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Probe for EPMA v. 12.8.9

# User's Guide and Reference

*Xtreme Edition*

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# Introduction



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## Description

Probe for EPMA Xtreme Edition is a complete acquisition, automation and analysis package for WDS (wavelength dispersive spectrometry) EPMA (Electron Probe Micro-Analysis) running under the Windows OS on the Intel CPU platform. Probe for EPMA can acquire, automate and analyze x-ray intensities on JEOL 8800/8900/8100/8200/8500/8230 and 8530 and Cameca SX100/SXFive microprobes with WDS/EDS spectrometers.

One can also run Probe for EPMA on the Mac OS using the Parallels virtual machine on a compatible Mac computer or laptop.

*The Xtreme Edition includes all the function and features of the original Probe for EPMA and many new additional features and enhancements including support for integrated imaging, analysis of beta lines, duplicate quantitative elements and support for analysis of elements up to atomic number 100.*

Probe for EPMA Xtreme can also be run off-line to simply re-process previously acquired x-ray intensity data. Because Probe for EPMA stores all standard, unknown and wavescan sample data and retains all experimental conditions in a single Microsoft Access relational database file (Access MDB file), it is extremely simple to transfer Probe for EPMA Xtreme intensity data to another computer for off-line reprocessing with another copy of Probe for EPMA Xtreme running in "demonstration" mode.

Probe for EPMA is written primarily in Visual Basic 6.0 (Professional) and utilizes several external DLLs (Dynamic Link Libraries) for data access, automation and graphics written in C++ v. 9.0.

This Reference Manual is essentially a description of what each software dialog item or menu does and should be used in conjunction with the Getting Started Manual and Advanced Topics Manual written by Dan Kremser and Dave Adams which describes how to perform specific operations with the software.

This user's guide and reference manual was written by John J. Donovan and edited by John Fournelle, Dan Kremser and Karsten Goemann.

In addition, many suggestions, features and capabilities have been provided by all users of Probe for EPMA but I would like to especially thank Paul Carpenter for his tireless testing and many helpful discussions.

I thank everyone for the enormous contribution of time and energy they have devoted to this software project.

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## License

Probe for EPMA Xtreme has a flexible site license policy. You are allowed to use Probe for EPMA on only one (1) computer for automation and data acquisition on an electron microprobe or SEM. However you may install Probe for EPMA Xtreme on as many computers as you require for the processing of off-line data from microprobe or SEM.

Probe for EPMA Xtreme requires specialized hardware for operation under the real-time interfaces; however no special hardware is required for operation in "demonstration" mode. Probe for EPMA Xtreme provides full functionality for quantitative analysis and graphical or ASCII file output in "demonstration" mode.

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## Special Thanks

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## Technical Support

Technical support for Probe for EPMA is provided by Probe Software, Inc. Please contact John Donovan at (541) 343-3400 for any questions or difficulties you may have with Probe for EPMA.

If an error message or software bug is seen, please note the exact error message and record as many details of the incident as you can to help us in determining a solution to your problem. Suggestions regarding new features or improvements to Probe for EPMA are always welcome and every effort will be made to incorporate them in future releases. If you have an idea for a new feature, we would like to hear about it. Please write John Donovan at the address below or call anytime.

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## Analysis Concepts

One significant difference between Probe for EPMA and other microprobe analysis programs is that Probe for EPMA can acquire and analyze all samples, regardless of whether they are standard or unknown samples, in exactly the same manner. This begins with the data acquisition; Probe for EPMA can acquire a complete analysis for every standard sample, just as it would for an unknown sample. In addition all standard, unknown and wavescan (and digitized coordinate) data is stored together in each user's probe run database so that your calibrations cannot be overwritten by subsequent users. Of course there are several very flexible options for "quick" standardization in which the program will automatically determine which standards are required for acquisition based on standard, interference or background calibration assignments or based on a concentration criterion in the standard.

The concept is much like a laboratory notebook where you have your private lab notes, which are protected from casual or accidental modification. Of course each user can import any standard calibration or sample setup from any run for their own use at any time.

### Standard and Unknowns Easily Run Under Identical Conditions

For example, if in a particular run you are analyzing for Si, Al, Na and K, then by default, Probe for EPMA can acquire data for all four elements on each standard. This means that even if a standard is only used to calibrate Si, the x-ray count data for the other three elements, Al, Na, and K can also be acquired on that standard. Measuring all samples in the same manner under the same conditions can eliminate errors that may be introduced in the analysis from beam and sample interactions, especially when beam charge buildup, carbon buildup or Time Dependent Intensity (TDI) element effects are present in the samples.

Although the time needed to acquire standard data is not any longer for this method when the analysis is measuring only one element on each spectrometer, it is true that acquiring more than one element on a spectrometer will increase the time required for standard calibration. This might be considered a small price to pay for the increased accuracy and flexibility of measuring both standards and unknown for the same elements. However, to save time, Probe for EPMA does allow the acquisition of "quick" standards for those standards used in the primary calibration. Note that acquisition of these "quick" standards will restrict the use of some of the unique analysis features discussed below.

### Easy Wavescan Acquisition and Graphical Off-Peaks Assignment

Probe for EPMA provides a unique wavescan acquisition and off-peak assignment capability. With one mouse click one can acquire wavescans on and about the on-peak region for any and all current elements in a run. For example, if one is analyzing for 12 elements using a three spectrometer instrument, one mouse click will start the automatic acquisition of all elements and the data will be automatically saved.

Off-peak assignment is even easier. Simply select the element to display and use the mouse to place the high and low peak positions exactly where you want them on the wavescan plotted data for subsequent acquisitions.

### Easy and Flexible Standard Re-assignment Capability

Aside from the obvious value in keeping the acquisition conditions constant for all samples, having the complete data for each standard at hand, provides the analyst with several useful capabilities. Among these is a unique standard re-assignment feature. Since all elements in the run are acquired for all standards, any standard containing a particular element can be assigned as the calibration standard for that element at any time, even when processing off-line.

Another benefit to complete standard analysis is the ability to calculate each standard analysis as if it were an unknown. This means that every standard is also a secondary standard. In this way, one can easily determine the accuracy of each element in the run (and the overall accuracy of the run by noting the totals on each standard). If an element is present in a

standard but is not assigned as the calibration standard for that element, then the program will calculate the relative error in the analysis based on the assigned standard. In this way one can check for agreement between various standards in the run. If the element is not present in the standard, the analysis provides a check on the accuracy of the off-peak or MAN background correction and also as a check for possible spectral interferences.

## Automatic Interference Calibration

In fact, the quantitative interference correction in Probe for EPMA uses this additional data to calculate the effect of an interfering element on an interfered channel. Again, since all elements are acquired for each standard and the composition of each standard is of course already known, the count to the concentration ratio of the interfering element can be calculated. This in turn can be used in the ZAF composition iteration to quantitatively calculate the interference correction in an unknown sample (Donovan, et. al., 1993).

## Single Relational Database for Data Integrity

Another unique feature of Probe for EPMA is that all data for all samples including unknowns, standards and wavelength scans, along with all conditions and digitized coordinates are stored in a single relational database. This allows the analyst to easily transfer all essential raw data to another computer for off-line processing without having to track down numerous files scattered over the hard disk.

This also means that the wavescan feature for checking for off-peak interferences is intelligent, easy to use and can be started with a single mouse click- no more writing down off-peak positions on a piece of paper and typing them into another part of the program! To change an off-peak position simply click the new position with the mouse and the program will automatically remember it for the next standard or unknown acquisition.

Probe for EPMA Xtreme Edition is based on the philosophy that the analyst should have maximum control and flexibility. The software should open up possibilities, not restrict the ability to achieve good results. Following this, the terminology used in Probe for EPMA makes a distinction between *acquisition* of x-ray intensities, and the processing of that data or the *analysis* as it is called here. By breaking what is traditionally called "doing an analysis" into two distinct steps of first acquisition, then analysis (which are 2 of the 4 main divisions of the program -- Acquire!, Analyze!, Automate! and Plot!), unlimited options are available for post processing of data.

## History of Probe for EPMA

The origins of Probe for EPMA lie in the distant past of computer history when 64K was a large amount of memory and a hard disk was a luxury (8 inch floppies were the norm at the time). In 1979, Mark Rivers (now at Argonne National Laboratory running the synchrotron microprobe beam line) bought a PDP-11 clone computer kit, a DEC RT-11 operating system and a FORTRAN IV DEC compiler and connected it to the UC Berkeley ARL SEMQ microprobe that had been using an IBM card puncher to record intensity data. Prior to the PDP-11 computer interfacing by Mark, these punch cards had to be carried by hand over to the central computer center and submitted (along with the program punch cards) as an overnight job for processing using Bence-Albee matrix corrections. One had to wait 12 hours or so to find out if one had analyzed epoxy.

After Mark's pioneering programming efforts (called PRMAIN), UCB had one of the first microcomputers ever interfaced to an electron microprobe. But more importantly from the programmers point of view was that Mark implemented many novel methods for acquisition and analysis that were far ahead of his time, including direct access binary file I/O, automatic standard drift corrections, automatic beam drift corrections, linear Bence-Albee corrections (using two coefficients that provided a significant improvement over the single coefficient factors used by other laboratories at that time) and a hierarchical sample and data structure that allowed for easy access to large data sets each with its own dynamically calculated average and standard deviation.

A later version called PRSUPR was modified by John Donovan and provided for automation of the stage and spectrometers (previously the spectrometers were turned using hand cranks). This was all integrated in a single vertical application that overcame the 64K RAM limit of the PDP-11 by using the dreaded "disk overlay" feature which was essentially a primitive virtual memory function. This was not for the faint hearted because on RT-11 the disk overlays

were not interrupt driven and so one could destroy one's return path (code-wise) by calling the wrong function at the wrong time. Up to 8 levels of disk overlay were utilized in this early version of Probe.

This PDP-11 FORTRAN code was eventually ported by John Donovan in 1986 to an IBM PC using Microsoft FORTRAN which also included a translation and modification of John Armstrong's CITZAF (ZAF and Phi-Rho-Z) code into FORTRAN and called Probe or Probe for DOS. After the 64K RAM limits of the PDP-11, even 640K seemed like an enormous amount of memory space.

Subsequently the program was converted to Visual Basic 3 by John Donovan in his spare time (weekends and a lot of burned midnight oil) as a 16 bit Windows application and finally into a 32 bit Windows application using Visual Basic 5 and called Probe for Windows. Subsequently it was modified to include larger element arrays (up to element 100), EDS and integrated imaging support and called Probe for Windows Enterprise Edition. The latest iteration, now called Probe for EPMA Xtreme Edition, is available from Probe Software for modern TCP/IP based EPMA instruments. The software incorporates a simply enormous feature set as this manual demonstrates.

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# Installation

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## Platform Requirements

Probe for EPMA requires a fairly fast computer platform for reasonable calculation and data access times. The following system requirements are the minimum and preferred hardware that can be used to run Probe for EPMA Xtreme.

### Minimum

- Windows XP Professional (32 bit only)
- 160 GB hard disk with at least 500 MB free disk space
- 2 GB RAMm 2 GB video RAM
- 24" color monitor (1920 x 1200 x 64K colors)
- two network connections

### Preferred

- Windows 7, 8 or 10 (32 or 64 bit)
- 500 GB hard disk with at least 200 GB free disk space
- 8 GB RAM, 6 GB video RAM
- 3 network connections
- 30" color monitor

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## Installation

Be sure that you have at least 2 GB of free disk space on the installation hard drive. The Probe for EPMA installation requires less than this, but the run time files require additional space for the creation of data and table files.

If attempting to perform a "clean" installation, be sure to remove any existing copies of Probe for EPMA by running the Add/Remove Software applet from the Windows Control Panel.

Probe for EPMA will only replace the executable files in the **C:\Probe Software\Probe for EPMA** application folder and certain internal data files in the **C:\ProgramData\Probe Software\Probe for EPMA** program data folder. Always backup your user/site specific files: STANDARD.MDB, PROBEWIN.INI, MOTORS.DAT, SCALERS.DAT, ELEMENTS.DAT, DETECTORS.DAT, CRYSTALS.DAT and any other program data files that you have created or modified since the original installation.

## Installation Using Probe for EPMA.msi and CalcZAF.msi

Double-click on CalcZAF.msi to install the base files, then double-click on ProbeForEPMA.msi to perform the installation. Note that both CalcZAF.msi and ProbeForEPMA.msi must be installed to obtain a complete installation. Subsequent updates can be performed using the Help menu to download and re-run the ProbeForEPMA.msi file.

The installer will confirm the installation when it is complete. If any error messages are observed during the installation, please note the exact error message text and call or e-mail for technical support.

## Files Used by Probe for EPMA

The following files are installed by the Probe for EPMA Setup program. The following section details where they are ultimately installed and what they are used for.

### Files that are installed to the Probe for EPMA application directory (usually C:\Probe Software\Probe for EPMA) :

|                                |   |
|--------------------------------|---|
| STANDARD.EXE                   | ' Standard database application   |
| PROBEWIN.EXE                   | ' Probe for EPMA main application   |
| PROBEWIN.CHM                   | ' Probe for EPMA help file (Win7)   |
| EVALUATE.EXE                   | ' Application utility for evaluation of standard compositions (for John Fournelle)                    |
| STARTWIN.EXE                   | ' Start utility application   |
| USERWIN.EXE                    | ' User database application   |
| STAGE.EXE                      | ' Stage utility application   |
| COAT.EXE                       | ' Coating utility applet  |
| CALCZAF.EXE                    | ' ZAF/Phi-Rho-Z calculation application   |
| DRIFT.EXE                      | ' Standard intensity drift utility  |
| STRIPCHART.EXE                 | ' Application utility that simulates a strip chart recorder digitally                                 |
| CALMAC.EXE                     | ' Utility to calculate mass absorption coefficients for x-ray emitters and arbitrary ranges of energy |
| SEARCH.EXE                     | ' Utility to search for probe database files based on a number of user defined criteria               |
| FARADAY.EXE                    | ' Application utility for controlling the faraday cup   |
| TESTFID.EXE                    | ' Fiducial transformation program (for conversion to/from other instrument stage coordinates)         |
| MONITOR.EXE                    | ' System monitor utility (JEOL only)  |
| VACUUM.EXE                     | ' Vacuum status utility   |
| CALIBRATE.EXE                  | ' Instrument calibration utility (JEOL only)  |
| GUNALIGN.EXE                   | ' Gun alignment utility (JEOL/SX100/SXFive only)  |
| PROBEUSERWIZARD.EXE            | ' Probe for EPMA "Wizard" utility   |
| PROBEWIN.PDF                   | ' Pdf version of this Reference Manual  |
| PROBE FOR EPMA_QUICK START.PDF | ' Quick Start manual  |
| GRAPHPPR.HLP                   | ' Plot graphics window help file  |
| PROBEHELP.INI                  | ' Context sensitive help INI file   |
| EQUATION4.RTF                  | ' RTF interference correction equations for debugging   |
| EQUATION4A.RTF                 | ' RTF interference correction equations for debugging   |
| EQUATION4B.RTF                 | ' RTF interference correction equations for debugging   |
| EQUATION8.RTF                  | ' RTF interference correction equations for debugging   |

### Files installed to the Windows SYSTEM32 sub directory (usually C:\Winnt\System32 for WinNT/2000/XP/Vista-32bit/Win7-32bit or Windows\SysWOW64 for Windows 7 or 8 64 bit) :

|              |                               |
|--------------|-------------------------------|
| CSFTPCTL.OCX | ' Secure socket FTP utility   |
| CSHTPCTL.OCX | ' Secure socket HTTP utility  |
| CSMSGCTL.OCX | ' Secure socket EMAIL utility |
| CSMTPCTL.OCX | ' Secure socket SMTP utility  |
| CSTCPCTL.OCX | ' Secure socket TCP utility   |

|                 |  |
|-----------------|--|
| DAO350.DLL      | ' Microsoft 3.5 DAO  |
| DAO360.DLL      | ' Microsoft 3.6 DAO  |
| GRAPH32.OCX     | ' Graphics Server graph control                                |
| GRAPHPR.HLP     | ' Graphics Server support file                                 |
| GSJPG32.DLL     | ' Graphics Server support file                                 |
| GSW32.EXE       | ' Graphics Server support file                                 |
| GSWAG32.DLL     | ' Graphics Server support file                                 |
| GSDLL32.DLL     | ' Graphics Server support file                                 |
| MSCOMM32.OCX    | ' Microsoft Communication control                              |
| MSVBVM50.dll    | ' Microsoft VB5 support file                                   |
| Psi_utility.dll | ' Probe Software utility DLL                                   |
| Richtx32.ocx    | ' Microsoft rich text control                                  |
| VB5DB.dll       | ' Microsoft 3.6 DAO support file                               |
| WSATRACE.DLL    | ' Secure socket trace utility (for debugging network problems) |

**Files that are installed to the Probe for EPMA program data directory (usually C:\ProgramData\Probe Software\Probe for EPMA) :**

|              |  |
|--------------|--|
| PROBEWIN.INI | ' JEOL INI file (sample)   |
| MOTORS.DAT   | ' JEOL MOTOR file (sample)   |
| SCALERS.DAT  | ' JEOL SCALER file (sample)  |
| CRYSTALS.DAT | ' Crystal data (defined spectrometer crystals)                                     |
| ELEMENTS.DAT | ' Element data   |
| STANDARD.MDB | ' Default standard composition database  |
| VERSION.TXT  | ' ASCII file of version changes  |
| EMPAPF.DAT   | ' Empirical APF factors ASCII file   |
| EMPMAC.DAT   | ' Empirical MACs ASCII file  |
| EMPFAC.DAT   | ' Empirical alpha-factors ASCII file   |
| EMPPHA.DAT   | ' Empirical PHA ASCII file   |
| XEDGE.DAT    | ' X-ray absorption edge data binary file   |
| XFLUR.DAT    | ' X-ray fluorescence yield data binary file  |
| XLINE.DAT    | ' X-ray emission line data binary file   |
| ABSORB.DAT   | ' McMaster MAC coefficients ASCII file   |
| CITZMU.DAT   | ' Cal-Tech (Heinrich) MACs data binary file  |
| LINEMU.DAT   | ' Henke/Cal Tech MACs data binary file   |
| MCMASTER.DAT | ' McMaster MACs data binary file   |
| MAC30.DAT    | ' Caltech MACs data binary file  |
| XRAY.MDB     | ' NIST x-ray database ASCII file   |
| TECH1.DOC    | ' Technical document (Word 97)   |
| TECH2.DOC    | ' Technical document (Word 97)   |
| TECH3.DOC    | ' Technical document (Word 97)   |
| GRIDBB.BAS   | ' Partial OLE script used for creating new scripts (Surfer 6)                      |
| GRIDCC.BAS   | ' Partial OLE script used for creating new scripts (Surfer 7)                      |
| TEST3D.DAT   | ' Sample polygon for polygon grid  |
| LINES2.DAT   | ' Line and edge energies used for creating MAC30.DAT                               |
| DEMO1.BMP    | ' Demo image for Image Digitize feature  |
| MACMATK.DAT  | ' Text file of K mass absorption coefficients from Armstrong (used for LINEMU.DAT) |
| MACMATL.DAT  | ' Text file of L mass absorption coefficients from Armstrong (used for LINEMU.DAT) |
| MACMATM.DAT  | ' Text file of M mass absorption coefficients from Armstrong (used for LINEMU.DAT) |
| MODAL.DAT    | ' Sample input file for modal analysis feature                                     |
| COAT.DAT     | ' Sample input file for Coat.exe   |
| LINES.DAT    | ' Text file from Armstrong for creating XLINE.DAT file                             |
| MACJTA.DAT   | ' John Armstrong MAC file (binary format)  |
| BLUERED.FC   | ' False color palette file   |

|                                |   |
|--------------------------------|---|
| RAINBOW.FC                     | ' False color palette file  |
| THERMAL.FC                     | ' False color palette file  |
| 0395_MAGNETITE.BMP             | ' Demonstration standard bitmap file                                      |
| 0012_MGO.BMP                   | ' Demonstration standard bitmap file                                      |
| 0023_V2O3.BMP                  | ' Demonstration standard bitmap file                                      |
| 0140CALCITE.BMP                | ' Demonstration standard bitmap file                                      |
| 0298_VG2 GLASS.BMP             | ' Demonstration standard bitmap file                                      |
| HOLDERCIW6.WMF                 | ' Demonstration JEOL stage vector file                                    |
| HOLDERCIW4A.WMF                | ' Demonstration JEOL stage vector file                                    |
| CALTECH1.5.WMF                 | ' Demonstration standard bitmap file                                      |
| NISTBIN2.DAT                   | ' NIST binary k-ratio file for testing matrix correction routines         |
| NISTBIN3.DAT                   | ' NIST binary k-ratio file for testing matrix correction routines         |
| CHANTLER2005-KA.DAT            | ' Input files for creating FFAST MAC file                                 |
| CHANTLER2005-KB.DAT            | ' Input files for creating FFAST MAC file                                 |
| CHANTLER2005-LA.DAT            | ' Input files for creating FFAST MAC file                                 |
| CHANTLER2005-LB.DAT            | ' Input files for creating FFAST MAC file                                 |
| CHANTLER2005-MA.DAT            | ' Input files for creating FFAST MAC file                                 |
| CHANTLER2005-MB.DAT            | ' Input files for creating FFAST MAC file                                 |
| FFAST.DAT                      | ' FFAST MAC file (NIST, 2005)   |
| DHZ.MDB                        | ' DHZ composition database (for matching and searching)                   |
| AMCSRD.MDB                     | ' American Mineralogist composition database (for matching and searching) |
| ALL_WEIGHTS-MODIFIED.TXT       | ' American Mineralogist mineral composition input file                    |
| AUAGCU.DAT                     | ' Binary k-ratio test file (NIST, 1979, 52.5 degrees)                     |
| AUCU_NBS-K-RATIOS.DAT          | ' CalcZAF input format k-ratio test file (NIST, 1979, 52.5 degrees)       |
| OLIVINE PARTICLE-JTA-1.0UM.DAT | ' CalcZAF input format particle test data file                            |
| OLIVINE PARTICLE-JTA-0.5UM.DAT | ' CalcZAF input format particle test data file                            |
| PKZIP25.EXE                    | ' Unzip utility   |
| DEADTIME_CALC.DOC              | ' Deadtime spreadsheet documentation (Paul Carpenter)                     |
| DEADTIME_CALC.XLS              | ' Deadtime spreadsheet (Paul Carpenter)                                   |

#### **NIST MQ executable (Probe for EPMA program data directory)**

|            |   |
|------------|---|
| MQ.EXE     | ' NIST Monte Carlo software executable      |
| EDGES.TSR  | ' NIST Monte Carlo line table               |
| MCARLO.BAT | ' NIST Monte Carlo batch file for input     |
| MCARLO.INP | ' NIST Monte Carlo sample input file        |
| CDONLY.DAT | ' NIST Monte Carlo data file for MCARLO.BAT |

#### **Files that are installed to the Probe for EPMA program data directory but also copied to the Userdata directory (usually C:\UserData) :**

|             |   |
|-------------|---|
| DHZ.DAT     | ' standard compositions from DHZ (1st Ed.)              |
| ORE.DAT     | ' standard compositions from Dana's                     |
| SRM.DAT     | ' standard compositions from NIST SRMs                  |
| CALCZAF.DAT | ' ZAF/Phi-Rho-Z sample data                             |
| GRIDMIN.DAT | ' sample boundary data for demonstration                |
| JEOLOX.MDB  | ' <b>Probe for EPMA</b> sample data (oxide)             |
| JEOLEL.MDB  | ' <b>Probe for EPMA</b> sample data (elemental)         |
| AUAGCU2.DAT | ' CalcZAF binary intensity data file                    |
| NISTBIN.DAT | ' CalcZAF binary intensity data file                    |
| POUCHOU.DAT | ' CalcZAF binary intensity data file                    |
| XYSCAN.BAS  | ' sample Surfer script (run from GSScripter) (Surfer 6) |
| XYSCAN.BLN  | ' sample Surfer boundary file (Surfer 6)                |

|                |   |
|----------------|---|
| XYSCAN.DAT     | ' sample Surfer data file (Surfer 6)  |
| XYSCAN2.BAS    | ' sample Surfer script (run from GSScripter) (Surfer 7 or 8 or 9)               |
| XYSCAN2.BLN    | ' sample Surfer boundary file (Surfer 7 or 8 or 9)                              |
| XYSCAN2.DAT    | ' sample Surfer data file (Surfer 7 or 8 or 9)                                  |
| NISTBINA20.DAT | ' Sample binary calculation input for CALCZAF                                   |
| NISTBINZ10.DAT | ' Sample binary calculation input for CALCZAF                                   |
| POUCHOUA20.DAT | ' Sample binary calculation input for CALCZAF                                   |
| POUCHOUZ10.DAT | ' Sample binary calculation input for CALCZAF                                   |
| CALCBIN.DAT    | ' Sample calculation input for CALCZAF  |
| DEMO2.GIF      | ' Demonstration "PictureSnap" image file  |
| DEMO2.JPG      | ' Demonstration "PictureSnap" image file  |
| DEMO2.ACQ      | ' Demonstration "PictureSnap" image file  |
| DEMO2.BMP      | ' Demonstration "PictureSnap" image file  |
| CALCZAF2.DAT   | ' CalcZAF standard composition variance test data (see CalcZAF Analytical menu) |
| DHZ.MDB        | ' DHZ composition database (for matching and searching)                         |
| AMCS.DMDB      | ' American Mineralogist composition database (for matching and searching)       |

**Files that are created by Probe for EPMA:**

|              |   |
|--------------|---|
| POSITION.MDB | ' position database                                     |
| SETUP.MDB    | ' element setup database                                |
| USER.MDB     | ' user database   |
| STANDARD.DAT | ' standard composition import/export ASCII file         |
| SETUP.DAT    | ' element setup import/export ASCII file                |
| USER.DAT     | ' user database import or export ASCII file             |
| *.POS        | ' position import or export ASCII files                 |
| *.OUT        | ' Log window ASCII files                                |
| DETECTOR.DAT | ' Configuration file for specifying detector parameters |
| USERMAC.DAT  | ' User Defined MAC table for user specified values      |

## Running Probe for EPMA for the First Time

Simply double-click the Probe for EPMA icon to allow the program to configure these and any other necessary directories and files that it will need for analytical calculations and make sure that all of the analytical files are installed. Several sample data files are supplied to test the default installation.

Once this step is completed, the configuration of the hardware specific configuration files (PROBEWIN.INI, MOTORS.DAT and SCALERS.DAT) may be performed at this time using a text editor such as NotePad or TextPad. See below, under the Configuration Files section.

The JEOL 8900/8200/8500/8x30 and Cameca SX100/SXFive interfaces require a network connection with TCP/IP transport. Contact Probe Software for help with these modifications if necessary.

## Other Programs Supplied With Probe for EPMA

Several very useful utilities are provided with Probe for EPMA that are sometimes a subset of the complete Probe for EPMA application but also provide unique features. These utilities and a brief description are listed here (full details are provided in additional sections of this document):

|                  |  |
|------------------|--|
| <b>CalcImage</b> | <b>Quantitative Imaging (requires ProbeImage acquisition package)</b>              |
| <b>CalcZAF</b>   | <b>Standalone ZAF with electron/x-ray range and detection modeling</b>             |
| <b>Calibrate</b> | <b>JEOL (8200/8900/8500 only) spectrometer and stage limit calibration utility</b> |
| <b>CalMAC</b>    | <b>Mass absorption coefficient calculations</b>                                    |
| <b>Coat</b>      | <b>Calculates evaporation coating thickness from wire diameter and length</b>      |

|                   |  |
|-------------------|--|
| <b>Drift</b>      | <b>Search all files for standard intensities for a specified condition</b>                   |
| <b>Evaluate</b>   | <b>Plot k-ratios or intensities versus composition of standards</b>                          |
| <b>Faraday</b>    | <b>Faraday cup and beam utility</b>  |
| <b>GunAlign</b>   | <b>JEOL (8200/8900/8500 only) and Cameca (SX100/SXFive) Gun alignment utility</b>            |
| <b>Monitor</b>    | <b>JEOL (8900/8200/8500 only and Cameca (SX100/SXFive) instrument monitor utility</b>        |
| <b>Remote</b>     | <b>Automation utility for Excel to instrument interface, optional</b>                        |
| <b>Search</b>     | <b>Utility to search for text or samples within a probe database file</b>                    |
| <b>Stage</b>      | <b>General stage and digitizing utility</b>  |
| <b>StartWin</b>   | <b>General spectrometer and counting utility</b>   |
| <b>StripChart</b> | <b>Digital stripchart recorder</b>   |
| <b>TestEDS</b>    | <b>EDS interface test utility for Demo, Bruker or Thermo EDS</b>                             |
| <b>TestFid</b>    | <b>Standalone fiducial conversion utility for stage import/export from other instruments</b> |
| <b>TestType</b>   | <b>Test utility for interface functions (available upon request)</b>                         |
| <b>UserWin</b>    | <b>User hourly usage utility</b>   |
| <b>Vacuum</b>     | <b>JEOL (8200/8900/SXFive) and Cameca (SX100/SXFive) vacuum utility</b>                      |

## **Removing Probe for EPMA**

To properly uninstall Probe for EPMA Xtreme, it is highly recommended to use the Add/Remove Programs applet in the Control Panel. This will insure that any "shared" components are properly removed from the system. Some files created by the user may need to be removed manually after the uninstall. Please contact Probe Software if necessary for more information.

## **Copy Protection**

Probe for EPMA has no copy protection for off-line processing of data or for running the program in "demonstration" mode. Please see the License Agreement section above for details on how you may copy Probe for EPMA Xtreme.

However, to run Probe for EPMA under any real-time interface mode (See "InterfaceType" in the PROBEWIN.INI file) other than demonstration mode requires a registration code, only available from Probe Software.

The first time you run the software and make a connection to your microprobe the program will prompt you with a serial number and request a corresponding registration code. This registration code is available from Probe Software by supplying the displayed serial number. Once you obtain the registration code from Probe Software, simply enter it in the text field and you will never be prompted again.

If you get the error: "Error returned from DCC20AX\_MasterReset (error code = 273)" then either your special hardware is not properly configured or it is not present and cannot therefore be run in any mode other than the demonstration interface (or off-line reprocessing mode).

---

## **Configuration Files**

There are several configuration file used by Probe for EPMA and its companion programs. They are all editable ASCII files and may be edited (carefully!) by the user for customizing the microprobe configuration by using any text editor such as Windows NotePad. Please contact Probe Software for help with these procedures if necessary. Always remember to make copies of your original and current configuration files before making any changes to them. The following files are used by Probe for EPMA for configuration purposes and read in the order listed :

**PROBEWIN.INI**  
**CRYSTALS.DAT**  
**ELEMENTS.DAT**  
**MOTORS.DAT**  
**SCALERS.DAT**  
**DETECTORS.DAT**

The PROBEWIN.INI file and other Probe for EPMA configuration files will be in the **C:\ProgramData\Probe Software\Probe for EPMA** directory. These files will be automatically installed in the correct directories by the setup program.

## Constants for array declarations

Some configuration file parameter allowable ranges are defined using the following constant declarations.

```

' New integrated intensity background fit parameter (on each side of scan)
Global Const MAX_INTEGRATED_BGD_FIT% = 50
Global Const MAX_ENERGY_ARRAY_SIZE% = 10
Global Const MAX_THROUGHPUT_ARRAY_SIZE% = 20

' Based on Cameca SX100 set times
Global Const KILOVOLT_SET_TIME! = 6#
Global Const BEAMCURRENT_SET_TIME_CAMECA! = 10#
Global Const BEAMCURRENT_SET_TIME_JEOL! = 12#
Global Const BEAMSIZE_SET_TIME! = 2#
Global Const HYSTERESIS_SET_TIME! = 12#
Global Const COLUMNCONDITION_SET_TIME! = 25#
Global Const BEAMMODE_SET_TIME! = 1#

Global Const PENEPMATERIAL_MATERIAL_TIME# = 4#           ' in seconds to init Penepma for demo EDS (running
material.exe, per compositional element)                  ' in seconds to init Penepma for demo EDS (starting
Global Const PENEPMASTARTUP_STARTUP_TIME# = 15#          penepma.exe)
Global Const PENEPMAWDS_WDS_SYNTHESIS_TIME# = 20#         ' in seconds for demo WDS spectrum synthesis (per
analyzed element)                                         ' number of boundary points for cluster digitize

Global Const BOUNDARYNUMBEROFPOLYGOONS# = 100

' Special folders for system
Global Const SpecialFolder_CommonAppData = &H23           ' for all Windows users on this computer [Windows
2000 or later]                                         ' for the current Windows user (roaming), on any
Global Const SpecialFolder_AppData = &H1A                ' for the current Windows user (non roaming), on
computer on the network [Windows 98 or later]           ' the Documents folder for the current Windows user
Global Const SpecialFolder_LocalAppData = &H1C             this computer only [Windows 2000 or later]
Global Const SpecialFolder_Documents = &H5

' VB trappable errors
Global Const VB_OutOfMemory& = 7
Global Const VB_FileNotFound& = 53
Global Const VB_FileAlreadyOpen& = 55
Global Const VB_UnrecognizedDatabaseFormat& = 3343

' Global Const SW_HIDE& = 0
Global Const SW_SHOWNORMAL& = 1
' Global Const SW_SHOWMINIMIZED& = 2
' Global Const SW_SHOWMAXIMIZED& = 3
' Global Const SW_SHOWNOACTIVATE& = 4
' Global Const SW_SHOW& = 5
' Global Const SW_MINIMIZE& = 6
' Global Const SW_SHOWMINNOACTIVE& = 7
' Global Const SW_SHOWNA& = 8
' Global Const SW_RESTORE& = 9
' Global Const SW_SHOWDEFAULT& = 10

' New constants for non-traditional emission lines
Global Const MAXRAY_OLD% = 6                                ' maximum xray symbols (ka,kb,la,lb,ma,mb)
Global Const MAXRAY% = 13                                 ' maximum xray symbols (ka,kb,la,lb,ma,mb,ln,lg,lv,ll,mg,mz,
") including blank for non-analyzed

```

```

' Constants for array declarations
Global Const MAXINTERFACE% = 5          ' maximum number of instrument interfaces (0 to
MAXINTERFACE%)
Global Const MAXINTERFACE_EDS% = 6        ' maximum number of EDS spectra interfaces (0 to
MAXINTERFACE_EDS)
Global Const MAXINTERFACE_IMAGE% = 10      ' maximum number of imaging interfaces (0 to
MAXINTERFACE_IMAGE)
Global Const MAXINTERFACE_CL% = 4          ' maximum number of CL spectra interfaces (0 to
MAXINTERFACE_CL)

Global Const MAXCHAN% = 72
Global Const MAXCHAN1% = MAXCHAN% + 1
Global Const MAXSTD% = 132
Global Const MAXROW% = 500
Global Const MAXVOLATILE% = 400
per sample
Global Const MAXEDG% = 9
Global Const MAXELM% = 100
AbsorbGetMAC)
Global Const MAXEMP% = 20
Global Const MAXSAMPLE% = 19999
Global Const MAXINTF% = 6
Global Const MAXINDEX% = 10000
as of 2-27-2007)
Global Const MAXMAN% = 112
112 08-23-2017)
Global Const MAXSET% = 30
Global Const MAXCRYSTYPE% = 60
Global Const MAXCRY% = 6

Global Const MAXCOND% = 64
sample

Global Const MAXSPEC% = 6
Global Const MAXAXES% = 3
Global Const MAXMOT% = MAXSPEC% + MAXAXES%          ' maximum spectrometers per run (spectro 0 = EDS)
                                                    ' maximum stage axes (x, y, z)
                                                    ' maximum motors (spectrometer + stage)

Global Const MAXSPECTRA_CL% = 4096                  ' maximum number of CL channels

Global Const MAXDIM% = 3
Global Const MAXCOEFF% = 3
Global Const MAXCOEFF4% = 4
Global Const MAXCOEFF9% = 9
Global Const MAXBITMAP% = 12
Global Const MAXLINE% = 99999
Global Const MAXDET% = 12
Global Const MAXMULTI% = 12
background acquisition
Global Const MAXCATION% = 100
oxygens (0 to MAXCATION% - 1)
Global Const MAXCI% = 5
Global Const MAXPLOTPONTS% = 6000
Global Const MAXORDER% = 72
(sample().OrderNumbers)
Global Const MAXEND% = 4
Global Const MAXPHASCAN% = 256
code)
Global Const MAXBRAGG% = 9
Global Const MAXELEMENTSORTMETHODS% = 3            ' maximum analytical Bragg order
                                                    ' 0 to MAXELEMENTSORTMETHODS% (0 = none)

Global Const MAXCORRECTION% = 6
1,2,3,4 = alpha fits, 5 = calibration curve, 6 = fundamental parameters)
Global Const MAXBINARY% = 11
Global Const MAXDENSITY# = 30#
Global Const BLANKINGVALUE! = 1.70141E+38    ' Surfer blanking grid value
Global Const MAXPATHLENGTH% = 255
Global Const MINCPSPERNA! = 0.00000000001 ' default minimum bkgd count rate for P/B calculations
Global Const SIMULATION_ZERO! = 0.0000000001   ' zero intensity

```

```

Global Const MAXEMPFAC% = 100          ' maximum empirical alpha factors (from EMPFAC.DAT)
Global Const MAXCALIBRATE% = 5          ' maximum number of elements for multiple peak calibration
Global Const LIF2D! = 4.0267           ' 2d spacing of LIF crystal

Global Const SCROLLBARWIDTH% = 325      ' scroll bar width for sizing grid column widths (twips)
Global Const WINDOWBORDERWIDTH% = 175    ' window border width for sizing image width
Global Const FRAMEBORDERWIDTH% = 120     ' window border width for sizing controls
Global Const GRIDCOLUMNWIDTH% = 1100     ' width of a single grid column

Global Const PPMPERWTPERCENT# = 10000#   ' PPM per weight percent
Global Const DEFAULTDENSITY! = 5#         ' default density for sample (needs user input)

Global Const ANGPERMICRON& = 10000      ' angstroms per micron
Global Const NMPERMICRON& = 1000        ' nano-meters per micron
Global Const UMPERMICRON& = 1            ' micro-meters per micron
Global Const MMPERMICRON# = 0.001         ' milli-meters per micron
Global Const CMPERMICRON# = 0.0001        ' centi-meters per micron
Global Const MPERMICRON# = 0.000001       ' meters per micron
Global Const MICRONSPERCM& = 10000       ' microns per centimeter
Global Const MICRONSPERMM& = 1000        ' microns per millimeter

Global Const ANGPERNM& = 10             ' angstroms per nanometer
Global Const NMPERANG# = 0.1            ' nanometers per angstrom

Global Const CMPERANGSTROM# = 0.00000001 ' centi-meters per angstrom

Global Const MICROINCHESPERMICRON# = 39.37007874      ' micro-inches per micron
Global Const MILLIINCHESPERMICRON# = 0.03937007874     ' milli-inches per micron
Global Const INCHESPERMICRON# = 0.00003937           ' inches per micron

Global Const NAPA# = 1000000000#          ' nano-amps per amp
Global Const APNA# = 0.000000001         ' amps per nano-amps
Global Const PAPA# = 1000000000000#       ' pico-amps per amp
Global Const PAPERNA# = 1000#            ' pico-amps per nano-amp
Global Const NAPERMA# = 1000#            ' nano-amps per milli-amp

Global Const ANGKEV! = 12.39854          ' angstrom per KeV (and visa versa)
Global Const ANGEV! = 12398.54           ' angstrom per eV (and visa versa)
Global Const EVPERKEV# = 1000#          ' eV per keV
Global Const MILLIVOLTPERVOLT# = 1000#   ' millivolt per volt

Global Const MSPS! = 1000000#            ' micro-secs per second
Global Const TENTHMSECPERSEC# = 10000#   ' 1/10th millsecs per second
Global Const MSECPERSEC# = 1000#         ' milli-seconds per second
Global Const SECPERMIN# = 60#            ' seconds per min
Global Const SECPERHOUR# = 3600#         ' seconds per hour
Global Const SECPERDAY# = 86400#         ' seconds per day
Global Const HOURPERDAY# = 24#           ' hours per day

Global Const CPSPERKCPS# = 1000#         ' cps per kcps

Global Const PASCALSPERTORR# = 131.578   ' Pascals per Torr conversion
Global Const PASCALSPERMBAR# = 100#       ' Pascals per mBar conversion

Global Const MAXCOUNT& = 100000000      ' default maximum counts for statistics based counting
Global Const MAXMINIMUM! = 10000000000#   ' (reversed) for calculating minimums
Global Const MAXMAXIMUM! = -10000000000#  ' (reversed) for calculating maximums
Global Const MAXMINIMUM2& = 2147483647  ' (reversed) for calculating minimums
Global Const MAXMAXIMUM2& = -2147483648# ' (reversed) for calculating maximums
Global Const MAXMINIMUM3% = 32767        ' (reversed) for calculating minimums
Global Const MAXMAXIMUM3% = -32768        ' (reversed) for calculating maximums

Global Const MAXCRYSTAL2D_NOT_LDE! = 30# ' maximum 2d for non LDE crystal
Global Const MAXCRYSTAL2D_LARGE_LDE! = 100# ' maximum 2d for non large LDE crystal
Global Const ANALYTICALMAGTHRESHOLD# = 50000#   ' minimum mag for analytical warning

Global Const PI! = 3.14159              ' close enough!
Global Const PID# = 3.141592653        ' closer!
Global Const PIDD# = 3.14159265358979  ' even closer!

Global Const NATURALE# = 2.718281828   ' natural log constant

```

```

Global Const MAXLOGEXPD! = 709.6          ' maximum exponent for double precision natural log
(e^MAXLOGEXPD!)
Global Const MAXLOGEXPS! = 88.721         ' maximum exponent for single precision natural log
(e^MAXLOGEXPS!)

Global Const MININTEGER% = -32768          ' minimum integer value
Global Const MAXINTEGER% = 32767           ' maximum integer value
Global Const MINLONG& = -2147483648#       ' minimum long
Global Const MAXLONG& = 2147483647        ' maximum long
Global Const MINSINGLE! = -3.402823E+38    ' minimum single precision
Global Const MAXSINGLE! = 3.402823E+38      ' maximum single precision
Global Const MINDOUBLE# = -1.79769E+308     ' minimum double precision
Global Const MAXDOUBLE# = 1.79769E+308      ' maximum double precision

Global Const MAXOFFBGDTYPES% = 8            ' maximum off-peak background correction types (0 to 8, 0 =
default linear)
Global Const MAXMINTYPES% = 6                ' maximum mineral end-member types (including zero for none)
Global Const MAXZAF% = 10                  ' maximum number of ZAF correction options
Global Const MAXZAFCOR% = 8                 ' maximum number of stored ZAF corrections (analysis
structure)
Global Const MAXLIST% = 4999                ' maximum number of xray database listbox items
Global Const MAXMACTYPE% = 7                 ' maximum number of mass absorption files
Global Const MAXRELDEV% = 9999               ' maximum size for printout of % relative standard
deviations
Global Const MAXGRIDROWS% = 4096             ' maximum number of rows allowed in grid control

Global Const ROWLAND_JEOL# = 140#            ' default JEOL focal circle
Global Const ROWLAND_CAMECA# = 160#           ' default Cameca focal circle

Global Const MAXEDS% = 48                   ' maximum number of eds spectrum elements
Global Const MAXSPECTRA% = 8192              ' maximum number of eds spectrum channels
Global Const MAXSTROBE% = 128                ' maximum number of strobes (resolution calibrations)

Global Const BIT24& = 16777215            ' maximum 24 bit depth 0-16777215
Global Const BIT23& = 8388607             ' maximum 23 bit depth 0-8388607
Global Const BIT16& = 65535               ' maximum 16 bit depth 0-65535
Global Const BIT15& = 32767               ' maximum 15 bit depth 0-32767
Global Const BIT14& = 16383               ' maximum 14 bit depth 0-16383
Global Const BIT13& = 8191                ' maximum 13 bit depth 0-8191
Global Const BIT12& = 4095                ' maximum 12 bit depth 0-4095
Global Const BIT11& = 2047                ' maximum 11 bit depth 0-2047
Global Const BIT10& = 1023                ' maximum 10 bit depth 0-1023
Global Const BIT8& = 255                  ' maximum 8 bit color palette 0-255
Global Const BIT7& = 127                  ' maximum 7 bit depth 0-127
Global Const BIT6& = 63                   ' maximum 6 bit depth 0-63
Global Const BIT5& = 31                   ' maximum 5 bit depth 0-31
Global Const BIT4& = 15                   ' maximum 4 bit depth 0-15
Global Const BIT3& = 7                    ' maximum 3 bit depth 0-7
Global Const BIT2& = 3                    ' maximum 2 bit depth 0-3
Global Const BIT1& = 1                    ' maximum 1 bit depth 0-1

Global Const SIZE_65536_BYTES& = 65536   ' 65536 byte constant
Global Const SIZE_32768_BYTES& = 32768   ' 32768 byte constant
Global Const SIZE_16384_BYTES& = 16384   ' 16384 byte constant
Global Const SIZE_8192_BYTES& = 8192     ' 8192 byte constant
Global Const SIZE_4096_BYTES& = 4096     ' 4096 byte constant
Global Const SIZE_2048_BYTES& = 2048     ' 2048 byte constant
Global Const SIZE_1024_BYTES& = 1024     ' 1024 byte constant
Global Const SIZE_512_BYTES& = 512       ' 512 byte constant
Global Const SIZE_256_BYTES& = 256       ' 256 byte constant
Global Const SIZE_128_BYTES& = 128       ' 128 byte constant
Global Const SIZE_64_BYTES& = 64          ' 64 byte constant
Global Const SIZE_32_BYTES& = 32          ' 32 byte constant
Global Const SIZE_16_BYTES& = 16          ' 16 byte constant
Global Const SIZE_8_BYTES& = 8           ' 8 byte constant
Global Const SIZE_4_BYTES& = 4            ' 4 byte constant
Global Const SIZE_2_BYTES& = 2            ' 2 byte constant

Global Const IMAGESIZE128& = 128          ' image size 128 x 128 (96 @ 4/3)
Global Const IMAGESIZE256& = 256          ' image size 256 x 256 (192 @ 4/3)
Global Const IMAGESIZE512& = 512          ' image size 512 x 512 (384 @ 4/3)

```

```

Global Const IMAGESIZE1024& = 1024          ' image size 1024 x 1024 (768 @ 4/3)
Global Const IMAGESIZE2048& = 2048          ' image size 2048 x 2048 (1536 @ 4/3)

Global Const INT_ZERO% = 0                   ' for parameter passing
Global Const INT_ONE% = 1                   ' for parameter passing
Global Const INT_TWO% = 2                   ' for parameter passing

Global Const MAXMODELS% = 6                 ' maximum number of PTC geometric models
Global Const MAXDIAMS% = 6                 ' maximum number of PTC particle diameters
Global Const MAXMONITOR% = 4                ' maximum number of Monitor app lists
Global Const MAXMONITORLIST% = 13           ' maximum number of Monitor app list items
Global Const MAXSTDIMAGE% = 5               ' maximum number of standard images displayed per standard
number

Global Const MAXAUTOFOCUSPOINTS& = 1010    ' maximum autofocus data points (1010 + 14 = 1024 longs)
Global Const MAXAUTOFOCUSSCANS% = 3         ' maximum autofocus scans (fine, coarse, 2nd fine)
Global Const MAXIMAGES% = 2000              ' maximum number of sample images in run
Global Const MAXIMAGEIX% = 2048             ' maximum number of x pixels (SX100 = 2048, all others 1024)
Global Const MAXIMAGEIY% = 2048             ' maximum number of y pixels (SX100 = 2048, all others 1024)
Global Const MAXROMSCAN% = 1000             ' maximum points per ROM scan
Global Const MAXIMAGESIZES& = 4            ' dimensioned 0 to MAXIMAGESIZES&
Global Const MAXPALETTE% = 4                ' maximum number of color palettes

Global Const MINTAKEOFF! = 10#              ' minimum takeoff in degrees
Global Const MINKILOVOLTS! = 1#              ' minimum beam size in kilovolts
Global Const MINBEAMCURRENT! = 0.01          ' minimum beam current in nA
Global Const MINBEAMSIZE! = 0#               ' minimum beam size in microns

Global Const MAXTAKEOFF! = 75                ' maximum takeoff in degrees
Global Const MAXKILOVOLTS! = 100#            ' maximum beam size in kilovolts
Global Const MAXBEAMCURRENT! = 2000#          ' maximum beam current in nA
Global Const MAXBEAMSIZE! = 500#              ' maximum beam size in microns

Global Const MINREPLICATES% = 1              ' minimum number of replicate samples
Global Const MAXREPLICATES% = 500             ' maximum number of replicates samples

Global Const MAXBEAMCALIBRATIONS% = 32       ' maximum number of beam scan calibration array members

' Integrated intensity constants
Global Const MININTEGRATEDINTENSITYINITIALSTEP! = 800#
Global Const MAXINTEGRATEDINTENSITYINITIALSTEP! = 10#
Global Const MININTEGRATEDINTENSITYMINIMUMSTEP! = 10000#
Global Const MAXINTEGRATEDINTENSITYMINIMUMSTEP! = 20#
Global Const INTEGRATEDINTENSITYINITIALSTEP! = 100#
Global Const INTEGRATEDINTENSITYMINIMUMSTEP! = 400#

Global Const SCALERSDATLINESNEW% = 90        ' number of lines in new SCALERS.DAT file

Global Const MAXSCANS& = 99999               ' maximum number of PHA, bias, gain or peaking
scans
Global Const MAXAPERTURES% = 4                ' maximum number of apertures in system
Global Const SMALLAMOUNTFRACTION_HALF! = 0.0005 ' small amount fraction for motion limits (times
0.5)
Global Const SMALLAMOUNTFRACTION! = 0.001      ' small amount fraction for motion limits
Global Const SMALLAMOUNTFRACTIONx5! = 0.005     ' small amount fraction for motion limits (times
5)
Global Const SMALLAMOUNTFRACTIONx10! = 0.01     ' small amount fraction for motion limits (times
10)
Global Const MAXROMPEAKTYPES% = 6              ' maximum number of ROM scan peaking types
Global Const DEFAULTVOLATILEINTERVALS% = 5       ' default number of volatile intervals

Global Const JEOL_SPECTRO_JOG_SIZE# = 0.6      ' additional room for JEOL spectro low limit jog
(in mm)
Global Const JEOL_IMAGE_SHIFT_FACTOR! = 163.8    ' image_shift = um_shift * 163.8

Global Const MAXKLMORDER% = 20                 ' maximum number of KLM higher order lines to display
Global Const MAXKLMORDERCHAR% = 5              ' maximum number of KLM higher order Roman characters to
display
Global Const MAXSAMPLETYPES% = 3                ' maximumn types of samples (standard, unknown, wavescan)
Global Const MAXFORBIDDEN% = 20                 ' maximum number of forbidden elements

```

```

Global Const NOT_ANALYZED_VALUE_SINGLE! = 0.00000001      ' 10^-8
'Global Const NOT_ANALYZED_VALUE_DOUBLE# = 0.00000001      ' 10^-8      (do not utilize as it causes
a problem for Pro Essentials with .NullDataValueX property)

Global Const MAX_EXCEL_2003_COLS% = 256      ' maximum number of columns supported by Excel 2003 (version
11)

Global Const LOTSOFGRIDPOINTS% = 1000      ' lots of polygon points (hide FormAUTOMATE)
Global Const TOOMANYGRIDSTEPS% = 2000      ' too many grid steps
Global Const MAXTITLELENGTH% = 80          ' maximum graph title length
Global Const MAX_PATH% = 259              ' maximum file and path length for Dir$ command

Global Const FONT_REGULAR% = 0            ' regular format
Global Const FONT_BOLD% = 1              ' bold
Global Const FONT_ITALIC% = 2            ' italic
Global Const FONT_UNDERLINE% = 4          ' underline
Global Const FONT_STRIKETHRU% = 8          ' strikethru

```

## PROBEWIN.INI

The main configuration file used by Probe for EPMA is PROBEWIN.INI. This file is located in the Probe for EPMA program data (**usually C:\ProgramData\Probe Software\Probe for EPMA**). This is a windows initialization file used only by **Probe for EPMA** and its companion programs. A number of "shared" components are installed to the Windows system folder and they are automatically registered by the installation program. All single value parameters (e.g., the number of stage motors) used by **Probe for EPMA** and its companion programs are specified in this PROBEWIN.INI file.

The PROBEWIN.INI file has a standard Windows .INI file structure. That is, it consists of several distinct sections designated by a section name enclosed in square [ ] brackets. Each section then contains one or more distinct keywords to specify various single value probe configuration values. These values can be integer or string values. Strings need to be enclosed in double quotes if the string contains blanks. Real number values are read as strings and then converted to single or double precision real numbers. Boolean (true or false) values are read as integer values where 0 is false and any non-zero value is true.

The following section describes each section and the keyword definitions for each. The allowable range and the default value loaded if the keyword is not found in the file is given for each keyword.

### **[General]**

```

KiloVolts=15
TakeOff=40
BeamCurrent=30.
BeamSize=2
Magnification=400
MagnificationAnalytical=4000
MagnificationImaging=100
BeamMode=1           ; 0 = analog spot, 1 = analog scan, 2 = digital spot
Aperture =1          ; aperture 1 is default (JEOL only)
OxideOrElemental=2   ; 1 for oxide or 2 for elemental
PeakCenterMethod=1    ; 0 for interval, 1 for parabolic, 2 for ROM based
DebugMode=0
UserName="Probe User"
Title="Probe for EPMA Xtreme Demonstration Run for JEOL Hardware"
Description="By John Donovan and Probe Software, Inc."
FileViewer="NOTEPAD.EXE"
CustomLabel1="Department"
CustomLabel2="Account #"
CustomLabel3="Group"
CustomText1="Analytical Chemistry"

```

```
CustomText2=""  
CustomText3="Microscopy"  
SMTPServerAddress="smtp.probesoftware.com"  
SMTPAddressFrom="donovan@probesoftware.com"  
SMTPAddressTo="donovan@probesoftware.com"  
SMTPUserName="donovan"  
UseWavFileAfterAutomationString=""  
PeakCenterSkipPBCheck=0  
NominalBeam=1.0  
InstrumentAcknowledgementString=""  
BeamModeAfterAcquisition=0
```

#### **KiloVolts=15**

This keyword specifies the typical operating voltage of the microprobe. This value will be used as a default value for initialization. The default value can be changed at any time during the data acquisition process from the Acquire! window in **Probe for EPMA** or from the Analytical | Operating Conditions menu item in Standard. The allowed kilovolt range is 1 to 100 keV. The default value is 15 keV.

If the OperatingVoltagePresent flag is true then the software will attempt to set the operating voltage through the software interface when the first data acquisition is performed.

#### **TakeOff=40.0**

This keyword specifies the actual x-ray take-off angle of the microprobe. This value can be changed during the data acquisition process, but is generally not modified. However for modeling various experimental effects, this value can be changed from the Analytical | Operating Conditions menu item in Standard. Typically JEOL and Cameca = 40 and ARL = 52.5. The allowed takeoff range is 1 to 90 degrees. The default value is 40 degrees.

#### **BeamCurrent=30. ! SX-50/51/100 and JEOL 8900/8200 only**

This keyword is used to determine the default beam current in nano-amps. This parameter is used simply for documentation purposes unless the hardware interface supports beam current control as specified by the BeamCurrentPresent keyword in the [Hardware] section below. The allowed beam current range is 0.1 to 1000 nA. The default value is 30 nA.

#### **BeamSize=2 ! SX-50/51/100 and JEOL 8900/8200 only**

This keyword is used to determine the default beam size in microns. This parameter is used simply for documentation purposes unless the hardware interface supports beam size control as specified by the BeamSizePresent keyword in the [Hardware] section below. The allowed beam size range is 1 to 1000 u. The default value is 2 u.

#### **Magnification=400 ! SX-50/51/100 and JEOL 8900/8200 only**

This parameter is used to set the default magnification for general use. The default is 400 and the allowed range is 10 to 100000.

#### **MagnificationAnalytical=4000 ! SX-50/51/100 and JEOL 8900/8200 only**

This parameter is used to set the default magnification for analytical acquisition (standard, unknown and wescan samples). The default is 4000 and the allowed range is 10 to 100000.

#### **MagnificationImaging=100 ! SX-50/51/100 and JEOL 8900/8200 only**

This parameter is used to set the default magnification for image acquisition (Imaging and Digitize Image). The default is 100 and the allowed range is 10 to 100000.

#### **BeamMode=1**

This parameter sets the default beam mode for the program. A value of 0 forces an analog spot mode (for normal analysis), 1 for analog scan (for normal analysis at high magnification or analog imaging) and 2 for digital spot (only for digital imaging and not normally selected as the default beam mode). This value can be changed by the user in the Analytical Conditions dialog.

**Aperture =1 ; aperture 1 is default (JEOL only)**

The keyword defines the default aperture. Note that this value is only used by the JEOL 8900/8200/8500 interface for setting the beam current. See [hardware] section JeolCondenserCalibrationSetting keywords. The default is aperture 1 and the allowable range is 1 to 4.

**OxideOrElemental=2**

This keyword is used to determine the default option for oxide calculations (oxygen calculated by stoichiometry based on the element cation ratios for each sample) for starting the first sample of a new **Probe for EPMA** run. It can be changed at any time during the acquisition or analysis process by the user. For default elemental analysis enter 2, for default oxide analysis, enter 1. The default value is 2 for elemental analysis.

**PeakCenterMethod=1**

This keyword is used to determine the default peak center method used by the program. The valid values are :

- 0 for interval halving
- 1 for parabolic peak fit
- 2 for ROM based peak center      ! **SX100/SXFIVE, JEOL 8900/8200/8500/8x30 only**

The default is the parabolic peak fit. Note that JEOL 8900/8200 probes have options for various ROM peaking methods including parabolic ROM, Brent's Maxima ROM and Gaussian ROM in addition to two Dual ROM methods (Maxima for LiF crystals and Parabolic for other crystals and Maxima for LiF and Gaussian for other crystals plus a new Highest Intensity option (using smoothed intensities).

To increase the default ROM scan width decrease the Peakscan Size (line 16) or increase the LiF Peaking Start Size (line 19) in the SCALERS.DAT file

Note that within the application, the ROM based peak scans are based on the Peaking Count Time divided by 4 and the number of Peak Scan Points. The ROM scan width is determined by the Peaking Start Size.

**DebugMode=0**

This keyword specifies if the program is to start up in the so called "debug" mode. This option may be useful for certain trouble-shooting situations. The **Probe for EPMA** program will always save a user data file with DebugMode turned off. Change this parameter in the PROBEWIN.INI to any non-zero value to force the program to start up in "debug" mode for trouble-shooting purposes. The default is 0 to not start in debug mode.

**UserName="Probe User"**

This keyword specifies the default user name for the file information table in the various **Probe for EPMA** database files. It may be modified for a maximum character length of 64 characters. The default value is "User Name".

**Title="Probe for EPMA Demonstration Run"**

This keyword specifies the default run title for the file information table in the various **Probe for EPMA** database files. It may be modified for a maximum character length of 64 characters. The default value is "File Title".

**Description="By John Donovan and Probe Software, Inc."**

This keyword specifies the default file description for the file information table in the various **Probe for EPMA** database files. It may be modified for a maximum character length of 64 characters. The default value is "File Description".

**FileViewer="NOTEPAD.EXE"**

This keyword is used to determine the default file viewer that is to be run to view the disk log file. The default file viewer is Windows NotePad. Note, that a more robust file viewer such as TextPad may be substituted instead. TextPad is a shareware text editing program that is available any many FTP sites. Please contact **Probe Software** for help in obtaining TextPad.

**CustomLabel1="Department"****CustomLabel2="Account #"****CustomLabel3="Group"**

This keyword is used to define the first custom database field label in the user database application. These fields are also visible in the File Information window for each **Probe for EPMA** application database. The defaults are "Department", "Account #" and "Group".

**CustomText1="Analytical Chemistry"**

**CustomText2=""**

**CustomText3="Microscopy"**

This keyword is used to define the defaults for first, second and third custom database fields in the user database application. These fields are also visible in the File Info window for each **Probe for EPMA** application database. The defaults are blank.

**SMTPServerAddress="smtp.probesoftware.com"**

**SMTPAddressFrom="donovan@probesoftware.com"**

**SMTPAddressTo="donovan@probesoftware.com"**

**SMTPUserName="donovan"**

These keywords are to be used for e-mail notification of real-time automation errors. If these SMTP fields are valid and the option "E-mail Notification of Errors" is checked in the Automation Options dialog, then any real-time errors (for example, a blown filament message) will be trapped and forwarded to the address indicated. This option is only available for automation procedures initiated from the Automate! window.

SMTPServerAddress: must be a secure email server

SMTPAddressFrom: only used for documentation purposes

In addition, a "progress report" message will be sent every 8 hours to the e-mail address specified in order to give positive notification of the normal functioning of the instrument during especially long runs.

Note that the first time the automated acquisition is started and the E-mail Notification of Errors option is checked then the program will prompt the user for the password for the specified user email account. This password will be retained only for the duration of the currently open probe run.

**UseWavFileAfterAutomationString=""**

This parameter specifies a .WAV audio file to be played after automation in ProbeWin or Stage has been completed. If the .WAV file is located in the application folder or a folder in the system path then the filename only can be specified. If the file is located in another folder, the complete path must be designated.

**PeakCenterSkipPBCheck=0**

This parameter sets the default value for the "Skip P/B Check Before Peaking" option in the Peaking dialog. If this option is non-zero the program will automatically check that box when the dialog is loaded. Until it is unchecked by the user.

**NominalBeam=1.0**

This keyword specifies the beam current normalization constant. The x-ray intensities will be displayed normalized to this value. For example, if the nominal beam current is set to 20 nA, then all intensities will be displayed as cps per 20 nA. If the nominal beam current is 1.0 then the x-ray intensities will be displayed as cps per nA.

The default nominal beam current is equal to the default beam current unless the value is explicitly defined in the PROBEWIN.INI file.

**InstrumentAcknowledgementString=""**

This keyword is utilized in the Report button in the Analyze! window to acknowledge funding and or other institutional support. If this keyword is not empty, the program will print this string at the end of the report output to the log window for the user.

Generally this string is edited for something along the lines of "This instrument was supported by funding from NSF (EAR-0394756382) and NASA (NNX45409AF08G) and matching support from the University of California at Berkeley"

**BeamModeAfterAcquisition=0**

This is the beam mode the program sets after the (manual) acquisition is complete. 0 = spot, 1 = scan.

**[Software]**

```
LogWindowFontName="Courier New"
LogWindowFontSize=10
AcquirePositionFontSize=9
AcquireCountFontSize=10
LogWindowInterval=0.5
RealTimeInterval=0.2
AutomateConfirmDelay=10.0
EnterPositionsRelative=1
UpdatePeakWaveScanPositions=1
MaxMenuFileArray=4
ExtendedFormat=0
MACTypeFlag=1
PositionImportExportFileType=2
DeadtimeCorrectionType=1
AutoFocusStyle=1
AutoFocusInterval=5
BiasChangeDelay=2.0
UseEmpiricalPHADefaults=0
KilovoltChangeDelay=1.0
BeamCurrentChangeDelay=1.0
BeamSizeChangeDelay=0.5
LogWindowBufferSize=524288
PeakOnAssignedStandards=0
PrintAnalyzedAndSpecifiedOnSameLine=0
NoMotorPositionLimitsChecking=0
UserDataDirectory="C:\UserData"
ExtendedMenu=0
AutoAnalyze=0
FaradayAlwaysOnTop=0
ColumnConditionChangeDelay=5.0
SurferOutputVersionNumber=7
SelPrintStartDoc=1
UseMultiplePeakCalibrationOffset=0
UseWideROMPeakScanAlways=0
UseCurrentConditionsOnStartUp=1
UseCurrentConditionsAlways=0
DefaultVacuumUnitsType=0
DefaultCorrectionType=0
DefaultZAFType=1
PENDBASE_Path="C:\Userdata\Penepma12\Pendbase"
PENEPMa_Path="C:\Userdata\Penepma12\Penepma"
PENEPMa_Root="C:\Userdata\Penepma12"
PENEPMa_PAR_Path="C:\Userdata\Penepma12\Penfluor"
ForceNegativeKratiosToZero=0
AutoIncrementDelimiterString="_"
UseLastUnknownAsWavescanSetup=0

UserSpecifiedOutputSampleName=1
UserSpecifiedOutputLineNumber=1
UserSpecifiedOutputWeightPercent=1
```

UserSpecifiedOutputOxidePercent=0  
UserSpecifiedOutputAtomicPercent=1  
UserSpecifiedOutputTotal=0  
UserSpecifiedOutputDetectionLimits=0  
UserSpecifiedOutputPercentError=0  
UserSpecifiedOutputStageX=1  
UserSpecifiedOutputStageY=1  
UserSpecifiedOutputStageZ=1  
UserSpecifiedOutputRelativeDistance=0  
UserSpecifiedOutputOnPeakTime=0  
UserSpecifiedOutputHiPeakTime=0  
UserSpecifiedOutputLoPeakTime=0  
UserSpecifiedOutputOnPeakCounts=0  
UserSpecifiedOutputOffPeakCounts=0  
UserSpecifiedOutputNetPeakCounts=0  
UserSpecifiedOutputKraw=1  
UserSpecifiedOutputDateTime=1  
UserSpecifiedOutputKratio=0  
UserSpecifiedOutputZAF=0  
UserSpecifiedOutputMAC=0  
UserSpecifiedOutputStdAssigns=0  
UserSpecifiedOutputSampleNumber=1  
UserSpecifiedOutputSampleConditions=0  
UserSpecifiedOutputFormula=0  
UserSpecifiedOutputTotalPercent=0  
UserSpecifiedOutputTotalOxygen=0  
UserSpecifiedOutputTotalCations=0  
UserSpecifiedOutputCalculatedOxygen=0  
UserSpecifiedOutputExcessOxygen=0  
UserSpecifiedOutputZbar=0  
UserSpecifiedOutputAtomicWeight=0  
UserSpecifiedOutputOxygenFromHalogens=0  
UserSpecifiedOutputHalogenCorrectedOxygen=0  
UserSpecifiedOutputChargeBalance=0  
UserSpecifiedOutputFeCharge=0

UserSpecifiedOutputSpaceBefore=0  
UserSpecifiedOutputAverage=0  
UserSpecifiedOutputStandardDeviation=0  
UserSpecifiedOutputStandardError=0  
UserSpecifiedOutputMinimum=0  
UserSpecifiedOutputMaximum=0  
UserSpecifiedOutputSpaceAfter=0

UserSpecifiedOutputUnkIntfCorsFlag=0  
UserSpecifiedOutputUnkMANAbsCorsFlag=0  
UserSpecifiedOutputUnkAPFCorsFlag=0  
UserSpecifiedOutputUnkVolElCorsFlag=0  
UserSpecifiedOutputUnkVolElDevsFlag=0

UserSpecifiedOutputOxideMolePercentFlag=0

UserSpecifiedOutputStandardPublishedValuesFlag=0  
UserSpecifiedOutputStandardPercentVariancesFlag=0  
UserSpecifiedOutputStandardAlgebraicDifferencesFlag=0  
UserSpecifiedOutputRelativeLineNumber=1

```

UserSpecifiedOutputBeamCurrent=0
UserSpecifiedOutputAbsorbedCurrentFlag=0
UserSpecifiedOutputBeamCurrent2=0
UserSpecifiedOutputAbsorbedCurrent2Flag=0

NthPointAcquisitionInterval=10
GeologicalSortOrder=0      ; 0 = no sorting, 1 = traditional, 2 = low to high Z, 3 = high to low Z
DefaultLIFPeakWidth=0.08
ThermoNSSLocalRemoteMode=0
MonitorFontSize=10
JEOLSecurityNumber=0
UseCurrentBeamBlankStateOnStartUpAndTermination=0
ShowAllPeakingOptions=0
ForceSetPHAParametersFlag=0
CalcZAFDATFileDirectory= "C:\UserData\CalcZAFDATData"
ColumnPCCFileDirectory= "C:\UserData\ColumnPCCData"
SurferDataDirectory="C:\UserData\SurferData"
DemoImagesDirectory="C:\UserData\DemoImages"

DoNotRescaleKLM=0
UsePenepmaKratiosLimit=0
PenepmaKratiosLimitValue=90
PenepmaMinimumElectronEnergy=1.0
SurferPlotsPerPage=4      ; must be 1, 4 or 9

UserImagesDirectory="C:\UserImages"
UserEDSDirectory="C:\UserEDS"
UserCLDirectory="C:\UserCL"
UserEBSDDirectory="C:\UserEBSD"

GrapherAppDirectory=""
SurferAppDirectory=""
SurferPlotsPerPagePolygon=3 ; must be 1, 3 or 8
ProbeSoftwareInternetBrowseMethod=0
UseFluorescenceByBetaLinesFlag=1

StrataGEMVersion=6

MenuFlagArray[n]=""      ' do not edit

```

**LogWindowFontName="Courier New"**

Enter the default font for use in the log window. Generally a fixed spacing (not proportional) font such as Courier or New Courier should be used for best results. The font must be already be installed in Windows. The log window font can also be changed from the Output | Log Window Font menu item in all programs. The default font name is "Courier New".

**LogWindowFontSize=10**

Enter the default font point size for use in the log window. If the actual point size is not available, Windows will use the nearest available font size. The log window font can also be changed from the Output | Log Window Font menu item. The allowable range is 6 to 32. The default font size is 10.

**AcquirePositionFontSize=12**

Enter the font size for the Acquire Window motor position display. Generally use the largest font that will allow the motor positions to fill the display area without wrapping to the next line. The allowable range is 6 to 32. The default position font size is 12.

**AcquireCountFontSize=12**

Enter the font size for the Acquire Window time and counter display. Generally use the largest font that will allow the timer and counter values to fill the display area without wrapping to the next line. The allowable range is 6 to 32. The default count font size is 12.

**LogWindowInterval=0.5**

Enter the time interval in seconds for the log window buffer to be updated. The allowable range is 0.1 to 10 seconds. The default value is 0.5 seconds.

**RealTimeInterval=0.2**

Enter the time interval in seconds for the microprobe position and counter displays to be refreshed. The allowable range is 0.1 to 10 seconds. The default is 0.2 seconds.

**AutomateConfirmDelay=10.0**

Specifies the amount of time in seconds to wait after removing the faraday cup before starting the sample x-ray count acquisition. Generally used for microprobes equipped with a beam current aperture to allow time for beam stabilization or stage x, y, z position adjustment. The allowable range is 0 to 100 seconds. The default value is 10 seconds.

**EnterPositionsRelative=1**

Specifies whether the Element Setup dialog uses relative or absolute off-peak position entry as a default. Specify 1 for relative off-peak position entry for the default. The default value is 0 for absolute off-peak position entry.

**UpdatePeakWavescanPositions=0**

Specifies whether the peak center routine updates the peakscan and wavescan limits (in addition to the off-peak positions) after a peak center. Specify 1 to have the program to update the peakscan and wavescan limits after a peak center. The default value is 0 for no update.

**MaxMenuFileArray=4**

Specifies the number of previously opened **Probe for EPMA** files that appear on the File menu. Simply click the file name in the file list and the file will be opened. The program will automatically create entries in the INI file that will appear similar to "MenuFlagArray[n] =" where n is an integer and a recently opened file will be inserted are the equal sign. These lines should not be edited.

**ExtendedFormat=0**

Specifies whether the default log window output is extended format mode (more than 8 elements per row) or not. The default value is false for 8 elements per row of output. This mode is best for portrait mode printing, but uses additional vertical screen space.

**MACTypeFlag=1**

Specifies the default MAC (mass absorption coefficient) file used in the matrix calculations. Note that the MCMASTER.DAT file does not contain values for emitting energies below 1 keV.

**LINEMU.DAT (Henke < 10 keV) = 1**

**CITZMU.DAT (Heinrich, Armstrong) = 2**

**MCMASTER.DAT McMaster (from Rivers) = 3**

**MAC30.DAT Heinrich (fit to Goldstein table) = 4**

**MACJTA.DAT (Armstrong MACCALC program) = 5**

**FFAST.DAT (Chantler 2005 tabulation from Ritchie) = 6**

**USERMAC.DAT (User defined MAC table for user specified values) = 7**

To obtain mass absorption coefficients for elements 93 and above, the USERMAC.DAT file should be created using the MAC30.DAT file as its “basis” and then updated using the USERMAC.TXT file from CalcZAF. The USERMAC.TXT file contains the database values from the Institute of Transuranium Elements. The reference is:

I. R. Farthing and C. T. Walker, Heinrich's Mass Absorption Coefficients for the K, L and M Lines, The European Commission, Institute for Transuranium Elements, Karlsruhe, Report K0290140, 1990

#### **PositionImportExportFileType=1**

Specifies the default position file import and export format. There are two types (type = 1 or type = 2) used by **Probe for EPMA** and its companion programs.

Type = 1 format is the original (default) format specified in the Import or Export Position Samples section below.

Type = 2 format is a new import/export position file format that contains two additional integer and a string field which allows the analytical sample setup number and digitized autofocus flag and file setup name to be specified. Note that the sample setup number and the file setup name should be the same for all positions in a single position sample. To convert an original format .POS file (type=1) to the new format (type=2), simply add two columns of zeros and a column of double quotes to the file using a text editor or spreadsheet program (comma, space or tab delimited).

*If it is intended to use an off-line stage for digitizing of analysis positions, it is recommended that the operator use the type = 2 format for standard, unknown and wavescan digitizing with sample setup numbers or file setup names. The only valid values for this parameter are 1 or 2.*

#### **DeadtimeCorrectionType=1**

Specifies the default deadtime correction type. There are two types supported, the normal expression (type = 1) and a high precision variation (type = 2) for use when the count rate exceeds 50K cps. The default expression is the normal expression. The only valid values for this parameter are 1 or 2.

Use the high precision deadtime expression when the instrument utilizes an electronically forced deadtime. In this case the deadtime is truly a constant parameter and can be experimentally determined to two or even three significant digits, allowing the precision deadtime expression to be used to significant advantage.

#### **AutoFocusStyle=1 ! SX-50/51/100 and JEOL 8900/8200 only**

Specifies the default auto focus style used during automated acquisitions. This option is ignored if the AutoFocusPresent parameter in the [Hardware] section is not set. There are several varieties for the auto focus style, as shown in the following list:

- 1 = auto focus on a new position sample only**
- 2 = auto focus on every point**
- 3 = auto focus using the digitized auto focus flags**
- 4 = auto focus using the specified auto focus interval**

#### **AutoFocusInterval=5 ! SX-50/51/100 and JEOL 8900/8200 only**

Specifies the default auto focus interval used during automated acquisitions if the auto focus feature is selected and the interval auto focus option is selected. This option is ignored if the AutoFocusPresent parameter in the [Hardware] section is not set. The default is an auto focus interval of 5 which means that the program will attempt an auto-focus every 5 positions during an automated acquisition.

#### **BiasChangeDelay=2.0**

Specifies the default time delay used when a change in detector bias is performed on a spectrometer PHA. This delay is used to allow the electronics sufficient time to settle before an acquisition is started. The default is 2.0 seconds and the value must be greater than or equal to zero and less than 100 seconds.

#### **UseEmpiricalPHADefaults**

Specifies whether to calculate PHA baseline, window, gain and bias defaults based on fit coefficients from the EMPPHA.DAT file. Only applicable when manually entering element setups. The default is to not use empirical PHA defaults. Change the UseEmpiricalPHADefaults flag to any non-zero value to use empirical PHA defaults calculated from the fit coefficients from the EMPPHA.DAT file. The format of the file is as shown here:

```

1 "TAP" 32 1340 0.00 "Baseline" 0.001 1.00 0.000 0.00
1 "TAP" 32 1340 0.00 "Window" 0.001 10.00 0.000 0.00
1 "TAP" 32 1340 0.00 "Gain" 0.000 0.00 0.000 0.00
1 "TAP" 32 1340 0.00 "Bias" -89.430 1668.35 0.000 0.00
1 "LDEB" 64 1380 0.00 "Baseline" -0.070 0.56 0.000 0.00
1 "LDEB" 64 1380 0.00 "Window" 0.001 10.00 0.000 0.00
1 "LDEB" 64 1380 0.00 "Gain" 0.000 0.00 0.000 0.00
1 "LDEB" 64 1380 0.00 "Bias" -58.160 1701.24 0.000 0.00
1 "LDE1" 64 1390 0.00 "Baseline" 0.040 0.95 0.000 0.00
1 "LDE1" 64 1390 0.00 "Window" 0.001 10.00 0.000 0.00
1 "LDE1" 64 1390 0.00 "Gain" 0.000 0.00 0.000 0.00
1 "LDE1" 64 1390 0.00 "Bias" -89.030 1656.12 0.000 0.00
1 "PET" 32 1240 5.00 "Baseline" 1.200 -0.27 2.030 -2.09
1 "PET" 32 1240 0.00 "Window" 0.001 5.00 0.000 0.00
1 "PET" 32 1240 0.00 "Gain" 0.000 0.00 0.000 0.00
1 "PET" 32 1240 0.00 "Bias" -100.570 1670.39 0.000 0.00
2 "TAP" 16 1340 0.00 "Baseline" 0.001 1.00 0.000 0.00
2 "TAP" 16 1340 0.00 "Window" 0.001 10.00 0.000 0.00
2 "TAP" 16 1340 0.00 "Gain" 0.000 0.00 0.000 0.00
2 "TAP" 16 1340 0.00 "Bias" -89.430 1668.35 0.000 0.00
2 "LDEC" 32 1390 0.00 "Baseline" 0.060 0.72 0.000 0.00
2 "LDEC" 32 1390 0.00 "Window" 0.001 10.00 0.000 0.00
2 "LDEC" 32 1390 0.00 "Gain" 0.000 0.00 0.000 0.00
2 "LDEC" 32 1390 0.00 "Bias" -59.120 1688.58 0.000 0.00
3 "LiF" 64 1440 8.00 "Baseline" 1.080 -0.38 0.630 -0.67
3 "LiF" 64 1440 0.00 "Window" 0.001 10.00 0.000 0.00
3 "LiF" 64 1440 0.00 "Gain" 0.000 0.00 0.000 0.00
3 "LiF" 64 1440 0.00 "Bias" -98.430 1841.70 0.000 0.00
3 "PET" 64 1480 0.00 "Baseline" 0.180 1.06 0.000 0.00
3 "PET" 64 1480 0.00 "Window" 0.001 5.00 0.000 0.00
3 "PET" 64 1480 0.00 "Gain" 0.000 0.00 0.000 0.00
3 "PET" 64 1480 0.00 "Bias" -106.100 1859.76 0.000 0.00
4 "TAP" 32 1340 0.00 "Baseline" 0.001 1.00 0.000 0.00
4 "TAP" 32 1340 0.00 "Window" 0.001 10.00 0.000 0.00
4 "TAP" 32 1340 0.00 "Gain" 0.000 0.00 0.000 0.00
4 "TAP" 32 1340 0.00 "Bias" -76.380 1749.55 0.000 0.00
4 "PET" 16 1540 5.40 "Baseline" 1.470 -0.46 0.840 -0.73
4 "PET" 16 1540 0.00 "Window" 0.001 5.00 0.000 0.00
4 "PET" 16 1540 0.00 "Gain" 0.000 0.00 0.000 0.00
4 "PET" 16 1540 0.00 "Bias" -95.530 1764.11 0.000 0.00
5 "LIF" 32 1370 8.00 "Baseline" 1.080 -0.38 0.630 -0.67
5 "LIF" 32 1370 0.00 "Window" 0.001 10.00 0.000 0.00
5 "LIF" 32 1370 0.00 "Gain" 0.000 0.00 0.000 0.00
5 "LIF" 32 1370 0.00 "Bias" -98.430 1841.70 0.000 0.00
5 "PET" 64 1680 0.00 "Baseline" 0.180 1.06 0.000 0.00
5 "PET" 64 1680 0.00 "Window" 0.001 5.00 0.000 0.00
5 "PET" 64 1680 0.00 "Gain" 0.000 0.00 0.000 0.00
5 "PET" 64 1680 0.00 "Bias" -106.100 1859.76 0.000 0.00

```

The first column indicates the “target” spectrometer number, the second column the “target” crystal, the third column the “target” gain value (JEOL instruments), the fourth column the “target” bias value (Cameca instruments), the fifth column the keV threshold for whether to use the low or high fit coefficients, the sixth column the data type (Baseline, Window, Gain or Bias only). The next two columns are the low threshold linear fit coefficients (slope and intercept) used to calculate the PHA defaults. The last two columns are the high threshold linear fit coefficients (slope and intercept) used to calculate the PHA defaults.

In JEOL instruments the gain is considered “fixed” and the baseline, window and bias are calculated. In Cameca instruments either the gain or bias could be considered ‘fixed’, but typically the bias is set just above the detector response plateau and therefore can be considered “fixed”. In this case the bias (+/- 10 volts) is the target value and the baseline, window and gain are calculated.

The expression used to calculate the PHA parameters is shown here:

$$y_{PHA} = m \cdot x + b$$

where:      m      =      the slope coefficient  
                 b      =      the intercept coefficient  
                 x      =      the natural log of the emission line energy in keV

The program will automatically load PHA defaults values from the SCALERS.DAT file if any of the following conditions are true in the EMPPHA.DAT file:

- the spectrometer and crystal pair are not found
- the coefficient slope and intercept are both zero
- the PHA data type is not found

Also, if the keV threshold is zero, the program will always use the low slope and intercept (assuming that they are not both zero).

**KilovoltChangeDelay=1.0 ! SX-50/51/100 and JEOL 8900/8200 only**

Specifies the default time delay used when a change in the operating voltage is performed. This parameter is only utilized if the OperatingVoltagePresent flag is true. The default is 1.0 seconds and the valid range is from 0.0 to 100 seconds. A change in the operating voltage will force a change in the beam current (if available).

**BeamCurrentChangeDelay=1.0 ! SX-50/51/100 and JEOL 8900/8200 only**

Specifies the default time delay used when a change in the beam current is performed. This parameter is only utilized if the BeamCurrentPresent flag is true. The default is 1.0 seconds and the valid range is from 0.0 to 100 seconds. A change in the beam current will force a change in the beam size (if available).

**BeamSizeChangeDelay=0.5 ! SX-50/51/100 and JEOL 8900/8200 only**

Specifies the default time delay used when a change in the beam size is performed. This parameter is only utilized if the BeamSizePresent flag is true. The default is 0.5 seconds and the valid range is from 0.0 to 100 seconds.

**LogWindowBufferSize=32786**

Specifies the default size of the Log window text buffer. The size is limited to 32K on Win95 systems and only by the available memory on Win NT systems. The default is 512 kbytes (524288).

**PeakOnAssignedStandards=0**

Specifies the default mode for whether calibration of peak positions for each element should be attempted on all standards or for the standard assigned for that element. This value will be used for a new run to load the Calibrate on Assigned Standards check box in the Automate! window. The default is zero or false to attempt peaking on all standards.

**PrintAnalyzedAndSpecifiedOnSameLine=0**

Specifies the default mode for whether analyzed and specified element results in the log window should be printed out on the same line. This flag may be used in conjunction with the Extended Format flag to get all elements analyzed and specified (up to 32) all printed out on one line in the log window. The default is zero or false to print the specified elements on several lines first and then print the analyzed elements on subsequent lines next.

**NoMotorPositionLimitsChecking=0 ! AM interface only**

Specifies whether the spectrometer motor position electrical limit software check should be implemented during the crystal flip procedure. This flag only applies to the AM (MC API) interface using the original JEOL crystal flipping electronics. If this flag is set to a non-zero value then the software will not check the motor position limits during a crystal flip in both the manual and automated software procedures. The default is zero or false to check the motor position limits during a crystal flip procedure.

**UserDataDirectory="C:\UserData"**

Specifies the default directory that the user data files are stored in. Normally this is "C:\UserData" but any other valid path may be used.

**ExtendedMenu=0**

This flag is used to indicate if additional menus should be visible in CalcZAF. These menus add calculations for comparing atomic, mass and electron fraction first approximation calculations and are not generally utilized by most

users. The default is zero to have these menu items not visible. Set the flag to a non-zero value to make these items visible.

#### **AutoAnalyze=0**

This flag is used to indicate if **Probe for EPMA** should automatically perform a quantitative analysis after each analyzed standard or unknown data point. The default is zero to not perform a quantitative analysis after each acquisition. Set the flag to a non-zero value to have the program automatically perform a quantitative analysis after each standard or unknown acquisition.

#### **FaradayAlwaysOnTop=0**

This flag will specify whether the main window of the Faraday applet (beam and faraday cup control) will be "always on top" of all other forms. The default is 0 for not always on top. Set this flag to a non-zero number if it is preferred that the Faraday program main window should be "always on top".

#### **ColumnConditionChangeDelay=10.0**

Specifies the default time delay used when a change in the column condition is performed on the microprobe. This delay is used to allow changes in the electron column configuration sufficient time to complete before an acquisition is started. The default is 10.0 seconds and the value must be greater than or equal to zero and less than 100 seconds.

#### **SurferOutputVersionNumber=7**

Specifies the default output format for the Surfer (from Golden Software) OLE scripting .BAS file. This is necessary due to the fact that there are subtle differences in the way VBA scripting is implemented in different versions of Surfer. The default is 7 for Surfer version 7.0 and the only allowed values are 6 for Surfer 6.0 or 7 and higher for Surfer 7.0. (Surfer 8 uses the same format as Surfer 7). See the GRID file format in the Glossary for more information.

#### **SelPrintStartDoc=1**

Specifies whether the print option for the Log window should send nested StartDoc and EndDoc commands to the printer (the default behavior). Because some printers will lock up when they receive a nested StartDoc and EndDoc commands this option allows this default behavior to be disabled if the flag is set to zero. The default is non-zero for allowing nested StartDoc and EndDoc commands to be sent to the printer.

#### **UseMultiplePeakCalibrationOffset=0**

This parameter specifies whether the software will calculate the default spectrometer offset (from the theoretical peak position to the actual peak position) using a fixed offset or based on coefficients stored in the PROBEWIN-X.CAL file (where X is Ka, Kb, La, Lb, Ma or Mb representing each of the x-ray line families). Using this method the user can calibrate each crystal on each spectrometer so that the default peak positions are automatically updated for the proper position offsets based on the spectrometer position and the measured offsets.

The PROBEWIN-X.CAL file contains, for each crystal on each spectrometer, a number of elements and standards used for the peak calibration process. Up to 5 element/x-ray/standard combinations may be specified for each spectrometer/crystal calibration in the system. The software will also store the theoretical and actual (measured) peak positions for each peak center along with the fit coefficients calculated using a 2<sup>nd</sup> order polynomial fit. The fit is based on the actual peak position versus the measured offset from the theoretical peak position. For example the spectrometer position offset is calculated using the following equation:

$$P_O = P_T - P_A$$

Where  $P_T$  is the theoretical spectrometer peak position  
 $P_A$  is the actual (measured) spectrometer peak position

The calculated offset is applied to the theoretical peak position as follows:

$$P_A = P_T - P_O$$

Where  $P_T$  is the theoretical spectrometer peak position  
 $P_O$  is the calculated spectrometer position offset from above

Use the Peak/Scan Options dialog in program StartWin to maintain and update these spectrometer peak positions offset coefficients. The default offset is zero until the user performs multiple peak calibrations. Note that if only a single x-ray line is specified for the calibration procedure on a particular spectrometer/crystal combination, the software will apply that measured offset as a constant over the entire spectrometer range. If two elements are specified, the software will apply a straight line interpolation over the spectrometer range. If three elements, then a polynomial fit is used.

**UseWideROMPeakScanAlways=0**

The “UseWideROMPeakScanAlways” forces the application to always specify a “wide” ROM based peak scan for peaking the spectrometers (SX100/SXFive only).

**UseCurrentConditionsOnStartUp=0**

**UseCurrentConditionsAlways=0**

The “UseCurrentConditionsOnStartUp” flag forces the application to read the current kilovolts, beam current and beam size state of the instrument when the application is first started.

The “UseCurrentConditionsAlways” flag forces the application to read the current kilovolts, beam current and beam size state of the instrument when the application is first started ***and to also use the current instrument conditions when starting new samples***. This means that if you are in scan mode but the sample analytical conditions are set to spot mode, the software will use the current instrument scan mode (and magnification) for the next sample that is acquired.

Use a non-zero value to turn on either flag in the software. If the “UseCurrentConditionsOnStartUp” or “UseCurrentConditionsAlways” flags are set, the column conditions and PHA parameters existing on the instrument are used as the default column conditions and PHA values. The default for both flags is off (zero).

**DefaultVacuumUnitsType=0**

Default vacuum units display for vacuum.exe. Values: 0 = Pascals, 1 = Torr, 2 = mbar, default = 0 for Pascal vacuum units.

**DefaultCorrectionType=0**

**DefaultZAFType=1**

Allows the user to set the default matrix correction type and the default ZAF or Phi-Rho-Z type. The allowable values for Default Correction Type are:

- 0 = "ZAF or Phi-Rho-Z calculations"
- 1 = "Constant Alpha Factors (single coefficient)"
- 2 = "Linear Alpha Factors (double coefficient)"
- 3 = "Polynomial Alpha Factors (triple coefficient)"
- 4 = "Calibration Curve (multi-standard)"

The allowable values for Default ZAF Type are:

- 1 = "Armstrong/Love Scott (default)"
- 2 = "Conventional Philibert/Duncumb-Reed"
- 3 = "Heinrich/Duncumb-Reed"
- 4 = "Love-Scott I"
- 5 = "Love-Scott II"
- 6 = "Packwood Phi(pz) (EPQ-91)"
- 7 = "Bastin (original) Phi(pz)"
- 8 = "Bastin PROZA Phi(pz) (EPQ-91)"
- 9 = "Pouchou and Pichoir - Full"
- 10 = "Pouchou and Pichoir - Simplified"

You may not use a DefaultZAFType of 0 (individual selections) as the default ZAF Type.

**PENDBASE\_Path="C:\Userdata\Penepma12\Pendbase"**  
**PENEPMA\_Path="C:\Userdata\Penepma12\Penepma"**

**PENEPMARoot="C:\Userdata\Penepma12"**

**PENEPMAPARPath="C:\Userdata\Penepma12\Penfluor"**

The path for application files for creating Penepma (Penelope Monte-Carlo simulation software for EPMA) input files are specified here. Contact Probe Software if you are interested in this utilizing this option to obtain the necessary PENEPMARoot and sample files.

The PENEPMARoot is the folder where the base input files are stored to optimize production of characteristic x-rays, backscatter electrons, continuum or secondary x-rays.

Probe for EPMA (Standard.exe) supports Penepma06, Penepma08 and Penepma12. With Penepma12 Probe for EPMA allows calculation of secondary fluorescence from boundaries quickly using Penfluor.exe, Fitall.exe and Fanal.exe.

The PENEPMAPARPath keyword can be utilized for performing parallel calculations for PAR files with multiple applications running simultaneously. This is to allow the more rapid calculation of pure element and binary compositional range PAR files for the entire periodic table in reasonable times.

For this purpose the PENEPMAPARPath should be set to a shared network folder and the CalcZAF or Probe for EPMA setup.exe should be run multiple times and installed in multiple folders, each with its own unique Penepma path statements with the exception of the PENEPMAPARPath statements which should all point to the shared network folder for storing the .PAR files.

#### **ForceNegativeKratiosToZero=0**

This flag is used to force negative k-ratios to zero during the matrix iteration. Normally this flag is zero to allow accurate calculation of trace element concentrations where the average value is close to zero. However the ForceNegativeKratiosToZero flag can be used to force negative k-ratios to zero when, for example, the background position offsets are interfered with and the resulting negative concentrations are affecting the overall matrix correction.

To force negative k-ratios to zero as the default simply set the value to any non-zero value.

#### **AutoIncrementDelimiterString="\_"**

This parameter specifies the Auto Increment delimiter string used in the Digitize window for automatically incrementing a number (\_001, \_002, \_003, etc.) that appended on the current position sample name. The default delimiter string is an underscore. Any string up to 12 characters long may be used.

#### **UseLastUnknownAsWavescanSetup=0**

This parameter allows the user to force the program to load wavescan samples based on the last unknown sample as opposed to the last wavescan sample. This is useful if you prefer to always have the next wavescan sample based on the last unknown sample in case it has been re-peaked or elements added or subtracted from it.

The default is false (do not load wavescan samples based on last unknown sample) to be backward compatible. Set this flag to any non-zero number to force the program to load wavescan samples based on the last unknown sample instead.

**UserSpecifiedOutputSampleName=1**

**UserSpecifiedOutputLineNumber=1**

**UserSpecifiedOutputWeightPercent=1**

**UserSpecifiedOutputOxidePercent=0**

**UserSpecifiedOutputAtomicPercent=1**

**UserSpecifiedOutputTotal=0**

**UserSpecifiedOutputDetectionLimits=0**

**UserSpecifiedOutputPercentError=0**

**UserSpecifiedOutputStageX=1**

**UserSpecifiedOutputStageY=1**

**UserSpecifiedOutputStageZ=1**

**UserSpecifiedOutputRelativeDistance=0**

**UserSpecifiedOutputRelativeLineNumber=1**

**UserSpecifiedOutputOnPeakTime=0**

```
UserSpecifiedOutputHiPeakTime=0
UserSpecifiedOutputLoPeakTime=0
UserSpecifiedOutputOnPeakCounts=0
UserSpecifiedOutputOffPeakCounts=0
UserSpecifiedOutputNetPeakCounts=0
UserSpecifiedOutputKraw=1
UserSpecifiedOutputDateTime=1
UserSpecifiedOutputKratio=0
UserSpecifiedOutputZAF=0
UserSpecifiedOutputMAC=0
UserSpecifiedOutputStdAssigns=0
UserSpecifiedOutputSampleNumber=1
UserSpecifiedOutputSampleConditions=0
UserSpecifiedOutputFormula=0
UserSpecifiedOutputTotalPercent=0
UserSpecifiedOutputTotalOxygen=0
UserSpecifiedOutputTotalCations=0
UserSpecifiedOutputCalculatedOxygen=0
UserSpecifiedOutputExcessOxygen=0
UserSpecifiedOutputZbar=0
UserSpecifiedOutputAtomicWeight=0
UserSpecifiedOutputOxygenFromHalogens=0
UserSpecifiedOutputHalogenCorrectedOxygen=0
UserSpecifiedOutputChargeBalance=0
UserSpecifiedOutputFeCharge=0

UserSpecifiedOutputSpaceBefore=0
UserSpecifiedOutputAverage=0
UserSpecifiedOutputStandardDeviation=0
UserSpecifiedOutputStandardError=0
UserSpecifiedOutputMinimum=0
UserSpecifiedOutputMaximum=0
UserSpecifiedOutputSpaceAfter=0

UserSpecifiedOutputUnkIntfCorsFlag=0
UserSpecifiedOutputUnkMANAbsCorsFlag=0
UserSpecifiedOutputUnkAPFCorsFlag=0
UserSpecifiedOutputUnkVolElCorsFlag=0
UserSpecifiedOutputUnkVolElDevsFlag=0

UserSpecifiedOutputOxideMolePercentFlag=0

UserSpecifiedOutputStandardPublishedValuesFlag=0
UserSpecifiedOutputStandardPercentVariancesFlag=0
UserSpecifiedOutputStandardAlgebraicDifferencesFlag=0

UserSpecifiedOutputBeamCurrent=0
UserSpecifiedOutputAbsorbedCurrentFlag=0
UserSpecifiedOutputBeamCurrent2=0
UserSpecifiedOutputAbsorbedCurrent2Flag=0
```

These flags are used to set the default data types for the User Specified Output dialog which is accessed from the Probewin.exe Output menu. The selections in the dialog are automatically saved to your current probe database. The output data defaults are shown above (zero = false, non-zero = true).

**NthPointAcquisitionInterval=10**

This keyword indicates the default value for the Nth Point background acquisition method. In this method, the program will measure the off-peak backgrounds on the first point on each sample, but then re-use that point's background intensity measurements on subsequent data points instead of measuring the off-peak intensity for each data point.

The program will continue to re-use the background intensities until NthPointAcquisitionIntervals have been acquired (or a new sample started) at which time it will re-measure the off-peak intensities and use those previously measured intensities for subsequent off-peak intensities for another NthPointAcquisitionIntervals.

#### **GeologicalSortOrder=0**

The Geological Sort Order defines the element sort order for the sort button in the Analyze! window and for the sort option in the JJD-2, H.W. and user specified custom output formats available from the Output menu. This flag can be changed within a probe run from the Analytical | Analysis Options menu. The default is 0 for no sorting, 1 = traditional geological sorting (SiO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, etc.). Or enter 2 for low to high Z (atomic number) sorting or 3 for high to low Z (atomic number) sorting.

#### **DefaultLIFPeakWidth=0.08**

Used to set the default LIF peak width for nominal interference overlap percent calculations. Not used for actual quantitative interference calculations. The default is 0.08 which is calibrated for SX100/SXFive instruments with a 160mm focal circle. JEOL instruments may require a slightly larger value for improved accuracy of the nominal interference. Valid values range from 0.01 to 0.1.

#### **ThermoNSSLocalRemoteMode=0**

This flag indicates whether the Thermo NSS portal server is installed on the local or a remote computer. See also EDS\_IPAddress in the [hardware] section.

The default is 0 for local mode which means that both Probe for EPMA and Thermo NSS are installed on the same computer. Change the value to 1 to indicate that the Thermo NSS server system is installed on a remote computer.

#### **MonitorFontSize=10**

Sets the default font size for the Monitor app display. Default = 10, valid range is between 6 and 32.

#### **JEOLSecurityNumber=0**

JEOL security number for connecting to 8230 and 8530 instruments. This number can be obtained from JEOL and needs to be specified before a connection can be made to the instrument.

#### **UseCurrentBeamBlankStateOnStartUpAndTermination=0**

This flag specifies that the program will use the current beam blank state the instrument is already in, upon startup or termination. The default is to use blank the beam upon startup or termination. Set the flag to a non-zero value to allow the program to use the current beam blank state when starting up or terminating.

#### **ShowAllPeakingOptions=0**

This flag specifies that the program will show or not show all spectrometer peaking options if the interface type is 2 or 5 (JEOL 8900/8200/8500 or Cameca SX100/SXFive). The default is to not show all peaking options if the interface is JEOL 8900/8200 or 8500 or Cameca SX100/SXFive. To show all peaking options set the flag to a non-zero value.

#### **ForceSetPHAParametersFlag=0**

This flag will force the PHA parameters to be set during acquisition even if the current PHA parameters on the instrument are already set to the specified values. This flag is only for special situations.

#### **CalcZAFDATFileDirectory= “C:\UserData\CalcZAFDATData”**

This keyword specifies the default location of the CalcZAF Data files (\*.DAT) used by the CalcZAF.exe application for importing intensity and compositional data. The default is the C:\UserData\CalcZAFDATData.

#### **ColumnPCCFileDirectory= “C:\UserData\ColumnPCCData”**

This keyword specifies the default location of the Column Data files (\*.PCC) used by Probe for EPMA applications to save and load instrument column condition settings. The default is the C:\UserData\ColumnPCCData.

**SurferDataDirectory="C:\UserData\SurferData"**

This keyword specifies the default location of the Surfer demonstration script files (\*.BAS) used by Golden Software's Scripter application to automate gridding, plotting and presentation graphics. The default is the C:\UserData\SurferData.

**DemoImagesDirectory="C:\UserData\DemoImages"**

This keyword specifies the default location of the demonstration image files (\*.JPG, \*.GIF, \*.BMP) used by Probe for EPMA applications demonstrate PictureSnap features. The default is the C:\UserData\DemoImages.

**DoNotRescaleKLM=0**

Flag to specify the default zoom mode for re-scaling KLM markers in the Plot Graph dialog. Set to a non-zero value to force the program to NOT re-scale the KLM markers when the wavescan plot is zoomed. The user can override this option in the Plot Graph window by checking or unchecking the appropriate checkbox in the KLM Markers frame.

**UsePenepmaKratiosLimit=0****PenepmaKratiosLimitValue=90**

These flags indicate whether Penepma derived k-ratios are to be used for overloading alpha-factors calculated from one of the analytical models in the Analytical | ZAF, Phi-Rho-Z, Alpha factor and Calibration Curve Selections menu are to be limited to concentrations below the specified limit value.

Set UsePenepmaKratiosLimit to a non-zero number to enable this option. If the flag is true then the software will not overload Penepma derived k-ratios for weight percent concentration greater than the limit value specified (e.g., 90).

This option is to avoid using k-ratios that may be subject to large imprecisions and that are normally a small component of the calculation (i.e., at high concentrations of the emitting element, the alpha factor for that element approaches 1.0).

The allowed range for the PenepmaKratiosLimitValue is from 50 to 99 percent.

**PenepmaMinimumElectronEnergy=1.0**

This keyword specifies the minimum electron energy the Penepma Monte Carlo simulations will go down to for photon-electron calculations. The default is 1.0 keV (1000 eV), the allowed range is 0.001 to 10 keV.

**UserImagesDirectory="C:\UserImages"****UserEDSDirectory="C:\UserEDS"****UserCLDirectory="C:\UserCL"****UserEBSDDirectory="C:\UserEBSD"**

These keywords specify the default location of other data types for integration into Probe for EPMA. The UserImagesDirectory keyword specifies the location of Probe Image PrbImg image files for background correction and matrix quantification using CalcImage. The other keywords are for other data types for utilizing the Hyper-Imaging features in CalcImage.

**SurferPlotsPerPage=4**

This keyword specifies the number of plots per page to output from the Surfer Scripter application. The only legal values are 1, 4 and 9 plots per page.

**GrapherAppDirectory=""****SurferAppDirectory=""**

These are the application folders where the Golden Software Grapher and Surfer Scripter.exe applications are found. This is usually updated by Probe for EPMA automatically along with the SurferOutputVersionNumber.

**SurferPlotsPerPagePolygon=3 ; must be 1, 3 or 8**

This parameter sets the default plots per page for the CalcImage quantitative polygon extraction output. These values can also be changed in the Surfer Scripter script for customized output.

**ProbeSoftwareInternetBrowseMethod=0**

Use this keyword to specify the Internet browse mode. A 0 (default) is for WWW, 1 for DVD browsing locally without an Internet connection. Requires Probe Software subscription DVDs.

#### **UseFluorescenceByBetaLinesFlag=1**

This flag enables fluorescence correction of beta lines to be calculated. To skip this correction set this flag to a non-zero value. The default is zero.

#### **StrataGEMVersion=6**

This flag is used to add a “\$End” statement at the end of the StrataGEM import file. Beginning with version 6 of StrataGEM a \$End statement is required at the end of the import file.

However, versions of StrataGEM prior to version 6 \*cannot\* have a \$End statement at the end of the StrataGEM import file according to JF Thiot. Whatever happened to backwards compatibility?

Anyway, the default StrataGEM version is 6, so Probe for EPMA will add a \$End statement to the end of the import file automatically, unless the StrataGEMVersion keyword in the Probewin.ini file [software] section is set to 4 or 5. Which means that users with older versions of StrataGEM will have to edit their Probewin.ini files manually.

### **[Hardware]**

InterfaceType=0

NumberOfTunableSpecs=5

NumberOfStageMotors=3

JoystickXPolarity=0

JoystickYPolarity=1

JoystickZPolarity=0

SpecBacklashFlag=0

StageBacklashFlag=0

SpecBacklashType=1

StageBacklashType=1

FilamentStandbyPresent=0

FilamentStandbyType=0

FilamentStandbyExternalScript=""

EDSSpectraInterfacePresent=0

EDSSpectralInterfaceType=0

EDSThinWindowPresent=0

EDSInsertRetractPresent=0

EDSMaxEnergyThroughputPresent=0

EDSMCSInputsPresent=0

EDS\_IPAddress=""

EDS\_ServicePort=""

EDS\_ServerName="Local Server"

EDS\_LoginName="edx"

EDS\_LoginPassword="edx"

WDS\_IPAddress=""

WDS\_IPAddress2=""

WDS\_ServicePort=0

WDS\_ServicePort2=0

```

DriverLoggingLevel=0
JeolMonitorInterval=400
JeolEOSInterfaceType=1
JeolEIKSVersionNumber=4
ThermalFieldEmissionPresent=0
LAB6FieldEmissionPresent=0

OperatingVoltagePresent=0
OperatingVoltageType=0
OperatingVoltageTolerance=0.002

BeamCurrentPresent=0
BeamCurrentType=0
BeamCurrentTolerance=0.02
BeamCurrentToleranceSet=0.01

BeamSizePresent=0
BeamSizeType=0

BeamModePresent=0
BeamModeType=1
MagnificationPresent=0
MagnificationType=1

ColumnConditionPresent=0
ColumnConditionType=0
ColumnConditionMethod=0
ColumnConditionString=""

Jeol8900PreAcquireString="" ; use commas to separate multiple commands
Jeol8900PostAcquireString="" ; use commas to separate multiple commands

AutoFocusPresent=0
AutoFocusType=0 ; 0 = parabolic, 1 = Gaussian, 2 = maximum value
AutoFocusOffset=0
AutoFocusMaxDeviation=30
AutoFocusThresholdFraction=0.33
AutoFocusMinimumPtoB=1.4
AutoFocusRangeFineScan=100 ; 50-1000 points
AutoFocusRangeCoarseScan=600
AutoFocusTimeFineScan=20 ; 1-500 msec
AutoFocusTimeCoarseScan=20

ROMPeakingPresent=0
ROMPeakingType=0 ; 0 = internal, 1 = parabolic, 2 = maxima, 3 = Gaussian
ROMPeakingParabolicThresholdFraction=0.33
ROMPeakingMaximaThresholdFraction=0.50
ROMPeakingGaussianThresholdFraction=0.33
ROMPeakingMaxDeviation=20.

AlwaysPollFaradayCupState=0

ScanRotationPresent=0
ScanRotation=0.0

DetectorSlitSizePresent=0

```

```
DetectorSlitSizeType=0
DetectorSlitPositionPresent=0
DetectorSlitPositionType=0
DetectorModePresent=0
DetectorModeType=0

MoveAllStageMotorsHardwarePresent=0

JeolCondenserCoarseCalibrationSettingLow=25
JeolCondenserCoarseCalibrationSettingMedium=35
JeolCondenserCoarseCalibrationSettingHigh=45
JeolCondenserFineCalibrationSetting=128

JeolCondenserCoarseCalibrationMode=0
JeolCondenserCoarseCalibrationBeamLow=30.4
JeolCondenserCoarseCalibrationBeamMedium=7.3
JeolCondenserCoarseCalibrationBeamHigh=1.68
JeolCondenserNumberOfApertures=1
JeolCoarseCondenserCalibrationDelay=0.1

SampleExchangePositionX=44.5
SampleExchangePositionY=1
SampleExchangePositionZ=11
SampleExchangePositionW=1

ReflectedLightIntensity=63
TransmittedLightIntensity=63

DisableSpectrometerNumber=0
SpectrometerROMScanMode=0 ; SX100/SXFive only, 0 = absolute scan, 1 = relative scan
FilamentWarmUpInterval=2.0
TurnOffSEDetectorBeforeAcquisition=0
AutomationOverheadPerAnalysis=10.0

ReflectedLightPresent=0
TransmittedLightPresent=0
HysteresisPresent=0

SX100MoveSpectroMilliSecDelayBefore=100
SX100MoveSpectroMilliSecDelayAfter=10

SX100MoveStageMilliSecDelayBefore=100
SX100MoveStageMilliSecDelayAfter=10

SX100ScanSpectroMilliSecDelayBefore=200
SX100ScanSpectroMilliSecDelayAfter=200

SX100FlipCrystalMilliSecDelayBefore=200
SX100FlipCrystalMilliSecDelayAfter=200

MinMagWindow=40
MaxMagWindow=12000000

ImageShiftPresent=0
ImageShiftType=0
```

CLSSpectraInterfacePresent=0  
CLSSpectraInterfaceType=0  
CLInterfaceInsertRetractPresent=0

ThermoNSSVersionNumber="3.0"

MoveStageToleranceX=0.1  
MoveStageToleranceY=0.1  
MoveStageToleranceZ=0.1

InsertFaradayDuringStageJogFlag=0

**InterfaceType=0**

Specify the hardware interface type to be used for microprobe interface control. The following values are allowed :

**0 = Demonstration mode (no hardware interface)**

**1 = Unused WDS**

**2 = JEOL 8900/8200/8500/8230/8530 interface**

**3 = Unused WDS**

**4 = Unused WDS**

**5 = Cameca SX100/SXFive interface**

Note that specifying the wrong hardware interface could cause the computer to "lock-up" if the hardware interface defined is not actually present so be sure to consider the proper interface. The default value is 0 for demonstration mode (no hardware interface) and is always a safe mode.

**Special note: when using the InterfaceType=2 (JEOL 8200/8900/8500/8x30 TCP/IP direct socket interface), the parameter JeolEOSInterfaceType must be defined as described below (1=8200, 2=8900, 3=8230/8530).**

You may receive a "Function Level Timeout in Driver" error message when you attempt to connect to the JEOL instrument for one of the following reasons:

1. Any component of the TCP/IP communication is not set up correctly or has been broken after setup, this includes:
  - a. Ethernet cable from the Probe for EPMA PC is not connected to the Ethernet switch (also Ethernet cable from switch to JEOL SC board is not connected)
  - b. Power to Ethernet switch is not on.
  - c. IP address on Probe for EPMA computer is not properly set (should be manual IP assignment, no gateway, no DNS).
2. JEOL driver DLL file is not present in expected location on the Probe for EPMA PC.
3. TCP/IP communications from the JEOL SC board, EOS board, EDS board, and OS-9 computer have been interrupted. This lack of activity can be observed on the Ethernet switch. If interrupted the normal fix is to recycle OPE power. On JEOL 8500 systems the fix is to restart SC communications.
4. The JEOL software can be used to attempt to restart the monitor between the SUN/HP and the SC board. This is accessed from the JEOL Menu - Connect EPMA System. This is sometimes successful but if all the JEOL boards have been interrupted OPE power cycle is necessary.

Cycling OPE Power ("white switch") should be done by the microprobe operator. The switch is located on the back of the 8900 console or on the front of the 8200 column (same for 8500).

**Special note: when using InterfaceType=5 (Cameca SX100/SXFive direct socket interface), the spectrometer dynamic offsets should always be zero. Therefore, use of the Cameca Verify Spectrometers or SXLocal "veri spec" commands are discouraged, to ensure that the recorded spectrometer positions are in absolute spectrometer values.**

That is to say that the Cameca Verify Spectrometer command may be used, but the multi-peak spectrometer calibration feature will not provide an accurate calibration of the theoretical spectrometer offsets since all spectrometer positions will be relative, not absolute. However, the use of the Stage verify command is perfectly fine.

**NumberOfTunableSpecs=5**

Enter the actual number of tunable spectrometers (scanning) on the microprobe. The allowable range is 0 to MAXTUNABLE%. The default is 0 for no tunable spectrometers.

**NumberOfStageMotors=3**

Enter the actual number of stage motors on the microprobe. For no stage control enter 0, for X and Y motion only, enter 2, for X, Y and Z motion, enter 3. The allowable range is 1 to MAXAXES%. The default value is 0 for no stage motors.

**JoyStickXPolarity=0**

The joystick X axis polarity. Applied to the X axis on the joystick to reverse the default direction of the motor. Change this value to "1" to reverse the polarity of the X axis joystick axis. The default value is 0.

*Note: Joystick polarity values are used to set the Move window spin button stage polarity even if a joystick is not used in the system.*

**JoyStickYPolarity=0**

The joystick Y axis polarity. Applied to the Y axis on the joystick to reverse the default direction of the motor. Change this value to "1" to reverse the polarity of the Y axis joystick axis. The default value is 0.

*Note: Joystick polarity values are used to set the Move window spin button stage polarity even if a joystick is not used in the system.*

**JoyStickZPolarity=0**

The joystick Z axis polarity. Applied to the Z axis on the joystick to reverse the default direction of the motor. Change this value to "1" to reverse the polarity of the Z axis joystick axis. The default value is 0. This parameter applies to 3-D joysticks only.

*Note: Joystick polarity values are used to set the Move window spin button stage polarity even if a joystick is not used in the system.*

**JeolEOSInterfaceType=1**

This parameter is used only for the JEOL instruments using the TCP/IP direct socket interface (InterfaceType = 2). Values from 1 to 3 are valid. The default is 1 for the 8200. The reason for this parameter is that the EOS (Electron Optics System) uses a different TCP/IP packet structure for the 8200/8500 and 8230/8530 compared to the older 8900 packet structure.

|   |                                |
|---|--------------------------------|
| JeolEOSInterfaceType = 1, ThermalFieldEmissionPresent = 0 | 8200                           |
| JeolEOSInterfaceType = 1, ThermalFieldEmissionPresent = 1 | 8500                           |
| JeolEOSInterfaceType = 2                                  | 8900 (TFE flag not applicable) |
| JeolEOSInterfaceType = 3, ThermalFieldEmissionPresent = 0 | 8230                           |
| JeolEOSInterfaceType = 3, ThermalFieldEmissionPresent = 1 | 8530                           |

*Special note for the 8230/8530: All Probe for EPMA functions work only if the JEOL PC-SEM software is in Observation mode. Probe for EPMA returns errors for every EIKS function called if in Comparison or Analysis mode.*

#### **JeolEIKSVersionNumber=4**

The JEOLEIKSVersionNumber is used by the software to indicate the current version of the JEOL EIKS interface. Use 3 for the original JEOL EIKS (2009), use 4 or 5 for the JEOL EIKS 2012. The default is 4. You can download the latest JEOL EIKS client software from our ftp site.

#### **[hardware] JEOLEIKSVersionNumber=3**

- Read mag
- Set mag
- Read hv
- Set hv

#### **[hardware] JEOLEIKSVersionNumber=4**

- Read beam size
- Set beam size
- Read probe scan (on/off)
- Set probe scan (on/off)

#### **[hardware] JEOLEIKSVersionNumber=5**

- Calibrate beam current (read condenser lenses)
- Set beam current (set condenser lenses)
- Read "sampling mode" spot vs. scan
- Set "sampling mode" spot vs. scan
- Read beam deflection position
- Set beam deflection position
- Read optical light (reflected/transmitted)
- Set optical light (reflected/transmitted)
- Set Analog channel (SE, BSE, etc.)

#### **ThermalFieldEmissionPresent=0**

This parameter determines whether a thermal field emission gun is present. This parameter only affects the JEOL 8200/8500 and 8230/8530 and Cameca SXFive instruments at the present time. The allowable values are 0 for not present and non-zero for present. The default is zero for not present.

#### **LAB6FieldEmissionPresent=0**

This parameter determines whether a LaB6 emission gun is present. This parameter is only valid for Cameca instruments at the present time. The allowable values are 0 for not present and non-zero for present. The default is zero for not present.

#### **SpecBacklashFlag=0**

This parameter is used to specify the default spectrometer backlash mode for all spectrometer motion. Enter 0 for false or any non-zero value for true. The default is 0 for no spectrometer backlash.

#### **StageBacklashFlag=0**

This parameter is used to specify the default stage backlash mode for stage motion from the Move dialog.

To set stage backlash options for standards, unknowns and wavescans separately for when the automation is used, refer to the Acquisition Options button in the Acquire! dialog. Note that the StageBacklashFlag in the Move dialog overrides the backlash options in the Acquisition Options dialog.

Enter 0 for false or any non-zero value for true. The default is 0 for no stage backlash.

#### **SpecBacklashType=1**

This parameter is used to indicate the spectrometer backlash type. Specify 1 for **Probe for EPMA** to control the spectrometer backlash, or 2 for ROM based backlash. The software backlash values are specified in the MOTORS.DAT file, line 7. The hardware ROM option is only available for certain hardware interfaces that support machine based automatic backlash or jog options (JEOL).

For JEOL 8900/8200/8500 instruments, this flag will enable the JEOL spectrometer backlash parameters. The actual backlash values are read from the MOTORS.DAT file, line 21 because the JEOL read backlash values command does not function properly. If the JEOL backlash values are changed in the JEOL software, be sure that the MOTORS.DAT values are also edited if it is desired that they be the same for both software packages.

#### **StageBacklashType=1**

This parameter is used to indicate the stage backlash type. Specify 1 for **Probe for EPMA** to control the stage backlash, or 2 for ROM based backlash. The software backlash values are specified in the MOTORS.DAT file, line 7. The hardware ROM option is only available for certain hardware interfaces that support machine based automatic backlash or jog options (JEOL).

For JEOL 8900/8200/8500 instruments, this flag will enable the JEOL stage backlash parameters. The actual backlash values are read from the MOTORS.DAT file, line 21 because the JEOL read backlash values command does not function properly. If the JEOL backlash values are changed in the JEOL software, be sure that the MOTORS.DAT values are also edited if it is desired that they be the same for both software packages.

#### **FilamentStandbyPresent=0**

This parameter is used to specify whether the microprobe hardware interface supports a filament standby mode for turning the filament off using the software automation. Enter 0 for false or any non-zero value for true. The default is 0 for no filament standby hardware interface.

#### **FilamentStandbyType=0**

The filament standby type for tungsten instruments (ThermalFieldEmissionPresent=0). The default is zero to just reduce the heat to the standby level. To reduce the heat AND turn off the HV, set FilamentStandbyType = 1. To only reduce the HV but leave the heat on (LaB6 filaments), set FilamentStandbyType = 2. To run an external script, set FilamentStandbyType = 3. To load a previously saved probe column condition file (.PCC), set FilamentStandbyType = 4.

If the instrument is a JEOL 8530 the FilamentStandbyType has no effect, and this feature will simply close the gun valve (V1) in filament standby mode (ON) and open the gun valve in filament standby mode (OFF)- assuming the specimen vacuum is within the allowable range.

#### **FilamentStandbyExternalScript=""**

The name of the external script or .PCC probe column condition file to be run if the FilamentStandbyType = 3 or FilamentStandby = 4. For the external script this can be any executable, e.g., .exe, .bat, etc. The full path to the external script must be specified, e.g., "C:\UserData\Shutdown.bat". For the .PCC probe column condition file, the full path to the .PCC file must be specified, e.g., "C:\UserData\ColumnPCCData\Filament\_Standby.PCC"

#### **EDSSpectraInterfacePresent=0**

This parameter defines whether a "spectrum" EDS interface is present. This EDS spectrum interface acquires a complete EDS spectrum for each analysis and saves it to the current probe database. This way the user may change or add the elements to analyze by EDS at any time, even in off-line mode.

Note that this spectral interface automatically acquires EDS spectrum for standard samples as well as unknown samples since the full net intensity calculation and matrix correction is performed in **Probe for EPMA**. Enter 0 for false or any non-zero value for true. The default is 0 for no EDS spectrum interface.

#### **EDSSpectraInterfaceType=0**

The EDS spectrum interface type. This interface acquires a full spectrum for each acquisition and stores it in the probe database for subsequent processing. Unlike the above EDS "weight percent" interface, the user does not have to pre-define the elements to be acquired by EDS. Note that EDS spectrum acquisition is required for standards containing the

element of interest for EDS quantification. The following low level (full spectrum acquisition) EDS interfaces are supported:

|                                |          |          |  |
|--------------------------------|----------|----------|--|
| <b>EDSSpectraInterfaceType</b> | <b>=</b> | <b>0</b> | <b>' Demonstration EDS spectrum mode</b>                     |
| <b>EDSSpectraInterfaceType</b> | <b>=</b> | <b>1</b> | <b>' Unused EDS</b>  |
| <b>EDSSpectraInterfaceType</b> | <b>=</b> | <b>2</b> | <b>' Bruker RTIFCCLIENT spectrum interface</b>               |
| <b>EDSSpectraInterfaceType</b> | <b>=</b> | <b>3</b> | <b>' Oxford Aztec spectrum interface (not supported yet)</b> |
| <b>EDSSpectraInterfaceType</b> | <b>=</b> | <b>4</b> | <b>' Unused EDS</b>  |
| <b>EDSSpectraInterfaceType</b> | <b>=</b> | <b>5</b> | <b>' Thermo NSS TEPortal spectrum interface</b>              |
| <b>EDSSpectraInterfaceType</b> | <b>=</b> | <b>6</b> | <b>' JEOL OEM spectrum interface</b>                         |

**Note that the Thermo NSS interface requires the TEPortal.Dll and Socketdll.Dll files to be copied to the PROBE application folder (or a folder in the system path such as System32 or SysWOW64). These files can be found in the Thermo application folders in the TEPortal folder under Program Files\Thermo Scientific\NSS.**

**Note that the Bruker RTIFCCLIENT interface requires the RTIFCCLIENT.DLL file to be copied to the PROBE application folder. Contact Probe Software if it is not available.**

#### **EDSThinWindowPresent=0**

If an ultra-thin window is used in the EDS system and combined EDS-WDS analyses are being performed, the optical light source must be turned off. This parameter is used to specify that an ultra-thin window is present that could transmit some of the optical light signal. Enter 0 for false or any non-zero value for true. The default is 0 for no EDS thin window.

#### **EDSInsertRetractPresent=0**

#### **EDSMaxEnergyThroughputPresent=0**

#### **EDSMCSInputsPresent=0**

This keyword indicates if the EDS detector hardware supports automatic insertion and retraction. The default is 0 for not present. Change to a non-zero value to indicate the insert/retract hardware is present.

The next keyword indicates that the maximum energy scale and pulse processing time is read/write. The default is 0 for not present. Change to a non-zero value to indicate the set/get max energy and pulse processing throughput hardware is present.

The EDSMCSInputsPresent keyword is used to set the WDS multi-channel spectrometer inputs for the proper element, x-ray and crystal in the Thermo or Bruker software, if the MCS hardware is present. Change to a non-zero value to indicate the MCS WDS inputs channel hardware is present.

#### **EDS\_IPAddress=""**

For certain EDS interfaces the IP address of the PC EDS acquisition server must be specified. The address must be in the correct TCP/IP "dot" format, e.g. "90.0.0.3

If the EDS software server is installed on the same computer as the **Probe for EPMA** software, use the loopback address of 127.0.0.1. If the EDS software is installed on another computer, the IP number will be the IP address of the remote computer.

The default for the EDS\_IPAddress is no IP number. This value is only used by the Thermo and JEOL EDS spectrum interfaces.

#### **EDS\_ServicePort=""**

For certain EDS interfaces the service port number of the acquisition server must be specified. The service port number must be an integer between 1 and 32768. This number is used only by the JEOL OEM EDS interface (must be 5000).

This number is not used by the Thermo or Bruker EDS servers (in Thermo it is specified in the application Service section (default = 5800 decimal), for Bruker it is specified in the Client remote server configuration).

#### **EDS\_ServerName="Local Server"**

Used to specify the Bruker remote client server interface. If installed on the local server, the server name is usually simply "Local Server". For remote clients, the server name must match a server defined by the Bruker Esprit **client** application Configuration menu (install Esprit using "Client" option).

Note that the Bruker remote server name must match exactly the server name specified in the Esprit client and is case sensitive. See the Bruker Configuration document for further details on setting up the Bruker server client for integrated EDS/WDS acquisition.

#### **EDS\_LoginName="edx"**

Used to specify the Bruker remote client user. This is defined in the Bruker Esprit **server** application (click on the icon in the system tray). For local server interfacing the login name is usually "edx" and the password is usually "edx".

#### **EDS\_LoginPassword="edx"**

Used to specify the Bruker remote client user password. This is defined in the Bruker Esprit server application (click on the icon in the system tray). For local server interfacing the login name is usually "edx" and the password is usually "edx".

#### **WDS\_IPAddress=""**

For certain WDS interfaces (JEOL 8900, 8200 and Cameca SX100/SXFive) the IP address of the microprobe system controller must be specified. The address must be in the correct TCP/IP "dot" format, e.g. "128.32.146.11".

Note that for the JEOL 8900 and the JEOL 8200 the default IP address for the system controller is 192.6.1.11 and for the client computer it is 192.6.1.1. For the 8200, a second IP address needs to be specified for EOS (device = SEM) (see WDS\_IPAddress2)

#### **WDS\_IPAddress2=""**

For certain WDS interfaces (JEOL 8200) the IP address of the microprobe EOS controller must be specified. The address must be in the correct TCP/IP "dot" format, e.g. "128.32.146.11". For the JEOL 8200 this second address is the SEM EOS interface.

#### **WDS\_ServicePort=0**

For certain WDS interfaces (JEOL 8900 and SX100/SXFive) the service port number of the microprobe system controller must be specified. The service port number must be an integer between 1 and 32768.

Note that for the JEOL 8900 and the JEOL 8200 the default service port number for the system controller (EPMA) must be 2785. For the JEOL 8900 only this one service port needs to be specified. For the 8200, a second service port needs to be specified (see WDS\_ServicePort2)

#### **WDS\_ServicePort2=0**

For certain WDS interfaces (JEOL 8200) a second service port number of the microprobe EOS controller must be specified. For the JEOL 8200 this second address is the SEM EOS interface and must be 22200. The service port number must be an integer between 1 and 32768.

#### **Summary of JEOL parameter settings (InterfaceType = 2):**

|  |   |
|--|---|
| <b>JeolEOSInterfaceType = 1, ThermalFieldEmissionPresent = 0</b> | <b>8200</b>                               |
| <b>JeolEOSInterfaceType = 1, ThermalFieldEmissionPresent = 1</b> | <b>8500</b>                               |
| <b>WDS_ServicePort = 2785, WDS_ServicePort2 = 22200</b>          |   |
| <br>   |   |
| <b>JeolEOSInterfaceType = 2</b>                                  | <b>8900 (Thermal flag not applicable)</b> |
| <b>WDS_ServicePort = 2785, WDS_ServicePort2 = 2785</b>           |   |

|  |             |
|--|-------------|
| <b>JeolEOSInterfaceType = 3, ThermalFieldEmissionPresent = 0</b> | <b>8230</b> |
| <b>JeolEOSInterfaceType = 3, ThermalFieldEmissionPresent = 1</b> | <b>8530</b> |
| <b>WDS_ServicePort = 22200, WDS_ServicePort2 = 22210</b>         |             |

**JeolMonitorInterval=400**

Allows the user to specify the default monitor packet send interval in milli-seconds. Default is 400 msec and the minimum value is 100 msec and the maximum is 10000 msec.

**OperatingVoltagePresent=0**

This parameter is used to specify whether the microprobe hardware interface supports control of the accelerating voltage. Enter 0 for false or any non-zero value for true. The default is 0 for no operating voltage hardware interface.

**OperatingVoltageType=0**

The operating voltage hardware interface type. Not utilized.

**OperatingVoltageTolerance=0**

The operating voltage tolerance is used to specify how close the operating voltage read from the system must be to the desired operating voltage in order to allow the program to skip re-setting the operating voltage. The parameter is in fraction units, that is 0.01 equals 1% and the default is 0.002 or 0.2%. The allowed range is 0.0001 (0.01%) to 0.1 (10%).

**BeamCurrentPresent=0**

This parameter is used to specify whether the microprobe hardware interface supports control of the beam current. Enter 0 for false or any non-zero value for true. The default is 0 for no beam current hardware interface.

**BeamCurrentType=0**

The beam current hardware interface type. Only utilized for the JEOL 8x30 interface. 0 = normal beam current iteration method, 1 = new direct set beam current call using firmware.

**BeamCurrentTolerance=0.02**

The beam current tolerance is used to specify how close the beam current read from the system must be to the desired beam current in order to allow the program to skip re-setting the beam current. The parameter is in fraction units, that is 0.01 equals 1% and the default is 0.02 or 2%. The allowed range is 0.0001 (0.01%) to 0.2 (20%).

**BeamCurrentToleranceSet=0.01**

This is the tolerance value utilized by the JeolSetBeamCurrentMDA function to set the beam current (0.01 = 1%). This value determines how close the iteration will approach the desired beam current value. The BeamCurrentTolerance value (see above) is the tolerance value the program uses before deciding to re-set the beam current. The BeamCurrentToleranceSet value is defaulted to 0.01 and the BeamCurrentTolerance is defaulted to 0.02.

This parameter is only used by the JEOL 8200, 8900 and 8500 instruments. The BeamCurrentToleranceSet value must be less than the BeamCurrentTolerance value.

**BeamSizePresent=0**

This parameter is used to specify whether the microprobe hardware interface supports control of the beam size. Enter 0 for false or any non-zero value for true. The default is 0 for no beam size hardware interface.

**BeamSizeType=0**

The beam size hardware interface type. Not utilized.

**BeamModePresent=0**

This parameter is used to specify whether the microprobe hardware interface supports control of the beam scan mode. Enter 0 for false or any non-zero value for true. The default is 0 for no beam scan mode control hardware interface.

**BeamModeType=1**

This parameter is not currently utilized.

**MagnificationPresent=0**

This parameter is used to specify whether the microprobe hardware interface supports control of the scan magnification. Enter 0 for false or any non-zero value for true. The default is 0 for no beam scan magnification hardware interface.

**MagnificationType=1**

The beam scan magnification hardware type. This parameter is only used to specify the JEOL 6300/6400 or 8600 type magnification control (same command in both cases).

|                              |   |
|------------------------------|---|
| <b>MagnificationType = 0</b> | <b>no magnification column control available (documentation only)</b> |
| <b>MagnificationType = 1</b> | <b>JEOL 6400 serial interface magnification column control</b>        |
| <b>MagnificationType = 2</b> | <b>JEOL 8600 serial interface magnification column control</b>        |

Note that if the MagnificationType = 0 the software will only use the user specified magnification for documentation purposes, for example to calibrate the scale bar of captured images from the SIS image acquisition system.

**ColumnConditionPresent=0****ColumnConditionType=0****ColumnConditionMethod=0****ColumnConditionString=""**

These keywords are used to specify if a column condition interface is present and if so how it should be utilized. Normally, microprobes without column interfaces use the kilovolt, beam current and beam size parameters simply for documentation purposes.

However, on instruments with a column interface, these kilovolt, beam current and beam size parameters are actually used to also set the operating condition of the electron column. On other instruments, these conditions cannot be specified individually and (or in addition), instead a "column condition string" is specified that configures the microscope for the desired operating conditions. Again, if the column condition interface is not actually present, then this field may be used simply for documentation purposes.

Some instruments, may have both the capability of setting the individual kilovolt, beam current and beam size parameters and also the capability of setting a "column condition string". In this situation either method may be used to actually set the electron column condition, however it must be noted that the even if the column condition method is utilized, the individual kilovolt (especially this parameter), beam current and beam size parameters must also be correctly specified so that the software can perform a proper quantitative matrix correction of the intensity data. This is because there is no rigorous method to determine the actual operating voltage from an arbitrary column condition string.

The ColumnConditionMethod is used to specify the default method to set the electron column operating conditions. If the ColumnConditionMethod is not specified or is zero, then the kilovolts, beam current and beam size parameters are individually specified for configuring the electron column. If the ColumnConditionMethod is 1, then the specified column condition string is utilized to set the electron column conditions.

The ColumnConditionString is used to specify the default column condition string. Normally this is blank.

**AutoFocusPresent=0      ! Cameca SX100/SXFive and JEOL 8900/8200/8500/8x30 only**

This parameter is used to specify whether the microprobe hardware interface supports control of the auto focus. This parameter must be set for the use of Cameca ROM based or JEOL software enabled auto focus options during automated acquisitions. Only the SX100/SXFive microprobe supports a ROM based auto focus feature, the JEOL auto-focus is software based and requires additional parameters described below. Enter 0 for false or any non-zero value for true. The default is 0 for no auto focus hardware interface. See also, the parameter EDSThinWindowPresent above.

To test the JEOL autofocus use the TestType.exe program and click the Plot Last Auto Focus button to see the optical data, threshold level, parabola fit, fit coefficients, standard deviation, and calculated centroid.

**AutoFocusType=0**

The autofocus hardware fit method. If the parameter is zero a parabolic fit is attempted based on the thresholded light intensities. If the value is one then a Gaussian fit to all the intensity data is attempted. If the value is 2 then the software will determine the autofocus based on the maximum intensity value (this is the OEM JEOL method). The default is 2 for a maximum value fit and this parameter and most of the following auto-focus parameters applies only to the JEOL 8900/8200/8500 interface.

**0 = Parabolic fit**

**1 = Gaussian fit**

**2 = Maximum value (JEOL method)**

Note that in DebugMode the program will display a modeless window showing the autofocus data and fit. Click the three buttons one at a time to see the fine scan, coarse scan and 2<sup>nd</sup> fine data displayed respectively.

There is no thresholding applied to the optical data for the maximum value fit method.

#### **AutoFocusOffset=0**

The z-axis stage offset for the autofocus adjustment. Use this parameter to compensate for small but systematic offset errors in the auto-focus procedure. This parameter is added to the auto-focus Z stage position returned by the ROM based (hard coded) procedure. The value must be specified in microns and must be between -100 and +100.

Note that this value does not apply to Cameca SX100/SXFive microprobes, only JEOL 8900, 8200/8500, and 8230/8530 instruments. For setting the auto-focus offset for Cameca instruments use the OEM configuration procedure.

#### **AutoFocusMaxDeviation=30**

This is the maximum percent deviation allowed for the parabolic fit to the optical data returned from the JEOL 8200/8500, 8900, 8230/8530 ILSM command for the auto-focus procedure. If the percent deviation fit from the fine auto-focus range (+/-100um) is greater than this the procedure will issue a coarse auto-focus range (+/-500um). If this is successful a fine autofocus range is attempted using the newly found z-position.

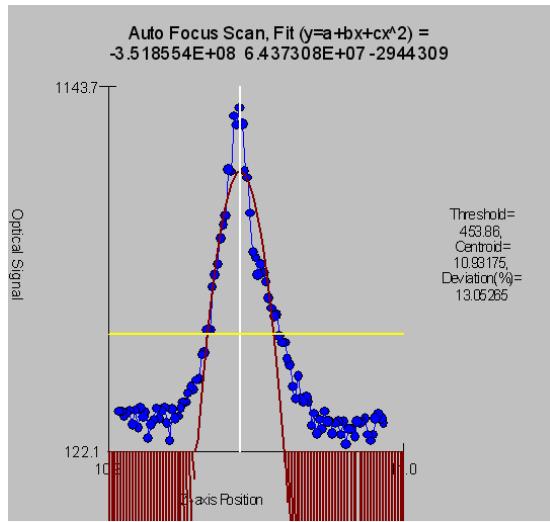
The minimum allowed percent deviation is 1 the maximum is 60 and the default is 30.

#### **AutoFocusThresholdFraction=0.33**

This value specified the threshold fraction above which the optical data is fit for finding the auto-focus z-axis position for data returned from the JEOL 8200/8900 ILSM command. The default is 0.33 the allowable values are 0.01 to 0.99. For example, if the AutoFocusThresholdFraction is 0.33 then all optical signal data point values in the lower 1/3 of the (maximum – minimum) range will not be included in the centroid fit. Obviously this parameter does not apply to the AutoFocusType = 2 (maximum intensity value).

#### **AutoFocusMinimumPtoB=1.4**

This value determines whether a sufficient peak in the optical z-axis stage scan was acquired. This parameter applies only to JEOL 8900 and 8200 instruments. The default value is 1.4, the range of allowable values are from 1.05 to 10. The image below (from TestType) shows a properly acquired autofocus scan.



**AutoFocusRangeFineScan=100**

**AutoFocusRangeCoarseScan=600**

These values determine the auto-focus scan ranges for fine and coarse focus. Used only by the JEOL 8200/8900 interface. The default is 100 um for fine and 600 um for coarse. The range is 10 to 1000 um for fine and 60 to 6000 um for coarse.

**AutoFocusPointsFineScan=200**

**AutoFocusPointsCoarseScan=100**

These values determine the number of points for the auto-focus fine and coarse scan ranges. Used only by the JEOL 8200/8900 interface. The default is 200 points for the fine scan and 100 points for the coarse scan. The range is 50 to 1000 points for both the fine and coarse scan ranges.

**AutoFocusTimeFineScan=20 ; 1-500 msec**

**AutoFocusTimeCoarseScan=20**

These values determine the integration time per point for the auto-focus fine and coarse scan ranges. Used only by the JEOL 8200/8900 interface. The default is 20 msec for both the fine scan and the coarse scan. The range is 1 to 500 msec for both the fine and coarse scan ranges.

**ROMPeakingPresent=0**

This parameter is used to specify whether the microprobe hardware interface supports control of the ROM based peak centering hardware that is internal to the microprobe. Enter 0 for false or any non-zero value for true. The default is 0 for no ROM based peak centering hardware interface. Note that following parameters are utilized for non ROM based and ROM based scans:

#### Non-**ROM** scan based

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

#### **ROM** scan based

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

**ROMPeakingType=0**

The ROM based peak center fit type. Applies only to Cameca SX100/SXFive and JEOL 8200/8900/8500 microprobes.

0 = Internal (the instrument ROM peak method which applies only to Cameca)

1 = Parabolic (spectrometer scan data is fit to a parabolic fit)

2 = Maxima (spectrometer scan data is fit to Brent's Maxima function)

3 = Gaussian (spectrometer scan data is fit to a Gaussian fit)

- 4 = Dual ROM (Maxima for LiF crystals, Parabolic for other crystals)
- 5 = Dual ROM (Maxima for LiF and Gaussian for other crystals)
- 6 = Highest Intensity (using smoothed intensities)

To increase the default ROM scan width decrease the Peakscan Size (line 16) or increase the LiF Peaking Start Size (line 19) in the SCALERS.DAT file. Note that within the application, the ROM based peak scans are based on the Peaking Count Time divided by 4 and the number of Peak Scan Points. The ROM scan width is determined by the Peaking Start Size.

The internal ROM type is available only on Cameca instruments. The parabolic and maxima fits require at least 3 data points above the threshold (see below). The parabolic, Gaussian and maxima methods have a user defined threshold that can be specified in the INI file and within the program.

The default is 2 for Brent's Maxima (inverted to obtain the minima).

**ROMPeakingParabolicThresholdFraction=0.33**  
**ROMPeakingMaximaThresholdFraction=0.50**  
**ROMPeakingGaussianThresholdFraction=0.33**  
**ROMPeakingMaxDeviation=20.**

The ROM peaking thresholds are designed to allow the user to define the intensity values above which are used for one of the three ROM fitting methods (parabolic, maxima or Gaussian). The default is 0.33 which means that all intensity values 1/3 above the minimum to maximum intensity range are used in the fit.

Note that increasing the threshold decreases the number of points that can be fit. The parabolic and Gaussian methods require at least 4 data points. The minimum threshold is 0.1 and the maximum is 0.9.

The ROMPeakingMaxDeviation is the maximum percent deviation allowed in the ROM peak fit. It applies only to the parabolic and Gaussian fit options. The default is 20% and the minimum is 5% and the maximum is 80%.

**ScanRotationPresent=0**

**ScanRotation=0.0**

These parameters are used to indicate that a scan rotation interface is present and what the default scan rotation should be set to upon startup. The default is 0 for no scan rotation interface present and 0.0 for zero degrees default scan rotation. Any non-zero value is used to indicate that the scan rotation interface is present and any value from 0 to 360 is valid for the default scan rotation.

**DetectorSlitSizePresent=1**  
**DetectorSlitSizeType=0**  
**DetectorSlitPositionPresent=0**  
**DetectorSlitPositionType=0**  
**DetectorModePresent=1**  
**DetectorModeType=0**

These parameters are used to indicate that an interface is present for various detector parameters. Specifically for slit size, slit position and detector mode. The default is 0 for no detector interface present and 0 for the interface type (not used at this time). Any non-zero value is used to indicate that the detector interface is present.

See the DETECTORS.DAT file for specific parameter values that can be specified by the user for configuring the various detector values.

**MoveAllStageMotorsHardwarePresent=0**

This parameter (intended for the JEOL stage control interfaces only), allows **Probe for EPMA** Xtreme to issue a stage move control in a single interface command. This is implemented to support interfaces that cannot move the stage motors individually. Set this value to a non-zero value to force **Probe for EPMA** to use a single command to move all three (or two) stage motors simultaneously.

**Jeol8900PreAcquireString=""** ; use commas to separate multiple commands

**Jeol8900PostAcquireString=""** ‘ use commas to separate multiple commands

These parameters are used by the JEOL 8900 (not the JEOL 8200) interface to set special conditions before and after the acquisition as specified by the user. For example, it might be desirable to give the command "PB OFF" before an acquisition to ensure that the beam is not scanning during the x-ray counting followed by the command "PB ON" to restore the beam scan after the acquisition is completed.

Use commas to separate multiple commands. For example, to send a PB ON and MG 4000 command the string should be "PB ON, MG 4000".

Alternatively the user might prefer to issue a change in the magnification instead, "MG 100000" before the acquisition and "MG 40" after the acquisition. The default is "PB OFF" for the pre-acquire command string and "PB ON" for the post-acquire string, otherwise the default is blank.

**AlwaysPollFaradayCupState=0**

This parameter determines whether the faraday cup position is constantly polled through the interface. This applies to the “demo”, JEOL and Cameca interfaces.

**DriverLoggingLevel=0**

This parameter determines whether the driver (JEOL 8200/8900 and SX100/SXFive) will log debug information to a file with the name of the application and an extension of .LOG. Note that enabling driver logging will overwrite the existing PROBEWIN.LOG file that logs all errors within all **PROBE** applications. This only applies to the JEOL 8200/8900, and SX100/SXFive interfaces. The logging level may be set to 0 for disabled (no driver debug information logged), 1 for basic logging of driver information and 2 for detailed information. Note that enabling driver level debug information may slow down the application response slightly.

**JeolCondenserCoarseCalibrationSettingLow=20****JeolCondenserCoarseCalibrationSettingMedium=35****JeolCondenserCoarseCalibrationSettingHigh=45****JeolCondenserFineCalibrationSetting=128**

These parameters are for the JEOL 8900 and 89200 to calibrate the beam current setting (auto-md). The program uses the three condenser coarse values to read the beam current and fit the log(beamcurrent) vs. condenser coarse for setting the approximate beam current. If necessary the program will iterate the condenser fine setting (starting at the specified setting) to get the value of the beam current based on the value of the BeamCurrentTolerance value.

The default values for both the JEOL 8200 and 8900 are 25, 35 and 45 and for the condenser fine calibration setting value is 128 for the 8900 and 255 for the 8200, and 511 if a thermal field emission gun is specified. See keyword ThermalFieldEmissionPresent above.

The beam current increases as the condenser lens values are lowered, however note that the JEOL 8200/8500 Condenser lens values are displayed inverted on the instrument console. Internally they are identical to the 8900.

**JeolCondenserCoarseCalibrationMode=0****JeolCondenserCoarseCalibrationBeamLow=55.13****JeolCondenserCoarseCalibrationBeamMedium=7.9****JeolCondenserCoarseCalibrationBeamHigh=1.68****JeolCondenserNumberOfApertures=1****JeolCoarseCondenserCalibrationDelay=0.1**

These values are used to provide a file based calibration curve for JEOL 8900 and 8200 instruments using aperture 1 through the number of apertures defined for the purpose of setting the coarse condenser lens value for initial iteration adjustment of the beam current.

Set JeolCondenserCoarseCalibrationMode to 0 for acquiring a fresh calibration curve and using the measured calibration curve within the program. To use a static calibration curve, set JeolCondenserCoarseCalibrationMode to 1 to use the JeolCondenserCoarseCalibrationBeamLow, JeolCondenserCoarseCalibrationBeamMedium and JeolCondenserCoarseCalibrationBeamHigh values from the INI file for setting the initial coarse condenser value when the program is adjusting the beam current.

Note that due to a subtle problem in the JEOL 8900 system, one should always set the JeolCondenserCoarseCalibrationMode value to 1 and define a manually measured condenser calibration curve. **Or use the Calibrate Set beam Current button the Analytical Conditions dialog to automatically perform a beam current calibration.** Here is a typical JEOL 8900 condenser calibration curve defined for a 2 aperture system:

```
JeolCondenserCoarseCalibrationMode=1
JeolCondenserCoarseCalibrationSettingLow=4,16
JeolCondenserCoarseCalibrationSettingMedium=21,30
JeolCondenserCoarseCalibrationSettingHigh=30, 40

JeolCondenserFineCalibrationSetting=128

JeolCondenserCoarseCalibrationBeamLow=197, 110.85
JeolCondenserCoarseCalibrationBeamMedium=18.61, 13.115
JeolCondenserCoarseCalibrationBeamHigh=4.33, 2.939
JeolCondenserNumberOfApertures=2
JeolCoarseCondenserCalibrationDelay=0.1
```

The beam current values are in nA and must correspond to the above JeolCondenserCoarseCalibrationSettingLow, JeolCondenserCoarseCalibrationSettingMedium and JeolCondenserCoarseCalibrationSettingHigh condenser lens values. Note that as the condenser lens values are decreased the beam current increases, however the displayed value for the 8200 in the HP software is inverted. The following is a typical (internal) response curve for the 8200 and 8900:

#### **8200:**

```
JeolCondenserCoarseCalibrationSettingLow=20, JeolCondenserCoarseCalibrationBeamLow=55.13
JeolCondenserCoarseCalibrationSettingMedium=35, JeolCondenserCoarseCalibrationBeamMedium=7.9
JeolCondenserCoarseCalibrationSettingHigh=45, JeolCondenserCoarseCalibrationBeamHigh=1.68
```

#### **8900:**

```
JeolCondenserCoarseCalibrationSettingLow=20, JeolCondenserCoarseCalibrationBeamLow=203
JeolCondenserCoarseCalibrationSettingMedium=35, JeolCondenserCoarseCalibrationBeamMedium=25.4
JeolCondenserCoarseCalibrationSettingHigh=55, JeolCondenserCoarseCalibrationBeamHigh=0.931
```

The allowable range of values are:

```
JeolCondenserCoarseCalibrationBeamLow > 0, JeolCondenserCoarseCalibrationBeamLow < 1000
JeolCondenserCoarseCalibrationBeamMedium > 0, JeolCondenserCoarseCalibrationBeamMedium < 1000
JeolCondenserCoarseCalibrationBeamHigh > 0, JeolCondenserCoarseCalibrationBeamHigh < 1000
```

*Note: as of 09/21/2008 PROBE supports multiple apertures for the JEOL 8900/8200/8900/8500. Normally 8900 and 8200 instruments always use aperture 1 (the default). However, some 8500 instruments that are capable of high beam currents are configured to use aperture 2 as the default aperture. In cases where more than one aperture needs to be defined, simply indicate the number of apertures to be defined and add additional comma separated values on each line for each aperture.*

*In cases where multiple apertures are defined, the first value on each line indicates the lens setting and beam values for aperture 1 and the second value (comma separated) indicates the values for aperture 2 (and so on).*

For the JEOL 8230/8530 the condenser lens values are opposite the 8900/8200. Therefore low condenser lens values correlate with low beam currents and high condenser lens values correlate with high beam current values. The following is an example of a 4 aperture calibration for a JEOL 8530:

#### **8230/8530:**

```
JeolCondenserCoarseCalibrationSettingLow=65,71,78,80 ; CC high current ~several hundred nA
JeolCondenserCoarseCalibrationSettingMedium=40,57,65,75 ; CC medium current ~25nA
JeolCondenserCoarseCalibrationSettingHigh=36,43,36,36 ; CC low current ~ 1nA
```

|   |   |
|---|---|
| JeolCondenserFineCalibrationSetting=255                           | ; middle of fine cl range is 255 for 8530                   |
| JeolCondenserCoarseCalibrationMode= 1                             | ; use pre-calibrated calibration curve                      |
| JeolCondenserCoarseCalibrationBeamLow=609.9,107.71,111.71,37.85   | ; low condenser value beam current                          |
| JeolCondenserCoarseCalibrationBeamMedium=13.836,10.2,10.354,14.09 | ; medium condenser value beam current                       |
| JeolCondenserCoarseCalibrationBeamHigh=7.491,1.1001,.10031,.023   | ; high condenser lens value beam current                    |
| JeolCondenserNumberOfApertures=4                                  | ; number of sets of probe current calibration data expected |
| JeolCoarseCondenserCalibrationDelay=1.0                           |   |

The JeolCoarseCondenserCalibrationDelay parameter is used to insert a small delay (between 0 and 2 seconds) during the coarse condenser calibration for lens hysteresis effects. The default is 0.1 seconds (100 msec). For 8230/8530 instruments, use a value of 1 or 2 seconds.

#### **SampleExchangePositionX=44.5**

#### **SampleExchangePositionY=1**

#### **SampleExchangePositionZ=11**

#### **SampleExchangePositionW=1**

These values define the stage position for performing a sample exchange. The values are defaulted to the above values for the JEOL (direct) interface and to X=0, Y=LoLimit, Z=0 for the Cameca SX100/SXFive.

#### **ReflectedLightIntensity=63**

#### **TransmittedLightIntensity=63**

These values define the default optical light intensities for the reflected and transmitted light optics. The default is 32 for the Cameca SX100/SXFive and 63 for the JEOL 8900/8200/850/8x30. The allowable range is 0 to 64 for the Cameca SX100/SXFive and 0 to 127 for the JEOL 8900/8200/8500/8x30.

#### **DisableSpectrometerNumber=0**

This value allows the user to specify a single spectrometer to be disabled on the instrument. This can be useful in the case of a non-functional spectrometer that is preventing the instrument from being used at all. Default is zero for no disabled spectrometer, allowable values are from zero to NumberofTunableSpecs.

#### **SpectrometerROMScanMode=0 ; SX100/SXFive only, 0 = absolute scan, 1 = relative scan**

This flag determines whether the Sx100/SXFive utilizes an absolute or relative spectrometer ROM scan function call for spectrometer scanning. 0 = absolute position (default) and 1 = relative position.

Note that the older SX100/SXFive instruments (spectro board type = "old") only support ROM scanning from low to high spectrometer positions (otherwise the scan will start and run but no data is returned). This means that the MOTORS.DAT backlash factors MUST be negative for these older SX100 instruments. For newer SX100 instruments (spectro board type = "new") the scan can be run in either direction (backlash factors can be negative or positive).

#### **FilamentWarmUpInterval=2.0**

This interval in seconds defines the amount of time delay between heat steps during the filament warmup. The default is 2 seconds and the allowable range is from 0.1 to 1000 seconds. This parameter applies only to the SX100/SXFive and JEOL 8900/8200 and 8500 interfaces.

#### **TurnOffSEDetectorBeforeAcquisition=0**

This flag tells the software whether to turn off the SE detector before each quantitative (standard or unknown) or qualitative (wavescan) acquisition. The default is zero to not turn off the SE detector before acquisition. Set it to one to have the program turn off the SE detector before each acquisition.

#### **AutomationOverheadPerAnalysis=10.0**

This parameter allows the user to override the default automation overhead for each single automated analysis to adjust the accuracy of the estimated automation time (e.g., for overnight runs). The default is 10 seconds and the valid range is from 0.1 seconds to 100.0 seconds.

**ReflectedLightPresent=0**

**TransmittedLightPresent=0**

These parameters specify if the reflected light optics or transmitted light optics hardware is interfaced. The default is 0 (false) for all interfaces except for the JEOL 8900/8200/8500/8x30 and Cameca SX100/SXFive interfaces where it is 1 (true).

**HysteresisPresent=0**

This parameter specifies whether the hardware supports a call to perform a hysteresis nulling to the objective lens. If this flag is set to a non-zero value the software will send a hysteresis command to the instrument after the beam size is set.

Currently only Cameca SX100 and SXFIVE instruments are supported.

**SX100MoveSpectroMilliSecDelayBefore=100**

**SX100MoveSpectroMilliSecDelayAfter=10**

**SX100MoveStageMilliSecDelayBefore=100**

**SX100MoveStageMilliSecDelayAfter=10**

**SX100ScanSpectroMilliSecDelayBefore=200**

**SX100ScanSpectroMilliSecDelayAfter=200**

**SX100FlipCrystalMilliSecDelayBefore=200**

**SX100FlipCrystalMilliSecDelayAfter=200**

These keywords allow the user to specify various delays before and after spectro and stage moves and before and after spectrometer ROM scans. These are apparently necessary for some SX100/SXFive instruments that utilize the PFE software backlash feature and SXFive instruments for the ROM scanning. The symptoms are occasional “axis busy” or “task creation” errors.

The crystal flip delays are necessary for the SXFive and should be 200 millisecs.

The values might have to be increased slightly for some instruments to 200 or 300 millisecs.

**MinMagWindow=40**

**MaxMagWindow=12000000**

These keywords determine the minimum and maximum magnification available on the instrument. The default is 40/12000K for the JEOL and 63/12000K for the Cameca.

**ImageShiftPresent=0**

**ImageShiftType=0**

These keywords are used to specify if image shift hardware is supported. Default = True for JEOL 8900/8200/8500/8230/8530 and Cameca SX100/SXFive. The ImageShiftType is not currently utilized.

**CLSpectraInterfacePresent=0**

**CLSpectraInterfaceType=0**

**CLInterfaceInsertRetractPresent=0**

These keywords are to specify the CL spectrum acquisition interface for Probe for EPMA. To indicate a CL interface is present, change CLSpectraInterfacePresent to any non-zero number. The following CL interfaces will be supported:

0 = Demo CL

1 = Ocean Optics (OmniDrive driver)

2 = Gatan

3 = Newport

4 = Avantes (driver v. 8.3 or later)

#### **ThermoNSSVersionNumber="3.0"**

This keyword defines the Thermo NSS application version number. Probe for EPMA needs to know this in order to ask whether you can specify an NSS project file for use as a template for EDS quantification.

With the release of Thermo PathFinder software, version 1.x of PathFinder should be considered version 5 with regard to the ThermoNSSVersionNumber keyword in the Probewin.ini file. Therefore to utilize the new beam deflection API in PathFinder from Probe for EPMA, edit the ThermoNSSVersionNumber keyword to 5.

#### **MoveStageToleranceX=0.1**

#### **MoveStageToleranceY=0.1**

#### **MoveStageToleranceZ=0.1**

These keywords define the minimum stage distance required to initiate a stage move for the X, Y and Z stage axes in microns. If the stage move for the given axis is less than the move stage tolerance (or all three for the JEOL stage), the stage axis move will not be initiated. The default is 0.1 microns for each axis.

#### **InsertFaradayDuringStageJogFlag=0**

This flag (if set to a non-zero value) instructs the software to insert the faraday cup when performing a stage jog (if it is not already inserted).

### **[Image]**

ImageInterfacePresent=0

ImageInterfaceType=0

ImageInterfaceNameChan1="SE"

ImageInterfaceNameChan2="BSE"

ImageInterfaceNameChan3="CL"

ImageInterfacePolarityChan1=0

ImageInterfacePolarityChan2=0

ImageInterfacePolarityChan3=0

ImageInterfaceImageIxIy=1.0

ImageInterfaceCalNumberOfBeamCalibrations=1

ImageInterfaceCalKeV="15.0"

ImageInterfaceCalMag="400.0"

ImageInterfaceCalXMicrons="805.0"

ImageInterfaceCalYMicrons="803.0"

ImageInterfaceCalScanRotation="0.0"

ImageInterfaceBeamXPolarity=0

ImageInterfaceBeamYPolarity=0

ImageInterfaceStageXPolarity=0

ImageInterfaceStageYPolarity=0

ImageInterfaceDisplayXPolarity=0

ImageInterfaceDisplayYPolarity=0

ImagePaletteNumber=1

ImageDisplaySizeInCentimeters=38.0

ImageAutoBrightnessContrastSEGain=350

ImageAutoBrightnessContrastSEOffset=480

ImageAutoBrightnessContrastBSEGain=350

ImageAutoBrightnessContrastBSEOffset=480

ImageAlternateScaleBarUnits=0

ImageShiftMinimumMag=1267

ImageMosaicSizeX="10.0"

ImageMosaicSizeY="10.0"

**ImageMosaicMag="400"**

**ImageRGB1\_R="Fe"**  
**ImageRGB1\_G="Mg"**  
**ImageRGB1\_B="Ca"**

**ImageInterfacePresent=0**

This parameter indicates if an imaging interface is available for use by **Probe for EPMA** in digitizing stage positions graphically. A non-zero value indicates that the imaging interface is available. The default is zero for no imaging interface.

**ImageInterfaceType=0**

This parameter indicates the type of imaging interface available. A value of zero is the demonstration imaging interface. The default is zero for the demonstration imaging interface. The current interfaces that are defined are:

**0 = Demonstration imaging interface**

**1 = Unused**

**2 = Unused**

**3 = Unused**

**4 = JEOL 8200/8500, 8900, 8230/8530 interface (TCP/IP) imaging interface**

**5 = SX100/SXFive (TCP/IP) imaging interface**

**6 = SX100/SXFive Video (TCP/IP) imaging interface (normally used for analog imaging)**

**7 = JEOL OEM EDS/Imaging interface (TCP/IP)**

**8 = Unused**

**9 = Bruker Quantax Server**

**10 = Thermo TE Portal Server**

**ImageInterfaceNameChan1="SE"**

**ImageInterfaceNameChan2="BSE"**

**ImageInterfaceNameChan3="CL"**

This keywords are used to indicate the signal types for the analog input channels. Usually either "SE" or "BSE" signals are used for image digitizing purposes. Leave empty to disable the channel.

**ImageInterfacePolarityChan1=0**

**ImageInterfacePolarityChan2=0**

**ImageInterfacePolarityChan3=0**

These keywords specify whether the analog signal obtained from the image interface is inverted or not. A normal signal (ImageInterfacePolarityChan1=0) is inverted, that is black = 255 and white = 0. If ImageInterfacePolarityChan1 is set to a non-zero number, then the analog signal is treated as non-inverted, that is black = 0 and white = 255.

**ImageInterfaceImageIxIy=1.0**

This parameter is used to specify the aspect ratio of X over Y, for the normal beam scan generated by the imaging interface. A value of one is the default and would specify a square beam scan. A value greater or less than one would specify a rectangular beam scan. Normally the SX100/SXFive Video interface is a 4/3 ratio (or 1.333), so for example 1024 x 768.

**ImageInterfaceCalNumberOfBeamCalibrations=1**

This new field specifies the number of beam scan calibrations that are utilized by the software to calibrate the beam scan images. The minimum (default) is 1, the maximum is 32.

Basically the beam scan calibration is based on the current keV and magnification and returns the X or Y scan width or height and the scan rotation. If only one beam scan calibration is specified, the application always utilizes that value adjusted for the current magnification assuming a linear 1:1 relationship in magnification.

If more than one beam scan calibration is specified, the values must be separated by commas and enclosed in double quotations as shown here:

```
ImageInterfaceCalNumberOfBeamCalibrations=2
ImageInterfaceCalKeV="15.0,15.0"
ImageInterfaceCalMag="400.0,800.0"
ImageInterfaceCalXMicrons="805.0,402.5"
ImageInterfaceCalYMicrons="803.0, 401.5"
ImageInterfaceCalScanRotation="0.0,0.1"
```

The program will search for matching keV calibrations. If none are found it will use the first beam scan calibration. If only one is found to match the specified keV, it will perform a linear extrapolation based on a 1:1 relationship in magnification.

Otherwise, if multiple calibrations are found the program will find the two nearest neighbor beam scan calibrations for the specified keV and interpolate them.

#### **ImageInterfaceCalKeV="15.0"**

This parameter specifies the operating voltage in kilovolts used for the ImageInterfaceCalXMicrons and ImageInterfaceYMicrons calibration. The default is 15 and the minimum is 1 and maximum is 50.

#### **ImageInterfaceCalMag="400.0"**

This parameter specifies the magnification used for the ImageInterfaceCalXMicrons and ImageInterfaceYMicrons calibration. The default is 400 and the minimum is 10 and maximum is 10000.

#### **ImageInterfaceCalXMicrons="805.0"**

#### **ImageInterfaceCalYMicrons="803.0"**

These parameters specify the actual X and Y beam scan size at the kilovolt and magnification parameters specified above. These parameters are used to calculate the stage position of the beam deflection. The default ImageInterfaceCalXmicrons is 800 and the default ImageInterfaceCalYmicrons is 800 and the minimum is 10 and maximum is 10000.

#### **ImageInterfaceCalScanRotation="0.0"**

This parameter specifies the returned or interpolated scan rotation for then specified keV and magnification. The default is 0 and the minimum is 0 and maximum is less than 360.

#### **ImageInterfaceBeamXPolarity=0**

#### **ImageInterfaceBeamYPolarity=0**

#### **ImageInterfaceStageXPolarity=0**

#### **ImageInterfaceStageYPolarity=0**

#### **ImageInterfaceDisplayXPolarity=0**

#### **ImageInterfaceDisplayYPolarity=0**

These six parameters specify the polarity of the image display, beam deflection and stage registration coordinate systems. That is, the display polarity for the orientation of the displayed image, the beam polarity for the beam deflection polarity and the stage polarity for the stage registration polarity. The default is zero for normal (graphical) registration using lower left as minimum and upper right as maximum (SX100/SXFive default). JEOL microprobes use a reversed Cartesian reference system. Any non-zero value will re-orient the beam, stage or display registration.

#### **Typical Settings for Image Orientation based on Imaging Interface Type:**

**ImageInterfaceType= 0 Demonstration Mode (usually JEOL orientation)**

**ImageInterfaceBeamXPolarity=1 ; depends on emulation**

**ImageInterfaceBeamYPolarity=0 ; depends on emulation**

|   |                        |
|---|------------------------|
| <b>ImageInterfaceStageXPolarity=1</b>   | ; depends on emulation |
| <b>ImageInterfaceStageYPolarity=1</b>   | ; depends on emulation |
| <b>ImageInterfaceDisplayXPolarity=1</b> | ; depends on emulation |
| <b>ImageInterfaceDisplayYPolarity=1</b> | ; depends on emulation |

**ImageInterfaceType= 4 JEOL Imaging**

|   |
|---|
| <b>ImageInterfaceBeamXPolarity=1</b>    |
| <b>ImageInterfaceBeamYPolarity=0</b>    |
| <b>ImageInterfaceStageXPolarity=1</b>   |
| <b>ImageInterfaceStageYPolarity=1</b>   |
| <b>ImageInterfaceDisplayXPolarity=1</b> |
| <b>ImageInterfaceDisplayYPolarity=1</b> |

**ImageInterfaceType= 5 Cameca SX100/SXFive Imaging**

|   |
|---|
| <b>ImageInterfaceBeamXPolarity=0</b>    |
| <b>ImageInterfaceBeamYPolarity=1</b>    |
| <b>ImageInterfaceStageXPolarity=0</b>   |
| <b>ImageInterfaceStageYPolarity=0</b>   |
| <b>ImageInterfaceDisplayXPolarity=0</b> |
| <b>ImageInterfaceDisplayYPolarity=0</b> |

**ImageInterfaceType= 6 Cameca SX100/SXFive Video Imaging**

|   |
|---|
| <b>ImageInterfaceBeamXPolarity=0</b>    |
| <b>ImageInterfaceBeamYPolarity=1</b>    |
| <b>ImageInterfaceStageXPolarity=0</b>   |
| <b>ImageInterfaceStageYPolarity=0</b>   |
| <b>ImageInterfaceDisplayXPolarity=0</b> |
| <b>ImageInterfaceDisplayYPolarity=0</b> |

#### **ImagePaletteNumber=1**

The default palette used for image display can be specified here. The options are:

- 0      Use grayscale palette (calculated)
- 1      Use palette file "THERMAL.FC"
- 2      Use palette file "RAINBOW.FC"
- 3      Use palette file "BLUERED.FC"
- 4      Use palette file "CUSTOM.FC"

Note that the CUSTOM.FC file is not supplied but is calculated if missing. However this file, once generated, can be modified by using the False Color dialog in MicroImage to any palette desired by the user. The other false color palette files should not be modified by the user.

#### **ImageDisplaySizeInCentimeters=38.0**

This parameter specifies the size of the display image in **Probe for EPMA**. It is utilized to calculate magnification only for the SX100/SXFive instrument which does not utilize magnification but rather a “field of view parameter” (FOV) which is normally scaled to a display size of 38cm (15 inches) by default (100x = 3800um FOV).

**ImageAutoBrightnessContrastSEGain=350**  
**ImageAutoBrightnessContrastSEOffset=480**  
**ImageAutoBrightnessContrastBSEGain=350**  
**ImageAutoBrightnessContrastBSEOffset=480**

These parameters are used to set starting values for the automatic brightness/contrast video adjustment. The default values are 350 for the contrast (gain) and 480 for the offset (brightness) for the SX100/SXFive.

Lower the Offset value to automatically increase the PM value (gain).

#### **ImageAlternateScaleBarUnits=0**

Specify the alternate scale bar unit label for the image display dialog. The default is zero for no alternate scale bar unit display. Specify values 1 through 8 for other units (in addition to um or microns).

1 = nm, 2 = um, 3 = mm, 4 = cm, 5 = meters, 6 = microinches, 7 = milliinches, 8 = inches

#### **ImageShiftMinimumMag=1267**

This is the minimum magnification for which the image shift values can be set and applies only to the Cameca Sx100/SXFive instruments. It is actually the magnification which the first scan coil changes to the second scan coil.

The default is 1267, but should be edited for the actual value, e.g., 1301.

#### **ImageMosaicSizeX="10.0"**

#### **ImageMosaicSizeY="10.0"**

#### **ImageMosaicMag! = "400."**

These fields are to specify the initial area of the mosaic imaging area and the beam scan magnification for acquisition of BSE, SE, etc. image mosaics over large areas in the Stage.exe application.

The units for the stage size is in stage units (mm for JEOL and um for Cameca). The defaults are 10 mm for JEOL and 10,000 um (10 mm) for Cameca.

#### **ImageRGB1\_R="Fe"**

#### **ImageRGB1\_G="Mg"**

#### **ImageRGB1\_B="Ca"**

These keywords define the default RGB elements for creating RGB mixed images from raw or quant data in CalcImage. The defaults are Red for Fe, Green for Mg and Blue for Ca.

### **[Serial]**

Port=1

HandShaking=1

Baud=9600

Parity="N"

DataBits=8

StopBits=1

These serial port parameters are obsolete and no longer utilized.

#### **Port=1**

The serial communication port used for hardware interfacing to the microprobe hardware. Allowable serial communication port values are 1 - 4. The default is 1 for COM1.

#### **HandShaking=1**

HandShaking defines the data flow control. The allowable values are :

**0 = no handshaking**

**1 = XON/XOFF**

**2 = RTS**

**3 = RTS and XON/XOFF**

The default is 1 for XON/XOFF handshaking data flow.

#### **Baud=9600**

The serial port baud rate. The allowable values are : 1200, 2400, 4800, 9600 and 19200. The default is 9600 for 9600 baud.

**Parity="N"**

The serial port parity. The allowable values are :

"N" = **none**  
"E" = **even**  
"O" = **odd**  
"M" = **mark**  
"S" = **space**

The default is N for no parity checking on the serial port.

**DataBits=8**

The serial port data bits. The allowable values are 4 or 8. The default is 8 for 8 databits.

**StopBits=1**

The serial port stop bits. The allowable values are 1 or 2. The default is 1 for 1 stop bit.

**[Faraday]**

FaradayCountAverages=3.0  
FaradayCupType=0  
FaradayWaitInTime=0.2  
FaradayWaitOutTime=0.2  
AbsorbedCurrentPresent=0  
AbsorbedCurrentType=0  
DefaultBlankBeamFlag=1  
MinimumFaradayCurrent=1.0  
FaradayBeamCurrentSafeThreshold=500.0  
FaradayStagePresent=0  
FaradayStagePositionsX=0.0  
FaradayStagePositionsY=0.0  
FaradayStagePositionsZ=0.0  
FaradayStagePositionsW=0.0

**FaradayCountAverages=5.0**

The FaradayCountAverages specifies the number of beam current measurements to average. In this case, the default is 1 for one beam current average. Note that the FaradayCountAverages value is also used for the Absorbed Current measurement (if indicated).

**FaradayCupType=0**

This option specifies the faraday cup type. It must be set to zero for automatic recording of the beam current. For manual entry of the beam current (some older SEM instruments), change this value to any non-zero value. In this case, the program will prompt the user for the beam current value. The default is zero for automatic beam current measurement.

**FaradayWaitInTime=0.5**

Specifies the amount of time in seconds to wait after inserting the faraday cup before starting the beam current acquisition. The allowable range is 0 to 100 seconds. The default value is 0.5 seconds.

**FaradayWaitOutTime=0.5**

Specifies the amount of time in seconds to wait after removing the faraday cup before starting the x-ray count acquisition. The allowable range is 0 to 100 seconds. The default value is 0.5 seconds.

**AbsorbedCurrentPresent=0**

This Boolean option specifies whether an absorbed current interface and associated hardware is present on the microprobe. If non-zero, the program will automatically acquire the absorbed current along with the faraday cup beam current.

#### **AbsorbedCurrentType=0**

This option specifies the absorbed current type. It is not used at this time.

#### **DefaultBlankBeamFlag=1**

This parameter is used to specify the default beam blank mode. Enter 0 for false or any non-zero value for true. If set to a non-zero value, the program will automatically leave the beam blanked after a stage motion command. If 0, the program will ask the user to confirm the beam unblank operation and then unblank the beam. The default is 1 to insert the faraday cup or blank the beam as the default.

#### **MinimumFaradayCurrent=1.0**

This parameter defines the threshold that the program uses to decide if the filament may have blown. The default threshold is 0.1 nA. The range of allowable values is 0.001 to 1000.

#### **FaradayBeamCurrentSafeThreshold=500.0**

This option sets the maximum safe threshold for the beam current so as to force the software to confirm with the user that it is safe to remove the faraday cup (or unblank) the beam. The default is 500 nA and the allowable range is 1 to 10,000 nA. Set this value lower to have the software confirm a manual faraday remove at lower currents.

#### **FaradayStagePresent=0**

This flag tells the software if a specimen (stage) mounted faraday cup is present. If this device is present a beam current reading will be made using the sample (specimen) current. Also the program will move the stage to the faraday stage position for each beam current measurement. Any non-zero value indicates that the faraday stage is present.

#### **FaradayStagePositionsX=0.0**

#### **FaradayStagePositionsY=0.0**

#### **FaradayStagePositionsZ=0.0**

#### **FaradayStagePositionsW=0.0**

These parameters allow the user to specify the default stage position (in x, y, z, (and w) ) for the specimen faraday measurement. These values can be updated in the program. These parameters must be in bounds for the x, y, z, (and w) positions.

### **[Counting]**

OnPeakCountTime=10.0

OffPeakCountTime=5.0

PeakingCountTime=8.0

WavescanCountTime=6.0

QuickscanCountTime=0.5

UnknownMaxCounts=100000000

#### **OnPeakCountTime=10.0**

This option specifies the default on peak counting time for both standard and unknown quantitative samples. The allowable range is 0.01 to 1000 seconds. The default value is 10 seconds.

#### **OffPeakCountTime=5.0**

This option specifies the default high and low off-peak counting time for both standard and unknown quantitative samples. The allowable range is 0.01 to 1000 seconds. The default value is 5 seconds.

#### **PeakingCountTime=8.0**

This option specifies the default spectrometer peak counting time for the **Probe for EPMA** interval halving, parabolic or ROM based peak methods. If a spectrometer pre-scan is specified, then **Probe for EPMA** uses a count time of 1/4 of the PeakingCountTime for the spectrometer wavelength pre-scan acquisition. The allowable range is 0.01 to 1000 seconds. The default value is 8 seconds.

**WavescanCountTime=6.0**

This option specifies the default spectrometer wavelength scan counting time for the **Probe for EPMA** for normal wavescans. The allowable range is 0.01 to 1000 seconds. The default value is 6 seconds.

**QuickscanCountTime=0.5**

This option specifies the default spectrometer wavelength scan counting time for the **Probe for EPMA** for "quick" spectrometer wavescans. The allowable range is 0.01 to 1000 seconds. The default value is 0.5 seconds. Note the following parameters apply to non-ROM based and ROM based quick scan parameters:

**Non-ROM scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

**ROM scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

**UnknownMaxCounts=100000000**

This option specifies the default maximum counts for unknown samples (that is precision based counting). The allowable range is 1000 to 100000000 seconds. The default value is 100000000 counts.

**[PHA]**

PHAHardwarePresent=1

PHAHardwareType=0

PHAGainBiasPresent=0

PHAGainBiasType=0

PHAInteDiffPresent=0

PHAInteDiffType=0

PHADeadTimePresent=0

PHADeadTimeType=0

PHACountTime=0.5

PHAIrradiation=20

PHAAdjustPresent=0

BiasScanCountTime=0.5

BiasScanIntervals=40

GainScanCountTime=0.5

GainScanIntervals=30

PHAFirstTimeDelay=0.0

PHAMultiChannelMin=0.805 ; Cameca MCA PHA minimum x-axis voltage

PHAMultiChannelMax=5.637 ; Cameca MCA PHA maximum x-axis voltage

**PHAHardwarePresent=0**

This Boolean option specifies whether PHA baseline and window voltage control hardware is implemented. Enter 0 for none or any non-zero value if present. The default is 0 for no PHA baseline and window hardware interface.

**PHAHardwareType=0**

Specifies the PHA acquisition type. The default is zero for the traditional PHA acquisition (scan baseline and window voltages across the voltage range). If PHA multi-channel analyzer hardware is available (JEOL 8230/8530 and Cameca SX100/SXFive), set PHAHardwareType to "1" for fast MCA PHA acquisition.

**PHAHardwareType = 0 for traditional PHA hardware acquisition (the default)**

**PHAHardwareType = 1 for PHA acquisition using multi-channel analyzer (MCA)**

**PHAGainBiasPresent=0**

This Boolean option specifies whether PHA gain and bias voltage control hardware is implemented. Enter 0 for none or any non-zero value if present. Note that the Gain and Bias interface may be enabled even if the Baseline and Window interface is not. This is to allow the user to leave the PHA baseline and window parameters "wide open" and instead utilize the microprobe's gain and bias adjustments to keep the pulse height properly adjusted. This method is actually preferred for the best quantitative results.

**PHAGainBiasType=0**

Specifies the PHA gain and bias hardware type. Not used at this time.

**PHAInteDiffPresent=0**

This Boolean option specifies whether PHA integral/differential control hardware is implemented. Enter 0 for none or any non-zero value if present.

**PHAInteDiffType=0**

Specifies the PHA integral/differential hardware type. Not used at this time.

**PHADeadtimePresent=0 ! SX-50/51/100 only**

This Boolean option specifies whether PHA deadtime control hardware is implemented. Enter 0 for none or any non-zero value if present. This flag is usually set true if the hardware interface supports an "enforced" non-extendable deadtime on the system.

**PHADeadTimeType=0**

Specifies the PHA deadtime hardware type. Not used at this time.

**PHACountTime=.5**

Specified the default count time for PHA distributions. The valid range is .1 to 1000 seconds. The default is 0.5 seconds.

**PHAIrvals=5**

Specifies the default number of PHA measurements (intervals) for PHA distributions. The valid range is 5 to 500 intervals. The default is 20 intervals.

**PHAAjustPresent=0**

Use this flag to specify if the hardware interface supports automatic PHA adjustment. Currently only the SX100/SXFive interface supports auto PHA adjustment (not currently implemented).

**BiasScanCountTime=0.5**

**BiasScanIntervals=40**

**GainScanCountTime=0.5**

**GainScanIntervals=30**

These parameters are used to set the PHA bias and gain scans performed in the PHA dialogs. See the parameters defined in line 36-41 in the SCALERS.DAT file for more information.

**PHAFirstTimeDelay=0.0**

This parameter is used to delay the program after the PHA parameters are set for the very first time. This can be used to deal with problems in the detector count rate and bias amplification response time when a large bias voltage change is applied. The default value is 0.0 (no delay) and the range is zero to 100 seconds. Note that this delay is only applied after the first time the PHA bias is set within the program for each spectrometer.

**PHAMultiChannelMin=0.805** ; Cameca MCA PHA minimum x-axis voltage  
**PHAMultiChannelMax=5.637** ; Cameca MCA PHA maximum x-axis voltage

These parameters are for demo and Cameca interfaces only that support the multi-channel analyzer (MCA) PHA hardware. The default values are indicated above but might need to be modified to 0 and 5 volts if the instrument is a "newer" Sx100 or SXFive and has "dynamic baseline hardware".

### **[Plot]**

MinimumKLMDisplay=0.5  
GraphType=1  
GraphTypeWave=1  
NumberofForbiddenElements=8  
ForbiddenElements="43,61,84,85,86,87,88,89"

#### **MinimumKLMDisplay=0.01**

The default minimum intensity for display of KLM markers with wavescan data. To view fewer low intensity lines, increase this value. The allowable range is 0.01 to 10. The default value is 0.5.

#### **GraphType=1**

The default graph type specified in the Plot! window. The default value is 1 for a line graph (lines or lines and symbols), 0 for symbols only, 2 for Linear-Log plot and 3 for 3D plot (three axes).

#### **GraphTypeWave=1**

The default wavescan graph type specified in the Plot! window. The default value is 1 for a line graph (lines or lines and symbols), 0 for symbols only, 2 for Linear-Log plot.

#### **NumberofForbiddenElements=8**

**ForbiddenElements="43,61,84,85,86,87,88,89"**

These keywords specify elements that should never show up in the KLM markers. The default forbidden elements are Tc, Pm, Po, At, Rn, Fr, Ra, and Ac. You may add or delete elements from this list but be sure to properly indicate the number of elements in the list. Note that x-ray lines do not exist for H and He so they are automatically excluded from the KLM database.

### **[Standards]**

IncrementXForAdditionalPoints = 4  
IncrementYForReStandardizations = 6  
StandardPointsToAcquire=5  
StageBitMapCount=1  
StageBitMapFile="CalTech1.5.WMF"  
StageBitMapXmin = 0  
StageBitMapXmax = 32  
StageBitMapYmin = 0  
StageBitMapYmax = 50  
StandardPOSFileDirectory="C:\UserData\StandardPOSData"  
MatchStandardDatabase="DHZ.MDB"  
StandardCoatingFlag = 1 ; 0 = not coated, 1 = coated  
StandardCoatingElement = 6 ; assume carbon  
StandardCoatingDensity = 2.1  
StandardCoatingThickness = 200 ; in angstroms  
LoadFormulasFromStandardDatabase=0  
LoadCalculateOxygenFromStandardDatabase=0

**IncrementXForAdditionalPoints=4**

This option is used to specify the stage step size in microns for automatically adjusting the x-axis stage position, if the POSITION.MDB position database contains an insufficient number of digitized coordinates for a specific standard number. The allowable range is -100 to 100 microns. The default value is 0 microns.

**IncrementYForReStandardizations=6**

This option is used to specify the stage step size in microns for automatically adjusting the y-axis stage when the program is acquiring an additional standard set. The allowable range is -100 to 100 microns. The default value is 0 microns.

**StandardPointsToAcquire=5**

This option is used to specify the default number of data points to acquire on each standard sample when using the automation features. The allowable values are 1 - 50. The default is 5.

**StageBitMapCount=1**

This option is used to specify the number of stage bitmap files used for the display of the Stage Bit Map move window. This window, accessible from the Position Database window (see the Stage button) allows the user to indicate a stage position to move to by simply double-clicking a point on the stage bit map image. Up to MAXBITMAP% .WMF files may be specified on this line.

The remaining stage bitmap keywords in the INI file must contain this number of values separated by commas on each line. Up to MAXBITMAP% Windows Meta-Files may be utilized. Typically, several stage files are specified so that all typical sample mount configurations can be viewed by the user.

**StageBitMapFile="CalTech1.5.WMF"**

This line must contain exactly "StageBitMapCount" files separated by commas. The files must be Windows Meta-Files (.WMF). These files may be created using most Windows drawing programs. Up to MAXBITMAP% .WMF files may be specified on this line separated by commas.

Note that if more than one stage bitmap file is entered on this line, then all files must be enclosed in double quotes. For example : StageBitMapFile="SX50\_35.WMF,SX50\_12.WMF,SX50\_7.WMF".

**StageBitMapXmin=0**

**StageBitMapXmax=30**

**StageBitMapYmin=0**

**StageBitMapYmax=40**

This option is used to specify the x-axis and y-axis limits of the StageBitMapFile in stage coordinates. It is used to calibrate the stage image for purposes of motion and position sample plotting. The values should correspond to the corners of the image used for the StageBitMapFile. Note that these values are NOT necessarily the stage limits but instead the edges of the stage bit map file as displayed in the WMF file. If the user happens to click a region of the stage image that is outside the stage limits, the program will of course prevent the move.

The program requires that the min and max values be unequal. To achieve the correct polarity for the stage coordinates, the X or Y min and max values may be swapped in the INI file. Up to MAXBITMAP% .WMF coordinates may be specified on this line.

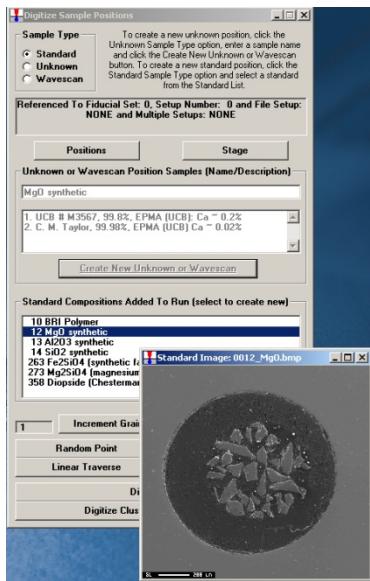
**StandardPOSFileDirectory="C:\UserData\StandardPOSData"**

This string specifies the folder to look for the standard \*.POS file for importing into (or exporting from) the POSITION.MDB database. This keyword replaces the older look up standard file names that were necessary with older 16 bit DOS and Windows file naming restrictions. Since the new file systems allow naming files up to 256 characters, simply rename your old standard .POS file to reflect what they contain. The string defaults to the C:\UserData\StandardPOSData directory if it is not specified.

A new feature is that the program displays BMP electron image files of the standards in the standard blocks. The program looks for any BMP files in the StandardPOSFileDirectory as defined in the PROBEWIN.INI file [Standards] section, (defaults to UserDataDirectory if not defined) and if it finds a match to any file (first 4 characters) of the standard number it loads it. For example standard MgO entered as 12 in the STANDARD.MDB database could be a file

called "0012\_MgO\_Block2.bmp" or "0012\_Periclase\_Block4.BMP". Both image files will be loaded during standard digitization (when the standard is clicked in the Digitize Sample Positions dialog ).

These BMP files should be placed in the Standard POS data folder as well, along with the \*.POS files, so the Probe for EPMA programs can find them when digitizing coordinates on your standards



This can be used to check that you are on the correct standard or the image could be annotated to suggest places or grains to avoid during the standard digitization or acquisition process.

#### **MatchStandardDatabase="DHZ.MDB"**

The MatchStandardDatabase specifies the default standard database to be used for compositional matching in the Standard.exe application and the Probewin.exe application in the Analyze! window. The user may select other databases from three Match dialog.

The DHZ.MDB database is supplied as the default match database (Deere, Howie and Zussman Rock Forming Minerals, student edition), but the SRM.MDB (NIST Standard Reference Materials) and the DANA.MDB (non-silicate minerals from Dana's Mineralogy) can also be specified. Note that the SRM.MDB and DANA.MDB standard databases must first be imported to a new database for use by the match feature.

The user may create additional standard databases as desired that are specific to their compositional matching requirements.

**StandardCoatingFlag = 1 ; 0 = not coated, 1 = coated**

**StandardCoatingElement = 6 ; assume carbon**

**StandardCoatingDensity = 2.1**

**StandardCoatingThickness = 200 ; in angstroms**

These parameters are the default parameters for the conductive coating on the standards. Values for all standards or individual standards can be edited in the program under the Standards menu. Note that these parameters are NOT used unless the user turns on this option for an unknown sample or group of unknown samples (see Calculation Options) from the Analytical | Analysis Options menu dialog.

#### **LoadFormulasFromStandardDatabase=0**

#### **LoadCalculateOxygenFromStandardDatabase=0**

These flags specify that the program load the formula basis and/or the calculate oxygen by stoichiometry parameters from the standard database.

Note that because there is no “Calculate Oxygen By Stoichiometry” flag in the standard database, the program will load the stoichiometric oxygen flag in Probe for EPMA based on the value of the “Display As Oxide” flag in the standard database.

### **[monitor]**

**ScanComboLabel1="Image Source"**

**ScanComboLabel2="Scan Mode"**

**ScanComboLabel3="Scan Speed"**

**ScanComboLabel4="EOS Mode"**

**ScanComboCommand1="IMS"**

**ScanComboCommand2="SM"**

**ScanComboCommand3="SS"**

**ScanComboCommand4="EM"**

**ScanComboNumberof1=10**

**ScanComboNumberof2=4**

**ScanComboNumberof3=5**

**ScanComboNumberof4=4**

**ScanComboNames1="SEI,COMPO,TOPO,AUX,XR1,XR2,XR3,XR4,XR5,EDS"**

**ScanComboNames2="PIC,CROSS,LSP,SPOT"**

**ScanComboNames3="TV,SR,S1,S2,S3"**

**ScanComboNames4="NOR,EMP,LDF,ECP"**

**ScanComboParameters1="SEI,COM,TOP,AUX,XR1,XR2,XR3,XR4,XR5,EDS"**

**ScanComboParameters2="PIC,BUP,LSP,SPT"**

**ScanComboParameters3="TV,SR,S1,S2,S3"**

**ScanComboParameters4="NOR,EMP,LDF,ECP"**

**ScanComboLabel1="Image Source"**

**ScanComboLabel2="Scan Mode"**

**ScanComboLabel3="Scan Speed"**

**ScanComboLabel4="EOS Mode"**

This section is utilized only by the MONITOR.EXE utility application which “monitors” the state of the JEOL 8900 and 8200/8500 microprobes. The utility could be adapted for other microprobe interfaces, but is currently only implemented for the JEOL interface.

The following parameters can be used to define four different drop down menus in the MONITOR.EXE application for setting various electron optical system parameters. The default settings are shown in the following examples, but they can be changed to suit the user’s particular preferences.

The ScanComboLabel[1,2,3,4] parameters are for defining the main menu labels as displayed in the program interface.

**ScanComboCommand1="IMS"**

**ScanComboCommand2="SM"**

**ScanComboCommand3="SS"**

**ScanComboCommand4="EM"**

The ScanComboCommand[1,2,3,4] parameters are for defining the actual commands to be sent by the program for each of the four menus.

**ScanComboNumberof1=10**

**ScanComboNumberof2=4**

**ScanComboNumberof3=5**

**ScanComboNumberof4=4**

The ScanComboNumberof[1,2,3,4] parameters are the number of list items in each menu list box.

**ScanComboNames1="SEI,COMPO,TOPO,AUX,XR1,XR2,XR3,XR4,XR5,EDS"**

**ScanComboNames2="PIC,CROSS,LSP,SPOT"**

**ScanComboNames3="TV,SR,S1,S2,S3"**

**ScanComboNames4="NOR,EMP,LDF,ECP"**

The ScanComboNames[1,2,3,4] are the names for each item in the list boxes as seen by the user in the program interface.

**ScanComboParameters1="SEI,COM,TOP,AUX,XR1,XR2,XR3,XR4,XR5,EDS"**

**ScanComboParameters2="PIC,BUP,LSP,SPT"**

**ScanComboParameters3="TV,SR,S1,S2,S3"**

**ScanComboParameters4="NOR,EMP,LDF,ECP"**

The ScanComboParameters are the parameters for defining the actual parameters sent with each command.

## ELEMENTS.DAT

The data file used to define element parameters. This file is normally not edited unless the default oxide cations need to be changed permanently. The file structure consists of 100 lines (one line per element for H to Fm). The lines MUST be in atomic number order!

Each element line contains the element atomic number, lower case symbol, upper case symbol, default x-ray line, default crystal name, atomic weight, default number of oxide cations and default number of oxide oxygens.

The default x-ray lines must be either "ka", "kb", "la", "lb", "ma" or "mb". These are the only allowable x-ray lines for analysis. Because the physical parameters of the beta series lines are not known with great accuracy, their use should be limited in quantitative analysis. In practice this could be of concern when spectral interferences are present. However, **Probe for EPMA** provides a powerful and completely quantitative spectral interference correction that can correct for interfering lines down to the trace level (Donovan, et al., 1993).

The default crystal names must correspond to a valid entry in the CRYSTALS.DAT file. The number of default oxide cations must be between 1 and 9 and the number of default oxide oxygens must be between 0 and 9.

|    |      |      |      |          |          |   |   |
|----|------|------|------|----------|----------|---|---|
| 1  | "h " | "H " | "ka" | "WC144"  | 1.00800  | 2 | 1 |
| 2  | "he" | "He" | "ka" | "WC144"  | 4.00300  | 1 | 0 |
| 3  | "li" | "Li" | "ka" | "WC144"  | 6.93900  | 2 | 1 |
| 4  | "be" | "Be" | "ka" | "WC144"  | 9.01200  | 1 | 1 |
| 5  | "b " | "B " | "ka" | "WC144"  | 10.81100 | 2 | 3 |
| 6  | "c " | "C " | "ka" | "NiCrBN" | 12.01100 | 1 | 2 |
| 7  | "n " | "N " | "ka" | "NiCrBN" | 14.00700 | 2 | 5 |
| 8  | "o " | "O " | "ka" | "NiCrBN" | 16.00000 | 1 | 0 |
| 9  | "f " | "F " | "ka" | "NiCrBN" | 18.99800 | 1 | 0 |
| 10 | "ne" | "Ne" | "ka" | "NiCrBN" | 20.17900 | 1 | 0 |
| 11 | "na" | "Na" | "ka" | "TAP"    | 22.99000 | 2 | 1 |
| 12 | "mg" | "Mg" | "ka" | "TAP"    | 24.30500 | 1 | 1 |
| 13 | "al" | "Al" | "ka" | "PET"    | 26.98200 | 2 | 3 |
| 14 | "si" | "Si" | "ka" | "PET"    | 28.08600 | 1 | 2 |
| 15 | "p " | "P " | "ka" | "PET"    | 30.97400 | 2 | 5 |
| 16 | "s " | "S " | "ka" | "PET"    | 32.06400 | 1 | 3 |
| 17 | "cl" | "Cl" | "ka" | "PET"    | 35.45300 | 1 | 0 |
| 18 | "ar" | "Ar" | "ka" | "PET"    | 39.94800 | 1 | 0 |
| 19 | "k " | "K " | "ka" | "PET"    | 39.10200 | 2 | 1 |
| 20 | "ca" | "Ca" | "ka" | "LiF"    | 40.08000 | 1 | 1 |
| 21 | "sc" | "Sc" | "ka" | "LiF"    | 44.95600 | 2 | 3 |
| 22 | "ti" | "Ti" | "ka" | "LiF"    | 47.90000 | 1 | 2 |
| 23 | "v " | "V " | "ka" | "LiF"    | 50.94200 | 2 | 3 |
| 24 | "cr" | "Cr" | "ka" | "LiF"    | 51.99600 | 2 | 3 |
| 25 | "mn" | "Mn" | "ka" | "LiF"    | 54.93800 | 1 | 1 |
| 26 | "fe" | "Fe" | "ka" | "LiF"    | 55.84700 | 1 | 1 |
| 27 | "co" | "Co" | "ka" | "LiF"    | 58.93300 | 1 | 1 |
| 28 | "ni" | "Ni" | "ka" | "LiF"    | 58.71000 | 1 | 1 |
| 29 | "cu" | "Cu" | "ka" | "LiF"    | 63.54600 | 2 | 1 |
| 30 | "zn" | "Zn" | "ka" | "LiF"    | 65.37000 | 1 | 1 |
| 31 | "ga" | "Ga" | "ka" | "LiF"    | 69.72000 | 2 | 3 |
| 32 | "ge" | "Ge" | "ka" | "LiF"    | 72.59000 | 1 | 2 |

|     |      |      |      |          |           |   |   |
|-----|------|------|------|----------|-----------|---|---|
| 33  | "as" | "As" | "la" | "LiF"    | 74.92200  | 2 | 3 |
| 34  | "se" | "Se" | "la" | "LiF"    | 78.96000  | 1 | 2 |
| 35  | "br" | "Br" | "la" | "LiF"    | 79.90400  | 2 | 5 |
| 36  | "kr" | "Kr" | "la" | "LiF"    | 83.80000  | 1 | 0 |
| 37  | "rb" | "Rb" | "la" | "PET"    | 85.47000  | 2 | 1 |
| 38  | "sr" | "Sr" | "la" | "PET"    | 87.62000  | 1 | 1 |
| 39  | "y"  | "Y"  | "la" | "PET"    | 88.90500  | 2 | 3 |
| 40  | "zr" | "Zr" | "la" | "PET"    | 91.22000  | 1 | 2 |
| 41  | "nb" | "Nb" | "la" | "PET"    | 92.90600  | 2 | 5 |
| 42  | "mo" | "Mo" | "la" | "PET"    | 95.94000  | 1 | 3 |
| 43  | "tc" | "Tc" | "la" | "PET"    | 99.00000  | 1 | 4 |
| 44  | "ru" | "Ru" | "la" | "PET"    | 101.07000 | 1 | 2 |
| 45  | "rh" | "Rh" | "la" | "PET"    | 102.90500 | 2 | 3 |
| 46  | "pd" | "Pd" | "la" | "PET"    | 106.40000 | 1 | 1 |
| 47  | "ag" | "Ag" | "la" | "PET"    | 107.86800 | 2 | 1 |
| 48  | "cd" | "Cd" | "la" | "PET"    | 112.40000 | 1 | 1 |
| 49  | "in" | "In" | "la" | "PET"    | 114.82000 | 2 | 1 |
| 50  | "sn" | "Sn" | "la" | "LiF"    | 118.69000 | 1 | 2 |
| 51  | "sb" | "Sb" | "la" | "LiF"    | 121.75000 | 2 | 3 |
| 52  | "te" | "Te" | "la" | "LiF"    | 127.60000 | 1 | 2 |
| 53  | "i"  | "I"  | "la" | "LiF"    | 126.90400 | 2 | 5 |
| 54  | "xe" | "Xe" | "la" | "LiF"    | 131.30000 | 1 | 0 |
| 55  | "cs" | "Cs" | "la" | "LiF"    | 132.91000 | 2 | 1 |
| 56  | "ba" | "Ba" | "la" | "LiF"    | 137.34000 | 1 | 1 |
| 57  | "la" | "La" | "la" | "LiF"    | 138.91000 | 2 | 3 |
| 58  | "ce" | "Ce" | "la" | "LiF"    | 140.12000 | 2 | 3 |
| 59  | "pr" | "Pr" | "la" | "LiF"    | 140.90700 | 2 | 3 |
| 60  | "nd" | "Nd" | "la" | "LiF"    | 144.24000 | 2 | 3 |
| 61  | "pm" | "Pm" | "la" | "LiF"    | 147.00000 | 2 | 3 |
| 62  | "sm" | "Sm" | "la" | "LiF"    | 150.35000 | 2 | 3 |
| 63  | "eu" | "Eu" | "la" | "LiF"    | 151.96000 | 2 | 3 |
| 64  | "gd" | "Gd" | "la" | "LiF"    | 157.25000 | 2 | 3 |
| 65  | "tb" | "Tb" | "la" | "LiF"    | 158.92400 | 2 | 3 |
| 66  | "dy" | "Dy" | "la" | "LiF"    | 162.50000 | 2 | 3 |
| 67  | "ho" | "Ho" | "la" | "LiF"    | 164.93000 | 2 | 3 |
| 68  | "er" | "Er" | "la" | "LiF"    | 167.26000 | 2 | 3 |
| 69  | "tm" | "Tm" | "la" | "LiF"    | 168.93400 | 2 | 3 |
| 70  | "yb" | "Yb" | "la" | "LiF"    | 173.04000 | 2 | 3 |
| 71  | "lu" | "Lu" | "la" | "LiF"    | 174.97000 | 2 | 3 |
| 72  | "hf" | "Hf" | "la" | "LiF"    | 178.49000 | 1 | 2 |
| 73  | "ta" | "Ta" | "la" | "LiF"    | 180.94800 | 2 | 5 |
| 74  | "w"  | "W"  | "la" | "LiF"    | 183.85000 | 1 | 3 |
| 75  | "re" | "Re" | "la" | "LiF"    | 186.20000 | 1 | 2 |
| 76  | "os" | "Os" | "la" | "LiF"    | 190.20000 | 1 | 2 |
| 77  | "ir" | "Ir" | "la" | "LiF"    | 192.20000 | 1 | 2 |
| 78  | "pt" | "Pt" | "la" | "LiF"    | 195.09000 | 1 | 1 |
| 79  | "au" | "Au" | "la" | "PET"    | 196.97000 | 2 | 3 |
| 80  | "hg" | "Hg" | "la" | "PET"    | 200.59000 | 1 | 1 |
| 81  | "tl" | "Tl" | "ma" | "PET"    | 204.37000 | 2 | 3 |
| 82  | "pb" | "Pb" | "ma" | "PET"    | 207.19000 | 1 | 1 |
| 83  | "bi" | "Bi" | "ma" | "PET"    | 208.98000 | 2 | 3 |
| 84  | "po" | "Po" | "la" | "LiF220" | 210.00000 | 1 | 1 |
| 85  | "at" | "At" | "la" | "LiF220" | 210.00000 | 1 | 1 |
| 86  | "rn" | "Rn" | "la" | "LiF220" | 222.00000 | 1 | 0 |
| 87  | "fr" | "Fr" | "la" | "LiF220" | 223.00000 | 2 | 1 |
| 88  | "ra" | "Ra" | "la" | "LiF220" | 226.00000 | 1 | 1 |
| 89  | "ac" | "Ac" | "la" | "LiF220" | 227.00000 | 2 | 3 |
| 90  | "th" | "Th" | "ma" | "PET"    | 232.03800 | 1 | 2 |
| 91  | "pa" | "Pa" | "ma" | "PET"    | 231.00000 | 2 | 5 |
| 92  | "u"  | "U"  | "ma" | "PET"    | 238.03000 | 1 | 2 |
| 93  | "np" | "Np" | "la" | "LiF220" | 237.00000 | 2 | 5 |
| 94  | "pu" | "Pu" | "la" | "LiF220" | 244.00000 | 1 | 2 |
| 95  | "am" | "Am" | "ma" | "PET"    | 243.00000 | 1 | 2 |
| 96  | "cm" | "Cm" | "ma" | "PET"    | 247.00000 | 1 | 2 |
| 97  | "bk" | "Bk" | "ma" | "PET"    | 247.00000 | 1 | 2 |
| 98  | "cf" | "Cf" | "ma" | "PET"    | 251.00000 | 1 | 2 |
| 99  | "es" | "Es" | "ma" | "PET"    | 254.00000 | 1 | 2 |
| 100 | "fm" | "Fm" | "ma" | "PET"    | 253.00000 | 1 | 2 |

## CHARGES.DAT

The data file used to define elemental atomic charge parameters. This file is normally not edited unless the default atomic charge defaults need to be changed permanently. The file structure consists of 100 lines (one line per element for H to Fm). The lines MUST be in atomic number order!

Each element line contains the element atomic number, upper case symbol, and the atomic charge. The atomic charge must be between -10 and +10.

## CRYSTALS.DAT

The data file used to define crystal parameters. The file structure consists of one line per crystal. Generally this file is edited to specify special analyzing crystals such as layered dispersive elements (LDEs). The maximum number of defined crystals is determined by the MaxCrysType parameter in **Probe for EPMA** (currently set to 60). Each line contains the crystal name, the 2d spacing, the crystal K reflection parameter, followed by the default peaking element and x-ray line (not used at this time).

The crystals can be defined in any order, although ordered by 2d spacing is most convenient for editing purposes.

The spectrometer position of a specific wavelength is calculated according to the following formula. Note that **Probe for EPMA** always assumes a 1st order line for the purposes of the calculation :

$$\sin \phi = \frac{N\lambda}{2d * \left(1 - \frac{K}{N^2}\right)}$$

Where :

N      is the order  
d      is the d spacing of the analyzing crystal  
 $\lambda$     is the wavelength  
K      is the crystal reflection factor

|          |        |          |      |      |
|----------|--------|----------|------|------|
| "LiF420" | 1.802  | 0.       | "fe" | "ka" |
| "LiF220" | 2.848  | 0.       | "fe" | "ka" |
| "LIF"    | 4.0267 | 0.000058 | "fe" | "ka" |
| "NaCl"   | 5.641  | 0.       | "fe" | "ka" |
| "Si"     | 6.2709 | 0.       | "ca" | "ka" |
| "Ge"     | 6.532  | 0.       | "ca" | "ka" |
| "QTZ011" | 6.686  | 0.       | "ca" | "ka" |
| "QTZ010" | 8.50   | 0.       | "ca" | "ka" |
| "PET"    | 8.750  | 0.000144 | "ca" | "ka" |
| "EDT"    | 8.808  | 0.       | "ca" | "ka" |
| "ADP"    | 10.648 | 0.000476 | "ca" | "ka" |
| "GYPSUM" | 15.19  | 0.       | "si" | "ka" |
| "MICA"   | 19.8   | 0.       | "si" | "ka" |
| "KAP"    | 26.63  | 0.       | "si" | "ka" |
| "RAP"    | 26.12  | 0.       | "si" | "ka" |
| "TAP"    | 25.745 | 0.00218  | "si" | "ka" |
| "WSi55"  | 55.0   | 0.       | "o"  | "ka" |
| "WSi60"  | 60.0   | 0.01483  | "o"  | "ka" |
| "WSi63"  | 63.0   | 0.       | "o"  | "ka" |
| "NiCrBN" | 65.0   | 0.       | "o"  | "ka" |
| "WC144"  | 144.0  | 0.       | "c"  | "ka" |
| "PbSTEA" | 100.6  | 0.       | "c"  | "ka" |
| "OV 95"  | 96.8   | 0.       | "c"  | "ka" |
| "OV 200" | 197.4  | 0.       | "b"  | "ka" |
| " "      | 0.     | 0.       | " "  | " "  |
| " "      | 0.     | 0.       | " "  | " "  |
| " "      | 0.     | 0.       | " "  | " "  |
| " "      | 0.     | 0.       | " "  | " "  |
| " "      | 0.     | 0.       | " "  | " "  |

```
" "
0.          0.          " "
" "
0.          0.          " "
" "
```

## MOTORS.DAT

The data file used to define motor parameters. The motor array size (the number of data values on each line) is defined as the sum of the "NumberOfTunableSpecs" and the "NumberOfStageMotors" as specified in the PROBEWIN.INI file. **Note that a comment string enclosed in double quotes, MUST be present at the end of each line.** Several unused parameter lines are at the end of the file for future expansion. Note that the tunable spectrometer motors must be listed first on each line, followed by the stage motors.

```
"SP1"   "SP2"   "SP3"   "SP4"   "SP5"   "X"     "Y"     "Z"     "motor labels"
60.4    82.4    60.4    60.4    60.4    1.5    1.8    9.0    "low limit spect/stage motors"
254.0   254.0   254.0   254.0   254.0   99.2   99.2   12.9   "high limit spect/stage motors hilim"
742.8646 742.8646 742.8646 742.8646 742.8646 27593.425 27593.425 27593.425 "unit to steps conversion"
.014381 .014381 .014381 .014381 .014381 1000.0 1000.0 1000.0 "unit to LiF200 angstrom or micron conversion"
5       5       5       5       5       20      20      20      "completion step tolerance for motors"
-400.   -400.   -400.   -400.   -400.   -400.   -400.   -400.   "backlash size, (hilimit - lolimit)/backlash size"
200    200    200    200    200    200    200    200    "derivative gain"
750    750    750    750    750    700    700    700    "integral gain"
700    700    700    700    700    700    700    700    "integration limit"
2000   2000   2000   2000   2000   2000   2000   2000   "following error"
20     20     20     20     20     300    300    300    "acceleration"
200000 200000 200000 200000 200000 400000 400000 400000 "velocity"
600    600    600    600    600    700    700    700    "gain"
11     11     11     11     11     11     11     11     "hard limit mode (low xor high xor smooth)"
1.0    1.0    1.0    1.0    1.0    1.0    1.0    1.0    "joystick sensitivity"
1.4    1.4    1.4    1.4    1.4    1.4    1.4    1.4    "joystick acceleration"
.002   .002   .002   .002   .002   .002   .002   .002   "backlash tolerance"
180.0  180.0  180.0  180.0  180.0  12     12     10     "park positions"
500000 500000 500000 500000 500000 400000 400000 40000  "JEOL velocities"
50000  50000  50000  50000  50000  -5000  -5000  5000  "Jeol Backlash"
0      0      0      0      0      0      0      0      "SX100 velocities"
0      0      0      0      0      0      0      0      "SX100 minimum speeds"
0      0      0      0      0      0      0      0      "unused"
```

### Line 1 (spectrometer and stage motor labels)

```
"1"   "2"   "3"   "4"   "X"   "Y"   "Z"   "motor labels"
```

The motor labels are used to load several dialog controls to allow the user to select a specific motor axis for a specified operation. The motor labels must be enclosed in double quotes.

### Line 2 (motor low limits)

```
60.4   82.4   60.4   60.4   60.4   1.5   1.8   9.0   "low limit spect/stage motors"
```

The motor low limit positions are used to define a software low limit before the electrical limit switch position has been reached. This is to prevent the user from accidentally moving to a position outside the normal range of motion. The motor low limits are defined in spectrometer and stage units.

### Line 3 (motor high limits)

```
254.0  254.0  254.0  254.0  254.0  99.2   99.2   12.9   "high limit spect/stage motors hilim"
```

The motor high limit positions are used to define a software high limit before the electrical limit switch position has been reached. This is to prevent the user from accidentally moving to a position outside the normal range of motion. The motor high limits are defined in spectrometer and stage units. The motor high limits must be greater than the motor low limits.

### Line 4 (not used at this time)

```
742.8646 742.8646 742.8646 742.8646 742.8646 27593.425 27593.425 27593.425 "unit to steps conversion"
```

### Line 5 (spectrometer and stage units to angstrom or micron conversion factors)

```
.014381 .014381 .014381 .014381 .014381 1000.0 1000.0 1000.0 "unit to LiF200 angstrom or micron conversion"
```

These parameters define the conversion factor for converting from spectrometer units to angstroms (assuming an LiF crystal present) or stage units to microns. The conversion factors must not be equal to zero.

Note that although almost any spectrometer units may be used (sin theta, L-units, Bragg angle, etc.) , the spectrometer units chosen by the user MUST be directly proportional to angstrom units. For this reason, the program cannot utilize energy units (for example keV) since these units are inversely proportional to angstrom units.

| Micropoke | Spectrometer | Stage  |
|-----------|--------------|--------|
| JEOL      | 0.014381     | 1000.0 |
| Cameca    | 4.0267 E-5   | 1.0    |

The spectrometer conversion factors are used to convert spectrometer position to angstroms for wavescan plotting. Note that the 100 mm Rowland circle JEOL spectrometer makes internal adjustments so that it reads out in 140 mm L-units, so use the same conversion factor as the JEOL 140 mm Rowland circle spectrometer (if this was \*not\* the case, the JEOL 100 mm Rowland circle would need a conversion factor of 0.0201335). The Cameca spectrometers (160 mm and 180 mm) read directly in sin theta units so they use the same readout also.

The stage motor conversion factors are used to calculate stage coordinates in microns. This allows the program to calculate output of traverse data in relative microns (accumulated number of microns between each data point). For example if the stage is read in mm units, the stage units to micron conversion factor will be 1000.

Note that some interfaces where the Rowland circle are variable, the conversion factor is calculated on the fly and this parameter is therefore only "nominal".

### ***Some other helpful expressions :***

JEOL L-Unit Crystal Conversion factors (d/R, where R = 140.0)

Angstroms = d/R \* Spectrometer Reading

Cameca Sin $\phi$  Crystal Conversion factors (2d)

Angstroms = 2d \* Spectrometer Reading

**Line 6**                    (not used at this time)

0                    0                    0                    0                    0                    0                    "unused"

**Line 7**                    (backlash size parameter)

400.                  400.                  400.                  400.                  400.                  400.                  "backlash size, (hilimit - lolimit)/backlash size"

These parameters define the size of the backlash motions used for the spectrometer and stage motors. The absolute value of each backlash size parameter must be greater than 10.0. Note that the larger the value, the smaller the resulting backlash motion will be. The calculation for the backlash size is :

$$B_{\text{Size}} = \frac{\text{ABS}(P_{\text{HiLim}} - P_{\text{LoLim}})}{B_{\text{factor}}}$$

Where :

B<sub>Size</sub>                    is the size of calculated backlash motion in motor units

P<sub>HiLim</sub>                    is the motor high limit

P<sub>LoLim</sub>                    is the motor low limit

B<sub>factor</sub>                    is the backlash size parameter for the motor

Note that if the backlash size parameter is negative, all wavelength scans will scan from the low end to the high end and, if the backlash size parameter is positive, all wavelength scans will scan from the high end to the low end. In addition, each motor (spectrometer or stage) can have either polarity. That is, one spectrometer can have a negative backlash, while the others have positive backlash values.

**Note that the older SX100 instruments (spectro board type = "old") only support ROM scanning from low to high spectrometer positions (otherwise the scan will start and run but no data is returned). This means that the MOTORS.DAT backlash factors MUST be negative for these older SX100 instruments. For newer SX100/SXFive instruments (spectro board type = "new") the scan can be run in either direction (backlash factors can be negative or positive).**

Normally the backlash factor for each motor should be selected to produce the smallest backlash size that can be tolerated to reduce the time required for the backlash correction.

Note that the program will attempt to determine if a backlash is required based on the last direction of motion for each motor. If the backlash factor is positive and the last motion was from a higher to a lower position and the size of the motion was larger than the calculated backlash size, then a backlash will not be performed on the next occurrence of the backlash adjustment for that motor.

**Line 8-15 (not used at this time)**

|   |   |   |   |   |   |   |          |
|---|---|---|---|---|---|---|----------|
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | "unused" |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | "unused" |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | "unused" |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | "unused" |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | "unused" |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | "unused" |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | "unused" |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | "unused" |

**Line 16-17 (not used at this time)**

|   |   |   |   |   |   |   |          |
|---|---|---|---|---|---|---|----------|
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | "unused" |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | "unused" |

**Line 18 (backlash tolerance)**

|      |      |      |      |      |      |      |                      |
|------|------|------|------|------|------|------|----------------------|
| .002 | .002 | .002 | .002 | .002 | .002 | .002 | "backlash tolerance" |
|------|------|------|------|------|------|------|----------------------|

These motor parameters are used to allow the user to "tune" the backlash tolerance which the program uses to decide whether to apply a "smart" backlash to the current move. The default is 0.002 (0.2%) and the values must be between 0.1 and 0.000001. Use a smaller value to have the program apply the backlash more often.

The backlash tolerance is also used to determine when the StageMap window picture should be refreshed. If the stage move is less than the backlash tolerance than the StageMap is not refreshed.

**Line 19 (park positions)**

|       |       |       |       |       |    |    |    |                  |
|-------|-------|-------|-------|-------|----|----|----|------------------|
| 180.0 | 180.0 | 180.0 | 180.0 | 180.0 | 12 | 12 | 10 | "park positions" |
|-------|-------|-------|-------|-------|----|----|----|------------------|

These motor parameters are used to set the "park" positions for the spectrometer and stage motors. The park spectrometer and park stage buttons are available from the Move window. If no position is entered (read zero), then the motor hilimit is used for the spectrometer park positions and the midway point between the hilimit and lolimit is used for the stage park positions.

The stage park position can be specified so as to be used for the instrument sample exchange position if desired.

**Line 20 (JEOL velocities)**

|        |        |        |        |        |        |        |       |                   |
|--------|--------|--------|--------|--------|--------|--------|-------|-------------------|
| 500000 | 500000 | 500000 | 500000 | 500000 | 200000 | 200000 | 20000 | "JEOL velocities" |
|--------|--------|--------|--------|--------|--------|--------|-------|-------------------|

The JEOL velocities are used to restore spectrometer and stage speeds to normal operating speeds after they have been reduced for special operations. Currently only used for spectrometer scanning for peaking and "quick" spectrometer scanning. The values must be specified in units of 1/100<sup>th</sup> micrometers per second. Must be between 2000 (.02mm/sec) and 500000 (5mm/sec). JEOL 8900 is limited to 400000 (4mm/sec). The default is 500000 (400000 for 8900) for spectrometers and 400000 for stage axes.

**Line 21 (JEOL backlash)**

|       |       |       |       |       |       |       |      |                 |
|-------|-------|-------|-------|-------|-------|-------|------|-----------------|
| 50000 | 50000 | 50000 | 50000 | 50000 | -5000 | -5000 | 5000 | "JEOL Backlash" |
|-------|-------|-------|-------|-------|-------|-------|------|-----------------|

The JEOL backlash values are for restoring the spectrometer and stage backlash values after they have been turned off for special operations. Currently only used for the auto-focus. The values must be specified in units of 1/100<sup>th</sup> micrometers. The defaults are 50000 (500um) for spectrometers and -5000 (-50um) for X and Y and 5000 (50um) for Z. Must be between 100 and 100,000.

**Line 22 (SX100 and SXFive motor velocities)**

|      |      |      |      |      |      |      |                    |
|------|------|------|------|------|------|------|--------------------|
| 5000 | 5000 | 5000 | 5000 | 2000 | 2000 | 2000 | "SX100 velocities" |
|------|------|------|------|------|------|------|--------------------|

The SX100 and SXFive velocities are used to restore spectrometer and stage speeds to normal operating speeds after they have been reduced for special operations. Currently only used for spectrometer scanning for peaking and "quick" spectrometer scanning. Must be between 1000 and 4000 for spectrometers and between 500 and 15000 for X and Y stage

motors and between 1 and 200 for the Z stage motor. The defaults are 3000 for spectrometers, 10000 for X and Y stage axes (this is slightly slower than the OEM default) and 50 for the Z stage axis.

Note: SX100 Z speed range is between 10 and 200, and SXFive Z speed range is between 1 and 10.

#### Line 23 (SX100 minimum speeds)

0 0 0 0 0 0 "SX100 minimum speeds"

The SX100 minimum speeds are used to determine how slow a spectrometer or stage motor can be driven. At this time, only the spectrometer ROM scanning is affected by this parameter. Note that some spectrometers will scan at very slow speeds down to 5 steps per second, while some spectrometers will only scan as low as 20 steps per second.

The default is 10 for spectrometers (must be between 2 and 1000) and for stage axes the default is 100 for X and Y and 5 for Z (must be between 10 and 1500 for X and Y and between 2 and 100 for Z).

#### Line 24 (not used at this time)

0 0 0 0 0 0 "unused"

## SCALERS.DAT

The data file used to define scaler parameters. The scaler array size (the number of data values on each line) is defined as the "NumberOfTunableSpecs" in the PROBEWIN.INI file. **Note that a comment string enclosed in double quotes MUST be present at the end of each line.** Several unused parameter lines are at the end of the file for future expansion.

The following example SCALERS.DAT is for an instrument with 4 tunable spectrometers.

```
"1"      "2"      "3"      "4"      "5"      "scaler labels"
" "      " "      " "      " "      " "      "fixed scaler elements"
" "      " "      " "      " "      " "      "fixed scaler x-rays"
2       2       2       2       2       "crystal flipping flag"
253.   253.   253.   253.   253.   "crystal flipping position"
2       2       4       2       2       "number of crystals"
"PET"   "LIFH"   "PET"   "PET"   "LIF"   "crystal types1"
"PETH"   "PETH"   "TAB"   "TAB"   "PET"   "crystal types2"
" "      "WSI60"   " "      " "      " "      "crystal types3"
" "      "NiCrBN"   " "      " "      " "      "crystal types4"
" "      " " "      " "      " "      "crystal types5"
" "      " " "      " "      " "      "crystal types6"
1.0    1.0    1.0    1.0    1.0    "deadtime in microseconds"
60.    60.    60.    60.    60.    "off-peak size, (hilimit - lolimit)/off-peak size"
40.    40.    30.    40.    40.    "wavescan size, (hilimit - lolimit)/wavescan size"
80.    80.    50.    80.    80.    "peakscan size, (hilimit - lolimit)/peakscan size"
100.   100.   100.   100.   100.   "wavescan steps"
40.    40.    40.    40.    40.    "peakscan steps"
0.08   0.08   0.08   0.08   0.08   "LiF peaking start size"
0.004  0.004  0.004  0.004  0.004  "LiF peaking stop size"
30.    30.    30.    30.    30.    "maximum peaking cycles"
5.     5.     5.     5.     5.     "minimum peak to background"
10.   10.   10.   10.   10.   "minimum peak intensity"
1.    1.    1.    1.    1.    "default PHA baseline voltages"
9.    9.    9.    9.    9.    "default PHA window voltages"
32.   32.   32.   64.   16.   "default PHA gain"
1500. 1750. 1450. 1650. 1760. "default detector bias"
1.    1.    1.    1.    1.    "PHA baseline full scale factors"
1.    1.    1.    1.    1.    "PHA window full scale factors"
1.    1.    1.    1.    1.    "PHA gain scale factors"
1.    1.    1.    1.    1.    "Detector bias scale factors"
140.  100.  140.  140.  140.  "Roland Circle (mm)"
2     2     2     2     2     "Crystal flip delays"
400.  400.  400.  400.  400.  "Spec offset warning factors"
0     0     0     0     0     "Cameca integer deadtimes"
1500. 1500. 1500. 1500. 1500. "Bias scan low limits"
1800. 1800. 1800. 1800. 1800. "Bias scan high limits"
4     4     4     4     4     "Gain scan low limits"
128. 128. 128. 128. 128. "Gain scan high limits"
4     4     4     4     4     "Scan Baselines"
.1   .1   .1   .1   .1   "Scan Windows"
1.   1.   .3   2.   .45   "default PHA baseline voltages1"
1.   1.   1.   .5   .5   "default PHA baseline voltages2"
0.   0.   1.   0.   0.   "default PHA baseline voltages3"
0.   0.   1.   0.   0.   "default PHA baseline voltages4"
0.   0.   0.   0.   0.   "default PHA baseline voltages5"
0.   0.   0.   0.   0.   "default PHA baseline voltages6"
9.   9.   9.7   8.   9.55  "default PHA window voltages1"
9.   9.   9.   9.   9.5   "default PHA window voltages2"
0.   0.   9.   0.   0.   "default PHA window voltages3"
0.   0.   9.   0.   0.   "default PHA window voltages4"
0.   0.   0.   0.   0.   "default PHA window voltages5"
```

```

0.      0.      0.      0.      0.      "default PHA window voltages6"
32.     16.     32.     64.     16.     "default PHA gain1"
32.     32.     64.     64.     32.     "default PHA gain2"
0.      0.      32.     0.      0.      "default PHA gain3"
0.      0.      8.      0.      0.      "default PHA gain4"
0.      0.      0.      0.      0.      "default PHA gain5"
0.      0.      0.      0.      0.      "default PHA gain6"
1674.   1764.   1750.   1750.   1790.   "default detector bias1"
1750.   1800.   1800.   1700.   1770.   "default detector bias2"
0.      0.      1740.   0.      0.      "default detector bias3"
0.      0.      1740.   0.      0.      "default detector bias4"
0.      0.      0.      0.      0.      "default detector bias5"
0.      0.      0.      0.      0.      "default detector bias6"
0.      0.      0.      0.      0.      "default PHA inte/diff modes1"
0.      0.      0.      0.      0.      "default PHA inte/diff modes2"
0.      0.      0.      0.      0.      "default PHA inte/diff modes3"
0.      0.      0.      0.      0.      "default PHA inte/diff modes4"
0.      0.      0.      0.      0.      "default PHA inte/diff modes5"
0.      0.      0.      0.      0.      "default PHA inte/diff modes6"
1.23    1.31    1.56    1.32    1.04    "default detector deadtimes1"
1.21    1.16    1.31    1.14    1.03    "default detector deadtimes2"
0.      0.      1.23    0.      0.      "default detector deadtimes3"
0.      0.      1.18    0.      0.      "default detector deadtimes4"
0.      0.      0.      0.      0.      "default detector deadtimes5"
0.      0.      0.      0.      0.      "default detector deadtimes6"
0.      0.      0.      0.      0.      "Cameca large area crystal flag1"
0.      0.      0.      0.      0.      "Cameca large area crystal flag2"
0.      0.      0.      0.      0.      "Cameca large area crystal flag3"
0.      0.      0.      0.      0.      "Cameca large area crystal flag4"
0.      0.      0.      0.      0.      "Cameca large area crystal flag5"
0.      0.      0.      0.      0.      "Cameca large area crystal flag6"
0.      0.      0.      0.      0.      "unused"

```

#### **Line 1 (scalar labels)**

"1"      "2"      "3"      "4"      "5"      "scalar labels"

These scalar labels are used to load several dialog controls to specify the counting channel labels. The scalar labels must be enclosed in double quotes.

#### **Line 2 (not used at this time)**

0      0      0      0      0      "unused"

#### **Line 3 (not used at this time)**

0      0      0      0      0      "unused"

#### **Line 4 (tunable spectrometer crystal flip flags)**

0      0      0      0      0      "crystal flipping flag"

Indicate the type of automated crystal flipping supported by the hardware. Be aware that flipping crystals within a quantitative run may reduce the accuracy of the analysis. The allowable range of values is 0 to 4.

- 0**      no automated crystal flipping
- 1**      automated crystal flipping in any position
- 2**      automated crystal flipping at "crystal flipping position"
- 3**      automated crystal flipping at any position greater than "crystal flipping position"
- 4**      automated crystal flipping at any position less than "crystal flipping position"

#### **Line 5 (tunable spectrometer crystal flip positions)**

0.      0.      0.      0.      "crystal flipping position"

This line is used to indicate the crystal flipping position used when flipping the crystal (if required). See also "crystal flipping flag" above. The values must be greater than zero and within the mechanical range of the spectrometer. Note that the software will disable the software limit checking when performing a crystal flip since some spectrometer mechanisms require that the crystal flip occur at the motor limit position.

#### **Line 6 (number of crystals on each tunable spectrometer)**

2      2      2      2      2      "number of crystals"

This line is used to indicate the actual number of crystals on each tunable spectrometer. The range of allowable values is 1 to 6.

#### **Line 7-12 (crystal types on each tunable spectrometer)**

```

"PET"      "LIFH"      "PET"      "PET"      "LIF"      "crystal types1"
"TAP"      "PETH"      "TAP"      "TAP"      "PET"      "crystal types2"
" "        " "          "WS160"    " "        " "        "crystal types3"
" "        " "          "NiCrBN"   " "        " "        "crystal types4"
" "        " "          " "        " "        " "        "crystal types5"
" "        " "          " "        " "        " "        "crystal types6"

```

Use these lines to specify the crystal types on each tunable spectrometer. The crystal types must be enclosed in double quotes and must match one of the crystal types as defined in the **C:\ProgramData\Probe Software\Probe for EPMA\CRYSTALS.DAT** file.

#### **Line 13            (detector deadtime)**

```
1.0        1.0        1.0        1.0        1.0        "deadtime in microseconds"
```

Use these lines to specify the default detector deadtime in micro-seconds. These values are used by the *software* to calculate the deadtime for quantitative analyses. The range of allowable values is from 0.1 to 10 microseconds.

The detector deadtime value specified here is NOT used for setting the non-extendable or "enforced" (DTIM) deadtime for those microprobe hardware that support it (Cameca SX100/SXFive only). The non-extendable or "enforced" value deadtime value is instead specified on line 35 below. Note that in this case, because the SX100/SXFive interface only supports an integer deadtime value, the actual deadtime of the system may vary slightly from the integer values specified.

However careful deadtime calibration measurements should be performed (using program StartWin or the optional Excel deadtime acquisition and calculation spreadsheets provided with the Remote OLE interface option) at the "nominal" integer setting to determine the precise actual deadtime coefficient of the SX100/SXFive pulse processor, and these should be specified here for the analysis correction routine for the most rigorous deadtime correction possible.

See also the option for the "precision" deadtime correction in the PROBEWIN.INI file under the [Software] section and in the ANALYSIS window options for high count rates.

*Note that this section is now superseded by the new deadtime configuration lines below so that a separate deadtime value can be specified for each spectrometer and crystal combination. See lines 72-77 below.*

#### **Line 14-16        (off-peak/wavescan/peakscan offset size parameters)**

```
0        0        0        0        "unused"
90.      90.      90.      90.      "off-peak size, (hilimit - lolimit)/off-peak size"
60.      60.      60.      60.      "wavescan size, (hilimit - lolimit)/wavescan size"
80.      80.      80.      80.      "peakscan size, (hilimit - lolimit)/peakscan size"
```

Use these lines to specify the default off-peak, wavescan and peakscan offset sizes respectively. These factors are the fraction of the total spectrometer range used for the off-peak position and wavelength scan ranges for x-ray lines that use an LiF (200) analyzing crystal and are at the high theta spectrometer limit. They must be between 20 and 500 (20 and 10000 for the peakscan parameters).

X-ray lines analyzed using other crystals and at lower thetas will have their off-peak, wavescan and peakscan ranges adjusted for the 2d spacing of the crystal and the spectrometer position. The actual expression used is shown below for (in this example) calculating the default off-peak positions. Of course all off-peak and wavelength scan ranges may be changed at any time by the user within the run.

$$P_{OffPeak} = P_{OnPeak} \pm \frac{ABS(P_{HiLim} - P_{LoLim})}{O_{factor}} * \sqrt[3]{\frac{2d_{OnPeak}}{2d_{LiF200}}} (* 3) \sqrt{\frac{P_{HiLim}}{P_{OnPeak}}}$$

Where :

- $P_{OffPeak}$         is the calculated default off-peak position
- $P_{OnPeak}$         is the spectrometer on-peak position
- $P_{HiLim}$         is the spectrometer high limit
- $P_{LoLim}$         is the spectrometer low limit
- $2d_{OnPeak}$         is the 2d spacing of the analyzing crystal
- $2d_{LiF200}$         is the 2d spacing for LiF (4.0267)

$O_{\text{factor}}$  is the off-peak/wavescan/peakscan factor (SCALERS.DAT lines 14-16)

Note the (\*3.0) factor, which is applied if the 2d spacing of the crystal is 30 angstroms or larger in order to account for the poorer resolution of pseudo-crystals such as stearate or layered dispersive element (LDE) analyzers.

### Pre and Post Scan Ranges

The peakscan range (for post and prescan peakscans) is based on the peakscan range calculated above but the counting time is further modified as shown here:

$$\text{PreScanCountTime} = \text{PeakingCountTime} / 4$$

$$\text{PostScanCountTime} = \text{PeakingCountTime}$$

Where:

$\text{PeakingCountTime}$  is from the PROBEWIN.INI [counting] section.

### ROM Peak Scan Range

The ROM peak scan range is also related to the crystal 2d and spectrometer position peaking start size parameter as shown here:

$$\text{PeakingStartSize} = \text{ScalLiFPeakingStartSize} * \text{Factor}$$

where:

$\text{ScalLiFPeakingStartSize}$  is from line 19 of the SCALERS.DAT file

and:

$\text{Factor}$  is calculated from the following expression:

$$\text{Factor} = \sqrt[3]{\frac{2d_{\text{OnPeak}}}{2d_{\text{LiF200}}}} (* 3) \sqrt{\frac{P_{\text{HiLim}}}{P_{\text{OnPeak}}}}$$

where:

$P_{\text{OnPeak}}$  is the spectrometer on-peak position

$P_{\text{HiLim}}$  is the spectrometer high limit

$2d_{\text{OnPeak}}$  is the 2d spacing of the analyzing crystal

$2d_{\text{LiF200}}$  is the 2d spacing for LiF (4.0267)

Note that if the analyzing crystal 2d is greater than 30 (for LDE analyzers) the scaling factor is increased by an additional factor of three. The actual ROM peakscan range is further adjusted based on the expression:

$$\text{ROMPeakScanRange} = \frac{(P_{\text{HiLim}} - P_{\text{LoLim}})}{\text{ScalPeakScanSizeFactor}} * \frac{\text{PeakingStartSize}}{\text{ScalLiFPeakingStartSize}}$$

where:

$\text{ScalPeakScanSizeFactor}$  is from line 16 of the SCALERS.DAT file

$\text{ScalLiFPeakingStartSize}$  is from line 19 of the SCALERS.DAT file

$\text{PeakingStartSize}$  is the calculated from "Factor" above and is user adjustable in PFE

$P_{\text{HiLim}}$  is the spectrometer high limit

$P_{\text{LoLim}}$  is the spectrometer low limit

Note that the SCALERS.DAT parameters are designed to place these parameters in approximate relative scale for normal and high intensity spectrometers while the PeakingStartSize can be edited by the user from the Peak/Scan Options dialog for a specific analytical situation to increase or decrease the ROM peaking scan range.

**Line 17-18 (number of wavescan or peakscan steps)**

```
100    100    100    100    "wavescan steps"  
40     40     40     40     "peakscan steps"
```

These parameters are used to define the default wavescan and peakscan (pre-scan before peak centering) number of steps. The number of steps must be greater than 5 and the maximum number of peakscan steps must be less than or equal to MAXROMSSCAN% (1000).

**Line 19-20 (spectrometer peak center start and stop sizes)**

```
0.001   0.001   0.001   0.001   "LiF peaking start size"  
0.0002  0.0002  0.0002  0.0002  "LiF peaking stop size"
```

These parameters are used to define the default start and stop step sizes which is used for all three types of spectrometer peaking (interval, parabolic and ROM based).

### **Software Based Peaking**

For interval halving spectrometer peak centering, the peaking start size is the spectrometer step size used to begin the interval halving peak center assuming an LiF (200) crystal and a theta at the highest spectrometer setting. For example, K  $\alpha$  on an LiF crystal (SEMQ probe) or Ti  $\alpha$  on a LiF crystal (Cameca probe). Each time the interval halving peaking procedure reverses direction, the interval size is halved. When the interval size is less than the peaking stop size the spectrometer is considered to be properly peaked.

For parabolic fit peak centering, the peaking start size determines the offset used to measure the high and low side peak intensities. The peaking start size should be adjusted to yield an intensity approximately 1/2 the peak intensity for both the high and low side of the peak, to obtain a good fit to the peak shape. If the parabolic fit spectrometer positions need adjustment, the program shifts the middle, high and low positions by 1/2 the peaking start size and tries again.

### **ROM Based Peaking**

For the ROM based peak centering (if available), the peaking start size is again utilized for best results, depending on the interface type. Note the following parameters apply to non-ROM based and ROM based peak scan parameters:

#### **Non-ROM scan based**

1. Wavescan: # of points, wave count time
2. Quickskan: Spectro scan speed (%), quick count time

#### **ROM scan based**

1. Wavescan: # of points, wave count time
2. Quickskan: Spectro scan speed (%), quick count time

#### **ROM Based Peaking Scan:**

- fine scan uses peaking time divided by 4 times 1.0
- coarse scan uses peaking time divided by 4 divided by 2.0
- fine scan uses peaking time divided by 4 times 1.5

Several ROM based peak center fit types are available. Applies only to Cameca SX100/SXFive and JEOL 8200/8900/8500/8x30 micropores.

0 = Internal (the instrument ROM peak method which applies only to Cameca)

1 = Parabolic (spectrometer scan data is fit to a parabolic fit)

2 = Maxima (spectrometer scan data is fit to Brent's Maxima function)

3 = Gaussian (spectrometer scan data is fit to a Gaussian fit)

The internal ROM type is available only on Cameca instruments. The other options are only available on JEOL 8200/8900/8500/8x30 and Cameca SX100/SXFive instruments. The parabolic and maxima fits require at least 3 data points above the threshold (see below). The parabolic and maxima methods have a user defined threshold that can be specified in the INI file and the program.

The ROM peaking thresholds are designed to allow the user to define the intensity values above which are used for one of the three ROM fitting methods (parabolic, maxima or Gaussian). The default is 0.33 which means that all intensity values 1/3 above the minimum to maximum intensity range are used in the fit.

**JEOL 8900/8200/8500/8x30 Direct (InterfaceType=2)-** The "Peaking Start Size" is used to calculate the spectrometer scan width for the peak scan procedure. The coarse scan width is 3 times larger than the fine scan width.

$$S_{ScanWidth} = \frac{(P_{HiLim} - P_{LoLim})}{P_{factor}} \cdot \frac{P_{StartSize}}{P_{LiFStartSize}}$$

Where :

|                    |   |
|--------------------|---|
| $S_{ScanWidth}$    | is the calculated peaking scan width                                      |
| $P_{HiLim}$        | is the spectrometer high limit (from MOTORS.DAT)                          |
| $P_{LoLim}$        | is the spectrometer low limit (from MOTORS.DAT)                           |
| $P_{factor}$       | is the peak scan size factor (from SCALERS.DAT)                           |
| $P_{StartSize}$    | is the crystal spectrometer peaking size (calculated for 2d and position) |
| $P_{LiFStartSize}$ | is the LiF spectrometer peaking size (from SCALERS.DAT)                   |

**SX100/SXFive (InterfaceType=5)-** The "Peaking Start Size" is modified to produce a number between 0 and 4 by utilizing the following expression :

$$pw = (M_{high} - M_{low}) * P / 3000000$$

where pw is the SX100 peak center width  
 $M_{high}$  is the motor high limit  
 $M_{low}$  is the motor low limit  
 $P$  is the Peaking Start Size

The program uses a larger start and stop size for lower thetas and larger crystal 2d spacings. A smaller stop peaking step size will force the program to more carefully adjust the spectrometer position, however this will require more time to achieve. Be sure that the start peaking size is large enough for the program to measure a significant count change. The start values must be larger than 1/6000th of the total spectrometer range. The stop peaking size must be less than the start peaking size. The actual calculation for a specific peak position for a specific crystal is shown here :

$$S_{actualstart} = S_{start} * \sqrt[3]{\frac{2d_{OnPeak}}{2d_{LiF200}}} (* 3) \sqrt{\frac{P_{HiLim}}{P_{OnPeak}}}$$

Where :

|                   |  |
|-------------------|--|
| $S_{actualstart}$ | is the actual calculated step size             |
| $S_{start}$       | is the spectrometer default start or stop size |
| $P_{HiLim}$       | is the spectrometer high limit                 |
| $P_{OnPeak}$      | is the spectrometer on-peak position           |
| $2d_{OnPeak}$     | is the 2d spacing of the analyzing crystal     |
| $2d_{LiF200}$     | is the 2d spacing for LiF (4.0267)             |

## **SCALERS.DAT Lines 21-41**

### **Line 21-23 (peaking cycles, P/B and minimum intensities)**

|     |     |     |     |                              |
|-----|-----|-----|-----|------------------------------|
| 30. | 30. | 30. | 30. | "maximum peaking cycles"     |
| 5.  | 5.  | 5.  | 5.  | "minimum peak to background" |
| 10. | 10. | 10. | 10. | "minimum peak intensity"     |

These parameters are used to define additional peak centering default parameters for the interval halving and parabolic peak center. The maximum peaking cycles is used to specify the maximum number of interval halving attempts the program will make to find the peak center. If the number of peaking cycles is small or the peak start size is small or the peak is too far from the starting position, the peak center may fail. The allowable range is 5 to 50 maximum peak cycles.

The minimum peak to background is the minimum peak to background ratio that the program will attempt a peak center on. The values must be greater than 1.5.

The minimum peak intensity is the minimum peak intensity in counts per second that the program will attempt a peak center on. The values must be greater than 10 counts per second.

### **Line 24-25 (PHA baseline, window default voltages)**

|     |     |     |     |                                 |
|-----|-----|-----|-----|---------------------------------|
| 1.  | 1.  | 1.  | 1.  | "default PHA baseline voltages" |
| 10. | 10. | 10. | 10. | "default PHA window voltages"   |

The default baseline and window PHA values are specified on this line. The values must be greater than zero.

### **Line 26-27 (PHA gain and bias defaults)**

|       |       |       |       |                         |
|-------|-------|-------|-------|-------------------------|
| 16.   | 16.   | 64.   | 32.   | "default PHA gain"      |
| 1350. | 1880. | 1850. | 1350. | "default detector bias" |

The default gain and bias values are specified on this line. The values must be greater than zero.

### **Line 28-31 (PHA baseline, window, gain and bias scale factors)**

|    |    |    |    |                                   |
|----|----|----|----|-----------------------------------|
| 1. | 1. | 1. | 1. | "PHA baseline full scale factors" |
| 1. | 1. | 1. | 1. | "PHA window full scale factors"   |
| 1. | 1. | 1. | 1. | "PHA gain scale factors"          |
| 1. | 1. | 1. | 1. | "Detector bias scale factors"     |

The PHA scale factors are used for PHA hardware that does not accept 0 to 10 volt control voltages. If the PHA hardware does give a 10 volt output for a 10 volt input control voltage then the scale factors will be 1. If the PHA hardware outputs full scale (10 volts) using a lower input control voltage, then the scale factors should be adjusted. For example, if the PHA hardware wants a .33 volt input to output 10 volts, then set the appropriate PHA scale factor to .33 for that channel. The values must be greater than 0.0.

These parameters are no longer utilized by any of the current instrument interfaces.

### **Line 32 (spectrometer Roland circle radius in mm)**

|      |      |      |      |                      |
|------|------|------|------|----------------------|
| 127. | 127. | 127. | 127. | "Roland Circle (mm)" |
|------|------|------|------|----------------------|

The values on this line are used to specify the spectrometer Roland circle. These values are not used at this time. The values must be greater than 0.0.

### **Line 33 (crystal flip delays in seconds)**

|    |    |    |    |                       |
|----|----|----|----|-----------------------|
| 0. | 0. | 0. | 0. | "Crystal flip delays" |
|----|----|----|----|-----------------------|

The values on this line are used to define the type and duration of the crystal flip delay used to wait for a crystal flip to complete. If the value is greater than zero then the program will simply delay for the duration specified before allowing the program to continue.

### **Line 34 (spectrometer offset warning factors)**

|      |      |      |      |                               |
|------|------|------|------|-------------------------------|
| 400. | 400. | 400. | 400. | "Spec offset warning factors" |
|------|------|------|------|-------------------------------|

The values on this line are used to define the size of the maximum offset for a spectrometer from its theoretical position for the purposes of printing a warning message to the log window. If the actual spectrometer position offset is less than the spectrometer range (as defined in the MOTORS.DAT file) divided by this spectrometer offset warning factor then the warning will not be printed. The default value is 400.

### **Line 35 (Cameca integer deadtimes)**

|    |    |    |    |                            |
|----|----|----|----|----------------------------|
| 0. | 0. | 0. | 0. | "Cameca integer deadtimes" |
|----|----|----|----|----------------------------|

This line is used to explicitly specify the integer deadtime constants used to set the Cameca PHA hardware (non-extensible or "enforced" deadtime. Therefore this data is used only by the Cameca SX100/SXFive hardware interface

and the values are not accessible from within the program. These integer deadtime values are distinct from the single precision deadtimes specified on line 72-77 below which are used to perform the actual deadtime correction in the analysis routines.

The typical procedure is to set the deadtimes on the Cameca PHA hardware all to zero and then to measure the "intrinsic" deadtime of the system using a range of beam currents from approximately 10 to 200 nA on a pure metal x-ray line such as Si Ka (PET and TAP) or Ti Ka (PET and LIF). A sufficient counting time should be used to obtain .2% precision or better. The best method is to find the "worst case" deadtime for each spectrometer since the deadtimes may vary somewhat as a function of detector bias and x-ray line energy.

Assume that the "intrinsic" deadtimes measured (when all spectrometers are set to "DTIM"=0) are 2.23, 3.14, 3.45, 3.78 and 2.15. The next step would be to set the DTIM deadtime parameter for each spectrometer to a value large enough to completely "mask" this intrinsic" deadtime, that is values of 3, 4, 4, 4 and 3. Now since these integer deadtime are not accurately set by the Cameca hardware, the operator must now re-run the deadtime calibration measurement using these new values and note the actual deadtimes. In this case depending on the instrument, the measured deadtimes will be somewhat larger, say, 3.75, 4.65, 4.12, 4.89 and 3.32.

In the example just described, the "DTIM" values of 3, 4, 4, 4 and 3 should be entered on line 35 for setting the Cameca hardware and the measured values that correspond to them, that is, 3.75, 4.65, 4.12, 4.89 and 3.32 should be entered on line 13 above to use in the software correction.

The integer "DTIM" deadtime values must be between zero and ten. If a value of zero is read, then the program will load the single precision deadtime from line 13 and truncate to integer each value for setting the Cameca PHA hardware to an approximate value.

#### **Lines 36-41 (Bias scan low and high, gain scan low and high, scan baseline and window values)**

```

0      0      0      0      "unused"
1500  1500  1500  1500  "Bias scan low limits"
1800  1800  1800  1800  "Bias scan high limits"
4      4      4      4      "Gain scan low limits"
128   128   128   128   "Gain scan high limits"
4.    4.    4.    4.    "Scan baselines"
0.1   0.1   0.1   0.1   "Scan windows"

```

These lines are used to specify the PHA bias and gain scan parameters used in the PHA dialogs. This characterization of the detector electronics is essential for non-Cameca (JEOL) instruments where the gain can only be adjusted in coarse steps. Normally one sets the bias scan baseline at 4 volts, the window at 0.1volts above the baseline and scans the detector bias between 1500 and 1800 volts to determine what bias value is necessary for the x-ray peak to obtain a 4 volt level. Note, the low bias scan voltage must be above the bias "plateau" for the correct determination of the bias value.

For other instruments the gain can be scanned to determine the actual gain necessary for a given detector bias. Both scan procedures use the same scan baseline and windows settings. The default count time and interval values are specified in the PROBEWIN.INI file in the [pha] section. Contact Paul Carpenter or John Donovan for more information on this issue.

#### **Extended Format SCALERS.DAT Lines 42-83**

#### **Lines 42-65 (default baseline, window, gain and bias and(deadtime PHA settings for each crystal)**

```

1.    1.    .3    .45    "default PHA baseline voltages1"
1.    1.    1.    1.    "default PHA baseline voltages2"
0.    0.    0.    0.    "default PHA baseline voltages3"
0.    0.    0.    0.    "default PHA baseline voltages4"
0.    0.    0.    0.    "default PHA baseline voltages5"
0.    0.    0.    0.    "default PHA baseline voltages6"
9.    9.    9.7   8.    "default PHA window voltages1"
9.    9.    9.    9.    "default PHA window voltages2"
0.    0.    0.    0.    "default PHA window voltages3"
0.    0.    0.    0.    "default PHA window voltages4"
0.    0.    0.    0.    "default PHA window voltages5"
0.    0.    0.    0.    "default PHA window voltages6"
32.   16.   32.   64.   "default PHA gain1"
32.   32.   64.   64.   "default PHA gain2"
0.    0.    0.    0.    "default PHA gain3"
0.    0.    0.    0.    "default PHA gain4"
0.    0.    0.    0.    "default PHA gain5"
0.    0.    0.    0.    "default PHA gain6"
1674. 1764. 1750. 1650. "default detector bias1"
1750. 1800. 1800. 1700. "default detector bias2"
0.    0.    0.    0.    "default detector bias3"
0.    0.    0.    0.    "default detector bias4"

```

```

0.      0.      0.      0.      "default detector bias5"
0.      0.      0.      0.      "default detector bias6"

```

These lines are used to set the individual default PHA settings for the baseline, window, gain and bias on the different spectrometer and crystal basis. These crystal based parameter values replace the older default PHA settings in lines 24-27. The older values are still read and used as defaults for backward compatibility.

You only need to enter values for as many crystals as you have for each spectrometer (up to six crystals per spectrometer). The range of allowable values is the same as the PHA parameters for lines 24-27.

#### **Lines 66-77 (default Inte/Diff mode and Deadtime PHA settings for each crystal)**

```

0      0      0      0      "default PHA inte/diff modes1"
0      0      0      0      "default PHA inte/diff modes2"
0      0      0      0      "default PHA inte/diff modes3"
0      0      0      0      "default PHA inte/diff modes4"
0      0      0      0      "default PHA inte/diff modes5"
0      0      0      0      "default PHA inte/diff modes6"
0      0      0      0      "default detector deadtimes1"
0      0      0      0      "default detector deadtimes2"
0      0      0      0      "default detector deadtimes3"
0      0      0      0      "default detector deadtimes4"
0      0      0      0      "default detector deadtimes5"
0      0      0      0      "default detector deadtimes6"

```

These lines are used to set the individual default PHA settings for the inte/diff mode and deadtime on the different spectrometer and crystal basis. These crystal based deadtime parameter values replace the older default settings in line 13.

This is mainly for non-Cameca microprobes where there is no deadtime pulse stretching electronics and the instrument may benefit from individual deadtime values for each crystal (energy range). The older deadtime values are still read and used as defaults for backward compatibility if these new values are zero.

There was no default inte/diff mode previously so the default inte/diff mode is zero (integral mode).

#### **Lines 78-83 (Cameca large area crystal flag)**

```

0      0      0      0      "Cameca large area crystal flag1"
0      0      0      0      "Cameca large area crystal flag2"
0      0      0      0      "Cameca large area crystal flag3"
0      0      0      0      "Cameca large area crystal flag4"
0      0      0      0      "Cameca large area crystal flag5"
0      0      0      0      "Cameca large area crystal flag6"

```

This section allows the user to specify whether any crystals are of the large area type. This flag applies only to the Cameca SX100/SXFive and is only used to properly set the correct index for crystal flipping. Two crystal spectrometer use indices 1 and 4 unless they are of the large area type in which case they use indices 2 and 4.

To indicate that a crystal is of the large area type edit the flag to be a non zero value for each large area crystal. Note that the Cameca very large area crystals are always index 1 and there is only one crystal per spectrometer (it cannot be flipped).

#### **Lines 84-90 (not used at this time)**

```

0      0      0      0      "unused"

```

## **DETECTORS.DAT**

The data file used to define detector parameters. The scalar array size (the number of data values on each line) is defined as the "NumberOfTunableSpecs" in the PROBEWIN.INI file. **Note that a comment string enclosed in double quotes MUST be present at the end of each line.** Several unused parameter lines are throughout the file for future expansion.

The example DETECTORS.DAT file shown here is for a 1 spectrometer instrument.

```

"1"      "Scaler Labels"
3        "Number of Slit Sizes"
"2.0mm"  "Slit Size Strings[1]"
"0.5mm"  "Slit Size Strings[2]"
"0.3mm"  "Slit Size Strings[3]"

```

```

" "
"Slit Size Strings[4]"
"Slit Size Strings[5]"
"Slit Size Strings[6]"
"Slit Size Strings[7]"
"Slit Size Strings[8]"
"Slit Size Strings[9]"
"Slit Size Strings[10]"
"Slit Size Strings[11]"
"Slit Size Strings[12]"
0
"Number of Slit Positions"
"Slit Position Strings[1]"
"Slit Position Strings[2]"
"Slit Position Strings[3]"
"Slit Position Strings[4]"
"Slit Position Strings[5]"
"Slit Position Strings[6]"
"Slit Position Strings[7]"
"Slit Position Strings[8]"
"Slit Position Strings[9]"
"Slit Position Strings[10]"
"Slit Position Strings[11]"
"Slit Position Strings[12]"
4
"Number of Detector Modes"
"Detector Mode Strings[1]"
"Detector Mode Strings[2]"
"Detector Mode Strings[3]"
"Detector Mode Strings[4]"
"Detector Mode Strings[5]"
"Detector Mode Strings[6]"
"Detector Mode Strings[7]"
"Detector Mode Strings[8]"
"Detector Mode Strings[9]"
"Detector Mode Strings[10]"
"Detector Mode Strings[11]"
"Detector Mode Strings[12]"
0
"Number of Unused"
"Unused Strings[1]"
"Unused Strings[2]"
"Unused Strings[3]"
"Unused Strings[4]"
"Unused Strings[5]"
"Unused Strings[6]"
"Unused Strings[7]"
"Unused Strings[8]"
"Unused Strings[9]"
"Unused Strings[10]"
"Unused Strings[11]"
"Unused Strings[12]"
2
"Slit Size Exchange Flags [0=none, 1=any position, 2=at position]"
0
"Slit Position Exchange Flags [0=none, 1=any position, 2=at position]"
1
"Detector Mode Exchange Flags [0=none, 1=any position, 2=at position]"
0
"Unused Exchange Flags [0=none, 1=any position, 2=at position]"
320.0
"Slit Size Exchange Positions"
0.0
"Slit Position Exchange Positions"
0.0
"Detector Mode Exchange Positions"
0.0
"Unused Exchange Positions"
185.0
"Slit Size Exchange Rowlands"
0.0
"Slit Position Exchange Rowlands"
0.0
"Detector Mode Exchange Rowlands"
0.0
"Unused Exchange Rowlands"
2
"Default Slit Size Indexes [1 to Number of Slit Sizes]"
1
"Default Slit Position Indexes [1 to Number of Slit Positions]"
4
"Default Detector Mode Indexes [1 to Number of Detector Modes]"
1
"Default Unused Indexes [1 to Number of Unused]"

```

### **Line 1 (scaler labels)**

"1"  
"Scaler Labels"

These scaler labels are used to load several dialog controls to specify the counting channel labels. The scaler labels must be enclosed in double quotes and must be identical to the values in the SCALERS.DAT file.

### **Line 2 (number of slit sizes)**

3  
"Number of Slit Sizes"

### **Lines 3-14 (slit size strings)**

```

"2.0mm"
"0.5mm"
"0.3mm"
" "
"Slit Size Strings[1]"
"Slit Size Strings[2]"
"Slit Size Strings[3]"
"Slit Size Strings[4]"
"Slit Size Strings[5]"
"Slit Size Strings[6]"
"Slit Size Strings[7]"
"Slit Size Strings[8]"
"Slit Size Strings[9]"
"Slit Size Strings[10]"
"Slit Size Strings[11]"
"Slit Size Strings[12]"

```

Line two indicates the number of valid slit size strings that will follow each scaler label. Do not enclose the number in quotes. The next 12 lines contain the actual slit size strings that will appear in the dialog for the user to choose from.

These must be enclosed in double quotes. The actual value that is set by the program is depends on the detector hardware interface.

**Line 15                    (number of slit positions)**

```
0         "Number of Slit Positions"
1
```

**Lines 16-27            (slit position strings)**

```
"" "Slit Position Strings[1]"
"" "Slit Position Strings[2]"
"" "Slit Position Strings[3]"
"" "Slit Position Strings[4]"
"" "Slit Position Strings[5]"
"" "Slit Position Strings[6]"
"" "Slit Position Strings[7]"
"" "Slit Position Strings[8]"
"" "Slit Position Strings[9]"
"" "Slit Position Strings[10]"
"" "Slit Position Strings[11]"
"" "Slit Position Strings[12]"
```

Line fifteen indicates the number of valid slit position strings that will follow each scaler label. Do not enclose the number in quotes. The next 12 lines contain the actual slit position strings that will appear in the dialog for the user to choose from. These must be enclosed in double quotes. The actual value that is set by the program is depends on the hardware interface.

**Line 28                    (number of detector modes)**

```
4         "Number of Detector Modes"
```

**Lines 29-40            (detector mode strings)**

```
"SPC- FPC-"
"SPC- FPC+"
"SPC+ FPC-"
"SPC+ FPC+"
"""
"Detector Mode Strings[1]"
"Detector Mode Strings[2]"
"Detector Mode Strings[3]"
"Detector Mode Strings[4]"
"Detector Mode Strings[5]"
"Detector Mode Strings[6]"
"Detector Mode Strings[7]"
"Detector Mode Strings[8]"
"Detector Mode Strings[9]"
"Detector Mode Strings[10]"
"Detector Mode Strings[11]"
"Detector Mode Strings[12]"
```

Line twenty-eight indicates the number of valid detector mode strings that will follow each scaler label. Do not enclose the number in quotes. The next 12 lines contain the actual detector mode strings that will appear in the dialog for the user to choose from. These must be enclosed in double quotes. The actual value that is set by the program is depends on the hardware interface.

*Note that these DetectorMode strings can also be used for other probe types (e.g., Cameca and JEOL) for specifying the detector gas type to allow the program to better calculate whether an Ar or Xe absorption edge is present between the on peak and off-peak measurement positions.*

For example, if the Xe filled detectors are specified using a string containing the sub-string “XPC” then the program will know that that detector is filled with Xe gas and subject to Xe edge effects.

' JEOL Detector types:

- ' GPC = gas flow proportional detector P-10 atmospheric pressure
- ' XPC = sealed detector Xenon proportional counter (no Ar gas)
- ' GPCH = gas flow prop detector P-10 atm press, on H-type spectrometer
- ' XPCH = sealed Xe detector on H-spectrometer (has different slit device compared to XPC) (no Ar gas)

For this purpose the number of “DetectorModes” should be left at zero as shown in the following example:

```
0         "Number of Detector Modes"
"GPC"    "XPCH"    "GPC"    "GPC"    "XPC"    "Detector Mode Strings [1]"
" "       " "       " "       " "       " "       "Detector Mode Strings [2]"
" "       " "       " "       " "       " "       "Detector Mode Strings [3]"
" "       " "       " "       " "       " "       "Detector Mode Strings [4]"
" "       " "       " "       " "       " "       "Detector Mode Strings [5]"
" "       " "       " "       " "       " "       "Detector Mode Strings [6]"
" "       " "       " "       " "       " "       "Detector Mode Strings [7]"
" "       " "       " "       " "       " "       "Detector Mode Strings [8]"
```

```

" "      " "      " "      " "      " "      "Detector Mode Strings [9]"
" "      " "      " "      " "      " "      "Detector Mode Strings [10]"
" "      " "      " "      " "      " "      "Detector Mode Strings [11]"
" "      " "      " "      " "      " "      "Detector Mode Strings [12]"

```

Similarly, Cameca instruments can use this string to define which detectors are one atmosphere and which are two atmosphere detectors for documentation purposes as shown in the following example:

```

0      0      0      0      0      "Number of Detector Modes"
"FPC-1" "FPC-1" "FPC-2" "FPC-1" "FPC-2"  "Detector Mode Strings [1]"
" "      " "      " "      " "      " "      "Detector Mode Strings [2]"
" "      " "      " "      " "      " "      "Detector Mode Strings [3]"
" "      " "      " "      " "      " "      "Detector Mode Strings [4]"
" "      " "      " "      " "      " "      "Detector Mode Strings [5]"
" "      " "      " "      " "      " "      "Detector Mode Strings [6]"
" "      " "      " "      " "      " "      "Detector Mode Strings [7]"
" "      " "      " "      " "      " "      "Detector Mode Strings [8]"
" "      " "      " "      " "      " "      "Detector Mode Strings [9]"
" "      " "      " "      " "      " "      "Detector Mode Strings [10]"
" "      " "      " "      " "      " "      "Detector Mode Strings [11]"
" "      " "      " "      " "      " "      "Detector Mode Strings [12]"

```

Since the Cameca detectors are always Ar filled, on Cameca instruments the program will never calculate a Xe absorption problem and always calculate an Ar edge problem (if it exists).

**Line 41            (number of unused)**

```

0      "Number of unused"
1

```

**Lines 42-53        (unused strings)**

```

" "      "Unused String[1]"
" "      "Unused String[2]"
" "      "Unused String[3]"
" "      "Unused String[4]"
" "      "Unused String[5]"
" "      "Unused String[6]"
" "      "Unused String[7]"
" "      "Unused String[8]"
" "      "Unused String[9]"
" "      "Unused String[10]"
" "      "Unused String[11]"
" "      "Unused String[12]"

```

Lines forty-one to fifty-three are unused for the time being and are reserved for future expansion of the detector parameter interface.

**Line 54            (slit size exchange flag)**

**Line 55            (slit position exchange flag)**

**Line 56            (detector mode exchange flag)**

**Line 57            (unused exchange flag)**

```

2      "Slit Size Exchange Flag"
0      "Slit Position Exchange Flag"
1      "Detector Mode Exchange Flag"
0      "Unused Exchange Flag"

```

Lines fifty four to fifty seven are flags that indicate the type of the detector exchange mechanism. The allowable range of values is 0 to 2.

The example lines shown above indicate that the slit size must be exchanged at the specified spectrometer position given below, while the slit position mechanism is not automated, and finally while the detector mode exchange can be automated in any spectrometer position.

```

0      no automated exchange
1      automated exchange in any position
2      automated exchange only at "exchange position"

```

**Line 58            (slit size exchange position)**

**Line 59            (slit position exchange position)**

```

Line 60      (detector mode exchange position)
Line 61      (unused exchange position)
  320.0      "Slit Size Exchange Position"
  0.0       "Slit Position Exchange Position"
  0.0       "Detector Mode Exchange Position"
  0.0       "Unused Exchange Position"
Line 62      (slit size exchange Rowland circle) (obsolete)
Line 63      (slit position exchange Rowland circle) (obsolete)
Line 64      (detector mode exchange Rowland circle) (obsolete)
Line 65      (unused exchange Rowland circle)
  185.0      "Slit Size Exchange Rowland"
  0.0       "Slit Position Exchange Rowland"
  0.0       "Detector Mode Exchange Rowland"
  0.0       "Unused Exchange Rowland"

```

Lines fifty eight to sixty five specify the spectrometer Rowland circle for the automated exchange mechanism. This parameter is obsolete..

```

Line 66      (Default slit size index)
Line 67      (Default slit position index)
Line 68      (Default detector mode index)
Line 69      (Default unused index)
  2          "Default Slit Size Index [1 to Number of Slit Sizes]"
  1          "Default Slit Position Index [1 to Number of Slit Positions]"
  4          "Default Detector Mode Index [1 to Number of Detector Modes]"
  1          "Default Unused Index [1 to Number of Unused]"

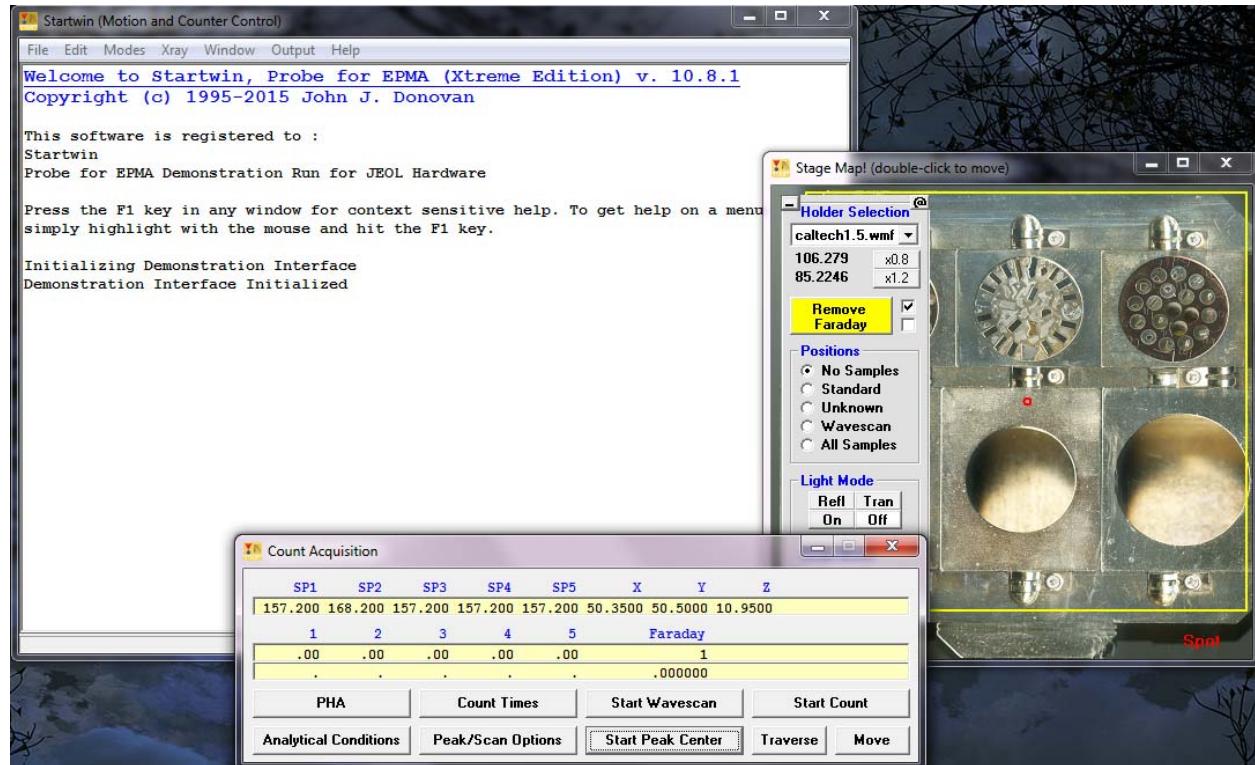
```

Lines sixty six to sixty nine specify the default detector settings for the given spectrometer scalar number. These are index numbers and must correspond with the slit size, slit position and detector mode strings in the previous sections. Values must be between 1 and the maximum number specified in the "number of slit sizes", "number of slit positions" and "number of detector modes" values.

# StartWin

## Overview

Program STARTWIN is used to provide a simple interface to the microprobe motion and counting hardware functions. From STARTWIN, the user can move the spectrometer and stage motors, flip spectrometer crystals, blank or unblank the beam and measure x-ray intensities.



## Description

Startwin is a utility application for general purpose control of the microprobe hardware. It supports stage and spectrometer motion, crystal flipping and scaler counting. It also includes features for peak centering, beam and detector stability, multiple spectrometer wavescans and PHA distribution tests and spectrometer reproducibility routines.

X-ray intensities in the log window are normalized to counts per second and corrected for beam drift by default. If you prefer to have the actual raw counts displayed, you can uncheck the Normalize to Counts Per Second and/or the Correct For Beam Drift options, respectively in the Count Times dialog. The actual normalization constant used for the beam

drift correction is, by default, the first non-zero beam current read when the program is started, but this value can be edited in the Count Times dialog. Enter 1.0 for counts normalized to counts "per nA" or zero for no beam drift correction. Note that to enable use of the +/- 3 S.D. option in the Graph window, the counts must not be normalized to counts per second.

The main window of STARTWIN is a scrollable text window which will record all output and data acquisition. Most of the functionality in STARTWIN is included in the larger **Probe for EPMA** application and help for these functions can be found in the **Probe for EPMA** documentation and help sections. The following specific functions are unique to STARTWIN :

## Beam and Detector Stability

To test the beam and/or detector stability select from the Modes menu the data acquisition options desired for the test. Normally to test beam and/or detector stability, one should make sure that the Measure Faraday menu item is checked (even for aperture microprobes) and from the Count Times button in the Count Acquisition window, confirm that the Use Beam Drift Correction check box is unchecked in order to disable the beam drift correction.

It may also be desirable to check the Cycle menu item to continuously acquire data for an extended period of time. Finally, when ready, simply click the Start Count button. After a suitable number of acquisitions have been acquired, click the Cancel button in the StartWin log window to stop the acquisition.

Next, the data may be graphically plotted by using the mouse to select the data in the log window to plot. When the desired data is selected, simply use the Output | Plot Count Data menu to graph the results. By default all data columns are selected, however to plot a single data column simply click the column label of the desired data and click the Graph button. The Use 3 S.D. check box will display +/- 3 standard deviation lines for evaluation purposes if checked.

## Spectrometer Reproducibility

To test spectrometer reproducibility, set up the count acquisition as before in the Counter and Detector Stability section, peak the spectrometer or spectrometers to test using the Peak/Scan Options and Start Peak center buttons, and then select the Move to Off Peaks menu item in the Modes menu to enable spectrometer motion.

Again, acquire a sufficient number of data acquisitions, select the data by dragging the mouse and use the Output | Plot Count Data menu to graph the results.

## Deadtime Calibration

To calibrate the deadtime constants for each spectrometer, StartWin may be utilized. StartWin is capable of producing a data set that is easily copied and pasted into Excel for use with Paul Carpenter's deadtime spreadsheet template.

First, set a sufficient count time (for example 60 seconds) for a precise measurement of intensities. If your interface supports sample (absorbed) current measurements, select this option also. Turn off the beam drift correction to acquire "raw" intensities. Then tune all spectrometers to the same element, for example Ti Ka on LiF and PET or Si Ka on PET and TAP. Use pure metal standards for high count rates and acquire 5 points at 10 different currents from about 10 nA to 200 nA.

Copy and paste the data set from the StartWin log window into a blank Excel spreadsheet and duplicate the beam current column if the absorbed current column is missing. See DEADTIME.DOC for more information on the data format required by DEADTIME.XLS. Finally create a column of the count time in the proper position (typically 1 second for data acquired by StartWin).

When the data is properly formatted, simply copy and paste the data set into the DEADTIME.XLS spreadsheet. The calculations on the new data set will be automatically updated and plotted. Use these new figures to update the values in the SCALERS.DAT file. Note that the actual calibrated deadtimes are entered on line 13, while the constant integer deadtime constants used to set the PHA interface, are specified on line 35 for the Cameca interface only.

Be aware however that deadtime is actually not a constant and can vary with the line energy of the x-ray being measured. For this reason, it is best if a pulse "stretcher" circuit can be placed in front of the counter-timer board to ensure that a "forced" deadtime is used to mask the actual deadtime of the spectrometer (as is done with the Cameca PHA hardware). If the pulse stretcher is adjusted to produce pulse widths greater than the worst deadtime seen on the spectrometer than the deadtime of the pulse stretcher (which is constant) can be used for the software deadtime correction. This will result in a much more accurate deadtime correction even at very high count rates.

## PHA

The StartWin PHA window allows the user to acquire PHA, Bias or Gain scans on all spectrometers at the same time. For the PHA scans, the program will measure the x-ray intensity as the baseline is varied from its current value up to the maximum baseline value with a window width based on the differential between the baseline and window and the number of points to be acquired.

**Pulse Height Analysis (PHA) Parameters**

| Define spectrometer PHA parameters |          |           |          |           |  |
|------------------------------------|----------|-----------|----------|-----------|--|
|                                    | Baseline | Window    | Gain     | Bias      | Inte/Diff                                  |
| 1                                  | 1.00     | 9.00      | 32.      | 1674.     | <input type="checkbox"/> Differential 1.23 |
| 2                                  | 1.00     | 9.00      | 16.      | 1764.     | <input type="checkbox"/> Differential 1.31 |
| 3                                  | .30      | 9.70      | 32.      | 1750.     | <input type="checkbox"/> Differential 1.56 |
| 4                                  | 2.00     | 8.00      | 64.      | 1650.     | <input type="checkbox"/> Differential 1.32 |
| 5                                  | .45      | 9.55      | 16.      | 1790.     | <input type="checkbox"/> Differential 1.04 |
| 6                                  |          |           |          |           | <input type="checkbox"/> Differential      |
| 7                                  |          |           |          |           | <input type="checkbox"/> Differential      |
| 8                                  |          |           |          |           | <input type="checkbox"/> Differential      |
| 9                                  |          |           |          |           | <input type="checkbox"/> Differential      |
|                                    | Bias Low | Bias High | Gain Low | Gain High | Scan Baseline                              |
| 1                                  | 1500.    | 1800.     | 4.       | 128.      | 4.00                                       |
| 2                                  | 1500.    | 1800.     | 4.       | 128.      | 4.00                                       |
| 3                                  | 1500.    | 1800.     | 4.       | 128.      | 4.00                                       |
| 4                                  | 1500.    | 1800.     | 4.       | 128.      | 4.00                                       |
| 5                                  | 1500.    | 1800.     | 4.       | 128.      | 4.00                                       |
| 6                                  |          |           |          |           |  |
| 7                                  |          |           |          |           |  |
| 8                                  |          |           |          |           |  |
| 9                                  |          |           |          |           |  |

| Detector Parameters |           |               |           |           |               |
|---------------------|-----------|---------------|-----------|-----------|---------------|
|                     | Slit Size | Slit Position | Det. Mode | Slit Size | Slit Position |
| 1                   |           |               | GPC       | 6         |               |
| 2                   |           |               | XPC       | 7         |               |
| 3                   |           |               | GPC       | 8         |               |
| 4                   |           |               | GPC       | 9         |               |
| 5                   |           |               | XPC       |           |               |

**Scan PHA**

Count Time
Intervals
Width

.5
20
.500000

**Scan Bias or Gain**

Use the bias or gain scan to determine the bias or gain value necessary for a given gain or bias setting to center the intensity in the PHA distribution

Count Time
Intervals
Width

.5
40
7.50000

**Scan Gain**

Count Time
Intervals
Width

.5
30
4.13333

For the Bias and Gain scans the baseline and window values are set to the Scan Baseline and Scan Window values while the bias or gain is scanned between the low and high range values. For bias scans the gain is set to the values displayed in the normal gain fields and for gain scans the bias is set to the value displayed in the normal bias fields.

Note that since JEOL instruments are limited to specific gain values, the gain values can only be modified using the spin buttons and the gain scanning fields and button are disabled.

## Peak/Scan Options

The Peak/Scan Options dialog allows the user to specify conditions for wavelength scans and peak calibrations (peak centering). To initiate a wavelength scan first indicate the start and stop positions in the Peak/Scan dialog and click OK. Then click the Start Wavescan button to start the spectrometers scanning. Data is saved to the log window and may also be plotted using the Output | Plot Count Data menu or exported to an ASCII file.

Peak centering is started by indicating the on-peak positions in the Peak/Scan dialog and clicking OK and then clicking the Start Peak Center button. Individual spectrometers may be disabled or enabled using the "Yes" checkboxes in the Peak/Scan dialog. Two additional peaking options are provided and described in the next section.

## Alignment Peak Center

From the Peak/Scan Options dialog the user may select "Alignment" peak centering. This option causes the program to perform a series of peak center procedures over a range of the Z stage axis. This test is useful in order to determine if the focal circle of the spectrometers are properly aligned. If the maximum intensity is obtained at the z-axis optical focus then the Rowland circle is properly adjusted.

Use the Alignment Parameters button to specify the z-axis range and number of peak calibrations to be performed for each spectrometer. To start the Alignment Peak Center check the Alignment Peak Center checkbox, click OK and click the Start Peak Center button.

## Calibrate Peak Center

The Calibrate Peak Center option allows the user to specify a number of elements and corresponding standards for the program to perform multiple peak calibrations. After the peak centering has been performed the program will save the theoretical spectrometer positions along with the actual measured peak positions and a least squares fit to the data for the purpose of calculating a variable offset function which in turn is used to calculate the actual peak position of any element on that spectrometer/crystal combination.

The default constant "offset" from actual spectrometer position is calculated as follows:

$$O_C = P_T - P_A$$

where:  $P_T$  = the theoretical spectrometer peak position

$P_A$  = the actual or measured spectrometer peak position

and is always applied unless the user performs the procedures described in this section. The default constant offset is usually sufficient if the miscalibration of the spectrometer mechanism is constant over the range of motion. However, if the offset varies as a function of spectrometer position, then the multiple peak calibration procedure will allow the use of variable offsets for each spectrometer-crystal combination for highest accuracy.

The multiple peak calibrations for each x-ray family (Ka, Kb, La, Lb, Ma and Mb) are stored in separate files which are named PROBEWIN-X.CAL where X is KA, KB, LA, LB, MA or MB depending on the line type. This due to the fact that slight differences in partially resolved alpha and beta-1,2 peaks behave differently for each line family. There the calibrations for each family are stored separately. This difference for K and L fits can be seen the figure at the end of this section.

Note that each spectrometer/crystal combination should be calibrated using multiple peaks. If less than three elements are specified the program will fit the data to a straight line. If less than two elements are specified the program will use a constant offset for the spectrometer correction which will have a similar accuracy to the default constant offset calculation.

Note also that a two crystal spectrometer will require two separate peak center procedures, one for each spectrometer/crystal combination although all tunable spectrometers can be calibrated automatically in a single run.

See the keyword "UseMultiplePeakCalibrationOffset" in the [software] section of the PROBEWIN.INI file for further instructions. This parameter must be set to a non-zero value to use this feature. Note that the calibration data is saved to an ASCII file called PROBEWIN-X.CAL and can be viewed using a text editor, but should not be edited by hand.

**Calibrate Spectrometers (multiple peak method)**

| Element Peak Parameters (choose 1 to 5 elements to peak) |                                  |                                  |                                  |                                  |                                  |                          |
|--|----------------------------------|----------------------------------|----------------------------------|----------------------------------|----------------------------------|--------------------------|
| Spectrometer   | Element 1                        | Element 2                        | Element 3                        | Element 4                        | Element 5                        | Current Fit Coefficients |
| 1 PET  | <input type="button" value="▼"/> | .000000 .000000 0.00e+00 |
| 2 LIFH   | <input type="button" value="▼"/> | .000000 .000000 0.00e+00 |
| 3 PET  | <input type="button" value="▼"/> | .000000 .000000 0.00e+00 |
| 4 PET  | <input type="button" value="▼"/> | .000000 .000000 0.00e+00 |
| 5 LIF  | <input type="button" value="▼"/> | .000000 .000000 0.00e+00 |
| 6  | <input type="button" value="▼"/> |                          |

| Element Peak Standards (choose a standard for each element/xray) |                                  |                                  |                                  |                                  |                                  | Automate Flags                                   |
|--|----------------------------------|----------------------------------|----------------------------------|----------------------------------|----------------------------------|--|
| Spectrometer   | Standard 1                       | Standard 2                       | Standard 3                       | Standard 4                       | Standard 5                       |  |
| 1 PET  | <input type="button" value="▼"/> | <input type="checkbox"/> Calibrate Spectrometer! |
| 2 LIFH   | <input type="button" value="▼"/> | <input type="checkbox"/> Calibrate Spectrometer! |
| 3 PET  | <input type="button" value="▼"/> | <input type="checkbox"/> Calibrate Spectrometer! |
| 4 PET  | <input type="button" value="▼"/> | <input type="checkbox"/> Calibrate Spectrometer! |
| 5 LIF  | <input type="button" value="▼"/> | <input type="checkbox"/> Calibrate Spectrometer! |
| 6  | <input type="button" value="▼"/> | <input type="checkbox"/> Calibrate Spectrometer! |

| Element Peak Positions (edit the starting peak center position if necessary) |                      |                      |                      |                      |                      | OK                                  |
|--|----------------------|----------------------|----------------------|----------------------|----------------------|-------------------------------------|
| Spectrometer   | Position 1           | Position 2           | Position 3           | Position 4           | Position 5           | Cancel                              |
| 1 PET  | <input type="text"/> | <input type="radio"/> Use Ka Series |
| 2 LIFH   | <input type="text"/> | <input type="radio"/> Use Kb Series |
| 3 PET  | <input type="text"/> | <input type="radio"/> Use La Series |
| 4 PET  | <input type="text"/> | <input type="radio"/> Use Lb Series |
| 5 LIF  | <input type="text"/> | <input type="radio"/> Use Ma Series |
| 6  | <input type="text"/> | <input type="radio"/> Use Mb Series |

Load Calibrate Spectrometer Fields Using Previous Peak Positions

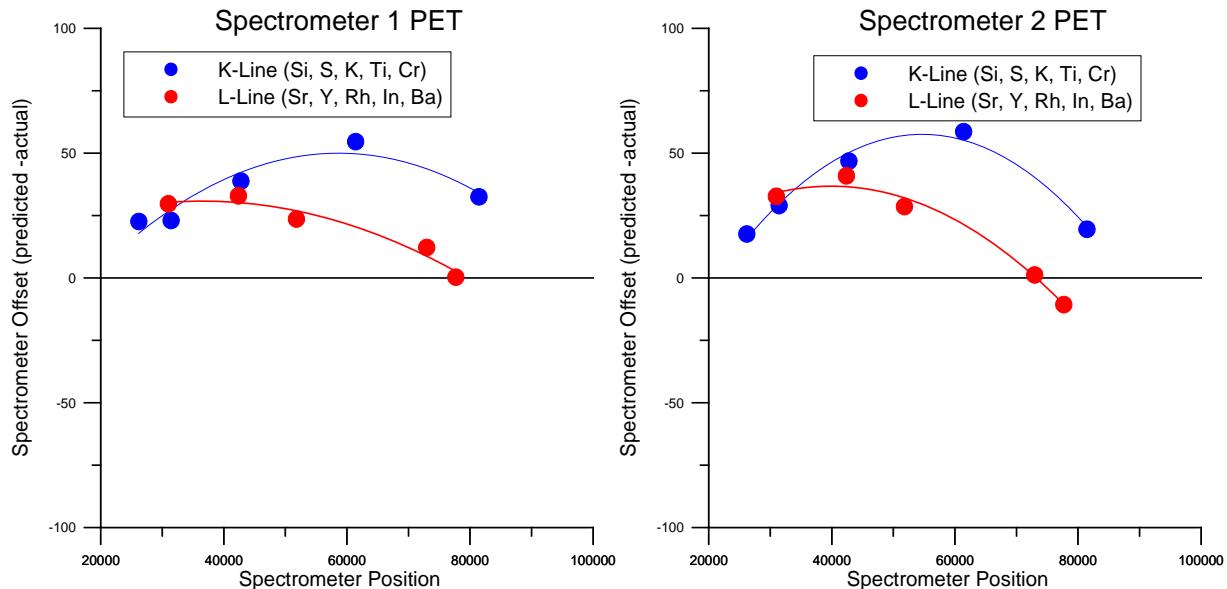
To start the multiple peak calibration acquisition click OK here and in the Peak/Scan Options dialog and then click the Start Peak Center button in the Count Acquisition window.

When performing multiple peak calibrations be sure to run graphical PHA distribution scans (from the PHA dialog which is accessed using the PHA button) using the lowest energy line specified for each spectrometer/crystal combination to make sure that the detector bias and gain are properly set. The baseline and window settings should be wide enough to allow for a range of energies since the entire spectrometer range will normally be utilized.

In addition, it is recommended that the count time in the Peak/Scan dialog be set to a sufficiently long time to provide proper statistics for the calibration, at least 20-40 seconds. Also each standard must be properly digitized and the position confirmed before the multiple peak calibration procedure is performed (it is important that the optical focus be precisely adjusted for each standard used in the procedure).

Use the Calibrate Parameters button to set the elements and standards for each spectrometer/crystal combination. To start a multiple peak center, check the Calibrate Peak Center checkbox, click OK and click the Start Peak Center button. To perform multiple peak centers on the other crystals, use the Move dialog to flip crystals and use the Calibrate Parameter dialog for each additional crystal.

When a spectrometer/crystal combination has been properly calibrated, the program will automatically load the "corrected" peak position for an element based on the fit to the measured spectrometer positions. This may be tested using the element/x-ray target positions in the Move dialog. The "corrected" spectrometer positions will also be utilized in the Elements/Cations dialog of **Probe for EPMA** for new elements not loaded from the element Setup database or another file or sample setup.



### Spectrometer Calibration Offsets for Ka and La lines on PET Crystals

A modification allows the user to specify the starting peak position for the peak center procedure for each element on each spectrometer. This is especially useful in cases where the theoretical peak position is completely off the peak due to severe misalignment of the spectrometer. The starting peak center position loaded is by default based on the theoretical peak position or the previously stored actual peak position from the .CAL file (if available). Which default starting peak position is loaded depends on whether the Load Peak Center Start From Previous Peak Positions checkbox in the Calibrate Peak dialog box is checked (default is checked).

A new post-scan option allows the user to confirm that the peak position has been accurately determined by the software peak center method. Note that the post-scan range is 5% (+/- 2.5%) of the pre-scan range.

### **Multiple Peak Calibration for Spectrometer to Angstroms Calculations**

When **Probe for EPMA** calculates the calibrated angstrom of a given spectrometer position, by default the program bases the conversion on the constant offset calculated at the element peak position for each analytical channel using the following standard expression:

$$\text{angstroms} = \frac{(P_S + P_O) * K_S * 2d * (1 - K)}{2d_{LIF}}$$

where:  $P_S$  = the position for the spectrometer

$P_O$  = the measured or calibrated spectrometer offset from the theoretical (constant or variable)

$K_S$  = the conversion factor for spectrometer units to LIF angstroms (SCALERS.DAT)

$2d$  = the 2d spacing of the spectrometer crystal

$K$  = the crystal constant (CRYSTALS.DAT)

$2d_{LIF}$  = the 2d for LIF (4.0267)

To calculate a variable spectrometer offset, the program utilizes a series of peak center calibrations performed in the StartWin application as described above. The multiple peak calibration data is stored in the .CAL files described above and the following is a small part of such a file produced by running a series of peak calibrations on an SX-51 microprobe where the spectrometer positions range roughly from 22000 to 86000.

|      |           | "PET"        |       | 5   |               |
|------|-----------|--------------|-------|-----|---------------|
| "si" | "ka"      | 81454.48     | 81426 | 14  |               |
| "s"  | "ka"      | 61415.59     | 61354 | 327 |               |
| "k"  | "ka"      | 42776.79     | 42730 | 374 |               |
| "ti" | "ka"      | 31430.02     | 31403 | 22  |               |
| "cr" | "ka"      | 26186.62     | 26175 | 24  |               |
| 2    | -107.9718 | 5.904327E-03 |       |     | -5.182411E-08 |

The above calibrations are for the PET crystal on spectrometer two using K line intensities. The calibration is based on 5 peak centers using Si, S, K Ti and Cr. These elements are designed to cover the full range of the spectrometer crystal whenever practical. The first column of numbers are the theoretical calculated peak positions and the second column of numbers are the actual or measured peak positions. The offset between these two sets of spectrometer positions is fit to a 2<sup>nd</sup> order polynomial and the coefficients are stored on the last line of the data set as can be seen above. The actual application of the variable offset for the purposes of spectrometer position to angstrom conversion is as follows:

First, the program calculates the expected peak position for the calibration element used for the wavescan based on the variable offset obtained from the calibration coefficients measured above using the polynomial expression:

$$P_E = P_{On-Peak} + (K_1 + K_2 * P_{On-Peak} + K_3 * P_{On-Peak}^2)$$

where:  $K_1, K_2, K_3$  = the calibration coefficients from the multiple peak calibration polynomial fit

$P_{On-Peak}$  = the theoretical on peak position of the calibration element

This expected on-peak position from the multiple peak calibration coefficients is used to calculate the difference from the actual measured on-peak position as follows:

$$O_V = P_A - P_E$$

where:  $P_A$  = the actual or measured on-peak spectrometer position

$P_E$  = the expected spectrometer position based on the previous calculation

This spectrometer position offset is used to temporarily modify the variable offset calibration intercept coefficient (first term) obtained from the .CAL file as follows:

$$K'_1 = K_1 - O_V$$

where:  $K'_1$  = the intercept coefficient from the multiple peak calibration polynomial fit

$O_V$  = the difference between the actual on-peak and the expected on-peak from the previous equation

In this way, the modified expected position calculation will obtain a modified variable offset equal to the constant offset when the spectrometer position is at the actual on-peak position, but the calculated offset will still vary based on the slope and curvature terms as the spectrometer position departs from the on-peak position as follows:

$$P'_E = P + (K'_1 + K_2 * P + K_3 * P^2)$$

where:  $K'_1$  = the modified intercept term of the multiple peak calibration polynomial fit

$K_2, K_3$  = the slope and curvature coefficients of the multiple peak calibration polynomial fit

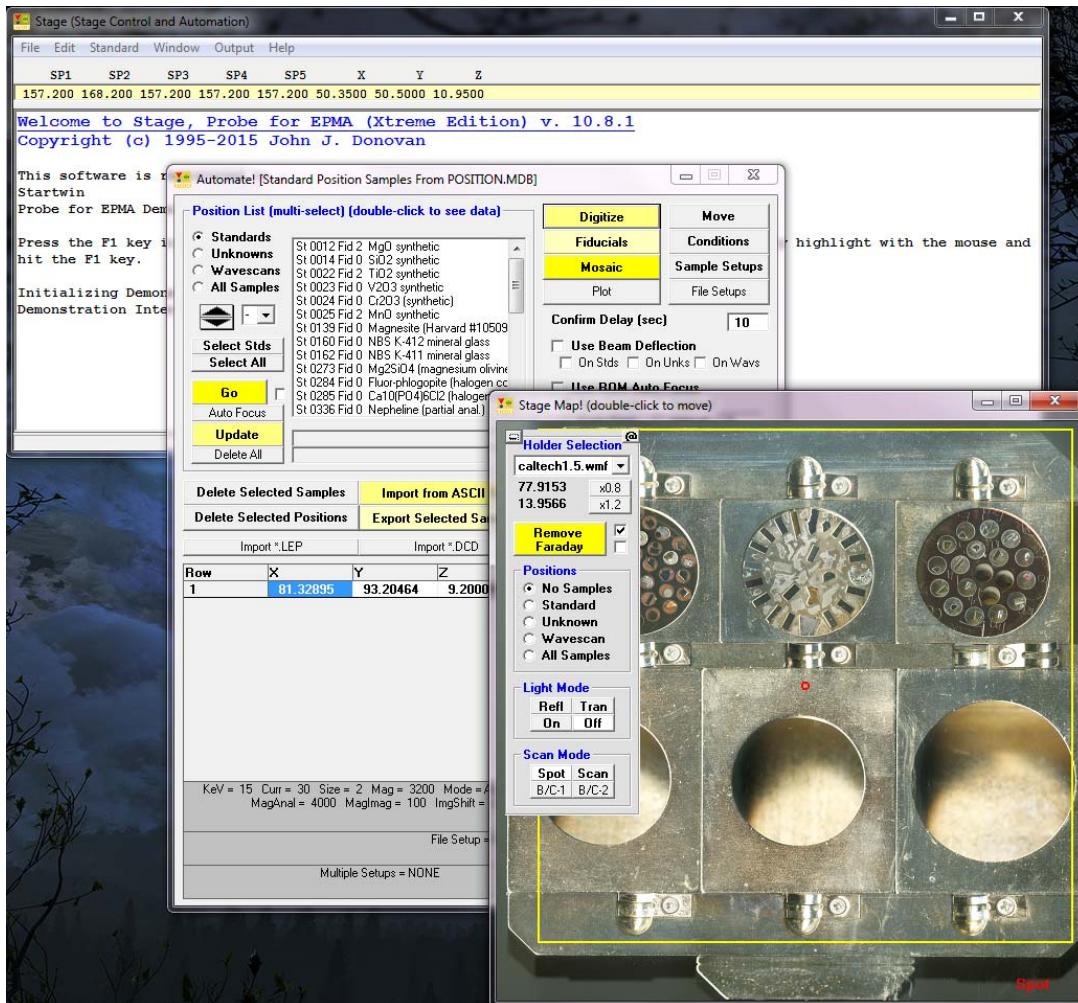
$P$  = the spectrometer position

By doing this, the angstrom calculation is based on the multiple peak calibration for high accuracy over the entire spectrometer range, but can still be re-calibrated simply by a single re-peaking of the spectrometer peak position in the future.

# Stage

## Overview

Program Stage is used to provide a simple interface for digitizing standard, unknown and wavescan position samples and ASCII import position files. Normally, the standard mounts are digitized using Stage and saved as ASCII .POS files so that they may be imported into the Position database file from the Automate! window of **Probe for EPMA** at any time later. Stage can also be used for coordinate transformation and confirmation of sample positions.



# Description

The STAGE application is a simple utility application for digitizing and confirmation of standard, unknown or wavescan coordinates. It is intended specifically for the digitization of pre-digitized standard mounts in the microprobe, but can also be used off-line on a suitably equipped light microscope for digitizing unknown samples referenced to fiducial markings for precise relocation of small features in the electron microprobe.

Most of the functionality in STAGE is included in the larger **Probe for EPMA** application and help for these functions can be found in the **Probe for EPMA** documentation and help sections. The following specific functions are unique to STAGE.

## Digitize Positions

The Digitize Positions menu item is found under the Window menu and is a subset of the functions found in the Automate! window of **Probe for EPMA**. Although STAGE contains the full Digitize button feature set of **Probe for EPMA**, the only automation action that can be initiated is through the Confirm button. This button will cause the program to automatically proceed to each selected position sample for confirmation of the digitized positions.

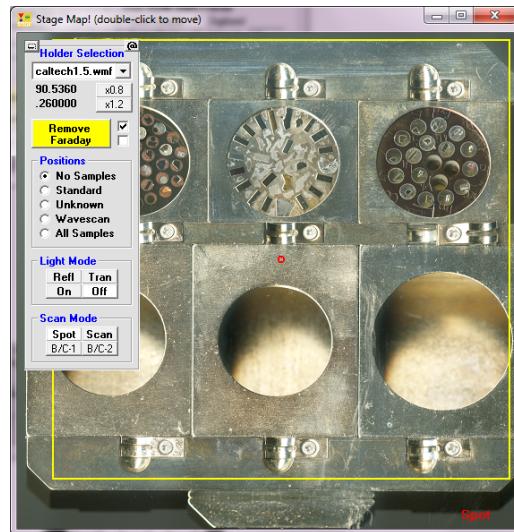
Although the Stage application only allows confirmation of position coordinates, it contains the full set of coordinate fiducial and transformation routines and can therefore be utilized on an off-line light microscope for digitizing of standard or unknown samples.

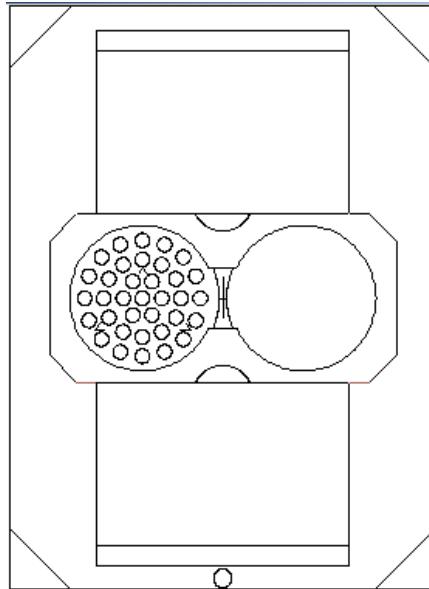
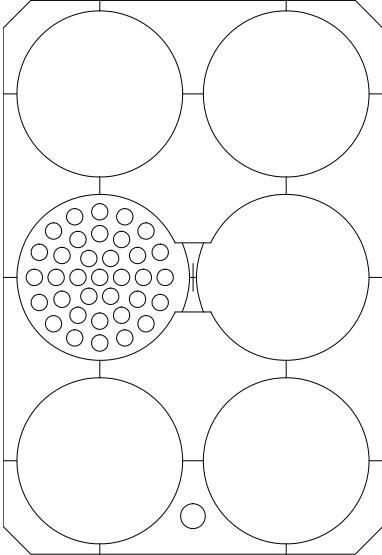
## Stage

The Stage button will open a Stage Bit Map window to allow the user to move to a stage position by simply double-clicking on a specific area on an image of the stage. More than one image file (up to MAXBITMAP%) can be specified in the PROBEWIN.INI file under the "Standards" section. To select another stage bit map simply select the file from the list box. The current stage position is indicated as a red circle.

The frame containing the stage bit map list box, cursor position and Faraday/Beam Blank button can be dragged to a different area of the window if the present position covers an area of interest. The minus button minimizes the stage bitmap selection and cursor position display. The "@" button loads a window that can be used to calibrate the currently displayed stage bit map. The calculated X and Y min and max values need to be entered into the PROBEWIN.INI file.

To re-size the Stage Bit Map window simply drag any corner of the window to the desired size and shape. To enable the Stage button or add additional Stage Bit Maps files to the program, see the [Standard] section in the PROBEWIN.INI file. Some sample Stage Bit Map files are shown here for the Cameca SX100/SXFive and JEOL 8900/8200/8500/8x30 respectively:





### **Position File (\*.POS) File Formats**

A data field is provided (see PositionImportExportFileType in the PROBEWIN.INI file section) to export and import the sample setup numbers to the .POS ASCII position files in addition to the sample name and coordinates. If this option is utilized, care must be taken to ensure that the setup numbers match the appropriate setups in the **Probe for EPMA** database file that they are intended to be acquired with.

Another data field is provided (see PositionImportExportFileType in the PROBEWIN.INI file section) to export and import the file setup names to the .POS ASCII position files in addition to the sample name and coordinates. If this option is utilized, care must be taken to ensure that the Probe file setups specified in the position file, actually exist as specified by the directory path.

### **Import \*.LEP**

This menu will load a custom position file. This format is a fixed length field ASCII file that contains off-line digitized data from an automated Leica light microscope. This format is intended for use with Canadian Geological Survey laboratory .LEP position files only. The file format is as follows:

|        |         |        |       |     |      |   |   |
|--------|---------|--------|-------|-----|------|---|---|
| REF #1 | -195279 | 80485  | -2130 | LEP | SH # | 2 | 1 |
| REF #2 | -55671  | 9769   | -5227 | LEP | SH # | 2 | 1 |
| REF #3 | -222595 | -40036 | 3740  | LEP | SH # | 2 | 1 |
| OXIDE  | -192879 | 80303  | -2237 |     |      |   | 1 |
| OXIDE  | -187449 | 80303  | -2237 |     |      |   | 1 |
| OXIDE  | -182519 | 80472  | -2237 |     |      |   | 1 |
| OXIDE  | -162776 | 80178  | -3229 |     |      |   | 1 |

See additional information on the "Import From ASCII File" button in the Automate! window and also the PositionImportExportFileType parameter in the PROBEWIN.INI file.

### **Import \*.DCD**

This menu will load a custom position file produced by Microbeam Services optical digitizer. This format is as follows:

The first two rows are the two reference points P1. The following S1.... are sample points.

Columns 1, 2, 3 are XYZ

Column 4 is the analysis setup, which can be pre-programmed.

Column 5 is the sample description

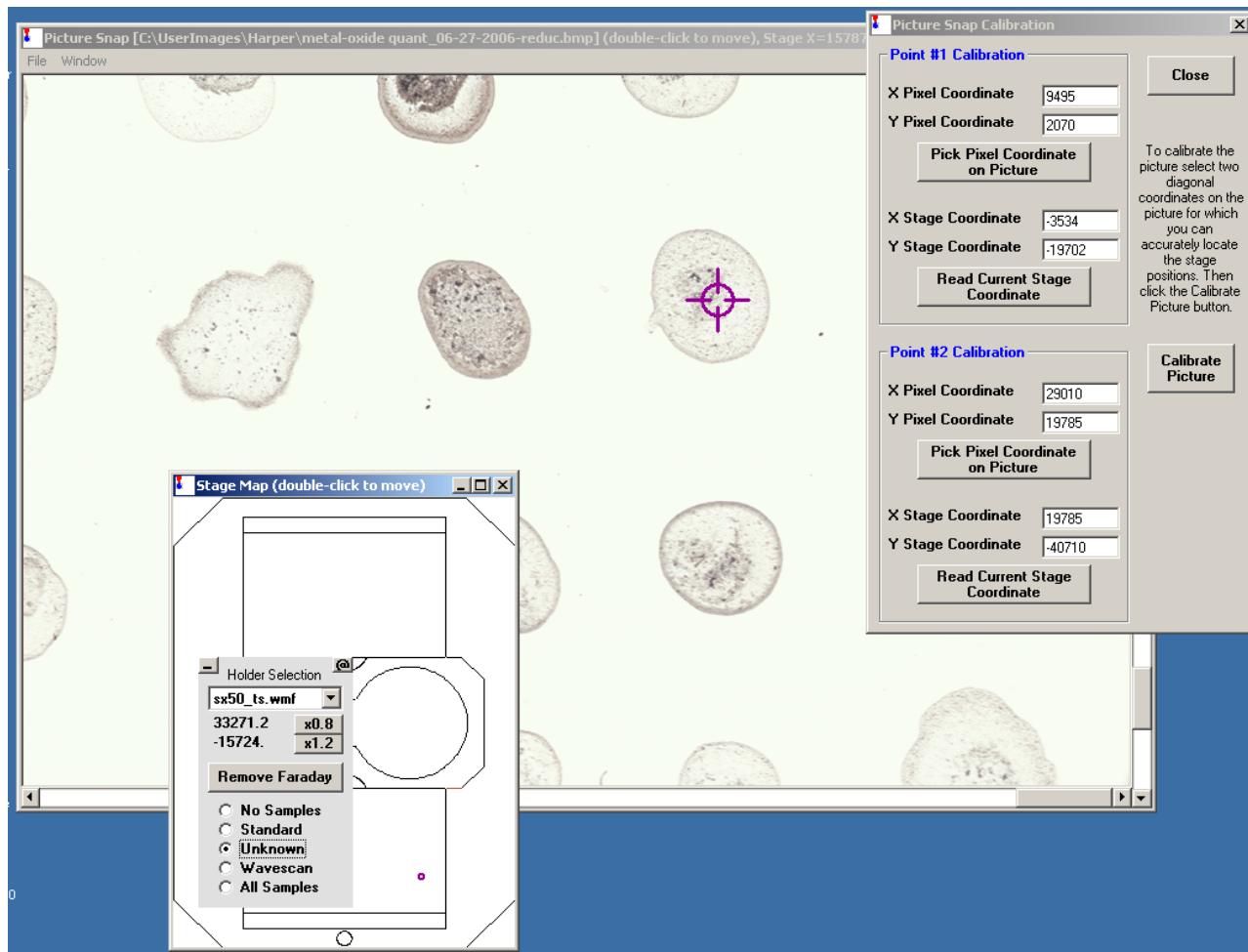
Column 6 is the sample number (P for reference, S for single point...)

Columns 7-9 are used as number within dataset etc. and not really important.

This format is intended for use with Microbeam Services .DCD position files only.

### **Picture Snap**

The Picture Snap window allows the user to import any image from a slide or flatbed scanner and calibrate the image to the probe stage coordinates using any two diagonal points or three points to account for sample rotation, on the image so that current stage position can be displayed on the scanned image and that sample areas of interest can be located easily.



The current stage position location is shown as a target cursor on the calibrated picture and is updated continuously as the stage position is adjusted by the user. To move to any point on the image simply double click the image coordinate.

### **Digitizing Standard Mounts with More than 128 Standards**

When creating digitized standard files for the standard mounts, it should be noted that the program has a limit of MAXSTD% standards (normally equals 128). However, this limitation is easily overcome for standard mounts that contain more than 128 standards. The following instructions will explain how to bypass the 128 standard limit in the STAGE digitize feature:

1. First clear the current position database (POSITION.MDB) by clicking the Standards option and then clicking Delete All from the Digitize! window.

2. Next from the Standard | Add/Remove Standards To/From Run menu (from the main STAGE window) select the first 128 standards to be digitized.
3. Now digitize these 128 standards using the Digitize Sample Positions dialog. Typically only a single random point need be digitized, but more will be utilized by the software if they are digitized explicitly.
4. Next, exit the STAGE program and re-start it. Open the Digitize! window. The first 128 standard sample positions will be listed.
5. Now, go back to the Standard | Add/Remove Standards To/From Run menu and add an additional (up to) 128 standards for the next phase of digitizing.
6. Digitize these next 128 standards, again using the Digitize Sample Positions dialog.
7. Repeat steps 4, 5 and 6 if necessary.

## **Import Positions From Cameca PeakSight**

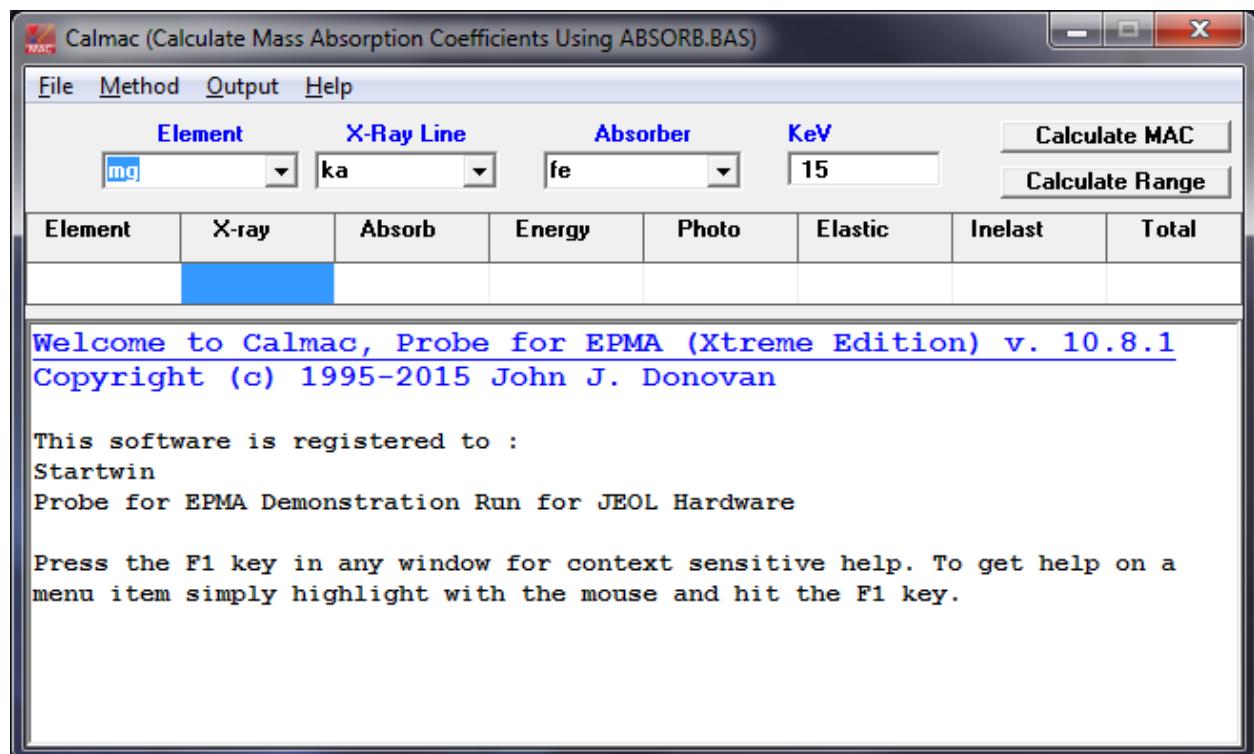
1. To import standard positions from the Cameca PeakSight software, select the Import Positions From Cameca PeakSight (Sx.mdb) menu and then select the Cameca PeakSight sx.mdb file to import. The program will automatically process the standard position data into the position database.

# CalMAC

## Overview

CalMAC is a small utility for calculating mass absorption coefficients (MACs) for arbitrary energies or ranges of energies. The calculations are based on the tables from McMaster et.al. and are valid for x-ray energies above 1 keV.

W. H. McMaster, N. Kerr Del Grande, J. H. Mallet and J. H. Hubbell, "Compilation of x-ray cross sections ", Lawrence Livermore Lab., 1969."



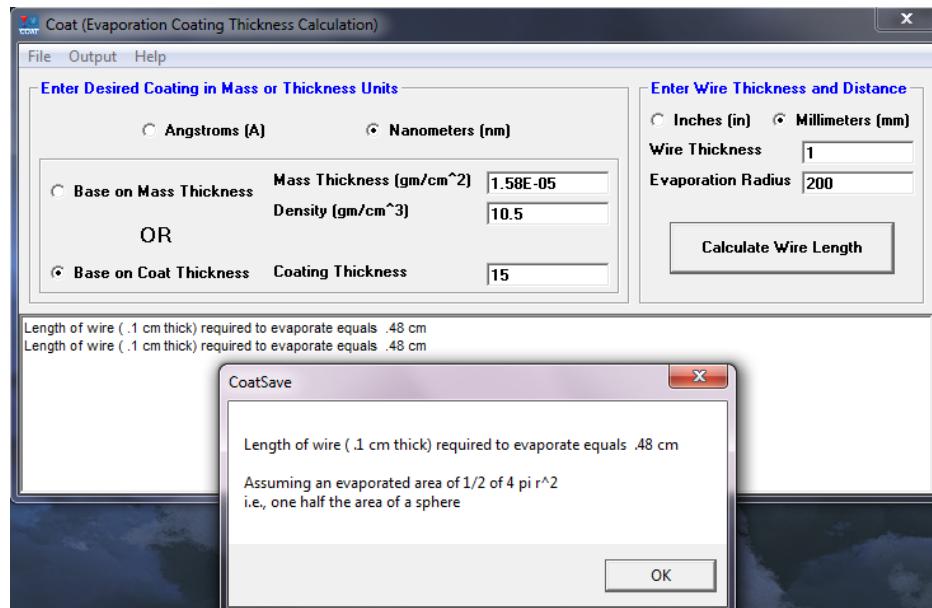
## Features

In the Method menu, one may also select calculations based on MACJTA (Armstrong) and MAC30 (Heinrich).

# Coat

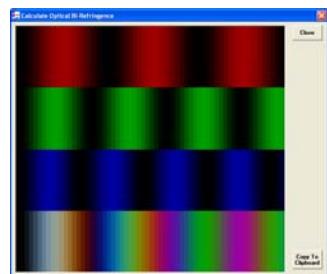
## Overview

Coat.exe is a small utility for calculating the length of wire of a given diameter necessary to evaporate a specified thickness of material at a given radius assuming a depositional hemisphere such as a relatively flat tungsten or tantalum evaporation "boat".



## Features

Coat will also display a synthetic birefringence graphic calculated by summing the wavelengths of red, green and blue light from their wavelengths.

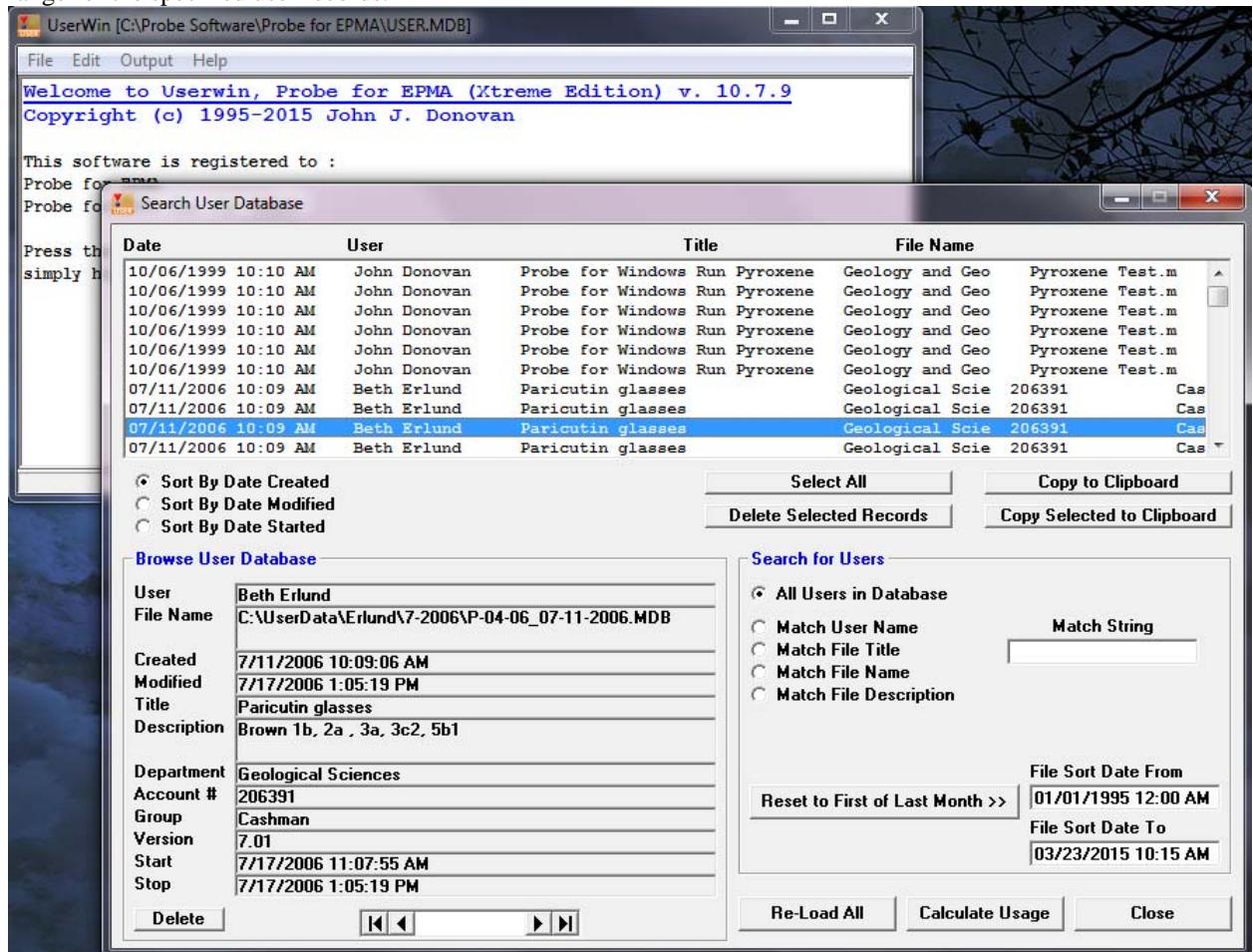


# UserWin

## Overview

Program USERWIN.EXE is another small applet for accessing the probe user database that is automatically maintained by **Probe for EPMA** whenever the program is opened in "real-time" mode (that is, interfaced to the microprobe hardware).

It contains extensive search and list capabilities and can calculate the hourly usage of the microprobe in a specified date range for the specified user records.



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## Description

The USERWIN application is a small utility for access to the real-time user database. All user activity (when interfaced to the microprobe hardware) is automatically recorded and can be accessed for statistical and recharge purposes.

Hourly usage can be calculated for the displayed records and the selection of records can be based on a number of criteria including name, title of run, filename and date range. The calculated hours can be printed out for laboratory documentation purposes and is also saved automatically to a tab delimited file.

### Creating a New User Database

The operator may find that it is necessary to create a new user database. This might be to take advantage of new database fields that may have been added to the program. For example, beginning in version 2.05, three additional user customizable fields were added for improved documentation purposes. If the current user database was created using a version prior to version 2.05, then a new user database will have to be created to access these new fields. To create a new user database and retain the information in the current user database, simply follow the steps below:

1. Backup your current user database. The user database is in the **Probe for EPMA** directory and is named **USER.MDB**. In fact, backup all files in the Probe for EPMA directory to be safe.
2. Run the Userwin application. The default user database is opened automatically. Move the Search for Users window to one side so that the main window is visible (do not click the Close button).
3. Click File | Export and confirm the **USER.DAT** export file. All user records will be exported. This takes only a second or two.
4. Click File | Close to close the current user database.
5. Click File | Import and select the import file. Click OK to select the previously created default **USER.DAT**. Next, select a new user database. Click OK to select the default **USER.MDB** and confirm the overwrite operation.
6. Modify the File Info fields as desired. The records will be imported in a few seconds and the Search for Users window will display the new user database.

### Calculating Hourly Usage

To calculate the hourly usage of all user records, simply click the Calculate Usage button in the Search for Users window. To calculate the hourly usage of the most recent records, click the Reset to First of Last Month button and then click the Update User Database List. Then click the Calculate Usage button again. In a similar fashion, any selected subset of records may be obtained using the various Search For Users options and the hourly usage calculated.

Each time the hourly usage is calculated, output is directed to both the log window and an ASCII tab delimited text file for importing into Excel or other spreadsheet software. The log window output or selection may also be copied to the clipboard or printed.

### Using the Data Cursor

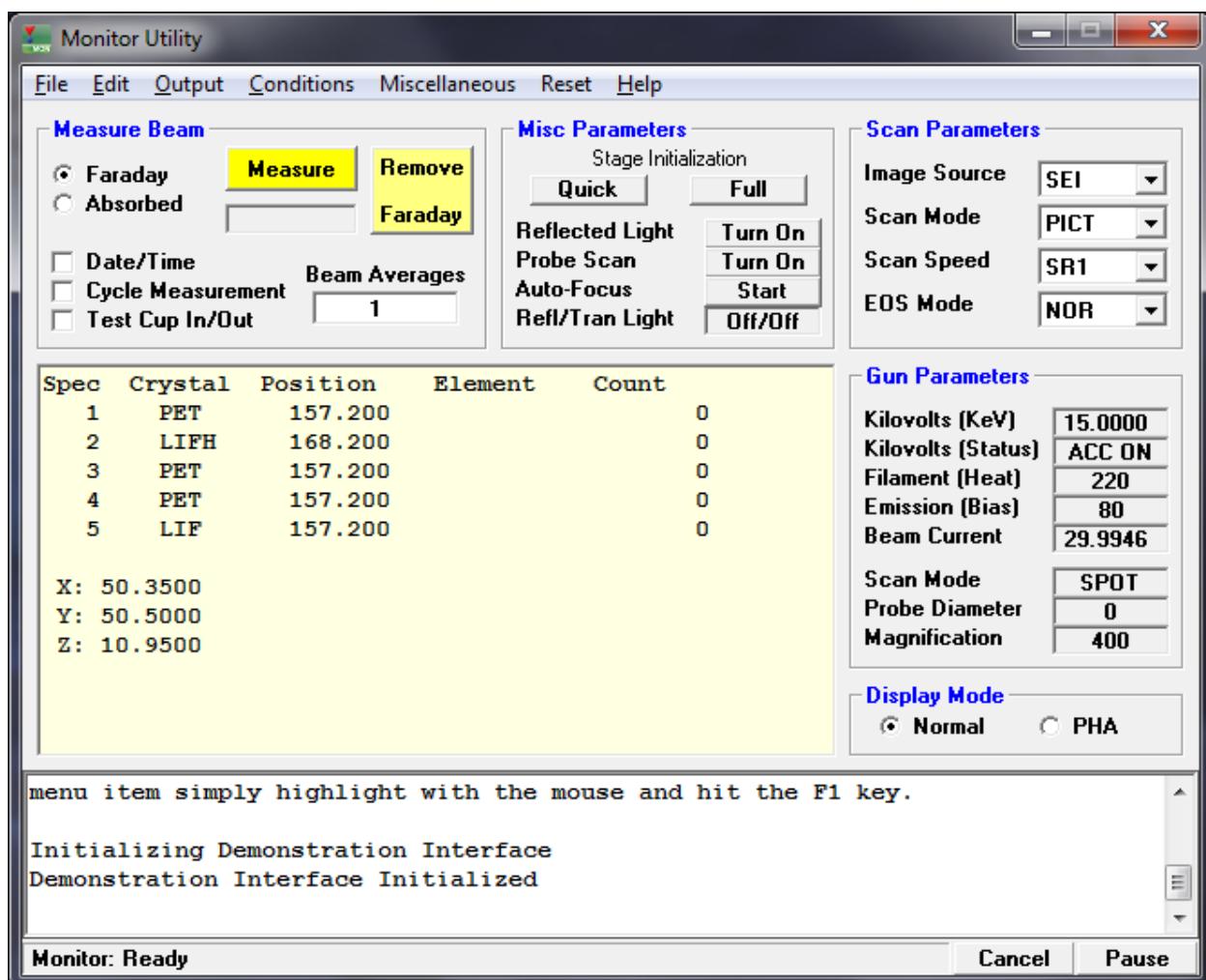
In the lower left of the Search User Database window a number of database fields and a data cursor are visible. Using the data cursor allows one to browse through all or selected records. The outside arrows on the data cursor move to the beginning or end of the database and the inside arrows move one record at a time forward or backward.

## **Modifying the Custom Data Fields**

Several user database fields may be defined by the user for special documentation or accounting purposes. They are all text fields, up to 64 characters in length. To change the field labels and/or the default text, see the instructions under the [general] section of the PROBEWIN.INI configuration file documentation above.

# Monitor

## Overview

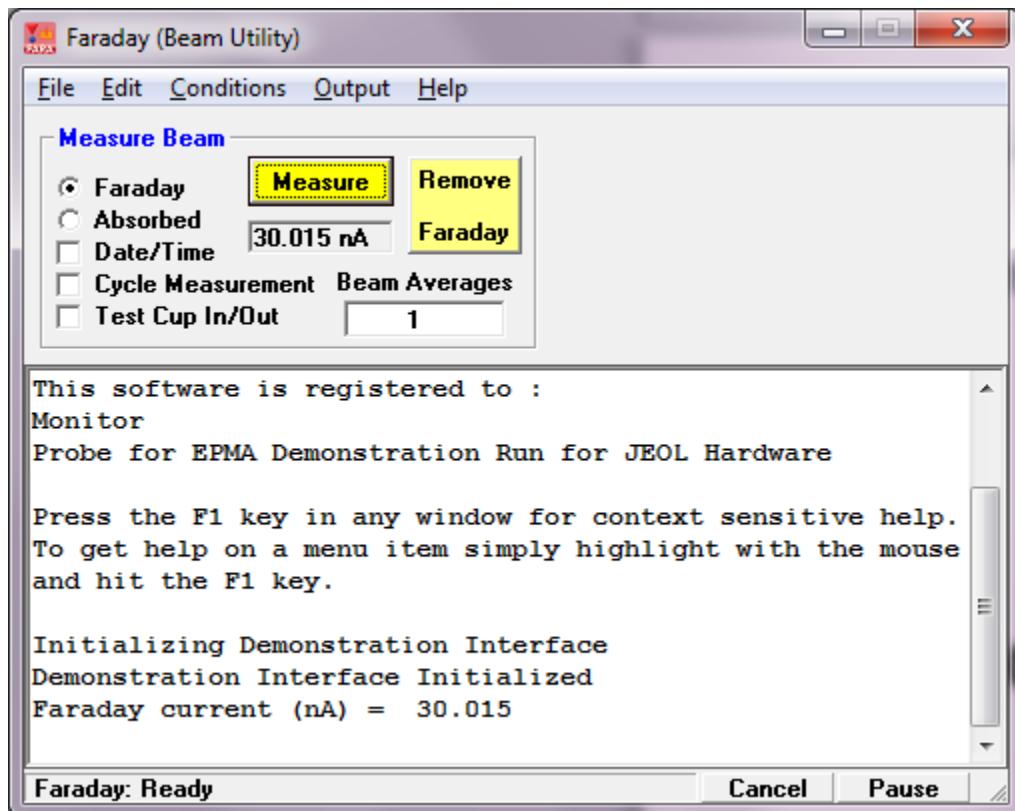


Program Monitor is only used for the JEOL 8900/8200 and 8500 instrument interfaces. It is essentially a PC version combining the EOS and WDS monitor applications on the JEOL workstation. Use Monitor to quickly change image sources, scan speeds and start an auto-focus. It can also load and save column condition files and perform re-sets of the JEOL system controller.

Note that the view can be easily toggled between Spectro/Stage and PHA display modes.

# Faraday

## Overview

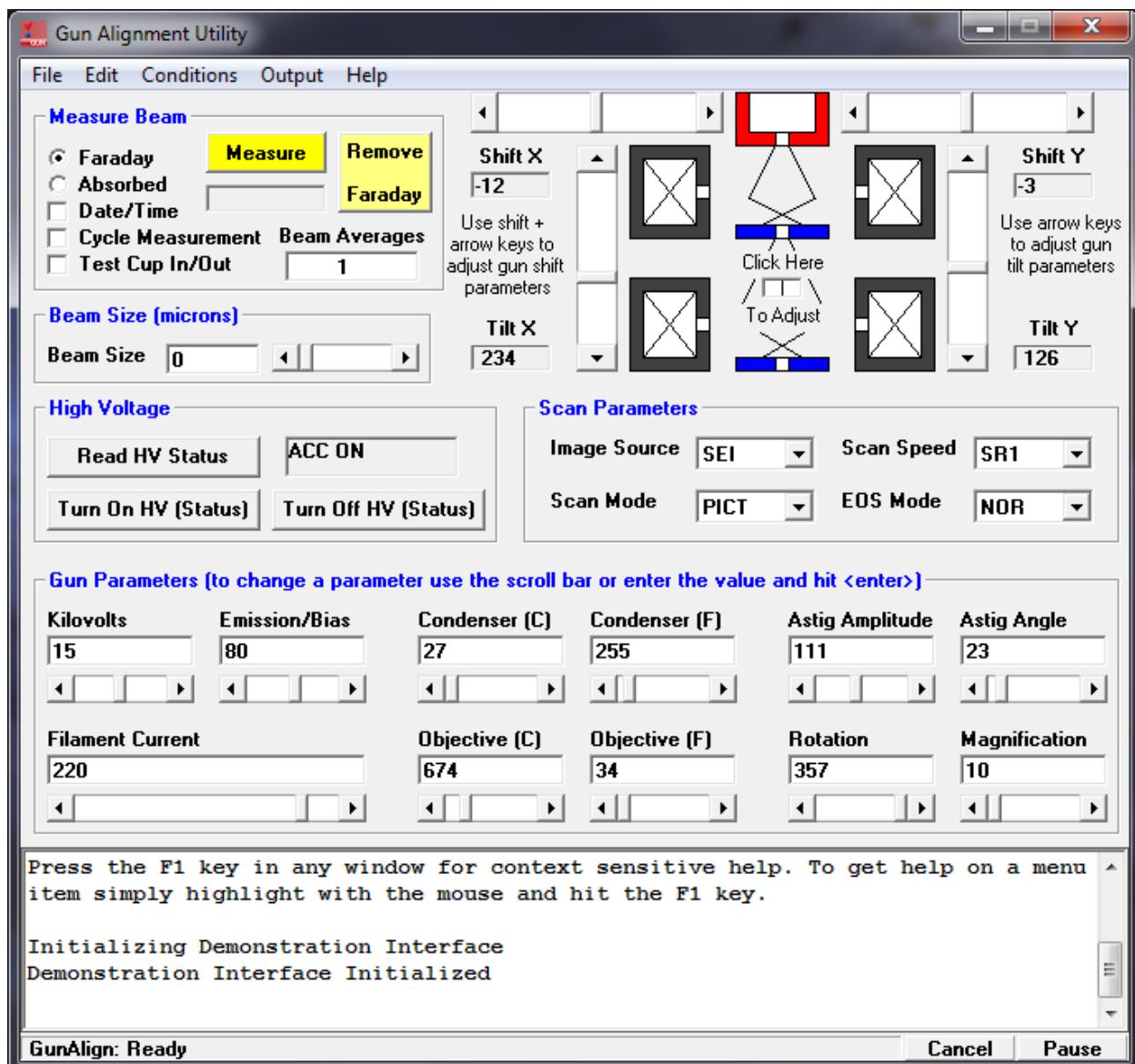


The Faraday application is a small utility useful for controlling the faraday cup and making beam current measurements. It has a cycle control which can be used to continuously monitor the beam current for stability measurements.

It can also move to the specified stage position for an in-stage faraday cup and can be used to load and save column conditions.

# GunAlign

## Overview



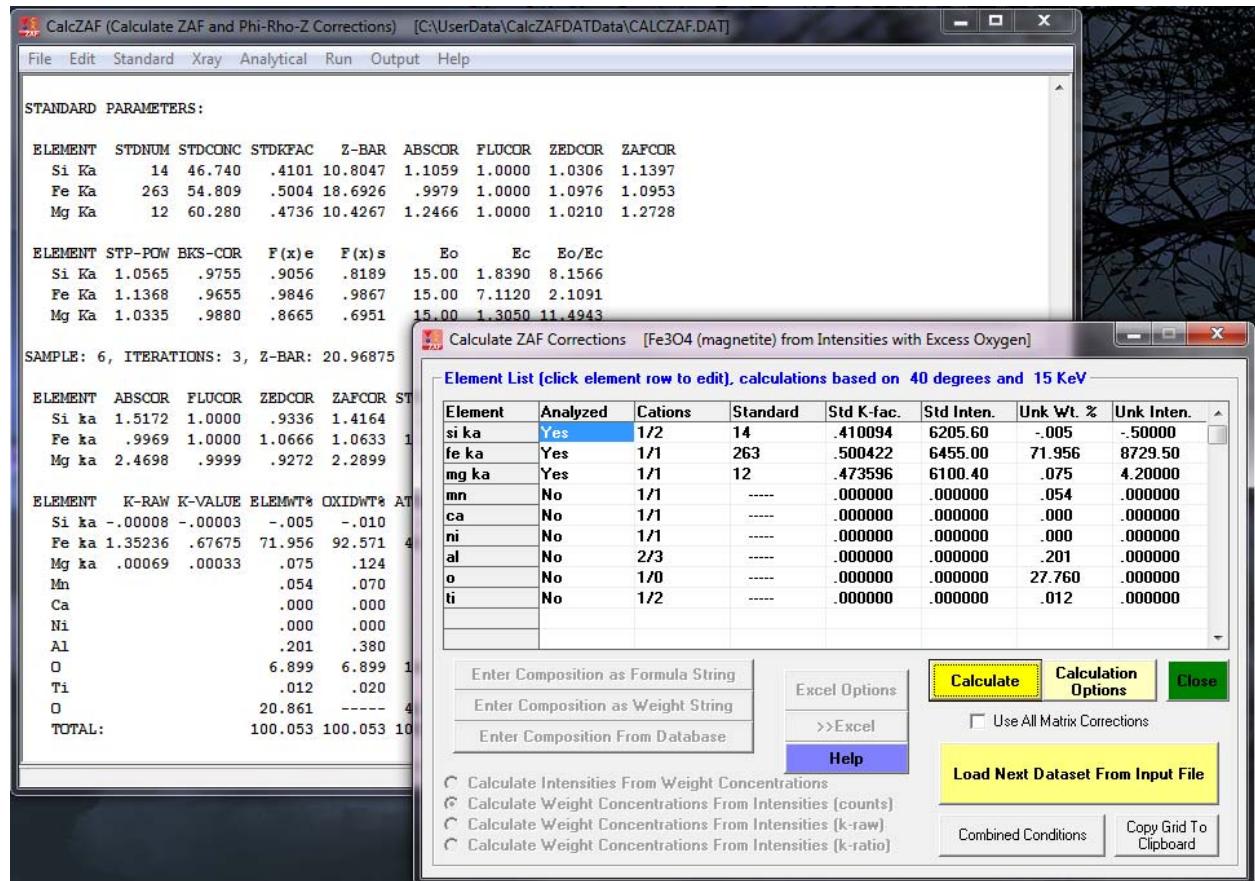
The GunAlign applet can perform gun tilt and shift alignment for SX100/SXFive and JEOL 8900/8200 and 8500 instruments. Note that for ease of tuning the cursor keys can be used to adjust the tilt and shift by clicking on the “Click Here To Adjust” field in the center of the column schematic and using the up and down cursor keys to control left and right gun tilt and the <shift> and cursors keys to adjust the gun shift.

To change the Gun Parameters one can use the scroll bars or click the text field above the control. The field will turn yellow to indicate that the commands are not being sent, until the user hits the <enter> key.

One can also load and save column conditions, measure beam current and change beam size using the various controls in this application.

# CalcZAF

## Overview



Program CALCZAF.EXE is another small applet for "stand-alone" calculation of ZAF and Phi-Rho-Z matrix correction parameters. CalcZAF can accept composition to calculate intensities (k-ratios) or calculate composition from intensities using normalized k-ratios, raw k-ratios or unknown and standard counts. K-ratios or counts measured on compound standards may be utilized, the software will load standard compositions from the Standard composition database.

CalcZAF can also calculate large amounts of binary composition data and create data suitable for plotting error histograms. In addition CalcZAF can apply geometric corrections to intensities for particles (cubes, prisms, spheres and

fibers) and thin films (only for those films or foils less than 1 microgram/centimeter<sup>2</sup> or assuming density = 1 approximately 0.01 um or 100 nm or 1000 angstroms) based on various adjustable parameters. Note that the accuracy of the particle method depends on bracketing a single particle in the scanned or defocused beam (overscanning) and applying judgement as to the particle shape and the distance along the beam axis. Note that the scan or defocus size must be equal to the particle dimension.

Finally CalcZAF has very powerful tools for calculating detection limits (concentration at given integration time or integration time given concentration) and also for calculating x-ray and electron distances in elements and compounds.

Note that Probe for EPMA can export a CalcZAF compatible input file of standard or unknown sample intensities or raw k-ratios for processing in CalcZAF. See the File | Open menu description.

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## Description

Program CalcZAF is a small utility program for stand-alone calculations using the ZAF and Phi-Rho-Z algorithms contained in the Probe for EPMA software

CalcZAF allows the user to perform various types of calculations for concentrations to intensities or intensities to concentrations:

Mode 0. Concentrations to intensities- this option calculates the elemental k-factors for the specified concentrations. This is similar to the normal standard k-factor calculation performed for the primary standards by Probe for EPMA. Note that although Probe for EPMA only performs an "elemental" standard k-factor calculation, CalcZAF can also perform an "oxide" standard k-factor calculation. Requires elemental concentrations.

Mode 1. Count intensities to concentrations- this calculation calculates elemental concentrations using both unknown and standard intensities and an assigned standard to calculate the standard k-factor for each "analyzed" element. Intensities for the standard and unknown must be in the same units.

The program will automatically calculate the standard k-factors for each element, however the correct standard must first be assigned from the list of standards in the run. See the Standards | Add/Remove Standards To/From Run menu item to add standards to the run that can be assigned to the "analyzed" elements.

Mode 2. Raw k-ratio intensities to concentrations- this calculation is similar to the above, except that the standard intensity is not required to be entered as it is already "included" in the raw unknown k-ratio. Note that a standard must still be assigned to each "analyzed" element.

Mode 3. K-ratio intensities to concentrations- this calculation is based on an already "normalized" elemental k-ratio (to 1.000). Therefore, only the element, x-ray and unknown "intensity" must be entered for the calculation to be performed. No standard assignments are required, however the program will assign a "dummy" standard assignment of 32767 to each analyzed element.

Output of results are normally to the log window although one can use the Copy Grid To Clipboard button or use the Open Link To Excel menu under the Output menu to create a live link to Excel that the user can utilize to export a number of data types to the spreadsheet as desired using the >>Excel button.

Note that the Output Oxide Weight Percents and Output Normalized Oxide Percents options will be disabled if both the Calculate With Stoichiometric Oxygen and Display Results As Oxide Formulas option are not selected. Note also that the Calculate With Stoichiometric Oxygen option cannot be selected if oxygen is an analyzed element.

A new feature is to use the Combined Conditions button to specify different operating voltages for each analyzed element.

## Importing Concentration or Intensity Data From a Disk File

CalcZAF allows the user to create ASCII files for importing concentration or intensity data from a disk file. An example file is supplied which is called CALCZAF.DAT. See the File | Open and File | Close menu for loading input data from file. Click the Calculate button to perform the calculations and click the Load Next Dataset from Input File button to load the next data set.

Each data set in the input file consists of 3 or more lines. The first line contains the calculation mode (defined above), number of analyzed and specified elements, operating voltage and takeoff angle. The second line contains the oxide or elemental calculation flag (1=oxide,2=elemental), the element by difference, element by stoichiometry to stoichiometric oxygen, the stoichiometric ratio, the element by stoichiometry relative to another element, the element to which the stoichiometry is calculated relative to and the relative ratio. The third (and subsequent lines) are the element data for each element including the element symbol, the x-ray symbol ("ka", "kb", "la", "lb", "ma", "mb" or " " for a specified concentration), the number of cation atoms, the number of oxygen atoms, the standard number assigned as the primary standard (from the Probe for EPMA STANDARD.MDB default database), the concentrations (for intensity calculations), the unknown intensity and the standard intensity (if required). This structure is repeated as often as desired for each data set. The program will automatically detect if the last data set has been reached.

The structure is shown below in pseudo-code:

```
' Read calculation mode (0, 1, 2, or 3), number of elements, kilovolts and takeoff, (optional sample name)
Input #3, CalcMode%, LastChan%, Kilovolts!, Takeoff!, (SampleName$), (X, Y, Z stage positions)

' Read oxide/elemental mode, difference, stoichiometry, relative
Input #3, OxideOrElemental%, DifferenceElement$, StoichiometryElement$, StoichiometryRatio!,
RelativeElement$, RelativeToElement$, RelativeRatio!

' Loop on each element
For i% = 1 To LastChan%
Input #3, Elsyms$(i%), Xrsyms$(i%), NumCat%(i%), NumOxd%(i%), StdAssigns%(i%), ElmPercents!(i%),
UnkCounts!(i%), StdCounts!(i%)
next i%
```

Note:

CalcMode% = 0 for calculation of k-ratios from concentrations

CalcMode% = 1 for calculation of concentrations from unknown and standard intensities

CalcMode% = 2 for calculation of concentrations from "raw" k-ratios (no standard intensities necessary)

CalcMode% = 3 for calculation of concentrations from "normalized" k-ratios (no standards necessary)

Note:

OxideorElemental%=1 calculate oxide output based on stoichiometry

OxideorElemental%=2 calculate as elemental output (default)

Note: all strings (element symbols, etc.) must be in double quotes, elements not analyzed (specified concentrations or calculated) are indicated by a blank (empty double quotes) x-ray line string. If the element is a specified concentration, be sure to give the concentration in elemental weight percent for the "ElmPercents!(I%)" parameter and leave the count intensity fields zero.

**An example of a CalcZAF input file demonstrating each calculation mode is shown here:**

```
0,2,15,40,"MgO K-ratio"
' 1st dataset (calculates MgO intensities)
2,"","","0.0","","0.0
"mg","ka",1,1,0,60.0,0.0,0.0
"o","ka",1,0,0,40.0,0.0,0.0
```

```

1,3,15,40.,"Fe2SiO4"          ' 2nd dataset (calculates concentrations from unk and std intensities)
2,"","","0.0","",",0.0
"fe","ka",1,1,895,0.0,7568.1,10265.7
"si","ka",1,2,14,0.0,1329.4,5268.2
"o","ka",1,0,895,0.0,2519.6,2498.1
2,3,15,40.,"Fe2SiO4"          ' 3rd dataset (calculates concentrations from "raw" k-ratios)
2,"","","0.0","",",0.0
"fe","ka",1,1,895,0.0,.96283,0.
"si","ka",1,2,914,0.0,0.00003,0.
"o","ka",1,0,895,0.0,1.09972,0.
3,2,20,40.,"MgO"              ' 4th dataset (calculates concentrations from normalized k-ratios)
2,"","","0.0","",",0.0
"mg","ka",1,1,0,0.,.418853,0.0
"o","ka",1,0,0,0.,.190763,0.0

```

## Calculating Error Histograms from Binary Composition Input Files

CalcZAF can also calculate error histograms from appropriate input files containing binary compositions and experimentally measured k-ratios. See the Analytical | Calculate Binary Intensities menu.

The user may select an input file of binary compositions for the calculation of error histograms. The calculations are based on the current ZAF and MAC selections. The output is filtered using the options in the Binary Calculation Options dialog.

Note that all input files for binary calculations have a .DAT extension and must conform to the format specified below. Do not confuse these files with CalcZAF input files or other files with the extension .DAT. Use a text editor to examine the input file if necessary.

Some sample input files of binary compositions are supplied, e.g., AUAGCU2.DAT, NISTBIN.DAT and POUCHOU.DAT. An example of the file format is seen here:

```

79  29   2   5  15.  52.5  .8015  .1983  .7400  .0
79  29   2   5  15.  52.5  .6036  .3964  .5110  .0
79  29   2   5  15.  52.5  .4010  .5992  .3120  .0
79  29   2   5  15.  52.5  .2012  .7985  .1450  .0

```

The data file format assumes one line for each binary. The first two columns are the atomic numbers of the two binary components to be calculated. The second two columns are the x-ray lines to use ( 1 = ka, 2 = kb, 3 = la, 4 = lb, 5 = ma, 6 = mb, 7 = Ln, 8 = Lg, 9 = Lv 10 = Li, 11 = Mg, 12 = Mz, 13 = specified, that is, do not calculate the intensity). The next two columns are the operating voltage and take-off angle. The next two columns are the weight fractions of the binary components. The last two columns contains the k-exp values for calculation of k-calc/k-exp. If the second element x-ray line is "specified" (iray = "13"), then no experimental k-ratio value is required.

Note that although the k-ratio file format example above has two emitting elements (so that two k-ratio error sets will be calculated), only one emitting element per line is actually required, as in the following example (where the x-ray line of "13" in the fourth column indicates an "absorber only", specifically an element by difference from 1.0):

```

13  26   1   13  20  52.5  0.241  .0  0.124  .0
13  26   1   13  25  52.5  0.241  .0  0.098  .0
13  26   1   13  30  52.5  0.241  .0  0.083  .0
26  13   1   13  20  52.5  0.759  .0  0.736  .0
26  13   1   13  25  52.5  0.759  .0  0.742  .0
26  13   1   13  30  52.5  0.759  .0  0.748  .0

```

---

## Log Window

The main window of CalcZAF is a scrollable text window which will record all output and calculations. This log window is a fully cut and paste enabled text window which can be used to transfer data or analysis results to any other Windows application such as a word processor or spreadsheet.

Simply click and drag the mouse to select text and use the <ctrl> c, <ctrl> x or <ctrl> v key combinations to copy, cut or paste text to or from the Windows Clipboard. Or use the Edit menu for these and other Clipboard functions.

The size of the log window buffer can be specified in the PROBEWIN.INI file and is limited only by the amount of memory available. If the Save Log to Disk File option is selected from the Output menu, then all log window output is mirrored to a text file, including any text manually entered by the user using the keyboard. The log window font and font size is specified in the PROBEWIN.INI file and may be changed during a run from the Output menu. To view a copy of the log file during a run, select the View Disk Log option from the Output menu.

# CalcZAF Window Details

## ZAF Calculation Window Details

The ZAF Calculation window provides an interface for viewing and entering x-ray compositions and intensities.

### Calculate

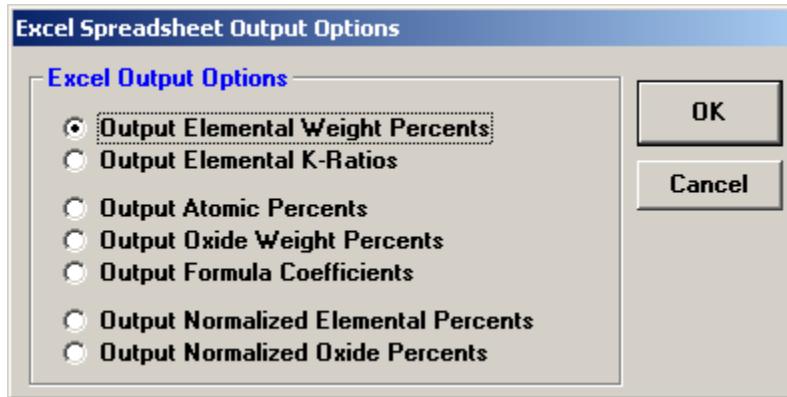
This button will start the current intensity or composition calculation. The results will be displayed in both the log window and the grid control. Use the Copy Grid to Clipboard button to copy the grid contents for pasting into another document.

### Close

This will close the currently open CalcZAF input data file. This is used in cases where it is desired to open another CalcZAF input data file.

### Excel Options

Use these option in conjunction with the Output | Open Link To Excel menu to select the data type to be exported to the currently open Excel spreadsheet.



Note that the Output Oxide Weight Percents and Output Normalized Oxide Percents options will be disabled if both the Calculate With Stoichiometric Oxygen and Display Results As Oxide Formulas option are not selected. Note also that the Calculate With Stoichiometric Oxygen option cannot be selected if oxygen is an analyzed element.

### >>Excel

Click this button to export the current calculation to the currently open Excel spreadsheet using the specified format in the Excel Options dialog. The column headings are automatically output if the elements or output mode changes.

## **Combined Conditions**

Use this dialog to specify different operating voltages for each analyzed element. Note that takeoff angle is not editable at this time.

## **Enter Composition As Atom String**

The button allows the user to enter a sample composition for matrix calculations based on a formula string entered by the user. Parentheses and subscripts are supported. Although all lower case chemical symbols are accepted, in situations where the atomic symbol is ambiguous always indicate the proper elements using proper chemical symbol capitalization. For example: caco3 will be interpreted as CoCO3 (cobalt carbonate), therefore use the capitalization CaCO3 which will be properly interpreted as calcium carbonate.

To utilize more significant digits simply multiple the actual formula by 10 or 100, for example: "Ca80Mg20C100O300".

## **Enter Composition As Weight String**

The button allows the user to enter a sample composition for matrix calculations based on a weight string entered by the user. For example, to enter magnesium iron olivine one could enter: "Si18Fe13Mg26O42".

## **Enter Composition As Standard**

The button allows the user to enter a sample composition for matrix calculations based on a standard composition from the standard database selected by the user.

## **Calculation Options**

### ***Calculate With Stoichiometric Oxygen***

CalcZAF allows the user to display the results of an analysis in oxide weight percents based on the cation ratios defined for each element. In addition the user may select to calculate oxygen by stoichiometry if oxygen is not an analyzed element. If oxygen is measured or calculated by stoichiometry, the program will automatically calculate the actual excess or deficit oxygen in the analysis if the Display As Oxide option is selected.

If the element to be calculated by formula basis, difference or stoichiometry is not already in the sample setup, it must be added as a "not analyzed" element using the Elements/Cations button first, by simply clicking any empty row in the element list and typing in an element symbol with a blank x-ray line.

Note that if the sample being analyzed is a standard and oxygen is not measured, then the program will automatically specify any oxygen from the standard database. However, if the sample is an unknown, then the program will add in any specified oxygen as "excess" oxygen into the matrix correction, if the user has specified any element concentrations.

### ***Calculate As Elemental***

This option calculates all elements as pure elements without the addition of stoichiometric oxygen in the matrix correction calculation.

### ***Element by Difference***

An element by difference can be included in this way also to account for their effect on the x-ray intensities. The element by difference must be one of the specified elements in the sample setup. Note that the this option should be utilized carefully, because the calculation will always result in a 100% total which could mask the results of a bad acquisition.

### ***Stoichiometry to Oxygen***

Another useful feature for the analysis of carbonate or borate samples in an oxide run is the use of the "element by stoichiometry to the stoichiometric element (oxygen)" option. With this feature the analyst can analyze just the cations (Fe, Mg, Ca, etc.) in a sample and have the oxygen calculated by stoichiometry and another specified element (usually C

for carbonates or B for borates) calculated relative to oxygen. In the case of carbonates, for example CaCO<sub>3</sub>, carbon is always in the ratio 1 to 3 to oxygen.

Therefore by simply specifying C by stoichiometry relative to the stoichiometric element (oxygen) at 0.333 (1 divided by 3) the correct amount of both oxygen and carbon will be incorporated into the ZAF matrix correction and totals without analyzing for either. This calculation should only be used with compounds where the ratio to oxygen is both known and unchanging.

Note that oxygen must be an analyzed or specified element before this calculation can be applied. One more point about element by stoichiometry to oxygen. Consider the example of a trace element analysis of several metals in an alumina (Al<sub>2</sub>O<sub>3</sub>) matrix. If Al and O are not to be analyzed, yet the user desires to have Al<sub>2</sub>O<sub>3</sub> added to the matrix correction, how can this be accomplished?

There are two ways this can be achieved. One way would be to simply specify Al by difference and calculate oxygen by stoichiometry. The program will then correctly add in the proper amount of stoichiometric Al<sub>2</sub>O<sub>3</sub> to the matrix correction for each analysis line. The other way is to use the element by stoichiometry to oxygen calculation as discussed below.

Adjust the cation ratios of the metals to elemental stoichiometry (one cation and zero anions). Next, select "element by stoichiometry to oxygen" and (in this example) select "al" as the element by stoichiometry. To achieve a 2 to 3 ratio, next enter "0.666" Al atoms per O atom. Run the calculation and note that Al<sub>2</sub>O<sub>3</sub> was not added to the matrix calculation! What happened? In this example, the user had selected a cation ratio for the analyzed elements of all elemental atoms, and since there was zero oxygen to begin the iteration, the program never got to add the Al which then never added the stoichiometric oxygen! How can this be avoided? Simply specify some small concentration of an oxide element (for instance SiO<sub>2</sub>) in the specified element concentration, say 0.05 %. This will give the iteration a chance to get started, and allow it to converge on a very close approximation of the Al<sub>2</sub>O<sub>3</sub> by difference!

### ***Stoichiometry to Another Element***

Yet another option for recalculation is the element relative to another element (by stoichiometry). This calculation is similar to the "element by stoichiometry to oxygen" as described below, however the user may select any other analyzed or specified element as the stoichiometric basis element.

### **Load Next Dataset From Input File**

This button will load the next sample dataset from the currently open CalcZAF input data file.

### **Copy Grid To Clipboard**

This button will copy the contents of the grid to the Windows clipboard. The contents can then be pasted into another windows document or spreadsheet.

# CalcZAF Menu Details

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## File

The File menu can be used to open an existing data file, close the current run, print the current log window contents or log window selection or exit the program.

### Open CalcZAF Input Data File

The Open menu allows the user to select an existing CalcZAF data file to process previously entered data. All CalcZAF input data files have an extension .DAT and must conform to the specific format described below. Do not confuse .DAT files for other applications with CalcZAF data files. If necessary inspect the input file with a text editor to determine if it actually conforms to the CalcZAF input format.

The structure is shown below in pseudo-code (all values separated by commas):

**' Read calculation mode (0, 1, 2, or 3), number of elements, kilovolts and takeoff, (optional sample name and stage coordinates)**  
Input #3, CalcMode%, LastChan%, Kilovolts!, Takeoff!, SampleName\$, Xpos!, Ypos!, Zpos!

**' Read oxide/elemental mode, difference, stoichiometry, relative**  
Input #3, OxideOrElemental%, DifferenceElement\$, StoichiometryElement\$, StoichiometryRatio!,  
RelativeElement\$, RelativeToElement\$, RelativeRatio!

**' Loop on each element**

For i% = 1 To LastChan%

Input #3, Elsyms\$(i%), Xrsyms\$(i%), NumCat%(i%), NumOxd%(i%), StdAssigns%(i%), ElmPercents!(i%),  
UnkCounts!(i%), StdCounts!(i%)

next i%

Note:

CalcMode% = 0 for calculation of k-ratios from concentrations

CalcMode% = 1 for calculation of concentrations from unknown and standard intensities

CalcMode% = 2 for calculation of concentrations from "raw" k-ratios (no standard intensities necessary)

CalcMode% = 3 for calculation of concentrations from "normalized" k-ratios (no standards necessary)

Note:

OxideorElemental%=1 calculate oxide output based on stoichiometry

OxideorElemental%=2 calculate as elemental output (default)

Note: all strings (element symbols, etc.) must be in double quotes, elements not analyzed (specified concentrations or calculated) are indicated by a blank (empty double quotes) x-ray line string. If the element is a specified concentration,

be sure to give the concentration in elemental weight percent for the "ElmPercents!(I%)" parameter and leave the count intensity fields zero.

Some sample CalcZAF input files are supplied, e.g., CALCZAF.DAT and CALCBIN.DAT.

### **Close CalcZAF Input Data File**

The Close menu allows the user to close the currently open CalcZAF database file. This can be used to close the current file before opening a different CalcZAF database file without re-starting the program.

### **Export CalcZAF Input Data File**

The export menu allows the user to save their work by exporting manually entered element data and intensities to an ASCII file for future use. The extension is .DAT and the file should be named so it can be recognized as a CalcZAF input data file.

### **Update CalcZAF Example Data Files**

Copies the latest distributed default CalcZAF example data files to the UserData folder for updated testing and demonstration.

### **Print Log**

This menu allows the user to print the entire contents of the log window or selected portions of the log window. Be sure to click the Selection field if only a text selection is to be printed, otherwise the program will print the entire log window text.

### **Print Setup**

This menu allows the user to change the default printer or printer setup options for the currently selected printer.

### **Exit**

This menu exits the program.

---

## **Edit**

### **Cut**

This menu cuts the selected text from the log window to the Windows Clipboard.

### **Copy**

This menu copies the selected text from the log window to the Windows Clipboard.

### **Paste**

This menu pastes text from the Windows Clipboard to the log window.

### **Select All**

This menu selects all text in the log window.

## **Clear All**

This menu clears all text from the log window.

---

# **Standard**

## **Standard Database**

This menu invokes a shell process to launch the Standard database application (STANDARD.EXE) as an asynchronous process. The standard database is used to maintain the database of standard compositions and also includes a number of features for exploring analytical conditions based on various standard compositions.

Since the shell process runs independently, it can be accessed during an automation or analysis procedure.

## **Select Standard Database**

This menu allows the user to load a non-standard database for various analytical situations. For example, if another microprobe laboratory sends you a probe database file that you would like to process but it is based on a different standard database than your default database, one can simply open the other laboratory's standard database by browsing to it using this menu which then temporarily assigns that database for the subsequent probe database operations.

Or perhaps you would like to process the same probe database with slightly different standard compositions to evaluate the effect of such differences on the same probe database intensities.

To return to using your default standard database select your default standard database using this menu or simply restart the program.

## **Edit Standard Coating Parameters**

The menu allows the user to specify conductive coating parameters for each standard in the run individually or all together. The coating element, thickness and density may be specified. The conductive coating parameters for the unknown specimen can be specified in the Calculation Options dialog available from the Calculate ZAF Corrections window.

Note that the coating parameters for standards and unknowns are ignored in the quantitative calculations until the electron absorption and x-ray transmission coating correction flags are explicitly turned on from the Analytical | Analysis Options menu dialog.

## **Add/Remove Standards To/From Run**

This menu allows the user to specify additional standards to be used in the currently open CalcZAF run.

Note that if the run is closed before any data is acquired for these newly added standards, then they will have to be added again, if the run is re-opened later to acquire data for them. This is because CalcZAF updates the list of standards in the run each time a database file is opened based on the standard samples that have been actually created.

All standards that need to be digitized, acquired or assigned, must be first added to the run using this window dialog.

---

# X-Ray

## X-Ray Database

This menu allows the user to view specified ranges of the NIST x-ray wavelength database. This database covers the wavelength range from approximately 0.5 to 100.0 angstroms and includes higher order reflections as well. Higher order reflections are reduced in intensity by 25% for each subsequent order to simulate the effect of PHA analysis.

The x-ray list range (Start Angstroms and Stop Angstroms) and minimum search intensity (Minimum Intensity) of the x-ray list can be specified by the user. Note that since the display is limited to about 1000 items, the program will automatically increase the minimum intensity until the returned list of x-ray lines is less than 1000. To view x-ray lines of lesser intensity, simply reduce the x-ray range and click the Re-Load button.

Note that angstrom values in the x-ray database list with Bragg reflection orders higher than one (Roman Numeral "I") are NOT corrected for refraction index corrections. However, KLM markers displayed in the Graph Data wavescan plot dialog are corrected using the equation  $A' = A * (1 - (K - (K / N^2)))$  where A is the uncorrected angstrom position, K is the refractive index from the CRYSTALS.DAT file and N is the Bragg reflection order.

Description of the X-ray Database (adapted from NIST documentation by C. Fiori)

The NIST x-ray database is based on 4985 (1st order) entries and includes all the measurable x-ray lines, satellites and absorption edges from under 100 eV to over 120 keV. Additionally, most of the x-ray lines and satellites are assigned a relative intensity (relative to the alpha-1 line in each family). The data base was assembled primarily from four sources:

- 1.) B.L. Doyle, W.F. Chambers, T.M. Christensen, J.M. Hall and G.H. Pepper "SINE THETA SETTINGS FOR X-RAY SPECTROMETERS", Atomic Data and Nuclear Data Tables Vol. 24, No 5, 1979.
- 2.) E.W. White, G.V. Gibbs, G.G. Johnson Jr. and G.R. Zechman "X-RAY WAVELENGTHS AND CRYSTAL INTERCHANGE SETTINGS FOR WAVELENGTH GEARED CURVED CRYSTAL SPECTROMETERS" Report of the Pennsylvania State Univ., 1964.
- 3.) J.A. Bearden "X-RAY WAVELENGTHS AND X-RAY ATOMIC ENERGY LEVELS" Rev. Mod. Phys., Vol. 39, No. 78, 1967.
- 4.) J.A. Bearden and A.F. Burr, "REEVALUATION OF X-RAY ATOMIC ENERGY LEVELS", Rev. Mod. Phys., Vol. 31, No. 1, 1967.

Each x-ray line or edge series as a function of atomic number was fit to a fourth degree polynomial. The fit was subtracted from the appropriate data and the residuals plotted and examined. In this way rogue entries could be identified and corrected. The resulting data base is considered to be sufficiently accurate for any application involving the Si (Li) x-ray detector and single crystal wavelength spectrometers.

Note that the last entry in the x-ray database window gives a code for the source of the entry. If the column is blank the source is reference 2. If the column contains the letter "C" the source is reference 1. If the letters "BB" appear, the source is reference 4. The letters "W,F" mean that reference 2 was used but the relative transition probability has been adjusted by Fiori. Reference 3 was used as a check since it is the source of many of the entries of reference 1.

In column 3 the notation KA1,2 means the entry is the weighted sum of the KA1 and KA2 in the ratio 2 to 1. For low atomic number the entries are not self-consistent since the data is from different sources. If the column begins with the capital letter S then the entry is a satellite line due to doubly ionized atoms. The relative transition values for these entries are only valid for electron excited specimens, and are, at best, estimates.

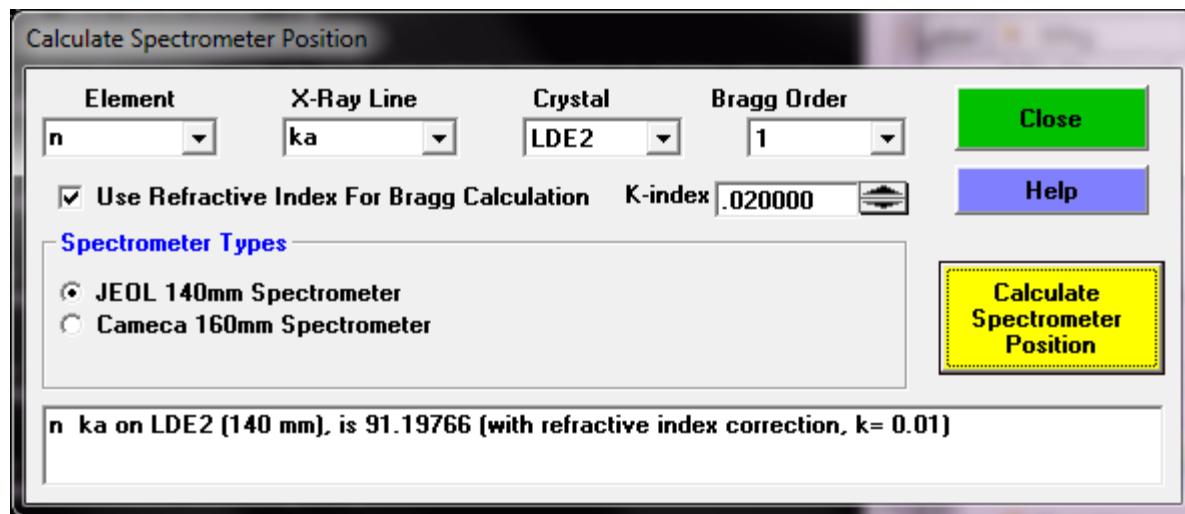
The following are Siegbahn to shell-transition notation conversions:

$$\begin{aligned} \text{KA} &= \text{KA1+KA2+KA3} \\ \text{KA1,2} &= (2*\text{KA1+KA2}) / 3 \end{aligned}$$

KA1 = K-L3  
KA2 = K-L2  
KA3 = K-L1  
KB = SUM(KBn)  
KBX = Metal  
KB1 = K-M3  
KB1' = KB1+KB3+KB5  
KB2 = (K-N3)+(K-N2)  
KB2' = K-N3  
KB2'' = K-N2  
KB3 = K-M2  
KB4 = (K-N4)+(K-N5)  
KB5 = (K-M4)+(K-M5)  
KB5' = K-M5  
KB5'' = K-M4  
Kd1 = K-O3  
Kd2 = K-O2  
LA = LA1+LA2  
LA1 = L3-M5  
LA2 = L3-M4  
LB1 = L2-M4  
LB10 = L1-M4  
LB15 = L3-N4  
LB17 = L2-M3  
LB2 = L3-N5  
LB3 = L1-M3  
LB4 = L1-M2  
LB5 = (L3-O4)+(L3-O5)  
LB6 = L3-N1  
LB7 = L3-O1  
LB9 = L1-M5  
LG1 = L2-N4  
LG11 = L1-N5  
LG2 = L1-N2  
LG3 = L1-N3  
LG4 = L1-O3  
LG4' = L1-O2  
LG6 = L2-O4  
LG8 = L2-O1  
Ll = L3-M1  
Ln = L2-M1  
Ls = L3-M3  
Lt = L3-M2  
Lu = (L3-N6)+(L3-N7)  
Lv = L2-N6  
MA1 = M5-N7  
MA2 = M5-N6  
MB = M4-N6  
MG = M3-N5  
MG2 = M3-N4  
MZ1 = M5-N3  
MZ2 = M4-N2  
Md = M2-N4  
Me = M3-O5

## Calculate Spectrometer Positions

This menu loads the Calculate Spectrometer Positions window which allows the user to calculate the spectrometer position for any element, x-ray and crystal combination and display the result in either JEOL or Cameca units.



There are also controls for specifying the crystal refractive index (k-value) to correct Bragg's Law for the refractive index of the crystal material. The traditional form of Bragg's Law is well known as:

$$n\lambda = 2ds\sin\phi$$

Taking the refractive index (k) into account (from Fournelle), the expression is:

$$n\lambda = 2ds\sin\phi * (1 - k/n^2)$$

## Emission Table

This menu lists a table of emission energies (or angstroms) for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

## Edge Table

This menu lists a table of absorption edge energies (or angstroms) for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

## Fluorescent Yield Table

This menu lists a table of fluorescent yield fractions for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

## MAC Table

This menu lists a table of MACs (mass absorption coefficients) for the specified element and its major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

The program will automatically load the values based on the selected default MAC table. See the Analytical | ZAF Selection section for a description of the MAC table choices.

## **MAC Table (Complete)**

This menu lists a table of MACs (mass absorption coefficients) for all element emitters and absorbers. Note that this output will be rather large, but is automatically saved to a text file for your convenience.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

The program will automatically load the values based on the selected default MAC table. See the Analytical | ZAF Selection section for a description of the MAC table choices.

## **Display MAC Emitter Absorber Pair**

This menu will list the mass absorption coefficient values for an emitter x-ray and absorber pair from each of the MAC tables existing in the Probe for EPMA folder. This is useful when trying to ascertain the accuracy of a particular mass absorption coefficient by comparing the values from available sources.

## **Edit X-Ray Table**

This menu items opens a small dialog for the purposes of editing single values in the default x-ray emission table. All values are entered in electron volt units (eV). Be aware that changes to this table will affect the calculation of x-ray intensities and compositions in the software.

## **Edit X-Edge Table**

This menu items opens a small dialog for the purposes of editing single values in the default x-ray edge table. All values are entered in electron volt units (eV). Be aware that changes to this table will affect the calculation of x-ray intensities and compositions in the software.

## **Edit X-Flur Table**

This menu items opens a small dialog for the purposes of editing single values in the default x-ray fluorescence yield table. Be aware that changes to this table will affect the calculation of x-ray intensities and compositions in the software.

## **Edit MAC Table**

This menu items opens a small dialog for the purposes of editing single values in the default mass absorption coefficient table. Be aware that changes to this table will affect the calculation of x-ray intensities and compositions in the software.

## **Convert ELEMINFO.DAT**

This menu will initiate a conversion routine to convert the original CITZAF element emission and edge energy ASCII files to the binary format used by CalcZAF. This will create the XLINE1.DAT, XEDGE1.DAT, XFLUR1.DAT x-ray files (rename to XLINE.DAT, XEDGE.DAT and XFLUR.DAT to be utilized by the Probe for EPMA applications).

## **Convert MACMAT\*.DAT (Create CITZMU.DAT)**

This menu will initiate a conversion routine to convert the original CITZAF mass absorption coefficient ASCII files to the binary format used by CalcZAF.

## **Create New X-ray Database (XRAY.ALL to XRAY.MDB)**

This menu will initiate a conversion routine to convert the binary x-ray data file XRAY.ALL (created by program MakeXray.exe) to the relational database format used by CalcZAF.

## **Create New McMaster MAC Table**

This menu item will use the McMaster mass absorption coefficient fit polynomials to calculate a new mass absorption coefficient table. This table may be selected from the ZAF, Phi-Rho-Z, Alpha Factor and Calibration Curve Selections menu by clicking the MACs button.

## **Create New MAC30 MAC Table**

This menu item will use the MAC30 (Heinrich fit to the Goldstein data) mass absorption coefficient fit polynomials to calculate a new mass absorption coefficient table. This table may be selected from the ZAF, Phi-Rho-Z, Alpha Factor and Calibration Curve Selections menu by clicking the MACs button.

To obtain mass absorption coefficients for elements 93 and above, the USERMAC.DAT file should be created using the MAC30.DAT file as its “basis” and then updated using the USERMAC.TXT file from CalcZAF. The USERMAC.TXT file contains the database values from the Institute of Transuranium Elements. The reference is:

I. R. Farthing and C. T. Walker, Heinrich's Mass Absorption Coefficients for the K, L and M Lines, The European Commission, Institute for Transuranium Elements, Karlsruhe, Report K0290140, 1990

## **Create New MACJTA MAC Table**

This menu item will use the Armstrong (simple Heinrich fit) mass absorption coefficient fit polynomials to calculate a new mass absorption coefficient table. This calculation is from Armstrong's MACCALC program. This table may be selected from the ZAF, Phi-Rho-Z, Alpha Factor and Calibration Curve Selections menu by clicking the MACs button.

## **Create New FFAST MAC Table**

This menu item will calculate a new FFAST table from the CHANTLER 2005 MAC data files provided by Nicholas Ritchie (NIST). You must have the Chantler2005-ka.dat, Chantler2005-kb.dat, etc., data files for this operation (contact Probe Software for more information).

## **Create New User Defined MAC Table**

This menu will allow the user to select a source for creating a user defined MAC table. This table can be edited manually by the user for specified values from the literature or their own measurements.

To obtain mass absorption coefficients for elements 93 and above, the USERMAC.DAT file should be created using the MAC30.DAT file as its “basis” and then updated using the USERMAC.TXT file from CalcZAF. The USERMAC.TXT file contains the database values from the Institute of Transuranium Elements. The reference is:

I. R. Farthing and C. T. Walker, Heinrich's Mass Absorption Coefficients for the K, L and M Lines, The European Commission, Institute for Transuranium Elements, Karlsruhe, Report K0290140, 1990

## **Update Existing User Defined MAC Table**

Using a comma, space or tab delimited input file defined below, this menu allows the user to overwrite individual MAC values in the USERMAC.DAT table.

Each line of the user selected input text file allows the user to define mass absorption coefficients for the Ka, Kb, La, Lb, Ma and Mb lines of an emitting element by an absorber. Any non-zero value from the user selected input file will get

read and used to update the table value in the USERMAC.DAT MAC table. The default name for the user selected input file is USERMAC.TXT but any text file with the appropriate format can be used.

The format of the input file is a text based comma, space or tab delimited file with the first line containing the column labels. For example:

```
zMeas  zAsorb  Ka      Kb      La      Lb      Ma      Mb
```

The subsequent lines of the file contain the emitting element atomic number, the absorbing element atomic number and the mass absorption coefficients of the Ka, Kb, La, Lb, Ma and Mb lines for the emitting element. An example file is shown here:

| zMeas | zAsorb | Ka     | Kb   | La    | Lb    | Ma | Mb |
|-------|--------|--------|------|-------|-------|----|----|
| 4     | 12     | 127000 | 0    | 0     | 0     | 0  | 0  |
| 5     | 12     | 59500  | 0    | 0     | 0     | 0  | 0  |
| 6     | 12     | 23900  | 0    | 0     | 0     | 0  | 0  |
| 7     | 12     | 11000  | 0    | 0     | 0     | 0  | 0  |
| 8     | 12     | 5170   | 0    | 0     | 0     | 0  | 0  |
| 9     | 12     | 2620   | 0    | 0     | 0     | 0  | 0  |
| 10    | 12     | 1405   | 1327 | 0     | 0     | 0  | 0  |
| 11    | 12     | 810    | 749  | 0     | 0     | 0  | 0  |
| 12    | 12     | 488    | 439  | 0     | 0     | 0  | 0  |
| 13    | 12     | 4169   | 3719 | 0     | 0     | 0  | 0  |
| 14    | 12     | 2822   | 2465 | 0     | 0     | 0  | 0  |
| 15    | 12     | 1947   | 1666 | 0     | 0     | 0  | 0  |
| 16    | 12     | 1367   | 1150 | 0     | 0     | 0  | 0  |
| 17    | 12     | 974    | 804  | 0     | 0     | 0  | 0  |
| 18    | 12     | 704    | 572  | 0     | 0     | 0  | 0  |
| 19    | 12     | 515    | 412  | 0     | 0     | 0  | 0  |
| 20    | 12     | 381    | 301  | 13720 | 13345 | 0  | 0  |
| 21    | 12     | 285    | 222  | 9682  | 9430  | 0  | 0  |
| 22    | 12     | 215    | 166  | 7039  | 6824  | 0  | 0  |
| 23    | 12     | 164    | 126  | 5189  | 5004  | 0  | 0  |
| 24    | 12     | 127    | 96   | 3902  | 3749  | 0  | 0  |
| 25    | 12     | 99     | 74   | 2972  | 2844  | 0  | 0  |
| 26    | 12     | 77     | 58   | 2290  | 2184  | 0  | 0  |

In the above example, the file contains emitting x-ray lines from B (4) to Fe (26) as absorbed by Mg (12). Integer or floating point numbers may be used. The program will read and process the input file until the last line is reached.

---

## Analytical

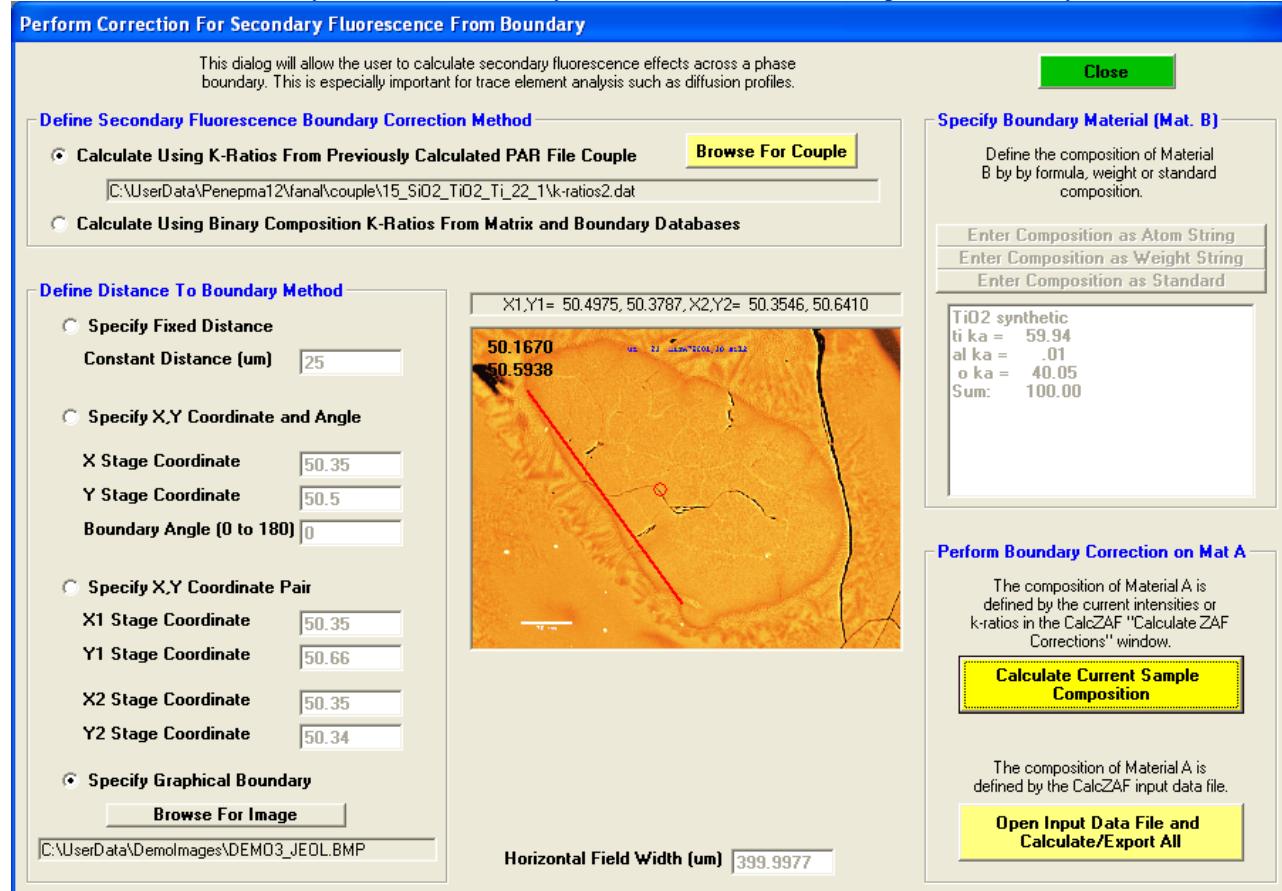
### Display Current Sample Elements

This menu item will make the ZAF Calculations window visible if it has been minimized or closed.

### Calculate Secondary Fluorescence

This menu will allow the user to specify secondary fluorescence effects due to the presence of nearby phases using a method developed by Xavier Llovet and Cesc Salvat. To perform the calculation the secondary fluorescence effects one must know the composition of the phase itself, the composition of the nearby phase and the distance to the nearby phase. The volume of the phase itself is assumed to be zero and at the surface for the calculation purposes.

To facilitate the secondary boundary fluorescence calculation please note that the Output | Save CalcZAF Output Format menu can be used to output Probe for EPMA intensity data for a sample for this boundary correction. The stage coordinates are automatically included to accurately calculate the distance to the specified boundary (see below).



First define the method for the boundary correction. Currently only the PAR file calculation option has been implemented. The MAT and PAR files can be calculated in Standard based on an existing standard composition or specified formula. Be sure to specify the density for each material. Also note that any analyzed sample in Probe for EPMA can be exported as a MAT file from the Analyze! Window Create Material File button.

Note that the beam incident, boundary and standard material must be calculated as PAR files in Standard.exe prior to this procedure. The final intensity extraction using Fanal.exe (from Standard.exe) must be performed using the same beam energy, element and x-ray line as the data was acquired with. The PAR file calculations in Standard.exe take approximately 10 hours each and are saved as a "k-ratios2.dat" file which must be browsed for in this dialog.

Second, define the boundary distance calculation method. One can define a specified fixed distance, a single stage coordinate and an angle (0 = vertical and 90 = horizontal), two stage coordinates or a graphical method based on a stage calibrated BMP file exported from Probe for EPMA.

Next load a sample from the main File menu and process each data point separately or open the input file from the secondary fluorescence window here and process all the data points. The position of each data point will be displayed on the image area.

## Calculate Elemental-To-Oxide Factors

This menu will calculate elemental to oxide conversion factors based on the current cation and oxide ratios (as defined by defaults in the ELEMENTS.DAT file).

## **Calculate Oxide-to-Elemental Factors**

This menu will calculate oxide to elemental conversion factors based on the current cation and oxide ratios (as defined by defaults in the ELEMENTS.DAT file).

## **Students' "t" Table**

Calculate a standard t-test table for confidence intervals from 60% to 99%.

## **Empirical MACs**

For complete details on this menu, see the help section under the heading: Probe Menu Details | Analytical | Empirical MACs.

## **ZAF, Phi-Rho-Z, Alpha Factor and Calibration Curve Selections**

For complete details on this menu, see the help section under the heading: Probe Menu Details | Analytical | ZAF, Phi-Rho-Z, Alpha Factor and Calibration Curve Selections.

## **Operating Conditions**

This menu allows the user to change the default Takeoff angle and keV for modeling different microanalytical conditions for the current composition or intensity data.

## **Particle and Thin Film**

This menu allows the user to quantitatively analyze particles (cubes, prisms, spheres and fibers) and thin films (only for those films or foils less than 1 milligram/centimeter<sup>2</sup> or assuming density = 1 approximately 100 um in thickness or less) based on various adjustable parameters. Films must be a single composition (not multiple layers) unsupported (TEM style) or on a low Z substrate. For more complex thin films use the Output | Save STRATAGem Format menu to create an export file suitable for post-processing with STRATAGem.

Note that the accuracy of the particle method depends on bracketing a single particle in the scanned or defocused beam (overscanning) and applying judgement as to the particle shape and the distance along the beam axis. Normal thick polished standards should be used (bulk specimens) for the intensity calibration. Note that the scan or defocus size must be equal to the particle width dimension.

Particles are normally deposited dry on a low Z substrate that does not contribute to the x-ray spectrum (or continuum) and is usually a double sticky carbon tab. Insulating particles should be carbon coated using a two axis rotation evaporation method or carbon sputtered coated for good conductivity.

### **Particle/Thin Film Model**

The geometric particle and thin film models are:

|  |   |
|--|---|
| Thin Film or Thick Polished Section (unsupported film or no substrate) |   |
| Rectangular Prism (flat top and flat sides or cube)                    | - sides orthogonal to spectrometer axis   |
| Tetragonal Prism (flat top and curved sides)                           | - sides 45 degrees to spectrometer axis   |
| Trigonal Prism (curved top and flat sides or fiber)                    | - long axis perpendicular to spectrometer |
| Square Pyramid (curved top and curved sides or sphere)                 |   |
| Backscatter-Modified Rectangular Prism (modified rectangular prism)    |   |

Note that the last model is the same as the previous "Rectangular Prism" model with a modified electron side-scattering correction.

Note that particle and thin film calculations are limited to the following standard Phi-Rho-Z correction methods:

|                                |                    |
|--------------------------------|--------------------|
| Select Individual Corrections  | (use with caution) |
| Armstrong/Love Scott (default) |                    |
| Packwood Phi(pz) (EPQ-91)      |                    |

Bastin (original) Phi(pz)  
Bastin PROZA Phi(pz) (EPQ-91)

Note also that if using "Select Individual Corrections", only the following absorption corrections are allowed:

Phi(pz) Absorption of Packwood-Brown 1982/XRS Alpha  
Phi(pz) Absorption of Bastin 1984/XRS Alpha  
Phi(pz) Absorption of Armstrong/Packwood-Brown 1981 MAS  
Phi(pz) Absorption of Bastin 1986/Scanning  
Phi(pz) Absorption of Riveros 1987/XRS  
Phi(pz) Absorption of Packwood (New)  
Phi(pz) Absorption of Bastin Proza (EPQ-91)

### **Particle Diameter**

The particle diameter is either set to an arbitrary large number (10,000 microns or 1 cm) for thick specimens or the actual distance the electron beam will travel through the particle or thin film in microns.

### **Particle Density**

The particle density in g/cm<sup>3</sup> is irrelevant for thick specimens, but critical for particles and thin films that are less than the electron range. Iron or calcium bearing silicates average around 3.0 and SiO<sub>2</sub> or high silica glasses are about 2.7.

### **Particle Thickness Factor**

This is the factor that defines the beam aspect ratio relative to the particle diameter. For example, 0.5 means that the particle thickness is half its diameter (a disk). This value should be 1 for thin films or thick polished specimens.

### **Numerical Integration Step**

This value is the step length (in g/cm<sup>2</sup>) for numerical integration. A typical value for 15 keV is 0.00001. For thinner films the step size should be decreased and for higher voltages the step size should be decreased slightly.

## **Use Conductive Coating Correction For Energy Loss**

Toggle the correction for adjusting the electron beam energy for electron energy loss due to the conductive coating. Important when working at low overvoltages. Uses a modified equation from Kerrick et. al., Amer. Min. 58, 920-925 (1973) and Springer (1974).

```
' Intensity correction from electron energy loss due to coating
ratio! = (100# - ((8.3 * sample(1).CoatingDensity! * sample(1).CoatingThickness! / ANGPERNM&) /
(sample(1).KilovoltsArray!(chan%) ^ 2 - (sample(1).LineEdge!(chan%) / EVPERKEV#) ^ 2))) / 100#
```

Note that the coating parameters for standards and unknowns are ignored in the quantitative calculations until the electron absorption and x-ray transmission coating correction flags are explicitly turned on from the Analytical | Analysis Options menu dialog.

## **Use Conductive Coating Correction For Absorption**

Toggle the correction for adjusting the x-ray intensities for x-ray absorption due to the conductive coating. Important when working with low energy emission lines or emission line energies just above the conductive coating absorption edges. Uses a modified equation from Kerrick et. al., Amer. Min. 58, 920-925 (1973) and Springer (1974).

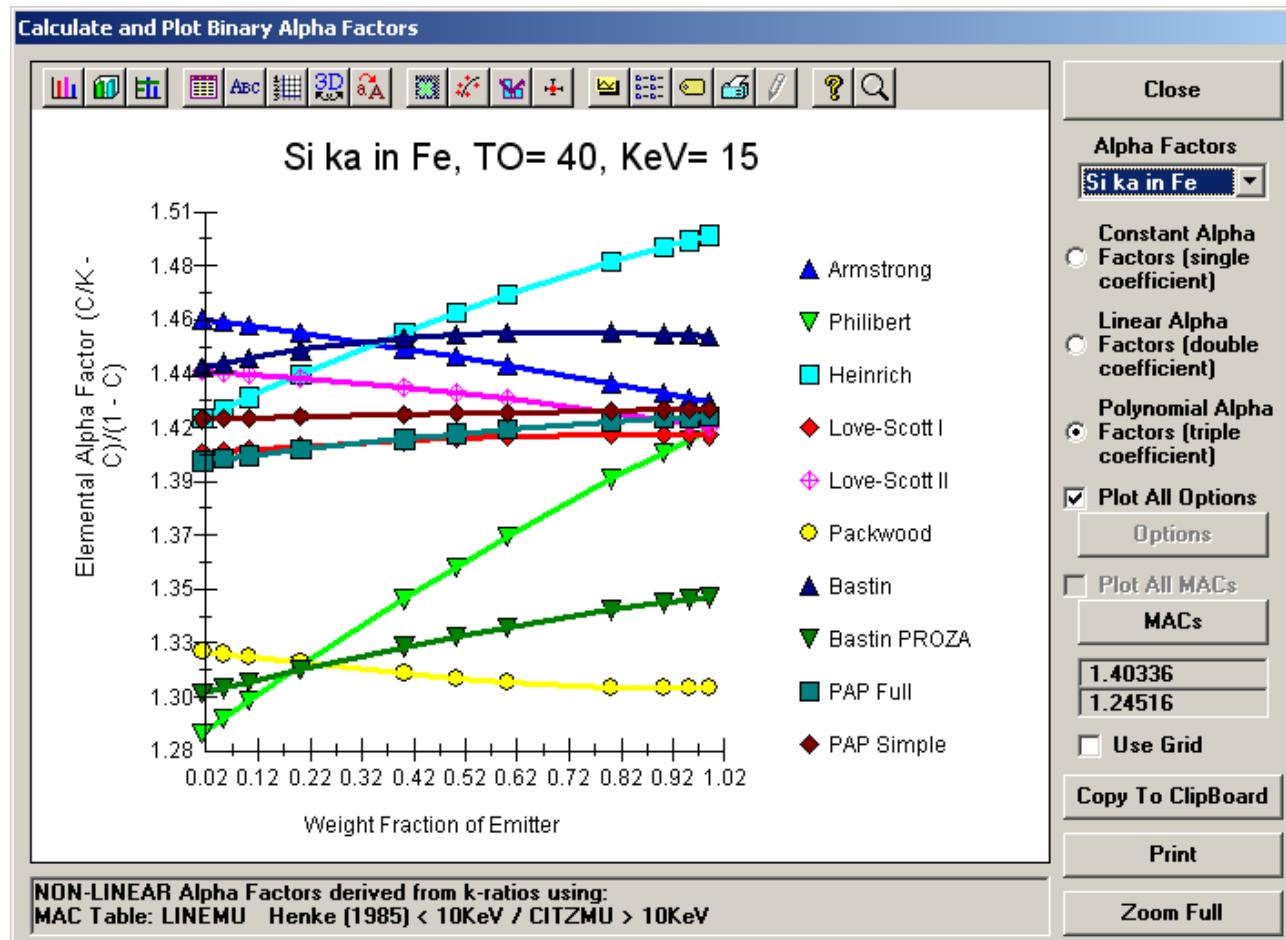
```
' Calculate x-ray transmission based on Sin thickness in angstroms
transmission! = NATURALE# ^ (-1# * atotal! * sample(1).CoatingDensity! * thickness! * CMPPERANGSTROM#)
```

Note that the coating parameters for standards and unknowns are ignored in the quantitative calculations until the electron absorption and x-ray transmission coating correction flags are explicitly turned on from the Analytical | Analysis Options menu dialog.

## Calculate and Plot Binary Alpha factors

This menu will allow the user to specify and plot binary alpha-factor coefficients, based on the current CalcZAF sample. The user may specify constant (single coefficient) alpha factors (original Bence-Albee type), linear (two coefficients) alpha-factors (UC Berkeley style) or polynomial (three coefficient) alpha-factors (Armstrong style).

One can also calculate alpha factor trends for all correction procedures and also all MAC tables as seen below:



## Calculate and Output Binary K-ratios and A-factors for Periodic Table

This menu will calculate tables for k-ratios (11 binary compositions from 1% to 99%) for all elements in the periodic table using the current ZAF and MAC selections. It will also calculate from these k-ratios constant, linear and non-linear (polynomial) elemental alpha factors for the periodic table. Finally it will calculate k-ratios for oxide end-member compositions for calculating oxide end-member alpha-factors externally.

All results can be automatically saved to disk in tab delimited files and may be exported to Excel tables for further manipulation.

## Binary Calculation Options

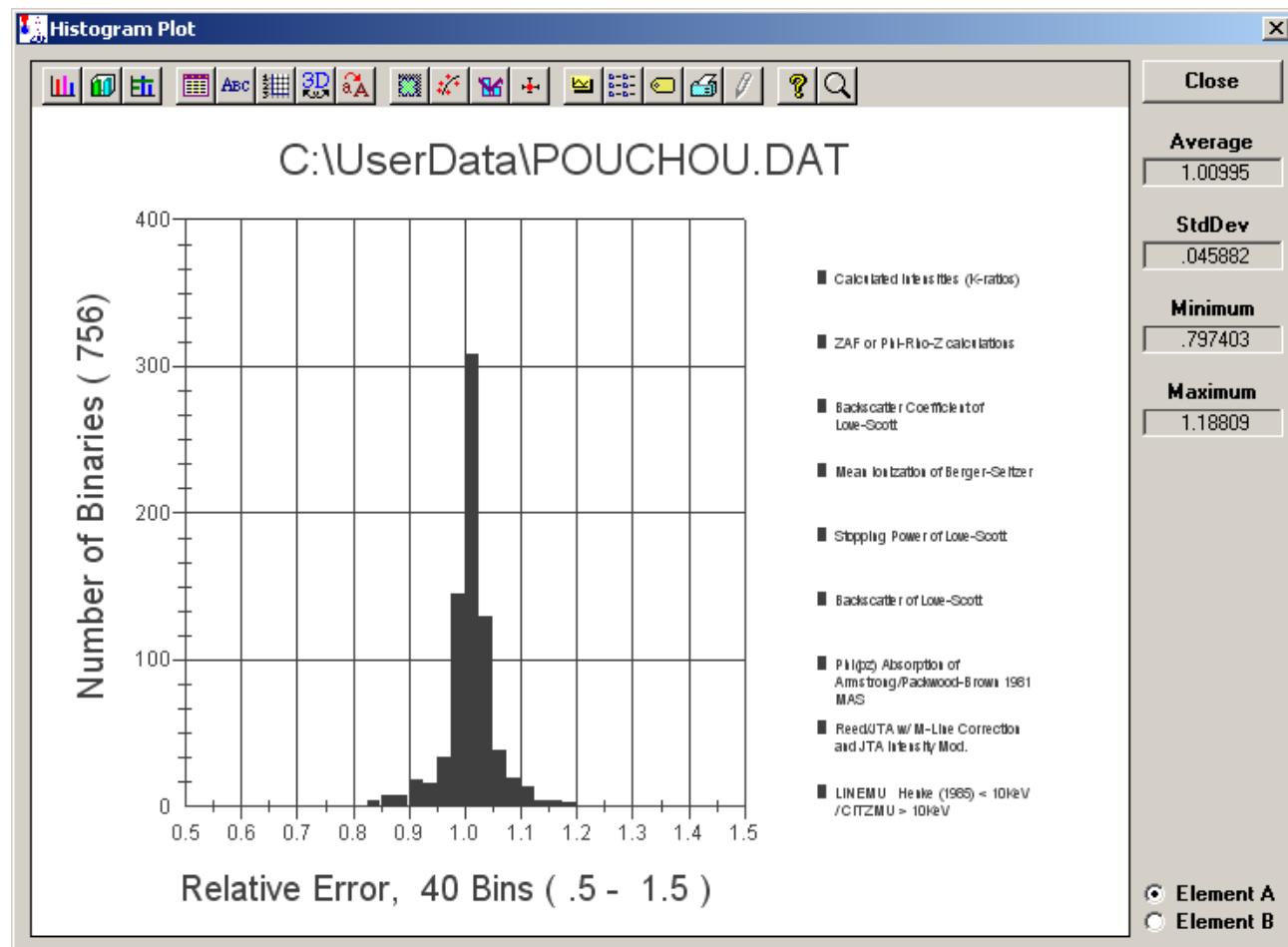
This menu allows the user to specify minimum and maximum absorption, fluorescence and atomic number corrections for the purposes of filtering the output to the data file.

## Binary Histogram Options

This menu allows the user to specify histogram limits (high and low) and the number of histogram bins for plotting error histograms from binary intensity compositional calculations.

## Calculate Binary Intensities (Output Calculated Intensities and Error Histogram)

This menu item will allow the user to select an input file of binary compositions for the calculation of error histograms. The calculations are based on the current ZAF and MAC selections. The output is filtered using the options in the Binary Calculation Options dialog.



Note that all input files for binary calculations have a .DAT extension and must conform to the format specified below. Do not confuse these k-ratio files with normal CalcZAF input files (File | Open menu) or other files with the extension .DAT.

Some sample input files of binary compositions are supplied, e.g.,

|                |   |
|----------------|---|
| AUAGCU2.DAT    | Au-Ag-Cu alloy k-ratios from NIST (measured at 52.5 takeoff)                      |
| NISTBIN.DAT    | NIST k-ratio database (compiled by Heinrich)                                      |
| NISTBINA20.DAT | NIST k-ratio database (compiled by Heinrich), Absorption correction > than 20%    |
| NISTBINZ10.DAT | NIST k-ratio database (compiled by Heinrich), Atomic Number correction > than 10% |
| POUCHOU.DAT    | Pouchou k-ratio database  |
| POUCHOUA20.DAT | Pouchou k-ratio database (compiled by Heinrich), Absorption correction > than 20% |

POUCHOUZ10.DAT Pouchou k-ratio database (compiled by Heinrich), Atomic Number correction > than 10%

An example of the file format is seen here:

|    |    |   |   |     |      |       |       |       |    |
|----|----|---|---|-----|------|-------|-------|-------|----|
| 79 | 29 | 2 | 5 | 15. | 52.5 | .8015 | .1983 | .7400 | .0 |
| 79 | 29 | 2 | 5 | 15. | 52.5 | .6036 | .3964 | .5110 | .0 |
| 79 | 29 | 2 | 5 | 15. | 52.5 | .4010 | .5992 | .3120 | .0 |
| 79 | 29 | 2 | 5 | 15. | 52.5 | .2012 | .7985 | .1450 | .0 |

The data file format assumes one line for each binary. The first two columns are the atomic numbers of the two binary components to be calculated. The second two columns are the x-ray lines to use ( 1 = ka, 2 = kb, 3 = la, 4 = lb, 5 = ma, 6 = mb, 7 = specified, that is, do not calculate the intensity). The next two columns are the operating voltage and take-off angle. The next two columns are the weight fractions of the binary components. The last two columns contains the k-exp values for calculation of k-calc/k-exp. If the second element x-ray line is "specified" (iray = "7"), then no experimental k-ratio value is required.

Note that although the k-ratio file format example above has two emitting elements (so that two k-ratio error sets will be calculated), only one emitting element per line is actually required, as in the following example (where the x-ray line of "7" indicates an element by difference):

|    |    |   |   |    |      |       |    |       |    |
|----|----|---|---|----|------|-------|----|-------|----|
| 13 | 26 | 1 | 7 | 20 | 52.5 | 0.241 | .0 | 0.124 | .0 |
| 13 | 26 | 1 | 7 | 25 | 52.5 | 0.241 | .0 | 0.098 | .0 |
| 13 | 26 | 1 | 7 | 30 | 52.5 | 0.241 | .0 | 0.083 | .0 |
| 26 | 13 | 1 | 7 | 20 | 52.5 | 0.759 | .0 | 0.736 | .0 |
| 26 | 13 | 1 | 7 | 25 | 52.5 | 0.759 | .0 | 0.742 | .0 |
| 26 | 13 | 1 | 7 | 30 | 52.5 | 0.759 | .0 | 0.748 | .0 |

## Calculate Binary Intensities (Output Calculated Intensities for All Matrix Corrections and MAC Files, Single Line, Single File)

This menu item will allow the user to select an input file (with the same format as above) containing a single binary composition for the calculation of k-ratio errors as in the previous menu, but will calculate all combinations of matrix corrections and MAC files (currently 70 combinations). The output will go to a single file and a string containing the ZAF and MAC combination options will be written to the file after each line. If the input file contains more than one line the program will only read the first line.

The average, standard deviation and minimum and maximum is written at the end of the output file for the calculated k-ratio error columns. The output is filtered using the options in the Binary Calculation Options dialog.

## Calculate Binary Intensities (Output Calculated Intensities for All Matrix Corrections and MAC Files, All Lines, Multiple Files)

This menu will allow the user to select an input file (with the same format as above) containing any number of binary compositions for the calculation of k-ratio errors as in the previous menus, and will calculate all combinations of matrix corrections and MAC files (currently 70 combinations), but the output will go to multiple files, one file for each ZAF and MAC combination.

The average, standard deviation and minimum and maximum is written at the end of the output file for the calculated k-ratio error columns. The output is filtered using the options in the Binary Calculation Options dialog.

## Calculate Standard Concentrations (Output Calculated Concentrations and Errors)

Calculate composition of standard from raw k-ratio ACII input data from .DAT file. Data file format assumes one line for each composition. See Output menu in Probe for EPMA to create an export file of all standards in a probe run. See sample file CALCZAF2.DAT for the input format.

This menu calculates standard composition using a special "Standard" single line input format that can be created using Probe for EPMA from previously acquired standard samples. The input file format is as follows:

- The first column is "standard name" in double quotes.
- The second column is the takeoff angle.
- The third column is the kilovolts.
- The fourth column is the oxide or elemental flag (1 = elemental, 2 = oxide).
- The fifth column is the number of elements.
- The next n columns are the element symbols (use double quotes).
- The next n columns are the element x-ray lines (use double quotes) (leave empty for specified concentration, must be last element(s)).
- The next n columns are the measured k-ratios for each element (blank for specified concentration).
- The next n columns are the "published" weight percent for each element.
- The next n columns are the primary standard number assignment for each element.
- The next n columns are the number of cations per element.
- The next n columns are the number of oxygens per element.

The following is an example of the output created using this menu. The first line has oxygen measured and the second line has oxygen specified using the value from the standard database

```
"Fayalite", 40.0, 15.0, 2, 3, "fe", "si", "o", "ka", "ka", "ka", .7372, .2523, 1.0086, 54.809,  
13.785, 31.407, 895, 14, 895, 1, 1, 1, 1, 2, 0  
"Fayalite", 40.0, 15.0, 2, 3, "fe", "si", "o", "ka", "", .7372, .2523, 0.0, 54.809, 13.785,  
31.407, 895, 14, 0, 1, 1, 1, 1, 2, 0
```

The output format is as follows:

|                      |   |
|----------------------|---|
| Line                 | Standard file row number (simple increment)   |
| Name                 | Standard name in double quotes  |
| keV                  | Standard keV  |
| Takeoff              | Standard take off angle" & VbDquote\$ & vbTab   |
| <element> Kraw       | Element raw k-ratio   |
| <element> Krat       | Element elemental (normalized) k-ratio  |
| <element> Publ-Krat  | Element elemental (normalized) k-ratio (from STANDARD.MDB composition)                |
| <element> Wt%        | Element elemental calculated weight percent   |
| <element> Wt%        | Element oxide calculated weight percent   |
| Total                | Calculated total  |
| <element> Publ       | Element elemental weight percent from STANDARD.MDB file                               |
| Publ Total           | Total from STANDARD.MDB file  |
| <element> Conc Error | Element relative error from calculated to published weight percent (1.0 = zero error) |
| <element> Pri F(Chi) | Element generated intensity   |
| <element> Sec F(Chi) | Element emitted intensity   |
| <element> Absorp     | Element absorption correction   |
| <element> Fluor      | Element fluorescence correction   |
| <element> Zed        | Element atomic number correction  |
| <element> Stp        | Element stopping power correction   |
| <element> Bks        | Element backscatter correction  |
| <element> ZAF        | Element ZAF (full) correction   |

## **Calculate Standard Concentrations for All Matrix Corrections and MAC Files, Single Line, Single File)**

Same as above but only reads the first line of the file and then calculates the standard concentrations using all combinations of matrix corrections and mass absorption coefficients and calculates errors. See .TXT file for a histogram data set of the calculation.

## **Calculate Binary Intensities (Output Atomic First Approximation)**

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file [software] section. It is used to calculate atomic fraction first approximations, but for only those binaries that meet the minimum or maximum correction factor output filter criteria are actually written to the file.

## **Calculate Binary Intensities (Output Mass First Approximation)**

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file [software] section. It is used to calculate mass fraction first approximations, but for only those binaries that meet the minimum or maximum correction factor output filter criteria are actually written to the file.

## **Calculate Binary Intensities (Output Corrected Electron First Approximation)**

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file [software] section. It is used to calculate electron fraction first approximations, but for only those binaries that meet the minimum or maximum correction factor output filter criteria are actually written to the file.

## **Calculate First Approximations Only (Atomic Fraction)**

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file [software] section. It is used to calculate atomic fraction first approximations, but without any correction factor output filter options.

## **Calculate First Approximations Only (Mass Fraction)**

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file [software] section. It is used to calculate mass fraction first approximations, but without any correction factor output filter options.

## **Calculate First Approximations Only (Electron Fraction)**

This menu is only visible if the ExtendedMenu flag is set in the PROBEWIN.INI file [software] section. It is used to calculate electron fraction first approximations, but without any correction factor output filter options.

## **Update Edge Line Flur Files**

This menu (never visible to users) updates x-ray files and add records for elements 95 - 100 (nrec 97-102). Called to convert older "Classic" PFW data files to the new Probe for EPMA data files that go up to element 100.

Input data is from:

R. B. Firestone, Table of isotopes, 8th Edition, Vol II: A+151-272, V.S. Shirley, ed. Lawrence Berkeley National Laboratory, John Wiley & Sons, Inc., New York

H. Kleykamp, "Wavelengths of the M X-Rays of Uranium, Plutonium, and Americium", Z. Naturforsch., 36a, 1388-1390 (1981)

H. Kleykamp, "X-ray Emission Wavelengths of Argon, Krypton, Xenon ' and Curium", Z. Naturforsch., 47a, 460-462 (1992).

## **Update Mac Files**

This menu (never visible to users) rewrites the LINEMU.DAT file from the 94 element version to the 100 element version for Xtreme **PROBE**. Only to be used when converting original MAC data files from the older PFW.

## **Convert Text To Data**

The menu (never visible to users) converts the "FLUR\_YIELD.TXT" fluorescence input file to the binary data file "XFLUR3.DAT".

---

## **Run**

This menu will output a variety of run information in summary format to the log window. This information can then be cut and pasted to another application or simply selected and printed using the File | Print Log menu.

## **List Standard Compositions**

This menu item will list the compositions of all standards in the current run based on the current values in the standard composition database.

## **List Current MACs**

This menu item will list the currently loaded MACs (mass absorption coefficients) from the last quantitative analysis. Note that any empirical MACs listed from this menu will be used to overwrite table based MACs automatically during calculations.

## **List Current Alpha Factors**

This menu will list the current alpha factor values and indicate any empirical and/or Penepma generated alpha factors for the current sample.

## **List Analysis Parameters**

This menu item will list the current analysis conditions.

## **Model Detection Limits**

This menu item will allow the user to estimate the 99% confidence (3 sigma) values for the minimum concentration detected based on a specified unknown count time or the count time required to detect a specified concentration. This will allow the user to design the required analytical setup for trace element determinations based on a given set of intensities from the standard on-peak and unknown background measurements.

The expression for minimum detection concentration is:

$$X_U = \frac{3\sqrt{I_U^B \cdot t_U^B \cdot B_U} \cdot C_S}{t_U^B \cdot B_U \cdot I_S^P}$$

The expression for counting time required for the detection of the given concentration is:

$$t_U^B = \frac{9 \cdot I_U^B \cdot (C_S)^2}{(X_U)^2 \cdot (I_S^P)^2 \cdot B_U}$$

where:  $I_U^B$  is the intensity of the background in cps/nA (divide the unknown counts by beam current)

- $t_U^B$  is the count time used for the unknown background or peak measurement (must be equal)  
 $B_U$  is the beam current used for the unknown measurement in nA  
 $C_S$  is the concentration of the element in the standard in weight percent (wt. %)  
 $I_S^P$  is the intensity of the standard in cps/nA (divide the standard intensity by standard beam current)  
 $X_U$  is the unknown concentration in weight percent (wt. %)

Note that all calculations are performed at 3 sigma (standard deviations) and assume that the background and peak measurement count times on the unknown are equal. There is no correction for unknown ZAF correction, but it could easily be applied by simply multiplying the ZAF factor times the resulting concentration or count time.

Remember that weight percent can be converted to ppm (parts per million) using the following expression:

$$ppm = wt\% \cdot 10,000$$

Therefore, 0.010 wt. % is equal to 100 ppm or 0.001 wt. % is equal to 10 ppm.

Note the calculation assumes that the counting time used for the unknown peak and background measurements are equal. This is usually the case for trace elements since at zero concentration both the peak and off-peak measurements have equal statistical weight. Note also that the unknown background time is the sum of both the high off-peak and low off-peak count times so that:

$$t_U^P = t_U^H + t_U^L = t_U^B$$

- where:  $t_U^P$  is the count time for the on-peak measurement  
 $t_U^H$  is the count time for the high off-peak measurement  
 $t_U^L$  is the count time for the low off-peak measurement  
 $t_U^B$  is the count time for the combined high and low off-peak measurements (used in the above calculations)

## Calculate Electron and X-ray Ranges

This menu pulls up a modeless dialog that can do three related range calculations. First given a specified composition (using either atom or weight fraction input), it will calculate the electron range (Kanaya-Okayama) in microns for a given density and incident beam energy, second, it will calculate the x-ray production range in microns based on the above and a specified element and x-ray line, third, it will calculate the x-ray transmission, in unit fraction, for the above conditions given a specified thickness.

The three equations (Goldstein, et. al., 1981) used for these calculation are shown here, where  $A$  is average atomic weight and  $Z$  is average atomic number (calculated using mass fraction averaging) and  $E$  is beam energy and  $E_C$  is critical excitation energy both in keV and  $t$  is thickness in cm and  $\rho$  is density in gm/cm<sup>3</sup>:

$$1. \quad R_{KO} = \frac{0.0276 \cdot A \cdot E^{1.67}}{\rho \cdot Z^{0.89}}$$

2.

$$R_{XR} = \frac{0.0276 \cdot A \cdot (E^{1.67} - E_C^{1.67})}{\rho \cdot Z^{0.89}}$$

3.

$$\frac{I}{I_o} = e^{-u \cdot \rho \cdot t}$$

## Calculate Sample Temperature Rise

This menu will calculate the approximate relative temperature rise in degrees kelvin (or C) for a material of specified thermal conductivity (in W/cmK) at a given beam condition.

The formula for this calculation in beam conditions units of keV, nA and um is as follows:

$$\Delta T = \frac{0.0048 \cdot E_o \cdot i}{C_t d_o}$$

The output is reflected to the log window.

## Output

### Log Window Font

This menu allows the user to modify the current log window font type, size and appearance using the font Common dialog. It is strongly recommended to use a non-proportional font, such a New Courier, so that numeric column data is properly aligned in the log window.

Note that the default font and size of the log window may be permanently defined in the PROBEWIN.INI file.

### Debug Mode

This menu toggles the program in and out of Debug mode. In Debug mode, the program generates an abundance of additional output to the log window for the purposes of debugging the program or viewing the analytical calculations in greater detail.

Debug mode is also useful to check the low level commands sent to the hardware interface. Note that in Debug mode the Form level events are not protected against unanticipated errors. Program CalcZAF normally runs in DebugMode for more analytical detail.

### Verbose Mode

This menu turns on even more detail than the DebugMode menu and should be used in conjunction with the DebugMode menu when the users desires an even more detailed output of the internal calculations.

### ZAF Equation Mode

Display the calculated ZAF correction factors and parameters in ZAF equation mode which is consistent with the values displayed by NIST's DTSA-II program.

### Extended Format

This menu toggles the output option for printout to the log window of element data. If unchecked, the program automatically wraps element data output to 8 elements per line. Note that wavescan data is wrapped 4 elements per line.

If this option is checked, the program does not wrap element data to the log window or disk file (if Save To Disk option is enabled), instead the data is written out as far as necessary to the right.

## **Save To Disk Log**

This menu toggles the output option to mirror all log window output to a user specified text file. This may be useful for importing text or data from the log window to another non-Windows program. The default extension is .OUT but another extension may be entered by the user.

If a large amount of output is to be generated, this option will capture all output to the log window so that it can be printed out afterwards. If the output file selected by the user already exists, the program offers the option to overwrite or append subsequent output to the already existing file.

## **View Disk Log**

This menu invokes the file viewer specified in the PROBEWIN.INI file (default file viewer is NotePad) to view the previously or currently opened disk log file.

If the file is currently open, the program will first close the disk log file before viewing it. Note that because the default file viewer (NotePad) is somewhat limited, it may be necessary to specify a more robust file viewer such as TextPad. See the section on editing the PROBEWIN.INI file above to change the default file viewer.

## **Open Link To Excel**

This menu will create an OLE (Object Linking and Embedding) link to an Excel application (if it is available). This link is available to both the StartWin and ProbeWin applications for sending both intensity and quantitative data to an Excel spreadsheet. This feature requires Excel 8 (Office 97) or higher.

From CalcZAF, the user can send calculated data automatically to an Excel spreadsheet along with the column labels, by clicking the "Excel >>" button.

## **Close Link To Excel**

This menu will close an open OLE (Object Linking and Embedding) link to an Excel application (if it is available).

---

## **Help**

### **About CalcZAF**

This menu displays the copyright notice, acknowledgments and contact information for users of CalcZAF NT/2000/XP/Vista.

### **Help On CalcZAF**

This menu opens this help file for hypertext help file viewing at the main table of contents. Note that both a searchable index and glossary are available for browsing by the user at any time during a run. Each help topic may be printed.

In addition, context sensitive help (that is, help that is applicable to the current program context) is available for each window in all applications by simply pressing the F1 key. To get help on a menu item simply highlight with the mouse and hit the F1 key.

## **Update CalcZAF**

This menu will connect the user to the CalcZAF update web page to download the latest version of CalcZAF. To update CalcZAF simply download the update zip file and extract the files to the CalcZAF application folder.

To update the Probe for EPMA application suite you should perform the automatic update procedure from the Probe for EPMA main application Help menu.

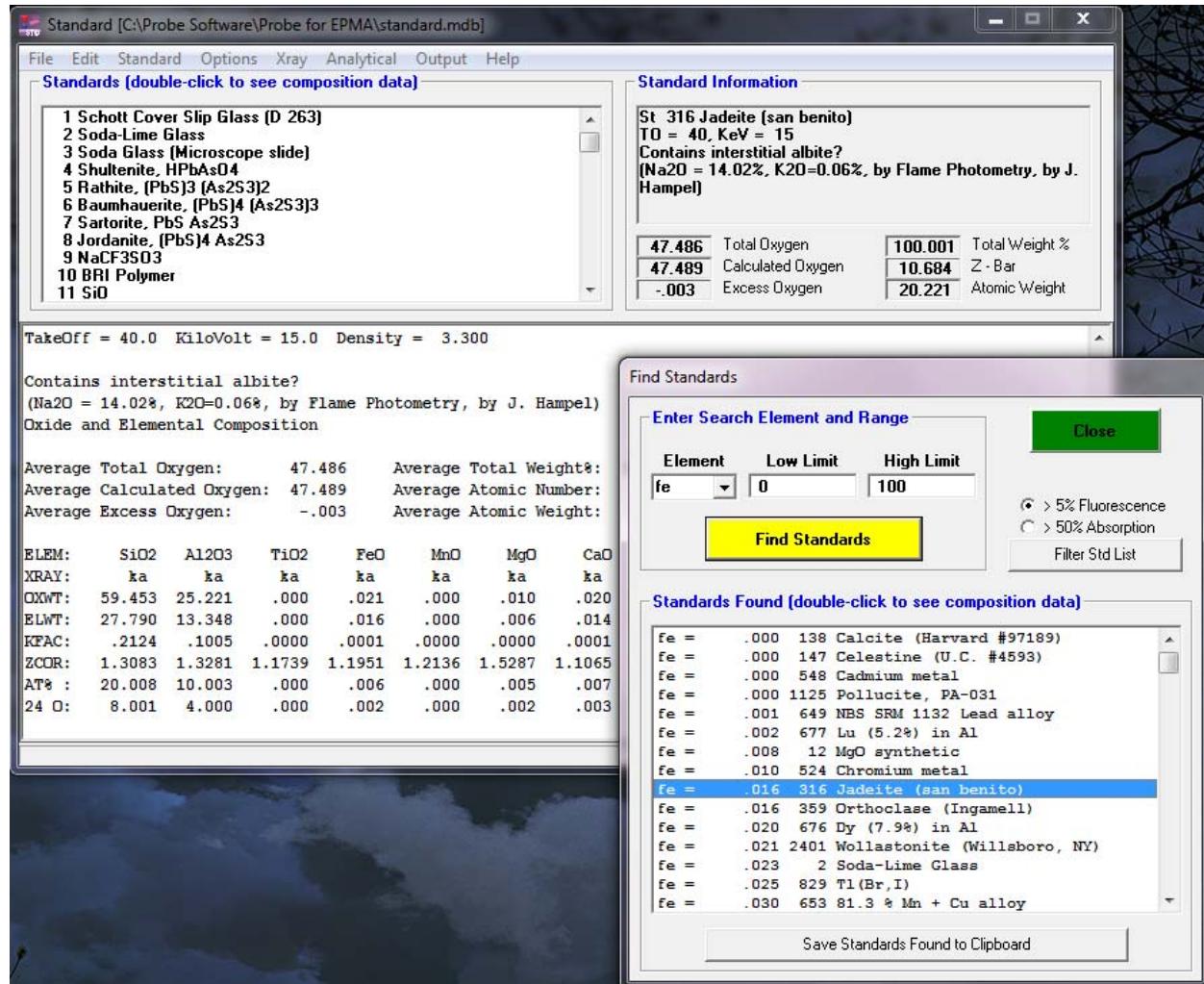
## **Probe Software On The Web**

Connect your browser to the Probe Software web site.

# Standard

## Overview

Program STANDARD.EXE maintains the standard composition database used by Probe for EPMA for quantitative analysis. A menu item is provided in the Probe for EPMA application to run Standard asynchronously.



The standard composition database is in Microsoft Access v. 3.5 format and can also be accessed using Microsoft Office Professional (although no direct modification of the tables using Access should normally be attempted). To import

standard compositional data from older versions of Probe for EPMA, first export the standard database to an ASCII file and then import the data using the Import menu item.

Standard stores all standard compositions as elemental concentrations. However, the user may elect to enter and display the composition in either oxide or elemental concentrations. In addition, the user may enter standard compositions based on a simple formula if creating a new standard composition.

Oxide standards are standards whose compositions are defined by cation concentrations and their corresponding cation and oxygen valence subscripts. Elemental standards are defined by elemental concentrations only. Whether a standard is entered and/or displayed as an oxide or elemental standard is determined by the value of the Enter Composition In and/or Display Composition As options in the File | Modify dialog. However, both oxide and elemental standards may be used to analyze either oxide or elemental calculated unknown samples in Probe for EPMA in any combination.

---

## Log Window

The main window of Standard is a scrollable text window which will record all output and calculations. This log window is a fully cut and paste enabled text window which can be used to transfer data or analysis results to any other Windows application such as a word processor or spreadsheet.

Simply click and drag the mouse to select text and use the <ctrl> c, <ctrl> x or <ctrl> v key combinations to copy, cut or paste text to or from the Windows Clipboard. Or use the Edit menu for these and other Clipboard functions.

The size of the log window buffer can be specified in the PROBEWIN.INI file and is limited only by the amount of memory available. If the Save Log to Disk File option is selected from the Output menu, then all log window output is mirrored to a text file, including any text manually entered by the user using the keyboard. The log window font and font size is specified in the PROBEWIN.INI file and may be changed during a run from the Output menu. To view a copy of the log file during a run, select the View Disk Log option from the Output menu.

All standards in the standard database are seen in the scrollable multi-select list box. Multi-select means that the list box supports the selection of more than one item. To select a range of items click the first item in the list and then holding the <shift> key, select the last item to be selected. To select individual items, click while holding the <ctrl> key down.

To see the compositional data for a particular standard, simply double click the standard in the list box. The standard composition and k-factors are output to the log window. Note that the k-factors calculated in Standard are based on the default operating voltage, take-off angle, x-ray lines, mass absorption coefficients and ZAF options for reference purposes only. Probe for EPMA calculates actual standard k-factors based on the actual analytical conditions of the specific sample. An example of this output is given here :

```
St 160 Set 0 NBS K-412 mineral glass
TakeOff = 40 KiloVolts = 15
Oxide and Elemental Composition

Elemental Wt. % Total : 99.260      Average Total Oxygen : 42.780
Average Calcu. Oxygen : 42.782      Average Excess Oxygen : -.002
Average Atomic Weight : 22.048      Average Atomic Number : 12.733

ELEM:    SiO2      FeO      MgO      CaO      Al2O3      MnO      O
XRAY:    ka        ka        ka        ka        ka        ka        ka
OXWT:   45.352   9.960   19.331   15.250   9.270    .099    -.002
ELWT:   21.199   7.742   11.657   10.899   4.906    .077   42.780
KFAC:   .1621    .0654    .0777    .1008    .0334    .0006   .1696
ZCOR:   1.3080   1.1836   1.5000   1.0817   1.4672   1.2043  2.5223
BETA:   .1632    .0631    .0732    .1023    .0312    .0004   .1634
ATWT:   16.766   3.079   10.653   6.040    4.039    .031   59.391
```

The keywords are defined here :

ELEM: Oxide formula or elemental symbol  
XRAY: Defined x-ray lines for k-factor calculation  
OXWT: Oxide formula weight percent (using defined oxides)  
ELWT: Elemental weight percent  
KFAC: Normalized elemental k-factor (using default Phi-Rho-Z)  
ZCOR: ZAF correction factor (using default Phi-Rho-Z)  
BETA: Beta correction factor (using default alpha-factors)  
ATWT: Normalized atomic weight percent of element

---

## Running Standard for the First Time

When Probe (or Standard) is run for the first time a demonstration standard database is automatically configured, for use with the demonstration probe data files JEOLEL.MDB and JEOLOX.MDB.

To create a site specific standard database, this demonstration standard database can be overwritten by clicking the File | New menus in the STANDARD program main window to create an empty standard database (click File | Close first, if necessary to close an already open database). Note that the demonstration probe data files JEOLEL.MDB and JEOLOX.MDB will be unable to run properly once this step is performed and should therefore these demo files should be deleted afterwards.

After the new standard database has been created, the user can either begin to type in their new standard compositions by hand or import them from ASCII import files. The format of the standard import ASCII file can be seen in the supplied example files DHZ.DAT, ORE.DAT or SRM.DAT.

# Standard Menu Details

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## File

The File menu can be used to create a new Probe for EPMA standard composition database, open an existing database, close the current database, import or export standard compositions from or to ASCII files, display database file information, print the log window or exit the program.

### New

The New menu opens a dialog to select a new Probe for EPMA standard database file. This option should only be used when creating a site specific standard composition database for the first time in order to overwrite the supplied demonstration database. The default standard database name must be STANDARD.MDB and this filename should be used in order that Probe for EPMA can access this file for quantitative calculations. Once a site specific standard database has been created it will be automatically loaded each time the Standard program is run.

If the file already exists, the program will prompt the user whether to overwrite the existing file. After the new file has been created the program will display a file information dialog to allow the user to personalize the standard database title and description for the specific site.

The program will automatically append a .MDB extension to the file name if one is not supplied by the user. The .MDB file name indicates Microsoft Database and is a Microsoft Access v. 3.5 database file.

The user can also use this menu to create a new searchable standard database before importing the DHZ.DAT, SRM.DAT or ORE.DAT ASCII files. It is suggested to name the files DHZ.MDB, SRM.MDB and ORE.MDB respectively.

### Open

The Open menu allows the user to select an existing Probe for EPMA standard database file to add or modify standard composition data. The program will check that the specified file is of the correct format and warn the user if not. Probe for EPMA generally uses a file extension of .MDB for its standard database file.

The default standard database is STANDARD.MDB and this file is automatically accessed by Probe for EPMA for quantitative calculations. Although the user may create as many additional standard composition databases as desired, Probe for EPMA uses only the STANDARD.MDB file in the **C: \ProgramData\Probe Software\Probe for EPMA** directory for analysis of unknown samples.

### Save As

The Save As menu allows the user to save the currently open Probe for EPMA standard database file to another file name. The program will automatically append a .MDB extension if necessary.

## Close

The Close menu allows the user to close the currently open Probe for EPMA standard database file. This menu can be used in order to open another Probe for EPMA standard database file. The program will ask the user to confirm the close operation and all compositions are automatically saved.

## Import ASCII File

The Import menu allows the user to import an ASCII file of standard compositions. This ASCII import file is usually named STANDARD.DAT and is primarily intended for users who wish to import ASCII standard composition data files into Probe for EPMA. Please contact Probe Software if your standard composition database is in another format and would like it imported into a Probe for EPMA standard database.

Several example import files are supplied :

|                           |  |
|---------------------------|--|
| DHZ.DAT silicate analyses | (Deer, Howie and Zussman)                    |
| ORE.DAT sulfide analyses  | (Dana's Mineralogy)                          |
| SRM.DAT SRM analyses      | (NIST standard reference alloys and glasses) |

The DHZ.DAT is a database of all the analyses in the student edition of "Rock Forming Minerals" by Deer, Howie and Zussman (1st Edition). The DHZ database also includes the Harvard oxygen mineral standards (entered as standards #2761-2773) which are available from Carl Francis at the Harvard Mineralogical Museum. The ORE.DAT file is sulfide minerals from Dana's Mineralogy entered in ideal formulas. The SRM.DAT is a database of SRM (standard reference materials) alloys and glasses from the NIST SRM catalog.

The import process can be repeated as often as desired to import additional standards into the Probe for EPMA standard database. Note that the program will abort the import process if a standard number already exists in the database.

Since almost any source can generate a suitable standard composition import file, a sample of a standard composition import file is shown here with a short explanation for each field:

```
2003          "Hortonolite, p. 4"
"""
-1 9
"si"    "ti"    "al"    "fe"    "mn"    "mg"    "ca"    "h"    "o"
1       1        2       1       1       1       1       2       1
2       2        3       1       1       1       1       1       0
15.912   .258     .482    32.402    .527    12.254    .579    .010   36.688
2004          "Fayalite, Pantelleria, p. 4"
"""
-1 8
"si"    "ti"    "al"    "fe"    "mn"    "mg"    "ca"    "o"
1       1        2       1       1       1       1       1
2       2        3       1       1       1       1       0
14.285   .432     .048    47.339    2.656    2.093    .808    32.650
```

Two standard compositions are shown in the example above. The fields may be comma, tab or space delimited.

The first line contains the standard number (must be unique and range from 1 - 32768), and the standard name in double quotes (generally site specific standard numbers should use values under 2000 to avoid conflicts with the supplied NIST SRM, DHZ and Dana ORE sample databases). The second line contains the standard description field in double quotes. It may be empty. The third line contains the DisplayAsOxide (0 = do not display as oxide composition, non-zero = display as oxide composition) flag and the number of elements in the standard composition. The fourth line contains the element symbols in double quotes for each element in the standard composition. The fifth and sixth lines contain the number of cations followed by the number of oxygens for each element (required even for elements not displayed as oxides). The seventh line contains the composition in elemental weight percent for each element in the standard composition.

Note that all elements must be specified, even oxygen for silicate standards (even if the DisplayAsOxide flag is true).

## **Export ASCII File**

The Export menu allows the user to export an ASCII file of standard compositions. This may be useful for transferring compositional standard data to another application or simply to have a text file backup of the standard compositions. This text format is suitable for importing back into Standard after manually editing the entries if desired.

## **Import ASCII File (single row format)**

The Import ASCII File (single row format) command is usually used to import standards into a new standard database from an Excel spreadsheet that has been saved as a tab delimited text file with a .DAT extension. Each standard must be on a single row with a separate column (H to Fm) for each element weight percent, number of cations and number of oxygens (for oxide formulas).

See the format of a test file produced by the Export ASCII File (single row format) menu.

## **Export ASCII File (single row format)**

The Export ASCII File (single row format) command is usually used to export all standards from the standard database to a format compatible for importing into an Excel spreadsheet. Each standard is exported to a single row with a separate column (H to Fm) for each element weight percent, number of cations and number of oxygens (for oxide formulas).

*Note: if you try to open this export file in Excel, you will have to use Excel 12 or higher. Previous versions of Excel only support 256 columns.*

## **Create AMCSD Database (American Mineralogist Database)**

This menu will create a standard database suitable for compositional matching using the American Mineralogist Crystal Structure Database compositions which contains over 9500 compositions based on formula stoichiometries.

## **Import Standards From Cameca PeakSight (Sx.mdb)**

To import standard compositions from the Cameca PeakSight software select the Import From Cameca PeakSight menu to first select the default standard database to create (usually STANDARD.MDB) and then select the Cameca PeakSight sx.mdb file to import. The program will automatically process the standard composition data into the default STANDARD.MDB database.

The standard numbers used will be the PhaseID numbers from the Cameca database. If different standard numbers are preferred, one can export the database to ASCII and edit the standard numbers and then re-import it, however the standard numbers in the position files will also need to be edited in that case.

## **Import Standards From JEOL Text File**

This menu allows the user to import standard compositions from a JEOL 8900/8200/8500 UNIX computer that have been properly formatted using a custom Perl script. See Paul Carpenter for more information.

## **File Information**

This menu displays and allows the user to change the run title and/or description fields for the currently open standard database.

## **Print Log**

This menu allows the user to print entire contents of the log window or the selected contents of the log window.

## **Print Setup**

This menu allows the user to change the default printer or printer setup options for the currently selected printer.

## **Exit**

This menu exits the Standard program. Note that Standard automatically saves any changes to the standard database automatically as they are made using transaction processing methods so there is no chance of data loss.

---

## **Edit**

### **Cut**

This menu cuts the selected text from the log window to the Windows Clipboard.

### **Copy**

This menu copies the selected text from the log window to the Windows Clipboard.

### **Paste**

This menu pastes text from the Windows Clipboard to the log window.

### **Select All**

This menu selects all text in the log window.

### **Clear All**

This menu clears all text from the log window.

---

## **Standard**

### **New**

To create a new standard composition simply click the Standard | New menu. From the Standard Composition window enter each element and its concentration by clicking on a blank element grid row. To change a previously entered element concentration or cation ratio for oxide entered or displayed standards, simply click an already existing element row.

The program will automatically specify a default standard number based on the next free standard number starting at 1.

**If it may be desirable to group the standards numbers using some sort of a numbering system. In this case it will be necessary to change the suggested standard number. Generally some thought should be given to arranging the various standards together into functional groups. For example, it might be helpful to reserve standard numbers 1 - 106 for the elemental metal standards based on atomic number. Or perhaps to reserve standard numbers 200 - 500 for silicate standards only.**

Note that if an already existing standard number is entered, the program will prompt whether the existing standard should be overwritten. If not, select another standard number. Note also that standard numbers between 1 and 2000 should normally be used to avoid conflicts or confusion with the standard numbers in the DHZ.DAT, SRM.DAT and ORE.DAT files.

Also enter the standard name and optionally a description or comment field to indicate the standard source or other pedigree information.

If the standard is a simple compound, it may be easier to enter the composition as a formula string. Click the Enter Atom Formula Composition to enter the formula string, for example fayalite entered as fe<sub>2</sub>sio<sub>4</sub>. To enter a formula string note that the parser is able to handle multiple occurrences of an element and even the use of parentheses. For example: "ca<sub>2</sub>mg<sub>5</sub>si<sub>8</sub>o<sub>22</sub>(oh)<sub>2</sub>"

All standard compositions are saved to the standard database as elemental concentrations, therefore, be sure to enter the oxygen concentration *if* oxygen is present in the compound. This applies to *all* standards, even those which are entered and/or displayed as oxide concentrations. To facilitate the data entry for the oxygen concentration of standard compositions which are entered as oxide concentrations, the program will display the calculated oxygen of the cations entered thus far. Therefore, if oxygen is entered as the last element in the standard, it is a simple matter to specify the displayed oxygen concentration from the calculated oxygen of the cations.

Be sure to also enter excess oxygen (for example minerals that contain ferric iron), into the standard composition since the presence of the excess oxygen will affect the overall calculation of the standard k-factors and mean atomic number.

For example, to enter hematite (Fe<sub>2</sub>O<sub>3</sub>) as a composition, one must enter two atoms of Fe and three atoms of O. Using elemental weight percent entry, enter 69.94 wt. % Fe and 30.06 wt. % O. Using the default oxide entry, of Fe expressed as FeO and elemental O, that would be two molecules of FeO and one molecule of O, or 89.98 wt. % FeO and 10.02 wt. % O (the excess oxygen). However, if the cations for Fe are changed to two atoms of Fe and three atoms of oxygen, then one can input the composition as oxide simply by entering 100% Fe<sub>2</sub>O<sub>3</sub>.

Note that "oxide" displayed standards that contain fluorine and/or chlorine will sum greater than 100 %, since these halogens actually replace some of the oxygen that is already being included in the oxide formulas of the other cations. The oxygen equivalent that is being included in the composition is usually small and can generally be ignored. However, for the most accurate analysis it is recommended that the standard containing fluorine and/or chlorine be entered as an elemental standard composition. That way, the oxygen can either be calculated by stoichiometry or measured directly along with the halogens. The only missing component then will be hydrogen present in water or hydroxyl which is usually less than 1 % of the total.

## **Formula Entry**

The Formula Entry compositional string must be entered as a string of characters for element symbols and numbers for subscripts. For example :

Fe<sub>2</sub>SiO<sub>4</sub> :

Enter as : "fe2sio4"

CaCO<sub>3</sub>

Enter as "caco3"

Note that subscripts of 1 need not be specified. If an element is present in the formula string more than once, the program will automatically sum the subscripts. Do not use fractional subscripts.

*Note that because some formulas are "ambiguous" when parsed one character at a time, use both upper and lower case characters to eliminate confusing the formula parser. For example: "yba2cu3o7" will be interpreted as yttrium, boron and an undefined element "a". Instead, enter the string as "YBa2Cu3O7" and the formula will be parsed correctly.*

## **Modify**

This menu item allows the user to modify an existing standard name or composition without re-entering all the element symbols and concentrations. The user must enter the compositional changes in elemental or oxide weight percent when modifying an existing standard composition. Simply click on the element row in the grid to pull up the Enter Composition dialog. Enter the indicated elemental or oxide weight percent and click OK to save the changes.

This window may also be used to temporarily change the default x-ray line and cations for a specific standard for calculation purposes. This is often useful to explore the effect on the k-ratios of various operating voltages or selection of alternative x-ray lines.

## **Duplicate**

This menu allows the user to easily add replicate standards (with unique standard numbers) that may be variations on the same standard or composition.

## **Delete**

Allows the user to delete an existing standard from the standards database file. Note that this is a permanent deletion from the standards database. The program will ask you to confirm the deletion. If it becomes necessary to use the standard again for quantitative analysis, it will have to be re-entered using the New menu item above.

**Be careful about deleting any standards from the database. Remember, if a standard has been deleted that has been used in a quantitative run and later one uses that run as a setup or simply wants to re-process the data again, the program will be unable to load that standard composition or even worse, might load the wrong composition if the standard was deleted and the same number was used for another standard composition.**

## **Delete Selected**

Allows the user to delete selected standards from the database. Each standard will require a confirmation before it can be deleted.

**Be careful about deleting any standards from the database. Remember, if a standard has been deleted that has been used in a quantitative run and one later uses that run as a setup or simply wants to re-process the data again, the program will be unable to load that standard composition or even worse, might load the wrong composition if the standard was deleted and the same number was used for another standard composition.**

## **List All Standard Names**

This menu item will type out all standard numbers and names to the log window. This list can then be printed out or cut or copied and pasted to another document for reference purposes.

## **List Elemental Standard Names**

This menu will output the names of all standards that are not flagged using the “Display As Oxide” flag.

## **List Oxide Standard Names**

This menu will output the names of all standards that are flagged using the “Display As Oxide” flag. Oxide standards will have the formula oxides displayed and the formula based on 24 oxygens if oxygen is present.

## **List Selected Standards**

This menu item will type out the standard compositions and k-factors for the selected standards to the log window. To select a standard just click a standard in the standard list, to select additional standards, simply hold the <ctrl> or <shift> keys and click additional standards. The <ctrl> key selects or deselects individual standards, the <shift> key selects all standards in a range.

If the list of standards is very long, it may be necessary to select the Save to Disk Log menu so that the output is automatically saved to disk. This file can then be opened using the View Disk Log menu and printed out if desired.

### **List All Standards**

This menu item will type out the standard compositions and k-factors for all standards in the standard database to the log window.

If the list of standards is very long, it may be necessary to select the Save to Disk Log menu so that the output is automatically saved to disk. This file can then be opened using the View Disk Log menu and printed out if desired.

### **List Elemental Standards**

This menu will type out the full composition of all elemental standards in the database. That is, all standards that are not flagged using the “Display As Oxide” flag.

### **List Oxide Standards**

This menu will type out the full composition of all oxide standards in the database. That is, all standards that are flagged using the “Display As Oxide” flag.

---

## **Options**

### **Search**

Use this option to have the program automatically search through the standard list as it finds letters that match your search criteria.

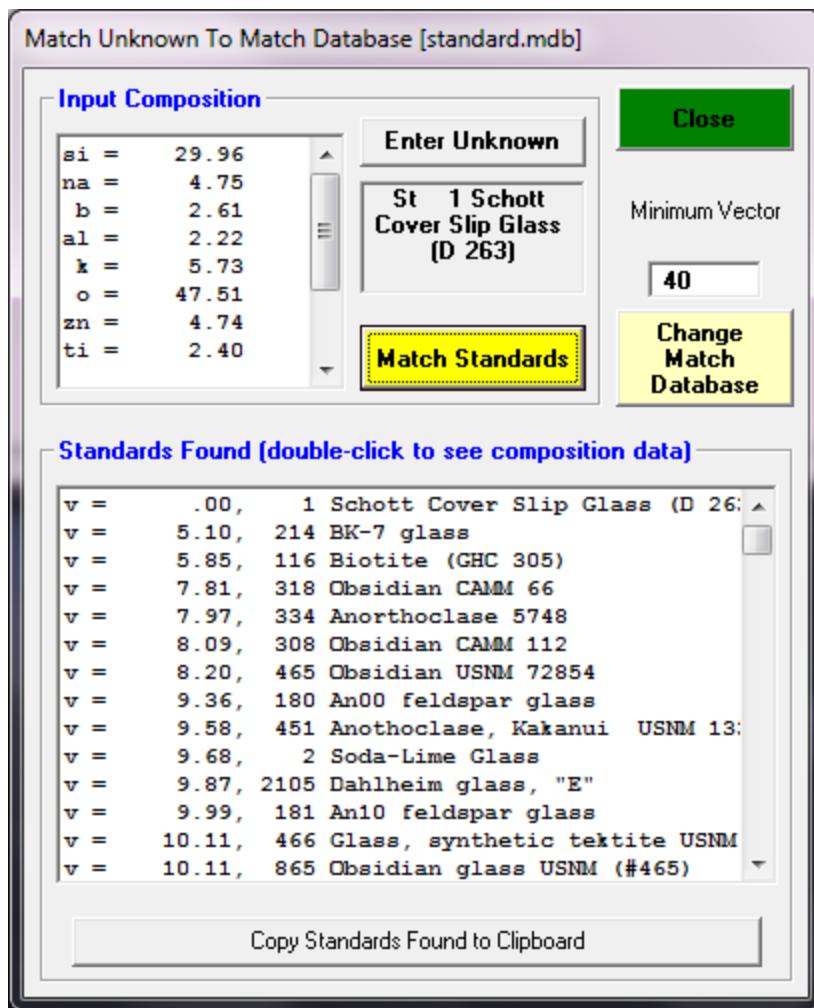
### **Find**

Use this option to obtain a list of all standards that contain a specified element within a specified concentration range. To see the composition of the standards, simply double click the standard in the returned list and the composition will be output to the log window. This command is useful for determining, for instance, what standards contain between 10 and 20 weight percent Fe.

To copy the list of standards found to the Window clipboard, click the Copy to Clipboard button.

### **Match**

This dialog is used to match a given standard or unknown composition to the standards in the standard database. Can be used to select the closest standard to an unknown sample for quantitative analysis or to suggest possible mineral identification.



The dialog will display the last selected standard as a default composition to match to. To match to an arbitrary unknown composition, click the Enter Unknown button and enter a compositional string in weight percent.

The routine automatically re-calculates all analyses in mole or atomic percents for the vector fit using a sum of the square of the differences algorithm. The following is an example of the output from the "match" command after entering a composition of 70.51% of FeO and 29.49% of SiO<sub>2</sub> (fayalite) using the DHZ database :

```
Unknown composition:
    fe      si
    70.51   29.49   .00   .00   .00   .00   .00   .00
Sum = 100.00
STD = 5 Fayalite, East Greenland, p. 4           MATCH = 6.718
STD = 4 Fayalite, Pantelleria, p. 4              MATCH = 12.81
STD = 68 Grunerite, U.S.A., p. 151             MATCH = 30.25
STD = 50 Orthoferrosilite, p. 105            MATCH = 34.24
STD = 183 Titaniferous magnetite, p. 427        MATCH = 38.97
```

Once the list of sorted standards has been loaded (decreasing match vector) each standard may be viewed by simply double clicking it in the list. The output will be automatically directed to the log window.

To copy the list of standards matched to the Window clipboard, click the Copy to Clipboard button.

It may be most useful to open one of the supplied databases for matching an unknown composition depending on the circumstances. See the section on the File | Import menu.

### **Weight Percent Entry**

The weight percent entry compositional string must be entered as a string of element and weight percent pairs. For example :

Albite :

ELEM: SiO<sub>2</sub> Al<sub>2</sub>O<sub>3</sub> Na<sub>2</sub>O O  
ELWT: 32.132 10.289 8.769 48.810

Enter as : "si32al10na9o48"

YAG :

ELEM: Y<sub>2</sub>O<sub>3</sub> Al<sub>2</sub>O<sub>3</sub> O  
ELWT: 44.939 22.721 32.339

Enter as : "y45al23o32"

Lanthanum Hexaboride :

ELEM: La B  
ELWT: 68.170 31.830

Enter as : "la68b31"

Note that numbers less than 1 percent must be entered as 1 since the string parser does not support fractional weight percents using a decimal point.

Note that because some formulas are "ambiguous" when parsed one character at a time, use both upper and lower case characters to eliminate confusing the formula parser. For example: "yba2cu3o7" will be interpreted as yttrium, boron and an undefined element "a". Instead, enter the string as "YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub>" and the formula will be parsed correctly.

### **Modal Analysis**

This menu item is used to perform a quantitative modal analysis of unknown compositions, based on a group of modal phases that are defined by selecting standard compositions from a standard database. Any database of standard compositions may be used to define the phases, since the modal groups thus defined are stored within each standard database and may be recalled for use with subsequent data sets.

Typically this procedure is performed on a large set of compositional data acquired using multiple traverses or gridded locations that are statistically representative of the material in question. The input file must contain the oxide or element symbols for each column of data enclosed in double quotes, in the first line of the data file. The file must not contain a totals column.

The number of matches and abundance of each phase in the group will be tabulated and the average and standard deviation of each phase calculated. A sample output is shown here for a silicate modal analysis :

```
Modal Group 1 silicate
Minimum Total = 95
```

```
Phase 1 plag
Minimum Vector = 4
End Member = Feldspar
```

301 albite (cazadero)  
 306 anorthite (Grass Valley) U.C.#16706

Phase 2 alka

Minimum Vector = 4  
 End Member = Feldspar  
 301 albite (cazadero)  
 374 orthoclase MAD-10

Phase 3 oliv

Minimum Vector = 4  
 End Member = Olivine  
 263 Fe<sub>2</sub>SiO<sub>4</sub> (synthetic fayalite)  
 273 Mg<sub>2</sub>SiO<sub>4</sub> (magnesium olivine) synthetic

Phase 4 opyx

Minimum Vector = 4  
 End Member = Pyroxene  
 350 enstatite 25-25  
 469 hypersthene, johnstown USNM #746

Phase 5 cpx

Minimum Vector = 4  
 End Member = Pyroxene  
 358 diopside (Chesterman)  
 453 augite, kakanui USNM 122142

| Line | Vector | Phase | End    | -      | Member        | Sum      | SiO <sub>2</sub> | FeO   | CaO   | Al <sub>2</sub> O <sub>3</sub> | MgO   | Na <sub>2</sub> O |
|------|--------|-------|--------|--------|---------------|----------|------------------|-------|-------|--------------------------------|-------|-------------------|
| 1    | .30    | opyx  | Wo     | 8.     | En 65. Fs 27. | . 98.70  | 52.80            | 17.70 | 4.06  | .44                            | 23.51 | .19               |
| 2    | .93    | opyx  | Wo     | 3.     | En 47. Fs 50. | . 98.48  | 50.08            | 29.90 | 1.44  | 1.23                           | 15.78 | .05               |
| 3    | 2.14   | opyx  | Wo     | 2.     | En 82. Fs 16. | . 98.94  | 50.55            | 12.18 | 1.22  | .62                            | 34.37 | .00               |
| 4    | .24    | oliv  | Fo     | 84.    | Fa 16.        | . 99.61  | 37.18            | 15.45 | .09   | .33                            | 46.56 | .00               |
| 5    | .98    | opyx  | Wo     | 1.     | En 87. Fs 13. | . 99.20  | 54.66            | 9.28  | .29   | .09                            | 34.88 | .00               |
| 6    | -----  | ----- | .      | .      | .             | . 82.11  | 40.66            | 7.28  | .21   | .08                            | 33.88 | .00               |
| 7    | .05    | oliv  | Fo     | 84.    | Fa 16.        | . 100.08 | 38.55            | 15.79 | .06   | .55                            | 45.13 | .00               |
| 8    | .00    | oliv  | Fo     | 85.    | Fa 15.        | . 101.46 | 40.37            | 14.91 | .08   | .00                            | 46.10 | .00               |
| 9    | .05    | oliv  | Fo     | 83.    | Fa 17.        | . 100.00 | 38.62            | 16.39 | .06   | .00                            | 44.93 | .00               |
| 10   | .06    | oliv  | Fo     | 83.    | Fa 17.        | . 100.16 | 38.47            | 16.23 | .03   | .29                            | 45.14 | .00               |
| 11   | .02    | oliv  | Fo     | 96.    | Fa 4.         | . 99.99  | 41.07            | 4.30  | .00   | .56                            | 54.06 | .00               |
| 12   | .68    | opyx  | Wo     | 1.     | En 87. Fs 12. | . 99.08  | 55.33            | 8.44  | .70   | .16                            | 34.45 | .00               |
| 13   | .08    | oliv  | Fo     | 83.    | Fa 17.        | . 97.86  | 37.46            | 15.97 | .01   | .03                            | 44.39 | .00               |
| 14   | .07    | oliv  | Fo     | 84.    | Fa 16.        | . 100.50 | 38.51            | 15.58 | .32   | .40                            | 45.69 | .00               |
| 15   | .01    | oliv  | Fo     | 84.    | Fa 16.        | . 101.09 | 39.76            | 15.53 | .05   | .00                            | 45.75 | .00               |
| 16   | .12    | oliv  | Fo     | 84.    | Fa 16.        | . 98.92  | 37.73            | 15.36 | .02   | .03                            | 45.78 | .00               |
| 17   | .00    | oliv  | Fo     | 84.    | Fa 16.        | . 101.15 | 40.03            | 15.59 | .01   | .00                            | 45.52 | .00               |
| 18   | 5.64   | ----- | .      | .      | .             | . 97.77  | 48.90            | 29.60 | 7.96  | 3.86                           | 6.87  | .58               |
| 19   | 17.73  | ----- | .      | .      | .             | . 98.53  | 51.92            | 31.90 | .00   | 1.85                           | .00   | 12.86             |
| 20   | .59    | cpx   | Wo     | 42.    | En 48. Fs 10. | . 98.64  | 52.92            | 6.20  | 19.97 | 2.80                           | 16.40 | .35               |
| 21   | -----  | ----- | .      | .      | .             | . 69.50  | 38.92            | 6.20  | 11.97 | 1.80                           | 10.40 | .21               |
| 22   | .17    | oliv  | Fo     | 83.    | Fa 17.        | . 100.64 | 37.94            | 16.66 | .04   | .02                            | 45.98 | .00               |
| 23   | .09    | oliv  | Fo     | 83.    | Fa 17.        | . 97.01  | 36.81            | 16.07 | .26   | .28                            | 43.59 | .00               |
| 24   | .05    | oliv  | Fo     | 84.    | Fa 16.        | . 100.08 | 38.55            | 15.79 | .06   | .55                            | 45.13 | .00               |
| 25   | .00    | oliv  | Fo     | 85.    | Fa 15.        | . 101.46 | 40.37            | 14.91 | .08   | .00                            | 46.10 | .00               |
| 26   | .02    | alka  | Ab100. | An     | Or            | . 98.65  | 67.84            | .05   | .00   | 19.65                          | .04   | 11.07             |
| 27   | .00    | plag  | Ab 85. | An 15. | Or            | . 100.33 | 64.10            | .17   | 3.26  | 22.66                          | .25   | 9.89              |
| 28   | .05    | oliv  | Fo     | 83.    | Fa 17.        | . 100.00 | 38.62            | 16.39 | .06   | .00                            | 44.93 | .00               |
| 29   | .06    | oliv  | Fo     | 83.    | Fa 17.        | . 100.16 | 38.47            | 16.23 | .03   | .29                            | 45.14 | .00               |
| 30   | .08    | oliv  | Fo     | 83.    | Fa 17.        | . 97.22  | 37.16            | 16.25 | .05   | .02                            | 43.74 | .00               |
| 31   | .00    | plag  | Ab 91. | An 9.  | Or            | . 100.44 | 66.12            | .00   | 2.02  | 21.66                          | .00   | 10.64             |
| 32   | .07    | oliv  | Fo     | 83.    | Fa 17.        | . 100.39 | 38.57            | 16.57 | .02   | .00                            | 45.23 | .00               |
| 33   | .97    | oliv  | Fo     | 83.    | Fa 17.        | . 99.96  | 42.15            | 14.88 | 1.17  | 1.82                           | 39.94 | .00               |
| 34   | .08    | oliv  | Fo     | 82.    | Fa 18.        | . 100.27 | 38.28            | 17.31 | .05   | .00                            | 44.63 | .00               |
| 35   | .01    | oliv  | Fo     | 83.    | Fa 17.        | . 97.14  | 38.14            | 15.69 | .23   | .25                            | 42.83 | .00               |
| 36   | 1.90   | oliv  | Fo     | 80.    | Fa 20.        | . 98.26  | 43.94            | 15.86 | 1.00  | 1.01                           | 36.45 | .00               |
| 37   | .07    | oliv  | Fo     | 82.    | Fa 18.        | . 99.87  | 38.17            | 17.35 | .05   | .00                            | 44.30 | .00               |
| 38   | .66    | opyx  | Wo     | 9.     | En 76. Fs 15. | . 96.47  | 50.60            | 9.91  | 4.57  | 4.07                           | 27.32 | .00               |
| 39   | .77    | opyx  | Wo     | 1.     | En 83. Fs 16. | . 98.26  | 53.16            | 11.34 | .42   | .59                            | 32.75 | .00               |

Results of Modal Analysis

InputFile : C:\SOURCE\PROBEWIN\modal.dat  
 OutputFile : C:\SOURCE\PROBEWIN\modal.out  
 Date and Time: 5/22/97 9:29:44 PM

Group Name : silicate  
 Total Number of Points in File : 39  
 Valid Number of Points in File : 37  
 Match Number of Points in File : 35

Minimum Total for Valid Points : 95.00  
 Percentage of Valid Points : 94.9

Percentage of Match Points : 89.7

| Phase    | #Match | %Total | %Valid | %Match | AvgVec |      |                  |       |       |                                |       |                   |       |
|----------|--------|--------|--------|--------|--------|------|------------------|-------|-------|--------------------------------|-------|-------------------|-------|
| plag     | 2      | 5.1    | 5.4    | 5.7    | .00    | Sum  | SiO <sub>2</sub> | FeO   | CaO   | Al <sub>2</sub> O <sub>3</sub> | MgO   | Na <sub>2</sub> O |       |
| Average: | Ab     | 88.    | An     | 12.    | Or     | .    | 100.38           | 65.11 | .09   | 2.64                           | 22.16 | .13               | 10.27 |
| Std Dev: | .      | 4.     | 4.     | .      | .      | .    | 1.43             | .12   | .88   | .71                            | .18   | .53               |       |
| Phase    | #Match | %Total | %Valid | %Match | AvgVec |      |                  |       |       |                                |       |                   |       |
| alka     | 1      | 2.6    | 2.7    | 2.9    | .02    | Sum  | SiO <sub>2</sub> | FeO   | CaO   | Al <sub>2</sub> O <sub>3</sub> | MgO   | Na <sub>2</sub> O |       |
| Average: | Ab     | 100.   | An     | .      | Or     | .    | 98.65            | 67.84 | .05   | .00                            | 19.65 | .04               | 11.07 |
| Std Dev: | .      | .      | .      | .      | .      | .    | 0.00             | .00   | .00   | .00                            | .00   | .00               |       |
| Phase    | #Match | %Total | %Valid | %Match | AvgVec |      |                  |       |       |                                |       |                   |       |
| oliv     | 24     | 61.5   | 64.9   | 68.6   | .18    | Sum  | SiO <sub>2</sub> | FeO   | CaO   | Al <sub>2</sub> O <sub>3</sub> | MgO   | Na <sub>2</sub> O |       |
| Average: | Fo     | 84.    | Fa     | 16.    | .      | .    | 99.72            | 38.96 | 15.46 | .16                            | .27   | 44.88             | .00   |
| Std Dev: | 3.     | 3.     | .      | .      | .      | 1.31 | 1.67             | 2.47  | .30   | .42                            | 2.93  | .00               |       |
| Phase    | #Match | %Total | %Valid | %Match | AvgVec |      |                  |       |       |                                |       |                   |       |
| opyx     | 7      | 17.9   | 18.9   | 20.0   | .92    | Sum  | SiO <sub>2</sub> | FeO   | CaO   | Al <sub>2</sub> O <sub>3</sub> | MgO   | Na <sub>2</sub> O |       |
| Average: | Wo     | 4.     | En     | 75.    | Fs     | 21.  | .                | 98.45 | 52.45 | 14.11                          | 1.81  | 1.03              | 29.01 |
| Std Dev: | 4.     | 15.    | .      | 14.    | .      | .    | 0.93             | 2.10  | 7.60  | 1.76                           | 1.39  | 7.24              | .03   |
| Phase    | #Match | %Total | %Valid | %Match | AvgVec |      |                  |       |       |                                |       |                   |       |
| cpxx     | 1      | 2.6    | 2.7    | 2.9    | .59    | Sum  | SiO <sub>2</sub> | FeO   | CaO   | Al <sub>2</sub> O <sub>3</sub> | MgO   | Na <sub>2</sub> O |       |
| Average: | Wo     | 42.    | En     | 48.    | Fs     | 10.  | .                | 98.64 | 52.92 | 6.20                           | 19.97 | 2.80              | 16.40 |
| Std Dev: | .      | .      | .      | .      | .      | .    | 0.00             | .00   | .00   | .00                            | .00   | .00               |       |

### Creating an Input File

The easiest way to create an input file for the modal analysis procedure is to use the Plot! window in Probe for EPMA to output a .DAT file of the oxide or elemental weight percent compositions to disk. Be sure to select output of column labels so that the modal analysis routine can identify the elements in the input file.

One can also use an input file from any other source, just so long as the oxide or element symbols are the first line of the file and the data is in weight percent. The weight percent data can be in any format since the file is read as unformatted ASCII. However, the element symbols in the first line must be enclosed within double quotes.

A sample input file is shown below :

|                     |       |       |                                   |       |                     |
|---------------------|-------|-------|-----------------------------------|-------|---------------------|
| "SiO <sub>2</sub> " | "FeO" | "CaO" | "Al <sub>2</sub> O <sub>3</sub> " | "MgO" | "Na <sub>2</sub> O" |
| 52.8                | 17.7  | 4.06  | .44                               | 23.51 | .19                 |
| 50.08               | 29.9  | 1.44  | 1.23                              | 15.78 | .05                 |
| 50.55               | 12.18 | 1.22  | .62                               | 34.37 | 0.0                 |
| 37.18               | 15.45 | .09   | .33                               | 46.56 | 0.0                 |
| 54.66               | 9.28  | .29   | .09                               | 34.88 | 0.0                 |
| 40.66               | 7.28  | .21   | .08                               | 33.88 | 0.0                 |
| 38.55               | 15.79 | .06   | .55                               | 45.13 | 0.0                 |
| 40.37               | 14.91 | .08   | .00                               | 46.10 | 0.0                 |
| 38.62               | 16.39 | .06   | .00                               | 44.93 | 0.0                 |
| 38.47               | 16.23 | .03   | .29                               | 45.14 | 0.0                 |
| 41.07               | 4.3   | 0.0   | .56                               | 54.06 | 0.0                 |
| 55.33               | 8.44  | .70   | .16                               | 34.45 | 0.0                 |
| 37.46               | 15.97 | .01   | .03                               | 44.39 | 0.0                 |
| 38.51               | 15.58 | .32   | .40                               | 45.69 | 0.0                 |
| 39.76               | 15.53 | .05   | .00                               | 45.75 | 0.0                 |
| 37.73               | 15.36 | .02   | .03                               | 45.78 | 0.0                 |
| 40.03               | 15.59 | .01   | .00                               | 45.52 | 0.0                 |
| 48.90               | 29.6  | 7.96  | 3.86                              | 6.87  | .58                 |
| 51.92               | 31.9  | 0.0   | 1.85                              | 0.0   | 12.86               |
| 52.92               | 6.2   | 19.97 | 2.8                               | 16.4  | .35                 |
| 38.92               | 6.2   | 11.97 | 1.8                               | 10.4  | .21                 |
| 37.94               | 16.66 | .04   | .02                               | 45.98 | 0.0                 |
| 36.81               | 16.07 | .26   | .28                               | 43.59 | 0.0                 |
| 38.55               | 15.79 | .06   | .55                               | 45.13 | 0.0                 |

### **Modal Analysis Options**

**Minimum Total-** Unknown compositions that sum below this value will not be used in the point count analysis. A default minimum sum of 90% or 95% is suggested. Increase the minimum total value to reject points that sum lower.

**Normalize Concentrations For Fit-** This option is used to specify if the standard and unknown concentrations should be normalized to 100% before the vector fit is calculated. Of course, this applies only to those concentrations that have already met the Minimum Total tolerance.

**Weight Concentrations For Fit-** This option is used to specify if the element concentrations for the standards that define each phase should be weighted based on the average composition of the element in that phase. If the major elements in a phase should have a greater influence in determining the vector fit, then this option should be selected. If all concentrations, regardless of magnitude should have equal weight in the vector fit, this option should not be selected.

**Minimum Vector-** The tolerance for the match to a defined phase. A default value of 4.0 is suggested. If a closer match is desired for one or more phases in the group, decrease the vector value for that phase. The vector is calculated by simply taking the square root of the sums of the differences squared to each n-dimensional phase. Note that all weight percents, for both standards and unknowns, are normalized to 100% prior to the vector fit. The calculation is shown below for a one dimensional phase :

$$V = \sqrt{\sum_{i=1}^n (X_i - X'_i)^2}$$

Where  $X_i$  and  $X'_i$  are the components (elements) to be compared

**Mineral end-member-** A mineral end-member calculation for one of the following phases, Feldspar, Olivine, Pyroxene and Garnet may also be specified.

### **Defining Modal Phases**

When defining the phase compositions to match, try to avoid over-determining the phase. For example, in defining an olivine phase you should only use two standards (preferably forsterite and fayalite) since the range of composition is linear. For example, if you enter more than two compositions in the above linear compositional system (for instance an Fo50 composition standard) you could over define the numerical system because the solution to the unknown composition is no longer unique. Note that the routine will always give a correct result, however the calculation will become more unstable as it is over-defined.

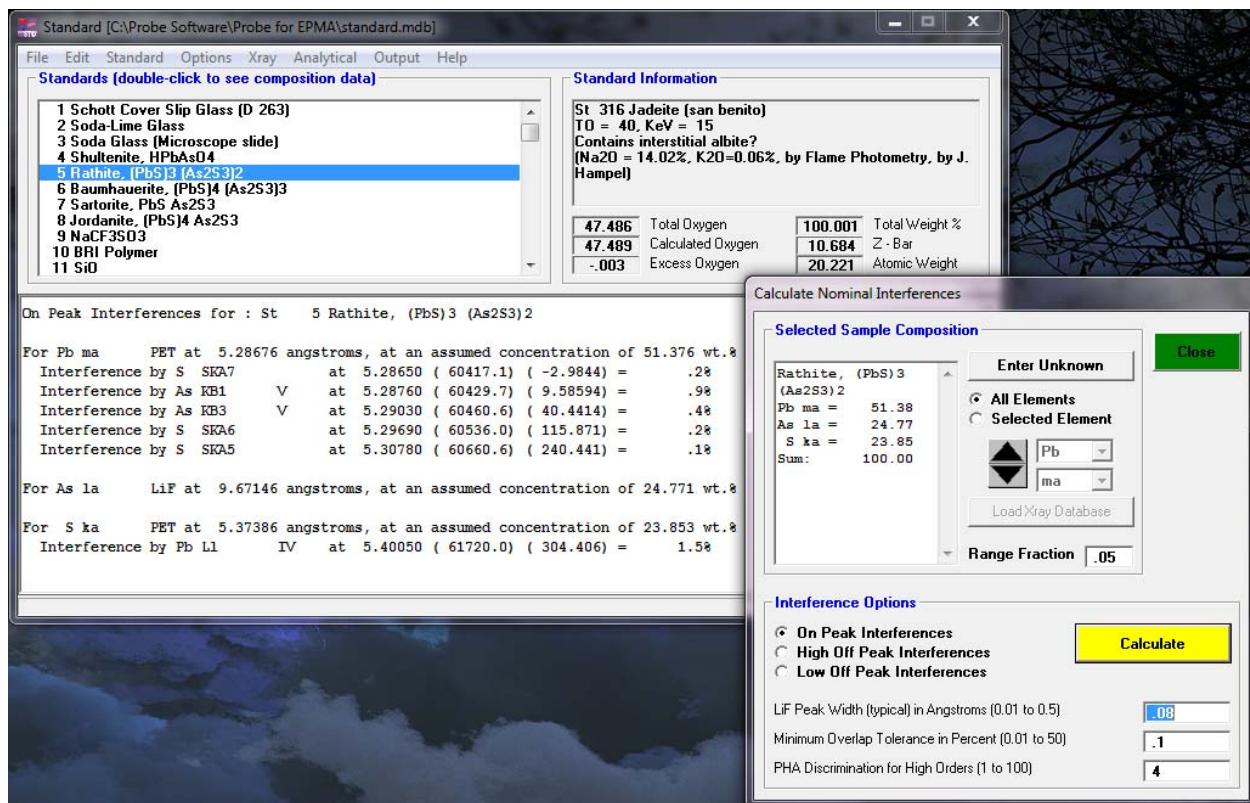
The entire printed output may be captured by selecting the Output | Save Log To Disk menu although an output file of the results for each input line is automatically saved. The output file will contain the vector fit, matched phase, end-member calculation (if selected), sum and composition of each line in the input file.

The printed summary lists the total number of analyses, the minimum total for a valid composition, number of valid points that sum above the minimum sum, the number of matched points and the percentage of points that were matched.

In addition, for each phase, the output will contain the phase name, the number of matches for that phase, the percent of matched points, valid points and total matched points for the matches in that phase. This is followed by the average end-member (if selected), weight percent sum and composition for that phase and the standard deviation for each element.

### **Interferences**

This command will accept a composition (of either a standard number in the standard database or an unknown composition entered by the user) and calculate the approximate percent spectral interference overlap on all interfered lines based on the concentrations, the x-ray line intensities of all known lines, a user defined Gaussian peak width, and a PHA discrimination factor for higher order lines.



The percent interference intensity is based on the actual concentrations of both the interfered and interfering elements. For example, if the concentration of the interfered element is stated as 5.3 wt. % and the percent interference is calculated to be 10%, then the apparent concentration that might be measured is 10% greater than the actual concentration, or 5.3 plus 0.53 (that is, 10% of 5.3) which equals 5.83 wt. %. Note that since this is only a nominal calculation, it should only be used as an indicator of possible problematic analytical situations.

The interference calculation uses the x-ray line and crystal defaults as defined in the ELEMENTS.DAT file (usually found in C:\ProgramData\Probe Software\Probe for EPMA). A sample interference output for a biotite standard is shown here :

```
*****
STD 112 biotite #3
MZbar= 10.688 Zbar= 13.413

LMNT: SiO2 Al2O3 FeO MgO CaO Na2O K2O TiO2
OXWT: 38.620 10.720 18.130 14.010 .020 .690 9.210 2.290
ELWT: 18.052 5.674 14.093 8.448 .014 .512 7.646 1.373
K-FAC .1435 .0407 .1206 .0571 .0001 .0027 .0696 .0120
Z-COR 1.2583 1.3931 1.1688 1.4808 1.0749 1.9290 1.0987 1.1443
MPCT: 34.560 5.653 13.568 18.690 .019 .599 5.257 1.541
APCT: 11.520 2.261 6.784 9.345 .010 .399 3.505 .514

LMNT: MnO BaO Rb2O Cl F H2O
OXWT: .950 .110 .030 .020 4.020 2.660
ELWT: .736 .099 .027 .020 4.020 .298
K-FAC .0062 .0007 .0002 .0002 .0130 .0000
Z-COR 1.1881 1.3255 1.3704 1.1777 3.0936 .0000
MPCT: .720 .039 .009 .030 11.378 7.939
APCT: .360 .019 .006 .030 11.378 5.293

TOTAL WEIGHT PERCENT : 101.480 TOTAL OXYGEN FROM CATIONS : 40.469
AVERAGE ATOMIC WEIGHT : 21.844 AVERAGE MOLECULAR WEIGHT : 54.564
```

```

LiF peak width =      .080 Angstroms
Overlap tolerance =   .100 Percent
PHA discrimination factor = 4.000

For Si  ka at    7.1262      38.62 wt.% :
For Al  ka at    8.3401      10.72 wt.% :
For Fe  ka at    1.9373      18.13 wt.% :
For Mg  ka at    9.8900      14.01 wt.% :
For Ca  ka at    3.3595      .02 wt.% :
Interference by K SKB^4   I   at    3.4040  is     1.2 % overlap.
Interference by K SKB'''  I   at    3.4116  is     .1 % overlap.

For Na  ka at    11.9101     .69 wt.% :
For K   ka at     3.7424      9.21 wt.% :
For Ti  ka at    2.7497      2.29 wt.% :
Interference by Ba LA1   I   at    2.7767  is     .4 % overlap.

For Mn  ka at    2.1031      .95 wt.% :
For Ba  la at    2.7759      .11 wt.% :
Interference by Ti SKA3   I   at    2.7324  is     .1 % overlap.
Interference by Ti SKA3'' I   at    2.7367  is     .3 % overlap.
Interference by Ti SKA'   I   at    2.7399  is     .5 % overlap.
Interference by Ti SKA''  I   at    2.7464  is     1.8 % overlap.
Interference by Ti KA1   I   at    2.7490  is     270.0 % overlap.
Interference by Ti KA1,2  I   at    2.7502  is     486.0 % overlap.
Interference by Ti KA2   I   at    2.7527  is     230.7 % overlap.
Interference by Rb KA1   III at    2.7771  is     .4 % overlap.
Interference by Rb KA1,2 III at    2.7813  is     .6 % overlap.
Interference by Rb KA2   III at    2.7897  is     .1 % overlap.

For Rb  la at    7.3183      .03 wt.% :
For Cl  ka at    4.7288      .02 wt.% :
Interference by Ba LB2   II  at    4.8092  is     .2 % overlap.

For F   ka at    18.3200     4.02 wt.% :
Interference by Fe LA2   I   at    17.5860  is     1.0 % overlap.
Interference by Fe LA1   I   at    17.5860  is     8.5 % overlap.
Interference by Mg SKB^7  II  at    18.4091  is     .1 % overlap.
Interference by Mn LB1   I   at    19.1034  is     .2 % overlap.

```

The default Gaussian peak width is just a nominal value, increasing the width will produce greater overlaps. The PHA discrimination factor can adjust the contribution from higher order lines. The default of 4.0 will divide the intensity of each subsequent order by 4, before calculating the percent overlap. This means that each higher order line will be only 1/4 or 25 % of the intensity of the previous order. To decrease the contribution of higher order lines, increase the PHA discrimination factor.

This calculation is for use only as a guideline and is not intended for use as a quantitative spectral interference correction. Probe for EPMA contains special algorithms to empirically determine the actual spectral interferences based on measured overlaps.

The dialog will use the composition of the currently selected standard as a default composition. See the section on Weight Percent Entry under the Options | Match menu for instructions on entering an arbitrary unknown composition for calculation of nominal interferences.

# X-Ray

## X-Ray Database

This menu allows the user to view specified ranges of the NIST x-ray wavelength database. This database covers the wavelength range from approximately 0.5 to 200.0 angstroms and includes higher order reflections as well. Higher order reflections are reduced in intensity by 25% for each subsequent order to simulate the effect of PHA analysis.

The screenshot shows the 'Xray Database' window with the title 'NIST Xray Lines (multi-select)'. The main table lists x-ray lines with columns for Xray Line, Angstroms, Energy, Intensity, and Reference. Several entries are highlighted in blue, including 'Pb SMA^3', 'Bi LI', 'Hf SLB2^2', 'Fe KB3', 'Fe KB1', 'Pb SMA^1', 'Hf SLB2^1', 'Fe SKB', 'Er Ln', 'Pt SLAS', 'Y LG3', 'Pb MA1', 'S SKA7', 'Am M1-N2', 'As KB1', 'Pu M2-N4', 'As KB3', 'Ho LG4', 'S SKA6', 'Pt LA2', and 'Pb MA2'. A 'Close' button is at the top right of the table area. To the right of the table is a sidebar with 'Highlight Element' and 'Periodic Table' buttons, and a 'Specify Range' section with dropdowns for Start Angstroms, Stop Angstroms, and Bragg Order. Below the table is a 'Select KLM Elements' dialog box containing a periodic table grid where several elements like As, Se, Br, Kr, Pb, Bi, Po, At, and Rn are highlighted in red. A note at the bottom left of the table area states: 'Note that angstrom values in the above table are not corrected for refraction index corrections. However, KLM markers displayed in the Graph Data wavescan plot dialog are corrected using the values in the table.' A 'Copy to Clipboard' button is located at the bottom left of the table area.

The x-ray list range (Start Angstroms and Stop Angstroms) and minimum search intensity (Minimum Intensity) of the x-ray list can be specified by the user. Note that since the list is limited to about 1000 items, the program will automatically increase the minimum intensity until the returned list of x-ray lines is less than 1000. To view x-ray lines of lesser intensity, simply reduce the x-ray range and click the Load New Range button.

Note that angstrom values in the x-ray database list with Bragg reflection orders higher than one (Roman Numeral "I") are NOT corrected for refraction index corrections. However, KLM markers displayed in the Graph Data wavescan plot

dialog are corrected using the equation  $A' = A * (1 - (K - (K / N^2)))$  where A is the uncorrected angstrom position, K is the refractive index from the CRYSTALS.DAT file and N is the Bragg reflection order.

### **Description of the X-ray Database (adapted from NIST documentation by C. Fiori)**

The NIST x-ray database is based on 4985 (1st order) entries and includes all the measurable x-ray lines, satellites and absorption edges from under 100 eV to over 120 keV. Additionally, most of the x-ray lines and satellites are assigned a relative intensity (relative to the alpha-1 line in each family). The data base was assembled primarily from four sources:

- 1.) B.L. Doyle, W.F. Chambers, T.M. Christensen, J.M. Hall and G.H. Pepper "SINE THETA SETTINGS FOR X-RAY SPECTROMETERS", Atomic Data and Nuclear Data Tables Vol. 24, No 5, 1979.
- 2.) E.W. White, G.V. Gibbs, G.G. Johnson Jr. and G.R. Zechman "X-RAY WAVELENGTHS AND CRYSTAL INTERCHANGE SETTINGS FOR WAVELENGTH GEARED CURVED CRYSTAL SPECTROMETERS" Report of the Pennsylvania State Univ., 1964.
- 3.) J.A. Bearden "X-RAY WAVELENGTHS AND X-RAY ATOMIC ENERGY LEVELS" Rev. Mod. Phys., Vol. 39, No. 78, 1967.
- 4.) J.A. Bearden and A.F. Burr, "REEVALUATION OF X-RAY ATOMIC ENERGY LEVELS", Rev. Mod. Phys., Vol. 31, No. 1, 1967.

Each x-ray line or edge series as a function of atomic number was fit to a fourth degree polynomial. The fit was subtracted from the appropriate data and the residuals plotted and examined. In this way rogue entries could be identified and corrected. The resulting data base is considered to be sufficiently accurate for any application involving the Si (Li) x-ray detector and single crystal wavelength spectrometers.

Note that the last entry in the x-ray database window gives a code for the source of the entry. If the column is blank the source is reference 2. If the column contains the letter "C" the source is reference 1. If the letters "BB" appear, the source is reference 4. The letters "W,F" mean that reference 2 was used but the relative transition probability has been adjusted by Fiori. Reference 3 was used as a check since it is the source of many of the entries of reference 1.

In column 3 the notation KA1,2 means the entry is the weighted sum of the KA1 and KA2 in the ratio 2 to 1. For low atomic number the entries are not self-consistent since the data is from different sources. If the column begins with the capital letter S then the entry is a satellite line due to doubly ionized atoms. The relative transition values for these entries are only valid for electron excited specimens, and are, at best, estimates.

The following are Siegbahn to shell-transition notation conversions:

|         |                       |
|---------|-----------------------|
| KA      | = KA1+KA2+KA3         |
| KA1 , 2 | = ( 2*KA1+KA2 ) / 3   |
| KA1     | = K-L3                |
| KA2     | = K-L2                |
| KA3     | = K-L1                |
| KB      | = SUM(KBn)            |
| KBX     | = Metal               |
| KB1     | = K-M3                |
| KB1'    | = KB1+KB3+KB5         |
| KB2     | = ( K-N3 ) + ( K-N2 ) |
| KB2'    | = K-N3                |
| KB2''   | = K-N2                |
| KB3     | = K-M2                |
| KB4     | = ( K-N4 ) + ( K-N5 ) |
| KB5     | = ( K-M4 ) + ( K-M5 ) |
| KB5'    | = K-M5                |
| KB5''   | = K-M4                |

```

Kd1    = K-O3
Kd2    = K-O2
LA     = LA1+LA2
LA1    = L3-M5
LA2    = L3-M4
LB1    = L2-M4
LB10   = L1-M4
LB15   = L3-N4
LB17   = L2-M3
LB2    = L3-N5
LB3    = L1-M3
LB4    = L1-M2
LB5    = (L3-O4)+(L3-O5)
LB6    = L3-N1
LB7    = L3-O1
LB9    = L1-M5
LG1    = L2-N4
LG11   = L1-N5
LG2    = L1-N2
LG3    = L1-N3
LG4    = L1-O3
LG4'   = L1-O2
LG6    = L2-O4
LG8    = L2-O1
Ll     = L3-M1
Ln     = L2-M1
Ls     = L3-M3
Lt     = L3-M2
Lu     = (L3-N6)+(L3-N7)
Lv     = L2-N6
MA1   = M5-N7
MA2   = M5-N6
MB    = M4-N6
MG    = M3-N5
MG2   = M3-N4
MZ1   = M5-N3
MZ2   = M4-N2
Md    = M2-N4
Me    = M3-O5

```

## **Emission Table**

This menu lists a table of emission energies (or angstroms) for all elements and their major analytical x-ray lines. An option is provided for printing the data as angstroms or electron volts.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries using the CalcZAF program.

## **Edge Table**

This menu lists a table of absorption edge energies (or angstroms) for all elements and their major analytical x-ray lines. An option is provided for printing the data as angstroms or electron volts.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries using the CalcZAF program.

## **Fluorescent Yield Table**

This menu lists a table of fluorescent yield fractions for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries using the CalcZAF program.

## **MAC Table**

This menu lists a table of MACs (mass absorption coefficients) for the specified element and its major analytical x-ray lines. Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries using the CalcZAF program. The program will automatically load the values based on the selected default MAC table. See the Analytical | ZAF Selection section for a description of the MAC table choices.

---

## **Analytical**

### **Empirical MACs**

This menu allows the user to selectively load empirical MACs (mass absorption coefficients) from a supplied ASCII file. These MACs are loaded from the EMPPMAC.DAT file in the Probe for EPMA directory.

See the documentation or help section under Probe for EPMA Menu Details for further information on using this feature.

The selections made here will be reflected in the calculation of ZAF correction factors and k-ratios in the Standard program. This may be used to determine the effect of various mass absorption coefficients on the correction factors for the standard compositions.

### **ZAF, Phi-Rho-Z, Alpha Factor and Calibration Curve Selections**

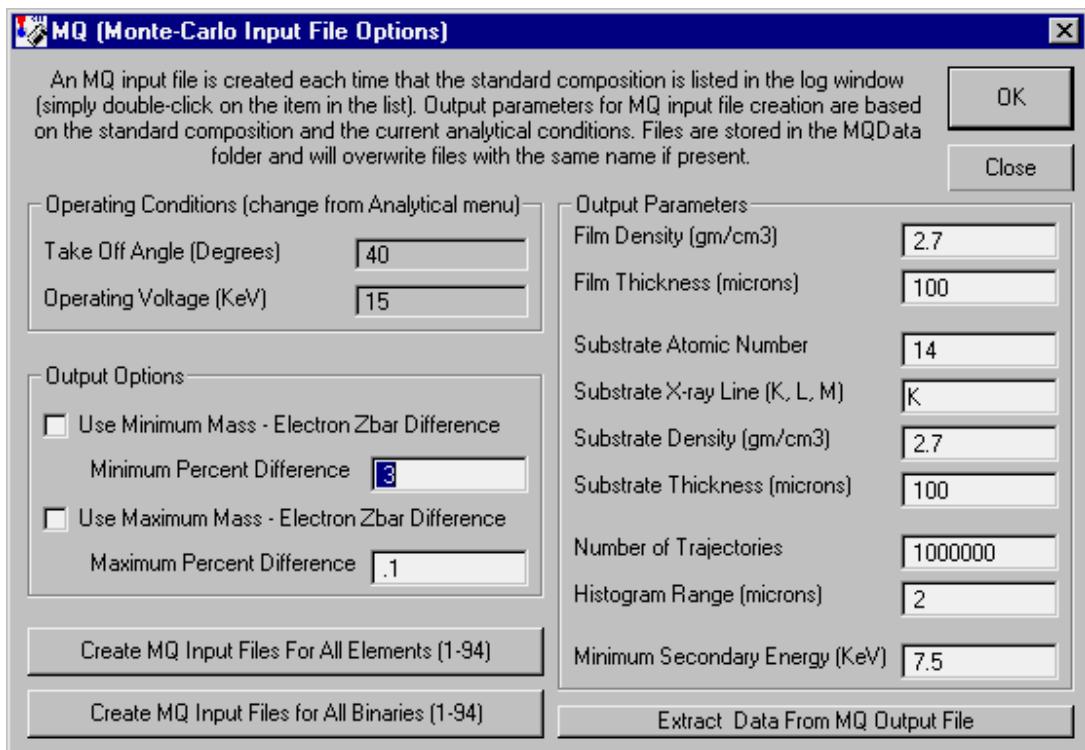
This menu allows the user to modify or display the ZAF or Phi-Rho-Z quantitative matrix correction options for all standards. The selections made here will be reflected in the calculation of ZAF correction factors and k-ratios in the Standard program.

Please refer to the section on ZAF, Phi-Rho-Z, Alpha Factor and Calibration Curve Selections for under the Probe for EPMA Menu details for further information on selecting these options.

### **Operating Conditions**

This menu allows the user to modify or display the accelerating voltage, takeoff angle or beam current for calculation purposes. No changes to the microprobe operation are performed. These parameters are not saved to the Standard database and are only intended for evaluating their effect on the various correction factors calculated by Standard. Of course, only the kilovolts and takeoff parameters have any effect on the calculations.

### **MQ (Monte-Carlo) Calculations**



This menu displays a dialog that is used in conjunction with the NIST (National Institute of Standards and Technology) Monte Carlo electron-solid interaction program (MQ.EXE). This software allows modeling of backscatter and generated and emitted x-ray intensities from bulk (infinitely thick) and thin film compositions and was written by Robert Myklebust and Dale Newbury at NIST. The letter "Q" in the "MQ" files names indicates the version of the NIST Monte Carlo software. This version is current as of Sept. 1999.

The options (and underlying routines) in the Probe for EPMA MQ Options dialog are designed to be used only with the "Q" version of the NIST software. Changes in the input or output format of previous or subsequent versions of the NIST Monte Carlo software could affect the ability of the program to create properly formatted input files or parse output files. The MQ program and source code is provided with the Probe for EPMA installation, with permission of the authors, to ensure compatibility.

### ***Creating MQ Input Files From Standard Database Compositions***

Whenever the Standard program lists data for a standard (whether the user double-clicks in the standard list or uses the List All or Selected Standards), the program will automatically output a properly formatted input file for use with the MQ software.

The compositions, default parameters and conditions in the input file created by Standard may be modified by editing the MQ Options dialog fields before the input files are created. All MQ input files created by his dialog are written to a sub-directory called MQData.

To use the input file simply issue the following command from the Windows command prompt:

```
mq < filename.inp
```

where "filename.inp" is the name of the input file created by Standard. The redirection symbol "<" causes MQ to read input from the ASCII file rather than from the keyboard. Because MQ is a console type FORTRAN program, be sure that input files do not contain spaces or other invalid characters.

## **Using the MCARLO.BAT Batch File**

For best results a batch file can also be created that automatically deletes the output file, copies the input file, runs MQ and copies the output file back to a sub directory. An example batch file (included in the installation) can be seen here:

```
REM This Batch file (MCARLO.BAT) MUST be executed from a data sub-directory!!!
REM Usage: CALL ..\MCARLO "inputfile"           (without extension)
REM (use double quotes if spaces exist in filename)
copy ..\cdonly.dat ..\cdonly.bat > nul
cd >> ..\cdonly.bat
copy %1.inp ..\mcarlo.inp
cd..
del mcarlo.dat
mq < mcarlo.inp
call cdonly
copy ..\mcarlo.dat %1.dat
del ..\cdonly.bat
```

Note that the MCARLO.BAT batch file shown here must reside in the directory containing the MQ executable, but must be run or called from a sub-directory of that directory. This sub-directory should be the directory containing MQ input files and will therefore generally contain the MQ input files previously created by the Standard program.

*The batch file shown above uses a small file called CDONLY.DAT. This file is an ASCII file that contains only the characters "cd ", that is the letters "cd" and a space. This is used to create a small batch file that contains the return path to the sub-directory and is executed once MQ has finished running.*

The MCARLO.BAT batch file shown above must therefore be called from the command line as seen here:

```
..\MCARLO "inputfile"
```

Where the "..\" indicates the parent directory. The MCARLO.BAT batch file will automatically process each input file (\*.INP) and save the output to an output file with that same name but a different extension (\*.DAT).

### **Multiple Batch Processing**

Or the MCARLO.BAT file may be called from another batch file (from the MQ input file sub-directory) as seen here for automating multiple MQ calculations:

```
CALL ..\MCARLO "inputfile1"
CALL ..\MCARLO "inputfile2"
CALL ..\MCARLO "inputfile3"
CALL ..\MCARLO "inputfile4"
CALL ..\MCARLO "inputfile5"
```

A multiple call batch file is automatically created for each type of input file to facilitate extended runs of many compositions. Calls to pure element input files (see next section) for each keV condition are contained in a batch file called 15-ELEMENT.BAT (in this case for 15 keV input files). Calls to the complete set of binary compositions (see next section) are contained in a batch file called 15-BINARY.BAT (again for 15 keV input files). Calls to input files created for compositions in the standard database are contained in a batch file called 15-STANDARD.BAT. All these batch files will be written by default to the MQ-DATA sub-directory.

## **Creating MQ Input Files For Pure Elements or Binary Compositions**

Two buttons on the lower left are used to create a set of MQ input files for each pure element in the periodic table and/or each binary in the periodic table. Once a set of pure element output files have been run through MQ (about 6 hours each for a 1,000,000 trajectory calculation), the "Extract Data From MQ Output File" button may be used to create a spreadsheet containing generated k-ratios and backscatter yields for output files from compound standard compositions.

## Parsing MQ Output Files

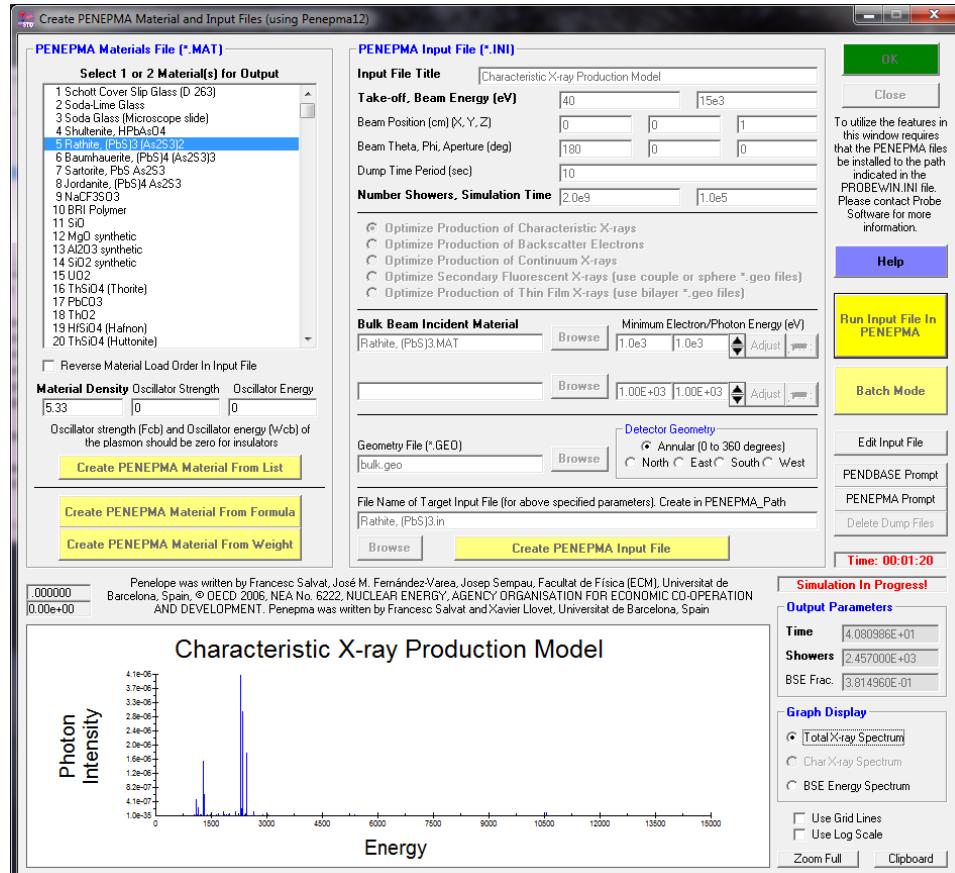
The "Extract Data From MQ Output File" button can be used to parse one or more MQ output files into a tab delimited data file that can then be used for further processing. First the program will ask the user to select the folder containing the output files previously calculated for all the pure elements in the periodic table. Although the program will appear to ask for a specific input file, simply select any pure element output file from the folder that contains the elemental output files.

Next it will prompt the user to select an output file from a compound composition. The program will then parse the compound composition output file and then automatically parse each pure element output file for each element in the compound to calculate elemental k-ratios and backscatter coefficients. The results for this and all subsequent compound output files selected (for this session), are automatically appended to a tab-delimited data file, which can then be easily imported into any plotting or spreadsheet program for further processing.

The data that is currently extracted from the MQ output files is not necessarily complete. If it is desired to automatically extract additional information from the output files, further modifications to the parsing routines can be made. Please contact Probe Software to discuss your specific needs.

## PENEPMa (Monte-Carlo) Calculations

This dialog allows the user to create material (\*.MAT) and input (\*.IN) files for PENEPMa processing. To utilize this option you will need to install the PENEPMa application files on your hard disk. These can be performed by downloading the PENEPMa.ZIP file and extracting them to the "C:\Userdata\Penepma08" folder. If the PENEPMa files are extracted to a different folder, the path keywords will need to be edited in the [software] section of the PROBEWIN.INI file.



## ***Creating Material and Input Files for PENEPMa***

To create material inputs files select up to 10 standard compositions from the standard list, enter a name for the material file and click the Create PENEPMa Material File button. The program will automatically create a material file for each composition and concatenate them into a single material file for input to the PENEPMa Material.exe program. The final material file is automatically copied from the Pendbase folder to the Penepma folder for Monte-Carlo calculations using Penepma.exe. Please be patient, the creation of the material file can take a minute or more if it contains multiple complex compositions.

To create an input file, select the production to optimize (characteristic x-rays, backscatter electrons, continuum x-ray or secondary fluorescent x-rays), a title for the input file, beam energy, beam position, etc. and a material and geometry file. Note that geometry files are complicated and two example files are supplied, bulk.geo and couple.geo. Bulk.geo describes a geometry with a simple flat disk. It can be used with any single composition material files. Generally one enters a beam position of 0, 0, 1 which has the beam position over the center of the disk with a 10 mm (1 cm) working distance.

The geometry file couple.geo is also a flat disk but consisting of two sides (half pie shapes) with different compositions. It can be used with material files that have two compositions. Generally one enters a beam position offset slightly in the x axis so that secondary fluorescent modeling can occur. Values of 1e-3, 0 and 1 would position the beam 10 um from the center of the material interface, centered in y and 10 mm above the sample.

Once the material and the input files are created, click the PENEPMa Prompt button and enter the command: “Penepma < (input file), where (input file) is the name of the input file you just created. Depending on the number of showers simulations the calculation can take up to 24 hour or longer.

Note that 4 sample production files are supplied. They are:

|             |  |
|-------------|--|
| Cu_Cha.in   | Sample characteristic x-ray production input file  |
| Cu_Back.in  | Sample backscatter electron production input file (use data column- “fractional transmitted”!) |
| Cu_Cont.in  | Sample continuum x-ray production input file   |
| CuFe_Sec.in | Sample fluorescent x-ray production input file   |

If the sample production files are edited they must be edited carefully or the program will not function properly. If that occurs it may be necessary to re-extract the sample production files to the root PENEPMa folder.

Consult the PENEPMa.PDF help file for more information.

## ***PENEPMa (Secondary Fluorescence Profile) Calculations***

This menu provides an easy graphical user interface to calculate secondary fluorescence profiles using Penepma12 FORTRAN executables.

This menu is available from both CalcZAF program and Probe for EPMA. Simply select the Standards | Standard Database menu to launch the Standard application. Accept the default (or other) standard database. From the Standard application click the Analytical | PENEPMa (Secondary Fluorescence Profile) Calculations menu.

The general procedure is to select two materials, one each for the two phases of the beam incident and boundary materials and a third phase, usually a pure element as a standard for the specified element and x-ray, for the calculation of k-ratio intensities. If the standard composition selected is NOT a pure element then the k-ratio intensities will be “raw” k-ratios as opposed to elemental k-ratios.

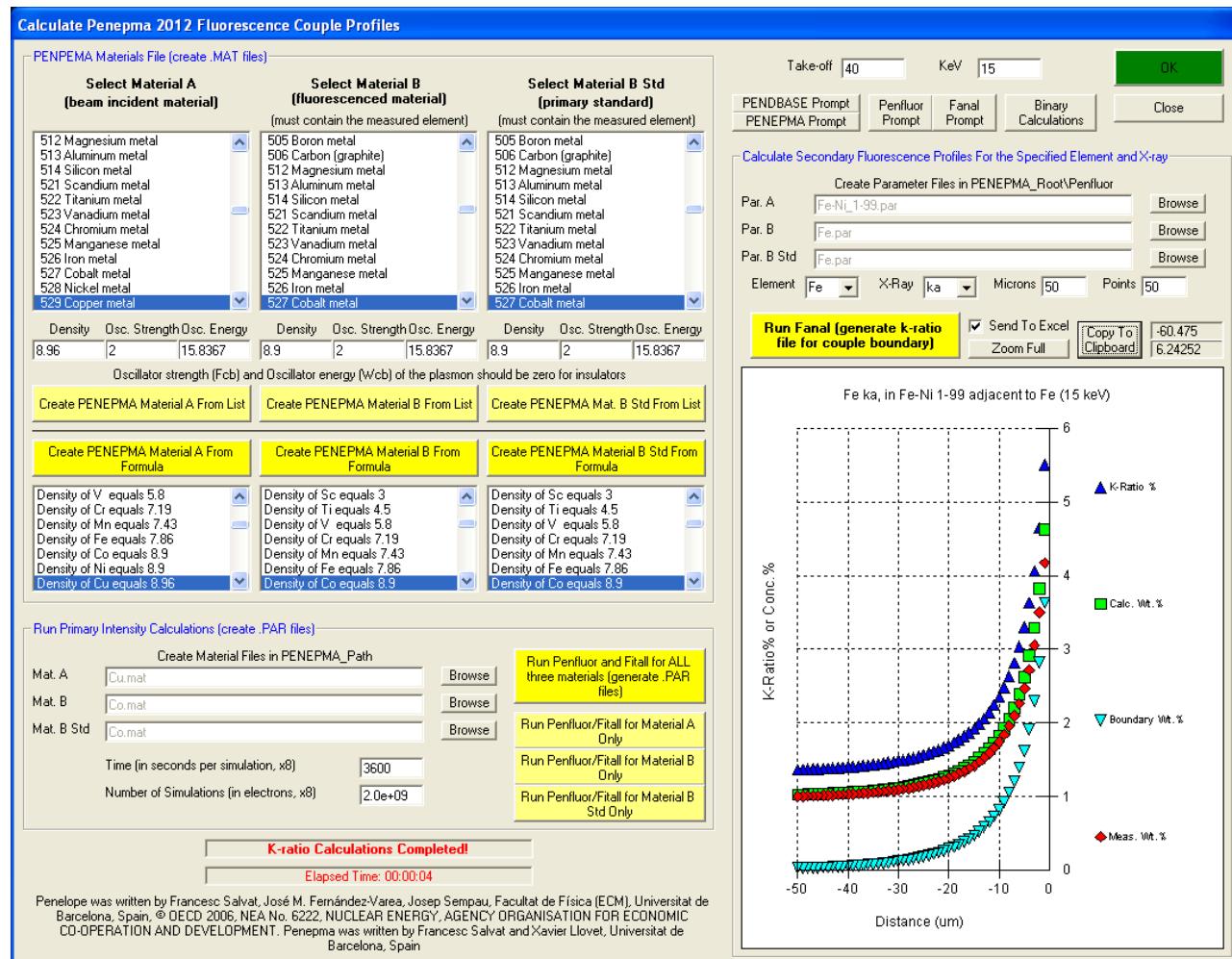
Note that material A is always on the left side of the geometry file, material B is on the right side of the geometry file and the electron beam is always incident on material A. Note that this is the opposite orientation of the normal couple.geo file used by Penepma for Monte Carlo calculations.

The element being fluoresced may only be present in material B or it may be in both material A and material B (or in neither material though this will not be as interesting).

If the same (compound) material is specified for both material A and material B then the calculation merely calculates the normal sample fluorescence (from both characteristic and continuum fluorescence) and also the full bulk matrix correction.

After calculating the three material files (or selecting them using the Browse buttons), click the Run Penfluor and Fitall buttons to calculate the .par files for use by Fanal.exe. The default time for each .par file is 8 hours (3600 seconds for each electron energy for 8 electron energies). The default lowest electron energy is 1 keV and the highest electron energy is 50 keV. The default is to calculate photon energies down to 1 keV although this default may be modified by editing the PenepmaMinimumElectronEnergy keyword in the [software] section of the PROBEWIN.INI file to obtain intensities for lower energy x-ray lines.

Once the PAR files are calculated one may use the Run Fanal button to run program Fanal.exe to calculate k-ratios for the boundary or matrix specified for Par. A, Par. B and Par. B Std. The following will describe the basic functions in more detail.



### PENEPEMA Material Files (create .MAT files)

This section allows the user to specify a material A, material B and/or material B Std for creating material files (.MAT) for use by Penfluor calculations.

Note that the user must specify the correct density for the subsequent distance calculations in Fanal to be accurate and if an insulator is specified the Oscillator Strength and Oscillator Energy values should be zero.

## **Run Primary Intensity Calculations (create .PAR files)**

This section allows the user to calculate parameter files (for use by Fanal) for the calculation of primary intensities. The user may calculate parameter files for material A only, B only, B Std only or for all three consecutively. Each parameter file by default takes about 8 hours to calculate.

## **Calculate Secondary Fluorescence Profiles**

This section is where the user may calculate matrix or boundary k-ratios based on the selected parameter files and the specified element and x-ray (at the specified takeoff angle and electron energy).

The parameter A file is the beam incident material and may or may not contain the element of interest. The parameter B file is the boundary material (it may be the same as the beam incident material for bulk matrix calculations) and may or may not contain the element of interest, though normally except for continuum fluorescent calculations it will. The parameter B Std material is the standard material and the actual composition is not critical for the purposes of these calculation though it must contain the element of interest and generally should contain a high concentration of the element of interest for highest possible precision. This means that one should always specify a pure element standard for most calculations. Note that if the B Std parameter file is NOT a pure element, the program will not be able to calculate a number of interesting parameters.

## **Send To Excel**

The Send To Excel option will ask the user if they desire to have the k-ratio and matrix correction calculations output to an Excel spreadsheet. The output data can be utilized for a number of purposes but is primarily intended to allow the user to estimate the contribution of secondary fluorescence from a boundary phase. The column labels are as follows:

| TO/keV/Elm/Xray |                 |               |                |               |                 |                 |                  |                     |                     |                     |
|-----------------|-----------------|---------------|----------------|---------------|-----------------|-----------------|------------------|---------------------|---------------------|---------------------|
| A               | B               | C             | D              | E             | F               | G               | H                | I                   | J                   | K                   |
| 1               | TO/keV/Elm/Xray | Distance (um) | Total Inten. % | Fluor. Only % | Fluor. A Only % | Fluor. B Only % | Pri. Int. Only % | Calc. Total Conc. % | Calc. A Flu Conc. % | Calc. B Flu Conc. % |
| 2               | 40/15/Fe/k      | -1            | 5.50758        | 4.55843       | 0.240717        | 4.317711        | 0.949151         | 4.628718            | 0.202305            | 3.8                 |
| 3               | 40/15/Fe/k      | -2            | 4.64879        | 3.69964       | 0.266705        | 3.432923        | 0.949151         | 3.823458            | 0.219355            | 2.82                |
| 4               | 40/15/Fe/k      | -3            | 4.06575        | 3.1166        | 0.284964        | 2.831636        | 0.949151         | 3.294455            | 0.230905            | 2.25                |
| 5               | 40/15/Fe/k      | -4            | 3.63443        | 2.68528       | 0.298843        | 2.386438        | 0.949151         | 2.912208            | 0.239458            | 1.91                |
| 6               | 40/15/Fe/k      | -5            | 3.30014        | 2.35099       | 0.309845        | 2.041143        | 0.949151         | 2.62124             | 0.246104            | 1.62                |
| 7               | 40/15/Fe/k      | -6            | 3.03305        | 2.08389       | 0.318805        | 1.765091        | 0.949151         | 2.392071            | 0.251431            | 1.39                |
| 8               | 40/15/Fe/k      | -7            | 2.81493        | 1.86578       | 0.32624         | 1.53954         | 0.949151         | 2.207104            | 0.255795            | 1.20                |
| 9               | 40/15/Fe/k      | -8            | 2.63383        | 1.68468       | 0.332504        | 1.352178        | 0.949151         | 2.056016            | 0.259433            | 1.05                |
| 10              | 40/15/Fe/k      | -9            | 2.48148        | 1.53233       | 0.33784         | 1.194487        | 0.949151         | 1.92812             | 0.262503            | 0.92                |
| 11              | 40/15/Fe/k      | -10           | 2.35192        | 1.40277       | 0.342425        | 1.060342        | 0.949151         | 1.820963            | 0.265121            | 0.82                |
| 12              | 40/15/Fe/k      | -11           | 2.24076        | 1.29161       | 0.346399        | 0.945214        | 0.949151         | 1.729577            | 0.267375            | 0.72                |
| 13              | 40/15/Fe/k      | -12           | 2.14467        | 1.19552       | 0.349864        | 0.84566         | 0.949151         | 1.650992            | 0.269329            | 0.65                |
| 14              | 40/15/Fe/k      | -13           | 2.06107        | 1.11192       | 0.3529          | 0.759017        | 0.949151         | 1.582937            | 0.271033            | 0.58                |
| 15              | 40/15/Fe/k      | -14           | 1.98792        | 1.03877       | 0.355577        | 0.68319         | 0.949151         | 1.523624            | 0.272529            | 0.52                |
| 16              | 40/15/Fe/k      | -15           | 1.92359        | 0.974443      | 0.357941        | 0.616502        | 0.949151         | 1.471651            | 0.273845            | 0.47                |
| 17              | 40/15/Fe/k      | -16           | 1.86679        | 0.917639      | 0.360044        | 0.557595        | 0.949151         | 1.425902            | 0.275011            | 0.42                |
| 18              | 40/15/Fe/k      | -17           | 1.81643        | 0.86728       | 0.361917        | 0.505365        | 0.949151         | 1.385454            | 0.276047            | 0.36                |

TO/keV/Elm/Xray      The takeoff, keV, element and x-ray calculated  
 Distance (um)      The linear distance from the boundary in microns  
 Total Inten. %      The total intensity emitted from A and B in % relative to the standard  
 Fluor. Only %      The total fluorescence from both material A and material B  
 Fluor. A Only %      The fluorescence intensity (characteristic and continuum) from A only  
 Fluor. B Only %      The fluorescence intensity (characteristic and continuum) from B only

Pri. Int. Only %      The primary intensity in % from electron excitation relative to the standard  
 Calc. Total Conc. %      The "calculated" concentration assuming the matrix correction from matrix A  
 Calc. A Flu Conc. %      The "calculated" concentration of the fluorescent intensity from material A  
 Calc. B Flu Conc. %      The "calculated" concentration of the fluorescent intensity from material B

|                      |   |
|----------------------|---|
| Calc. Pri. Conc. %   | The "calculated" concentration of the primary intensity from A and B          |
| Meas. Total Inten. % | The "measured" concentration of the fluorescent intensity from material A     |
| Meas. ZAF            | The bulk matrix correction utilized by CalcZAF or Probe for EPMA              |
| Meas. Total Conc. %  | The "measured" concentration assuming the bulk matrix correction from CalcZAF |
| Actual A Conc. %     | The actual concentration in material A  |
| Actual B Conc. %     | The actual concentration in material B  |
| Actual B Std Conc. % | The actual concentration in material B Std                                    |
| ZA u/s (Fanal)       | The absorption-atomic number matrix correction only from Fanal                |
| Flu u/s (Fanal)      | The fluorescence correction only from Fanal                                   |
| ZAF u/s (Fanal)      | The full matrix correction from Fanal k-ratios                                |
| ZA u/s (CalcZAF)     | The absorption-atomic number matrix correction only from CalcZAF              |
| Flu u/s (CalcZAF)    | The fluorescence correction only from CalcZAF                                 |
| ZAF u/s (CalcZAF)    | The full matrix correction from CalcZAF k-ratios                              |
| A Abs (CalcZAF)      | The CalcZAF absorption correction for material A                              |
| A Flu (CalcZAF)      | The CalcZAF fluorescence correction for material A                            |
| A Zed (CalcZAF)      | The CalcZAF atomic number correction for material A                           |
| A ZAF (CalcZAF)      | The CalcZAF matrix correction for material A                                  |
| B Abs (CalcZAF)      | The CalcZAF absorption correction for material B                              |
| B Flu (CalcZAF)      | The CalcZAF fluorescence correction for material B                            |
| B Zed (CalcZAF)      | The CalcZAF atomic number correction for material B                           |
| B ZAF (CalcZAF)      | The CalcZAF matrix correction for material B                                  |
| B Std Abs (CalcZAF)  | The CalcZAF absorption correction for material B Std                          |
| B Std Flu (CalcZAF)  | The CalcZAF fluorescence correction for material B Std                        |
| B Std Zed (CalcZAF)  | The CalcZAF atomic number correction for material B Std                       |
| B Std ZAF (CalcZAF)  | The CalcZAF matrix correction for material B Std                              |
| Distance (ug/cm2)    | Mass distance in micrograms per square centimeter                             |

In general, the user should subtract the elemental weight percent concentration reported by Probe for EPMA from the "Meas. Total Conc. %" in column L to obtain the actual fluorescence contribution from the presence of the boundary phase. These values can then be subtracted from the elemental weight percent concentration reported from Probe for EPMA to perform a boundary fluorescence correction assuming that appropriate densities were assumed in the material files and proper boundary distances were calculated.

Note that this is only a first order boundary correction since the matrix correction should then be recalculated after subtraction, but for correction of trace and even minor element concentrations the calculation will be very accurate. Eventually Probe for EPMA will incorporate this boundary fluorescence correction rigorously into the matrix iteration where the contribution from the boundary phases will be iteratively corrected for a full quantitative treatment.

### **Assumption Of Bulk Matrix Corrections**

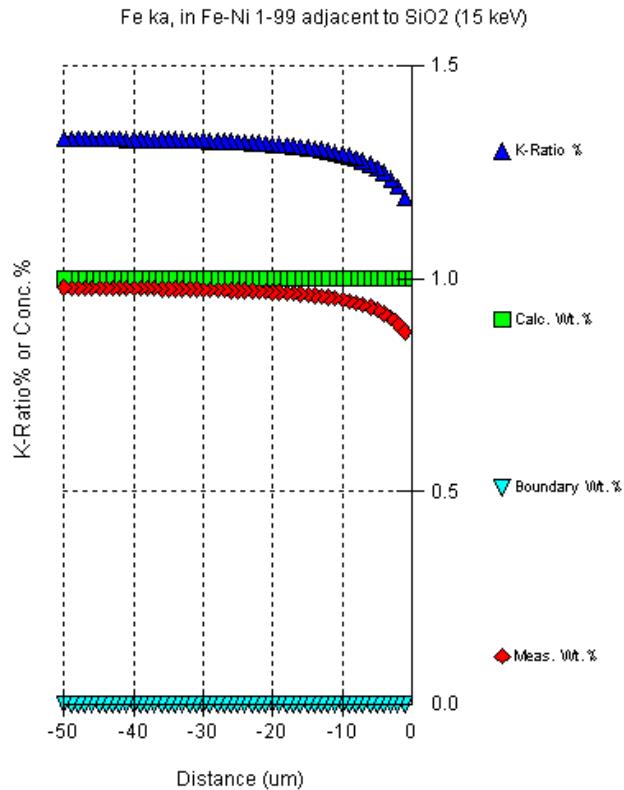
In the meantime it should be noted that when an EPMA measurement is made near a boundary, most EPMA analysis software will generally report a concentration based on the assumption of a matrix correction based on all the intensities measured. Since this is not actually correct the utilization of these calculated intensities from Fanal requires some careful thought when performing off-line corrections of boundary fluorescent effects.

Note that the difference between the "Calc. Total Conc. %" (column H) and the "Meas. Total Conc. %" (column L) is that the "calculated" values are calculated using a matrix correction derived from the internally self-consistent k-ratios calculated by Fanal (which assume that the intensity emitted from material B is only affected by material B), while the "measured" values are the concentrations calculated using the Probe for EPMA matrix correction assuming that the measured intensities from both the beam incident material and the boundary material represent a homogeneous material. In other words the assumption of a bulk matrix correction.

Note that this is not actually the physical situation of a measurement near a boundary phase, however in order to correct the reported values from Probe for EPMA (which assumes a bulk correction for the time being), the use of the "measured" concentrations is necessary for an "apples to apples" comparison of the concentrations.

### **Compositions Exhibiting Self Fluorescence Adjacent to a Non-Fluorescing Phase**

Note that if the beam incident material is self-fluorescing (e.g., materials containing Fe and Ni), and the boundary material is not fluoresced by the beam incident material, an inverse effect can be visible where the intensity from the beam incident material is less than expected leading one to observe a drop in intensity as the boundary is approached as seen here:



Please contact Probe Software for more information on these and other calculations.

## Output

### Log Window Font

This menu allows the user to modify the current log window font type, size and appearance using the font Common dialog.

Note that the default font and size of the log window may be permanently defined in the PROBEWIN.INI file.

### Debug Mode

This menu toggles the program in and out of Debug mode. In Debug mode, the program generates an abundance of additional output to the log window for the purposes of debugging the program or viewing the analytical calculations in greater detail.

### Extended Format

This menu toggles the output option for printout to the log window of element data. If unchecked, the program automatically wraps element data output to 8 elements per line. If checked, the program does not wrap element data to the log window or disk file (if Save To Disk option is enabled).

## **Save To Disk Log**

This menu toggles the output option to mirror all log window output to a user specified text file. This may be useful for importing text or data from the log window to another non-Windows program. In addition, this option may also be used to capture large amounts of output.

This disk log may be viewed using the Output | View Disk Log menu and therefore printed out as a hardcopy if desired.

## **View Disk Log**

This menu will cause the current disk log (if already opened in this session) to be closed and viewed using the specified file viewer in the PROBEWIN.INI file. The default file viewer is Windows NotePad, however another file viewer such as TextPad or Word might be substituted in the PROBEWIN.INI file "FileViewer" keyword.

## **Calculate Electron and X-ray Ranges**

This menu, when checked, will output calculated electron and x-ray ranges based on a range of densities based on the composition of the standard.

## **Calculate Alternative Z-bars**

This menu, when checked, will output an assortment of alternative Z-bars and various fractional models based on the composition of the standard.

## **Calculate Continuum Absorption**

This menu, when checked, will output information on continuum absorption calculations for a variety of x-rays ( $0.01 \sin \theta$  above the characteristic line) for the standard composition.

## **Calculate Charge Balance**

This menu, when checked, will perform a simple calculation of charge balance, based on the current cations and oxide ratios and the composition of the standard.

## **Calculate Total Cations**

This menu, when checked will calculate the total cation formula for the standard composition when displayed in the log window.

## **Display Amphibole Calculations**

Calculate amphibole formula and chemical classification based on Ague AMPHI.F formula re-calculation source code. Output to log window and also to AMPHI.OUT file (subsequent calculations are appended automatically).

## **Display Biotite Calculations**

Calculate biotite formula and chemical classification based on Brimhall, Ague and Donovan BIOTITE.F formula re-calculation source code. Output to log window and also to BIOTITE.OUT file (subsequent calculations are appended automatically).

## **Display ZAF Calculations**

This menu option will force the program to display the intermediate ZAF matrix correction values for each standard that is printed out. For example in addition to the overall ZAF correction factors, it will display the absorption, fluorescence, atomic number corrections along with the stopping power and backscatter factors.

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## **Help**

### **About Standard**

This menu display the copyright notice, acknowledgments and contact information for users of Probe for EPMA.

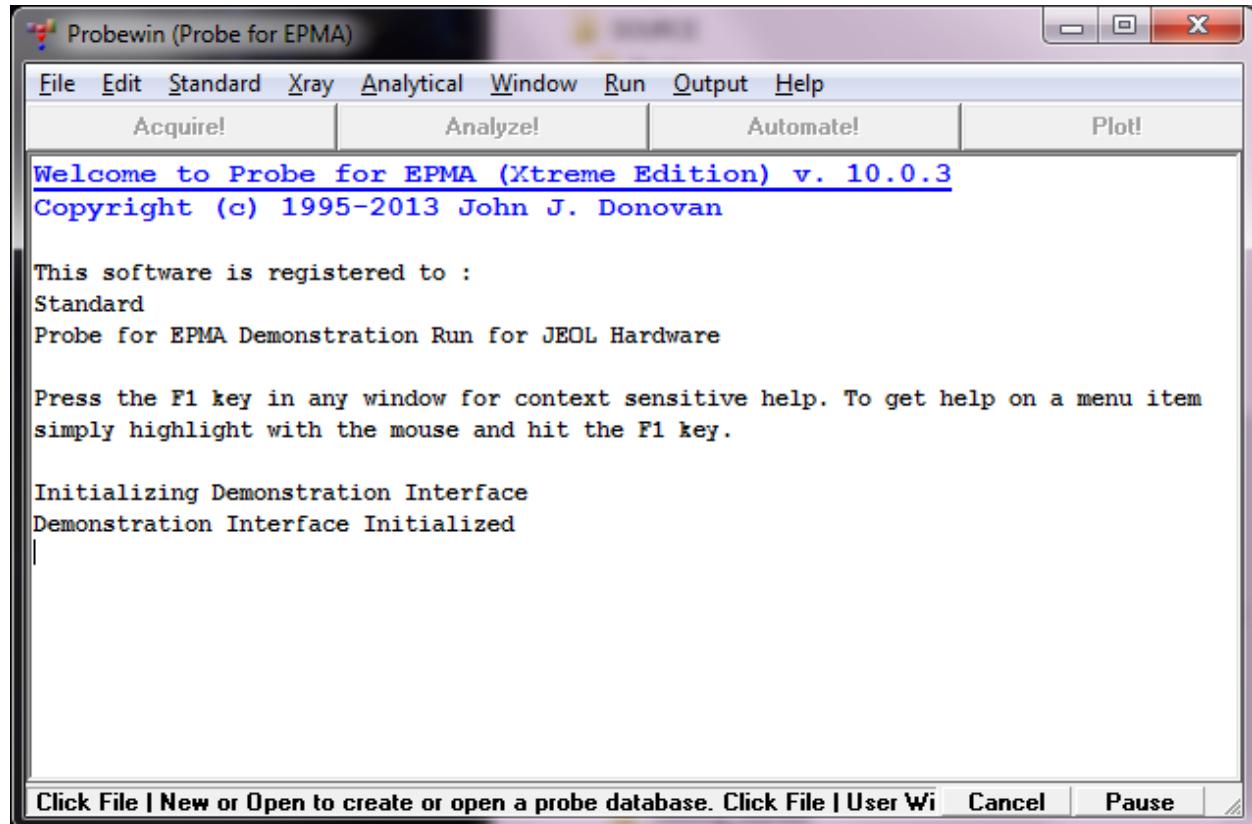
### **Help On Standard**

This menu opens this help file for hypertext help file viewing starting at the main table of contents.

# Probe for EPMA

## Overview

The main program for data acquisition and analysis is PROBEWIN.EXE. The menu for this program is found in the Probe for EPMA menu group under the Start button. Click the menu to start Probe for EPMA Xtreme.



When Probe for EPMA Xtreme is started it will ask first whether to attempt to interface to the microprobe hardware. If data acquisition or automation is desired, then click Yes for confirmation of the spectrometer, stage and crystal positions. If only re-processing previously acquired x-ray intensities, click No.

If the users clicks Yes, the program will attempt to communicate with the microprobe hardware based on the interface type defined in the PROBEWIN.INI file. Note that if the specified hardware is not actually present, this could cause the program to "hang". Be sure to click No if only processing microprobe data off-line.

One may also run Probe for EPMA in "demonstration" mode, e.g. for training new users offline. In that case, which normally will occur on a standalone computer not connected to a probe, you only need to have previously edited the probewin.ini file line under "Hardware" such that InterfaceType=0. Several other keywords or paths may need to be edited to match those in the computer (e.g., FileViewer under [Hardware], and UserDataDirectory under [Software]).

To open an existing Probe for EPMA database file, click File | Open. To open a new Probe for EPMA database file, click File | New. To browse available Probe for EPMA data files, click the Find File menu item.

Creation of a new PROBE file (Microsoft Access database file, actually) raises an important issue: how does one best name it? By user name? By mineral type? By date? Related to this is the issue of how the files are organized. There are many valid schemes; one that works is to organize a tree of folders (may be a couple levels deep, by Department, then Professor, then student), but where each user will have her/his own folder with all their data stored there. In this scenario there is no need for the user name in the new file title. From there, there are many options. One possibility would be something like YL-zirc-031102 that would allow the user to instantaneously recall that this file contains data from Yellowstone zircons that was initially begun on that date (the file date on the computer gets modified whenever the file is opened, so you cannot count on trusting that date). Or Eire-basalt-022201 could contain plagioclase, olivine, oxide and pyroxene data on a suite of basalts. All data -- from standards and unknowns, including wavelength scans on both -- are stored in one file, and easily retrieved and post processed later.

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## Log Window

The main window of Probe for EPMA is a scrollable text window which will record all acquisition and analysis results. This log window is a fully cut and paste enabled text window which can be used to transfer data or analysis results to any other Windows application such as a word processor or spreadsheet.

Simply click and drag the mouse to select text and use the <ctrl> c, <ctrl> x or <ctrl> v key combinations to copy, cut or paste text to or from the Windows Clipboard. Or simply use the Edit menu for these and other Clipboard functions.

The size of the log window buffer can be specified in the PROBEWIN.INI file and is limited only by the amount of memory available. If the Save Log to Disk File option is selected from the Output menu, then all log window output is mirrored to a text file, including any text manually entered by the user using the keyboard. The log window font and font size is specified in the PROBEWIN.INI file and may be changed during a run from the Output menu. To view a copy of the log file during a run, select the View Disk Log option from the Output menu.

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## Menus

The Probe for EPMA log window contains a main menu for easy access to many program functions. Most of these functions are file dependent and are only enabled if a Probe for EPMA database file is opened.

Menu items always available are the Standard | Standard Database and X-Ray menus. The Standard Database menu item launches the Standard application as a separate process, while the X-Ray Database menu item opens the NIST x-ray database as a modal window in Probe for EPMA. The other X-Ray menus list various x-ray data to the log window.

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## Buttons

The Probe for EPMA log window also contains four buttons for easy access to modeless windows that may be open at the same time. A modeless window is defined as a window that may remain open while allowing the focus to shift to another window. Probe for EPMA has four main modeless windows plus a number of smaller windows such as Move, Locate and Position Database modeless windows.

To activate an already open window, simply click the window. If you can't see one of the four main modeless windows because it is hidden by another window, simply click the corresponding button on the Probe for EPMA log window form, and it will be brought forward. The four buttons will in turn activate the Acquire!, Analyze!, Automate! or Plot!

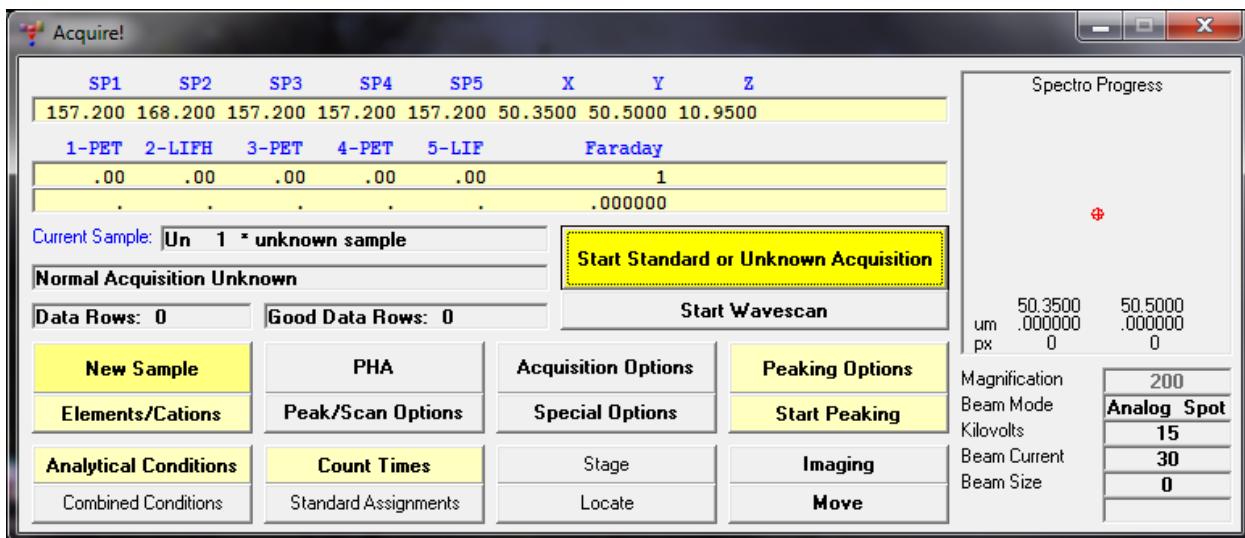
windows. To close a modeless window, simply double click the upper left corner of the window, however these windows will retain their size and position if left open or at least minimized for the duration of the run.

To conserve screen space, it might be wise to minimize windows that are not actively being used at a particular time. To do this, simply click the minimize button in the upper right corner of the window.

# Probe Window Details

## Acquire!

The Acquire! window is used to manually create new standard, unknown or wavescan samples, and to manually acquire x-ray intensity data for them. Other buttons from this window allow the operator to set up all acquisition parameters for the current or subsequent sample (the program will not allow acquisition parameters to be modified on samples that already contain data).



When the Acquire! window is first opened in a new run, the program will prompt the user that the nominal beam current may be acquired. The nominal beam current is used to provide a scaling factor for the beam drift correction (see below under beam drift correction). When the instrument conditions are properly configured (kilovolts and beam current), click Yes to start the nominal beam current acquisition.

To skip the acquisition of the nominal beam current until the next time the Acquire window is activated, click No, or to not acquire the nominal beam current simply click Cancel. In this last instance, the program will use load the nominal beam current from the default beam current from the PROBEWIN.INI file. To disable the beam drift correction or to manually change the nominal beam current, click the Count Times button and enter the new value in nA. A zero nominal beam current disables the beam drift correction.

Other than zero (no beam drift correction) two possible nominal beam current values are 1 (Cameca microprobes use this normalization constant, thus counts are always listed are counts/sec/1 nA) or the value you are actually using (i.e. 20, if you set your current in Analytical Conditions as 20), thus your counts listed are counts/sec/20 nA. There is no "incorrect" nominal value. However, the counts that are listed in the log or Analyze! window are already normalized to this value.

Thus if you were running 100 nA beam current, but you never adjusted the nominal value from a value of "1" that was set during initial installation, your count values would seem very low.

Manual standard and unknown sample data can be acquired on a line by line basis, while manual wavescan samples will automatically acquire a complete set of wavescan data for each element in the wavescan sample. If more than one element is assigned to a given spectrometer, then the program will automatically acquire the additional elements as soon as the previous element wavescan is completed.

At the top of the Acquire! window the current x, y, z stage coordinates, and the spectrometer/scanner positions are displayed as label fields. During data acquisition, real time counts, respective count times, the elements being acquired and the faraday beam counts can be observed as well.

In addition, the current sample name and the number of data points appended to it are displayed just above the New Sample button.

### **Beam Deflection and Column Condition Graphics**

A new feature is the beam deflection and column condition graphics. On the right side of the Acquire! window is displayed the current beam deflection. The actual position is indicated by a red circle and cross and the coordinates are relative to the current magnification. The readout is in microns and pixels (digital scan mode) from the center.

The imaging magnification is used for beam deflection acquisition for unknowns and wavescans (standards are always acquired using the analytical magnification).

The current conditions of the instrument (kilovolts, beam current, etc.) are displayed in the fields below the beam deflection graphic and are updated in real time as the manual or automated acquisition proceeds. During automated beam deflection acquisition (from the Automate!) window, the beam deflection graphic is updated during the acquisition. When deflecting the beam in manual mode from the Imaging window the beam deflection is also correctly displayed.

### **Current Sample**

**The current sample is, by definition, the last sample created by either the user or the automated procedures. This current sample is the only sample to which data can be acquired and appended to.** However, the name and description may be altered easily in the Analyze! window. This is useful when the operator has made an incorrect guess as to the exact phase in a multiphase sample prior to data acquisition and analysis.

Note that wavescan samples can only have data acquired and appended to them once. Be sure that the current sample is the sample to which data should be appended to. Otherwise be sure to create a new sample using the New Sample button.

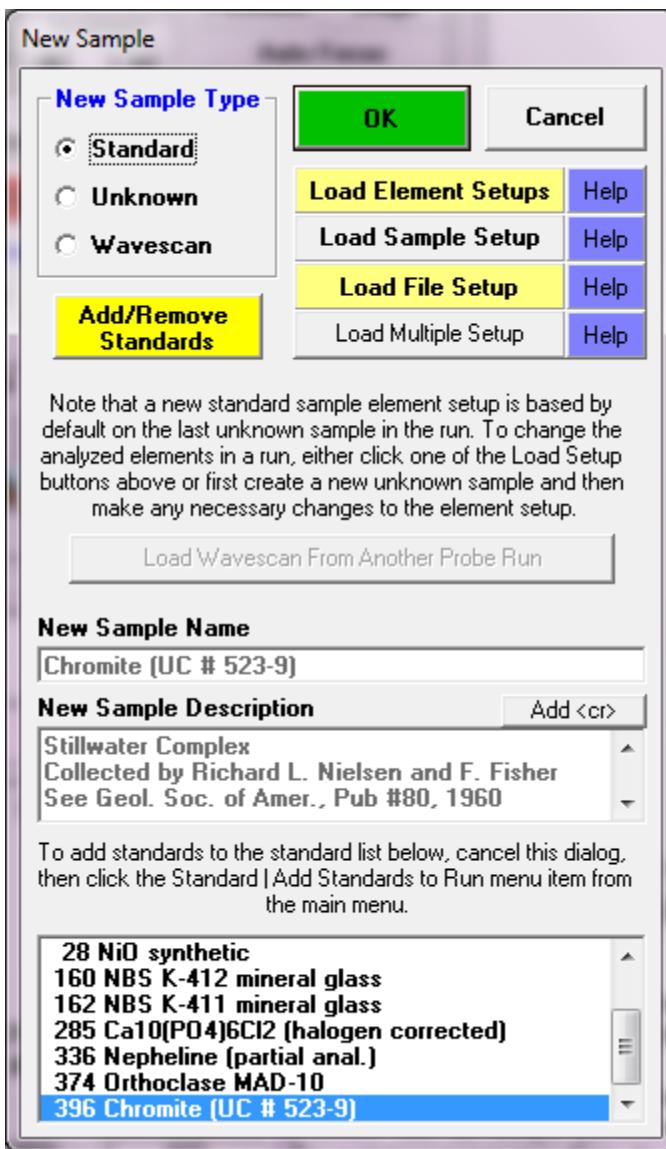
### **New Sample**

Click the New Sample button to start a new sample for the currently open run. This will open the New Sample dialog box. New users should create their first sample as an unknown sample which can then be utilized as a 'template' for creating other samples.

All subsequent new samples (standard, unknown or wavescan) are created based on the **last unknown sample** and the current real-time acquisition parameters specified by the user. Exceptions: if a wavescan sample already exists, the new wavescan is based on the last wavescan, or if no unknown samples exist, the new sample is based on the last standard sample, or if no unknown or standard samples exist, then the new sample is based on the last wavescan sample. The main reason for this is because if the last sample is a standard that was acquired using the "Quick" standard automation option, then all the unknown analyzed elements may not be present.

**It is important to keep in mind that because all assigned sample parameters such as standard assignments, interference assignments, calculation options, specified elements, etc. are loaded from the last unknown sample, if any changes to the sample assignments are *not* made to the last unknown sample, then these sample assignments will *not* be carried forward to the next new sample that is created.**

To change the analytical setup, it is required to either change the analytical setup of the last unknown sample (if it contains no data) or create a new unknown sample and modify it. Note that count time can be changed at any time, even for samples that already contain data.

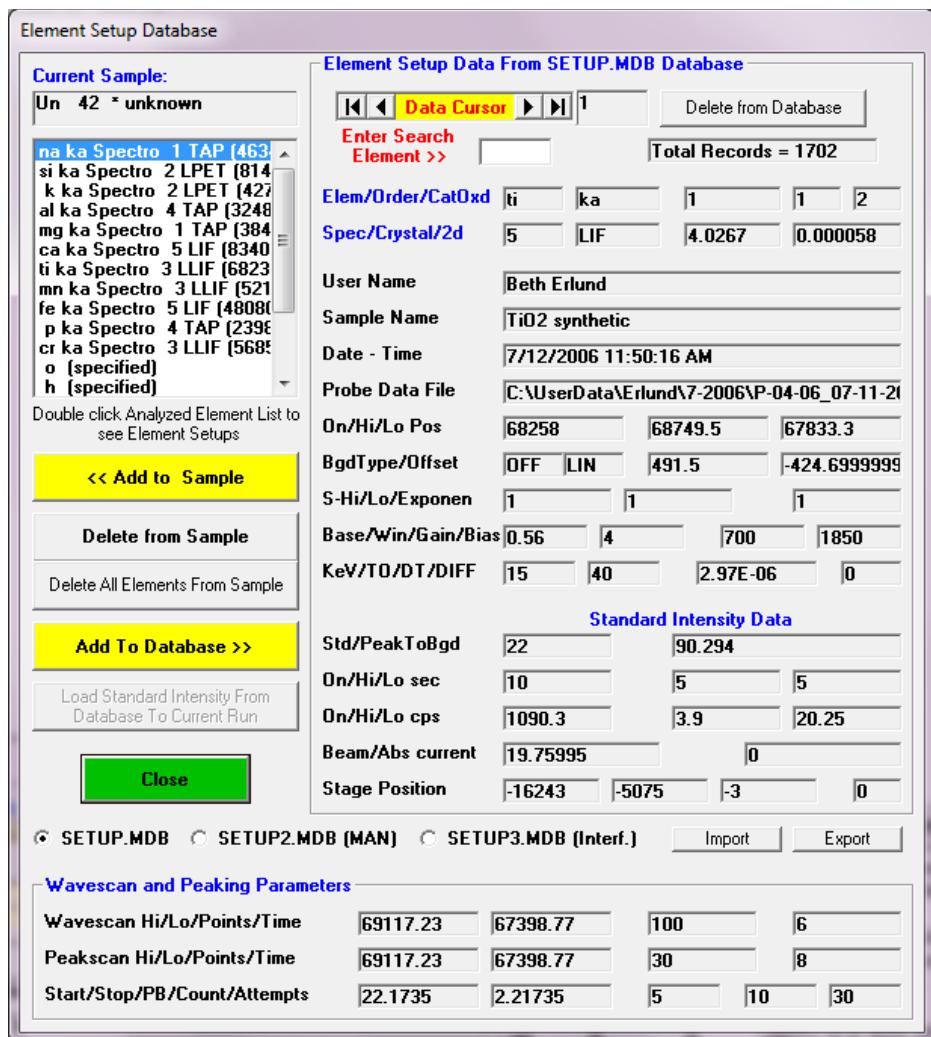


The New Sample dialog allows the user to create a new standard, unknown or wavescan sample. A new sample name and a sample description can be entered. The lower-most portion of the new sample dialog box lists which standards are currently in the run. In order for a standard sample to be created, it must first be added to the run. This is done by first clicking the Standard | Add/Remove Standards To/From Run menu item from the Probe for EPMA log window menus.

Once the sample type is selected and the name is entered, the user can simply click OK and the new sample will be created. If this is the first sample for a new run, the program will create a sample with no analyzed elements. The user may add analyzed elements using the Elements/Cation button described below.

### **Load Element Setup**

To add or remove elements from the sample, or to save or load element setups to or from the element setup database (SETUP.MDB), click the Load Element Setup button.



This dialog provides a method to save and load element setups that have been calibrated for peak positions and PHA parameters for use in creating new analytical setups. If the element setup is saved from a standard sample that has had data acquired for it, the intensity data is automatically saved to the element setup database for evaluation purposes. Note that element setups can be saved anytime, even when not interfaced to the microprobe hardware, from the Analyze! window using the Elements/Cations and Load Element Setup buttons.

**Probe for EPMA Xtreme also includes the complete wavescan and peaking parameter data for each element to be saved and recalled from the setup database. However, note that element setup data imported from the 16 bit version of Probe for EPMA will not contain any wavescan or peaking parameter data and therefore those fields will be blank, although when loaded into a new sample, those blank fields will be replaced with appropriate defaults.**

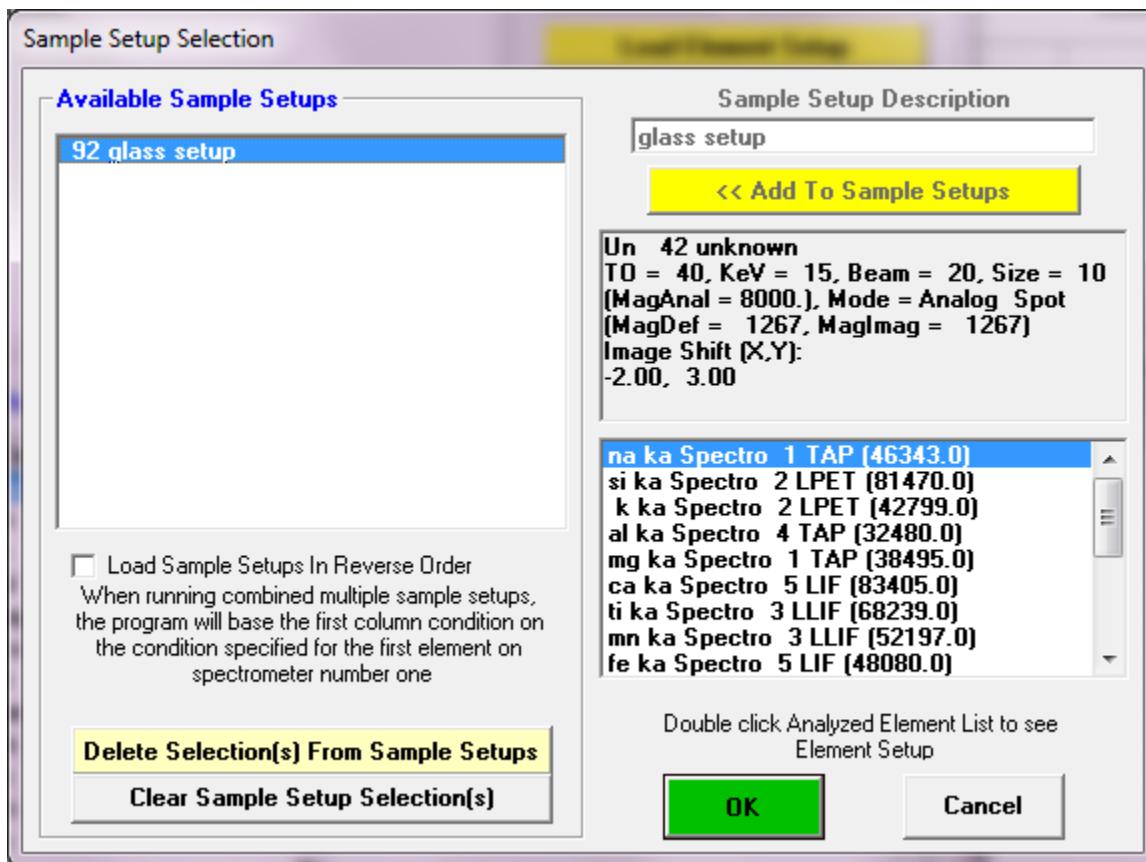
The element setup database can be browsed using the data cursor in the upper right of the window, or all occurrences of a specific element can be browsed by entering the element symbol in the Search Element field. To return the browser to display all elements, simply delete the element symbol from the Search Element field.

The browser displays element setups in the order that they were saved to the setup database. To go to the most recent setup, click the extreme right end of the data cursor. The number of element setups currently in the database is shown also.

Although element setups can be deleted, it may be desirable to keep all element setups for possible evaluation of position or intensity trends for microprobe performance records.

### Load Sample Setup

To save or load sample setups to or from the current run, click the Load Sample Setup button. This button allows the user to load sample setups previously saved **within the current run** for later use in acquisition of new samples.



Loading a previously saved sample setup (see Elements/Cations | Save Sample Setup buttons in the Analyze! Window) to a new sample means that all sample parameters for the new sample will be based on that sample setup. Any modifications to the real-time parameters from the Acquire! Window, such as kilovolts, beam current, beam size, count times, PHA, peak positions, etc. are applied only to the current sample and will not affect the original sample setup.

For example, it might be useful to create several sample setups within a run, if it is known that several different phases will be analyzed, each of which are significantly different in their elemental compositions. In other words, it is possible to create one sample setup containing the elements Fe, Ni and Cr as the analyzed elements and another sample setup containing the elements Al, Mg, and Cu as the analyzed elements, all within the same run.

These setups can be referenced for use with the automation actions for tremendous flexibility during the automated acquisition of different phases within a single run by using the Setups button in the Automate! window during the digitization of position samples.

However, if the sample pointed to by the sample setup contains any data, the element setup cannot be changed later on. For this reason it might be best to only reference samples that do not contain any data lines but only if one wishes to have the ability to modify the original sample setup.

Note that the sample setup is actually a pointer to the row number of the selected sample. Any changes in the selected sample parameters will be automatically included when the sample setup is selected later on. This row number may be viewed for all samples in the current run from the Run | List Sample Names menu.

Sample setups may be loaded (or saved for the current sample), if it does not contain any data, by clicking the Elements/Cations button from the Acquire! window and then clicking the Load Sample Setup button. A previously created sample can be saved as a sample setup by using the Elements/Cations button from the Analyze! window.

When saving a sample setups (from the Analyze! Window using the Elements/cations button) always enter a generic name to indicate the purpose of the sample setup. It may be best to name the sample setup something descriptive, such as, "olivine setup" or "volatile calibration setup" so that the correct sample setup may be easily remembered.

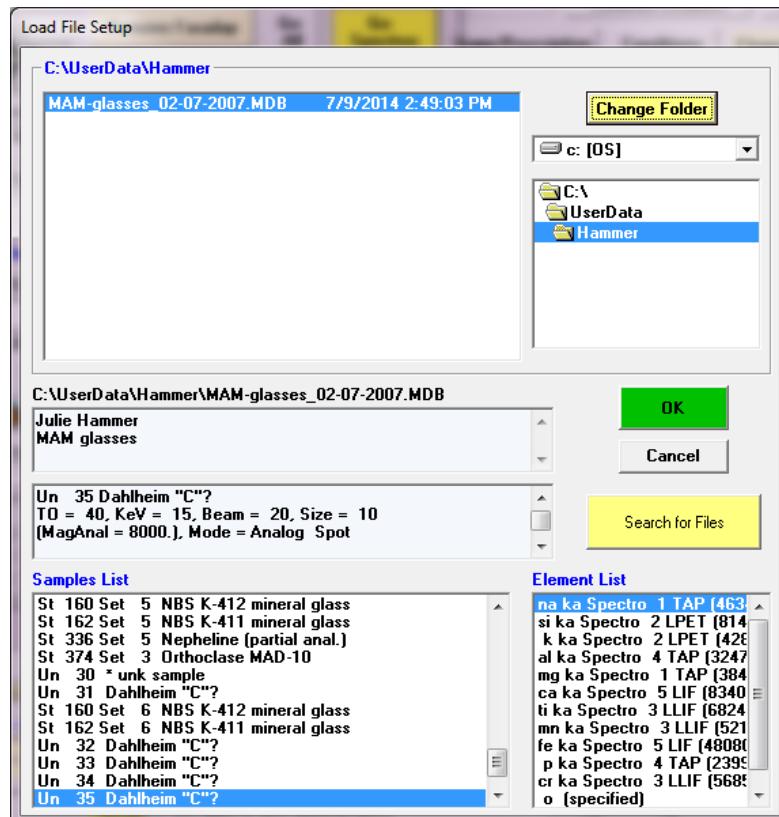
To utilize the full flexibility of PROBE for setting up separate setups for different phases, you will also want to make sure one has set the proper standard assignments (Analyze!-Standard Assignments), and Calculation Options (e.g. Oxygen by stoichiometry, maybe mineral formula type), as well as any various operating conditions in Acquire! (e.g., current, beam size). Once these are set, and the sample setup is saved, ALL these acquisition and data analysis parameters will be used for the appropriate phase.

Alternatively one can save all selected samples as sample setups using the existing sample names by clicking the Add To Setup button in the upper right of the Analyze! Window.

Note that the Save Setups button (in the Analyze! Window) saves all element setups (not sample setups) for standards in the run that are referenced in the selected sample based on the standard assignments.

### Load File Setup

To load the entire sample setup from another run file, including the standard list and MAN assignments, click the Load File Setup button. With this dialog, it is possible to load an entire sample setup from another run for immediate use or modification.



*Note that you will not be able to take advantage of new acquisition features in the software if you continue to append data to an old format probe database. Therefore you should generally create a new file for each probe run and if necessary use the Load File Setup button to load the setup from the previous database file. This way you get access to all the new features and also prevent the database from getting too large and unwieldy.*

Because this dialog provides a listing of the sample setup from each filename clicked, it is an easy matter to browse through all available runs to find a suitable sample setup to load.

Note that loading a file setup will overwrite all current run parameters for the Run, Sample and Element database tables. Only the Nominal Beam and Time Dependent Intensity (TDI) Element correction sample assignments are ignored from the file setup.

After, the file setup (based on the last unknown from the specified file) has been loaded, the program will ask whether to also load the standard intensities. Select Yes, to load the standard intensity data beginning with the last standard and going backwards to the beginning. The standard intensity data timestamp will retain be the original acquisition time. Note that if a standard intensity has already been loaded, the program will not load that particular standard intensity.

### **Load Multiple Setups**

This button is used to load multiple sample setups based on sample setups that have been previously saved sample setups within the current run. All considerations for Samples Setups apply for Multiple Setups (see Sample Setups above). To create sample setups for digitizing position samples, use the "Elements/Cations" button in the Analyze! Window or by clicking the Add To Setup button in the Analyze! window.

When more than one sample setup is specified by the user in the New Sample dialog, the program will automatically combine the specified sample setups into a single sample. This is useful in several situations, for example, when performing major and trace element acquisition it may be desired to acquire the major elements at one set of column conditions (kilovolts, beam current and/or beam size) and the trace elements using a different column condition (kilovolts, beam current, etc.).

In this case, each element will be evaluated and if different column conditions are required, all elements with similar column condition will be acquired before the program attempts acquiring elements with a different column condition. This is analogous to using the "Combine Multiple Setups" check box in the Automate! window. There are, however, important differences. When you "Load Multiple Setups" you create a NEW setup which now has say, two conditions. Those two conditions are applied immediately to each and every point, meaning that the column is constantly changing from the first condition to the second for each spot before proceeding to the next spot. The alternate procedure, using the Combine Multiple Setups checkbox in the Automate! window, applies both (if for example, two) setups to the digitized set of points, but here proceeds through the complete list of points only with the first setup; after the last point is acquired, it will return to the first point and now acquire with the second setup.

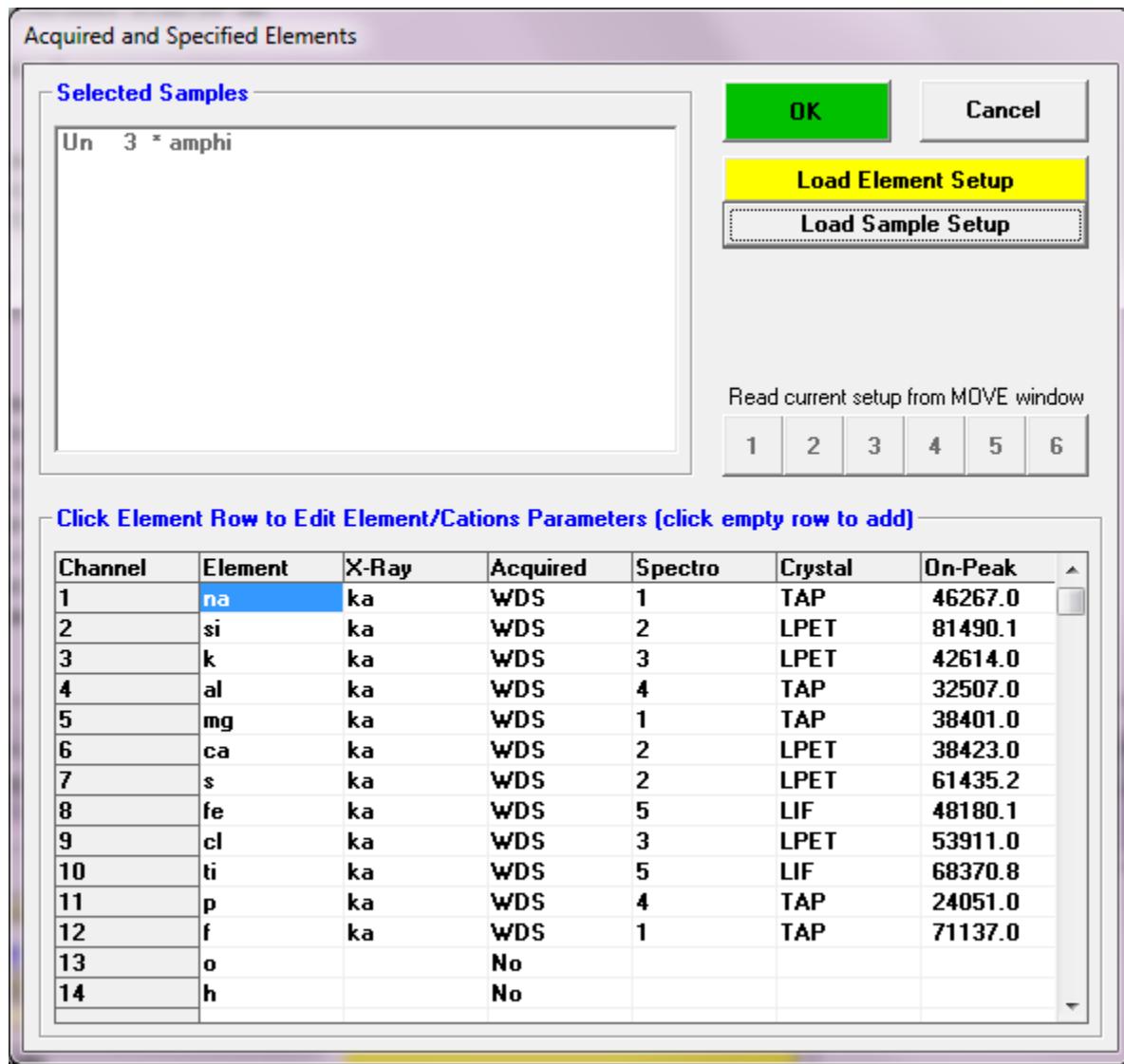
The way the program actually combines multiple setup with multiple column condition samples (in either the Acquire! Or Automate! is as follows: first the program looks at the first spectrometer used and finds the first element on that spectrometer. What the first element is, of course depends on the acquisition order option used. It might be channel order, ascending or descending angstroms or user defined order. Whatever conditions that "first" element uses, that is the first condition that is run. You must add a "dummy" element to the first spectrometer of the first setup if one is not present, to be sure that that setup actually is run first, if that is desired.

After all elements with that condition have been run, then the program looks for the next element (not already acquired) on the first used spectrometer and runs that condition and so on. So keep this in mind when creating "combined" column condition setups.

*See also the Combined Conditions button in the Acquire! window to change the acquisition order of elements using combined conditions so that elements with similar conditions are run as together.*

## Elements/Cations

Clicking the Elements/Cations button opens the analyzed and specified elements dialog box.



The analyzed and specified dialog box lists the currently selected elements for analysis. If this is the first sample of a new run then no analyzed elements are present. To edit or add elements, one of the following procedures may be performed :

1. Click the Load Element Setup button to load previously saved element setups from the element setup database. Element setups in samples created before the current sample may be saved as element setups from the Elements/Cations button in the Analyze! window.
2. Click the Load Sample Setup button to load a previously saved sample setups in the currently open run. Samples created before the current sample may be saved as a sample setup from the Elements/Cations button in the Analyze! window. If you are loading a multiple sample setup, only one column condition will be preserved. You need to check and set it for the appropriate setting for your next step, e.g. peaking.

- 3. Click the grid row of the element of interest or an empty row to add new elements. This action activates the element properties dialog box. One then selects an element by either typing the element symbol or by clicking the drop down list of all elements and scrolling to and highlighting the desired element.**

Default settings for the analyzed x-ray line, cations, and oxygen proportions are loaded automatically. These can be edited via the drop down menu associated with each based on the user's preferences.

The cation/oxygen ratios (subscripts) for Fe:O are calculated here for those performing "what if" calculations for testing the variations in the relative amounts of FeO and Fe<sub>2</sub>O<sub>3</sub> in cases where it is a valid exercise (e.g., typical volcanic glasses and some minerals). It is valid first, to show the effect of changing the oxygen abundance in a matrix correction and second, to see the effect on the analytical total:

| Fe | O | Fe <sub>2+</sub> | Fe <sub>3+</sub> |
|----|---|------------------|------------------|
| 1  | 1 | 100              | 0                |
| 8  | 9 | 85               | 15               |
| 7  | 8 | 83               | 17               |
| 6  | 7 | 80               | 20               |
| 5  | 6 | 75               | 25               |
| 7  | 9 | 60               | 40               |
| 3  | 4 | 50               | 50               |
| 5  | 7 | 33               | 67               |
| 2  | 3 | 0                | 100              |

Leave the x-ray line field blank to indicate an unanalyzed element (specified, by difference or by stoichiometry or by formula basis calculations). The defaults for these fields can be modified in the ELEMENTS.DAT file in the Probe for EPMA directory. Note that Probe for EPMA will retain any modifications of these defaults for the duration of the run.

### ***Disable Acq***

To disable an element for acquisition, check the Disable Acq checkbox. The element parameters will still be saved, but the acquisition routines will skip the element for standards, unknowns or wavescan samples. To enable the element for acquisition, first create a new sample and then uncheck the option before acquiring any more data.

### ***Disable Quant***

To disable the element for quantitative calculations, check the Disable Quant checkbox. The program will set the intensity to zero for the matrix correction calculations. To restore the element for quantitative calculations, simply uncheck the box. Normally this is only used for special reprocessing situations for example: analyzing duplicate elements.

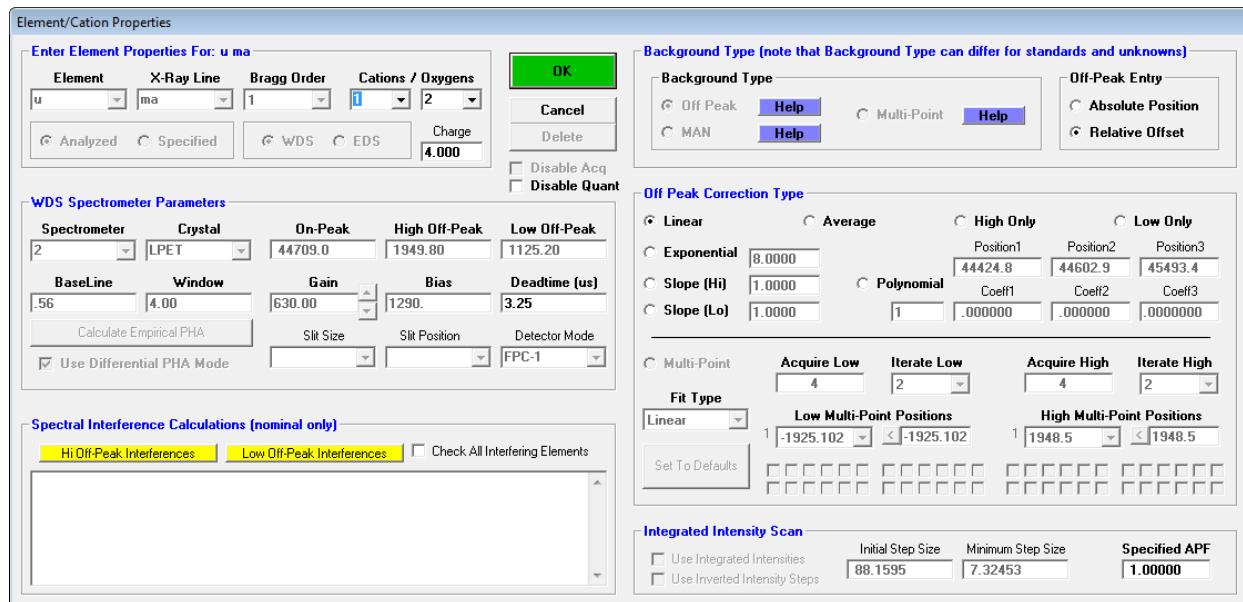
### ***Multiple Peak Calibration***

*Special note: if the "UseMultiplePeakCalibrationOffset" parameter is set in the PROBEWIN.INI file and the fit coefficients have been properly calibrated using the Peak/Scan Calibrate Parameters dialog in program StartWin, the program will load "corrected" spectrometer peak positions in the Elements/Cations dialog when the user selects an element and x-ray line. See the Calibrate Peak Center help in the StartWin section for more details.*

## Background Types

The choice of background measurement either off-peak or MAN can be specified here. Off-peak correction types include linear, average, high only, low only, exponential and polynomial. MAN backgrounds are based on a calibration curve fitted to appropriate standards that do not contain the element of interest (Donovan and Tingle, 1996). The MAN calibration is assigned from the Analytical | MAN Fits menu after the standards to be used in the MAN fit have been acquired.

Note that one can utilize on-peak intensities for MAN background calculations even if the standards were acquired using off-peak background types but selecting the "Use Off-Peak Elements for MAN Fit" menu (under the Analytical menu).



Note that because the background type flags may be specified differently for standards and unknowns, the Off/MAN flags specified here will be saved for only the current sample type. This means that if the current sample type is an unknown, then the MAN flags defined in the Element/Cation Properties dialog will only be applied to unknown samples. To define the MAN flags for both the standards and unknowns, select them from the Acquisition Options window by clicking the Acquisition Options button in the Acquire! window.

### Off-Peak Backgrounds

The off-peak background correction measures the background on the sample of interest (i.e., your standard or unknown) with the spectrometer adjusted to a position on each side of the analytical peak.

Probe for EPMA can individually off-peak correct each point analysis for standard and unknown samples. If the element concentration is very small, it may be necessary to use the off-peak corrected sample method. The program is very flexible in that it allows you to use all MAN or all off-peak background corrections or any combination of the two methods on each element of an acquisition.

Note that since the off-peak positions are saved on a sample by sample basis, one must always start a new standard or unknown sample after changing the off-peak positions or adjusting them graphically using the mouse from a wavelength scan from the Plot! window. Probe for EPMA uses a linear slope calculation as the default off-peak background correction. Because of this, unsymmetrically offset background position measurements can be easily handled. One can even position both of the off-peak measurements on the same side of a peak and extrapolate the background correction.

In addition, the user can also specify that the program calculate the off-peak counts using the average of the off-peak counts, or only the high or only the low off-peak counts. Finally, the user can specify an exponential slope calculation for curved backgrounds. If one changes the off-peak background type for an element, it may be necessary to change it for both the unknown samples, as well the standards, depending on the analytical situation. This is easily done in the Model

Background dialog which can be reached from the Graph dialog from the Plot! Window Output button, by selecting all samples that you wish to assign the new background type to and clicking Ok.

### Multi-Point Backgrounds

Multi-point backgrounds are utilized when performing high accuracy trace element analysis on sample with variable composition, e.g., monazites. In such cases the presence of minor and even trace levels of unexpected elements may cause a significant interference with the normal high and low off-peak positions of the analytical peak in question.

To avoid this situation the multi-point background acquisition was developed to allow the program to automatically acquire a number of off-peak intensities distributed on each side of the analytical peak (which can be specified precisely by the user) so that at least a few of the background measurements will not be affected by the unpredicted presence of various other elements.

This process is automatically performed iteratively when the background correction is calculated by looping on the data starting with the full set of background measurements and one by one throwing out the background points with the highest variances above the fitted background until the specified number of valid background positions is reached. See code here:

```
' Iterate from NumberofPointsAcquire to NumberofPointsIterate
k% = 1
npts% = MAXMULTI% * 2
nptshi% = MAXMULTI% * 2
nptslo% = MAXMULTI% * 2
Do While nptshi% > sample(1).MultiPointNumberofPointsIterateHi%(j%) Or nptslo% >
sample(1).MultiPointNumberofPointsIterateLo%(j%)

' Load data array for fitting
npts% = 0
nptshi% = 0
nptslo% = 0

' Load high side points
For m% = 1 To sample(1).MultiPointNumberofPointsAcquireHi%(j%)
If sample(1).MultiPointProcessManualFlagHi(i%, j%, m%) Or (Not
sample(1).MultiPointProcessManualFlagHi(i%, j%, m%) And Not badflaghi(m%)) Then
npts% = npts% + 1
nptshi% = nptshi% + 1
mxdata!(npts%) = sample(1).MultiPointAcquirePositionsHi!(j%, m%)
mydata!(npts%) = sample(1).MultiPointAcquireCountsHi!(i%, j%, m%)
End If
Next m%

' Load low side points
For m% = 1 To sample(1).MultiPointNumberofPointsAcquireLo%(j%)
If sample(1).MultiPointProcessManualFlagLo(i%, j%, m%) Or (Not
sample(1).MultiPointProcessManualFlagLo(i%, j%, m%) And Not badflaglo(m%)) Then
npts% = npts% + 1
nptslo% = nptslo% + 1
mxdata!(npts%) = sample(1).MultiPointAcquirePositionsLo!(j%, m%)
mydata!(npts%) = sample(1).MultiPointAcquireCountsLo!(i%, j%, m%)
End If
Next m%

' Re-fit the data using new points
If sample(1).MultiPointBackgroundFitType%(j%) = 1 Then
order% = 2 ' 2nd order polynomial
Else
order% = 1 ' linear
End If
Call LeastSquares(order%, npts%, mxdata!(), mydata!(), acoeff!())
If ierror Then Exit Sub

' Calculate deviations for high side and flag highest positive multi-point background as "bad"
If nptshi% > sample(1).MultiPointNumberofPointsIterateHi%(j%) Then
maxt! = 0#
maxm% = 0
```

```

For m% = 1 To sample(1).MultiPointNumberofPointsAcquireHi%(j%)
If Not sample(1).MultiPointProcessManualFlagHi(i%, j%, m%) And Not badflaghi(m%) Then

    ' Calculate fit and relative distance from fit for each high point
    temp! = acoeff!(1) + acoeff!(2) * sample(1).MultiPointAcquirePositionsHi!(j%, m%) + acoeff!(3) *
    sample(1).MultiPointAcquirePositionsHi!(j%, m%) ^ 2
    If temp! <> 0# Then
        temp! = (sample(1).MultiPointAcquireCountsHi!(i%, j%, m%) - temp!) / temp!
    If temp! > maxt! Then
        maxt! = temp!
        maxm% = m%
    End If
    End If

End If
Next m%

' Flag high side points if indicated
If maxt! <> 0# And maxm% <> 0 Then
    badflaghi(maxm%) = True
Else
    noneflaggedhi = True
End If
End If

' Calculate deviations for low side and flag highest positive multi-point background as "bad"
If nptslo% > sample(1).MultiPointNumberofPointsIterateLo%(j%) Then
    maxt! = 0#
    maxm% = 0
    For m% = 1 To sample(1).MultiPointNumberofPointsAcquireLo%(j%)
        If Not sample(1).MultiPointProcessManualFlagLo(i%, j%, m%) And Not badflaglo(m%) Then

            ' Calculate fit and relative distance from fit for each low point
            temp! = acoeff!(1) + acoeff!(2) * sample(1).MultiPointAcquirePositionsLo!(j%, m%) + acoeff!(3) *
            sample(1).MultiPointAcquirePositionsLo!(j%, m%) ^ 2
            If temp! <> 0# Then
                temp! = (sample(1).MultiPointAcquireCountsLo!(i%, j%, m%) - temp!) / temp!
            If temp! > maxt! Then
                maxt! = temp!
                maxm% = m%
            End If
            End If

        End If
        Next m%

        If maxt! <> 0# And maxm% <> 0 Then
            badflaglo(maxm%) = True
        Else
            noneflaggedlo = True
        End If
        End If

    ' Check if no points were flagged on the high and low side (could not iterate further possibly due to
    manual override points flagged)
    If noneflaggedhi And noneflaggedlo Then
        msg$ = "DataCorrectDataMultiPoint: Unable to iterate to Iterate Points in " & sample(1).Name$ & ", "
        & sample(1).Elsyms$(j%) & " " & sample(1).Xrsyms$(j%) & ", line " &
        Format$(sample(1).LineNumber&(i%))
        Call IOWriteLogRichText(msg$, vbNullString, Int(LogWindowSize%), vbMagenta, Int(FONT_REGULAR%), Int(0))
    End If

    ' Iterate next cycle
    k% = k% + 1
    If k% > maxiter% Then GoTo DataCorrectDataMultiPointTooManyIterations
Loop

' Iteration complete, calculate background based on last fit coefficients
If acoeff!(1) = 0# And acoeff!(2) = 0# And acoeff!(3) = 0# Then GoTo
DataCorrectDataMultiPointNoFitCoefficients

```

```

bgdcts! = acoeff!(1) + acoeff!(2) * sample(1).OnPeaks!(j%) + acoeff!(3) * sample(1).OnPeaks!(j%) ^ 2
sample(1).BgdData!(i%, j%) = bgdcts!

```

The user can alternatively utilize the combined background intensities by selecting one of the normal off-peak background calculations.

### **Off Peak Background Correction Types**

Probe for EPMA allows the user to select the off-peak type for the background correction. The default slope calculation is the most accurate, except in cases where the user has moved both off-peak positions to the same side of the analytical peak and adjusted the off-peaks excessively close together resulting in a large extrapolation. The off-peak correction types are as follows :

0. = linear slope interpolation or extrapolation
1. = average of both high and low off-peak counts
2. = use only the high off-peak counts
3. = use only the low off-peak counts
4. = use exponential off-peak calculation (based on exponent)
5. = slope-high (based on high off-peak and slope coefficient)
6. = slope-low (based on low off-peak and slope coefficient)
7. = polynomial (based on high and low off-peak and three coefficients)
8. = multi-point (user defined number of background positions on each side of peak)

These off-peak background correction types may be assigned on a sample by sample basis either on or off-line. Note that the actual values of the polynomial fit coefficients are dependent on the absolute magnitude of the count intensities. For this reason it is important to avoid changing the value of the nominal beam current once any polynomial fit coefficients have been assigned to samples in the run. The calculations for linear slope and exponential calculation are shown below :

### **Linear Slope Calculation**

$$I_{\text{off}} = I_{\text{low}} + (I_{\text{high}} - I_{\text{low}}) \cdot \frac{(P_{\text{on}} - P_{\text{low}})}{(P_{\text{high}} - P_{\text{low}})}$$

where :  $I_{\text{off}}$  = the off-peak count correction

$I_{\text{high}}$  = the x-ray counts for the high off-peak position

$I_{\text{low}}$  = the x-ray counts for the low off-peak position

$P_{\text{high}}$  = the high off-peak position

$P_{\text{low}}$  = the low off-peak position

$P_{\text{on}}$  = the on peak position

### **Exponential Slope Calculation**

$$I_{\text{off}} = \frac{ce^{-ap}}{P^N}$$

where :  $I_{\text{off}}$  = the off-peak background intensity

$P$  = the spectrometer position

$c$  = a fit parameter

|          |                             |
|----------|-----------------------------|
| <i>a</i> | = a fit parameter           |
| <i>e</i> | = the exponential           |
| <i>N</i> | = a user specified exponent |

See Donovan, et al. Amer. Min. (2011) for more details.

### **Exponential Background Source**

Source code for the exponential background fit is shown here. This first section is the background calculation itself.

```
' Use exponential interpolation
If sample(1).OffPeakCorrectionTypes%(j%) = 4 Then
    txdata!(1) = sample(1).LoPeaks!(j%)
    txdata!(2) = sample(1).HiPeaks!(j%)
    tydata!(1) = sample(1).LoPeakCounts!(i%, j%)
    tydata!(2) = sample(1).HiPeakCounts!(i%, j%)
    Call LeastExponential(txdata!(), tydata!(), sample(1).BackgroundExponentialBase!(j%), acoeff!())
    If ierror Then Exit Sub
    sample(1).BgdData!(i%, j%) = (coeff!(1) * Exp(-coeff!(2) * sample(1).OnPeaks!(j%))) /
        sample(1).OnPeaks!(j%) ^ coeff!(3)
End If
```

This next section is the subroutine used to calculate the exponential coefficients:

```
Sub LeastExponential(txdata() As Single, tydata() As Single, texp As Single, acoeff() As Single)
    ' Calculates the coefficients for a 2 point exponential fit of the form:
    ' y = (c * e^(-ax)) / x^n, where n is user specified

    ierror = False
    On Error GoTo LeastExponentialError

    Dim c As Single, a As Single, n As Single
    Dim temp1 As Double, temp2 As Double

    ' Init "coeff()"
    acoeff(1) = 0#
    acoeff(2) = 0#
    acoeff(3) = 0#

    ' Check for valid input data
    If txdata!(1) <= 0# Then Exit Sub
    If txdata!(2) <= 0# Then Exit Sub
    If tydata!(1) <= 0# Then Exit Sub
    If tydata!(2) <= 0# Then Exit Sub

    ' Load base
    If texp! < -6# Or texp! > 6 Then GoTo LeastExponentialBadExponent
    n! = texp!

    ' Calculate c
    temp1# = (txdata!(1) * Log(tydata!(2)) - txdata!(2) * Log(tydata!(1)))
    temp1# = temp1# + n! * txdata!(1) * Log(txdata!(2)) - n! * txdata!(2) * Log(txdata!(1))
    temp2# = txdata!(1) - txdata!(2)
    If temp2# = 0# Then Exit Sub
    c! = Exp(temp1# / temp2#)

    ' Calculate a
```

```

If c! < 0# Then Exit Sub
temp1# = (Log(tydata!(1)) + Log(tydata!(2)) + n! * (Log(txdata!(2)) + Log(txdata!(1))) - 2 * Log(c!))
temp2# = txdata!(1) + txdata!(2)
If temp2# = 0# Then Exit Sub
a! = -(temp1# / temp2#)

' Load coefficients (1=c, 2=a, 3=n)
acoeff!(1) = c!
acoeff!(2) = a!
acoeff!(3) = n!

Exit Sub

' Errors
LeastExponentialError:
MsgBox Error$, vbOKOnly + vbCritical, "LeastExponential"
ierror = True
Exit Sub

LeastExponentialBadExponent:
msg$ = "Exponent must be greater than -6 and less than 6"
MsgBox msg$, vbOKOnly + vbExclamation, "LeastExponential"
ierror = True
Exit Sub

End Sub

```

## ***Polynomial Background Source***

```

' Use polynomial
pxdata!(1) = sample(1).LoPeaks!(j%)    ' load three known positions
pxdata!(2) = sample(1).OnPeaks!(j%)
pxdata!(3) = sample(1).HiPeaks!(j%)

' Get three y values for these positions
pydata!(1) = sample(1).BackgroundPolynomialCoefficients!(1, j%) +
sample(1).BackgroundPolynomialCoefficients!(2, j%) * sample(1).LoPeaks!(j%) +
sample(1).BackgroundPolynomialCoefficients!(3, j%) * sample(1).LoPeaks!(j%) ^ 2
pydata!(2) = sample(1).BackgroundPolynomialCoefficients!(1, j%) +
sample(1).BackgroundPolynomialCoefficients!(2, j%) * sample(1).OnPeaks!(j%) +
sample(1).BackgroundPolynomialCoefficients!(3, j%) * sample(1).OnPeaks!(j%) ^ 2
pydata!(3) = sample(1).BackgroundPolynomialCoefficients!(1, j%) +
sample(1).BackgroundPolynomialCoefficients!(2, j%) * sample(1).HiPeaks!(j%) +
sample(1).BackgroundPolynomialCoefficients!(3, j%) * sample(1).HiPeaks!(j%) ^ 2

' Scale y values to current nominal beam
If UseBeamDriftCorrectionFlag Then
If sample(1).BackgroundPolynomialNominalBeam!(j%) <> 0# And NominalBeam! <> 0 Then
temp! = NominalBeam! / sample(1).BackgroundPolynomialNominalBeam!(j%)
pydata!(1) = pydata!(1) * temp!
pydata!(2) = pydata!(2) * temp!
pydata!(3) = pydata!(3) * temp!
End If
End If

' Re-fit the data using new scaled y values

```

```

order% = 2 ' 2nd order polynomial
npts% = 3
Call LeastSquares(order%, npts%, pxdata!(), pydata!(), acoeff!())
If ierror Then Exit Sub

If DebugMode Then
msg$ = "Polynomial re-fit coefficients for " & sample(1).Elsyms$(j%) & " (beam correction = " &
Format$(temp!) & ") " & Str$(coeff!(1)) & Str$(coeff!(2)) & Str$(coeff!(3))
Call IOWriteLog(msg$)
End If

' Calculate off-peak values based on new fit coefficients
temp1! = coeff!(1) + coeff!(2) * sample(1).HiPeaks!(j%) + coeff!(3) * sample(1).HiPeaks!(j%) ^ 2
temp2! = coeff!(1) + coeff!(2) * sample(1).LoPeaks!(j%) + coeff!(3) * sample(1).LoPeaks!(j%) ^ 2

' Calculate offsets from current off-peak measurements
temp3! = sample(1).HiPeakCounts!(i%, j%) - temp1!
temp4! = sample(1).LoPeakCounts!(i%, j%) - temp2!

' Average current offsets
temp! = (temp3! + temp4!) / 2#

' Calculate new y intercept for this sample based on offset
tintercept! = temp! + coeff!(1)

' Calculate actual background using new rescaled coefficients at on-peak position
bgdcts! = tintercept! + coeff!(2) * sample(1).OnPeaks!(j%) + coeff!(3) * sample(1).OnPeaks!(j%) ^ 2
sample(1).BgdData!(i%, j%) = bgdcts!

```

### **Background Options**

See the Model Background button the Graph dialog. This button is accessible when a wavescan sample is displayed from the Plot! Window.

### **Same Side Off-Peaks**

Beware of placing same-side off-peaks too close together. A large extrapolation can cause a very large error in the off-peak background correction if the background standard deviation is large and you are not using the average, high only or low only background type. The program will print a warning if it feels that the same-side off-peak extrapolation is excessive. In cases where the same side off-peak extrapolation is excessive, the user may want to use the average of the off-peak counts. Of course, averaging the off-peak counts is really only applicable in situations where the background slope is zero.

### **MAN Backgrounds**

MAN (mean atomic number) background corrections are utilized for major and minor elements when the P/B is sufficiently high (accuracy limit is ~100-200 PPM in oxide and silicates) and can also be used for trace element analysis if a suitable “blank” standard is available.

The user can specify the standards used to calibrate the background fit for the polynomial MAN iteration of an analyzed sample. The MAN background correction method works because the primary cause of background (aside from trace contamination and peak over-lap interferences) is the average atomic number of the sample. Therefore a function can be fit to appropriate standard data and applied to the analysis of unknown samples. Since the atomic number depends on the composition and that is unknown to begin with, the MAN background is not applied until the analytical calculation iteration. Remember :

- MAN backgrounds can be used whenever the ratio of x-ray peak to background is high (>10:1), and this is usually the case with WDS microanalysis

- Trace element concentrations generally require the use of off-peak measurements but MAN background corrections can be utilized if a suitable matrix matched “blank” standard is available (e.g., most pure elements, oxides, many simple sulfides and silicates).

### **Multi-Point Backgrounds**

There are a number of options to specify for the multi-point background acquisition. First one must define the number of positions to acquire on each side of the peak (maximum = MAXMULTI%) and the spectrometer position of each background.

Note that the adjustment of multi-point backgrounds is usually performed graphically using the wavescan plot Model Background dialog.

The number of points to iterate to and the background fit type (Linear or Polynomial) can be specified before or after the background intensity data has been acquired. Note that the sum of all multi-point intensities is automatically calculated and saved to the normal off-peak intensity arrays so that normal off-peak background calculations can also be performed.

### **Spectrometer Number and Analyzing Crystal**

The spectrometer used to analyze the element should be specified based on the crystal arrangement of the microprobe. Enter a number or use the drop down menu option. This automatically loads a default crystal type, on-peak and off-peak positions that may be modified.

### **Interferences**

The High and Low Off-Peak Interference buttons can be used when both the potential interfering and interfered elements are already present in the sample setup to determine the approximate magnitude of the off-peak interferences. This calculation is based on a Gaussian peak shape and various crystal defaults and is intended as a guideline only for off-peak position entry.

**The program assumes 100% of the interfering element and 0.1% of the interfered element in each binary pair calculation. For example, if the percent interference is calculated as 10%, then that indicates that instead of measuring 0.1 wt. % of the element, it is likely that a concentration of 10% greater than 0.1 wt. % or 0.11 wt. % will be measured. Note that since this is only a nominal calculation, it should only be used as an indicator of possible problematic analytical situations.**

### **PHA**

The PHA parameter settings (baseline, window, inte/diff (integral or differential) mode, gain, bias, and deadtime) can be specified here. Defaults are loaded from the configuration files and can be modified at any time.

**Note that if the UseEmpiricalPHADefaults flag is set in the [Software] section of the PROBEWIN.INI file, then the PHA defaults loaded, are actually calculated from coefficients stored in the EMPPHA.DAT file instead being loaded from the SCALERS.DAT file.**

The PHA parameters entered here will be used to configure the microprobe hardware, if supported by the interface type during the acquisition. The PHA settings for the current sample (if it contains no data) can also be modified from the PHA button in the Acquire! window. The current PHA settings from the microprobe can also be read (if supported by the hardware and specified in the PROBEWIN.INI file) by clicking the Get PHA button.

### **Detector Parameters**

If the proper microprobe interface support is present for detector control, the detector slit size, slit position and mode (usually FPC or SPC) may also be specified for automatic control during the analysis. If the microprobe interface does not support automated detector parameter control, these fields can be used simply to document the parameters. See the documentation on the DETECTORS.DAT file in the Configuration Files section above.

### **Integrated Intensity Options**

For standard and unknown samples, if the element is acquired on a tunable spectrometer, the program offers an option to acquire the integrated intensity over the peak instead of simply the peak intensity. If this option is checked, the program will start scanning at the low or high off-peak limit using the Initial Step Size specified and scan to the other off-peak limit.

The actual step size is modified based on the acquired intensity so that as the intensity increases, the step size decreases to the Minimum Step Size specified. This improves statistics where it is needed the most. The default step size is 1/80<sup>th</sup> of the off-peak range and the default minimum step size is 1/200<sup>th</sup> of the off-peak range.

The program accumulates the intensities over the off-peak range and this is stored as the on-peak data. The off-peak intensities are also measured normally and the integrated background is calculated based on the background type selected and the off-peak intensities. The integrated peak intensity itself is calculated using a cubic spline fit to the data. All off-peak options may be used with the integrated intensity option. The reported integrated intensities are in units of counts per second times spectrometer units divided by the spectrometer unit range (cps per second per spectrometer unit).

If an element is acquired using the integrated intensity option, the standard intensities for that element must also be acquired using the integrated option and preferably using the same off-peak range. See the Run | Display Integrated Intensities menu for a graphical display of the integrated intensity data and cubic spline fitting.

### **Variable Step Wavescan Options**

For wavescan samples, the user may specify the use of variable wavescan steps. In this mode the program will acquire wavescan samples using a step size that is inversely proportional to the intensity using the following expression:

$$S_{size} = \sqrt{\frac{I_B}{I}} \cdot S_{initial}$$

where:  $I_B$  is the background count rate  
 $I$  is the current count rate  
 $S_{initial}$  is the initial step size

From this we can see that the wavescan will begin with the initial step size and as the intensity increases (the peak), the step size will decrease according to the equation until the minimum step size specified is reached. This option may be useful in cases where improved accuracy is necessary on the peak maximum.

A new option "Use Inverted Intensity Steps" allows the user to perform exactly the opposite operation. That specifies that the minimum step size will be used on the background intensities while the step size is increased for higher peak intensities. This is useful for characterizing the background regions without spending too much time on the peak regions..

### **Specified Area Peak Factors (fixed composition APF)**

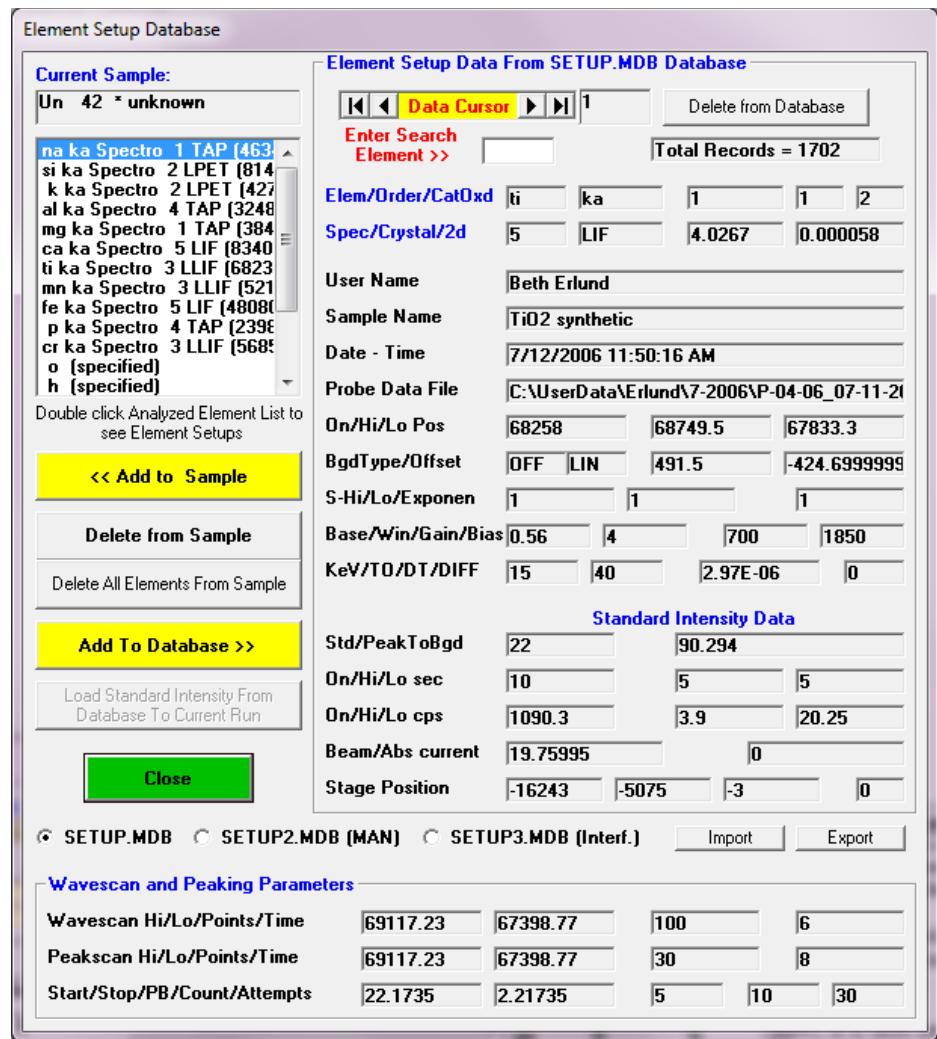
As an alternative to the "calculated" Area Peak Factors, Probe for EPMA offers the use of "fixed" composition Area Peak Factors. These are single values that are applied to the element intensity on an emitter basis (as opposed to calculated based on the actual absorber concentrations in the matrix).

These factors may be useful in certain situations where the compositional range is limited and binary Area Peak Factors are not available for some or all elements in the matrix (e.g., Na, K or Ca in an oxygen matrix). This option may be turned off or on in the Analysis Option dialog under the Analytical menu.

### **Load Element Setup**

This button opens the element setup database dialog. Here the user can add element setups to the new sample or save element setups to the setup database. Generally, one usually saves standard sample element setups for elements that are

assigned to that standard to the setup database. Later, one can retrieve these calibrated setups for use in creating new sample setups for acquisition in another run.

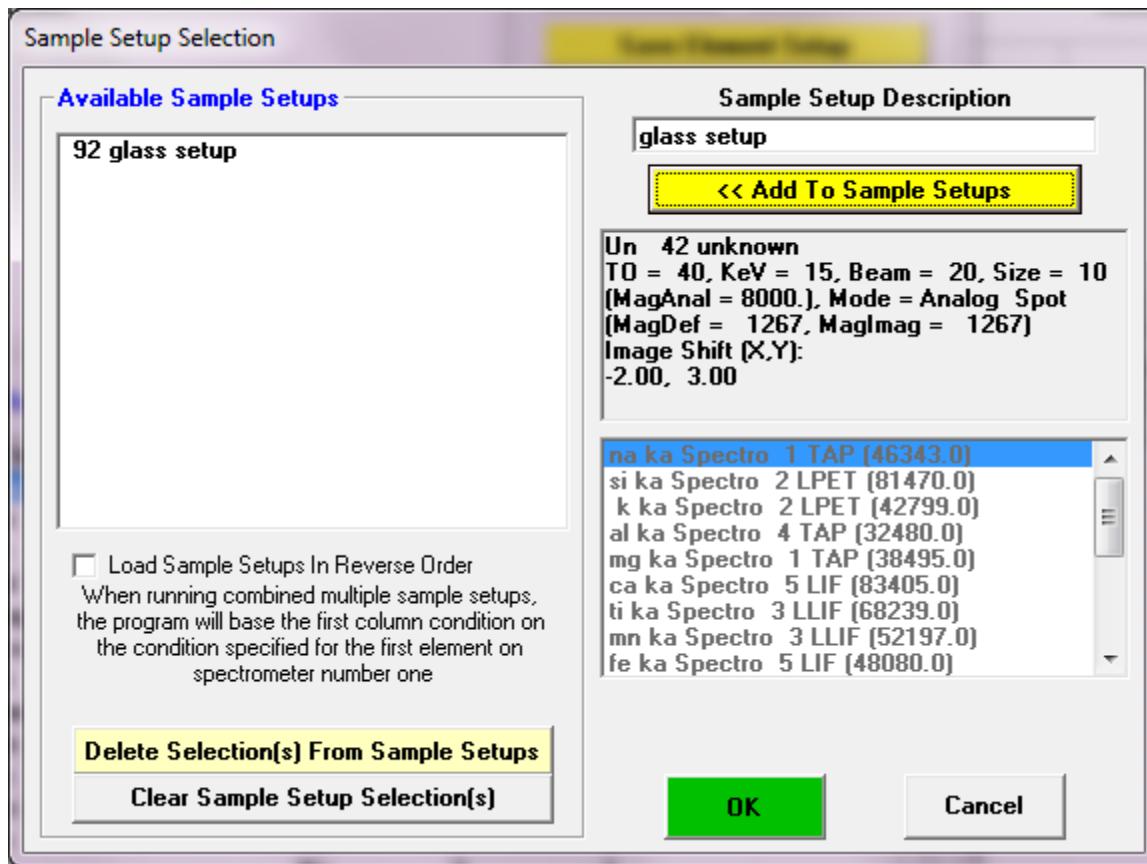


The element setup database can be browsed using the data cursor in the upper right of the window, or all occurrences of a specific element can be browsed by entering the element symbol in the Search Element field. To return the browser to display all elements, simply delete the element symbol from the Search Element field.

The browser displays element setups in the order that they were saved to the setup database. To go to the most recent setup, click the extreme right end of the data cursor. The number of element setups currently in the database is shown also.

### **Load Sample Setup**

To save or load sample setups to or from the current run, click the Load Sample Setup button. This button allows the user to load sample setups previously saved **within the current run** for later use in acquisition of new samples.



For example, it might be useful to create several sample setups within a run, if it is known that several different phases will be analyzed, each of which are significantly different in their elemental compositions. In other words, it is possible to create one sample setup containing the elements Fe, Ni and Cr as the analyzed elements and another sample setup containing the elements Al, Mg, and Cu as the analyzed elements, all within the same run.

These setups can be referenced for use with the automation actions for tremendous flexibility during the automated acquisition of different phases within a single run by using the Setups button in the Automate! window during the digitization of position samples.

However, if the sample pointed to by the sample setup contains any data, the element setup cannot be changed later on. For this reason it might be best to only reference samples that do not contain any data lines.

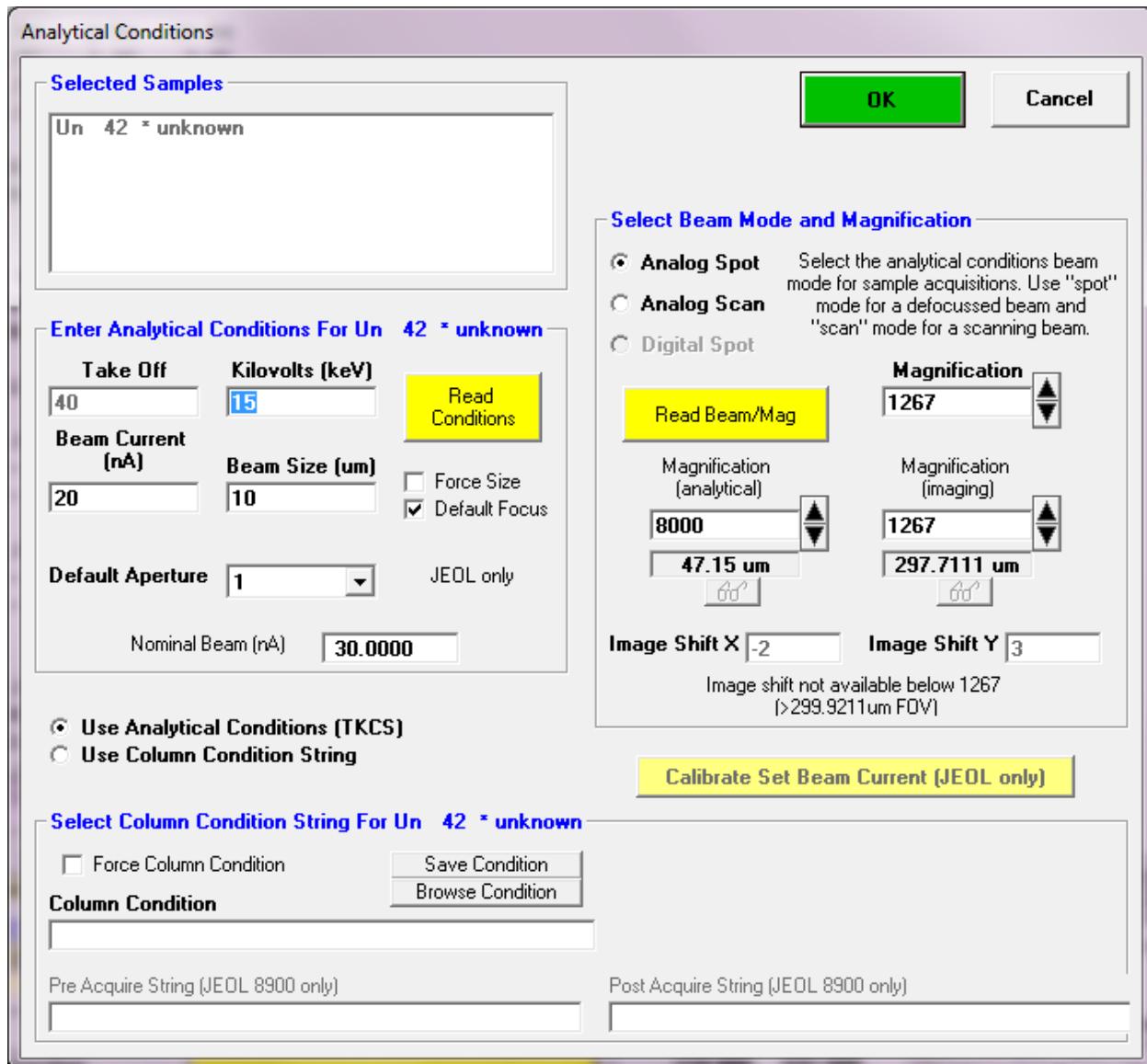
Note that the sample setup is actually a pointer to the row number of the selected sample. Any changes in the selected sample parameters will be automatically included when the sample setup is selected later on. This row number may be viewed for all samples in the current run from the Run | List Sample Names menu.

Sample setups may be loaded (or saved for the current sample), if it does not contain any data, by clicking the Elements/Cations button from the Acquire! window and then clicking the Load Sample Setup button. A previously created sample can be saved as a sample setup by using the Elements/Cations button from the Analyze! window.

When saving a sample setups always enter a generic name to indicate the purpose of the sample setup. It may be best to name the sample setup something descriptive, such as, "olivine setup" or "volatile calibration setup" so that the correct sample setup may be easily remembered.

## Analytical Conditions

Clicking the Analytical Conditions button opens the analytical conditions dialog box. The operator can then edit the kilovolts, beam current, and beam size settings for the selected sample. In addition, if the hardware interface is supported, the user may specify a column condition string to indicate the desired analytical conditions of the instrument.



Note that there are three magnifications defined in Probe for EPMA: a default magnification which is used for normal operation of the instrument between acquisitions, an analytical magnification which is used to acquire standards, unknowns and wavescans and an imaging magnification which is used to acquire images or for use with beam deflection acquisitions. Allowable range of magnifications for different platforms is documented here:

Minimum magnification = 40  
Maximum magnification = 300000

' JEOL 8900/8200/8500/8x30 (InterfaceType = 2)

Minimum magnification = 63  
Maximum magnification = 200000

' SX100/SXFIVE (InterfaceType = 5)

If the hardware support for these parameters is not present, then all parameters (other than kilovolts) are simply available for documentation purposes only. Note also that if the instrument has the ability to check the current analytical conditions, then the program will make sure that the analytical condition have not changed since they were last set. If the analytical conditions have changed by an amount greater than that specified by the "OperatingVoltageTolerance" and "BeamCurrentTolerance" in the PROBEWIN.INI file [hardware] section, then the program will force the analytical or column condition to be reloaded.

### **Analytical Conditions versus Column Conditions**

In the first method, (using specified values for kilovolts, beam current and beam size) the user directly specified the analytical conditions. In the second method, a specified string is used to indicate a column condition that is stored internally in the instrument. In this latter case, because the actual value of the operating voltage (kilovolts) in the column condition string is ambiguous, the user should be sure that the single value fields are also correctly specified based on the column condition string specified.

For example, the SX100/SXFIVE supports the use of externally stored column conditions. Therefore, if the column condition option is selected, and the column condition is specified as "15 keV, 20 nA, 5 um.PCC" (indicating a column condition at 15 keV), then the user should also be sure that the kilovolts field is correctly specified as 15 keV. Failure to properly specify the kilovolts field when using column condition strings will cause the ZAF or Phi-Rho-Z matrix corrections to be inaccurate. The beam current field is also used in some cases to check for beam drift.

For accurate documentation purposes it is also preferred that the beam current and beam size fields are also properly specified when using column condition strings. If, as in the above example, the "15 keV, 20 nA, 5 um.PCC" column condition file also includes a beam current of 20 nA and a beam size of 5 um, then those values should also be specified in the single values fields even though are not actually used in the acquisition procedure. This maintains the accuracy of the documented sample conditions.

Note that because samples at different operating voltages can be acquired in a single run, standards at the corresponding voltages must also be acquired for quantitative calculations.

### **Force Size or Force Column Conditions**

The Force Size option (when checked) specifies that the program should send the size command to the instrument for each acquisition. This is useful because with most instruments it is not possible to determine the current beam size. Therefore, if the user has manually changed the beam size then if the box is checked, the program will ensure a consistent beam size for acquisition. This option only applies to condition be set using the "Use Analytical Conditions" option (keV, beam current, beam size).

The Force Column Condition box (when checked) specifies that the program should send the column condition command for each acquisition. This is useful since the program is usually unable to determine the current column condition with most instruments. Therefore if the user has changed any column conditions, then the program can ensure that a consistent set of column conditions are loaded for each acquisition. This option only applies to the "Use Column Conditions" option.

### **Pre and Post Acquire Strings**

To allow the user to override analytical conditions or to customize the acquisition procedure the user can define pre acquire and post acquire strings. These strings are only applicable to the JEOL 8900 instruments. The commands are those commands allowed by the instrument (see vendor documentation).

For example, PB OFF for JEOL 8900. **Multiple commands may be sent to the interface by separating the command with commas. For example “PB OFF, LAMP OFF” for the JEOL 8900.**

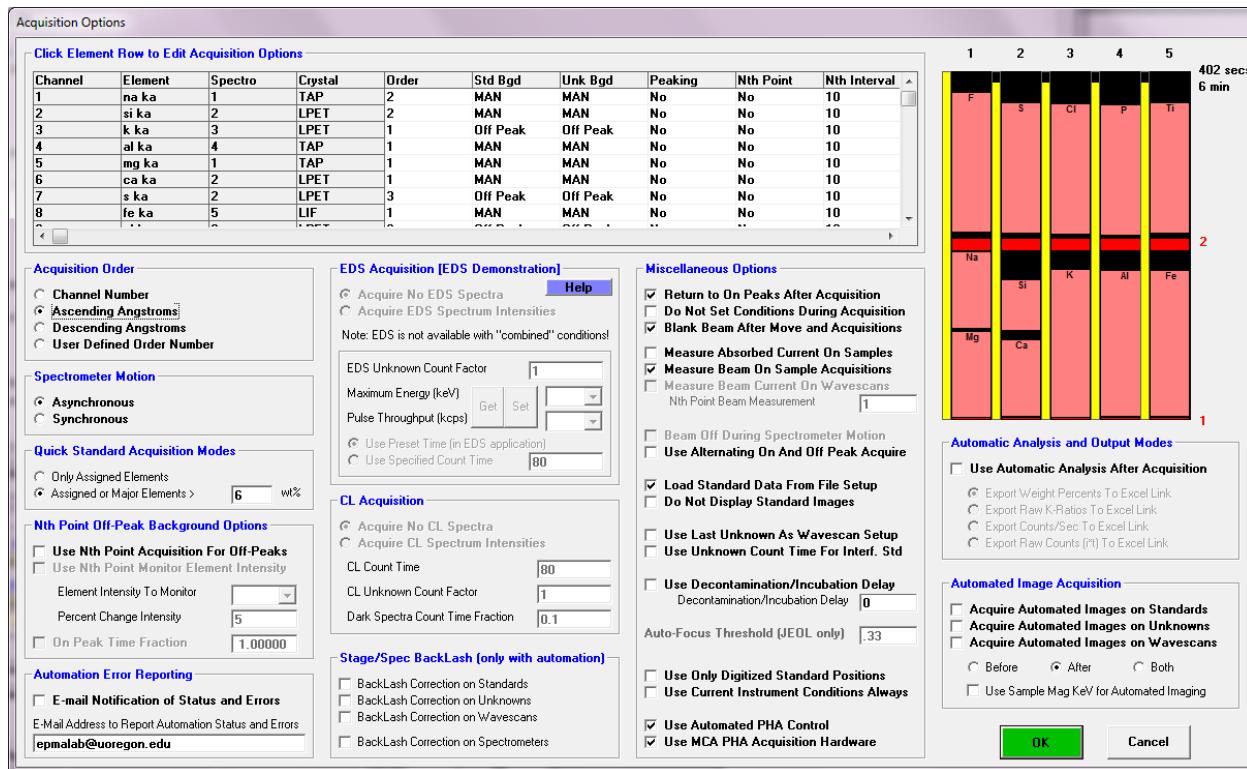
The pre acquire string is sent to the interface after all analytical conditions, magnification and beam mode commands have been sent and just prior to the removal of the faraday cup. The post-acquire string is sent after the acquisition is completed.

## Faraday Stage

A new feature is support for a specimen based faraday cup that is specified by stage coordinates. See the [Faraday] section of the PROBEWIN.INI file documentation for information on how to specify the default location of the stage faraday cup. The Faraday Stage button will bring up a dialog box that allows the user to update the faraday cup stage location in the program and make a manual measurement. During standard or unknown acquisitions, the software will move to the faraday stage location, make the measurement and then return the original stage location for the next analysis.

## Combined Conditions

The Combined Conditions dialog allows the user to specify individual analytical conditions (kilovolts, beam current and beam size), if supported by the hardware interface, for each element in the current sample separately. This allows for complete flexibility in the sample setup.



For example, the user may want to run the major elements first at a normal beam current and then run the minor and trace elements at an increased operating voltage and beam current.

The user can also change the acquisition order of elements (whether the sample contains combined conditions or not). Simply select the element and use the up/down spin button to move the selected element up the acquisition sequence or down. This feature is especially useful when sorting elements by combined conditions for most efficient acquisition.

## Locate

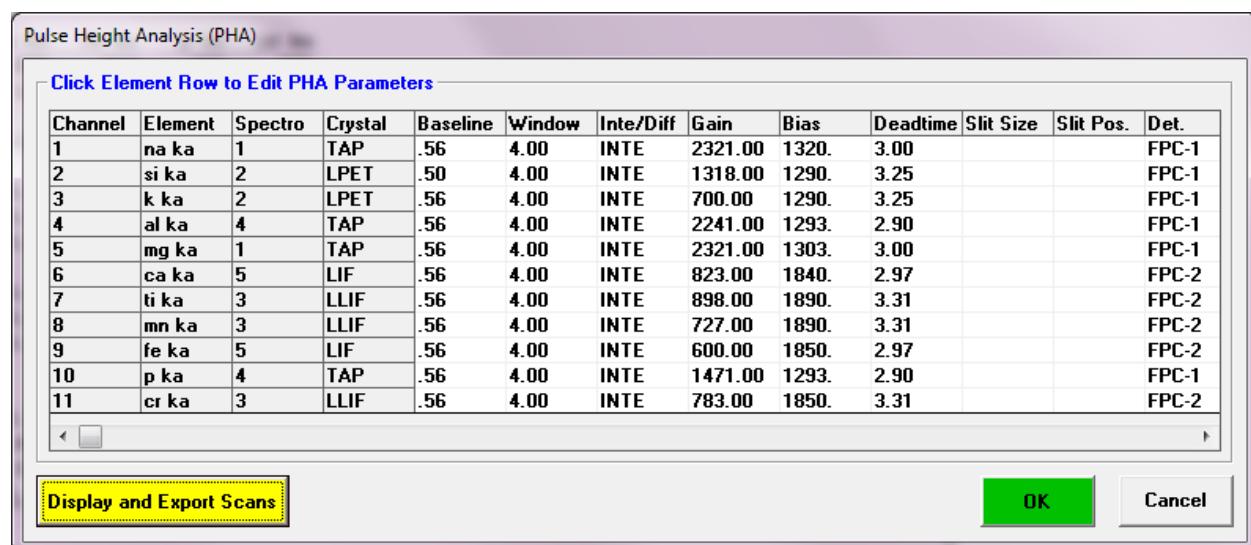
Clicking the Locate button opens the locate sample positions dialog box.

All acquired sample positions can be accessed from this dialog. Double-clicking on a sample entry displays the stage coordinates for each line acquired. To move to a sample position, highlight a position sample and click Go. This will move the stage to the first undisable stage coordinate of the sample selected. If a specific line coordinate is selected from the position grid, then clicking Go will move to that specific line coordinate (disabled or not).

By clicking the Copy Position Coordinates to Clipboard, the sample position coordinates shown will be copied to the Windows clipboard for pasting into another application if desired.

## PHA

Clicking the PHA (pulse height analysis) button opens the PHA parameters dialog box. Here all currently analyzed elements are listed detailing their respective baseline, window, inte/diff (integral or differential) mode, gain, bias, deadtime factors. To edit any of these values, simply click the element row to be edited and the element PHA parameters window will appear.



If the proper microprobe interface support is present for detector control, the detector slit size, slit position and mode (usually FPC or SPC) may also be specified for automatic control during the analysis. See the documentation on the DETECTORS.DAT file in the Configuration Files section above.

The PHA parameters dialog provides buttons to both set (write) PHA parameters to the microprobe and get (read) the PHA parameters from the microprobe (if the necessary hardware interface is available).

Note that if a gain and bias hardware interface is available, and the bias (detector high voltage) values on a given spectrometer are different for multiple elements, the acquisition will pause for two seconds before proceeding with the acquisition. For this reason the program will warn the user if the bias values are different between elements on a single spectrometer.

In addition a graphical PHA, Bias and Gain scan distribution may be acquired, based on the PHA count time and intervals specified on a single spectrometers at a time (for acquisition of all spectrometers simultaneously see the PHA window in the StartWin program). For the PHA scans, the program will measure the x-ray intensity as the baseline is varied from its current value up to the maximum baseline value with a window width based on the differential between the baseline and window and the number of points to be acquired.

For the Bias and Gain scans the baseline and window values are set to the Scan Baseline and Scan Window values while the bias or gain is scanned between the low and high range values. For bias scans the gain is set to the values displayed in the normal gain fields and for gain scans the bias is set to the value displayed in the normal bias fields.

Note that since JEOL instruments are limited to specific gain values, the gain values can only be modified using the spin buttons and the gain scanning fields and button are disabled.

The PHA settings can be adjusted using a fixed bias or a fixed gain. Normally JEOL users fix the gain (since they are limited to specific gain values) and scan the bias to find the proper adjustment. Cameca users usually fix the bias to a

value above the plateau level for stable response (lower energy x-rays require more bias, see below for more details) and scan the gain to determine the proper values.

Setting the bias above the "plateau" will provide the most stable count intensities since any small changes in the bias will not cause a change in the detector count rate. In this case the gain of the PHA amplifier is adjusted to place the PHA distribution is the approximate center of the amplifier range. If the argon escape peak is close to the signal peak, the gain should be adjusted so that the escape peak is either completely included or completely excluded from the PHA distribution.

## Count Times

Clicking the Count Times button opens the count times dialog box. This dialog also allows the user to modify the beam current integration time and the nominal beam current.

**Count Times**

**Click Element Row to Edit Count Times**

| Channel | Element | Spectro | Crystal | On-Peak | Hi-Peak | Lo-Peak | MaxCoun | Factor | Wave | Peak | Quick |
|---------|---------|---------|---------|---------|---------|---------|---------|--------|------|------|-------|
| 1       | na ka   | 1       | TAP     | 20.00   | 5.00    | 5.00    | 1000000 | 4.00   | 6.00 | 8.00 | 2.00  |
| 2       | si ka   | 2       | LPET    | 15.00   | 5.00    | 5.00    | 1000000 | 2.00   | 6.00 | 8.00 | 2.00  |
| 3       | k ka    | 2       | LPET    | 20.00   | 5.00    | 5.00    | 1000000 | 2.00   | 6.00 | 8.00 | 2.00  |
| 4       | al ka   | 4       | TAP     | 20.00   | 5.00    | 5.00    | 1000000 | 2.00   | 6.00 | 8.00 | 2.00  |
| 5       | mg ka   | 1       | TAP     | 30.00   | 5.00    | 5.00    | 1000000 | 2.00   | 6.00 | 8.00 | 2.00  |
| 6       | ca ka   | 5       | LIF     | 40.00   | 5.00    | 5.00    | 1000000 | 2.00   | 6.00 | 8.00 | 2.00  |
| 7       | ti ka   | 3       | LLIF    | 10.00   | 5.00    | 5.00    | 1000000 | 2.00   | 6.00 | 8.00 | 2.00  |
| 8       | mn ka   | 3       | LLIF    | 10.00   | 5.00    | 5.00    | 1000000 | 2.00   | 6.00 | 8.00 | 2.00  |
| 9       | fe ka   | 5       | LIF     | 30.00   | 5.00    | 5.00    | 1000000 | 2.00   | 6.00 | 8.00 | 2.00  |
| 10      | p ka    | 4       | TAP     | 20.00   | 5.00    | 5.00    | 1000000 | 3.00   | 6.00 | 8.00 | 2.00  |
| 11      | cr ka   | 3       | LLIF    | 15.00   | 5.00    | 5.00    | 1000000 | 2.00   | 3.00 | 8.00 | 2.00  |

**Beam Averages**

**Nominal Beam (nA)**

Change the Nominal Beam to modify the normalization constant used for the x-ray intensity display. For example, enter 1 (nA) for cps/nA intensity display.

**Return To On-Peak Time**

**Crystal Flip Time**

**Set Column (TKCS) Time**

171 secs  
2 min

Calculated Spectrometer Motion and Acquisition Time

0 secs

**OK** **Cancel** **Help** **Measure Nominal Beam**

This dialog displays the current analyzed elements and the counting time for on-peak, off-peak, wavescan, peaking and quick scans. Each element may utilize a different count time for each on and off-peak measurement.

## MultiPoint Background Counting Times

Note that the MultiPoint background count times are based on the corresponding Hi and Lo Off Peak Times divided by two, although the actual off-peak statistics will vary depending on how many "Iterate Points" are specified for the multi-point background fitting.

For example, let's suppose that an element is specified to use 200 seconds on-peak counting time and 100 seconds high off-peak and 100 seconds low off-peak. In the case of an element at zero concentration the total integration time on peak and off-peak are normally equal (200 seconds on-peaks equals 200 seconds off-peak) and therefore statistically

appropriate (obviously for element concentrations above zero the off-peak count time can be decreased relative to the on-peak counting time).

If the user switches to the MultiPoint background method and the number of points to acquire is say, 4 , then the software will calculate the off-peak acquisition time for each background as  $100/2 = 50$  seconds each, which yields 400 seconds for the four MultiPoint background measurements on both sides of the peak.

However, if the “Iterate Points” parameter is set to two (for each off-peak side), the MultiPoint background calculation will throw out 2 of those background measurements, so only 2 measurements on each side of the peak will remain in the calculated background. Hence, there will be  $4 * 50$  seconds or 200 seconds on both sides of the peak background compared to 200 seconds on-peak so the statistics are self-consistent.

### **Wavescan Counting Times**

The wavescan count time is applied to wavescan samples, the peak scan count time is applied to the peaking pre-scan acquired before a spectrometer peak center is run (if that option was selected) and the quickscan count time is applied to wavelength quick scans. Note the following parameters apply to non-*ROM* based and *ROM* based quick scan parameters:

#### **Non-*ROM* scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

#### ***ROM* scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

#### ***ROM* Based Peaking Scan:**

- fine scan uses peaking time divided by 4 times 1.0
- coarse scan uses peaking time divided by 4 divided by 2.0
- fine scan uses peaking time divided by 4 times 1.5

The count times for a range of elements may be specified by clicking and dragging a range of elements in the element list. Any changes to the count times dialog will be applied to all selected element in this case.

### **Max Counts and Unknown Count Factor Parameters**

The Max Counts is used to specify a statistics based count time. By default, Max Counts is set to 100,000,000 (100 million total counts), so that it will normally not affect the acquisition. However, it may be desirable to specify a specific maximum count that is to be acquired for each element. The default max Counts may be set in the PROBEWIN.INI file in the [counting] section.

For example, the count time and max counts could be configured so that each element will count for 30 seconds or 10000 total counts (that is 1% precision), whichever comes first. The advantage of this method is that on samples with high count rate elements, the actual time required to acquire data is shortened, while low count rate elements are counted for a full 30 seconds. In this way also, the statistical significance of all element count rates with the same Max Counts is more comparable.

The Unknown Count Time Factor is also displayed, which is actually used for trace element analysis in two different ways. The first method is as a factor applied to the default count time for acquiring unknown sample elements relative to the count time used for the standards. This is especially useful in trace element analysis when the standard only requires a 10 second count time but the unknown requires say, 100 seconds for the desired detection limit. By simply specifying an Unknown Count Time factor of 10, this can be achieved.

The other way in which the Unknown Count Time Factor can be used is when the Use Alternating On and Off Peak Acquisition flag is checked in the Acquisition Options window. In this case the total acquisition time is calculated as above but in addition, the program will automatically divide up the acquisition of on and off-peak count times into alternating intervals based on the Unknown Count Time Factor for each element.

For example, if the Use Alternating On and Off Peak Acquisition option is checked and the on peak count time for an element is 10 seconds and the off-peak count time is 5 seconds and the Unknown Count Time Factor is set to 3 then the program will first acquire 10 seconds on peak followed by 5 seconds on each off-peak, in three separate repetitions. The counts are automatically totaled at the completion of all repetitions. This option is especially useful in situations where the sample measurement could be affected by extended acquisition times due to beam damage, carbon contamination or surface charging.

Note that in both instances, the Unknown Count Time Factor entered, can be an integer or real number. Also be aware that the option exists to combine intensities for duplicate elements from multiple spectrometers. See Aggregate Intensity option.

### **Beam Current Measurement and Beam Drift**

The nominal beam current is used as a scaling factor for the beam drift correction. Any non-zero value will suffice, however to have the count rates reported close to the actual count rates, a nominal beam current should be used that is similar to the actual beam current used during the acquisition. That is, the counts displayed in the log and Analyze! Window is the beam drift corrected intensities (normalized to the nominal beam current in the Count Times dialog).

Microprobes which use a faraday cup (JEOL and Cameca) for the beam current measurement, require an extremely stable and regulated beam current since the beam current measurement occurs after the x-ray count acquisition. The benefit of the faraday cup beam current measurement method is that the electron optics geometry (i.e., sample current) can be changed during the quantitative run without affecting the beam drift calibration.

The beam drift correction calculation is shown here :

$$I_C = I_U \frac{B_N \frac{T_U}{T_N}}{B_U}$$

Where :

- $I_C$  is the beam drift corrected unknown intensity
- $I_U$  is the uncorrected unknown intensity
- $B_N$  is the nominal beam current
- $T_U$  is the count time for the unknown intensity
- $T_N$  is the count time for the nominal beam current measurement
- $B_U$  is the beam current for the unknown intensity

If you do not want a beam drift correction (it is recommended to always use a beam drift correction for the most quantitative work), one can either enter a nominal beam current of zero or disable the beam drift correction from the Analytical | Analysis Options menu.

### **Off-Peak Statistics**

The Off-Peak Statistics button is used to calculate a statistically equivalent off-peak counting time for the background position integration measurement. The parameters required for the calculation are the on-peak count time, the on-peak count rate (cps) and the off-peak count rate (cps). The calculated off-peak count time is divided between the two off-peak positions when the user clicks the Apply button. The calculation is from Scott and Love, 1983.

$$t_B = t_P \sqrt{\frac{I_B}{I_P}}$$

where:  $t_B$  = calculated background count time

$t_P$  = on-peak count time

$I_B$  = off-peak intensity

$I_P$  = on-peak intensity

## Standard Assignments

The standard assignments dialog is used to select the primary standard for the calibration of the quantitative analysis. Each analyzed element must have a standard assigned to it. This dialog is also used to specify the quantitative correction of on-peak interferences and the Time Dependent Intensity (TDI) element correction.

**Standard and Interference Assignments**

| Selected Samples       |                        |                        |                        |                        |  |  |
|------------------------|------------------------|------------------------|------------------------|------------------------|--|--|
| Un 35 MB2 IW2 C4-ext-4 | Un 36 MAM IW2 C3-ext-1 | Un 37 MAM IW2 C3-ext-2 | Un 38 MAM IW2 C3-ext-3 | Un 39 MB2 IW2 C3-ext-1 |  |  |

|   |   |   |   |   |   |
|---|---|---|---|---|---|
| 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|

**Click Element Row to Edit Standard/Interference/Time Dependent Intensity (TDI) Assignments**

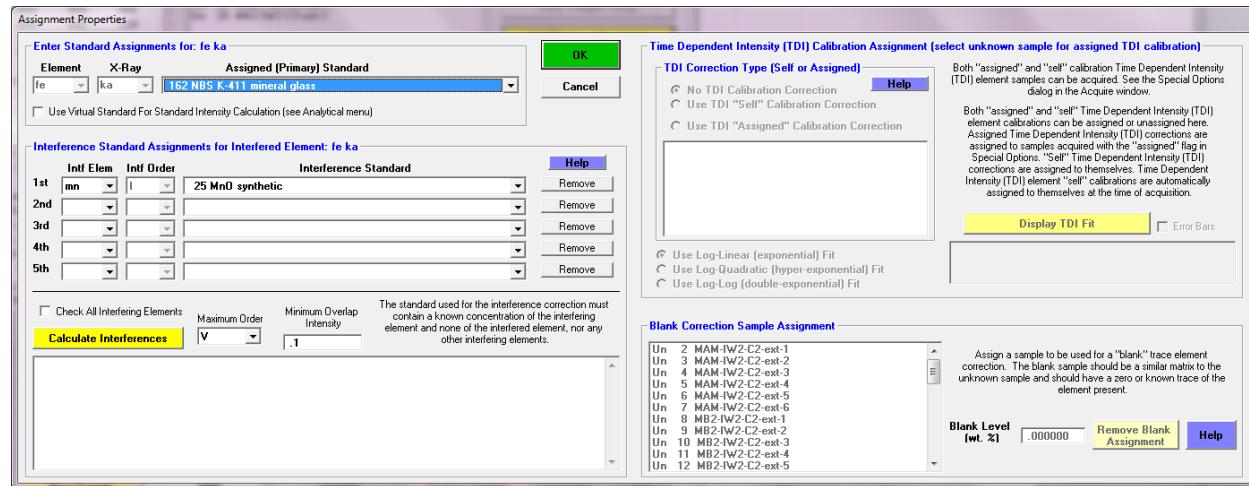
| Channel | Element | X-Ray | Analyzed | Standard | Interf-Ele | Interf-Std |
|---------|---------|-------|----------|----------|------------|------------|
| 1       | na      | ka    | Yes      | 336      | ....       | 0,0,0,0    |
| 2       | si      | ka    | Yes      | 162      | ....       | 0,0,0,0    |
| 3       | k       | ka    | Yes      | 374      | ....       | 0,0,0,0    |
| 4       | al      | ka    | Yes      | 336      | ....       | 0,0,0,0    |
| 5       | mg      | ka    | Yes      | 162      | ca....     | 285,0,0,0  |
| 6       | ca      | ka    | Yes      | 162      | ....       | 0,0,0,0    |
| 7       | ti      | ka    | Yes      | 22       | ....       | 0,0,0,0    |
| 8       | mn      | ka    | Yes      | 25       | ....       | 0,0,0,0    |
| 9       | fe      | ka    | Yes      | 162      | mn....     | 25,0,0,0   |
| 10      | p       | ka    | Yes      | 285      | ....       | 0,0,0,0    |
| 11      | cr      | ka    | Yes      | 396      | ....       | 0,0,0,0    |
| 12      | o       |       | No       | 0        | ....       | 0,0,0,0    |
| 13      | h       |       | No       | 0        | ....       | 0,0,0,0    |
| 14      | ba      |       | No       | 0        | ....       | 0,0,0,0    |
| ..      | ..      | ..    | ..       | ..       | ..         | ..         |

Remember that although the program will automatically load the standard assignments based on the last unknown sample, the standard assignments may be assigned at any time on a sample by sample basis, e.g. for post-processing of data.

The program will load a default set of standard assignments the first time this dialog is opened. These default standard assignments are simply based on the standard containing the highest concentration of each element. It will sometimes be necessary to modify these defaults standard assignments depending on the analytical situation.

Generally when assigning standards for use in the analytical calibration, it is best to use primary standards which contain a major concentration of the element. Standards not assigned but also containing the element are secondary standards and are valuable for checking the quality of the calibration. This can be seen by examining the "%VAR" line in the analysis printout, if they have been acquired as standards, not as unknowns.

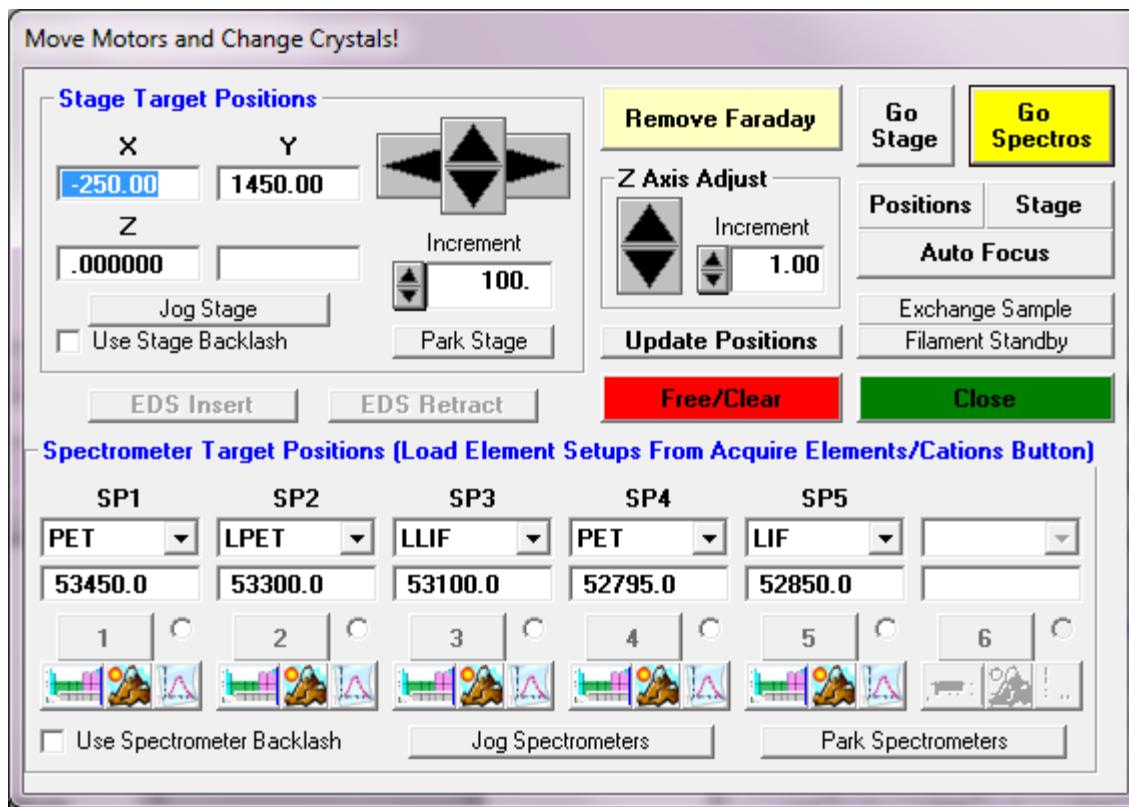
Although standards can be reassigned at any later time, the user should be aware that the use of "quick" standards may restrict one's ability to reassign standards off line since the required data may not have been acquired. When running connected to the microprobe (on line) however, you can rerun the affected "quick" standards after changing the standard assignments to obtain the necessary calibration data.



Users may also create subsets of the same elements to acquire on several standards, permitting maximum flexibility. Say you have a sample with feldspars and pyroxenes, and your complete element list holds Na, Mg, Al, Si, K, Ca, Ti, Cr, Mn, and Fe, and you have several silicate standards: albite, microcline, anorthite, wollastonite, enstatite, forsterite, fayalite (as well as others for Ti, Cr and Mn). It would take too long to acquire all elements on all standards, but you don't want to run quick standards either, as you'd like to acquire several elements on multiple standards for maximum flexibility. After peaking all elements and checking pulse height settings, you would save an "all elements" sample setup. Then create a new unknown, feldspar, and delete the Mg, Ti, Cr, and Mn. Save this sample setup as "feldspar". Then create a new unknown, this time recalling the "all elements" sample setup, this time deleting the K and Cr, and saving this as "pyroxene". You could then later "tag" the appropriate standard with the appropriate list of elements, under Automate!.

## Move

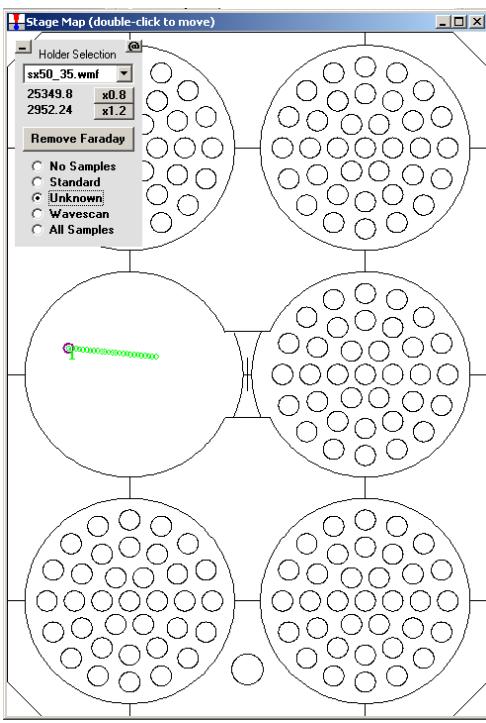
Clicking the Move button opens the move motors and change crystals dialog box. If crystal flipping is not supported by the interface hardware, then the crystal lists are disabled. The interface provides a quick and easy way to flip crystals, move the stage and spectrometers by clicking a single "Go" button.



The user can generally select crystals and element position using the Element and Xray selections or using the new Periodic Table dialog (see below) and then peak the elements and adjust the spectrometer PHA settings all from the Move dialog. Then using the Elements/Cations dialog from the Acquire! Window one can load the element setups into the current sample. If more elements are to analyzed than there are spectrometers, this process can be repeated as necessary.

## Stage

The stage button loads the Stage Bit Map window. This window allows the user to double click to move to any position on the stage using a graphical interface. Up to 12 different stage bit maps can be loaded as specified in the PROBEWIN.INI file. The file can be any photo, vector drawing or bitmap, but must be saved to the WMF (Windows Meta-File) format.



The standard, unknown and wavescan coordinates can be displayed on the stage bitmap. Also note the small “\_” button in the upper left which will minimize the Holder Selection frame as well as the small “@” just to the right which allows the user to easily calculate the exact stage bitmap calibration values which can then be edited into the PROBEWIN.INI file.

See below for more details.

### **Stage and Spectrometer Target Positions**

The operator can adjust the stage coordinates as well as the spectrometer motors positions and crystal positions.

**To manually flip a crystal when automated crystal flipping is not available, first drive to the crystal flip position (using the JOYWIN application if necessary) and manually flip the crystal. Then, after moving to a valid spectrometer position, exit the program and restart the application and confirm the new crystal positions in the Confirm dialog.**

The large four-way directional arrows can be used to increment the stage by the micron increment number listed in the cell below. The value of the micron increment can also be adjusted up or down using the spin controls or edited by hand. Note that the stage can also be incremented by using the keyboard cursor keys. Up and down arrow control the Y axis and left and right arrows control the X axis. Note that for the cursor keys to perform this function a text field in the Move! window must have the focus (be selected).

One can set stage, spectrometer and crystal targets by entering the positions manually or by selecting an element and x-ray symbol from the spectrometer target position lists. *Special note: if the "UseMultiplePeakCalibrationOffset" parameter is set in the PROBEWIN.INI file and the fit coefficients have been properly calibrated using the Peak/Scan Calibrate Parameters dialog in program StartWin, the program will load "corrected" spectrometer peak positions in the Move dialog when the user selects an element and x-ray line. See the Calibrate Peak Center help in the StartWin section for more details.*

This modeless window can be accessed using the Move button on either the Acquire! or Automate! windows. Note that during automated acquisitions this window will be automatically unloaded to prevent hardware access conflicts.

The Stage and Spectrometer backlash check boxes in the Move window are applied only to stage or spectrometer motion from the Move window. Exception: if the stage backlash option is unchecked in the Move window, no stage backlash will be applied during the automation actions. See the Acquisition Options dialog (accessed from the Acquire! window) to enable or disable stage and/or spectrometer backlash for sample acquisitions.

### ***Faraday In/Out or Beam Blanked/Unblanked***

The faraday cup on faraday equipped microprobes can be inserted or removed and the beam blank state can be toggled by the user using the Faraday In/Out or Beam Blanked/Unblanked button.

The insert and remove faraday delay times can be specified in the PROBEWIN.INI file. This may be useful to give the hardware sufficient time to complete the operation before proceeding.

An additional Faraday/Beam Blank button is provided in the Stage BitMap window used for graphical stage moving (Stage button).

### ***Update Positions***

This button when clicked, updates the Move dialog for the current stage and spectrometer positions for those interfaces that do not constantly poll the microprobe hardware. Normally, the Probe for EPMA TCP/IP interfaces access the hardware constantly to update the Move dialog, while other slower interfaces do not.

### ***Jog Stage***

Clicking this button causes the stage motors to jog by an amount specified in the PROBEWIN.INI file backlash factors. Use this button to remove any stage backlash that may be present when manually digitizing stage positions. The jog is based on the current stage positions for all stage motors.

Note that the stage backlash flag must be set (checked), in order to use the sample specific stage backlash flags as specified in the Acquisition Options windows for automated samples.

### ***Jog Spectrometers***

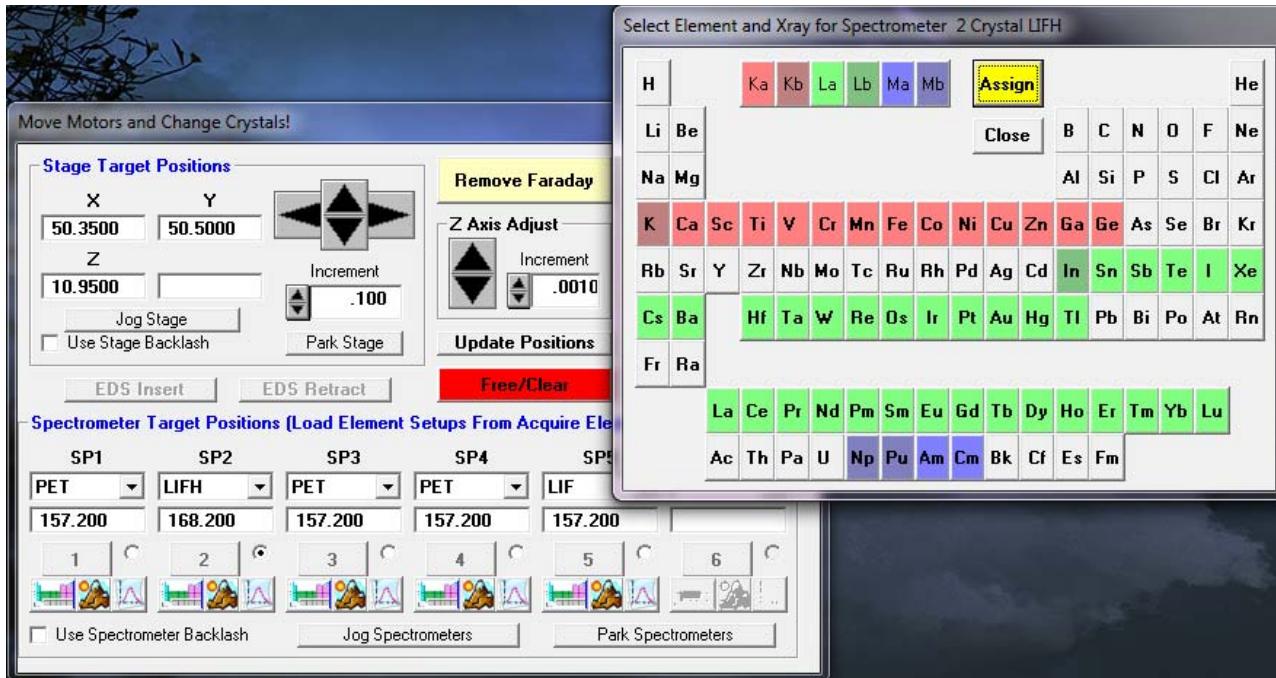
Clicking this button causes all spectrometers to jog by an amount specified in the PROBEWIN.INI file backlash factors. Use this button to remove any spectrometer backlash that may be present when manually adjusting the spectrometer positions. The jog is based on the current spectrometer positions for all spectrometer motors.

This flag is duplicated in the Acquisition Options window, which can be accessed by clicking the Acquisition Options button in the Acquire! window.

### ***Free/Clear***

This button stops and deactivates all current stage and spectrometer motor motion. It is equivalent to clicking the Cancel button on the Automation Status bar.

### ***Periodic/Peak and PHA buttons***



Three new buttons are available underneath the spectrometer crystal and position controls. These are the Periodic, Peak and PHA buttons.

The Periodic button allows the user to select available elements based on the current analyzing crystal for that specific spectrometer. The predicted spectrometer position and overvoltage (based on the current operating voltage) are displayed for evaluation and will be loaded upon clicking OK. The Peaking button will start a ROM based peak center procedure for the selected spectrometer. The peak center procedures can be performed in parallel. The third button is the PHA button and will display the Set PHA dialog allowing the user to manually or automatically (if available) adjust the PHA parameters for the selected spectrometer.

### **Positions**

Clicking this button opens the Position Database dialog box. This list of position samples is similar to that seen in the Automate! position list.

All currently digitized position sample stage coordinates, (standards, unknowns, and/or wavescans) for the position database can be displayed. Double-clicking on any entry will display the stage coordinates for that position sample.

One can move to any selected position sample by clicking the Go button on this window. The program will move to the first undisabled stage position for the selected sample. If a specific position coordinate is selected in the lower coordinate display grid, the program will move to that specific position.

The Re-Load will refresh the position sample list in case it was modified by another dialog.

The currently selected position sample coordinates can be updated using the Update button based on the current stage position.

The Edit button will allow the user to manually edit certain fields for the position samples including the position sample name fiducial set and setup number.

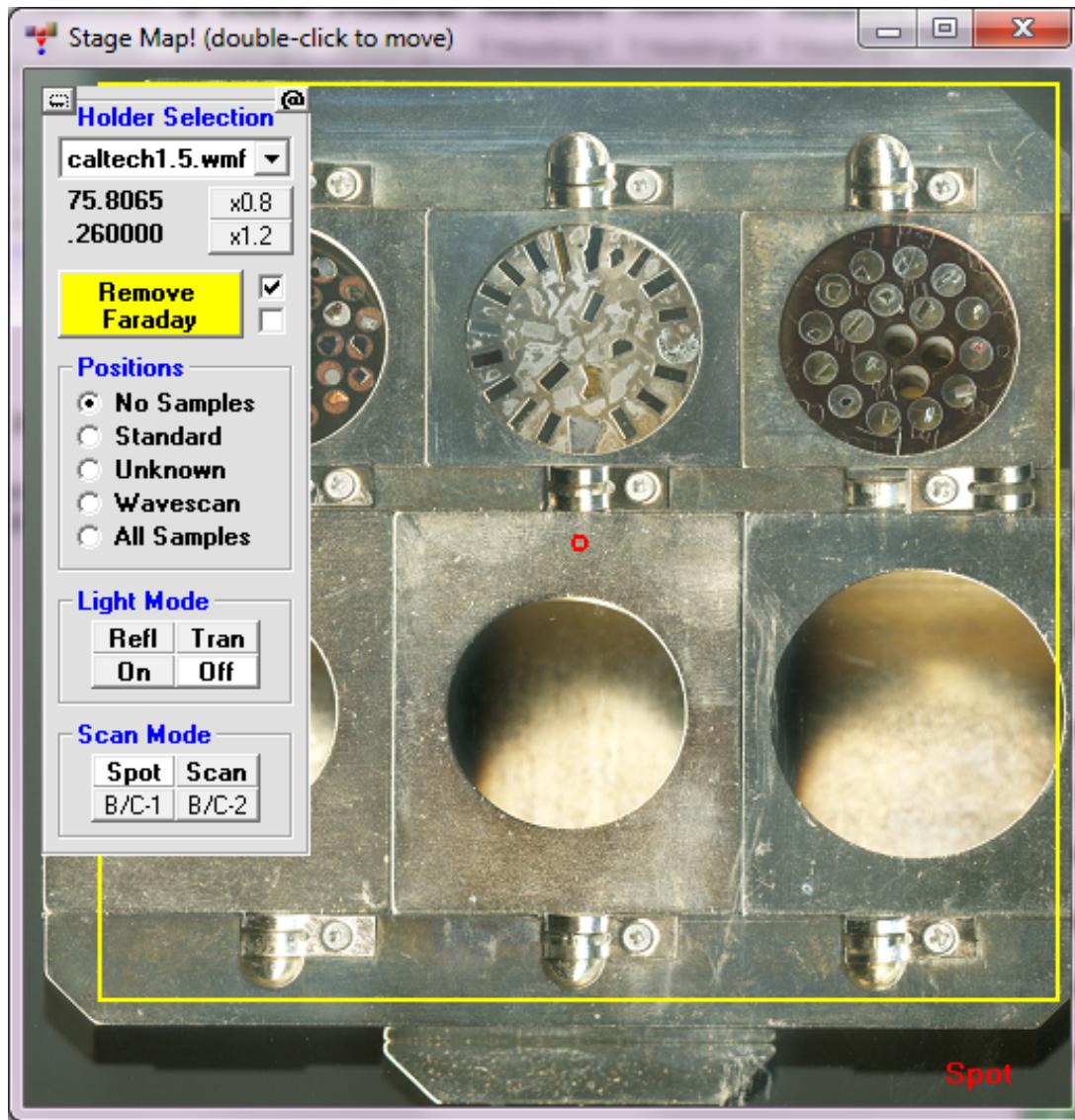
### **Stage**

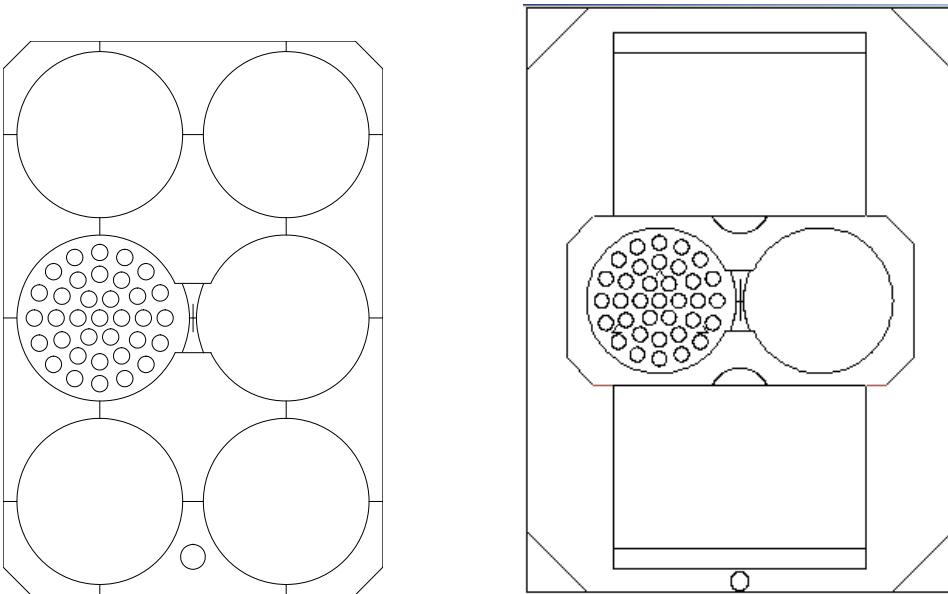
The Stage button will open a Stage Bit Map window to allow the user to move to a stage position by simply double-clicking on a specific area on an image of the stage. More than one image file (up to MAXBITMAP%) can be specified

in the PROBEWIN.INI file under the "Standards" section. To select another stage bit map simply select the file from the list box. The current stage position is indicated as a red circle.

The frame containing the stage bit map list box, cursor position and Faraday/Beam Blank button can be dragged to a different area of the window if the present position covers an area of interest. The minus button minimizes the stage bitmap selection and cursor position display. The "@" button loads a window that can be used to calibrate the currently displayed stage bit map. The calculated X and Y min and max values need to be entered into the PROBEWIN.INI file.

To re-size the Stage Bit Map window simply drag any corner of the window to the desired size and shape. To enable the Stage button or add additional Stage Bit Maps files to the program, see the [Standard] section in the PROBEWIN.INI file. Some sample Stage Bit Map files are shown here for the JEOL 8200/8500 and Cameca SX100/SXFive respectively:





### **Close**

Clicking this button will close the Move dialog box and return to the Acquire! window.

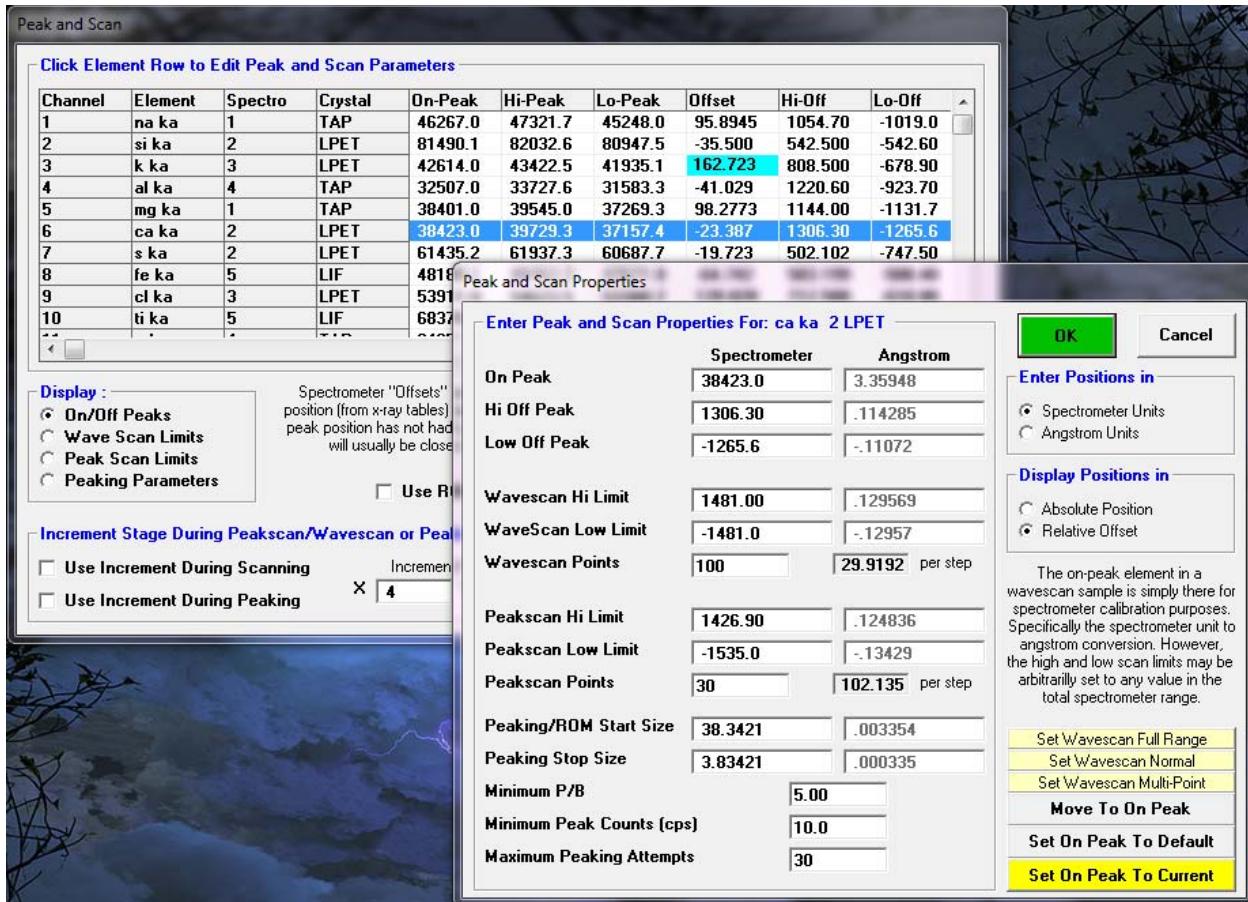
### **Go**

Clicking the Go button activates the motors to send all stage and/or spectrometers to their listed target positions. If the crystal positions were changed, the crystals will also be flipped before the spectrometers are driven to their final positions (if supported by the hardware interface).

Since the Go button is the default button in this window, typing <enter> from the keyboard will also cause the motors (and crystals) to move.

### **Peak/Scan Options**

Clicking the Peak/Scan Options button displays the spectrometer peak center and wavescan parameters for each element. All currently analyzed (tunable spectrometer) elements are displayed, along with their values for the current sample, for on and off-peaks, wavescan limits, peakscan limits, peaking parameters.



Note that the Peaking Start Size controls the start step size for the Increment Peaking and also the Parabolic Peaking distance for the side offsets for fitting the parabola to the peak centroid. This value should be small enough to avoid problems with asymmetrical peak profiles but large enough to ensure that there is at least one standard deviation difference in count intensities between the peak and the side offset positions. See the SCALERS.DAT file for more information.

Click an element row to edit any peak and scan parameter. Clicking any element row opens the peak and scan properties dialog box. Spectrometer positions may be entered in either spectrometer units or angstrom units and either in absolute position units or relative offsets.

The Move To On Peak button will actually move the current spectrometer to the on-peak position of the current element. The Update On Peak button will update the on-peak (and all other offset positions) to the current position of the spectrometer. This is useful when manually tuning a spectrometer position. The Move To On Peak button will also automatically set the PHA parameters if supported by the hardware.

Note that there is an option to have the stage position "bumped" during the wavescan acquisition. Because the spectrometers are usually operated in an asynchronous mode, the stage is incremented for the specified microns at each specified interval (0-60 seconds). For example, if the user has set the stage increment value to 5 microns and the stage increment interval to 10 seconds, then the program will increment the stage by 5 microns every 10 seconds, during the wavescan acquisition.

Note that the stage can also be incremented by using the keyboard cursor keys. Up and down arrow control the Y axis and left and right arrows control the X axis. Note that for the cursor keys to perform this function a text field in the Wavescan window must have the focus (be selected using the mouse).

## Spectrometer ROM scanning versus Step/Count Scanning

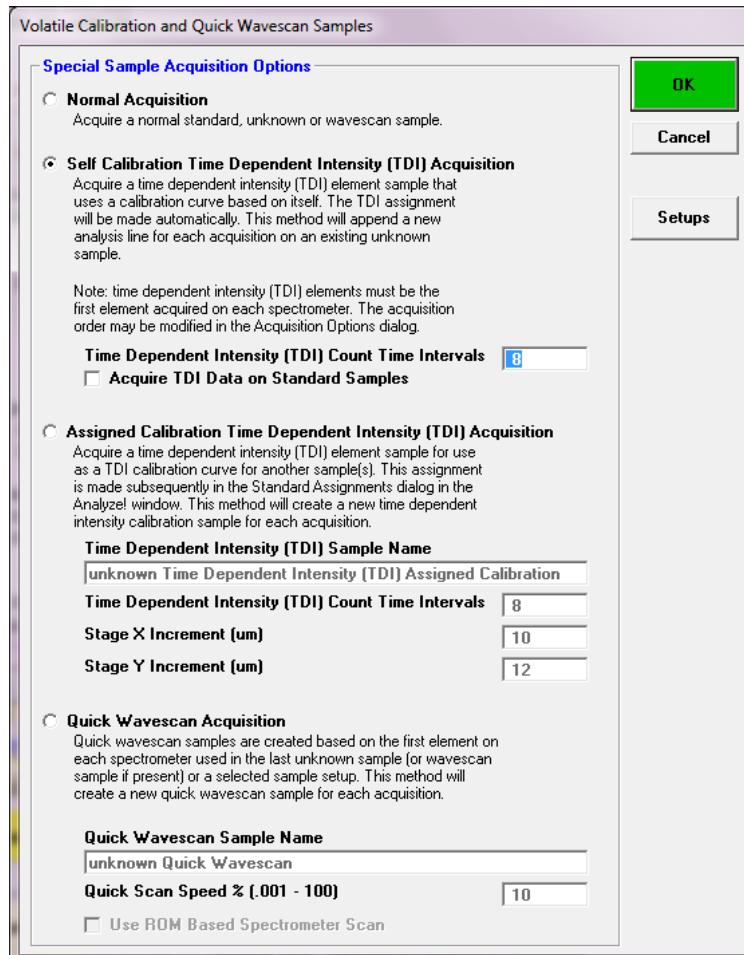
Note also the checkbox to utilize a "ROM" based spectrometer scan instead of the default step/count method for wavescans. The ROM scan option is only available on certain instruments, normally the JEOL 8900/8200/8500 and 8230 and 8530 and Cameca SX100/SXFive instruments. The difference is that a ROM spectrometer scan smoothly scrolls the spectrometer Bragg angle while counting x-rays as opposed to the normal wavescan which uses a defined spectrometer step size and count time interval method.

The ROM scan is more efficient at short count times due to low speed limitations, while the step/count method is more accurate because the spectrometer position is not "calculated" based on motor speed and also more sensitive because count time per step is not limited.

SX100 note: Be aware that because the Cameca ROM scan intensity digitization is limited to 18 bits of x-ray count data, very slow scans at high beam currents on pure samples may produce numerical intensity "wrapping" effects when the counters overflow (above 262144 counts) at the peak position intensity. The solution is to increase the scan speed, reduce the count time per point, decrease the beam current or use the step/count method for peakscan or wavescan acquisition.

## Special Options

Clicking the Special Options button opens the Time Dependent Intensity (TDI) Calibration and Quick Wavescan Samples dialog. There are two different methods that may be used in Probe for EPMA for the correction of Time Dependent Intensity (TDI) elements in unknown samples and another method for the acquisition of "quick" wavescan samples.



Time Dependent Intensity (TDI) element calibration corrections can only be applied to elements that are the first element acquired on a given spectrometer. The acquisition order of the elements may be modified in the Acquisition Options dialog. This option should be used for both elements "lost" during the acquisition (e.g. Na, K in glass) as well as elements "grown in" (e.g. Si and Al). Time Dependent Intensity (TDI) element fit types may be applied on an element by element basis in the Standard Assignments dialog from the Analyze! window.

"Quick" wavescan samples are spectrometer wavescans that automatically cover the entire range of each spectrometer available in the run.

### ***Self Calibration Time Dependent Intensity (TDI) Acquisition***

In the self calibration method, the software acquires the on-peak data for the Time Dependent Intensity (TDI) element during the normal sample acquisition process for unknown samples. Time Dependent Intensity (TDI) element fit types (linear and non-linear) may be applied on an element by element basis in the Standard Assignments dialog from the Analyze! window.

Note that this data is also displayed in the Run | Display Time Dependent Intensity (TDI) or Alternating On/Off Intensities menu.

Click the Self Calibration **Time Dependent Intensity (TDI)** Acquisition option so that the next sample acquisition will be acquired using the self calibration method. The number of count time intervals used is specified in the **Time Dependent Intensity (TDI)** Count Time Intervals text field. Up to 50 intervals may be used in a Time Dependent Intensity (TDI) acquisition.

For example, if the user enters 5 in the Time Dependent Intensity (TDI) Count Time Intervals text field, and the default on-peak count time is 10 seconds, then the software will automatically count five 2 seconds intervals for the on-peak elements that are first in spectrometer order. Off-peak acquisitions are not affected.

**Note that one can significantly reduce the time delay between when then beam is first unblanked and the x-ray counting is started by using the "Synchronous" option in the Acquisitions Option dialog.**

To view the Time Dependent Intensity (TDI) calibration data, click the Standard Assignments button in the Analyze! window and select the element to view. Click the Display Time Dependent Intensity (TDI) Fit to view the data for all lines in the sample. Note that the Time Dependent Intensity (TDI) calibration caption will indicate a self calibration correction. That is, the unknown number used for the calibration is itself.

### ***Assigned Calibration Time Dependent Intensity (TDI) Acquisition***

Another method for Time Dependent Intensity (TDI) element calibration corrections is the assigned calibration method. In this method an unknown sample is automatically acquired one element at a time by clicking the Assigned Calibration Time Dependent Intensity (TDI) Acquisition option to allow the Time Dependent Intensity (TDI) element effect to be calibrated precisely on a single sample. This sample will be started automatically using the sample name in the Time Dependent Intensity (TDI) Sample Name field the next time a sample acquisition is started.

The count time is adjusted based on the Time Dependent Intensity (TDI) Count Time Interval parameter. For example, if the user enters 5 in the Time Dependent Intensity (TDI) Count Time Intervals text field, and the default on-peak count time is 10 seconds, then the software will automatically count five 2 seconds intervals for the on-peak elements that are first in spectrometer order. Off-peak acquisitions are not affected.

The stage is automatically moved in X and Y for each element to ensure that a fresh sample spot is utilized for the Time Dependent Intensity (TDI) calibration.

The Time Dependent Intensity (TDI) element calibration can be assigned to any element in a sample or range of samples provided that it was acquired as the first element on that spectrometer. See the Analyze! window Standard Assignments button for the Time Dependent Intensity (TDI) element calibration display and assignment.

## **Setups**

Pressing the Setups button opens the sample setup window. Here an available sample setup can be selected and used as the basis for the assigned Time Dependent Intensity (TDI) calibration sample. This feature is intended for assigned calibration Time Dependent Intensity (TDI) corrections when it is desired to acquire a subset of the actual elements to which the Time Dependent Intensity (TDI) correction will be assigned.

### **Quick Wavescan Acquisition**

Clicking the Quick Wavescan Acquisition option will start a quick wavescan sample the next time a wavescan is started.

A sample name and scan speed percentage can be specified. The program will move each spectrometer currently being used to its extreme limit and continuously scan the spectrometer while acquiring count data at the same time. This is a very useful feature if an EDS detector is not available or WDS resolution is required.

The count time used for the quickscan is specified in the Count Times dialog. Note that the program uses the element configuration for the first acquired element (order=1) on each spectrometer in the sample to create the quickscan sample. Note the following parameters apply to non-*ROM* based and *ROM* based quick scan parameters:

#### **Non-*ROM* scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

#### ***ROM* scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

#### ***ROM* Based Peaking Scan:**

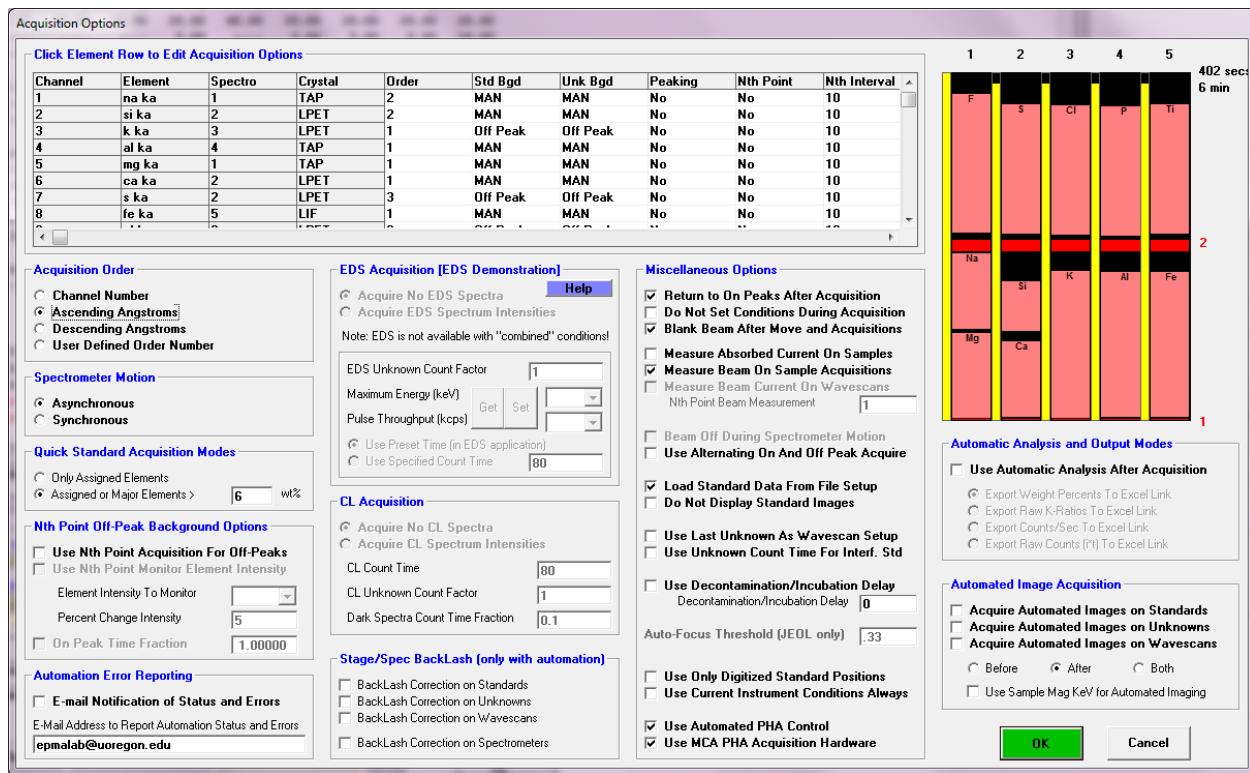
- fine scan uses peaking time divided by 4 times 1.0
- coarse scan uses peaking time divided by 4 divided by 2.0
- fine scan uses peaking time divided by 4 times 1.5

## **Setups**

Pressing the Setups button opens the sample setup window. Here an available sample setup can be selected and used as the basis for the quickscan.

## **Acquisition Options**

Clicking the Acquisition Options opens the Acquisition Options dialog box.



All elements analyzed in the current run are listed along with their order of analysis and background type (for both standard and unknown samples). The acquisition order for the elements on each spectrometer may be specified by channel number, ascending or descending angstroms or user-defined order number.

Asynchronous or synchronous spectrometer motion can be specified. In asynchronous mode each spectrometer moves and counts independently from the other spectrometers. This is the default mode for data acquisition. In synchronous mode the spectrometers move and count together and no spectrometer counts x-ray while another is moving (and visa versa).

**Note that one can significantly reduce the time delay between when the beam is first unblanked and the x-ray counting is started by using the “Synchronous” option in the Acquisitions Option dialog. This is especially useful for the Time Dependent Intensity (TDI) acquisition modes when the sample is extremely sensitive to intensity change during the first few seconds of beam exposure.**

Stage and spectrometer backlash flags for sample acquisitions can be set. Note that the sample specific stage backlash flag seen here are only used when performing automated sample confirmation or acquisition. These sample specific backlash flags will be ignored if the Stage Backlash option is not checked in the Move window before the automation is started.

**Note that the program will attempt to determine if a backlash is required based on the last direction of motion for each motor. If the backlash factor is positive and the last motion was from a higher to a lower position and the size of the motion was larger than the backlash size, then a backlash will not be performed on the next occurrence of the backlash adjustment.**

Other options include beam blanking after each acquisition, returning to on-peaks after acquisition, and beam current measurement modes. If an absorbed current interface is available, the measurement can be turned on or off here also.

The acquisition options for each element can be edited by clicking the desired element row. The spectrometer order number (if user defined was selected) and background type for both standards and unknowns can be specified. If entering

the spectrometer order number as user defined, be sure to set the acquisition order for all elements on that spectrometer before exiting the Acquisition Options dialog.

The Use Automatic PHA Control option only applies to those interfaces that support computer control of the baseline, window, gain, bias, integral/differential and/or deadtime parameters. If any of these parameters are under computer control and this option is checked (the default) then the computer will control them for acquisition and automation purposes. If this option is not checked then these parameters will not be set by the program and must be set manually outside the program.

The Use Alternating On And Off Peak Acquisition flag will cause the program to acquire the on and off peak intensities alternately based on the number of repetitions determined by the Unknown Count Time factor parameters in the Count Times dialog. This option applies only to unknown samples. This flag is ignored for Time Dependent Intensity (TDI) Assigned Calibration acquisition and also for elements (acquisition order = 1) that are acquired using the Time Dependent Intensity (TDI) Self Calibration acquisition.

The Load Standard Data From File Setup is used to specify whether standard intensity data from a file setup should automatically be imported during automated acquisition using the File Setup option. When a file setup is imported from the new Sample dialog, the user is offered a choice whether or not to load the standard intensity data.

### **Quick Standard Modes**

You select one of two “quick standard modes for automation. The first mode (acquire only assigned standards) will only acquire those elements in the standards that are actually assigned as standards, MAN backgrounds or interference standards. Normally this is used to save acquisition time after the appropriate standards and other assignments have been made. The second mode will acquire all elements in the standards that are present above the minimum quick standard level as specified by the user (between 0.1 and 100 percent). Therefore zero or minor element concentrations in the standards will generally not be acquired.

In both cases if no elements in a particular standard are determined valid for quick standard acquisition, then all elements in that standard will be acquired.

### **EDS Acquisition**

Probe for EPMA supports EDS spectrum acquisition on a variety of EDS hardware interfaces for combined WDS and EDS acquisition. The EDS Spectrum Intensity acquisition acquires the complete spectrum for each analysis (standard and unknown samples) and saves it to the probe data file to allow the user to add or change elements calculated from EDS at any time after the data is acquired.

Note that the EDS count time can be set to use the EDS application “preset” time, or a user specified count time. These count time parameters are saved in probe database files with version numbers after v. 6.32.

### **EDS Spectrum Intensity Acquisition**

To utilize this feature the Acquire EDS Spectrum Intensities check box must checked. Note that EDS spectrum data needs to be acquired on suitable standards for this EDS interface acquisition method. This is very important to remember since an element can only be added later to the analysis, if the standard intensity has also been acquired.

To include these EDS acquired elements in the analytical calculations, simply go to the Calculation Options window from the Analyze! window and make sure that the EDS Data Options has the Use EDS Spectrum Intensities option selected.

### **Automated Image Acquisition**

One can now automatically acquire an analog images either before, after or both before and after each sample acquisition for standards, unknowns and/or wavescans. Be sure that your image parameters (signal type, signal type, AD averages and magnification along with instrument brightness and contrast) are correctly specified before running automation of samples.

Note that there are three magnifications defined in Probe for EPMA: a default magnification which is used for normal operation of the instrument between acquisitions, an analytical magnification which is used to acquire standards, unknowns and wavescans and an imaging magnification which is used to acquire images or for use with beam deflection acquisitions.

This option is useful for automated recording of the sample before beam exposure and again after beam exposure. The acquired images can be viewed using the Display, Annotate and Export Images under the Run menu.

### **Peak Element Before Acquisition**

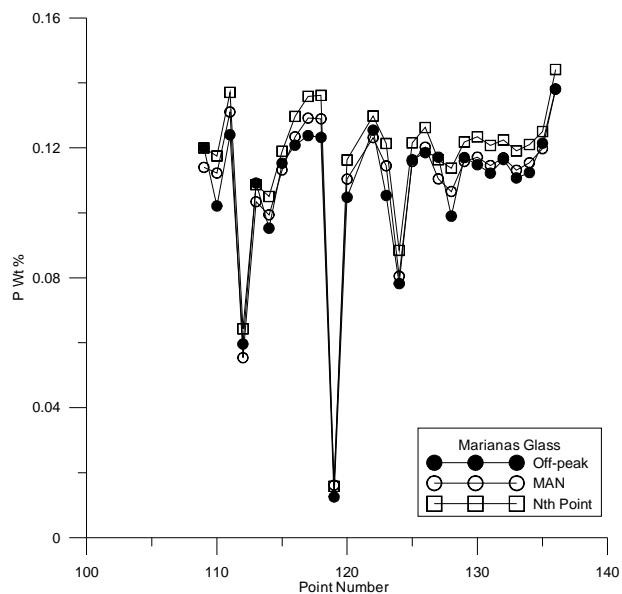
This option allows the user to define specific elements to be peaked just prior to the first data point for each unknown sample using either the Acquire or Automate windows. Subsequent acquisitions within the sample will use the spectrometer position from the peaking procedure from the first data point. Note that the element selected for peaking should have sufficient intensity for a precise peak position determination.

### **Nth Point Off-Peak Acquisition**

Another acquisition option method utilized to save time, is the Nth Point background method which simply acquires an off-peak background measurement every “N” points. That is, one performs an off-peak background measurement on the first point of a set of data and then re-uses that off-peak measurement for subsequent data points, instead of explicitly measuring the off-peak background for all points in the data set.

Typically this would be utilized on major and minor elements, with the trace elements continuing to be measured using normal off-peak background measurements.

The downside to this method is that if the composition changes, the average Z of the material will change and according to Kramer's Law, the continuum intensity will change as well. Although the change will be proportional to the degree to which the average Z changes and therefore will be small for a small difference in composition. However if the change in background intensity is greater than the precision of the measurement, it will affect the accuracy of the analysis.



A new option for the Nth Point off-peak background acquisition to handle samples with variable composition is the “smart” monitor element feature. Based on a user specified monitor element the software will automatically re-measure the off-peak backgrounds if the on-peak intensity of the monitor element changes by more than the user specified percentage.

Note that the monitor element should normally be a major element (with a statistical significance that is appropriate for the specified monitor element intensity change percent specified and also an element that finishes its on-peak count integration BEFORE the Nth Point off-peak ground elements in order to function properly.

## Peaking Options

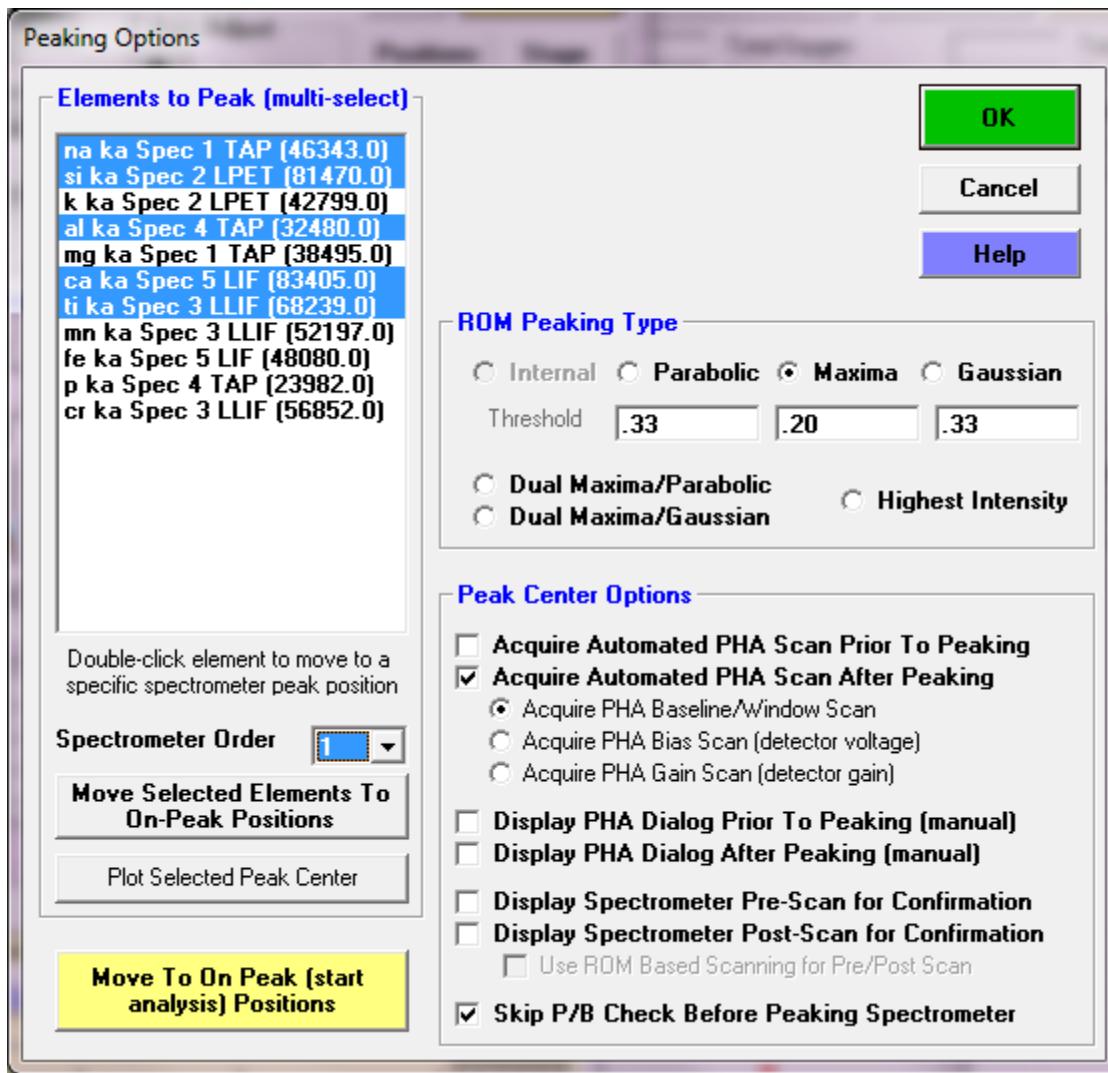
Clicking the peaking button opens the peak center window. All currently analyzed elements are listed. The Peak Center method can be specified. A choice of Interval Halving, Parabolic, ROM based (if supported by the hardware) and Manual peaking is available.

The default peak center method can be specified from the PROBEWIN.INI file. With all methods the program will perform a peak to background measurement to determine if the peak is statistically significant enough to attempt a peak center although this latter option can be skipped using the Skip P/B.

A button is available to move the spectrometers to their on-peak positions if selected in the element list. This option is useful for moving the spectrometers to calibrated peak positions for x-ray mapping purposes. One can also simple double-click an element in the list to move it to its on-peak position (and PHA parameters).

Another option forces the program to acquire a "pre-scan" wavescan just prior to the peak center procedure to allow the user to select an initial peak center start position. This last option is particularly useful in cases where the nominal spectrometer position is badly out of mechanical calibration and the actual peak position is some distance from the theoretical peak position. One can also specify a "post scan" to be executed after a peak center to confirm that the peak center method was properly performed. This option gives the operator maximum control over the automated determination of the peak "center". Using a post-scan, the peak center found by one of the 3 methods can be verified, by a slow step across the top of the centroid.

The Plot Selected Peak Center button will display graphically, the last peak center attempted for the selected element. The points displayed are numbered in the order acquired. Therefore, for example, if the parabolic peak center was performed, and an optimum peak center was obtained, there will be four points displayed: the three (on, hi, lo) for the parabolic fit and a fourth new on-peak based on the calculated peak center.



### Peak Center Options

- Acquire Automated PHA Scan Prior To Peaking
- Acquire Automated PHA Scan After Peaking

These two options will allow the program to automatically acquire a PHA scan for each element selected, either before, after or both before and after each peak centering. The results of the PHA scan are automatically saved to the user's probe database and can be displayed using the Run | Display and Export Scans menu.

- Display PHA Dialog Prior To Peaking
- Display PHA Dialog After Peaking

These two options will allow the program to display the manual PHA dialog for each element selected, either before, after or both before and after each peak centering. All three PHA scan types can be acquired: PHA, bias and gain scans. The results of the PHA scan are automatically saved to the user's probe database and can be displayed using the Run | Display and Export Scans menu.

## **Peak Center Methods**

The **Interval Halving** method is the most accurate, but the slowest. It is based on a peak crawl that changes direction and halves the step size whenever the count intensity drops by more than a standard deviation.

The **Parabolic** method peak center uses a moving three point fit on the peak top that is fit to a 2nd order polynomial. The centroid of the parabola is returned as the new peak center. This method is the fastest, if the spectrometer position is already close to the actual on-peak position. The **ROM Based** method peak center is available if the hardware interface supports a ROM peak center. The actual method used for the ROM Based peak center depends on the microprobe hardware. Note the following parameters apply to non-**ROM** based and **ROM** based peak scan parameters:

### **Non-**ROM** scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

### ****ROM** scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

### ****ROM** Based Peaking Scan:**

- fine scan uses peaking time divided by 4 times 1.0
- coarse scan uses peaking time divided by 4 divided by 2.0
- fine scan uses peaking time divided by 4 times 1.5

Several **ROM** based peak center fit types are available. Applies only to Cameca SX100/SXFive and JEOL 8200/8900 microprobes.

0 = Internal (the instrument **ROM** peak method which applies only to Cameca)

1 = Parabolic (spectrometer scan data is fit to a parabolic fit)

2 = Maxima (spectrometer scan data is fit to Brent's Maxima function)

3 = Gaussian (spectrometer scan data is fit to a Gaussian fit)

The internal **ROM** type is available only on Cameca instruments. The other options are only available on JEOL 8200/8900 and Cameca SX100/SXFive instruments. The parabolic and maxima fits require at least 3 data points above the threshold (see below). The parabolic and maxima methods have a user defined threshold that can be specified in the INI file and the program.

The **ROM** peaking thresholds are designed to allow the user to define the intensity values above which are used for one of the three **ROM** fitting methods (parabolic, maxima or Gaussian). The default is 0.33 which means that all intensity values 1/3 above the minimum to maximum intensity range are used in the fit.

### ****ROM** Based Peaking Scan Range**

The **ROM** peak scan range is related to the crystal 2d and spectrometer position peaking start size parameter as shown here:

$$\text{PeakingStartSize} = \text{ScalLiFPeakingStartSize} * \text{Factor}$$

where:

ScalLiFPeakingStartSize                          is from line 19 of the SCALERS.DAT file

and:

Factor    is calculated from the following expression:

$$Factor = \sqrt[3]{\frac{2d_{OnPeak}}{2d_{LiF200}}} (* 3) \sqrt{\frac{P_{HiLim}}{P_{OnPeak}}}$$

where:

|               |  |
|---------------|--|
| $P_{OnPeak}$  | is the spectrometer on-peak position       |
| $P_{HiLim}$   | is the spectrometer high limit             |
| $2d_{OnPeak}$ | is the 2d spacing of the analyzing crystal |
| $2d_{LiF200}$ | is the 2d spacing for LiF (4.0267)         |

Note that if the analyzing crystal 2d is greater than 30 (for LDE analyzers) the scaling factor is increased by an additional factor of three. The actual ROM peakscan range is further adjusted based on the expression:

$$ROMPeakScanRange = \frac{(P_{HiLim} - P_{LoLim})}{ScalPeakScanSizeFactor} * \frac{PeakingStartSize}{ScalLiFPeakingStartSize}$$

where:

|                           |   |
|---------------------------|---|
| $ScalPeakScanSizeFactor$  | is from line 16 of the SCALERS.DAT file                                 |
| $ScalLiFPeakingStartSize$ | is from line 19 of the SCALERS.DAT file                                 |
| $PeakingStartSize$        | is the calculated from "Factor" above and is user adjustable within PFE |
| $P_{HiLim}$               | is the spectrometer high limit  |
| $P_{LoLim}$               | is the spectrometer low limit   |

Note that the SCALERS.DAT parameters are designed to place these parameters in approximate relative scale for normal and high intensity spectrometers while the PeakingStartSize can be edited by the user from the Peak/Scan Options dialog for a specific analytical situation to increase or decrease the ROM peaking scan range.

The **Manual** method peak center is very simple, utilizing only a pre-scan, P/B check and post-scan. The peakscan range for post and prescan peakscans is based on the peakscan range calculated above but the counting time is further modified as shown here:

$$\begin{aligned} \text{PreScanCountTime} &= \text{PeakingCountTime} / 4 \\ \text{PostScanCountTime} &= \text{PeakingCountTime} \end{aligned}$$

Where:

$$\text{PeakingCountTime} \quad \text{is from the PROBEWIN.INI [counting] section.}$$

The program will automatically select elements in the element list that were not peaked in the previous peak center when the window is re-opened again. Note that the element list is multi-select so that any combination of elements can be peaked. If more than one element on the same spectrometer was selected, then the program will automatically peak center them one at a time. The Plot Selected Peak Center button may be used to display the last peak center count-position data for the selected element.

**The move to on-peak option is useful for moving a spectrometer (and setting the PHA conditions) to its on-peak position for imaging purposes. Remember : the elements to peak center (or move to on-peaks) must be highlighted before closing the dialog.**

## **Spectrometer Peak Center Scans**

All spectrometer peak center procedures may be prefaced with a spectrometer peak scan to allow the user to manually specify the nominal peak center position. This spectrometer peak scan is performed using the number of peak scan steps as defined in the Peak/Scan Options dialog. The count time for each peak scan point is based on the peak count time (divided by 4) as defined in the Count Times dialog.

## **Initial Peak-To-Background Measurements**

The program always performs a peak-to-background and peak intensity check before the actual peak center procedure. The peak-to-background is calculated by measuring the count rate at the nominal peak position and the current off-peak positions using the peak count time for each position. The measured count data must meet the required minimum peak-to-background and minimum peak count intensity as defined in the Peak/Scan Options dialog, before performing the actual peak center procedure.

If performing the peak center from the Automate! dialog, the program will not proceed with acquisition of sample data if one or more spectrometer fails to complete a peak center procedure for any reason.

## **Spectrometer Peak Center Procedure**

Three peak center procedures are used by Probe for EPMA. They are the Interval Halving, the Parabolic Fit and the ROM based peak center procedures.

### **Interval Halving**

Interval halving is a peak center procedure based on a peak crawl starting at the nominal peak position. The step size of the peak crawl is halved and the direction reversed whenever the count rate drops by more than a single standard deviation (square root of the intensity). The interval halving procedure is slow but extremely precise.

The initial peak step interval is always in a positive direction and the peak interval size is based on the Peaking Start Size as defined in the Peak/Scan Options dialog. Note that the default peaking start and stop sizes from the SCALERS.DAT file are modified by the following expression :

$$S_{actualstart} = S_{start} * \sqrt[3]{\frac{2d_{OnPeak}}{2d_{LiF200}}} (* 2) \sqrt{\frac{P_{HiLim}}{P_{OnPeak}}}$$

Where :

|                   |  |
|-------------------|--|
| $S_{actualstart}$ | is the actual calculated step size             |
| $S_{start}$       | is the spectrometer default start or stop size |
| $P_{HiLim}$       | is the spectrometer high limit                 |
| $P_{OnPeak}$      | is the spectrometer on-peak position           |
| $2 d_{OnPeak}$    | is the 2d spacing of the analyzing crystal     |
| $2 d_{LiF200}$    | is the 2d spacing for LiF (4.0267)             |

From this expression one can see that the actual start and stop sizes are unchanged at the spectrometer high limit when an LiF crystal is used, and are increased for lower spectrometer positions and larger crystal 2d spacings. The calculated start and stop sizes are also doubled when the crystal 2d spacing is larger than 30 angstroms (LDE crystals).

When the interval step size has been halved to a value less than the peaking stop size, the spectrometer is considered to be peak centered and the current position is saved as the new peak position.

If the number of intervals measured exceeds the "Maximum Peaking Cycles" as defined in the Peak/Scan Option dialog, the program terminates the peak center procedure with an error.

### **Parabolic Fit**

The parabolic peak center procedure is based on a moving three point triplet that attempts to fit an inverted parabola. The three positions measured are the middle, high and low positions. The middle position is initially the nominal peak position and the high and low positions are offset from the middle position based on the value of the Peaking Start Size, which is calculated using the process described in the interval peaking procedure above. Ideally the Peaking Start Size calculation should be adjusted to yield an offset which places the high and low positions at a position which produces approximately 1/2 the maximum intensity of the peak, for a more precise parabolic fit.

The peak center procedure then determines if the high and low position intensities are at least one standard deviation less than the middle position intensity. If the three intensities meet this criteria, the program calculates the centroid of a parabola based on the count-position data triplet. The results of the centroid peak fit can be viewed by placing the program into "Debug Mode" under the Output menu.

If the data cannot be fit to a parabola, the middle position is shifted by an amount equal to 1/2 of the Peaking Start Size and the middle, high and low position intensities are measured again.

If the number of count-position data triplets measured exceeds the "Maximum Peaking Cycles" as defined in the Peak/Scan Option dialog, the program terminates the peak center procedure with an error.

### **ROM Peaking**

The ROM peaking procedure is available for those interfaces that support an integrated ROM based peaking procedure. These are the JEOL 8900/8200/8500/8x30 and SX100/SXFive interface types. Note the following parameters apply to non-ROM based and ROM based peak scan parameters:

#### **Non-*ROM* scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

#### ***ROM* scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

#### ***ROM* Based Peaking Scan:**

- fine scan uses peaking time divided by 4 times 1.0
- coarse scan uses peaking time divided by 4 divided by 2.0
- fine scan uses peaking time divided by 4 times 1.5

The actual peaking procedure utilized by each interface is somewhat different but usually based on a parabolic fit of some variety. The program passes the "Peaking Start Size" to each interface specific ROM Peaking routine where it is modified if necessary.

Several ROM based peak center fit types are available. Applies only to Cameca SX100/SXFive and JEOL 8200/8900/8500/8x30 microprobes.

- 0 = Internal (the instrument ROM peak method which applies only to Cameca)
- 1 = Parabolic (spectrometer scan data is fit to a parabolic fit)
- 2 = Maxima (spectrometer scan data is fit to Brent's Maxima function)
- 3 = Gaussian (spectrometer scan data is fit to a Gaussian fit)
- 4 = Dual ROM (Maxima for LiF crystals, Parabolic for other crystals)
- 5 = Dual ROM (Maxima for LiF and Gaussian for other crystals)
- 6 = Highest Intensity (using smoothed intensities)

To increase the default ROM scan width decrease the Peakscan Size (line 16) or increase the LiF Peaking Start Size (line 19) in the SCALERS.DAT file. Note that within the application, the ROM based peak scans are based on the Peaking

Count Time divided by 4 and the number of Peak Scan Points. The ROM scan width is determined by the Peaking Start Size.

The internal ROM type is available only on Cameca instruments. The other options are only available on JEOL 8200/8900/8500/8x30 and Cameca SX100/SXFive instruments. The parabolic and maxima fits require at least 3 data points above the threshold (see below). The parabolic, maxima and Gaussian methods have a user defined threshold that can be specified in the INI file and the program.

The ROM peaking thresholds are designed to allow the user to define the intensity values above which are used for one of the three ROM fitting methods (parabolic, maxima or Gaussian). The default is 0.33 which means that all intensity values 1/3 above the minimum to maximum intensity range are used in the fit.

**JEOL 8900/8200/8500/8x30 (InterfaceType=2)-** The "Peaking Start Size" is not used. Instead a peak parameter of 1 (fine scan) is always used.

**SX100/SXFive (InterfaceType=5)-** The "Peaking Start Size" is modified to produce a number between 0 and 4 by utilizing the following expression :

$$pw = (M_{high} - M_{low}) * P / 3000000$$

where pw is the SX100/SXFive peak center width  
M<sub>high</sub> is the motor high limit  
M<sub>low</sub> is the motor low limit  
P is the Peaking Start Size

### **Manual Peaking**

The Manual method peak center is very simple. First a wavelength pre-scan is performed, after which the user is allowed to select the actual peak position. This is followed by a peak to background check, after which a wavelength post-scan is performed at a finer scale to allow the user to confirm the selected peak position. The user is then once again given the chance to adjust, confirm or cancel the peak center process.

Note that the final wavelength post-scan data will be saved to the Plot Peak Center array (see the Peaking dialog) for subsequent plotting or output by the user. Note that the post-scan range is 5% (+/- 2.5%) of the pre-scan range.

### **Final Peak-To-Background Measurements**

After a successful peak center procedure the peak-to-background is re-calculated by simply measuring the on-peak count rate at the new peak position. This result can be compared with the initial peak-to-background to evaluate the peak center procedure performance.

### **Post Scan Peak Confirmation**

This option gives the operator maximum control over the automated determination of the peak "center". Here the peak center found by one of the 3 methods can be verified, by a slow step across the top of the centroid. Optimal centering is left to the discretion of the user, but one strategy is to look for the small flat plateau that normally is present at the peak centroid, or looking at the centroid at 99% of maximum intensity. The values used are, respectively, the time value of the "peaking" in Count Times and the step width using 1/20 (5% or +/- 2.5%) of the width for peakscan limits, in Peak/Scan options.

### **Start Peaking**

Clicking Start Peak Center starts a manual peak center (and peak pre-scan and/or post scan if selected) on the elements selected from within the Peaking button window. If more than one element on the same spectrometer was selected, then the program will automatically peak center them one at a time.

Select the elements to be peak centered and the peak center method from the Peaking dialog accessed from the Acquire! window.

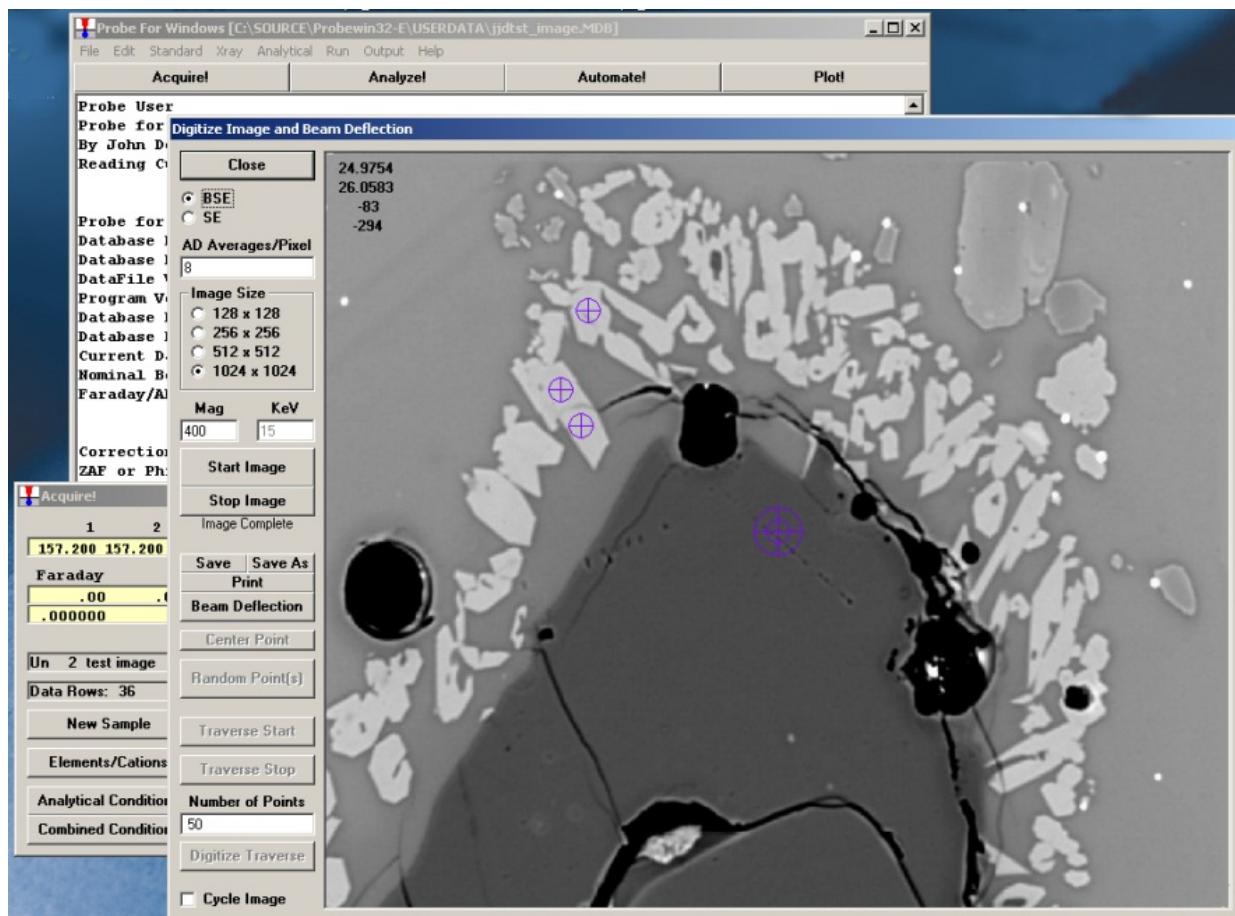
## Start Standard or Unknown Acquisition

Clicking the Start Sample Count button starts a standard or unknown sample acquisition based on the current sample. Data is automatically appended to the current sample which is displayed in the Acquire! window. Be sure that current sample is the correct sample to avoid appending data to the wrong sample.

The program will check the sample parameters before starting an acquisition to make sure that the sample is a valid sample for the configured hardware. For example, if the sample uses Fe ka LiF on spectrometer 2, but the current crystal on spectrometer 2 is PET and the crystal flip interface is not enabled, the program will warn the user that the crystal must be flipped manually before starting the acquisition.

## Imaging

The Imaging acquisition window is very similar to the Digitize Image dialog available from the Digitize window except that it does not allow the user to digitize stage coordinates based on the image. However, it does allow the user to acquire images manually and save them to the probe database. These images are referenced to the current sample and any data point coordinates associated with that sample can be subsequently displayed using the Display Images dialog available from the Run menu. Multiple images can be saved to a single sample.



Note that the image data is 32 bit and is stored in a separate binary file from the probe database but with the same name and a .BIM extension. All images are saved as un-normalized intensities. The file format is extremely simple and

consists entirely of long (4 byte) integers. The first 8 bytes are the X and Y pixel dimensions of the image and the following X by Y 4 byte values are the image data. This is repeated for each subsequent image.

**Note that there are three magnifications defined in Probe for EPMA: a default magnification which is used for normal operation of the instrument between acquisitions, an analytical magnification which is used to acquire standards, unknowns and wavescans and an imaging magnification which is used to acquire images or for use with beam deflection acquisitions.**

In addition, the window allows the user to manually deflect the beam using the mouse and then acquire data points at those beam deflection positions. The data point coordinates are recorded for those beam deflection positions in stage coordinates and can be displayed in the Display Image dialog available from the Run menu.

Note on the scan speed field:

**ImageInterfaceType = 4**      ' JEOL 8900/8200/8500/8x30

"AD Averages/Pixel"

Specify the number of A-D conversions to average per pixel (range 1 - 1000)

**ImageInterfaceType = 5**      ' SX100/SXFive mapping

"mSec/Pixel"

Specify the image dwell time in msec/pixel (range 1 - 1000)

**ImageInterfaceType = 6**      ' SX100/SXFive Video

"Scan Rate"

Specify the image scan speed (range = 1 - 7)

*The large cross hair displayed on the image represents the beam deflection position and the smaller cross hair represents the stage position. Use the beam deflection button to deflect the beam and the Move Stage button to move the stage to the image position. The yellow color indicates the whether the window is in beam deflection or stage move mode.*

### **Image Calibration**

So that the beam scan is calibrated accurately to the stage coordinate system, the user should utilize the "@" button to calibrate the beam scan based on an acquired image. The image calibration is performed by selecting 2 points on a diagonal in the image for which the stage coordinates are known (by centering the beam on those two stage positions).

A couple of points to consider when performing the image calibration:

- Scan rotation: not only does the scan rotation need to be set exactly (within 0.2 degrees or so) but the scan rotation need to be set to the stage orthogonal when the light optics are properly focused because the working distance changes the scan rotation.
- Scan speed: the scan speed affects the image shift significantly, especially in the X direction- the scan rotation (so long as it's a horizontal reference) is not very affected. But the use of a slow scan speed is very important for accurate image calibration.
- Electron beam focus: the scan rotation rotates dramatically when adjusting the focus. Be sure you are focused properly before setting the scan rotation.
- Prior to any image calibration efforts it is desirable to check the beam orthogonality of the instrument on a calibration standard.

### **Start Wavescan**

If the current sample is designated as a wavescan sample, then clicking this button opens the Wavescan Acquisition window and automatically starts a wavescan sample acquisition.

All elements in the wavescan sample will be scanned, even if more than one element is on a spectrometer. This provides an easy method to scan all elements in a quantitative sample for off-peak interferences and optimal background offset positions.

During the acquisition the wavescan graph may be viewed in greater detail, simply click the wavescan graph to toggle the display size. The wavescan window contains fields to display the mouse cursor position in spectrometer, angstrom and count units. In addition, options for loading the NIST x-ray database window based on the x-ray range of the wavescan graph clicked and a button to save the graph data to an ASCII file are available.

Note that angstrom values in the x-ray database list with Bragg reflection orders higher than one (Roman Numeral "I") are NOT corrected for refraction index corrections. However, KLM markers displayed in the Graph Data wavescan plot dialog are corrected using the equation  $A' = A * (1 - (K - (K / N^2)))$  where A is the uncorrected angstrom position, K is the refractive index from the CRYSTALS.DAT file and N is the Bragg reflection order.

Once the wavescan has been acquired, the user can view the off-peak interferences, by using the Plot! window to display the wavescan sample graphically.

Note that the current sample must be a wavescan sample without any data already acquired. To start a new manual wavescan sample use the New Sample button from the Acquire! window.

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## Analyze!

The Analyze window is a modeless dialog that lists both data and processed data for standard, unknown and wavescan samples. The average, standard deviation and other statistical calculations are shown in the upper data grid and the individual data points in the lower data grid. In addition buttons are provided to allow the user to specify elements by fixed composition, standard and interference assignments, Time Dependent Intensity (TDI) element assignments and plots, edit sample name and description, and specify sample calculation options and many other parameters. See the detailed descriptions below for more information.

*When making changes to the standard and/or unknown samples for any of these parameters, be sure that the last unknown sample in the list is also selected so that the changes made will be included in the next new sample that is created (unless loading a previously saved sample setup). Probe for EPMA, by default, always uses the last unknown sample as the basis for the next standard or unknown that is created.*

The **Select All** button will select all the currently displayed samples in the Analyze! Window list. This is useful for making changes to large numbers of samples with a single mouse click.

Note that if the samples were acquired with the self Time Dependent Intensity (TDI) correction (see the Special Options button in the Acquire! Window), one will probably not want to change the standard assignments for more than one sample at a time. The self Time Dependent Intensity (TDI) element correction requires that the assigned Time Dependent Intensity (TDI) sample number be the sample number of the sample itself.

The **Add To Setup** button will add the currently selected sample to the Sample Setup list. This list is accessed from the New Sample button in the Acquire! Window or the Sample Setups button in the Automate! window to allow the user to manually or automatically load different sample setups for acquisition and automation within a single run. To rename, add or delete the sample setups, go to the Elements/Cation button in the Analyze! Window and click the save Sample Setup button.

The **Save Setups** button is used to quickly save a pointer to an unknown sample so the sample can later be used as the basis for creating a new sample. It will save all selected samples as sample setups using the sample name already given. If it is desired to use a different name for the sample setup, use the Save Sample Setup button from the Elements/Cations dialog. The sample setup can be loaded for new samples by using the Load Sample Setup button in the New Sample dialog.

Note that one can double-click on any line in the currently analyzed sample and the program will display an additional grid (spreadsheet) with the analytical results in row format (analysis type in columns, elements in rows) for the selected line as shown below. In addition, if the row display spreadsheet is open and automatic analysis is turned on, the program will display the results for the last acquired line.

The screenshot shows the Analyze! window with the following details:

- Sample List (multi-select) (double-click to see intensity data):**
  - Selected: Standards
  - Options: Unknowns, Wavescans, All Samples
  - Buttons: Select All, Add To Setups, Save Setups
- Analyze Options:**
  - Buttons: Analyze, Raw Data, KRaw, >>Excel
  - Checkboxes: Pause Between Samples, Use All Matrix Corrections, Disable Selected Sample(s), Enable Selected Sample(s)
  - Buttons: Report, Match
- List Standard Intensities:**

|        | Total Oxygen | Total Weight % |
|--------|--------------|----------------|
| 42.979 | 99.795       |                |
| 42.651 | 12.887       |                |
| .328   | 21.734       |                |
- Calculation Options:**
  - Buttons: Combined Conditions, Count Times
  - Checkboxes: Sort Stat and Data Grids By Geological/Atomic Number, Do Not Output To Log
- Combine Selected Samples:**
  - Buttons: Combine Analysis Lines From Selected Samples, Combine Data Lines From Selected Samples, Sort Stat and Data Grids By Geological/Atomic Number, Do Not Output To Log
- Standard Assignments:**

|           | Na <sub>2</sub> O | SiO <sub>2</sub> | K <sub>2</sub> O | Al <sub>2</sub> O <sub>3</sub> | MgO    | CaO    | S      | FeO    | Cl     | TiO <sub>2</sub> |
|-----------|-------------------|------------------|------------------|--------------------------------|--------|--------|--------|--------|--------|------------------|
| Average:  | 2.614             | 40.544           | 2.029            | 14.718                         | 12.708 | 10.237 | .013   | 10.899 | .006   | 4.667            |
| Std Dev:  | .051              | .199             | .054             | .108                           | .079   | .067   | .021   | .054   | .017   | .084             |
| ZAF Corr: | 2.0206            | 1.3177           | 1.0964           | 1.4424                         | 1.5474 | 1.0759 | 1.2278 | 1.1795 | 1.2042 | 1.1713           |
| Std Err:  | .023              | .089             | .024             | .049                           | .026   | .020   | .009   | .024   | .009   | .027             |
| %Rel SD:  | 1.96              | .49              | 2.64             |                                |        |        |        |        |        |                  |
| Minimum:  | 2.525             | 40.192           | 1.970            |                                |        |        |        |        |        |                  |
| Maximum:  | 2.651             | 40.682           | 2.094            |                                |        |        |        |        |        |                  |
- Specified Concentrations:**

|   | Na <sub>2</sub> O | SiO <sub>2</sub> | K <sub>2</sub> O | Al <sub>2</sub> O <sub>3</sub> | MgO | CaO | S | FeO | Cl | TiO <sub>2</sub> |
|---|-------------------|------------------|------------------|--------------------------------|-----|-----|---|-----|----|------------------|
| St 468 Set 1 Hornblende (Kakanui) USNM 143965 (MagAnal = 10000., Mode = Analog, Spot) | 42.979            | 99.795           |                  |                                |     |     |   |     |    |                  |
|   | 42.651            | 12.887           |                  |                                |     |     |   |     |    |                  |
|   | .328              | 21.734           |                  |                                |     |     |   |     |    |                  |
- Name/Description:**

|   | Total Oxygen | Total Weight % |
|---|--------------|----------------|
| St 468 Set 1 Hornblende (Kakanui) USNM 143965 (MagAnal = 10000., Mode = Analog, Spot) | 42.979       | 99.795         |
|   | 42.651       | 12.887         |
|   | .328         | 21.734         |
- Conditions:**

| St 468 Set 1 Hornblende (Kakanui) USNM 143965 (MagAnal = 10000., Mode = Analog, Spot) |  |  |  |
|---|--|--|--|
- Elements/Cations:**

|   | Na <sub>2</sub> O | SiO <sub>2</sub> | K <sub>2</sub> O | Al <sub>2</sub> O <sub>3</sub> | MgO | CaO | S | FeO | Cl | TiO <sub>2</sub> |
|---|-------------------|------------------|------------------|--------------------------------|-----|-----|---|-----|----|------------------|
| St 468 Set 1 Hornblende (Kakanui) USNM 143965 (MagAnal = 10000., Mode = Analog, Spot) | 42.979            | 99.795           |                  |                                |     |     |   |     |    |                  |
|   | 42.651            | 12.887           |                  |                                |     |     |   |     |    |                  |
|   | .328              | 21.734           |                  |                                |     |     |   |     |    |                  |
- Results in Oxide Weight Percent:**

|           | Na <sub>2</sub> O | SiO <sub>2</sub> | K <sub>2</sub> O | Al <sub>2</sub> O <sub>3</sub> | MgO    | CaO    | S      | FeO    | Cl     | TiO <sub>2</sub> |
|-----------|-------------------|------------------|------------------|--------------------------------|--------|--------|--------|--------|--------|------------------|
| Average:  | 2.614             | 40.544           | 2.029            | 14.718                         | 12.708 | 10.237 | .013   | 10.899 | .006   | 4.667            |
| Std Dev:  | .051              | .199             | .054             | .108                           | .079   | .067   | .021   | .054   | .017   | .084             |
| ZAF Corr: | 2.0206            | 1.3177           | 1.0964           | 1.4424                         | 1.5474 | 1.0759 | 1.2278 | 1.1795 | 1.2042 | 1.1713           |
| Std Err:  | .023              | .089             | .024             | .049                           | .026   | .020   | .009   | .024   | .009   | .027             |
| %Rel SD:  | 1.96              | .49              | 2.64             |                                |        |        |        |        |        |                  |
| Minimum:  | 2.525             | 40.192           | 1.970            |                                |        |        |        |        |        |                  |
| Maximum:  | 2.651             | 40.682           | 2.094            |                                |        |        |        |        |        |                  |
- Disable Selected Line(s) / Enable Selected Line(s):**

|      | Na <sub>2</sub> O | SiO <sub>2</sub> | K <sub>2</sub> O | Al <sub>2</sub> O <sub>3</sub> | MgO | CaO | S | FeO | Cl | TiO <sub>2</sub> |
|------|-------------------|------------------|------------------|--------------------------------|-----|-----|---|-----|----|------------------|
| 71 G | 2.651             | 40.192           | 1.984            |                                |     |     |   |     |    |                  |
| 72 G | 2.618             | 40.617           | 2.071            |                                |     |     |   |     |    |                  |
| 73 G | 2.644             | 40.682           | 2.094            |                                |     |     |   |     |    |                  |
| 74 G | 2.632             | 40.607           | 1.970            |                                |     |     |   |     |    |                  |
| 75 G | 2.525             | 40.623           | 2.024            |                                |     |     |   |     |    |                  |
- Analyze Data:**

|     | K-Raw   | Counts | Elemental | Oxide   |
|-----|---------|--------|-----------|---------|
| Na  | .13200  | 290.6  | 1.961     | 2.644   |
| Si  | 1.01592 | 4320.4 | 19.016    | 40.682  |
| K   | .14013  | 476.5  | 1.739     | 2.094   |
| Al  | 1.60922 | 1618.2 | 7.766     | 14.673  |
| Mg  | .87720  | 1495.8 | 7.709     | 12.784  |
| Ca  | .66652  | 2054.2 | 7.364     | 10.304  |
| S   | .00057  | 1.7    | .036      | .036    |
| Fe  | .76088  | 2171.2 | 8.530     | 10.973  |
| Cl  | .00334  | 1.2    | .024      | .024    |
| Ti  | .04322  | 144.1  | 2.808     | 4.684   |
| P   | .00029  | .3     | -.007     | -.015   |
| F   | -.00045 | .1     | -.005     | -.005   |
| O   |         |        | 43.120    | .328    |
| H   |         |        | .105      | .938    |
| Mn  |         |        | .070      | .090    |
| Sum |         |        | 100.236   | 100.236 |

## Analyze

This button is used to convert previously acquired x-ray count data to elemental, oxide, atomic or formula results. The samples currently selected in the sample list will be analyzed and the analysis output is directed to both the Analyze! window and the log window.

The Analyze! window will display the totals, calculated oxygen (if any) and z-bar and atomic weight along with the last specified results in the following order :

- Elemental Weight Percents
- Oxide Weight Percents
- Atomic Weight Percents
- Formula Atoms

All calculations (including homogeneity statistics and mineral end-members) are also output in the above order to the log window as text. To reduce the detail level of the output, see the options in the Analytical | Analysis Options menu. To see a complete output of all intermediate steps of the analysis calculations use the Output | Debug Mode menu to turn on debug printout and re-run the analysis.

If more than one sample is selected for analysis, it may be desirable to check the Pause Between Samples check box. If this is selected, then the program will automatically pause after each analysis until the user clicks the Cancel or Next buttons on the Analysis Status window. This allows the user to view the results of each analysis and disable data points if necessary before proceeding to the next analysis. This is especially useful when analyzing the standards as unknowns after a standardization has been acquired to check for problems in the standard calibration data.

The Copy buttons in the two grids can be used to copy the displayed results into the Windows Clipboard buffer, from which it can be pasted into other applications as tab delimited text.

### **Error Messages**

Several problems can occur when performing analyses of quantitative samples. If data for a standard is missing, the program will print "No count data for standard \_\_ for \_\_". This means that either that the standard was not acquired at all or that the standard assignments were changed to a channel that was not acquired due to a "quick" standardization. Simply acquire data for the specified element on the specified standard or re-assign the standard to correct the problem.

If the message "No count data for interference standard \_\_ for \_\_" is seen, a similar problem exists. Note that standards used in the interference corrections should not be run as "quick" standards to avoid this problem. If the warning message persists, you may be assigning an interference correction where none exists. Check your wavelength tables to be sure. If you find no interference, then remove the interference correction for the element. Remember, interferences are quite dependent on spectrometer resolution (slit width) and PHA settings (for higher order lines) and so the magnitude or presence of the interference may change from run to run.

### **Problems With the Analysis**

Performing quantitative analysis is always beset with many difficulties and it is often difficult to pinpoint the cause of "bad" analyses. More often than not, the effect is the sum of more than one problem. However, there are several things that can be checked. First of all start by examining the standards. It is in situations like this that the use of secondary standards can really be helpful in determining what is going on. If the primary and secondary standards do not agree with each other, there is a problem.

First of all, are the standards really "good" standards? That is, have their compositions been determined by a reliable analytical method? In general, major element standard concentrations need to be determined using classical wet chemistry methods. Second, are the concentrations entered into the STANDARD.MDB database correct and without typographical errors? Third, was the right standard actually acquired? It is easy to get lost at 300-400 magnifications when using a standard mount which contains many standards.

Some other things to consider :

1. Is the operating voltage correct specified? Is the correct x-ray line tuned for each element? Check the on-peak position offsets from the Peak/Scan Option dialog and see if they are reasonable. The program will usually type a warning if the actual and calculated peak positions are very different. Be sure that the spectrometer is not tuned on a nearby line of another element if using multi-element standards.

2. Be sure that no bad data points are in the standards samples used for the quantitative calibration. The best way to check for this is to analyze each standard and examine the results to look for points with obviously bad or low totals (epoxy, inclusions, bad surface polish, bad carbon coat, etc.). If a bad point is found, one can disable it. Remember, one can always enable the data later on. A disabled point is simply not used in calculating the analytical calculations but is still present. A good rule of thumb is to only disable points that have low totals since generally most of the problems mentioned above will result in fewer x-ray counts. Avoid disabling points just to get better agreement between the primary and secondary standards. Points that have high totals should

not be arbitrarily disabled. It may be necessary to look for other problems such as points with low totals in the primary standards.

3. Look for interferences on the analyzed elements. One easy way to do this is to use the Interferences button in the Element Parameters window (see the Elements/Cations button in the Acquire! or Analyze! window). The best method is to perform a wavelength scan and display possible interfering peak markers. Then not only can the interferences be seen but one can also select background positions free from interferences. If the element causing the interference is present in significant concentrations in your unknowns, and is not being analyzed for, it may be necessary to add the interfering element to the run by creating a new sample with the interfering element as an analyzed element. Be sure that the proper standards are available to use for the interference correction.

4. If none of the above suggestions seem to help, try acquiring the standards again. Probe for EPMA uses an automatic standard drift corrections which can make a significant difference in situations where one or more of the standard intensities are drifting. Note that since the program will perform an automatic drift correction not only on the standards, but also the interference standards and the MAN background standards, it might be also be necessary to run additional sets of those standards or MAN standards.

5. In the case of trace or minor elements, also check to see that none of the off-peak positions are interfered with by another peak. This can cause a reduction in the on-peak counts, sometimes enough to result in a negative k-ratio. Always run at least one wavelength scan on a sample, using the same count time as your quantitative analyses, and if a peak is seen interfering with the off-peak marker, use the Low and/or High buttons in the Graph Data window to select a new off-peak position that is not interfered with.

6. Finally are the standards really "good" standards? That is, homogeneous and whose compositions are accurately and precisely known. Some may have been analyzed by classical "wet" chemical methods (e.g. Smithsonian mineral and glass standards) of small quantities of separates. Second, are the concentrations entered into the STANDARD.MDB database correctly? (no typos)

7. Low totals may result from charging due to a poor conductive (e.g. carbon) coating on insulated materials. Many year-old carbon coats have been known to fail. Also, a carbon-sputtered coating is not necessarily equivalent to a carbon-evaporated coating; both standards and unknowns may need to be recited by the same process

8. Peaks shifts both may need to be considered. The later are generally well recognized for the K lines of "light" elements (including Al), but less so for some L lines. If you recall that this results from the outer shell electron bonding or configuration being involved in X-ray production, those elements with M shell electrons (e.g. Co) also can have significant peak shifts.

9. Peak positioning: you may want to verify that the peak centering method you have selected is giving you optimal peak centers. It would not hurt to run the post-scan verification option where you step across the peak that the automation has chosen.

### **Description of the Analysis Printout**

When the analysis is printed out you will see the sample type (st or un), sample number and name printed on the first line. If the sample is a standard, then the sample set number is also shown. The microprobe takeoff angle and the operating voltage is shown.

The next line indicates the total number of lines (or data points) that are contained in the sample. One may have 1 to 100 points in each sample. In the same line are the number of points that have status of "G" for good, meaning that this many points are undisabled. A sample with all points disabled will have the number of "G" points equal to 0 and will not be used in any calibrations or recalculations and therefore cannot be quantitatively analyzed, unless using the Analyze Selected Lines button.

Next, the average elemental totals and the total, calculated and excess oxygen is displayed. The calculated oxygen is the amount of oxygen calculated by cation stoichiometry (if selected) and the excess oxygen is the difference between the measured and oxygen calculated if the Display As Oxide option was selected. This excess oxygen is often very useful in determining if the selected cation ratios are correct, especially for iron bearing oxides.

Next is the atomic weight and average Z-bar for the sample. This is followed by the average number of ZAF iterations needed to converge each data point for the sample and the MAN iterations, which are the iterations required to converge the MAN background, interference, APF (Area Peak factor) and Time Dependent Intensity (TDI) element corrections. The Z-bar is defined as the sum of the weight fraction of the atomic numbers of all elements in the sample including specified elements.

Following this are the weight percentages and standard deviation of any elements specified for the sample by fixed concentration, difference or stoichiometry.

The specified element type is listed for each specified element. The type "SPEC" means that the element concentration is truly specified. That is, either specified by the user or (for standard samples) loaded from the standard composition database. The other types are "DIFF" for element by difference, "CALC" for element by stoichiometry to stoichiometric oxygen and "RELA" for an element by stoichiometry to another element.

The next two lines indicate the type of background correction used for each element ("MAN" for mean atomic number corrected and "OFF" for off-peak corrected elements) and the average count times for each element in the sample. Remember that the count times for each element can be different for each line in the sample. To see the actual counting time details use the Data button in the Analyze! window with the Debug Mode menu checked.

Note that if MAN background corrected sample was analyzed, the program will also indicate the magnitude of the absorption correction to the continuum background counts in the line labeled "%ABS" if the element is MAN corrected.

The analyzed element symbols are printed next and listed below are the weight percentages calculated for each data point for each element. The elemental weight results are then followed by the average, "AVER" of each element column, the standard deviation, "SDEV" and the standard error, "SERR". The standard deviation is basically the range of the results and the standard error is essentially the precision of the average. Finally the percentage relative standard deviation, "%RSD" of the results are printed which is simply the standard deviation divided by the average times 100.

Note that one can perform analyses of samples which are not unknowns, e.g. it is possible to have the program analyze a standard as though it were an unknown. Therefore, if the sample is a standard sample, the program next lists the published, "PUBL" weight percentage value for the element as entered in the default standard database. If the element is not found in the standard database it is shown as "n.a." or "not analyzed". The next line lists the per cent variance, "%VAR" from the published value for each element compared to the actual measured average for the standard. This can provide a valuable check on the quality of your analyses for secondary standard elements. That is, element channels which are not assigned as the primary calibration for that element. After this, is the line labeled "DIFF" which is a simple difference between the "AVER" and "PUBL" values.

The actual primary standard used for each element is displayed on the next line "STDS" since the standard assignments can be made on a sample by sample basis.

Note that sometimes the average analyzed value of a standard that is assigned, appears to be not exactly the value shown in the "PUBL" line. It seems reasonable that they should always be exactly the same, but remember that because PROBE treats all samples (standards and unknowns) as unknowns when performing an analysis, you may see a small discrepancy if the standard contains other analyzed elements that are also not assigned. This is because Probe for EPMA calculates the correction factors for an analysis of a sample, based on the actual analyzed composition of the sample, not the theoretical composition in the STANDARD database. Of course, for the analytical calibration, Probe for EPMA uses the database composition for the calculation of standard k-factors.

Below this are shown the average standard k-factors "STKF" or the standard beta-factors "STBE" if using alpha-factors, the average standard counts "STCT" (all intensities are in counts per second per nominal beam) followed by the

normalized unknown k-ratio, "UNKF", the unknown count rate net intensity (Peak-Bkg) "UNCT", the unknown background counts "UNBG" (either MAN or off-peak measured), the ZAF correction factor, "ZCOR" if using ZAF or Phi-Rho-Z or the "UNBE", unknown beta-factor if using alpha-factors, and the average raw k-ratio, "KRAW", which is the normalized and background corrected unknown counts divided by the standard counts.

The "KRAW" value shown is corrected for all corrections except the matrix correction. These include the deadtime, count time, beam drift, MAN or off-peak background, and the quantitative interference and APF (area peak factor) corrections. The peak to background ratio for each element is shown in the "PKBG" line. A peak to background of close to 1.0 means that no peak was present.

Below this are listed the per cent correction to the counts for any interference corrections that were performed on the sample in the line labeled "%INT".

Finally, if the Time Dependent Intensity (TDI) element extrapolation was selected, the program will show the per cent change in the x-ray counts due to the Time Dependent Intensity (TDI) element extrapolation in the line labeled "VOL%" and the average deviation in the Time Dependent Intensity (TDI) fit data "DEV%". Also if any area peak factors (APF) were selected, the program will print the sum of the APFs (1.00 indicates no APF correction) for each element affected in the line labeled "APF:".

If the user elected to calculate the detection limits and/or sample statistics, the program will then print those calculations.

Following this are formula and atomic percent calculations if they were selected. All sample calculation options can be assigned to a single sample or a range of samples.

### **Summary of Analysis Printout**

TYPE: Element type (ANAL=analyzed, DIFF=by difference, STOI=by stoichiometry to oxygen, RELA=by stoichiometry relative to another element, CALC=oxygen by stoichiometry, EDS=specified concentration from EDS system, SPEC=specified as a fixed concentration

BGDS: Background type. INT=integrated intensity, LIN=linear interpolation, AVG=averaged, EDS=measured by EDS, HIGH=high off-peak only, LOW=low off-peak only, EXP=exponential fit, S-HI=slope from high off-peak, S-LO=slope from low off-peak, POLY=polynomial fit, MAN=mean atomic number fit model

ABS%: Percent continuum absorption correction (MAN backgrounds only)

TIME: Count time in seconds (average of all lines)

BEAM: Beam current in beam current units (usually nA) (if Aggregate mode it is the average of all lines and duplicate elements because detection limits are calculated based on *average* count time and beam current of all duplicate elements)

AGGR: Total number of aggregate (duplicate element) channels combined if using Aggregate Intensity option.

ELEM: Element symbol (if followed by a "-D" the quantitation is disabled)

AVER: Average

SDEV: Standard deviation

SERR: Standard error

%RSD: Relative percent standard deviation (SDEV/AVER \* 100)

PUBL: Published concentration from STANDARD.MDB database. (n.a.=not analyzed)

%VAR: Percent variance from the "published" value

DIFF: Algebraic difference from the "published" value

STDS: Standard number assignment (primary standard for this element)

STKF: Standard K-factor

STBE: Standard "beta-factor" (Bence-Albee correction only)

STCT: Standard count net intensity count rate (Peak-Bkg)

UNKF: Unknown K-factor

UNBE: Unknown "beta-factor" (Bence-Albee correction only)

UNCT: Unknown count net intensity rate (Peak-Bkg)

UNBG: Unknown background count rate

ZCOR: ZAF correction factor (matrix correction)

KRAW: Raw k-ratio (unknown counts/standard counts) corrected for all parameters (background, interferences, etc.)

PKBG: Peak to background ratio

INT%: Percent interference correction

APF: Area peak factor calculated from pure element area peak factors

APF\*: Specified area peak factor

TDI%: Percent Time Dependent Intensity (TDI) element correction

DEV%: Percent deviation in Time Dependent Intensity (TDI) element correction fit

TDI#: Time Dependent Intensity (TDI) assignment sample number (self-Time Dependent Intensity (TDI) correction is assigned it's own sample number)

TDIF: Time Dependent Intensity (TDI) fit type (0 = log-linear or exponential, 1 = log-quadratic or hyper-exponential)

TDIT: Total elapsed time for acquisition of the on-peak intensity data

TDII: Zero time intensity intercept in cps/per nominal beam (deadtime and beam drift but not background corrected)

TDIL: Zero time intensity intercept in Log(cps)/per nominal beam (deadtime and beam drift but not background corrected)

BLNK# Blank correction calibration unknown number for trace element background offset

BLNKL Blank correction calibration value (actual level of element in blank- usually zero) in wt%

BLNKV Blank correction measurement value (measured value of blank- should be equal to calibration level)

FIT1: Multi-standard calibration fit coefficients

FIT2: Multi-standard calibration fit coefficients

FIT3: Multi-standard calibration fit coefficients

DEV: Multi-standard calibration fit deviation

### **Example of Analysis Printout**

```
St 358 Set 4 Diopside (Chesterman)
TakeOff = 40.0 KiloVolt = 15.0 Beam Current = 30.0 Beam Size = 2
(Magnification (analytical) = 4000), Beam Mode = Analog Spot
(Magnification (default) = 3200, Magnification (imaging) = 100)
Pre Acquire String : PB OFF
Post Acquire String : PB ON
```

Twin Lakes, Fresno Co., CA

From Charles Chesterman (Ca Div. Mines)

Number of Data Lines: 5 Number of 'Good' Data Lines: 5

First/Last Date-Time: 10/15/2006 09:32:12 PM to 10/15/2006 09:34:43 PM

WARNING- Using Polynomial Off-Peak correction for al ka

|                            |      |                        |        |
|----------------------------|------|------------------------|--------|
| Average Total Oxygen:      | .000 | Average Total Weight%: | 99.966 |
| Average Calculated Oxygen: | .000 | Average Atomic Number: | 12.226 |
| Average Excess Oxygen:     | .000 | Average Atomic Weight: | 21.656 |
| Average ZAF Iteration:     | 4.00 | Average Quant Iterate: | 2.00   |

Results in Elemental Weight Percents

|       |       |       |       |       |       |      |
|-------|-------|-------|-------|-------|-------|------|
| ELEM: | Si    | Mg    | Ca    | Al    | Ti    | O    |
| TYPE: | ANAL  | ANAL  | ANAL  | ANAL  | ANAL  | SPEC |
| BGDS: | LIN   | LIN   | LIN   | POLY  | LIN   |      |
| TIME: | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 |      |

| ELEM: | Si     | Mg     | Ca     | Al     | Ti     | O      | SUM     |
|-------|--------|--------|--------|--------|--------|--------|---------|
| 20    | 25.955 | 10.999 | 18.658 | .000   | .020   | 44.316 | 99.949  |
| 21    | 25.992 | 11.249 | 18.438 | .008   | .000   | 44.316 | 100.003 |
| 22    | 25.977 | 11.319 | 18.459 | .001   | .000   | 44.316 | 100.071 |
| 23    | 26.011 | 11.255 | 18.652 | .000   | .000   | 44.316 | 100.234 |
| 24    | 25.742 | 11.242 | 18.274 | .000   | .000   | 44.316 | 99.574  |
| AVER: | 25.935 | 11.213 | 18.496 | .002   | .004   | 44.316 | 99.966  |
| SDEV: | .110   | .123   | .162   | .003   | .009   | .000   |         |
| SERR: | .049   | .055   | .072   | .002   | .004   | .000   |         |
| %RSD: | .4     | 1.1    | .9     | 202.1  | 223.6  | .0     |         |
| PUBL: | 25.925 | 11.192 | 18.489 | .016   | .000   | 44.316 | 99.938  |
| %VAR: | .04    | .19    | .04    | -89.21 | .00    | .00    |         |
| DIFF: | .010   | .021   | .007   | -.014  | .000   | .000   |         |
| STDS: | 301    | 160    | 358    | 305    | 22     | 0      |         |
| STKF: | .2520  | .0777  | .1707  | .1278  | .5581  | .0000  |         |
| STCT: | 2518.9 | 780.4  | 1710.4 | 1273.7 | 5563.0 | .0     |         |
| UNKF: | .2099  | .0774  | .1708  | .0000  | .0000  | .0000  |         |
| UNCT: | 2098.3 | 777.2  | 1711.0 | -.7    | -.8    | .0     |         |
| UNBG: | 19.0   | 26.0   | 22.8   | 18.1   | 29.8   | .0     |         |
| ZCOR: | 1.2355 | 1.4487 | 1.0831 | 1.4202 | 1.2051 | .0000  |         |
| KRAW: | .8330  | .9959  | 1.0003 | -.0006 | -.0001 | .0000  |         |
| PKBG: | 112.05 | 30.89  | 76.10  | .96    | .98    | .00    |         |

#### Results in Oxide Weight Percents

| ELEM: | SiO <sub>2</sub> | MgO    | CaO    | Al <sub>2</sub> O <sub>3</sub> | TiO <sub>2</sub> | O       | SUM     |
|-------|------------------|--------|--------|--------------------------------|------------------|---------|---------|
| 20    | 55.528           | 18.240 | 26.106 | .000                           | .034             | .041    | 99.949  |
| 21    | 55.606           | 18.654 | 25.799 | .015                           | .000             | -.071   | 100.003 |
| 22    | 55.575           | 18.770 | 25.827 | .001                           | .000             | -.102   | 100.071 |
| 23    | 55.646           | 18.664 | 26.098 | .000                           | .000             | -.175   | 100.234 |
| 24    | 55.071           | 18.643 | 25.569 | .000                           | .000             | .291    | 99.574  |
| AVER: | 55.485           | 18.594 | 25.880 | .003                           | .007             | -.003   | 99.966  |
| SDEV: | .235             | .204   | .226   | .007                           | .015             | .182    |         |
| SERR: | .105             | .091   | .101   | .003                           | .007             | .081    |         |
| %RSD: | .4               | 1.1    | .9     | 202.1                          | 223.6            | -5933.2 |         |
| PUBL: | 55.463           | 18.560 | 25.870 | .030                           | .000             | -.003   | 99.938  |
| %VAR: | .04              | .19    | .04    | -89.21                         | .00              | -7.23   |         |
| DIFF: | .022             | .034   | .010   | -.027                          | .000             | .000    |         |

#### Results in Atomic Percents

| ELEM: | Si     | Mg     | Ca     | Al   | Ti   | O      | SUM     |
|-------|--------|--------|--------|------|------|--------|---------|
| 20    | 20.036 | 9.812  | 10.093 | .000 | .009 | 60.050 | 100.000 |
| 21    | 20.038 | 10.021 | 9.961  | .006 | .000 | 59.973 | 100.000 |
| 22    | 20.016 | 10.078 | 9.967  | .001 | .000 | 59.939 | 100.000 |
| 23    | 20.027 | 10.014 | 10.064 | .000 | .000 | 59.895 | 100.000 |
| 24    | 19.904 | 10.045 | 9.901  | .000 | .000 | 60.150 | 100.000 |
| AVER: | 20.004 | 9.994  | 9.997  | .001 | .002 | 60.001 | 100.000 |
| SDEV: | .057   | .105   | .079   | .003 | .004 | .100   |         |

|       |      |      |      |       |       |      |
|-------|------|------|------|-------|-------|------|
| SERR: | .025 | .047 | .035 | .001  | .002  | .045 |
| %RSD: | .3   | 1.0  | .8   | 202.1 | 223.6 | .2   |

#### Results Based on Sum of 6 Cations

| ELEM: | Si    | Mg    | Ca    | Al    | Ti    | O     | SUM    |
|-------|-------|-------|-------|-------|-------|-------|--------|
| 20    | 3.009 | 1.474 | 1.516 | .000  | .001  | 9.019 | 15.019 |
| 21    | 3.004 | 1.502 | 1.493 | .001  | .000  | 8.990 | 14.990 |
| 22    | 2.998 | 1.509 | 1.493 | .000  | .000  | 8.977 | 14.977 |
| 23    | 2.996 | 1.498 | 1.506 | .000  | .000  | 8.961 | 14.961 |
| 24    | 2.997 | 1.512 | 1.491 | .000  | .000  | 9.056 | 15.056 |
| AVER: | 3.001 | 1.499 | 1.500 | .000  | .000  | 9.001 | 15.001 |
| SDEV: | .006  | .015  | .011  | .000  | .001  | .038  |        |
| SERR: | .002  | .007  | .005  | .000  | .000  | .017  |        |
| %RSD: | .2    | 1.0   | .7    | 202.2 | 223.6 | .4    |        |

## Raw Data

Click this button to see the count rate data for the sample. It will be either beam-corrected data or the raw count data, depending whether the "Use Beam Drift Correction" button in Analysis Options (Analytical menu) is or is not checked (use a Nominal Beam Current of 1 to see counts displayed in counts per second per nA). Note that the Display Count Intensities Unnormalized To Time (in Analyze!) option in Analysis Options will also affect the intensities displayed in the Analyze! window.

The count data will also be printed to the log window. This information includes the average "AVER", standard deviation "SDEV" and the one sigma (ideal) standard deviation, "1SIG", the sigma ratio (standard deviation divided by one sigma), "SIGR" and the standard error "SERR" and percent relative standard deviation "%RSD" which is the standard deviation divided by the average.

The upper data grid will display the count statistics for the sample averaged data including the standard deviation, one sigma, percent relative standard deviation, minimum and maximum values.

The one sigma values represent a predicted standard deviation based on the count rate and the counting time used for the acquisition. It is calculated by dividing the square root of the count rate by the square root of the average counting time. Therefore when counting at one second count times, the one sigma value will be exactly equal to the square root of the count rate. For sample sets using a 10 second counting time, the one sigma value will be smaller than the simple square root by the square root of 10 or approximately 3.162.

Use the one sigma value to evaluate whether a given average count rate represents a homogeneous set of sample data. Standard deviations larger than the one sigma value indicate the possibility of actual compositional variation within the data set. The percent relative standard deviation is simply the standard deviation divided by the average count rate.

Use the Copy buttons to copy this data as tab delimited data to the Windows Clipboard for pasting into another application. The data will automatically formatted as tab delimited text.

The log window will contain additional information including the stage positions for each point in the sample, the on and off-peak positions (absolute and relative offset), the average on-peak and high and low off-peak count times, and for unknown samples only, the Unknown Count Factor multiplier "UNFAC:" and Maximum Counts "MAXCT:", and finally the Time Dependent Intensity (TDI) Calibration Assignment "VOL#" which is equal to the unknown sample number for the Time Dependent Intensity (TDI) self calibration or equal to another unknown sample number in the case of the assigned Time Dependent Intensity (TDI) calibration.

A new option allows the user to set the Time Dependent Intensity (TDI) element fit type to log-linear (exponential) or to log-quadratic (hyper-exponential). The hyper-exponential fit is useful in cases where the intensity decay is non-exponential. This is usually the case for Na loss in hydrous-alkali glasses where the intensity loss is greater than 50%.

This is followed by the background acquisition type of Mean Atomic Number (MAN), or off-peak (OFF), spectrometer motor number, crystal type, and acquisition order and the MAN or off-peak corrected counts for the sample.

After this, the calculated off-peak counts are also printed out along with the background calculation type (if applicable) of Linear interpolation (LINEAR), high off-peak only (HIGH), low off-peak only (LOW), average of high and low (AVER), slope high (S-HIGH), slope low (S-LOW), exponential interpolation (EXPON) and polynomial (POLY).

### **Summary of Data Printout (standards and unknowns)**

|        |  |
|--------|--|
| ELEM:  | Element symbol (for combined sample setups)  |
| KILO:  | Combined sample setup kilovolts  |
| CURR:  | Combined sample setup beam currents  |
| SIZE:  | Combined sample setup beam sizes   |
| ELEM:  | Element symbols  |
| ONPEAK | On-peak spectrometer position  |
| OFFSET | Spectrometer offset from theoretical peak position (in spectrometer units)                           |
| HIPEAK | Hi-off peak position   |
| LOPEAK | Low off-peak position  |
| HI-OFF | High off-peak offset from on-peak position   |
| LO-OFF | Low off-peak offset from on-peak position  |
| DEAD:  | Deadtime correction (software)   |
| BASE:  | PHA baseline (volts)   |
| WINDOW | PHA window (volts)   |
| MODE:  | PHA mode (0 = integral, -1 = differential)   |
| GAIN:  | PHA gain (arbitrary units)   |
| BIAS:  | PHA detector bias (volts)  |
| ELEM:  | Element symbol   |
| BGD:   | Background type (see below for explanation)  |
| SPEC:  | Spectrometer number  |
| CRYST: | Spectrometer crystal   |
| ORDER: | Spectrometer order of acquisition  |
| ONTIM: | On-peak count time (standards or base time for unknowns)   |
| HITIM: | High off-peak count time (standards or base time for unknowns)                                       |
| LOTIM: | Low off-peak count time (standards or base time for unknowns)  |
| UNFAC: | Unknown count factors (also used for alternating on/off peak acquisition)                            |
| ONTIME | Unknown on-peak count time   |
| HITIME | Unknown high off-peak count time   |
| LOTIME | Unknown low off-peak count time  |
| MAXCNT | Maximum counts for statistics based counting   |
| AGGR:  | Total number of aggregate (duplicate element) channels combined if using Aggregate Intensity option. |
| KILO:  | Operating voltage for each element   |
| ENERGY | Emission line energy   |
| EDGE:  | Edge excitation energy   |
| Eo/Ec: | Oversupply ratio   |
| TDI#:  | Time Dependent Intensity (TDI) calibration unknown sample number (same = self, different = assigned) |
| BLNK#  | Blank correction calibration unknown number for trace element background offset                      |
| BLNKL  | Blank correction calibration value (actual level of element in blank- usually zero) in wt.%          |

APP\*: Use specified Area-Peak-Factor value (not calculated from binary APPFs)  
 INTE: Acquire integrated intensity flag  
 PEAK: Peak spectrometer before acquisition flag  
  
 ELEM: Element symbols  
 XXXX Line number ("G" indicates a good or undisabled line and "B" indicates a bad or disabled line)  
  
 INIT: Integrated intensity initial step size  
 MINI: Integrated intensity minimum step size  
  
 ELEM: Element symbols  
 BGD: Background type (INTEGRA=integrated, EDS=energy dispersive, OFF=off-peak, MAN=mean atomic number)  
 TYPE: Off-peak background type (INTEGRA=integrated, LINEAR=linear fit, AVERAGE=averaged, HIGH=high off-peak only, LOW=low off-peak only, EXPONEN=exponential fit, SLOPEHI=slope high off-peak, SLOPELO=low off-peak, POLYNOM=polynomial fit, NONE=no off-peak correction)  
  
 COEF1: Exponential, Polynomial or Multi-Point (last line only) background fit coefficients  
 COEF2: Exponential, Polynomial or Multi-Point (last line only) background fit coefficients  
 COEF3: Exponential, Polynomial or Multi-Point (last line only) background fit coefficients  
  
 MOTOR: Spectrometer number  
 CRYST: Analyzing Crystal  
 ORDER: Acquisition order of element on spectrometer  
  
 AVER: Average count rate (counts per second per nominal beam)  
 SDEV: Standard deviation  
 1SIG: Predicted one sigma deviation (normalized to count rate to compare to SDEV)  
 SIGR: Sigma ratio  
 SERR: Standard error  
 %RSD: Percent relative deviation (SDEV/AVER \* 100)

### ***Example of Data Printout (standards and unknowns)***

```

St 358 Set 4 Diopside (Chesterman)
TakeOff = 40.0 KiloVolt = 15.0 Beam Current = 30.0 Beam Size = 2
(Magnification (analytical) = 4000), Beam Mode = Analog Spot
(Magnification (default) = 3200, Magnification (imaging) = 100)
Pre Acquire String : PB OFF
Post Acquire String : PB ON
  
```

```

Twin Lakes, Fresno Co., CA
From Charles Chesterman (Ca Div. Mines)
Number of Data Lines: 5 Number of 'Good' Data Lines: 5
First/Last Date-Time: 10/15/2006 09:32:12 PM to 10/15/2006 09:34:43 PM
  
```

#### Stage (or Beam Deflection) Coordinate Positions:

| Z        | X        | Y        | Z        | W        | X        | Y        |          |          |
|----------|----------|----------|----------|----------|----------|----------|----------|----------|
| W        | 20G      | 50.34346 | 50.56176 | 9.200766 | 2.000000 | 21G      | 50.34346 | 50.56176 |
| 9.200766 | 2.000000 |          |          |          |          |          |          |          |
| 22G      | 50.34346 | 50.56176 | 9.200766 | 2.000000 | 23G      | 50.34346 | 50.56176 |          |
| 9.200766 | 2.000000 |          |          |          |          |          |          |          |
| 24G      | 50.34346 | 50.56176 | 9.200766 | 2.000000 |          |          |          |          |

On and Off Peak Positions:

ELEM: si ka mg ka ca ka al ka ti ka  
 ONPEAK 77.6740 107.798 107.519 90.9050 191.217  
 OFFSET -.00002 .000175 .000381 .000046 -.00012  
 HIPEAK 84.8933 113.926 111.802 97.5783 193.696  
 LOPEAK 70.4547 101.670 103.237 84.2318 188.738  
 HI-OFF 7.21930 6.12801 4.28300 6.67330 2.47900  
 LO-OFF -7.2193 -6.1280 -4.2820 -6.6732 -2.4790

PHA Parameters:

|        |       |       |       |       |       |
|--------|-------|-------|-------|-------|-------|
| DEAD:  | 1.00  | 1.00  | 1.00  | 1.00  | 1.00  |
| BASE:  | 1.00  | 1.00  | 1.00  | 1.00  | 1.00  |
| WINDOW | 9.00  | 9.00  | 9.00  | 9.00  | 9.00  |
| MODE:  | 0     | 0     | 0     | 0     | 0     |
| GAIN:  | 32.   | 32.   | 16.   | 64.   | 32.   |
| BIAS:  | 1900. | 1900. | 1760. | 1850. | 1800. |

Last (Current) On and Off Peak Count Times:

|        |       |       |       |       |       |
|--------|-------|-------|-------|-------|-------|
| ELEM:  | si ka | mg ka | ca ka | al ka | ti ka |
| BGD:   | OFF   | OFF   | OFF   | OFF   | OFF   |
| SPEC:  | 1     | 3     | 5     | 4     | 2     |
| CRYST: | TAP   | TAP   | PET   | TAP   | LiF   |
| ORDER: | 1     | 1     | 1     | 1     | 1     |
| ONTIM: | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 |
| HITIM: | 5.00  | 5.00  | 5.00  | 5.00  | 5.00  |
| LOTIM: | 5.00  | 5.00  | 5.00  | 5.00  | 5.00  |

On-Peak (off-peak corrected) or MAN On-Peak X-ray Counts (and Faraday Current):

|        |        |       |        |        |        |        |
|--------|--------|-------|--------|--------|--------|--------|
| ELEM:  | si ka  | mg ka | ca ka  | al ka  | ti ka  | BEAM   |
| BGD:   | OFF    | OFF   | OFF    | OFF    | OFF    |        |
| MOTOR: | 1      | 3     | 5      | 4      | 2      |        |
| CRYST: | TAP    | TAP   | PET    | TAP    | LiF    |        |
| ORDER: | 1      | 1     | 1      | 1      | 1      |        |
| 20G    | 2102.2 | 761.7 | 1726.4 | -.9    | 1.7    | 29.989 |
| 21G    | 2102.6 | 780.0 | 1705.8 | .6     | -.7    | 30.022 |
| 22G    | 2100.8 | 785.0 | 1707.8 | .0     | -2.6   | 29.977 |
| 23G    | 2104.5 | 780.3 | 1725.3 | -2.0   | -1.4   | 29.988 |
| 24G    | 2081.3 | 779.1 | 1689.4 | -1.3   | -.7    | 30.000 |
| AVER:  | 2098.3 | 777.2 | 1711.0 | -.7    | -.8    | 29.995 |
| SDEV:  | 9.6    | 9.0   | 15.4   | 1.0    | 1.6    | .017   |
| 1SIG:  | 14.5   | 8.8   | 13.1   | .0     | .0     |        |
| SIGR:  | .66    | 1.02  | 1.18   | .00    | .00    |        |
| SERR:  | 4.3    | 4.0   | 6.9    | .5     | .7     |        |
| %RSD:  | .5     | 1.2   | .9     | -143.2 | -206.2 |        |

Off-Peak (calculated) X-ray Counts (and Absorbed Current):

|        |        |        |               |         |        |      |
|--------|--------|--------|---------------|---------|--------|------|
| ELEM:  | si ka  | mg ka  | ca ka         | al ka   | ti ka  | BEAM |
| TYPE:  | LINEAR | LINEAR | LINEAR        | POLYNOM | LINEAR |      |
| COEF1: | .0000  | .0000  | .0000134.1732 | .0000   |        |      |
| COEF2: | .0000  | .0000  | .0000         | -2.5453 | .0000  |      |
| COEF3: | .0000  | .0000  | .0000         | .0140   | .0000  |      |
| 20G    | 18.2   | 24.4   | 22.9          | 18.7    | 26.8   | .000 |
| 21G    | 18.9   | 27.0   | 22.6          | 17.3    | 30.6   | .000 |
| 22G    | 21.2   | 26.6   | 22.3          | 16.8    | 32.1   | .000 |
| 23G    | 18.7   | 24.9   | 22.2          | 19.1    | 31.5   | .000 |

|       |      |      |      |      |      |      |
|-------|------|------|------|------|------|------|
| 24G   | 17.9 | 27.3 | 24.1 | 18.3 | 28.2 | .000 |
| AVER: | 19.0 | 26.0 | 22.8 | 18.1 | 29.8 | .000 |
| SDEV: | 1.3  | 1.3  | .8   | 1.0  | 2.2  | .000 |

Raw Hi-Peak X-ray Counts:

| ELEM: | si ka | mg ka | ca ka | al ka | ti ka |
|-------|-------|-------|-------|-------|-------|
| 20G   | 19.9  | 23.9  | 22.0  | 18.8  | 26.3  |
| 21G   | 17.5  | 27.2  | 21.0  | 17.1  | 31.0  |
| 22G   | 21.4  | 26.5  | 22.2  | 17.3  | 32.3  |
| 23G   | 16.6  | 26.5  | 20.8  | 19.0  | 31.4  |
| 24G   | 17.9  | 28.4  | 26.0  | 20.3  | 29.8  |
| AVER: | 18.7  | 26.5  | 22.4  | 18.5  | 30.2  |
| SDEV: | 1.9   | 1.7   | 2.1   | 1.3   | 2.3   |
| 1SIG: | 1.9   | 2.3   | 2.1   | 1.9   | 2.5   |
| SIGR: | 1.01  | .72   | 1.00  | .67   | .95   |

Raw Lo-Peak X-ray Counts:

| ELEM: | si ka | mg ka | ca ka | al ka | ti ka |
|-------|-------|-------|-------|-------|-------|
| 20G   | 16.5  | 25.0  | 23.8  | 20.0  | 27.3  |
| 21G   | 20.2  | 26.8  | 24.1  | 18.6  | 30.2  |
| 22G   | 21.0  | 26.7  | 22.4  | 17.6  | 31.9  |
| 23G   | 20.8  | 23.3  | 23.6  | 20.5  | 31.5  |
| 24G   | 17.9  | 26.3  | 22.2  | 17.6  | 26.6  |
| AVER: | 19.3  | 25.6  | 23.2  | 18.9  | 29.5  |
| SDEV: | 2.0   | 1.5   | .9    | 1.3   | 2.4   |
| 1SIG: | 2.0   | 2.3   | 2.2   | 1.9   | 2.4   |
| SIGR: | 1.01  | .65   | .41   | .69   | 1.00  |

### **Summary of Data Printout (wavescans)**

|        |  |
|--------|--|
| ELEM:  | Element and x-ray symbol   |
| SPEC:  | Spectrometer number  |
| CRYST: | Bragg crystal  |
| CRY2D: | Bragg crystal 2d spacing   |
| CRYK : | Bragg crystal refractive index   |
| ORDER: | Bragg order of diffraction   |
| WVTIM: | Wavescan count time per point  |
| ONPEAK | Nominal on-peak position of element and x-ray                              |
| WAVHI: | High limit of wavescan   |
| WAVLO: | Low limit of wavescan  |
| HI-OFF | Analytical high off-peak position  |
| LO-OFF | Analytical low off-peak position   |
| WIDTH: | Wavescan scan width  |
| VARFLG | Variable step acquisition flag   |
| VARTYP | Variable step type (normal or inverted intensity)                          |
| INISTP | Initial step size for normal variable setp, minimum step size for inverted |
| MINSTP | Minimum step size for normal variable step, initial step size for inverted |
| WAVESP | Quickscan acquisition speed (percent of normal)                            |
| WAVEPT | Number of points per normal wavescans                                      |

Example of Data Printout (wavescans)

Wa 9 wavescan sample

TakeOff = 40.0 KiloVolt = 15.0 Beam Current = 30.0 Beam Size = 2  
 (Magnification (analytical) = 4000), Beam Mode = Analog Spot  
 (Magnification (default) = 3200, Magnification (imaging) = 100)  
 Image Shift (X,Y): -2, 3  
 Pre Acquire String : PB OFF  
 Post Acquire String : PB ON  
 Number of Data Lines: 68 Number of 'Good' Data Lines: 68  
 First/Last Date-Time: 11/01/2009 01:39:37 PM to 11/01/2009 01:42:01 PM

Stage position of first data point:

252G 50.3500 50.5000 9.20000

| ELEM:  | Angstro | fe ka | Angstro | si ka | Angstro | mg ka | BEAM |
|--------|---------|-------|---------|-------|---------|-------|------|
| SPEC:  |         | 3     |         | 2     |         | 4     |      |
| CRYST: |         | LIFH  |         | PETH  |         | TAP   |      |
| CRY2D: | 4.0267  |       | 8.7500  |       | 25.7450 |       |      |
| CRYK : | .00006  |       | .00014  |       | .00218  |       |      |
| ORDER: |         | 1     |         | 1     |         | 1     |      |
| WVTIM: |         | .30   |         | .30   |         | .30   |      |
| ONPEAK | 133.727 |       | 228.075 |       | 107.793 |       |      |
| WAVHI: | 142.176 |       | 234.691 |       | 121.582 |       |      |
| WAVLO: | 125.278 |       | 221.460 |       | 94.0044 |       |      |
| HI-OFF | 8.44899 |       | 6.61551 |       | 13.7886 |       |      |
| LO-OFF | -8.4490 |       | -6.6155 |       | -13.789 |       |      |
| VARFLG |         | -1    |         | -1    |         | 0     |      |
| VARTYP |         | 0     |         | 1     |         | 0     |      |
| INISTP | .200000 |       | .882074 |       | 1.83844 |       |      |
| MINSTP | .063367 |       | .044104 |       | .091922 |       |      |
| WAVEPT |         | 100   |         | 100   |         | 100   |      |

Corrected Wavescan Angstroms and Counts:

|              |      |         |      |         |      |        |
|--------------|------|---------|------|---------|------|--------|
| 252G 2.05875 | 1.71 | 7.33311 | .76  | 11.1546 | .75  | 30.004 |
| 253G 2.05596 | 1.13 | 7.33151 | 1.12 | 11.1295 | .83  | 30.004 |
| 254G 2.05234 | 1.03 | 7.32966 | .97  | 11.1044 | .53  | 30.007 |
| 255G 2.04871 | .80  | 7.32826 | .76  | 11.0784 | .92  | 30.007 |
| 256G 2.04461 | 1.58 | 7.32720 | .94  | 11.0529 | .94  | 29.984 |
| 257G 2.04171 | 1.64 | 7.32561 | 1.01 | 11.0277 | .87  | 29.984 |
| 258G 2.03884 | 1.43 | 7.32416 | 1.37 | 11.0021 | 1.16 | 30.005 |
| 259G 2.03578 | 1.89 | 7.32201 | .68  | 10.9763 | 1.06 | 30.005 |
| 260G 2.03308 | 1.13 | 7.32088 | .71  | 10.9502 | .79  | 30.004 |
| 261G 2.02949 | 1.62 | 7.31977 | 1.18 | 10.9251 | 1.11 | 30.004 |
| 262G 2.02649 | 1.87 | 7.31782 | 1.14 | 10.8995 | .96  | 29.991 |
| 263G 2.02370 | 1.50 | 7.31643 | 1.22 | 10.8735 | .62  | 29.991 |
| 264G 2.02056 | 1.47 | 7.31496 | .94  | 10.8479 | .68  | 30.026 |
| 265G 2.01738 | 1.82 | 7.31376 | .58  | 10.8230 | .90  | 30.026 |
| 266G 2.01455 | .98  | 7.31244 | .93  | 10.7973 | .49  | 29.976 |
| 267G 2.01082 | 1.39 | 7.31072 | .99  | 10.7722 | 1.20 | 29.976 |
| 268G 2.00766 | 1.62 | 7.30944 | .87  | 10.7461 | 1.10 | 29.993 |
| 269G 2.00469 | 1.81 | 7.30783 | 1.48 | 10.7211 | .78  | 29.993 |
| 270G 2.00184 | .91  | 7.30621 | .95  | 10.6954 | .80  | 30.019 |
| 271G 1.99787 | 1.56 | 7.30431 | 1.01 | 10.6696 | .39  | 30.019 |
| 272G 1.99483 | 1.77 | 7.30242 | .97  | 10.6442 | 1.07 | 29.992 |
| 273G 1.99191 | 1.80 | 7.30089 | .77  | 10.6186 | .58  | 29.992 |
| 274G 1.98903 | 1.71 | 7.29937 | .75  | 10.5929 | .61  | 29.984 |
| 275G 1.98625 | 1.27 | 7.29781 | 1.03 | 10.5678 | .78  | 29.984 |
| 276G 1.98292 | 1.41 | 7.29629 | .57  | 10.5419 | 1.06 | 30.026 |
| 277G 1.97985 | 1.74 | 7.29541 | .83  | 10.5158 | .97  | 30.026 |

|      |         |      |         |      |         |       |        |
|------|---------|------|---------|------|---------|-------|--------|
| 278G | 1.97695 | 1.56 | 7.29420 | 1.40 | 10.4909 | .78   | 30.012 |
| 279G | 1.97401 | 1.72 | 7.29230 | 1.40 | 10.4646 | .40   | 30.012 |
| 280G | 1.97110 | 1.53 | 7.29027 | .81  | 10.4390 | 1.00  | 29.979 |
| 281G | 1.96800 | 1.10 | 7.28894 | .72  | 10.4138 | .89   | 29.979 |
| 282G | 1.96435 | 1.00 | 7.28743 | 1.34 | 10.3886 | .39   | 30.013 |
| 283G | 1.96068 | 1.38 | 7.28552 | 1.13 | 10.3627 | .61   | 30.013 |
| 284G | 1.95749 | 1.83 | 7.28383 | 1.51 | 10.3369 | .99   | 30.012 |
| 285G | 1.95481 | 1.56 | 7.28200 | .77  | 10.3121 | .71   | 30.012 |
| 286G | 1.95172 | 1.34 | 7.28062 | 1.09 | 10.2863 | .80   | 30.026 |
| 287G | 1.94854 | 1.82 | 7.27870 | 1.17 | 10.2605 | 1.01  | 30.026 |
| 288G | 1.94580 | 1.68 | 7.27695 | 1.41 | 10.2347 | 1.23  | 29.988 |
| 289G | 1.94282 | 1.43 | 7.27532 | .63  | 10.2094 | .42   | 29.988 |
| 290G | 1.93972 | 2.21 | 7.27434 | 1.56 | 10.1837 | .85   | 30.000 |
| 291G | 1.93723 | 1.82 | 7.27228 | 1.30 | 10.1586 | .97   | 30.000 |
| 292G | 1.93440 | 1.23 | 7.27051 | 1.14 | 10.1330 | .41   | 29.981 |
| 293G | 1.93101 | 1.93 | 7.26905 | 1.30 | 10.1073 | .55   | 29.981 |
| 294G | 1.92826 | 1.08 | 7.26715 | 1.10 | 10.0813 | .43   | 29.989 |
| 295G | 1.92464 | 1.75 | 7.26555 | .80  | 10.0565 | .83   | 29.989 |
| 296G | 1.92174 | 1.69 | 7.26388 | 1.39 | 10.0307 | .67   | 30.009 |
| 297G | 1.91895 | 1.22 | 7.26185 | 1.31 | 10.0047 | .82   | 30.009 |
| 298G | 1.91563 | 1.03 | 7.26020 | 1.51 | 9.97945 | 3.33  | 30.013 |
| 299G | 1.91190 | 1.44 | 7.25840 | 1.49 | 9.95364 | 11.46 | 30.013 |
| 300G | 1.90880 | 1.36 | 7.25616 | 1.24 | 9.92876 | 33.55 | 29.982 |
| 301G | 1.90556 | 1.10 | 7.25461 | 1.04 | 9.90268 | 69.58 | 29.982 |
| 302G | 1.90197 | 1.58 | 7.25264 | .76  | 9.87726 | 67.81 | 29.988 |
| 303G | 1.89899 | 1.42 | 7.25125 | .78  | 9.85166 | 38.16 | 29.988 |
| 304G | 1.89584 | 1.09 | 7.24985 | 1.09 | 9.82617 | 11.24 | 30.002 |
| 305G | 1.89231 | 1.47 | 7.24845 | .78  | 9.80051 | 3.29  | 30.002 |
| 306G | 1.88919 | .78  | 7.24717 | 1.60 | 9.77499 | 1.15  | 29.998 |
| 307G | 1.88503 | 1.21 | 7.24486 | .98  | 9.74971 | .51   | 29.998 |
| 308G | 1.88156 | 1.35 | 7.24331 | 1.18 | 9.72408 | 1.04  | 29.996 |
| 309G | 1.87830 | 1.93 | 7.24129 | .74  | 9.69828 | .79   | 29.996 |
| 310G | 1.87561 | 1.74 | 7.23978 | 1.04 | 9.67259 | .96   | 30.026 |
| 311G | 1.87275 | 1.08 | 7.23803 | 1.14 | 9.64764 | .89   | 30.026 |
| 312G | 1.86923 | 1.30 | 7.23615 | .62  | 9.62184 | .82   | 30.013 |
| 313G | 1.86585 | 1.82 | 7.23518 | 1.59 | 9.59642 | .66   | 30.013 |
| 314G | 1.86304 | 1.56 | 7.23304 | .78  | 9.57034 | .36   | 29.985 |
| 315G | 1.85994 | 1.51 | 7.23158 | 1.22 | 9.54516 | 1.06  | 29.985 |
| 316G | 1.85688 | 1.61 | 7.22994 | .83  | 9.51917 | .96   | 30.023 |
| 317G | 1.85397 | 1.25 | 7.22854 | .83  | 9.49342 | .75   | 30.023 |
| 318G | 1.85063 | 1.64 | 7.22704 | .81  | 9.46877 | .80   | 30.018 |
| 319G | 1.84761 | .79  | 7.22553 | 1.27 | 9.44309 | .58   | 30.018 |

## KRaws

Click this button after performing a quantitative analysis and the raw k-ratios will be displayed in the Analyze! grid. This button is disabled until an analysis is performed.

The raw k-ratio is defined as the unknown count rate divided by the standard count rate. The calculation uses background corrected count rates.

Note that Probe for EPMA also corrects the raw k-ratios for MAN backgrounds, on-peak interferences, APF (Area Peak factors) and Time Dependent Intensity (TDI) element corrections. This provides very useful information for importing into other recalculation programs (film thin analysis software for example). There is also a special output option in the Output menu for raw k-ratio export to an ASCII file format. This format can be imported by STRATAGem thin film processing software.

Use the Copy buttons to copy this data as tab delimited data to the Windows Clipboard for pasting into another application.

### **Example of K-Raw Output**

| Line | si ka Off | fe ka Off | mg ka Off | ca ka Off | al ka Off |
|------|-----------|-----------|-----------|-----------|-----------|
| 41 G | .50845    | .00100    | .16490    | .99360    | -.00042   |
| 42 G | .51184    | .00054    | .16137    | 1.00339   | .00007    |
| 43 G | .50878    | .00065    | .16337    | 1.00663   | .00027    |
| 44 G | .50874    | .00076    | .16369    | .99344    | .00098    |
| 45 G | .50920    | .00057    | .15963    | 1.00294   | .00036    |

### **Analyze Selected Line(s)**

To analyze a subset of the data lines in the selected sample, first select the lines in the data grid and then click this button. The results, average and statistics of only the selected lines will be calculated and displayed even if they were previously disabled.

The sample lines not selected and analyzed are temporarily flagged as disabled and are therefore eliminated from the analytical calculations and averages.

### **Excel**

This button sends the currently displayed data in the Analyze! Window to an Excel spreadsheet if a link has been opened from the Output menu (in the main Log Window). The button is disabled if no Excel link is currently open.

One may also automatically send data to a spreadsheet when the data is acquired by selecting the Use Automatic Analysis and the type of data in the Acquisition Options dialog in the Acquire! Window.

### **Combine Selected Samples**

This button will combine the currently selected samples into a single sample and calculate the matrix corrections as though they were a homogenous composition. This is useful if the multiple sample setups option was used in the Automate! window and the major and trace elements were acquired separately. It can also be used to demonstrate the effect of non-homogenous samples.

### **Pause Between Samples**

This check box will force the program to wait for the user to click Next for each sample, when more than one sample is selected in the Analyze! List for data output or analysis.

### **Use All Matrix Corrections**

This check box will calculate the composition of the selected sample(s) using all available matrix corrections, output the detailed results to tab-delimited files, optionally export to Excel and summarize the averages to the log window for a quick comparison. This calculation will be performed with the currently selected mass absorption coefficient table and empirical MAC selections.

This option is not available for Bence-Albee and calibration curve calculations. Currently the ZAF and Phi-Rho-Z selections utilized for this feature are:

- 1 = "Armstrong/Love Scott (default)"
- 2 = "Conventional Philibert/Duncumb-Reed"
- 3 = "Heinrich/Duncumb-Reed"
- 4 = "Love-Scott I"
- 5 = "Love-Scott II"
- 6 = "Packwood Phi(pz) (EPQ-91)"

- 7 = "Bastin (original) Phi(pz)"
- 8 = "Bastin PROZA Phi(pz) (EPQ-91)"
- 9 = "Pouchou and Pichoir - Full"
- 10 = "Pouchou and Pichoir - Simplified"

## **Disable Selected Sample(s)**

Click this button to disable the currently selected samples. The program will flag each data line in the sample as "B" for bad. This will prevent the data in this sample from being used in any analytical calculation or calibrations (if the sample is a standard).

However, if the Analyze Selected Lines option is used, even the disabled lines will be analyzed if they were selected using this option.

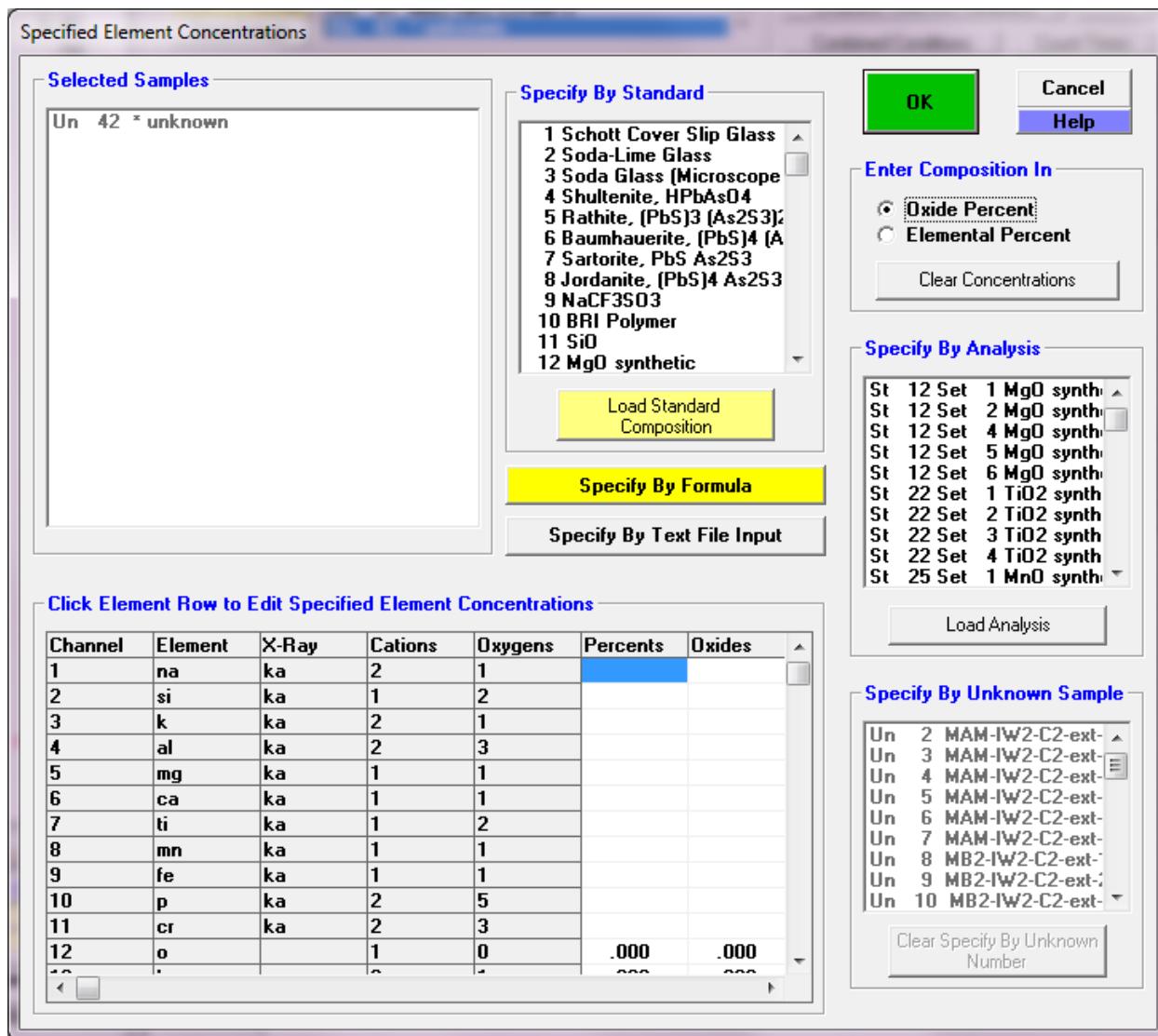
Any disabled line or sample can be enabled by using the Enable buttons described below. An enabled line is displayed with a "G" flag for good.

## **Enable Selected Sample(s)**

Click this button to enable the currently selected samples. The program will flag each data line in the sample as "G" for good. This will allow the data in this sample to be used in any analytical calculation or calibrations (if the sample is a standard).

## **Specified Concentrations**

Probe for EPMA allows the user to specify concentrations of elements not analyzed for in the following dialog:



Consider that in order for the program to compute matrix corrections properly, the program must know the concentration of all significant element concentrations. This means that the program should be able to compute a total of at least 90-95% to calculate matrix effects properly. Element concentrations which you have previously measured or estimated but are not analyzing for, can be manually specified.

This feature is especially useful when adding excess oxygen from oxide (T-fO<sub>2</sub>) ferrous-ferric calculations or H<sub>2</sub>O for hydrous phases. The effect of absorption by oxygen upon Si and Al x-rays is not insignificant and must be considered for the most accurate results.

To specify the concentration of an element not analyzed for in an unknown sample (standard samples will have elements not analyzed for (declared but not acquired), loaded automatically from the standard database), first select the samples to assign the specified concentrations to from the Analyze! sample list box.

First make sure the element(s) to be calculated as a specified element is/are in the sample setup by clicking the Elements/Cations button. If the element is not in the sample element list, add it as a specified element by clicking any empty element row and typing the element in the element field. Be sure to select an x-ray line of none by clicking a blank x-ray line. Then click OK or type <enter> to accept the element.

Then go back to the Specified Concentrations button and click the specified element. Enter the composition and click OK or type <enter>.

Remember that the specified concentrations for unknown samples will be automatically loaded into each new unknown sample that is created based on the last unknown sample.

### **Specify By Standard**

This option allows the user to select a standard composition from the standard database for loading of specified element concentrations. For example, if the sample was a standard but acquired as an unknown.

### **Specify By Formula**

This options allows the user to specify a formula for the specified elements. For example if one is analyzing Ti in quartz, but not analyzing Si, the user can specify SiO<sub>2</sub> as the matrix for the specified elements.

### **Specify By Analysis**

The option allows the user to specify a sample to load the specified concentrations from. The specified element concentrations are loaded based on the current specified concentrations already specified in the selected sample.

### **Specify By Unknown Sample**

This option allows the user to specify an unknown sample in the run to load the specified concentrations from. The program will automatically update the specified concentrations in the selected unknown each time the sample analysis is performed. This is useful if the specified concentrations are calculated during the analysis, for example an element calculated by stoichiometry.

### **Specify By Text File Input**

This button allows the user to select a tab delimited text file to load specified concentrations, for example from ICP-MS analyses. The user can then select the sample line from the text file and the program will automatically load all the element concentrations (in elemental weight percent only).

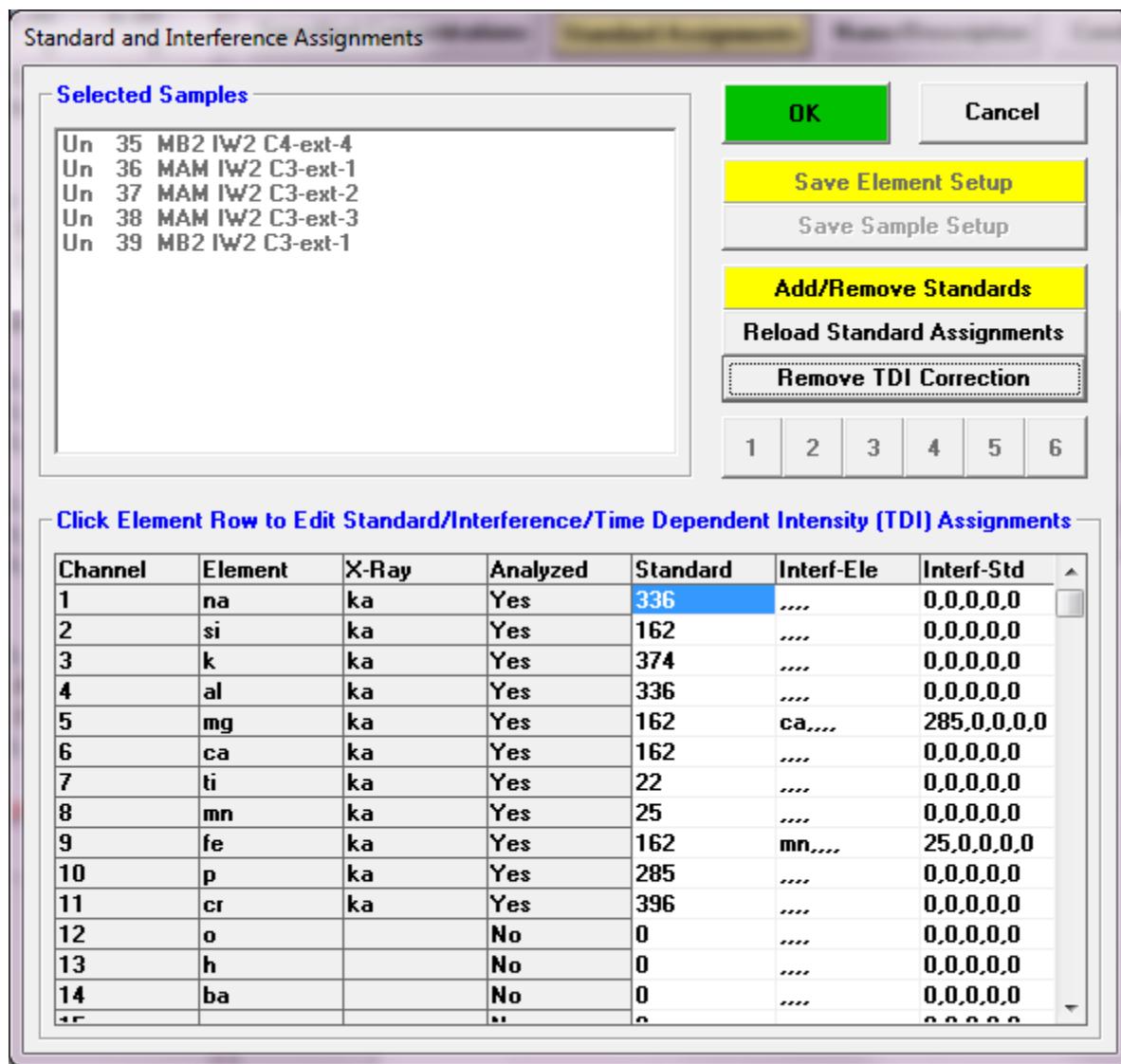
A sample tab delimited file for loading specified concentrations is shown here.

| %          | Si     | As     | La     | Ce     | Pr     |
|------------|--------|--------|--------|--------|--------|
| NYPK6-1.1  | 0.2308 | 0.0000 | 0.0112 | 0.1193 | 0.0506 |
| NYPK6-1.2  | 0.2784 | 0.0000 | 0.0078 | 0.0822 | 0.0343 |
| NYPK6-10.1 | 0.2285 | 0.0000 | 0.0131 | 0.1216 | 0.0474 |
| NYPK6-10.2 | 0.2950 | 0.0000 | 0.0080 | 0.0826 | 0.0354 |
| NYPK6-2.1  | 0.2499 | 0.0000 | 0.0070 | 0.0760 | 0.0329 |
| NYPK6-2.2  | 0.3949 | 0.0002 | 0.0161 | 0.1672 | 0.0621 |
| NYPK6-3.1  | 0.2743 | 0.0001 | 0.0069 | 0.0756 | 0.0323 |
| NYPK6-4.1  | 0.2189 | 0.0000 | 0.0129 | 0.1334 | 0.0504 |
| NYPK6-4.2  | 0.2409 | 0.0000 | 0.0102 | 0.1117 | 0.0469 |
| NYPK6-4.3  | 0.2423 | 0.0000 | 0.0169 | 0.1679 | 0.0649 |
| NYPK6-5.1  | 0.2540 | 0.0000 | 0.0077 | 0.0822 | 0.0352 |
| NYPK6-5.2  | 0.2525 | 0.0000 | 0.0118 | 0.1097 | 0.0426 |

Note that the elements can be in any order, but the first column must contain the sample names, and the first row of the file must contain the element symbols.

## **Standard Assignments**

The standard assignments dialog is used to select the primary standard for the calibration of the quantitative analysis. Each analyzed element must have a standard assigned to it. This dialog is also used to specify the quantitative correction of on-peak interferences and the Time Dependent Intensity (TDI) element correction.

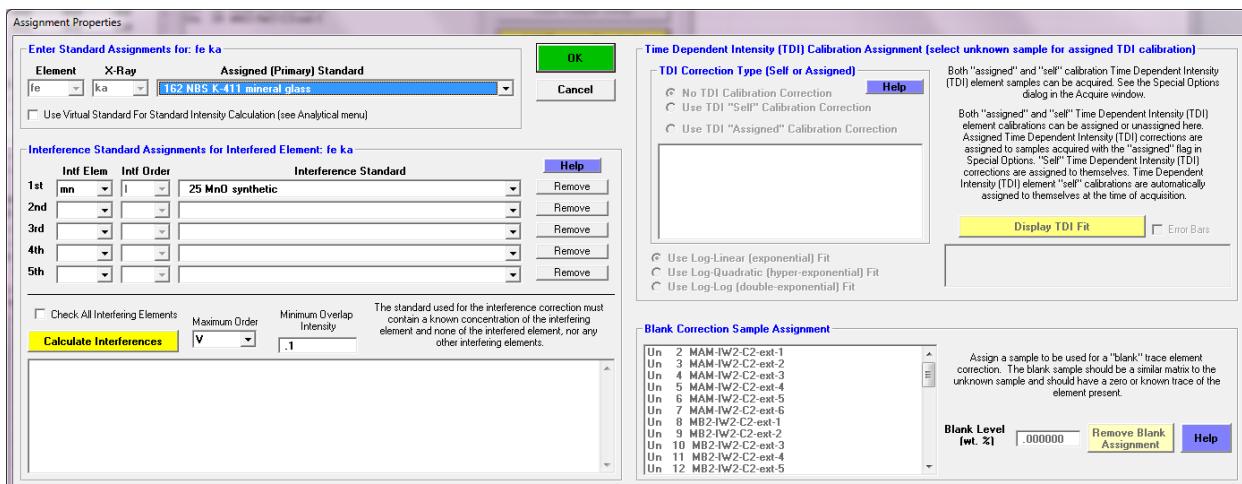


Remember that although the program will automatically load the standard assignments based on the last unknown sample, the standard assignments may be assigned at any time on a sample by sample basis, e.g. for post-processing of data.

The program will load a default set of standard assignments the first time this dialog is opened. These default standard assignments are simply based on the standard containing the highest concentration of each element. It will sometimes be necessary to modify these defaults standard assignments depending on the analytical situation.

Generally when assigning standards for use in the analytical calibration, it is best to use primary standards which contain a major concentration of the element. Standards not assigned but also containing the element are secondary standards and are valuable for checking the quality of the calibration. This can be seen by examining the "%VAR" line in the analysis printout, if they have been acquired as standards, not as unknowns.

Although standards can be reassigned at any later time, the user should be aware that the use of "quick" standards may restrict one's ability to reassign standards off line since the required data may not have been acquired. When running connected to the microprobe (on line) however, you can rerun the affected "quick" standards after changing the standard assignments to obtain the necessary calibration data.



Users may also create subsets of the same elements to acquire on several standards, permitting maximum flexibility. Say you have a sample with feldspars and pyroxenes, and your complete element list holds Na, Mg, Al, Si, K, Ca, Ti, Cr, Mn, and Fe, and you have several silicate standards: albite, microcline, anorthite, wollastonite, enstatite, forsterite, fayalite (as well as others for Ti, Cr and Mn). It would take too long to acquire all elements on all standards, but you don't want to run quick standards either, as you'd like to acquire several elements on multiple standards for maximum flexibility. After peaking all elements and checking pulse height settings, you would save an "all elements" sample setup. Then create a new unknown, feldspar, and delete the Mg, Ti, Cr, and Mn. Save this sample setup as "feldspar". Then create a new unknown, this time recalling the "all elements" sample setup, this time deleting the K and Cr, and saving this as "pyroxene". You could then later "tag" the appropriate standard with the appropriate list of elements, under Automate!.

### ***Virtual Standard Intensities***

A new option is the use of "virtual" standard intensities. These are calculated intensities based on interpolation and matrix corrected similar (atomic numbers) elements measured on the same crystal, spectrometer and voltage as the unknowns. This may be useful in cases where a physical standard is not available (e.g., Ar).

See the Calculate Virtual Standard Intensity menu under the Analytical menu for the interactive dialog for use in calculating and storing these parameters.

### ***Spectral Interferences***

Probe for EPMA allows the user to select a fully quantitative correction for spectral interferences. To help inform the user of possible on-peak interferences, the program will give the user the option of displaying all possible interferences based on the current set of analyzed and interfered elements. However, remember that the program can only correct for interferences if both the interfered and interfering element intensities are acquired. In addition, an interference calibration standard must be acquired that contains a major concentration of the interfering element and none of the interfered element or any other elements that interfere with the interfered element.

The interference correction used by Probe for EPMA is similar to that used by some investigators (Snetsinger, Bunch and Keil, 1968), but is rigorously quantitative and more general, and can be applied easily to the analysis even if the spectral line type of the interfering element is not known. In the case where duplicate elements (but different x-ray lines) have been specified by the analyst (for example Zn ka and Zn la with Na), either channel may be assigned for the interference correction. It really doesn't make any difference to the interference correction except for the fact that the matrix correction will be inaccurately calculated if the duplicated element is present at a significant concentration causing the total to be excessively high. In the case where both the interfering element and x-ray line are duplicated within a single sample (for example Zn ka and Zn ka with Na), the analyst must use the "Disable Quant" feature in the Elements/Cations dialog to temporarily remove the duplicated element and x-ray from the analysis.

To specify an interference simply select the element that is interfered with from the Standard and Interference Assignments window and select the interfering element from the element list of analyzed elements. If the interfering element is not already an analyzed element, it must be added to the sample setup using the Elements/Cations button in the Acquire! window. Next select a standard to be used for the interference calibration from the list of standards in the run. If the standard is not currently in the run, it must be added to the run using the Standards | Add/Remove Standards To/From Run menu item and have data acquired for it.

Standards which may be used in the interference corrections should not be run as "quick" standards unless they have already been assigned for the unknown sample. Data for the interfered channel is necessary to calibrate the correction which may not be present with a "quick" standard. If you find that you need to apply an interference correction which has already been run as a "quick" standard, you can simply re-run the standard again, this time though **not** as a "quick" standard. It is not necessary to disable the previous "quick" standard sample as the program will automatically find the necessary intensities.

**Note that once a sample setup has had interferences assigned, the program will automatically acquire the necessary intensity data even if the quick standard option has been selected.**

As an example of the interference assignment, consider the following : when analyzing for iron and manganese, you may notice that when high concentrations of manganese are present, that small amounts of iron will also appear to be present due to an interference of Mn K $\beta$  with the Fe K $\alpha$  analytical line. To correct for this interference you need a standard that contains manganese but does not contain any iron. Often, you can assign the same standard that is used for the primary standard calibration, also for the interference calibration, in this case, a MnO synthetic crystal. You could also use any other manganese standard not containing appreciable iron such as a synthetic manganese silicate.

Note that if you needed to analyze for Fe, but Mn was not present in any significant quantity, you could justifiably ignore the interference, since you would never see it. What can be considered an insignificant interference is something that needs special consideration.

One useful suggestion is to use either the Standard program's Interference menu item or, even better, use the Interferences button in the standard assignment dialog to provide an approximate calculation of the magnitude of the interference. The program will calculate the nominal interference based on a Gaussian peak shape by assuming a worst case of 0.1% of the analyzed element and 100% of each of the other analyzed (and possibly interfering elements).

Note that in the above case, Mn interferes with Fe, but the reverse is not true : Fe does not interfere with Mn. However in some cases both elements interfere with each other. Probe for EPMA can also handle this situation because the interference correction is an iterated solution. For example : consider the case of Ti K $\alpha$  and Ba L $\alpha$ . Both lines interfere with each other and both lines are used for quantitative analysis. To correct for this double Ti and Ba interference, the analyst will be required to analyze for both Ti and Ba. In addition, two interference standards will be required. To correct for the interference on Ti, a standard will be needed that contains Ba, but no Ti. To correct for the interference on Ba, a standard will be needed that contains Ti, but no Ba. In practice, if you used TiO<sub>2</sub> for the Ti and barite (BaSO<sub>4</sub>) for Ba, as the analytical standards, you can also use Barite as the Ti interference standard and TiO<sub>2</sub> as the Ba interference standard.

The actual form of the quantitative interference within the ZAF iteration loop is given below :

$$C_A^u = \frac{C_A^s}{[ZAF]_{\lambda_A}^s} [ZAF]_{\lambda_A}^u \frac{I^u(\lambda_A) - \frac{[ZAF]_{\lambda_A}^{\bar{s}}}{C_B^{\bar{s}}} \frac{C_B^u}{[ZAF]_{\lambda_A}^u} I_B^{\bar{s}}(\lambda_A)}{I_A^s(\lambda_A)}$$

Where the following notation has been adopted :

$C_i^j$  is the concentration of element i in matrix j

|                       |  |
|-----------------------|--|
| $[ZAF]_{\lambda_i}^j$ | is the ZAF (atomic #, absorption and fluorescence) correction term for matrix j (Z and A are for wavelength $\lambda_i$ and F is for the characteristic line at $\lambda_i$ for element i) |
| $I_i^j(\lambda_i)$    | is the measured x-ray intensity excited by element i in matrix j at wavelength $\lambda_i$ .   |

$\bar{s}$  refers to an interference standard which contains a known quantity of the interfering element B, but none of the interfered with element A.

The quantitative interference corrections in Probe for EPMA allow the user to correct for up to 5 interfering elements per channel. Since the solution is iterated the program can correct for interferences when two (or more) analytical lines interfere with each other. See Donovan, et al., (1993) for more details on the derivation of the quantitative interference correction used here.

*Note: To observe the intermediate interference calculations in your data file, use the VerboseMode menu and click the Analyze button. The program will print out to the log window the interference equations utilized and the values of the interference parameters used in each interference calculation.*

Note also that you can use a standard for the analytical calibration, even if the element channel used for the calibration is interfered with, as long as you correct for the interference as described above. In the case of a standard that contains interfered elements, the correction of the standard counts is simple, since the composition, is by definition, already known from the standard database. Therefore, in the case of standards at least, an iterated procedure is not required.

### **Area Peak Factors and Standard Assignments**

Another consideration in assigning standards concerns the use of Area Peak Factors (APFs) for corrections to effects of peak shift and shape changes. These occur for light element K lines as well as L lines of certain elements, e.g. Co (recall that this is due to involvement of electrons from the valence shell, L shell for K lines, M shell for L lines). These changes are most noticeable on TAP and Pb-stearate, barely if at all with the layered synthetic diffractors. When analysis of the affected elements/lines is to be performed (typically oxygen, nitrogen, carbon and boron but possibly silicon, aluminum and sulfur), you need to plan the use of APF corrections for the most accurate results. The actual APF values selected will depend upon the standard assigned, and also the specific crystal used on the spectrometer.

For example, when analyzing for C K $\alpha$  and using Fe<sub>3</sub>C as your carbon standard, your APF value for C K $\alpha$  in a Cr<sub>7</sub>C<sub>3</sub> matrix might be 0.80. However, if instead, you re-assigned your carbon standard to TiC, you must use APF values relative to TiC rather than Fe<sub>3</sub>C. If they are available in the Empirical APF database (EMPAPF.DAT), just change the APF values for C K $\alpha$ . Otherwise you would need to calculate the Fe<sub>3</sub>C APF values relative to TiC. Some typical Fe<sub>3</sub>C APF values for C K $\alpha$ , recalculated relative to TiC and Cr<sub>7</sub>C<sub>3</sub>, are shown in the table below :

| APF Relative To :              | Fe <sub>3</sub> C (APF/1.000) | TiC (APF/0.868) | Cr <sub>7</sub> C <sub>3</sub> (APF/0.80) |
|--------------------------------|-------------------------------|-----------------|---|
| Fe <sub>3</sub> C              | 1.000                         | 1.152           | 1.25                                      |
| TiC                            | 0.868                         | 1.000           | 1.085                                     |
| Cr <sub>7</sub> C <sub>3</sub> | 0.80                          | 0.92            | 1.00                                      |

### **Checking (Secondary) Standards**

Once the primary standards are assigned it is recommended to analyze the standards to check for internal consistency. For this, the acquisition of secondary standards is preferred. Secondary standards are standards in which no elements (or only some) are not assigned as the primary standard for the analyzed elements.

There are two ways to check your standards, either using the Evaluate utility to graphically view together all standards that have an element acquired, or by viewing the result (in Analyze!) of using the primary standard to analyze a secondary standard.

**The application EVALUATE.EXE runs as a standalone utility, with the MDB file being read in and the user then selecting an element-spectrometer pair. All standards with counts on that pair (e.g. Si Ka-Sp4) will be plotted with the declared element wt.% on the X axis. The user selects the specific type of count data to be plotted on the Y axis, e.g. ZAF-corrected average counts. A set of good standards would then all fall on a straight line through all the points as well as (0,0). This is discussed in more detail elsewhere.**

The other method is to review the secondary standards one by one, in the Analyze! Window. First select all the standards. Then check the Pause Between Sample check box. Then click the Analyze button. The program will pause between each sample analysis to allow the user to view the results. Look over the results carefully. Standards whose x-ray intensities have been assigned as the standard for that element will normally look close to perfect since they are by definition the primary standard. The other unassigned elements are treated as unknowns and by observing the "%VAR" (percent variance) line, one can get an idea of the quality of the standardization by examining these secondary standards.

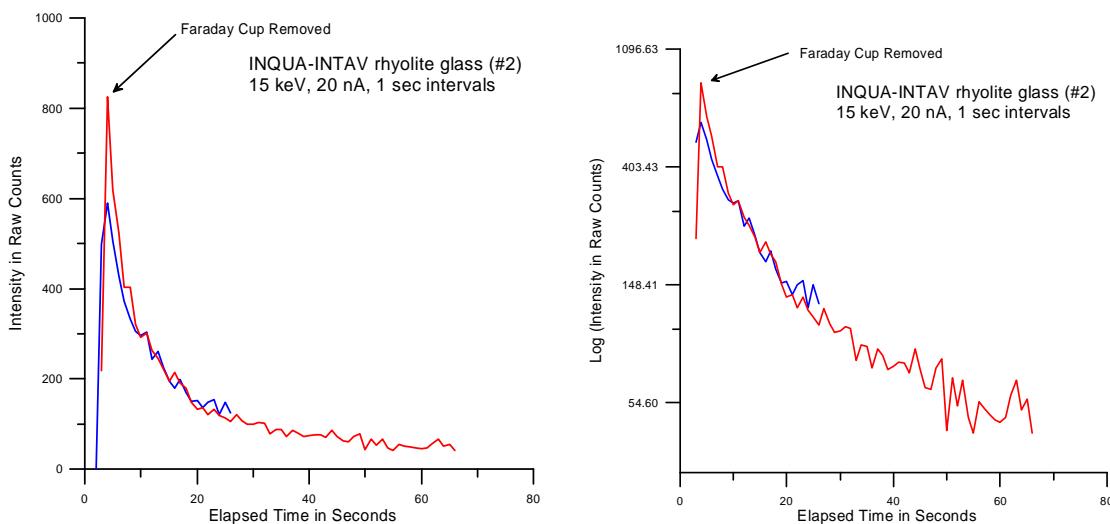
For example, assume that you are analyzing for Si, Al, Na and K and your assigned standards are  $\text{SiO}_2$  for Si,  $\text{NaAlSi}_3\text{O}_8$  (albite) for Al and Na, and  $\text{KAlSi}_3\text{O}_8$  (orthoclase) for K. In this case  $\text{SiO}_2$  is your primary standard for Si, and both albite and orthoclase can serve as secondary standards to check the quality of the Si calibration since they are measured for but not assigned as calibration standards for Si. In addition, the orthoclase standard can be used as a secondary standard in the example above, to check the Al calibration. Therefore, orthoclase is the primary standard for K, but is a secondary standard for both Si and Al, and albite is the primary standard for Al and Na, but is a secondary standard for Si only, while  $\text{SiO}_2$  is the primary standard for Si only. Note that a pure standard such as synthetic  $\text{SiO}_2$  can be used to check the quality of the background correction on the Al, Na and K channels in this example, since pure  $\text{SiO}_2$  should not contain those elements in significant quantities.

While looking over the standard results, look carefully for "bad" points. By this, it is meant points whose total compositions are anomalously low. These could be points where the beam was on epoxy or perhaps the surface was poorly polished. In any case you will see this as a low total. These points may be disabled.

Note that points whose totals are high, on the other hand, should not be disabled arbitrarily. High totals indicate that something else is wrong. Check for low total points on another standard or for the presence of spectral interferences or inclusions.

### ***Time Dependent Intensity (TDI) Element Corrections***

It is well known that measurements of x-ray intensity in glass matrices of certain elements, such as Na and K, show a sharp drop off in count rate under normal beam currents (10 or 20 nA). This has been traditionally referred to as "volatile element" behavior. This is not strictly correct for all of the elements involved, and we will instead introduce and use the phrase Time Dependent Intensity (TDI) element behavior, since current technical explanations suggest electron implantation in insulators (static charge layers) and/or heating cause migration of atoms either deeper into the material, or out of the sample into the vacuum (C. E. Linebarger *J. Am. Chem. Soc.*, 1895, 17 (8), pp 615–652). The intensity loss (or gain) appears to be instantaneous as seen in this glass for 2 separate measurements of Na Ka:

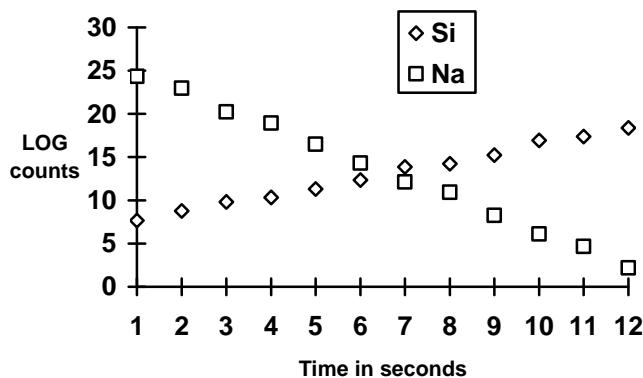


Note however, the slight curvature in the Lin-Log plot above (right hand side). This curvature in log space may indicate two separate exponential processes with different time constants overlapping in time. The “hyper-exponential” TDI correction described below is utilized to deal with this situation.

There are several ways to minimize this effect (lower beam current, defocus beam), but they may not be useful in certain analytical circumstances requiring both sensitivity and high spatial resolution.

Coincident with the effective loss of one element, there may be an increase in x-ray counts of other elements in the material (such as Si and Al), referred to as "grow in" (Morgan and London, 1996) as seen in the plot below. This is probably due to a decreasing matrix absorption effect for less mobile elements such as Si and Al, that, as the more mobile elements such as Na and K migrate to greater depths attracted by the sub-surface electron charge, the emission intensity of the Si and Al increases over time.

The TDI correction generally assumes that the change in counts is linear versus time when the natural LOG of the x-ray counts are plotted (Nielsen and Sigurdsson, 1981) as shown here :



Note that one can significantly reduce the time delay between when the beam is first unblanked and the x-ray counting is started by using the “Synchronous” option in the Acquisitions Option dialog. This is especially useful for the Time Dependent Intensity (TDI) acquisition modes when the sample is extremely sensitive to intensity change during the first few seconds of beam exposure.

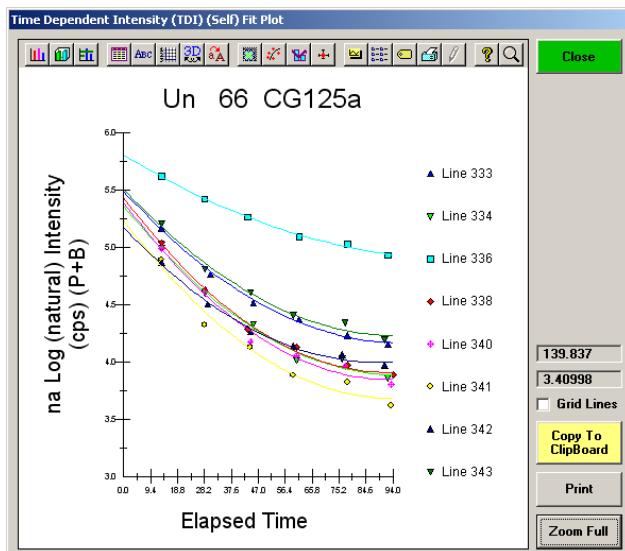
Probe for EPMA offers a **Time Dependent Intensity (TDI)** element extrapolation, whereby a short time series of measurements are made for the element(s) in question. This can be done for both the **Time Dependent Intensity (TDI)**

element and any grow-in elements, as well as for other situations where X-ray intensity changes during beam irradiation, e.g. carbon contamination, beam damage, etc. This correction is useful for samples that are too small to utilize a defocused beam, and allows the user to apply higher beam currents to improve the analytical sensitivity.

Two different methods are utilized by Probe for EPMA to correct for **Time Dependent Intensity (TDI)** element behavior: the first is an assigned method where an unknown sample is acquired and then later assigned for use as a calibration curve for one or more samples. The other is a "self" calibration where intermediate intensities are saved as the data is acquired and later used as the basis for calculating the **Time Dependent Intensity (TDI)** correction.

### **Mathematical Method**

In both cases, the slope coefficient is calculated using a linear fit of intensity versus elapsed time. In this method intensities of different magnitude can be corrected using the same slope (as opposed to using the Y-intercept). Intensities that are less than or equal to zero are ignored. The count data is normalized to counts per second and the natural log taken. The elapsed time is converted to seconds and the linear (straight line) fit is calculated from the midpoint of the elapsed time. Only the slope parameter is saved for the actual **Time Dependent Intensity (TDI)** correction to allow the correction to be applied to a variety of samples that may contain different absolute intensities at different times (due to beam or standard drift) although the third coefficient is utilized for the quadratic exponential **Time Dependent Intensity (TDI)** correction.



This new option allows the user to set the Time Dependent Intensity (TDI) element fit type to log-linear (exponential) or to log-quadratic (hyper-exponential) or even to log-log (double exponential) fit, on an element by element basis. The hyper-exponential fit is useful in cases where the intensity decay is non-exponential. This is usually the case for Na loss in hydrous-alkali glasses where the intensity loss is greater than 50%.

Note that the linear TDI correction utilizes only the slope of the TDI fit in log space. However the "hyper-exponential" TDI correction utilizes a 2<sup>nd</sup> order quadratic fit to the TDI data. While the log-log (double exponential) fit utilizes only the slope fit term similar to the log-linear fit, but with a LOG(elapsed-time) term.

Once the slope is calculated it is used in the following expression to adjust the unknown intensity for **Time Dependent Intensity (TDI)** element loss (or gain):

$$I_C = e^{\log(I_R) - m \cdot t \cdot 0.5}$$

where:  $I_R$  = Raw (uncorrected) intensity in counts per second

|     |   |
|-----|---|
| $m$ | = Time Dependent Intensity (TDI) slope coefficient from linear fit of Log intensity versus time |
| $t$ | = Total elapsed integration time  |

### Typical Situation for Time Dependent Intensity (TDI) Correction

For instance, when sodium loss is observed in an alkali glass sample, a corresponding gain in silicon and aluminum x-rays may be noted. The extrapolation correction used in Probe for EPMA can be applied to some or all elements in an sample, regardless of whether the x-ray intensities are decreased or increased during the acquisition (**as long as the elements to be corrected are acquired as the first element on each spectrometer, i.e., order number = 1**).

Depending on the sample, this may or may not be a valid assumption. Under certain conditions, with very beam sensitive hydrous alkali glasses, the change in count rate may actually decrease more quickly than a simple log decay. In this case, it may be necessary to defocus the beam slightly or reduce the beam current before acquisition.

### How to Use the Time Dependent Intensity (TDI) Correction

To utilize the Time Dependent Intensity (TDI) correction first decide if you require an assigned calibration curve type correction or a self-calibration curve type correction. In the self calibration correction the user simply selects the "Self-Calibration" option (see Special Options button in the Acquire! Window) and acquires the data point(s) as one would normally. In the assigned Time Dependent Intensity (TDI) correction the user first selects a sample that is similar in Time Dependent Intensity (TDI) characteristics to the sample to which the assigned calibration curve will be assigned to (it could be the actual sample itself) and then selects the "Assigned Calibration" option (see Special Options button in the Acquire! Window). The next acquisition that is started will create a calibration curve sample that can be assigned to one or more samples for the purpose of correcting Time Dependent Intensity (TDI) loss or grow-in.

*Note that if the Aggregate Intensity Analysis Option is selected and an element is also using the TDI correction, the slope correction will be applied from that determined by the first channel of the aggregate intensity element.*

### Self-Calibration vs Assigned Calibration

The difference between the two methods is that the assigned calibration curve will apply the same slope correction to all points within the assigned sample. Therefore the variation seen in the (assigned calibration) corrected sample concentrations reflects only the actual variation in the sample. On the other hand, the self calibration method acquires a calibration curve separately for each data point and therefore the variation from point to point may be partially due to statistical variation between the self-calibration curves.

Both of the calibration corrections are quantitative in that the correction is based on a calibration curve acquired during the run, on the sample itself or another sample of similar characteristics, and is applied during the ZAF or Phi-Rho-Z iteration phase of the analysis to correct for changes in the matrix correction due to the extrapolation correction. Any number of calibration samples may be acquired and any sample or range of samples can be assigned to use any of the calibration samples for the extrapolation correction. Note that the a previously assigned Time Dependent Intensity (TDI) element correction is automatically loaded for new unknown samples.

*Note that the correction uses the actual elapsed time for all calculations to produce the best quantitative results. Nevertheless, the user should be aware that the technique must be applied carefully to obtain the best results.*

### Time Dependent Intensity Corrections for Standards

There are two options for acquiring standard counts, if you intend to utilize the TDI procedure for your unknowns. One is to use the normal counting interval on a robust standard where the elements (e.g. Na, K, Al, Si) do not change with time under the beam. However, this might introduce some error, as it introduces conditions different from those used for the unknown. A second and potentially more correct approach would be to use a standard similar to the unknown (e.g. a glass standard, if that is what the unknown is) and acquire counts on it using the TDI method.

### Time Dependent Intensity Corrections and Acquisition Order

Another concern is with samples that contain more than one affected element on a given spectrometer. Probe for EPMA only applies the Time Dependent Intensity (TDI) element correction for elements that are acquired first on a given spectrometer. A user may wish to run some tests on the second element on the spectrometer, comparing the counts to

those acquired if it were the first element, and calculate a correction factor, which could be entered in the "specified APF" field for the element. This would be valid only to the extent that the correction were applied to unknown samples behaving the same as the one the test was performed on.

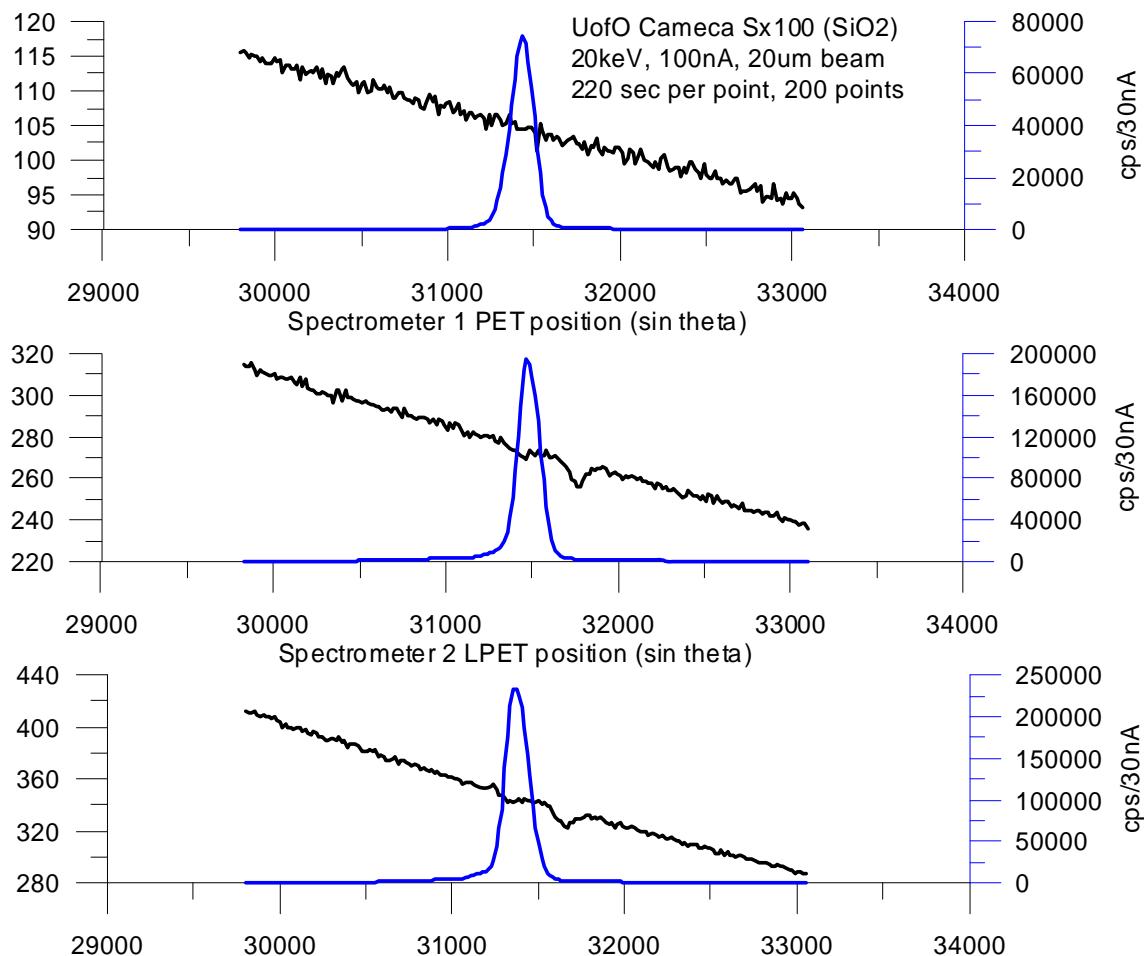
### **Blank Correction**

The blank correction can be used to ensure absolute accuracy when measuring trace element levels close to zero provided a suitable sample exists that is similar to the unknown matrix but contain none of the measured element or a known trace level of the measured element.

Simply acquire an unknown sample on the blank calibration sample, for example when measuring Ti in quartz, one could measure Ti in a synthetic quartz that contains no Ti. From the Standard Assignment dialog, the user simply selects the measured blank calibration sample (using the same conditions if possible) and sets the blank level to zero (the default) in this case and the program will automatically apply the blank correction when the analysis is performed.

The correction is performed in the matrix iteration by calculating the corresponding intensity based on the measured blank offset and this data is used to adjust the background correction. In this way the P/B statistics and k-ratios are always self-consistent.

This correction is necessary because of analyzing crystal artifacts which produce systematic errors in the measurement of peak intensities as seen in the following graphs which contain scans on both SiO<sub>2</sub> and TiO<sub>2</sub> at the Ti Ka peak position. Note that spectrometers 2 and 3 show "holes" in the background which will affect the accuracy of trace element analysis. The blank correction will correct for this systematic error.



The following analysis is of a standard synthetic quartz with a Ti concentration of zero. The blank correction has been applied to itself to demonstrate the validity of the correction by calculating the intensity offset from the measured concentrations and calculating the resulting concentrations (which should be zero). Note the fact that the P/B values are very close to unity.

```

Un      4 SiO2 synthetic
TakeOff = 40.0 KiloVolt = 20.0 Beam Current = 100. Beam Size = 20
(Magnification (analytical) = 8000), Beam Mode = Analog Spot
(Magnification (default) = 600, Magnification (imaging) = 100)
Number of Data Lines: 12 Number of 'Good' Data Lines: 12
First/Last Date-Time: 05/31/2007 04:41:29 PM to 05/31/2007 06:12:21 PM
WARNING- Using Blank Trace Correction
WARNING- Using Alternating On and Off Peak Acquisition
WARNING- Using Polynomial Alpha-Factor Matrix Corrections

Average Total Oxygen:      53.257      Average Total Weight%: 100.000
Average Calculated Oxygen: 53.257      Average Atomic Number: 10.805
Average Excess Oxygen:     .000      Average Atomic Weight: 20.029
Average BET Iteration:    2.00      Average Quant Iterate: 3.00

```

Oxygen Calculated by Cation Stoichiometry and Included in the Matrix Correction

WARNING- Duplicate analyzed elements are present in the sample matrix!!

Use Aggregate Intensity option or Disable Quant feature for accurate matrix correction.

#### Results in Elemental Weight Percents

| ELEM:  | Ti      | Ti      | Ti      | Ti      | Ti      | Si      | O       |                     |
|--------|---------|---------|---------|---------|---------|---------|---------|---------------------|
| TYPE:  | ANAL    | ANAL    | ANAL    | ANAL    | ANAL    | SPEC    | CALC    |                     |
| BGDS:  | LIN     | LIN     | LIN     | LIN     | LIN     |         |         |                     |
| TIME:  | 200.00  | 200.00  | 200.00  | 200.00  | 200.00  |         |         |                     |
| ELEM:  | Ti      | Ti      | Ti      | Ti      | Ti      | Si      | O       | SUM                 |
| XRAY:  | (ka)    | (ka)    | (ka)    | (ka)    | (ka)    | ()      | ()      |                     |
| 421    | .00007  | .00028  | -.00001 | .00074  | -.00022 | 46.7430 | 53.2576 | 100.001             |
| 422    | -.00011 | -.00009 | .00010  | -.00039 | .00001  | 46.7430 | 53.2567 | 99.9992             |
| 423    | .00006  | .00002  | .00019  | .00011  | .00055  | 46.7430 | 53.2576 | 100.002             |
| 424    | .00033  | -.00045 | -.00007 | -.00106 | -.00013 | 46.7430 | 53.2561 | 99.9977             |
| 425    | .00019  | .00021  | -.00013 | .00078  | -.00004 | 46.7430 | 53.2577 | 100.002             |
| 426    | .00000  | .00013  | -.00008 | .00015  | -.00008 | 46.7430 | 53.2571 | 100.000             |
| 427    | -.00023 | .00029  | .00005  | .00076  | -.00061 | 46.7430 | 53.2572 | 100.000             |
| 428    | -.00034 | -.00002 | .00015  | -.00047 | -.00015 | 46.7430 | 53.2564 | 99.9986             |
| 429    | -.00023 | -.00003 | -.00001 | -.00009 | .00022  | 46.7430 | 53.2569 | 99.9998             |
| 430    | -.00010 | .00002  | -.00011 | -.00003 | .00045  | 46.7430 | 53.2572 | 100.000             |
| 431    | .00016  | -.00007 | -.00009 | -.00035 | .00037  | 46.7430 | 53.2570 | 100.000             |
| 432    | .00019  | -.00029 | .00002  | -.00016 | -.00037 | 46.7430 | 53.2566 | 99.9990             |
| AVER:  | .00000  | .00000  | .00000  | .00000  | .00000  | 46.7430 | 53.2570 | 100.000             |
| SDEV:  | .00020  | .00022  | .00011  | .00056  | .00034  | .00000  | .00049  |                     |
| SERR:  | .00006  | .00006  | .00003  | .00016  | .00010  | .00000  | .00014  |                     |
| %RSD:  | ----    | ----    | ----    | ----    | ----    | .0      | .0      |                     |
| STDS:  | 22      | 22      | 22      | 22      | 22      | 0       | 0       |                     |
| STBE:  | 3.4651  | 3.4651  | 3.4651  | 3.4651  | 3.4651  | .0000   | .0000   |                     |
| STCT:  | 57993.6 | 22162.3 | 69644.5 | 3194.7  | 20247.7 | .0      | .0      |                     |
| UNBE:  | 1.1935  | 1.1935  | 1.1935  | 1.1935  | 1.1935  | .0000   | .0000   |                     |
| UNCT:  | .0      | .0      | .0      | .0      | .0      | .0      | .0      | ← blank corrected   |
| UNBG:  | 81.1    | 30.8    | 105.1   | 2.4     | 27.7    | .0      | .0      |                     |
| KRAW:  | .00000  | .00000  | .00000  | .00000  | .00000  | .00000  | .00000  |                     |
| PKBG:  | 1.00002 | 1.00004 | 1.00000 | 1.00079 | 1.00007 | .00000  | .00000  | ← blank corrected   |
| BLNK#: | 4       | 4       | 4       | 4       | 4       | ----    | ----    | ← blank assignment  |
| BLNKL: | .000000 | .000000 | .000000 | .000000 | .000000 | ----    | ----    | ← blank level       |
| BLNKV: | -.00043 | -.00009 | -.00053 | .000039 | .000704 | ----    | ----    | ← blank measurement |

The calculation is performed by calculating the intensity correction for the unknown sample from the quantitative measurement of the blank sample and subtracting that intensity from the measured intensity in the matrix iteration using the following expression:

$$I^U = \frac{[ZAF]^S}{[ZAF]^U} I^S \frac{(C^P - C^L)}{C^S}$$

Where:

- $[ZAF]^S$  is the ZAF correction for the primary standard
- $[ZAF]^U$  is the ZAF correction for the unknown (iterated)
- $I^S$  is the measured standard intensity in the primary standard
- $C^P$  is the measured concentration of the element in the blank sample
- $C^L$  is the blank level concentration of the element in the blank sample (usually zero)
- $C^S$  is the concentration of the element in the primary standard

## Name/Description

Probe for EPMA allows the user to change the sample name and description fields at any time. Use the Insert <cr> button to add a carriage return to the description field for multi-line comments.

The name field is limited to 64 characters and the description field is limited to 255 characters. Any printable characters may be used along with spaces for the name or description of the sample(s).

## Conditions

The conditions dialog can be used to display the sample conditions. These include the operating voltage in kilovolts, the takeoff angle in degrees, the beam current in nA and the beam size in microns. These fields are used for documentation purposes only, unless the hardware interface supports controlling these instrument parameters.

**Note that there are three magnifications defined in Probe for EPMA: a default magnification which is used for normal operation of the instrument between acquisitions, an analytical magnification which is used to acquire standards, unknowns and wavescans and an imaging magnification which is used to acquire images or for use with beam deflection acquisitions.**

## Combined Conditions

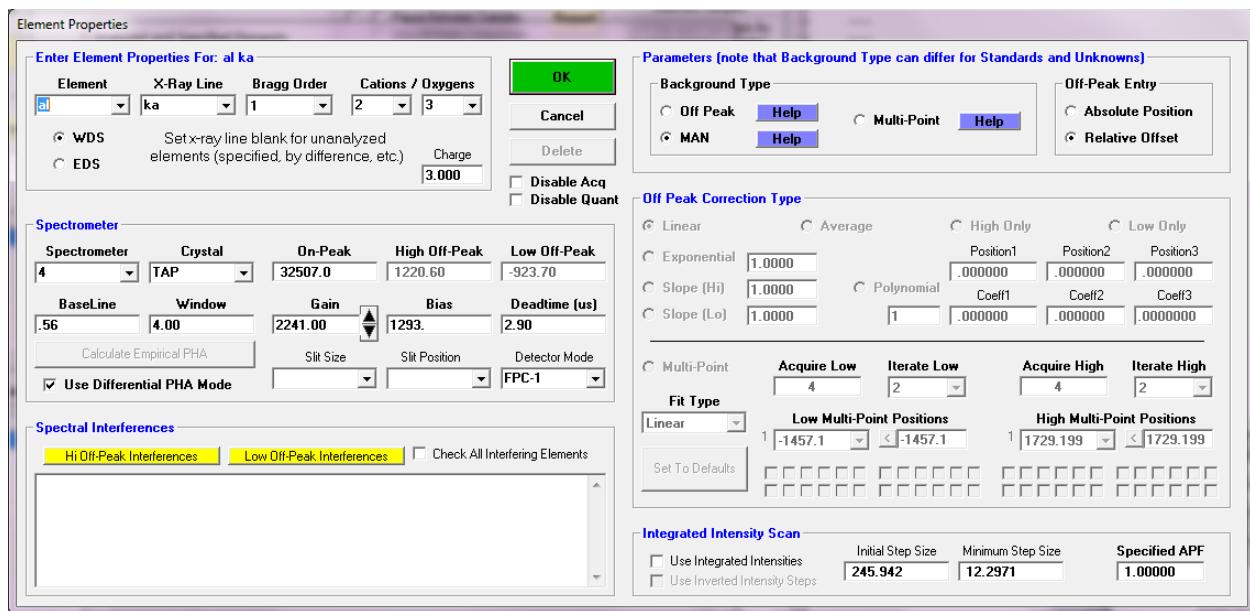
The Combined Conditions dialog allows the user to specify individual analytical conditions (kilovolts, beam current and beam size), if supported by the hardware interface, for each element in the current sample separately. This allows for complete flexibility in the sample setup.

For example, the user may want to run the major elements first at a normal beam current and then run the minor and trace elements at an increased operating voltage and beam current.

The user can also change both the display order and the combined-condition acquisition order of elements (whether the sample contains combined conditions or not). Simply select the element and use the up/down spin button to move the selected element up the acquisition sequence or down. This feature is especially useful when sorting elements by combined conditions for most efficient acquisition. In other words it is best to group elements with the same conditions together.

## Elements/Cations

Clicking the Elements/Cations button opens the analyzed and specified elements dialog box. The analyzed and specified dialog box lists the currently selected elements for analysis and calculations.



Use this button to add specified elements (elements calculated as fixed concentrations or by difference or stoichiometry) to the selected samples. Also the element cations and oxygens can be changed on a sample by sample basis for the calculation of oxide weight percents.

**Use the Save Element Setup button to save element setups to the element setup database for use in future runs. Normally one should save the element setup of an element that is assigned as the standard for that element. In this case the standard intensity data is saved to the element setup database for reference purposes.**

**Use the Save Sample Setup button to save the currently selected sample as a sample setup for use in creating subsequent samples within the current run. Note that selecting a previous sample setup will update the current element setup with the selected sample setup.**

If the element was disabled for acquisition the Disable Acq checkbox will be checked. To uncheck this option for a sample containing data, first create a new sample and then uncheck the Disable Acq option.

To disable the element for quantitative calculations, check the Disable Quant checkbox. The program will set the intensity to zero for the matrix correction calculations. To restore the element for quantitative calculations, simply uncheck the box. Normally this is only used for special reprocessing situations.

From the Analyze! window, only samples with no data can have their acquisition parameters modified. Parameters for samples that contain data can only be viewed. However, the off-peak type and deadtime parameter can be modified in either case. Off-peak correction types include linear, average, high only, low only, and exponential in addition to slope and polynomial fit backgrounds. Plot a wavescan sample(s) in the Plot! Dialog and click the Model Backgrounds button to see the various background options graphically.

Probe for EPMA uses a linear slope calculation as the default off-peak background correction. Because of this, unsymmetrically offset background position measurements can be easily handled. One can even position both of the off-peak measurements on the same side of the peak or even ignore one background measurement if an error is discovered after-the-fact, that one off-peak position was interfered with.

### Off Peak Background Correction Types

Probe for EPMA allows the user to select the off-peak type for the background correction. The default slope calculation is the most accurate, except in cases where the user has moved both off-peak positions to the same side of the analytical

peak and adjusted the off-peaks excessively close together resulting in a large extrapolation. The off-peak correction types are as follows :

0. = linear slope interpolation or extrapolation
1. = average of both high and low off-peak counts
2. = use only the high off-peak counts
3. = use only the low off-peak counts
4. = exponential off-peak calculation (based on exponent)
5. = slope-high (based on high off-peak and slope coefficient)
6. = slope-low (based on low off-peak and slope coefficient)
7. = polynomial (based on high and low off-peak and three coefficients)
8. = multi-point (user defined number of background positions on each side of peak)

These off-peak background correction types may be assigned on a sample by sample basis either on or off-line. Note that the actual values of the polynomial fit coefficients are dependent on the absolute magnitude of the count intensities. For this reason it is important to avoid changing the value of the nominal beam current once any polynomial fit coefficients have been assigned to samples in the run. The calculations for linear slope and exponential calculation are shown below :

### **Linear Slope Calculation**

$$I_{\text{off}} = I_{\text{low}} + (I_{\text{high}} - I_{\text{low}}) \cdot \frac{(P_{\text{on}} - P_{\text{low}})}{(P_{\text{high}} - P_{\text{low}})}$$

where :  $I_{\text{off}}$  = the off-peak count correction

$I_{\text{high}}$  = the x-ray counts for the high off-peak position

$I_{\text{low}}$  = the x-ray counts for the low off-peak position

$P_{\text{high}}$  = the high off-peak position

$P_{\text{low}}$  = the low off-peak position

$P_{\text{on}}$  = the on peak position

### **Exponential Slope Calculation**

$$I_{\text{off}} = \frac{ce^{-aP}}{P^N}$$

where :  $I_{\text{off}}$  = the off-peak background intensity

$P$  = the spectrometer position

$c$  = a fit parameter

$a$  = a fit parameter

$e$  = the exponential

$N$  = a user specified exponent

### **Polynomial Background Source**

' Use polynomial

```
pxdata!(1) = sample(1).LoPeaks!(j%)    ' load three known positions
pxdata!(2) = sample(1).OnPeaks!(j%)
```

```

pxdata!(3) = sample(1).HiPeaks!(j%)

' Get three y values for these positions
pydata!(1) = sample(1).BackgroundPolynomialCoefficients!(1, j%) +
sample(1).BackgroundPolynomialCoefficients!(2, j%) * sample(1).LoPeaks!(j%) +
sample(1).BackgroundPolynomialCoefficients!(3, j%) * sample(1).LoPeaks!(j%) ^ 2
pydata!(2) = sample(1).BackgroundPolynomialCoefficients!(1, j%) +
sample(1).BackgroundPolynomialCoefficients!(2, j%) * sample(1).OnPeaks!(j%) +
sample(1).BackgroundPolynomialCoefficients!(3, j%) * sample(1).OnPeaks!(j%) ^ 2
pydata!(3) = sample(1).BackgroundPolynomialCoefficients!(1, j%) +
sample(1).BackgroundPolynomialCoefficients!(2, j%) * sample(1).HiPeaks!(j%) +
sample(1).BackgroundPolynomialCoefficients!(3, j%) * sample(1).HiPeaks!(j%) ^ 2

' Scale y values to current nominal beam
If UseBeamDriftCorrectionFlag Then
If sample(1).BackgroundPolynomialNominalBeam!(j%) <> 0# And NominalBeam! <> 0 Then
temp! = NominalBeam! / sample(1).BackgroundPolynomialNominalBeam!(j%)
pydata!(1) = pydata!(1) * temp!
pydata!(2) = pydata!(2) * temp!
pydata!(3) = pydata!(3) * temp!
End If
End If

' Re-fit the data using new scaled y values
order% = 2 ' 2nd order polynomial
npts% = 3
Call LeastSquares(order%, npts%, pxdata!(), pydata!(), acoeff!())
If ierror Then Exit Sub

If DebugMode Then
msg$ = "Polynomial re-fit coefficients for " & sample(1).Elsyms$(j%) & " (beam correction = " &
Format$(temp!) & ") " & Str$(coeff!(1)) & Str$(coeff!(2)) & Str$(coeff!(3))
Call IOWriteLog(msg$)
End If

' Calculate off-peak values based on new fit coefficients
temp1! = coeff!(1) + coeff!(2) * sample(1).HiPeaks!(j%) + coeff!(3) * sample(1).HiPeaks!(j%) ^ 2
temp2! = coeff!(1) + coeff!(2) * sample(1).LoPeaks!(j%) + coeff!(3) * sample(1).LoPeaks!(j%) ^ 2

' Calculate offsets from current off-peak measurements
temp3! = sample(1).HiPeakCounts!(i%, j%) - temp1!
temp4! = sample(1).LoPeakCounts!(i%, j%) - temp2!

' Average current offsets
temp! = (temp3! + temp4!) / 2#

' Calculate new y intercept for this sample based on offset
tintercept! = temp! + coeff!(1)

' Calculate actual background using new rescaled coefficients at on-peak position
bgdcts! = tintercept! + coeff!(2) * sample(1).OnPeaks!(j%) + coeff!(3) * sample(1).OnPeaks!(j%) ^ 2
sample(1).BgdData!(i%, j%) = bgdcts!

```

## **Background and Interference Calculations**

Note that the slope and polynomial off-peak corrections are calculated for each sample line based on the actual measured off-peak intensities for the sample. The coefficients can be modeled from the Plot! Window by displaying a wavescan sample in the Graph dialog and clicking the Model Background button.

### **Same Side Off-Peaks**

Beware of placing same-side off-peaks too close together if you are using a background type other than average, high only or low only (for example, the default: linear extrapolation). A large extrapolation can cause a very large error in the off-peak background correction if the background standard deviation is large and using a linear fit. The program will print a warning if it feels that the same-side off-peak extrapolation is excessive. In cases where the same side off-peak extrapolation is excessive, the user may want to select using the average of the off-peak counts. Of course, averaging the off-peak counts is really only applicable in situations where the background slope is zero.

For example, there may be some situations where interferences either above or below the peak require placing both off-peak background measurements on one side. There is nothing wrong with this, as long as "average" is used. A linear extrapolation in many situations, especially when the 2 positions are very close together, will produce an obviously incorrect background model if the background standard deviation is large. The program will print a warning alerting the user to the possibility of possible incorrect background selection. Averaging the off-peak counts is applicable where the background (arithmetic) slope is zero (or equivalent to a "slope-factor" of 1.0).

### **Multi-Point Backgrounds**

There are a number of options to specify for the multi-point background acquisition. The number of points to iterate to and the background fit type (Linear or Polynomial) can be specified before or after the background intensity data has been acquired. Note that the sum of all multi-point intensities is automatically calculated and saved to the normal off-peak intensity arrays so that normal off-peak background calculations can also be performed.

## **Interferences**

The High and Low Off-Peak Interference buttons can be used when both the potential interfering and interfered with elements are already present in the sample setup to determine the approximate magnitude of the off-peak interferences. This calculation is based on a Gaussian peak shape and various crystal defaults and is intended as a guideline only for off-peak position entry.

**The program assumes a nominal concentration of 100% of the interfering element and 1% of the interfered with element in each binary pair calculation. For example, if the percent interference is calculated as 10%, then that indicates that instead of measuring 1 wt. % of the element, it is likely that a concentration of 10% greater than 1 wt. % or 1.1 wt. % will be measured. Note that since this is only a nominal calculation, it should only be used as an indicator of possible problematic analytical situations.**

### **Specified Area Peak Factors (fixed composition APF)**

As an alternative to the "calculated" Area Peak Factors, Probe for EPMA offers the use of "fixed" composition Area Peak Factors. These are single values that are applied to the element intensity on an emitter basis (as opposed to calculated based on the actual absorber concentrations in the matrix).

These factors may be useful in certain situations where the compositional range is limited and binary Area Peak Factors are not available for some or all elements in the matrix (e.g., Na, K or Ca in an oxygen matrix). This option may be turned off or on in the Analysis Option dialog under the Analytical menu.

### **Save Element Setup**

This button opens the element setup database dialog. Here the user can save element setups to the setup database for re-use in another run or sample setup.

Generally, one usually saves standard sample element setups for elements that are assigned to that standard to the setup database. Later, one can retrieve these calibrated setups for use in creating new sample setups for acquisition in another run.

It is recommended that the user save element setups after selecting the primary standard for that element. In this way the element setup database will preserve a record over time of the peak intensities and peak to background ratios for evaluation purposes.

### **Save Sample Setup**

This button activates the Sample Setup dialog box. Here the operator can save sample setups for starting a new sample based on the previous sample setup within the current run.

Subsequently the user can use the Load Sample Setup button in the New Sample dialog (accessed from the Acquire! window) to create new analytical samples or the Setup dialog from the Automate! window when digitizing position samples for automated acquisition.

Note that the saving of a sample setup actually saves only a pointer to the sample selected. This means that any changes to the sample setup calculation options, elements/cations, standard assignments, etc. will be utilized when a new sample is created based on the modified sample setup.

### **Disable Selected Line(s)**

Use this button to *disable* a single data point or a contiguous range of data points (using the mouse to click and drag a range). Any data points thus disabled will be flagged "B" for "bad" and will not be used in any analysis or calculations until it is enabled. Points already disabled are not affected.

**Select the lines to be disabled in the Data grid by clicking and dragging the mouse. Only contiguous ranges of lines of data may be disabled using this feature. Non-contiguous points will have to be disabled individually.**

When analyzing your standards, look carefully for "bad" points. By this, it is meant points whose total compositions are anomalously low. These could be points where the beam was on epoxy or perhaps the surface was poorly polished. In any case you will see this as a low total. These points should usually be disabled. Note that points whose totals are high, on the other hand, should not be disabled arbitrarily. High totals indicate that something else is wrong. Check for low total points on another standard, check for inclusions using BSE, or run a wavescan to look for the presence of spectral interferences both on and off-peak. Off peak interferences usually produce negative k-ratios while on-peak interferences can produce high values (high totals in extreme cases).

### **Enable Selected Line(s)**

Use this button to *enable* a single data point or a range of data points. Any data points thus undisabled will be flagged "G" for "good" and will be used in any analysis or calculations until it is disabled. Points already undisabled are not affected.

**Select the lines to be enabled in the Data grid by clicking and dragging the mouse. Only contiguous ranges of lines of data may be enabled using this feature. Non-contiguous lines must be enabled individually.**

### **List Standard Intensities**

This button will list all standard, background and interference calibration intensities that are associated with the currently selected sample(s) to the log window for inspection. This is useful to determine, for example, the drift in count rates from one standardization to another or the magnitude of the interference overlaps.

Because each sample can be assigned different standard and interference assignments, be sure that the desired sample is selected before clicking this button to see the specific standard and interference standard intensities. This output also includes the measured or calculated background count rates on each standard set.

It is possible to use this button on multiple selected samples, even if they contain different element setups and assignments. In this case, the program will print a complete report for each different element setup selected.

## **Report**

This button generates English sentences that describe the experimental conditions of the selected sample and it's calculation options and analytical sensitivities along with published references when appropriate.

## **Match**

This button opens a modal dialog that allows the user to search the current analyzed composition against a number of search databases. The default search database is DHZ.MDB which contains the compositions within the student edition of Deer, Howie and Zussman's "Rock Forming Minerals". Other databases supplied are SRM.MDB the NIST Standard Reference Materials database and DANA.MDB, the stoichiometric compositions of non-silicate minerals from Dana's Mineralogy.

A recently compiled database is the American Mineralogist "Standard Crystal Structure Database" which is AMCS.DMDB. This latter database contains over 9500 mineral species based on formula stoichiometry and therefore takes some time to search.

One may also search an arbitrary composition by editing a "weight percent" string of elemental compositions. See the Match section under the program Standard for details.

## **Calculation Options**

The following calculation options are available from the Analyze! window. Note that these options do not need to be explicitly set for use from the Plot! window with the exception of the Formula element and basis.

### ***Do Not Use/Use EDS Spectrum Intensities***

If EDS spectrum acquisition data is available for this sample then this option will allow the user to choose whether to use or not use the EDS data stored in the user data file when the matrix correction is calculated. See the Acquisition Options dialog for this acquisition option.

### ***Assign EDS Spectrum Elements***

If EDS spectra were acquired during the sample acquisition, then the program will allow the user to specify elements and x-ray lines to be analyzed for each sample and also to assign primary standards for quantification. Probe for EPMA will apply the full quantitative correction to EDS spectrum net intensities as the WDS intensities (with the exception of deadtime and background continuum corrections). For example, beam drift, standard drift, interferences, etc.

Note that standards for the EDS elements must be acquired in the same manner, using the same analytical conditions (operating voltage) or column conditions as the unknown samples. This merely means that EDS spectrum data must be acquired for any standard that might be required for the assigned standards, just like WDS elements. The simplest way to ensure this is to leave the EDS spectrum acquisition flag on all the time.

This dialog also allows the EDS spectra to be displayed (for each data point in the sample) and also output the spectrum and acquisition parameters) to the file format that the EDS spectrum data was acquired in (Edax, Thermo, Bruker, Oxford, etc.).

### **Lispix Output**

A new capability is the output of Lispix format spectrum image data files including the .RPL parameter file. Because Lispix expects the data in a rectangular array, be sure that samples selected for Lispix output correspond to EDS data acquired from a rectangular array of gridded points. Because the user will need to specify the X and Y grid array coordinate dimensions it will be necessary for the number of data rows and columns be noted, for example in the sample name or description fields, and be sure that the "Stagger The Grid Points", option was NOT used for the digitization of the stage coordinates.

## ***Do Not Use/Use Integrated Intensities***

If integrated WDS intensity data is available for this sample, then this option will allow the user to choose whether to use or not use the stored integrated intensities in the quantitative calculations. See the Elements/cations dialog for this acquisition option.

If this user chooses to use the integrated intensities then the program will calculate the deadtime and beam drift correction for the raw integration data and then calculate the integrated area using cubic spline fit. Finally the program will then calculate the integrated background based on the currently selected background model and subtract that from the integrated intensities. Note that standards acquired using the integrated intensity option must also be acquired for this option.

If the user chooses not to use the integrated intensities then the program will use the peak intensity from the integrated data. Again, for this mode, the standards must still be acquired using the integrated acquisition intensity option and also have the integrated intensities analysis option turned off.

The integrated peak intensity itself is calculated using a cubic spline fit to the data. All off-peak options may be used with the integrated intensity option. The reported integrated intensities are in units of counts per second times spectrometer units divided by the spectrometer unit range (cps per second per spectrometer unit).

If an element is acquired using the integrated intensity option, the standard intensities for that element must also be acquired using the integrated option and preferably using the same off-peak range. See the Run | Display Integrated Intensities menu for a graphical display of the integrated intensity data and cubic spline fitting.

## ***Display Results As Oxides and Calculate With Stoichiometric Oxygen***

Probe for EPMA allows the user to display the results of an analysis in oxide weight percents based on the cation ratios defined for each element. In addition the user may select to calculate oxygen by stoichiometry if oxygen is not an analyzed element. If oxygen is measured or calculated by stoichiometry, the program will automatically calculate the actual excess or deficit oxygen in the analysis if the Display As Oxide option is selected.

**If the element to be calculated by formula basis, difference or stoichiometry is not already in the sample setup, it must be added as a "not analyzed" element using the Elements/Cations button first, by simply clicking any empty row in the element list and typing in an element symbol with a blank x-ray line.**

Note that if the sample being analyzed is a standard and oxygen is not measured, then the program will automatically specify any oxygen from the standard database. However, if the sample is an unknown, then the program will add in any specified oxygen as "excess" oxygen into the matrix correction, if the user has specified any element concentrations.

## ***Formula and Mineral Calculations***

The program can compute formulas based on any number of oxygens for oxide runs or any analyzed or specified element in elemental runs. In addition you may select either an olivine, feldspar, garnet (2 varieties) or pyroxene end-member calculation if you choose a formula based on oxygen.

It should be noted that the olivine, feldspar, garnet and pyroxene formula calculations are based only on atomic weight and do not consider charge balance and site occupancy for the calculations.

New formula calculations based on Jay Ague's AMPHI.F and HALOG.F formula re-calculation source code are now available. Output to log window and also to AMPHI.OUT and HALOG.F files (subsequent calculations are appended automatically to the text file).

### **Amphibole (AMPHI) Output:**

| Sample "468" |         |         |         |         |    |         |
|--------------|---------|---------|---------|---------|----|---------|
| 40.3718      | 5.90139 | 5.87380 | Si      |         |    |         |
| 4.72061      | .518955 | .516500 | Ti      |         |    |         |
| 14.9005      | 2.56756 | 2.55560 | 2.12620 | .429400 | Al | 8.99481 |
| .000000      | .000000 | .215300 | Fe+3    |         |    | 8.93467 |
| 10.9198      | 1.33495 | 1.11340 | Fe+2    |         |    |         |
| 12.8004      | 2.78858 | 2.77550 | Mg      |         |    |         |
| .090387      | .011192 | .011100 | Mn      |         |    |         |
| 10.2995      | 1.61320 | 1.60570 | Ca      |         |    |         |
| 2.60025      | .736983 | .733500 | Na      | .333100 |    |         |
|              |         |         |         | .400400 |    |         |

|         |         |         |         |         |         |         |         |         |         |         |         |         |
|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 2.05022 | .382347 | .380600 | K       |         |         |         |         |         |         |         |         |         |
| .000000 | .000000 | .000000 | F       |         |         |         |         |         |         |         |         |         |
| .000000 | .000000 | .000000 | 2.00000 | .400400 | Cl      |         |         |         |         |         |         |         |
| .000000 | .396690 | .713698 | .159057 | .073786 | .061343 | .001586 | -5.0000 | 2.45870 | 1.67770 | 2.55560 | .781000 | .837962 |
| .333100 | .400400 | 7.00000 | 1.60570 | 0       |         |         |         |         |         |         |         |         |

The first line is the sample number, the next 12 lines contain the following columns of data:

- AL: Wt% oxide, Formula fraction, Normalized fraction, Tetragonal, Octahedral, Element symbol, Press1,
- Press2
- NA: Wt% oxide, Formula fraction, Normalized fraction, M4, M12, Element symbol
- CL: Wt% oxide, Formula fraction, Normalized fraction, 2-(Cl+F), Element symbol
- Other: Wt% oxide, Formula fraction, Normalized fraction, Element symbol

The line (after the space) contains the following variables:

“LOG X-F/X-CL”  
 “LOG(X-MG/X-FE2+)”  
 “MG / (MG + FE2+)”  
 “X-FE2+”  
 “X-TI”  
 “X-ALVI”  
 “X-MN”  
 “LOG(X-F/X-OH)”  
 “LVI+2TI+A-SITE+FE3+”  
 “ALVI+2TI+FE3+”  
 “NORM AI”  
 “A-SITE”  
 “FE2+/(FE2+ + FE3+)”  
 “NORM SI”

The last line contains the following variables:

“NA M4”  
 “NA M12”  
 “TOTAL VI=”  
 “NORM CA”  
 “NCODE”

### Biotite (HALOG) Output:

|              |         |         |         |         |         |         |  |  |  |  |  |  |
|--------------|---------|---------|---------|---------|---------|---------|--|--|--|--|--|--|
| Sample "112" |         |         |         |         |         |         |  |  |  |  |  |  |
| 38.6218      | 2.96880 | Si      |         |         |         |         |  |  |  |  |  |  |
| 2.29024      | .132400 | Ti      |         |         |         |         |  |  |  |  |  |  |
| 10.7209      | .971200 | .971200 | .000000 | Al      |         |         |  |  |  |  |  |  |
| 18.1306      | 1.16540 | Fe+2    |         |         |         |         |  |  |  |  |  |  |
| 14.0110      | 1.60560 | Mg      |         |         |         |         |  |  |  |  |  |  |
| .019589      | .001600 | Ca      |         |         |         |         |  |  |  |  |  |  |
| .690164      | .102900 | Na      |         |         |         |         |  |  |  |  |  |  |
| .110533      | .003300 | Ba      |         |         |         |         |  |  |  |  |  |  |
| 9.21032      | .903100 | K       |         |         |         |         |  |  |  |  |  |  |
| 4.02000      | .977100 | F       |         |         |         |         |  |  |  |  |  |  |
| .020000      | .002600 | 1.02030 | Cl      |         |         |         |  |  |  |  |  |  |
| .950351      | .061900 | Mn      |         |         |         |         |  |  |  |  |  |  |
| 98.7955      | 2.57497 | .139162 | .541463 | .393013 | .044650 |         |  |  |  |  |  |  |
| .000000      | .020875 | -.01879 | -1.5096 | 47.9896 | 53.5200 | 1.01090 |  |  |  |  |  |  |
|              | 2.96530 |         |         |         |         |         |  |  |  |  |  |  |

The first line is the sample number, the next 12 lines contain the following columns of data:

- AL: Wt% oxide, Formula fraction, Normalized fraction, Tetragonal, Octahedral, Element symbol
- CL: Wt% oxide, Formula fraction, Normalized fraction, 2-(Cl+F), Element symbol
- Other: Wt% oxide, Formula fraction, Normalized fraction, Element symbol

The line (after the space) contains the following variables:

“TOTAL WT%”  
“LOG X-F/X-CL”  
“LOG(X-MG/X-FE2+)”  
“X-MG”  
“X-FE2+ “  
“X-TI“  
“X-ALVI“  
“X-MN“  
“LOG(X-F/X-OH)”

The last line contains the following variables:

“X-SID”  
“X-ANN”  
“X-PLOG”  
“BA+CA+MN+NA”  
“TI+OCTA+FE+MG+CL”

### **Calculate Atomic Percents**

The program can compute the analyses in atomic percent also. The program will calculate the results based on atomic weight and normalized to 100%. This calculation is sometimes known also as elemental mole percents.

**Note that this calculation should not be used if the elemental weight percent total indicates a serious problem with the analysis, i.e. less than 98 or more than 102 wt. %.**

### **Element by Difference**

An element by difference can be included in this way also to account for its effect on the x-ray intensities. The element by difference must be one of the specified elements in the sample setup. Note that this option should be utilized carefully, because the calculation will always result in a 100% total which could mask the results of a bad acquisition.

### **Stoichiometry to Oxygen**

Another useful feature for the analysis of carbonate or borate samples in an oxide run is the use of the "element by stoichiometry to the stoichiometric element (oxygen)" option. With this feature the analyst can acquire just the cations (Fe, Mg, Ca, etc.) intensities in a sample and have the oxygen calculated by stoichiometry and another specified element (usually C for carbonates or B for borates) calculated relative to oxygen. In the case of carbonates, for example  $\text{CaCO}_3$ , carbon is always in the ratio 1 to 3 to oxygen. Therefore by simply specifying C by stoichiometry relative to the stoichiometric element (oxygen) at 0.333 (1 divided by 3) the correct amount of both oxygen and carbon will be incorporated into the ZAF matrix correction and totals without analyzing for either. This calculation should only be used with compounds where the ratio to oxygen is both known and unchanging.

The following is an analysis example of carbon calculated by stoichiometry to oxygen in a carbonate sample for an oxide run :

```
Analysis of sample st 135 set 2 calcite (analyzed)
Total Number of Data Points = 5 Number of "G" Data Points = 5
Average Beam Counts = 45527. Average Base Time = 10.00
Average Sample Z-bar = 12.572 Average BNA Iterations = 11.000
Element : C is Calculated Relative to Stoich. Oxygen at .333 to 1.0 Atom
```

Results in Weight Percent :

|      | CO2   | Cl  | MgO | FeO | MnO | O   | SUB   |
|------|-------|-----|-----|-----|-----|-----|-------|
| SPEC | 43.82 | .00 | .00 | .00 | .00 | .00 | 43.82 |
| SDEV | .19   | .00 | .00 | .00 | .00 | .00 |       |
| BGD: | MAN   | MAN | MAN |     |     |     |       |

|      | CaO    | SO <sub>3</sub> | P2O <sub>5</sub> | SUM    |
|------|--------|-----------------|------------------|--------|
| 29   | 55.60  | .01             | -.01             | 99.12  |
| 30   | 56.18  | .04             | -.01             | 100.22 |
| 31   | 56.16  | .01             | .00              | 100.10 |
| 32   | 56.02  | -.01            | -.02             | 99.77  |
| 33   | 56.10  | -.01            | .01              | 100.00 |
| AVER | 56.01  | .01             | -.01             | 99.84  |
| SDEV | .24    | .02             | .01              |        |
| SERR | .11    | .01             | .00              |        |
| PUBL | 56.01  | n.a.            | n.a.             | 99.90  |
| %VAR | .00    | .00             | .00              |        |
|      | *      |                 |                  |        |
| BFAC | 1.0314 | .9925           | 1.0285           |        |
| KRAW | 1.0001 | .0001           | -.0001           |        |
| P/B: | 277.12 | 1.02            | .87              |        |

Note that oxygen **must** be an analyzed or specified element before this calculation can be applied.

One more point about element by stoichiometry to oxygen. Consider the example of a trace element analysis of several metals in an alumina ( $\text{Al}_2\text{O}_3$ ) matrix. If Al and O are not to be analyzed, yet the user desires to have  $\text{Al}_2\text{O}_3$  added to the matrix correction, how can this be accomplished?

There are two ways this can be achieved. One way would be to simply specify Al by difference and calculate oxygen by stoichiometry. The program will then correctly add in the proper amount of stoichiometric  $\text{Al}_2\text{O}_3$  to the matrix correction for each analysis line. The other way is to use the element by stoichiometry to oxygen calculation as discussed below.

Adjust the cation ratios of the metals to elemental stoichiometry (one cation and zero anions). Next, select "element by stoichiometry to oxygen" and (in this example) select "al" as the element by stoichiometry. To achieve a 2 to 3 ratio, next enter ".666" Al atoms per O atom. Run the calculation and note that  $\text{Al}_2\text{O}_3$  was **not** added to the matrix calculation!

What happened? In this example, the user had selected a cation ratio for the analyzed elements of all elemental atoms, and since there was zero oxygen to begin the iteration, the program never got to add the Al which then never added the stoichiometric oxygen! How can this be avoided? Simply specify some small concentration of an oxide element (for instance SiO<sub>2</sub>) in the specified element concentration, say 0.05 %. This will give the iteration a chance to get started, and allow it to converge on a very close approximation of the  $\text{Al}_2\text{O}_3$  by difference! The following is an example of how this calculation looks :

```

Analysis of lines:
 23  24  25  26  27  28  29  30
Total Number of Data Points =   8          Number of "G" Data Points =   8
Average Beam Counts =   98504.           Average Base Time =      10.00
Average Sample Z-bar =   10.816         Average ZAF Iterations =    5.000
Element : Al  is Calculated Relative to Stoich. Oxygen at .666 to 1.0 Atom
Element : O   is Calculated by Stoichiometry

```

Results in Weight Percent :

|      | Se  | O     | Al    | Si  | SUB   |
|------|-----|-------|-------|-----|-------|
| SPEC | .00 | 46.68 | 52.41 | .05 | 99.14 |
| SDEV | .00 | .02   | .02   | .00 |       |
| BGD: | OFF | OFF   | OFF   |     |       |

|      | Zn     | Cu     | Pd     | SUM    |
|------|--------|--------|--------|--------|
| 23   | .59    | .30    | .01    | 100.02 |
| 24   | .56    | .30    | .01    | 100.02 |
| 25   | .57    | .32    | .00    | 100.02 |
| 26   | .58    | .33    | .00    | 100.02 |
| 27   | .53    | .33    | .02    | 100.02 |
| 28   | .55    | .39    | .01    | 100.02 |
| 29   | .54    | .26    | .00    | 100.02 |
| 30   | .56    | .31    | .01    | 100.02 |
| AVER | .56    | .32    | .01    | 100.02 |
| SDEV | .02    | .04    | .01    |        |
| SERR | .01    | .01    | .00    |        |
| KRAT | .0047  | .0027  | .0000  |        |
| ZCOR | 1.2009 | 1.1976 | 1.2321 |        |
| KRAW | .0093  | .0027  | .0000  |        |
| P/B: | 2.70   | 2.29   | 1.02   |        |
| %INT | -.05   | .00    | .00    |        |

Results in Oxide Weight Percents using Custom Cation Oxide Ratios :

|      | Se   | O    | Al <sub>2</sub> O <sub>3</sub> | SiO <sub>2</sub> |
|------|------|------|--------------------------------|------------------|
| AVER | .000 | .000 | 99.029                         | .107             |
| SDEV | .000 | .000 | .040                           | .000             |
|      | Zn   | Cu   | Pd                             | SUM              |
| 23   | .592 | .302 | .006                           | 100.022          |
| 24   | .556 | .300 | .015                           | 100.022          |
| 25   | .574 | .323 | .000                           | 100.022          |
| 26   | .581 | .330 | .000                           | 100.022          |
| 27   | .528 | .332 | .018                           | 100.022          |
| 28   | .550 | .386 | .008                           | 100.022          |
| 29   | .543 | .262 | .001                           | 100.022          |
| 30   | .561 | .308 | .013                           | 100.022          |
| AVER | .561 | .318 | .008                           | 100.022          |
| SDEV | .021 | .035 | .007                           |                  |

### Stoichiometry to Another Element

Yet another option for recalculation is the element relative to another element (by stoichiometry). This calculation is similar to the "element by stoichiometry to oxygen" as described below, however the user may select any other analyzed (acquired) or specified element as the stoichiometric basis element.

This calculation can be used in combination with the other calculations to handle unusual analytical situations. For example, in the following analysis of an silica sample, the results revealed significant contamination of the material with Fe and Ca. Investigation using other techniques, implied that the Fe was in the form of Fe<sub>2</sub>O<sub>3</sub> (hematite), and the Ca was present as CaCO<sub>3</sub> (calcium carbonate). Aside from the effect of inhomogeneity on the matrix correction, it was desirable to improve the calculation by calculating Si by difference and C by stoichiometry to Ca as follows :

```

Analysis of sample un 2 seoj #3 random
Total Number of Data Points = 6 Number of "G" Data Points = 3
Average Beam Counts = 79092. Average Base Time = 10.00
Average Sample Z-bar = 11.480 Average ZAF Iterations = 2.333
Oxygen Calculated by Cation Stoichiometry and Included in Matrix Correction
Element : Si is Calculated by Difference from a Sum of 100.0 Weight Percent

```

Element : C is Calculated Relative to Element Ca at 1.000 to 1.0 Atom

Results in Weight Percent :

|      | O      | Si     | C      | La     | F     | SUB    |         |
|------|--------|--------|--------|--------|-------|--------|---------|
| SPEC | 51.526 | 39.743 | 1.134  | .000   | .000  | 92.403 |         |
| SDEV | 2.932  | 11.897 | 1.939  | .000   | .000  |        |         |
| BGD: | MAN    | MAN    | MAN    | MAN    | MAN   | MAN    |         |
|      | Fe     | Al     | Ca     | Mg     | Mn    | S      | SUM     |
| 133  | .054   | .018   | .012   | .009   | .000  | .000   | 100.000 |
| 134  | 10.696 | .467   | 11.253 | .010   | .054  | .000   | 100.000 |
| 135  | .119   | .013   | .083   | .004   | .000  | .000   | 100.000 |
| AVER | 3.623  | .166   | 3.782  | .008   | .018  | .000   | 100.000 |
| SDEV | 6.126  | .261   | 6.470  | .003   | .031  | .000   |         |
| SERR | 3.537  | .151   | 3.735  | .002   | .018  | .000   |         |
| KRAT | .0306  | .0013  | .0353  | .0001  | .0001 | .0000  |         |
| ZCOR | 1.1992 | 1.2245 | 1.0981 | 1.3804 | .9338 | .8713  |         |
| KRAW | .0451  | .0029  | .2072  | .0001  | .0002 | .0000  |         |
| P/B: | 14.07  | 4.78   | 43.69  | 1.45   | 1.07  | .92    |         |

Results in Elemental Mole Percents :

|      | O      | Si     | C     | La   | F    |      |         |
|------|--------|--------|-------|------|------|------|---------|
| AVER | 65.743 | 28.728 | 2.004 | .000 | .000 |      |         |
| SDEV | 1.571  | 7.841  | 3.430 | .000 | .000 |      |         |
|      | Fe     | Al     | Ca    | Mg   | Mn   | S    | SUM     |
| 133  | .019   | .014   | .006  | .007 | .000 | .000 | 100.000 |
| 134  | 4.069  | .368   | 5.965 | .009 | .021 | .000 | 100.000 |
| 135  | .043   | .010   | .041  | .003 | .000 | .000 | 100.000 |
| AVER | 1.377  | .130   | 2.004 | .006 | .007 | .000 | 100.000 |
| SDEV | 2.332  | .206   | 3.430 | .003 | .012 | .000 |         |

Results in Oxide Weight Percents using Custom Cation Oxide Ratios :

|      | O      | SiO2   | CO2    | La2O3 | F    |      |         |
|------|--------|--------|--------|-------|------|------|---------|
| AVER | .000   | 85.024 | 4.153  | .000  | .000 |      |         |
| SDEV | .000   | 25.452 | 7.104  | .000  | .000 |      |         |
|      | Fe2O3  | Al2O3  | CaO    | MgO   | MnO  | SO3  | SUM     |
| 133  | .077   | .035   | .017   | .014  | .000 | .000 | 100.000 |
| 134  | 15.293 | .883   | 15.745 | .017  | .070 | .000 | 100.000 |
| 135  | .170   | .024   | .116   | .006  | .000 | .000 | 100.000 |
| AVER | 5.180  | .314   | 5.292  | .013  | .023 | .000 | 100.000 |

Note that Fe was recalculated as Fe<sub>2</sub>O<sub>3</sub> and C was added relative to Ca by stoichiometry. This calculation can be applied in numerous other ways to a variety of analytical situations. Consider the example below. Here, the user decides to calculate CO<sub>2</sub> on the basis of moles of CaO, rather than by stoichiometry to oxygen.

```
Analysis of sample st 141 set 1 dolomite (Harvard #105064)
Total Number of Data Points = 3 Number of "G" Data Points = 3
Average Beam Counts = 9102. Average Base Time = 10.00
Average Sample Z-bar = 10.875 Average BNA Iterations = 5.000
CO2 is Calculated Relative to Oxide CaO at 2.000 to 1.0 Mole
```

Results in Weight Percent :

|      | CO2    | SUB    |         |
|------|--------|--------|---------|
| SPEC | 47.791 | 47.791 |         |
| SDEV | .071   |        |         |
| BGD: | MAN    | MAN    |         |
|      | CaO    | MgO    | SUM     |
| 9    | 30.501 | 21.735 | 100.109 |
| 10   | 30.424 | 21.769 | 99.947  |
| 11   | 30.420 | 22.135 | 100.301 |
| AVER | 30.448 | 21.880 | 100.119 |
| SDEV | .046   | .222   |         |
| SERR | .026   | .128   |         |
| PUBL | 30.560 | 21.880 | 100.186 |
| %VAR | -.365  | -.001  | *       |
| UNBE | 1.0496 | 1.2242 |         |
| KRAW | .5338  | 1.0000 |         |
| P/B: | .00    | .00    |         |

Results in Oxide Mole Percents :

|      | CO2    |        |         |
|------|--------|--------|---------|
| AVER | 50.002 |        |         |
| SDEV | .139   |        |         |
|      | CaO    | MgO    | SUM     |
| 9    | 25.053 | 24.841 | 100.000 |
| 10   | 25.028 | 24.917 | 100.000 |
| 11   | 24.922 | 25.233 | 100.000 |
| AVER | 25.001 | 24.997 | 100.000 |
| SDEV | .069   | .208   |         |

### Calculate Detection Limits

The user may also select a calculation of the single point detection limits, single point percent analytical sensitivity, average homogeneity, average range of homogeneity, average detection sensitivity and average weight percent analytical

sensitivity. The single point calculations are output for all data points, the sample average calculation are output only when the sample contains two or more data points and are expressed as t-test confidence intervals from .60 (60% confidence) to .99 (99% confidence). The t-test values will decrease detection limit and analytical sensitivity confidence significantly if only a few data points are acquired. Generally five to ten data points are necessary for a relatively robust confidence interval prediction.

#### **Single Point Detection Limits:**

The single point detection limit calculation is based on the standard counts and the unknown background counts and including the magnitude of the ZAF correction factor. The calculation is adapted from Scott and Lovet (1983). This detection limit calculation is useful in that it can be used even on inhomogeneous samples and can be quoted as the detection limit in weight percent for a single analysis line with a confidence of 99% (assumes 3 standard deviations above the background).

$$CDL = (ZAF) \frac{3}{I_s} \sqrt{I_B} \cdot 100$$

Where :       $ZAF$     is the ZAF correction factor for the sample matrix  
 $I_s$             is the count rate (cps/nA) on the (**pure element**) standard (apply std k-factor)  
 $I_B$             is the background count rate (cps/nA) on the unknown sample, see note below\*

\*In the above calculation be aware that before calculating the SQRT of the background count rate, one must first de-normalize the count rate (cps/nA) to raw counts by noting the beam current and integration time for the unknown intensity. Then take the SQRT, and finally re-normalize the background intensity to cps/nA before calculating the above k-ratio  $I_B/I_s$ . A sample DebugMode output is shown here for examination:

Line: 144 Intermediate Detection Limits Calculations (nominal beam = 1):

| Element/Xray  | ti      | ka | fe      | ka | al      | ka | mn      | ka | mg      | ka |
|---------------|---------|----|---------|----|---------|----|---------|----|---------|----|
| Bgd Count/Sec | 3.1     |    | 1.4     |    | 1.3     |    | .3      |    | .7      |    |
| Bgd CountTime | 1200.00 |    | 1200.00 |    | 1200.00 |    | 1200.00 |    | 1200.00 |    |
| Bgd Beam Curr | 100.10  |    | 100.10  |    | 100.10  |    | 100.10  |    | 100.10  |    |
| Bgd Raw Count | 369342. |    | 171492. |    | 154624. |    | 37771.2 |    | 84664.8 |    |
| Bgd Dev Raw   | 607.7   |    | 414.1   |    | 393.2   |    | 194.3   |    | 291.0   |    |
| Bgd Dev Cps   | .005059 |    | .003447 |    | .003274 |    | .001618 |    | .002422 |    |
| Std Count/Sec | 1894.82 |    | 1019.25 |    | 331.900 |    | 164.413 |    | 979.149 |    |
| Std K-Factor  | .5616   |    | .6862   |    | .1159   |    | .4052   |    | .4215   |    |
| Std Cps @100% | 3373.97 |    | 1485.35 |    | 2863.61 |    | 405.753 |    | 2322.84 |    |
| Unk ZAF Corr  | 1.2627  |    | 1.1927  |    | 1.6733  |    | 1.2235  |    | 2.1261  |    |

Detection limit at 99 % Confidence in Elemental Weight Percent (Single Line):

| ELEM: | Ti     | Fe     | Al     | Mn     | Mg     |
|-------|--------|--------|--------|--------|--------|
| 144   | .00057 | .00083 | .00057 | .00146 | .00067 |

#### **Single Point Analytical Sensitivities:**

After this, a rigorous calculation of analytical error also for single analysis lines is performed based on the peak and background count rates from both the unknown and the standard also from Scott and Love 1983. The results of the calculation are displayed after multiplication by a factor of 100 to give a percent analytical error of the net count rate. This analytical error result can be compared to the percent relative standard deviation (%RSD) displayed in the analytical calculation (both are one sigma standard deviation levels). The analytical error calculation is as follows:

$$\epsilon_{P-B} = \frac{\sqrt{\frac{N_P}{t_P^2} + \frac{N_B}{t_B^2}}}{\left( \frac{N_P}{t_P} - \frac{N_B}{t_B} \right)}$$

Where :  $N_P$  is the total peak counts  
 $N_B$  is the total background counts  
 $t_P$  is the peak count time  
 $t_B$  is the background count time

Note that the single point analytical sensitivity calculations utilize the interference, blank corrected, etc. on-peak intensities for the most accurate calculation.

#### Average Detection Limits and Analytical Sensitivity:

A more comprehensive set of calculations for analytical statistics is also performed on the averaged sample data if the sample contains two or more data points. These statistics are based on equations adapted from "Scanning Electron Microscopy and X-Ray Microanalysis" by Goldstein, et. al. (Plenum Press, 1992 ed., 1981) p. 432 - 436. All calculations are expressed for various confidence intervals from 60 to 99 % confidence.

Note that the average detection limit and analytical sensitivity calculations utilize the interference, blank corrected, etc. on-peak intensities for the most accurate calculation.

The calculations are based on the number of data points acquired in the sample and the measured average and standard deviation for each element. This is important because although x-ray counts theoretically have a standard deviation equal to square root of the mean, the actual standard deviation is usually larger due to variability of instrument drift, x-ray focusing errors, and x-ray production. The statistical calculations include :

The trace element detection limit is in elemental weight percent and indicates the minimum concentration detectability limit (CDL). Because this calculation utilizes the actual variance (standard deviation) of the measured composition, it should only be utilized for homogeneous materials.

$$C_{DL} = \frac{C_S}{\bar{N}_S - \bar{N}_{SB}} \frac{2^{1/2} (t_{n-1}^{1-\alpha}) S_C}{n^{1/2}}$$

The analytical sensitivity is in elemental weight percent and indicates what a statistically significant difference between two concentrations is. Because this calculation utilizes the actual variance (standard deviation) of the measured composition, it should only be utilized for homogeneous materials.

$$\Delta C = C - C' \geq \frac{2^{1/2} C (t_{n-1}^{1-\alpha}) S_C}{n^{1/2} (\bar{N} - \bar{N}_B)}$$

Where :  $C'$  is the concentration to be compared with  
 $C$  is the actual concentration in weight percent of the sample

- $C_s$  is the actual concentration in weight percent of the standard  
 $t_{n-1}^{1-\alpha}$  is the Student t for a  $1-\alpha$  confidence and  $n-1$  degrees of freedom  
 $n$  is the number of data points acquired  
 $S_C$  is the standard deviation of the measured values (count intensity)  
 $\bar{N}$  is the average number of counts on the unknown  
 $\bar{N}_B$  is the continuum background counts on the unknown  
 $\bar{N}_S$  is the average number of counts on the standard  
 $\bar{N}_{SB}$  is the continuum background counts on the standard

The detection limit calculation here is intended only for use with homogenous samples since the calculation includes the actual standard deviation of the measured counts. This detection limit can, however, be quoted for the sample average and of course will improve as the number of data points acquired increases. The analytical sensitivity calculation is ignored for elements whose concentrations are present at less than 1%.

An option exists for the Projected Detection Limits in which the program extrapolates for shorter and longer count times how much of an effect changing the counting time will have on the detection limit. Based on the assumption that doubling the count time will decrease the detection limit by  $\text{Sqr}(2)$ .

### ***Homogeneity Calculations***

The program will also calculate homogeneity evaluations based on the standard error statistics of the deviation of the mean.

The range of homogeneity in plus or minus weight percent.

$$W_{1-\alpha} = \pm C \frac{t_{n-1}^{1-\alpha}}{n^{1/2}} \frac{S_C}{\bar{N}}$$

The level of homogeneity in plus or minus percent of the concentration.

$$\pm \frac{W_{1-\alpha}}{C} = \pm \frac{(t_{n-1}^{1-\alpha})S_C(100)}{n^{1/2}\bar{N}}$$

- Where :
- $C'$  is the concentration to be compared with  
 $C$  is the actual concentration in weight percent of the sample  
 $t_{n-1}^{1-\alpha}$  is the Student t for a  $1-\alpha$  confidence and  $n-1$  degrees of freedom  
 $n$  is the number of data points acquired  
 $S_C$  is the standard deviation of the measured values (count intensity)  
 $\bar{N}$  is the average number of counts on the unknown

The homogeneity test merely compares the 99 % confidence range of homogeneity value with 1 % of the sample concentration for each element. If the range of homogeneity is less than 1 % of the sample concentration then the sample may be considered to be homogenous within 1 %. Note that the homogenous sample detection limit calculation are ignored for those elements which occur as minor or major concentrations ( $> 1\%$ ).

### **Alternate Homogeneity Calculations**

The program will also calculate an alternative range of homogeneity (Meisenkothen and Donovan, 2008) based on a revised Goldstein equation that calculates the range of homogeneity in elemental weight percent expected from statistics alone (the ideal range of homogeneity) and the actual range of homogeneity in the sample. The ranges are calculated as follows:

Ideal range of homogeneity:

$$W_{1-\alpha} = \pm C(t_{n-1}^{1-\alpha}) \frac{\sqrt{N}}{N}$$

Actual range of homogeneity:

$$W_{1-\alpha} = \pm C(t_{n-1}^{1-\alpha}) \frac{S_C}{\bar{N}}$$

The only difference between the traditional Goldstein equation and the “revised” Goldstein equation is the dropping of the SQRT(n-1) term in the denominator. Leaving this term in produces homogeneity ranges that change with the number of measurements. In fact the traditional Goldstein equation is actually a measure of the standard error of the mean homogeneity.

**Note that there is an alternative calculation for modeling the detection limit and/or required counting time in the program CalcZAF that is extremely useful for predicting sensitivity or analysis conditions.**

### **Use Particle/Film Calculations**

This button in the Calculation Options dialog of the Analyze! window allows the user to quantitatively analyze particles (cubes, prisms, spheres and fibers) and thin films (only for those films or foils less than 1 milligram/centimeter<sup>2</sup> or assuming density = 1 approximately 100 um in thickness) based on various adjustable parameters. Note that the accuracy of the particle method depends on bracketing a single particle in the scanned or defocused beam (overscanning) and applying judgement as to the particle shape and the distance along the beam axis. Normal thick polished standards should be used (bulk specimens) for the intensity calibration. Note that the scan or defocus size must be equal to the particle width dimension.

Particles are normally deposited dry on a low Z substrate that does not contribute to the x-ray spectrum (or continuum) and is usually a double sticky carbon tab. Insulating particles should be carbon coated using a two axis rotation evaporation method or carbon sputtered coated for good conductivity.

### **Particle/Thin Film Model**

The geometric particle and thin film models are:

Thin Film or Thick Polished Section (unsupported film or no substrate)

Rectangular Prism (flat top and flat sides or cube) - sides orthogonal to spectrometer axis

Tetragonal Prism (flat top and curved sides) - sides 45 degrees to spectrometer axis

Trigonal Prism (curved top and flat sides or fiber) - long axis perpendicular to spectrometer

Square Pyramid (curved top and curved sides or sphere)

Backscatter-Modified Rectangular Prism (modified rectangular prism)

Note that the last model is the same as the previous "Rectangular Prism" model with a modified electron side-scattering correction.

Note that particle and thin film calculations are limited to the following standard Phi-Rho-Z correction methods:

Select Individual Corrections (use with caution)

Armstrong/Love Scott (default)

Packwood Phi(pz) (EPQ-91)

Bastin (original) Phi(pz)

Bastin PROZA Phi(pz) (EPQ-91)

Note also that if using "Select Individual Corrections", only the following absorption corrections are allowed:

- Phi(pz) Absorption of Packwood-Brown 1982/XRS Alpha
- Phi(pz) Absorption of Bastin 1984/XRS Alpha
- Phi(pz) Absorption of Armstrong/Packwood-Brown 1981 MAS
- Phi(pz) Absorption of Bastin 1986/Scanning
- Phi(pz) Absorption of Riveros 1987/XRS
- Phi(pz) Absorption of Packwood (New)
- Phi(pz) Absorption of Bastin Proza (EPQ-91)

### **Particle Diameter**

The particle diameter is either set to an arbitrary large number (10,000 microns or 1 cm) for thick specimens or the actual distance the electron beam will travel through the particle or thin film in microns.

### **Particle Density**

The particle density in g/cm<sup>3</sup> is irrelevant for thick specimens, but critical for particles and thin films that are less than the electron range. Iron or calcium bearing silicates average around 3.0 and SiO<sub>2</sub> or high silica glasses are about 2.7.

### **Particle Thickness Factor**

This is the factor that defines the beam aspect ratio relative to the particle diameter. For example, 0.5 means that the particle thickness is half its diameter (a disk). This value should be 1 for thin films or thick polished specimens.

### **Numerical Integration Step**

This value is the step length (in g/cm<sup>2</sup>) for numerical integration. A typical value for 15 keV is 0.00001. For thinner films the step size should be decreased and for higher voltages the step size should be decreased slightly.

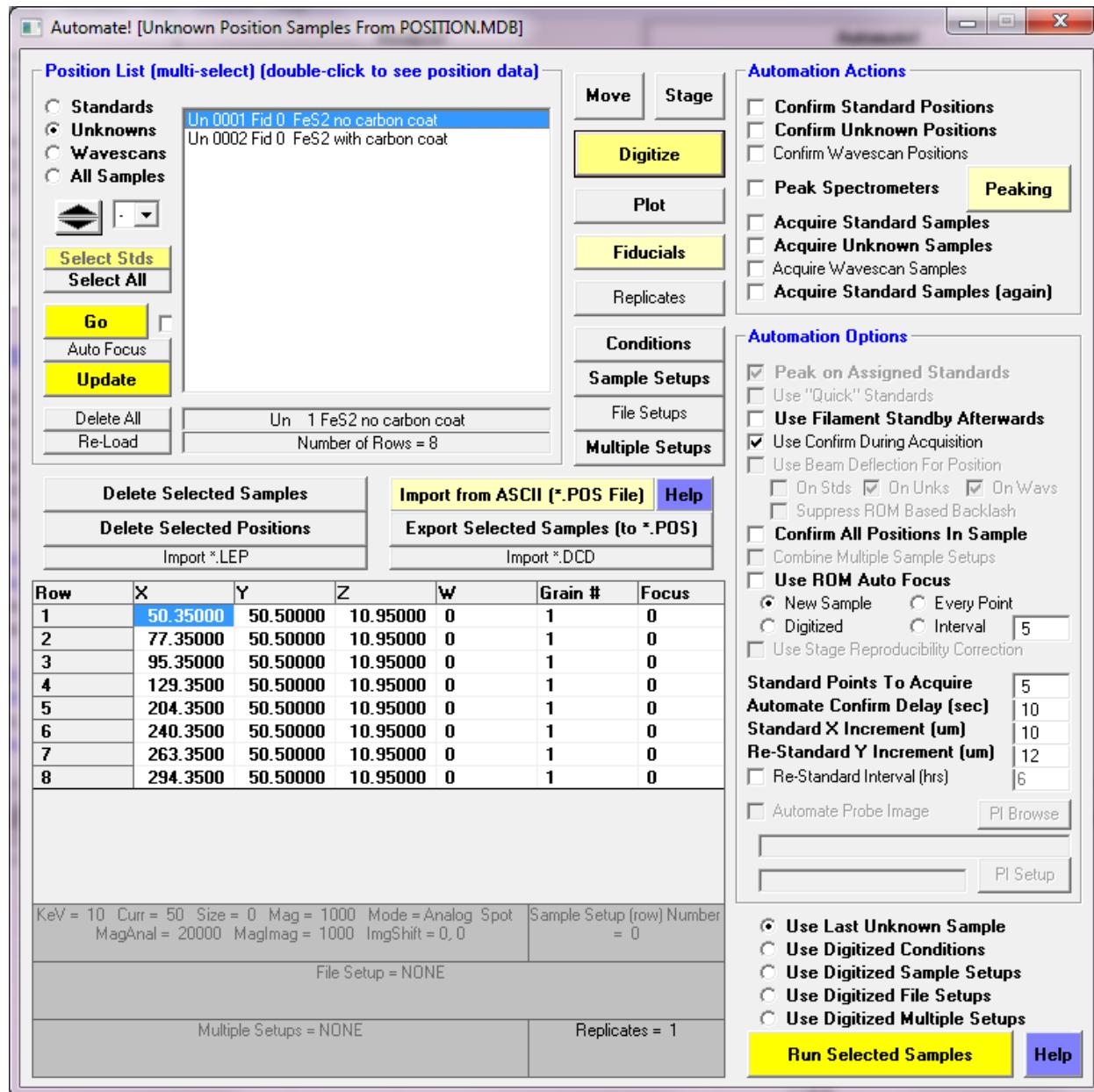
## **Analyze Data**

By double clicking a single analysis row in the data grid, the software will display the analysis is row format instead of the normal column format as shown here:

The screenshot shows the 'Analyze Data' dialog box. At the top left, there's a status bar with 'Un 34 MB2 IW2 C4-ext-3'. Below it is a table with various parameters: T0 = 40, KeV = 15, Beam = 20, Size = 10, MagAnal = 8000, Mode = Analog, Spot, MagDef = 1267, MagMag = 1267, and Image Shift (X,Y): .00, .00. To the right of the table, there's analytical data: Line: 301, Status: Good (Undeleted), Line: 2 of 6, Stage (XYZ): -2698.0 26354.0 177.000, Acquisition Date/Time: 03/15/2007 11:52:47 AM, Beam Current: 19.8309. The main part of the dialog is a data grid with columns: Copy, K-Raw, Counts, Elemental, Oxide, and Atomic. The data rows are as follows:

| Copy | K-Raw   | Counts  | Elemental | Oxide   | Atomic  |
|------|---------|---------|-----------|---------|---------|
| Na   | 09634   | 241.6   | 1.385     | 1.867   | 1.298   |
| Si   | 1.03918 | 10399.6 | 26.630    | 56.972  | 20.432  |
| K    | .04698  | 276.2   | .592      | .714    | .326    |
| Al   | .43437  | 3610.0  | 7.848     | 14.828  | 6.268   |
| Mg   | .45337  | 1282.8  | 3.860     | 6.401   | 3.422   |
| Ca   | .63699  | 213.3   | 7.122     | 9.965   | 3.829   |
| Ti   | .01068  | 68.8    | .701      | 1.169   | .315    |
| Mn   | .00370  | 55.3    | .328      | .423    | .129    |
| Fe   | .50475  | 302.9   | 5.691     | 7.322   | 2.196   |
| P    | .00822  | 79.4    | .186      | .427    | .130    |
| Cr   | .00124  | 6.5     | .044      | .064    | .018    |
| O    |         |         | 45.764    | .000    | 61.636  |
| H    |         |         | .000      | .000    | .000    |
| Sum  |         |         | 100.152   | 100.152 | 100.000 |

# Automate!



The Automate modeless window provides for automation of standard, unknown and wavescan samples, position confirmation and peak centering. Also import, export and digitization of standard, unknown and wavescan coordinates.

## Position List

The large list box at the top of the Automate! window holds all currently digitized positions resident in the POSITION.MDB file. This file is a separate file from the user's database and is intended as a temporary repository of the digitized positions that the current user is working with.

It can grow unnecessary large if it is not continually cleaned out; that is, each user should only have to contend with his or her standard, unknown and wavescan positions. Once the unknown and wavescan positions have been run and data acquired, those positions are saved in the user's run database, and there is no reason to keep them in the position.mdb file. As far as standards go, a user who will consistently be using the same set of standards should save the those standard positions, and then recall them at the next run. This is done with the Export Selected Samples, and Import from ASCII File buttons, described below.

To display all currently digitized standard position samples that are loaded click the Standard option, to display all the unknown position samples click the Unknown option and to display the Wavescan (wavelength scan) position samples click the Wavescan option. To see all currently loaded position samples, click the All Samples option.

**To display the coordinates for a particular sample, double-click the name of the position sample in the list. To move to a specific digitized coordinate position listed in the coordinate grid, simply double-click the coordinate row in the coordinate grid.**

In the coordinate grid the "Grain #" column is used to specify whether a beam blank is used between x, y, z coordinate positions within a position sample. The "Focus" column is used for specifying a "digitized" auto-focus action. See the Digitize window for more information.

### **Up/Down Arrow**

This "spin button" allows the user to change the acquisition order of unknown and wavescan samples. Select a single sample and then using the mouse click the arrow in the desired direction of placement.

### **Select Stds**

This button will select all standard position samples in the list that have been added to the run using the Standard | Add/Remove Standards To/From Run menu from the main menu.

This is useful for quickly selecting only those standard position samples of interest to the current run. Note that if a standard position sample is selected in the Position List and run as an automated acquisition, the program will automatically add the standard to the run.

### **Select All**

This button will select all position samples shown in the position sample list. Normally this button is used for exporting all listed position samples to an ASCII .POS file.

### **Delete All**

This button will delete all position samples shown in the position sample list. This is especially useful when starting a new run which will require a completely different position sample set from the previous user. New position samples are imported using the Import From ASCII File button described below.

Note that samples not shown in the list (unknowns or wavescans if the Standard option is selected) will **not** be deleted from the position database. To delete all samples in the database, first click the All Samples option and then the Delete All button.

Note that if all position samples that reference a fiducial calibration set are deleted, then the program will ask the user whether to delete the fiducial set that is no longer referenced by any position samples. Normally, the user should allow the program to delete unreferenced fiducial sets unless they plan to digitize additional position samples to the already confirmed fiducial set.

## **Re-Load**

This button will force the position database to be reloaded. This is useful when the position data in the position database has been modified by another application (for example STAGE.EXE while PROBEWIN.EXE is running).

## **Move**

This button will recall the Move dialog for manual stage (and spectrometer and crystal) motion. The Move window is a modeless window which may remain open and accessible during the digitization of position samples.

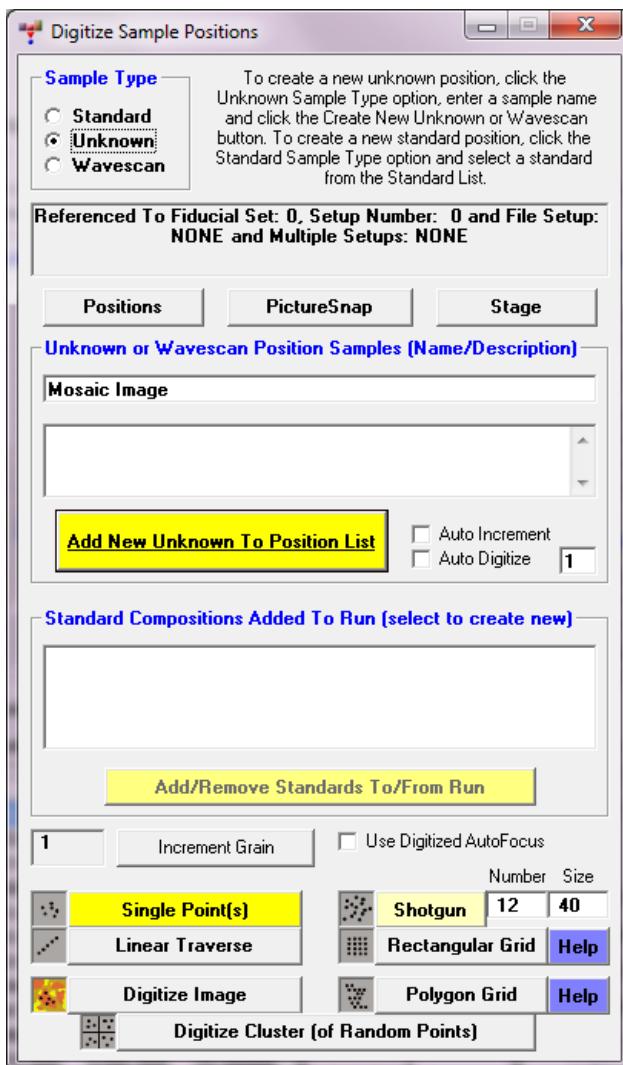
To move quickly and easily to a position already digitized in the Automate! (or Position) window lists, simply double-click the sample, to list the position coordinates for that sample, and then double-click on the specific coordinate (row) that one desires to move to. The program will automatically move to that stage position.

From the Move dialog one may select to open the Stage Map or the Position Database dialogs, both of which are very useful for digitization of standards and unknown position samples.

## **Digitize**

This button will load the Digitize dialog, where the user can digitize standard, unknown or wavescan positions based on random (individual) points, linear traverses or rectangular or polygon gridded areas. The “position samples” can be assigned to be automatically acquired based on the current sample, assigned sample setups, assigned analytical column conditions or assigned multiple sample setups (useful for acquisition of multi-voltage analysis for thin film samples).

The Digitize Cluster allows the user to specify randomly digitized areas using square or round regions for unbiased sampling.



In addition, the user may use the Digitize Image button to open a dialog that can be used to acquire an analog image for the purposes of digitizing sample coordinates by simply clicking on various locations on the image.

### **Creating Standard Position Samples**

To digitize standard positions, the standard to be digitized must first be added to the run using the Standard | Add/Remove Standards To/From Run menu (see menus in main log window of Probe for EPMA).

To create a new standard position sample, first select the sample type as Standard and then simply click the desired standard from the list box. The standard position sample will automatically be created (in the Automate! position sample list) if it did not already exist. Normally a single random (individual) digitized position is sufficient for the digitization of a standard position if the standard is large and homogeneous to permit automatic incrementing based on the X and Y increments in the Automate! window. The program will automatically increment the X and Y positions for subsequent point acquisitions based on the increment fields in the Automate! window. However, if specific positions are digitized for a standard position sample, the program will use those positions for automated acquisitions.

A new display standard image feature is that the program displays BMP electron image files of the standards in the standard blocks. The program looks for any BMP files in the StandardPOSFileDirectory as defined in the PROBEWIN.INI file [Standards] section, (defaults to UserDataDirectory\$ if not defined) and if it finds a match to any file (first 4 characters) of the standard number it loads it. For example standard MgO entered as 12 in the STANDARD.MDB database could be a file called "0012\_MgO\_Block2.bmp" or "0012\_Periclase\_Block4.BMP". Both

image files will be loaded during standard digitization (when the standard is clicked in the Digitize Sample Positions dialog ).

### ***Creating Unknown Position Samples***

To create unknown or wavescan position samples, first select the sample type as before and then enter the sample name in the sample name field. Optionally the user may enter a sample description field of up to 256 characters. To enter a new line in the sample description field use the <ctrl> and <enter> keys together.

Two new features are the Auto-Increment and Auto-Digitize check boxes. The Auto-Increment option will automatically increment the current sample name by appending a “\_001, \_002, \_003”, etc. suffix. Each time the Create New Unknown Or Wavescan button is clicked (or the <enter> key is pressed) a new sample will be created with an unique sample number. The Auto-Digitize feature will at the same time automatically digitize a number of random position coordinates based on the Shotgun digitize feature parameters. Note that if a single point is specified in the Shotgun parameters then a single point is digitized based on the current stage position.

Finally, click the Create New Unknown Or Wavescan Position Sample button and the position sample will appear in the Automate! position sample list. Next proceed to digitize points using the random, traverse or grid buttons.

### ***Picture Snap***

The Picture Snap window allows the user to import any image from a slide or flatbed scanner and calibrate the image to the microprobe stage coordinates using any two diagonal points (or three points to correct for sample rotation) on the image so that the sample areas of interest can be located easily.



The current stage position location is shown as a target cursor on the calibrated picture and is updated continuously as the stage position is adjusted by the user. To move to any point on the image simply double click the image coordinate.

Use the Display menu to display the currently digitized position samples. The Position Sample Labels menu will display the sample number alongside the plotted position.

Use the Save As GRD File menu to export the image data to a Surfer GRD format. If any sample labels are displayed the coordinates and labels will be exported to a .DAT file for using as a “post data” file in Surfer.

### ***Single Point(s)***

To digitize a sample of random (individual) points, simply click the Single Points button. Each time the button is clicked, it will add a digitized position based on the current position to the currently selected position sample.

Note that any number of random (individual) points can be digitized in a single position sample. At the time of acquisition the program will sub-divide them into appropriate sized samples using the keyword sample name "continued" for points in any subsequent samples.

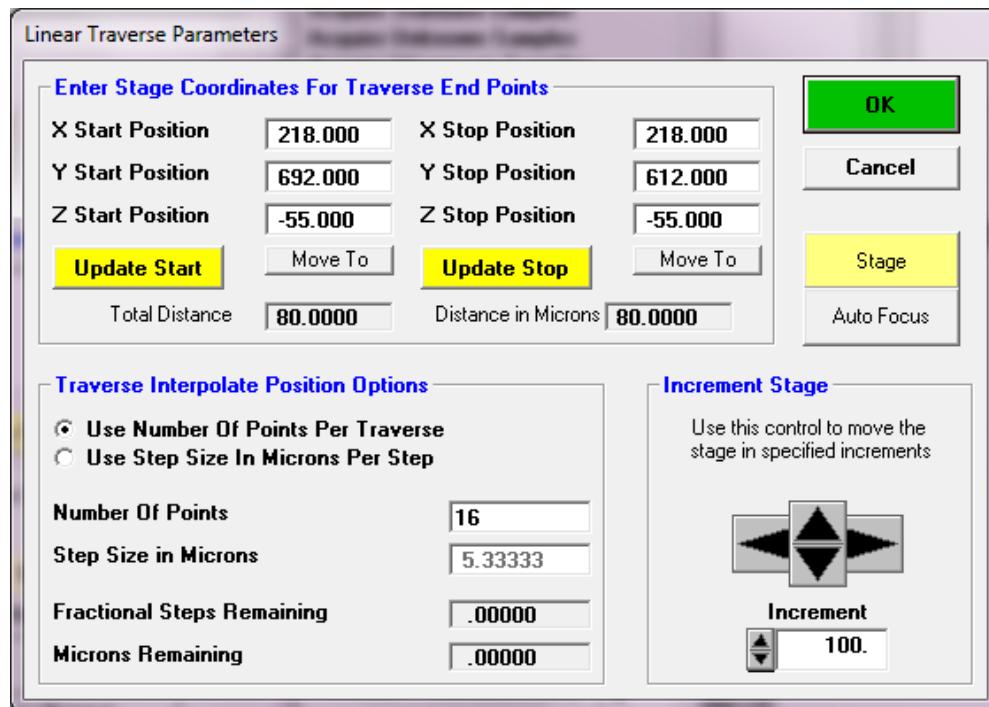
## **Shotgun**

The shotgun digitize method will automatically calculate a random sampling of data points based on Number and Size. The “Number” is the number of digitized points to digitize and the “Size” is the size (diameter) of the digitized area to cover specified in microns.

The software will automatically keep the distance between the digitized points to be a minimum of 1 micron or the beam size diameter, whichever is larger. If the software cannot calculate enough random points to fill the specified area, simply decrease the “Number” of points, increase the “Size” of the digitized area, or decrease the beam size.

## **Linear Traverse**

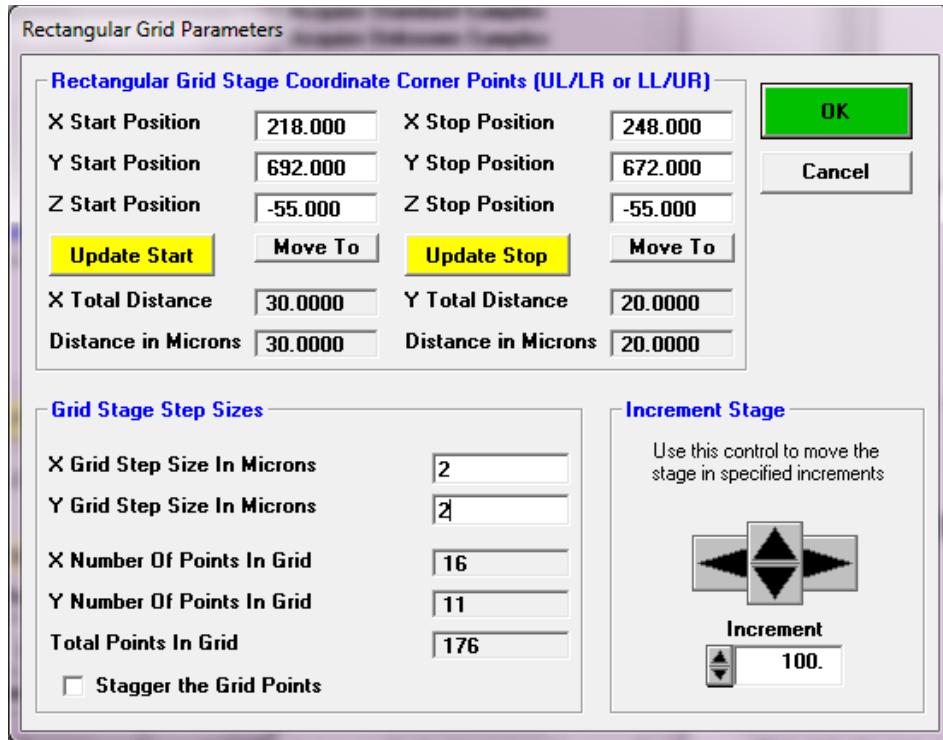
The linear traverse dialog allows for digitizing a straight line series of sample positions based on two given end-points. The default start and stop positions are loaded with the current position, therefore it is best to move to the desired start or stop position before clicking the Linear Traverse button.



Select either a specified number of points per traverse (including the end-points) or a specified distance between points.

## **Rectangular Grid**

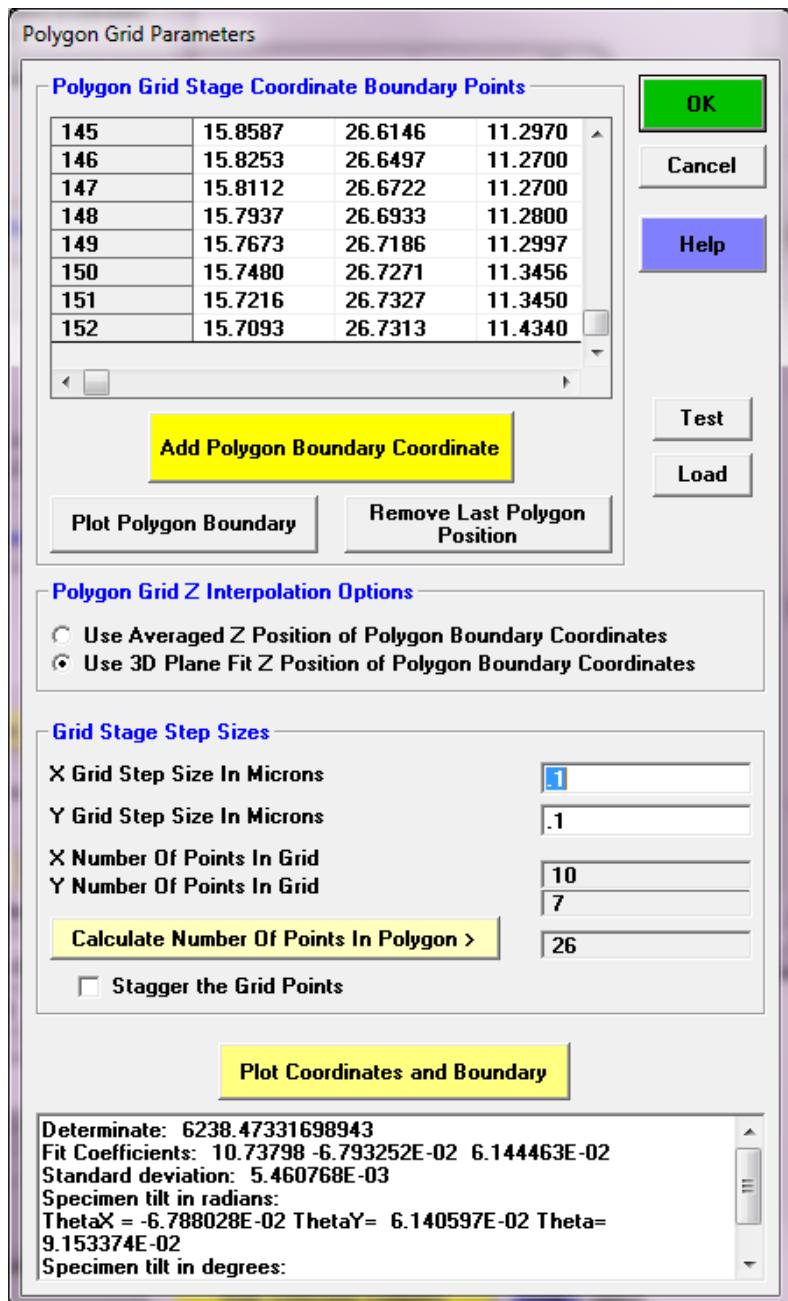
The Rectangular Grid dialog allows an offset grid of points to be digitized based on any two opposite corners. The grid spacing is calculated by the X and Y distance in microns specified by the user.



As the grid points are digitized, the positions are calculated in a "serpentine" manner to reduce stage motion to a minimum during acquisition.

### **Polygon Grid**

The Polygon Grid dialog allows the user to digitize an offset grid of points which is delimited by an arbitrary boundary digitized by the user. This boundary may contain any number of points and is saved along with the grid points for use in exporting to a .BLN boundary file used to create SURFER 3-D surface and/or contour output.



To create a boundary for the polygon grid, use the focused reflected light and/or BSE image to simply move the stage and then click the Add Polygon Boundary Coordinate button for each position to be included in the boundary. Depending on the desired level of detail, any number of coordinates may be used to digitize the boundary feature.

The boundary can be plotted during the digitization process by clicking the Plot Polygon Boundary button and the first and last points are automatically assumed to be closed to complete the polygon boundary. Because the grid algorithm is quite robust, boundaries of any arbitrary shape may be digitized.

The Test button is only visible in demonstration mode and is used to load a default polygon to demonstrate the polygon gridding process.

The Load button is used to load a previously digitized set of polygon grid boundary coordinates. This is useful when the user decides that a different grid spacing is preferred but with the same digitized boundary. By using this feature, the user is not required to re-digitize the boundary coordinates. Typically, one should create a new position sample (with no positions) when loading a previously digitized polygon boundary for the purpose of changing the grid step sizes.

Two methods for determining the Z axis interpolated position are available. The first is based on the average Z position of all boundary positions, the second is a 3-D plane fit to the Z position of all boundary positions.

Adjust the number of points to be gridded during the digitization by entering X and Y grid step sizes in microns. For example, a 100 x 100 micron rectangle (4 boundary points) will produce 121 analysis positions if a 10 micron spacing is used for both X and Y (11 by 11).

The program will prompt the user if the number of grid positions are excessively large by assuming a "rectangular" boundary. To see the actual number of grid points that will fit inside the digitized boundary, click the Calculate Number of Points in Polygon button to determine the actual number points that will be digitized.

Click OK when ready to digitize the grid based on the polygon boundary coordinates and the X and Y grid step sizes. Click Cancel to not digitize any polygon grid positions and not save the boundary coordinates.

An example of the type of output that is obtained when the polygon grid quant data is exported from the Plot! Window using the “SURFER .BAS File” checkbox (first click the “Send Data To ASCII File” option) and the resulting .BAS script is run from the Golden Software Scripter applet is shown here:



### **Digitize Image**

This button is used to open a dialog that can be used to digitize stage positions based on an SE or BSE analog image supplied by the system. This feature can acquire images from either the MicroImage-32 software, or the Win30 A/D-D/A board directly. The imaging system requires calibration in order to associate stage coordinates to beam deflection positions before it can be utilized for digitizing. See the parameters defined in the PROBEWIN.INI file [Image] section.

Both random points and traverse coordinates may be saved to the position database by simply clicking the mouse on the displayed image. If the image is supplied by MicroImage, any type of X/Y registered analog image that MicroImage is capable of acquiring may be used for the purpose (beam, stage or mosaic beam/stage scan images included) at any magnification supported by the instrument; in the case of the WIN30 direct interface only beam scan images may be acquired.



Note on the scan speed field:

**ImageInterfaceType = 4**      ' JEOL 8900/8200/850/8x30  
"AD Averages/Pixel"

Specify the number of A-D conversions to average per pixel (range 1 - 1000)

**ImageInterfaceType = 5**      ' SX100/SXFive mapping  
"mSec/Pixel"

Specify the image dwell time in msec/pixel (range 1 - 1000)

**ImageInterfaceType = 6**      ' SX100/SXFive Video  
"Scan Rate"

Specify the image scan speed (range = 1 - 7)

Use the Image Size options to select the image resolution, then click the analog source and click the Start Image button. The image acquisition will start. To halt the current image acquisition, click the Stop Image button. An image must be acquired before the user can begin to digitize position coordinates.

The Mag (magnification) and keV (kilovolts) fields are used by some interfaces (SX100/SXFive) to set the actual beam scan conditions for the image acquisition. For other interfaces the actual setting made to the instrument manually must be entered here so that the stage positions are correctly calculated.

**Note that there are three magnifications defined in Probe for EPMA: a default magnification which is used for normal operation of the instrument between acquisitions, an analytical magnification which is used to acquire standards, unknowns and wavescans and an imaging magnification which is used to acquire images or for use with beam deflection acquisitions.**

The Center Point button can be used to digitize the first point of the position sample in the exact center of the beam scan area if desired.

Once an image is acquired, use the Random (individual) Point(s) button to digitize one or more random (individual) position coordinates by simply clicking the image at the desired location(s). The stage coordinates of each mouse click is stored in the position database for sample automation purposes.

Use the Traverse Start to indicate the start position of a linear traverse, the Traverse Stop button to indicate the stop position of the linear traverse and the Number of Points field to specify the number of points in the traverse. Click the Digitize Traverse button to actually digitize the traverse and store it in the position database.

### ***Image Calibration***

So that the beam scan is calibrated accurately to the stage coordinate system, the user should utilize the “@” button to calibrate the beam scan based on an acquired image. The image calibration is performed by selecting 2 points on a diagonal in the image for which the stage coordinates are known (by centering the beam on those two stage positions).

A couple of points to consider when performing the image calibration:

- Scan rotation: not only does the scan rotation need to be set exactly (within 0.2 degrees or so) but the scan rotation need to be set to the stage orthogonal when the light optics are properly focused because the working distance changes the scan rotation.
- Scan speed: the scan speed affects the image shift significantly, especially in the X direction- the scan rotation (so long as it's a horizontal reference) is not very affected. But the use of a slow scan speed is very important for accurate image calibration.
- Electron beam focus: the scan rotation rotates dramatically when adjusting the focus. Be sure you are focused properly before setting the scan rotation.
- Prior to any image calibration efforts it is desirable to check the beam orthogonality of the instrument on a calibration standard.

### ***Digitize Cluster (of Random Points)***

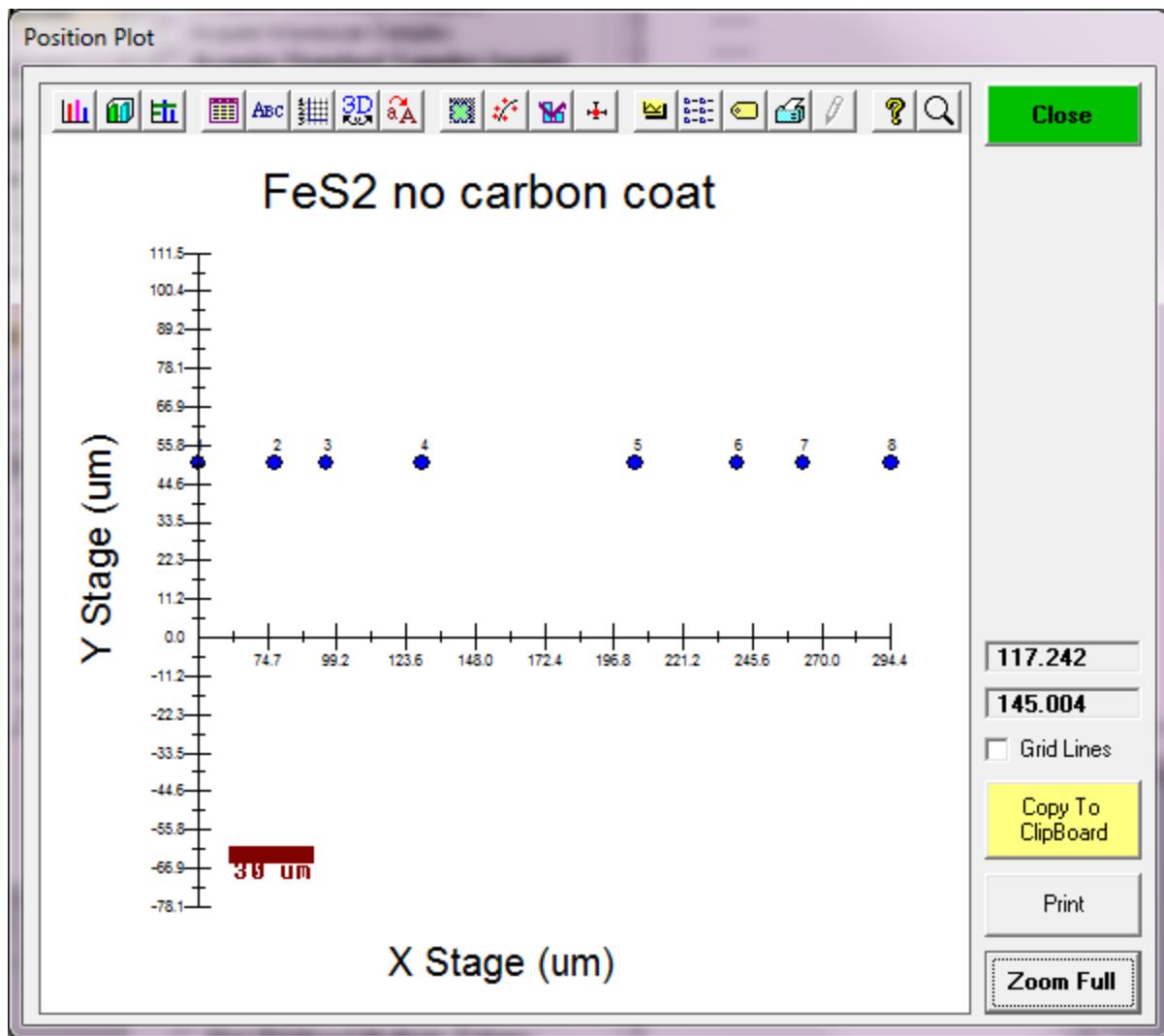
This dialog allows the user to define a circular or square areas of a given diameter or width and specify the number of randomly distributed positions within the area. There is also an option (by default) to save a polygon boundary of the area and the number of points to define the area (round area only).

In addition the user can specify the minimum distance between random points to prevent the beam from analyzing the same spot more than once. Note that if the minimum distance is too large or the cluster diameter or width is too small or the number of points is too large, the program may be unable to find enough evenly spaced coordinates.

This feature is useful when performing careful homogeneity tests when it is important to eliminate instrument drift and grid pattern artifacts. Note that the circular boundary created when digitizing round areas can be re-used in the polygon gridding dialog using the Load button.

### ***Plot***

The Plot button will display the digitized X and Y stage coordinates for the selected position sample. Multiple samples can be plotted at the same time.



The example above shows a plot of a Polygon grid consisting of 55 points digitized using three boundary points.

If the position sample is a polygon gridded position sample then the digitized polygon boundary will also be displayed. The coordinates of the cursor are displayed as the cursor is moved across the plot area.

## Fiducials

The Fiducials button will display the current digitized fiducial sets in the position database. To select a different fiducial set to reference subsequent digitized position to, simply select it from the list. In addition, this dialog will allow the user to modify, confirm, create new or delete fiducial sets in the position database.

Although standard positions are normally referenced to fiducial coordinates, it may also be desirable to reference unknown or wavescan coordinates to physical fiducial marks for the purposes of precisely relocating the acquired positions even after they have been removed and later re-loaded into the microprobe.

For this purpose, it is required that the sample mount have been previously scribed or otherwise marked to allow the user to locate 3 points that will be used for the fiducial coordinates. These fiducial coordinates must be permanent and easily re-located in the same order that they were defined to allow the program to calculate a transformation matrix when the sample is re-loaded later on. Once the positions have been referenced and digitized to a selected calibrated fiducial coordinate set, they can be exported to an ASCII file for subsequent use at another time.

See the section Import from ASCII File below for details on using the fiducial coordinates to transform digitized coordinates.

## Replicates

The Replicates button will allow the user to specify any number of replicate acquisitions for each point digitized in the position sample to be automated.

For example, if the number of replicates is 1 (the default), the program will acquire one data point per digitized point in the position sample. But if the number of replicates is, say 3, the program will acquire 3 data points for each digitized coordinate in the position sample. Note that each digitized position sample can have a different number of replicate acquisitions.

The "replicate" field only applies to unknown sample acquisitions run from the Automate! window.

## Conditions

Although Probe for EPMA normally acquires automated samples based on the last unknown sample conditions, the program will support the automatic acquisition of samples under various analytical conditions (if the interface hardware supports it).

There are two different options for analytical conditions, the first is using specified values for kilovolts, beam current and beam size, the second is using a specified string to indicate a column condition that is stored internally in the instrument. In this latter case, because the actual value of the operating voltage (kilovolts) in the column condition string is ambiguous, the user should be sure that the single value fields are also correctly specified based on the column condition string specified.

**Note that three different magnifications may be specified. A “default” magnification for the normal state of the instrument (after data or images are acquired), an “analytical” magnification for when acquiring standard, unknown or wavescan data when in Analog Scan mode, and an “imaging” magnification used for image acquisition. Allowable range of magnifications for different platforms is documented here:**

**Minimum magnification = 40**                          ‘ JEOL 8900/8200/8500/8x30 (InterfaceType = 2)  
**Maximum magnification = 300000**

**Minimum magnification = 63**                          ‘ SX100/SXFIVE (InterfaceType = 5)  
**Maximum magnification = 200000**

Therefore, if the column condition option is selected, and the column condition string is specified as "15\_keV\_30 nA\_0\_um.PCC" (indicating a column condition at 15 keV), then the user should also be sure that the kilovolts field is correctly specified as 15 keV. Failure to properly specify the kilovolts field when using column condition strings will cause the ZAF or Phi-Rho-Z matrix corrections to be inaccurate.

For accurate documentation purposes it is also preferred that the beam current and beam size fields are also properly specified when using column condition strings. If, as in the above example, the "15\_keV\_30 nA\_0\_um.PCC" column condition string also includes a beam current of 30 nA and a beam size of 0 um, then those values should also be specified in the single values fields even though they are not actually used in the acquisition procedure. This maintains the accuracy of the documented sample conditions.

By using either the analytical conditions and column condition methods, it is possible to automatically acquire a number of digitized positions using a different kilovolts/beam current/beam size values. This is useful for many purposes, for example, automated thin film analysis, where the acquisition of k-ratios at various operating voltages can improve the analysis considerably.

To utilize this acquisition type, be sure to select the Use Digitized Sample Conditions option before clicking the Run Selected Samples button.

## Sample Setups

Although Probe for EPMA normally acquires automated samples based on the last unknown sample conditions, the program will support the acquisition of samples based on previously saved sample setups with a variety of elements or multiple keV or nA conditions.

This is especially useful when the need to acquire data on samples with a variety of elements are present. For example, there may be a number of quite different phases present that each require a more or less different set of analyzed elements. By previously creating a number of sample setups and saving them, it is possible to have the program automatically acquire a different set of analyzed elements by referencing the various available sample setups during the digitization process.

**Sample setups may be created from the Acquire! window by first starting a new sample and then modifying the sample setup to the desired configuration. Then use the Save Sample Setup button from either the Analyze! Window or within the Elements/Cations window (accessed from the Analyze! window) to create a pointer to the sample setup for use later on during the creation of new analyzed samples or the digitizing of position samples.**

To utilize this automated acquisition method, you will need to "tag" each set of samples: with a sample (or set of them) highlighted, click the Sample Setups button and then highlight the particular sample setup to be used, and click OK. If there are other samples to be digitized using different sample setups, then tag those. Before clicking the Run Selected Samples button, make sure the Use Digitized Sample Setups option is checked (it will be done automatically by the program when you select the setups).

## File Setups

Although Probe for EPMA normally acquires automated samples based on the last unknown sample conditions, the program will support the acquisition of samples based on previously created file setups from other Probe for EPMA runs. These may be Probe database files that contain one or more specific sample setups and may or may not also contain standardization count intensity data.

This is especially useful when the need to acquire data on samples with a variety of elements are present. For example, there may be a number of quite different phases present that each require a more or less different set of analyzed elements. By previously creating a number of file setups, it is possible to have the program automatically acquire a different set of analyzed elements by referencing the various available file setups during the digitization process. Note that the specified file setup must be available to the program when the automation is run or an error will occur.

To utilize this automated acquisition method, be sure to select the Use Digitized File Setups option before clicking the Run Selected Samples button.

## Multiple Setups

This button is used to assign multiple sample setups to current or newly created digitized position samples. All considerations for Samples Setups apply for Multiple Setups (see Sample Setups above). To create sample setups for digitizing position samples, use the "Elements/Cations" button in the Analyze! Window.

This procedure is affected by the state of the "Combine Multiple Sample Setups" checkbox in the Automation Options frame. If this checkbox is not checked, then a separate sample, based on each multiple setup, will be acquired for each position sample. This is particularly useful for situations where the same elements need to be measured several times with different operating voltages, for example, in thin film analysis, when it is necessary to acquire different voltages on the sample position. Here one would specify two or more sample setups that have the same elements but different kilovolt operating conditions.

If the checkbox is checked, then all specified multiple setups will be combined into a single combined condition sample for the position sample, and the software will adjust the column condition as necessary to acquire the elements. When more than one sample setup is specified by the user, the automation routine will acquire the position sample for each sample setup that is specified. This is useful in several situations, for example, when performing major and trace element acquisition it may be desired to acquire the major elements at one set of sample conditions and the trace elements using a sample setup with different sample conditions (kilovolts, beam current, etc.).

In the former case of major and trace element analysis, each sample setup acquired will create a different analytical sample. To combine the elements into a single sample for quantitative analysis, simply select all the samples that are to be combined and click the "Analyze Selected Samples (Combined)" button in the Analyze! Window.

## Delete Selected Samples

This button will delete the currently selected position samples from the position database in the window above the button. If all position samples that reference a fiducial set are deleted, the program will ask whether to delete the fiducial set from the position database also. Normally one should allow the program to delete unreferenced fiducial sets to avoid confusion.

## Delete Selected Positions

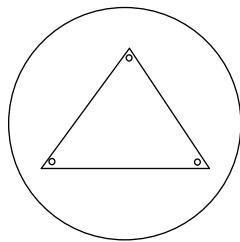
This button will delete the position coordinates that are currently selected in the position grid display below the button. This option will only delete contiguous position coordinates listed in the position grid. Delete non-contiguous digitized positions individually.

## Import From ASCII File (\*.POS)

The Import button allows the user to import an ASCII file of sample positions for automated acquisition. Standard, unknown and wavescan coordinates can be imported separately or in a single file. Normally all standard coordinates are stored in .POS files and imported into the POSITION.MDB database only as needed for current work.

### Fiducial Marks

The program will ask the user whether the imported coordinates should be transformed using a fiducial based matrix transformation. The fiducial positions on the standard or unknown sample mount should generally be laid out in an easy to re-locate pattern. Generally it is preferred to place the fiducials marks in the corners of a scribed (or partially scribed-corners only) triangle on the mount surface as shown here :



The fiducial marks should be deep enough to withstand repeated re-polishing but small enough to be precise for re-location of the digitized coordinates.

A sample import file of two unknown samples and corresponding positions is shown here. Note that a new standard, unknown or wavescan sample is created automatically by the program during the acquisition process, whenever the sample number changes (for standards), or if the sample name changes (for unknowns or wavescans).

**Special note:** there are three varieties of formats (type = 1 or type = 2 or type = 3) used in position import and export files. The format of the position import and export file is specified by the PositionImportExportFileType parameter in the PROBEWIN.INI file. If the parameter is not specified in the INI file then the default of type = 1 is used.

The first format is the original import/export format (type = 1) and contains the fields shown in the example below without the autofocus flag and analytical setup number fields shown in parentheses at the end of the position lines.

The second format is a newer import/export format (type = 2) which contains two additional integer fields and an additional string field for each position lines. These additional integer fields are used for an auto focus flag and an analytical setup number. The analytical setup number can be used by Probe for EPMA to specify which previously saved sample setup is to be used when acquiring data for the position sample. Note that all positions in a single position sample must use the same setup number. If more than one setup number is specified within a position sample, the program will use the last analytical setup specified. If the setup number specified is not available in the current Probe for EPMA run, then the program will base the automated acquisition on the last unknown sample. The autofocus flag is used to define a "digitized" autofocus (if supported by the hardware interface) for each position. If this autofocus flag is zero, then no autofocus is attempted, if the flag is -1 or 1 then an autofocus will be attempted at that position. The adjustment from the auto-focus will be applied to all positions in that position sample.

The additional string field is used to specify a file setup name that can be used for automated designation of a file setup from an existing Probe for EPMA data file.

The importing positions with a pre-defined file setups, analytical setup numbers (and autofocus flag) are useful when digitizing position samples off-line on an optical microscope using program STAGE. However, it is important when digitizing off-line to ensure that the setup number specified during the digitizing process will eventually correspond to the correct setup number created in the Probe for EPMA run in which the digitized position samples will be automatically acquired. Also that the specified file setup data file exists and is updated with valid standardization data.

```
0.0, 0.0, 0.0, 0.0
0.0, 0.0, 0.0, 0.0
0.0, 0.0, 0.0, 0.0
2, 1, "metallic phase #1", 15.234, 18.12, 10.873, 1.0, 1, (1, 4, "")
2, 1, "metallic phase #1", 15.547, 18.43, 10.873, 1.0, 1, (0, 4, "")
2, 1, "metallic phase #1", 15.698, 18.56, 10.873, 1.0, 1, (0, 4, "")
2, 2, "Si3N4 ceramic matrix", 15.747, 18.34, 10.873, 1.0, 1, (1, 7, "")
2, 2, "Si3N4 ceramic matrix", 15.747, 18.34, 10.873, 1.0, 1, (0, 7, "")
2, 2, "Si3N4 ceramic matrix", 15.747, 18.34, 10.873, 1.0, 1, (0, 7, "")
```

The format of these standard, unknown or wavescan position import files (the above example contains two unknown position samples with three positions each) is described below. Note that the first three lines are always used to define the coordinates of the 3 physical fiducial marks used for transformation of pre-digitized standard mounts. All parameters are comma, space or tab delimited.

Following the first 3 lines which define the x, y, z,coordinates for the three fiducial marks (used for transformation of pre-digitized standard mounts), each following position line has the following format (the fourth column is not utilized at this time) :

- the first column contains the sample type (1 = standard, 2 = unknown, 3 = wavescan)
- the second column contains the sample number, which for standards is the standard number for a standard as defined in the STANDARD.MDB database. For unknowns and wavescans it is an arbitrary number that is ignored.
- the third column is the sample name, which is optional for standard position samples and must be enclosed in double quotes
- the fourth, fifth, sixth and seventh columns contains the x, y and z coordinate positions (seventh column is not utilized at this time).
- the eighth column value is the grain number, which is used for automatically blanking the beam between successive points if different from the preceding point

- the ninth column value (only if PositionImportExportFileType = 2 or greater) is the autofocus flag, which is used for automatically performing an auto focus for each position. Specifically the third autofocus flag option (1= every sample, 2=every point, 3=digitized, 4=interval).
- the tenth column value (only if PositionImportExportFileType = 2 or greater) is the analytical setup number, which is used for automatically loading a previously created sample setup in a Probe for EPMA data file. If the specified analytical setup number is not found, the program will load a sample setup based on the last unknown sample in the run.
- the eleventh column value (only if PositionImportExportFileType = 3) is the analytical file setup name. If the specified file setup name is not found, an error is generated.

Note that standard coordinates can be imported in any order and all positions of a single standard will be appended to a single standard sample automatically. However, if a standard position sample already exists, the program will ask if the user wants to overwrite the existing standard coordinate data, so generally it is a good idea to group all coordinates for a single standard contiguously.

If no fiducial transformation of the imported positions is required, simply click "No" when asked whether to perform the fiducial transformation.

For unknown and wavescan positions these fiducial positions may be used for transformation of sample positions digitized on an optical microscope equipped with a digitizing stage using a different coordinate system than the microprobe stage. All linear transformations in scale, rotation and translation are handled. Although the use of three fiducial coordinates means that the procedure is capable of a three dimensional transformation (using x, y and z), the z coordinate transformation may be ignored, if desired by the user, for unknown or wavescan position transforms.

## Export Selected Samples

This button can be used to export selected position samples to an ASCII file. This operation is intended primarily for saving digitized standard positions to the .POS files; individual users can save specific subsets of their standard positions. Unknown and wavescan positions can also be exported to ASCII files.

If the selected position samples were referenced to fiducials, then the program will also export the fiducial positions for subsequent fiducial coordinates transformation at another time.

## Import \*.LEP

This menu will load a custom position file. This format is a fixed length field ASCII file that contains off-line digitized data from an automated Leica light microscope. This format is intended for use with Canadian Geological Survey laboratory .LEP position files only. The file format is as follows:

|        |         |        |       |          |     |      |   |   |
|--------|---------|--------|-------|----------|-----|------|---|---|
| REF #1 | -195279 | 80485  | -2130 |          | LEP | SH # | 2 | 1 |
| REF #2 | -55671  | 9769   | -5227 |          | LEP | SH # | 2 | 1 |
| REF #3 | -222595 | -40036 | 3740  |          | LEP | SH # | 2 | 1 |
| OXIDE  | -192879 | 80303  | -2237 | 201023-1 |     |      |   | 1 |
| OXIDE  | -187449 | 80303  | -2237 | 201023-2 |     |      |   | 1 |
| OXIDE  | -182519 | 80472  | -2237 | 201023-3 |     |      |   | 1 |
| OXIDE  | -162776 | 80178  | -3229 | 201023-4 |     |      |   | 1 |

See additional information on the "Import From ASCII File" button in the Automate! window and also the PositionImportExportFileType parameter in the PROBEWIN.INI file.

## Import \*.DCD

This menu will load a custom position file produced by Microbeam Services optical digitizer. This format is as follows:

The first two rows are the two reference points P1. The following S1.... are sample points.  
Columns 1, 2, 3 are XYZ

Column 4 is the analysis setup, which can be pre-programmed.  
Column 5 is the sample description  
Column 6 is the sample number (P for reference, S for single point...)  
Columns 7-9 are used as number within dataset etc. and not really important.

This format is intended for use with Microbeam Services .DCD position files only.

## Peak Spectrometers

Clicking the "Peaking" button opens the peak center window. All currently analyzed elements are listed. The Peak Center method can be specified. A choice of Interval Halving, Parabolic and ROM based (if supported by the hardware) is available. The default peak center method can be specified from the PROBEWIN.INI file. With all methods the program will perform a peak to background measurement to determine if the peak is statistically significant enough to attempt a peak center.

Three options are offered, the first an option move the spectrometers to their on-peak positions if selected in the element list. This option is useful for moving the spectrometers to calibrated peak positions for x-ray mapping purposes.

The second option forces the program to acquire a "pre-scan" wavescan just prior to the peak center procedure to allow the user to select an initial peak center start position. This last option is particularly useful in cases where the nominal spectrometer position is badly out of mechanical calibration and the actual peak position is some distance from the theoretical peak position. One can also specify as a third option a "post scan" to be executed after a peak center to confirm that the peak center method was properly performed. This option gives the operator maximum control over the automated determination of the peak "center". Here the peak center found by one of the 3 methods can be verified, by a slow step across the top of the centroid.

The Plot Selected Peak Center button will display graphically, the last peak center attempted for the selected element. The points displayed are numbered in the order acquired. Therefore, for example, if the parabolic peak center was performed, and an optimum peak center was obtained, there will be four points displayed: the three (on, hi, lo) for the parabolic fit and a fourth new on-peak based on the calculated peak center.

The **Interval Halving** method is the most accurate, but the slowest. It is based on a peak crawl that changes direction and halves the step size whenever the count intensity drops by more than a standard deviation.

The **Parabolic** method peak center uses a moving three point fit on the peak top that is fit to a 2nd order polynomial. The centroid of the parabola is returned as the new peak center. This method is the fastest, if the spectrometer position is already close to the actual on-peak position.

The **ROM Based** method peak center is available if the hardware interface supports a ROM peak center. The actual method used for the ROM Based peak center depends on the microprobe hardware.

Several peak center fit types are available. Applies only to Cameca SX100/SXFive and JEOL 8200/8900 micropoles.

- 0 = Internal (the instrument ROM peak method which applies only to Cameca)
- 1 = Parabolic (spectrometer scan data is fit to a parabolic fit)
- 2 = Maxima (spectrometer scan data is fit to Brent's Maxima function)
- 3 = Gaussian (spectrometer scan data is fit to a Gaussian fit)

The internal ROM type is available only on Cameca instruments. The other options are available on JEOL 8200/8900/8500/8x30 and Cameca SX100/SXFive instruments. The parabolic and maxima fits require at least 3 data points above the threshold (see below). The parabolic and maxima methods have a user defined threshold that can be specified in the INI file and the program.

The ROM peaking thresholds are designed to allow the user to define the intensity values above which are used for one of the three ROM fitting methods (parabolic, maxima or Gaussian). The default is

0.33 which means that all intensity values 1/3 above the minimum to maximum intensity range are used in the fit.

The **Manual** method peak center is very simple utilizing only a pre-scan, P/B check and post-scan.

The program will automatically select elements in the element list that were not peaked in the previous peak center when the window is re-opened again. Note that the element list is multi-select so that any combination of elements can be peaked. If more than one element on the same spectrometer was selected, then the program will automatically peak center them one at a time. The Plot Selected Peak Center button may be used to display the last peak center count-position data for the selected element.

**The move to on-peak option is useful for moving a spectrometer (and setting the PHA conditions) to its on-peak position for imaging purposes. Remember : the elements to peak center (or move to on-peaks) must be highlighted before closing the dialog.**

### **Spectrometer Peak Center Scans**

All spectrometer peak center procedures may be prefaced with a spectrometer peak scan to allow the user to manually specify the nominal peak center position. This spectrometer peak scan is performed using the number of peak scan steps as defined in the Peak/Scan Options dialog. The count time for each peak scan point is based on the peak count time (divided by 4) as defined in the Count Times dialog.

### **Initial Peak-To-Background Measurements**

The program always performs a peak-to-background and peak intensity check before the actual peak center procedure. The peak-to-background is calculated by measuring the count rate at the nominal peak position and the current off-peak positions using the peak count time for each position. The measured count data must meet the required minimum peak-to-background and minimum peak count intensity as defined in the Peak/Scan Options dialog, before performing the actual peak center procedure.

If performing the peak center from the Automate! dialog, the program will not proceed with acquisition of sample data if one or more spectrometer fails to complete a peak center procedure for any reason.

### **Spectrometer Peak Center Procedure**

Three peak center procedures are used by Probe for EPMA. They are the Interval Halving, the Parabolic Fit and the ROM based peak center procedures. Note the following parameters apply to non-ROM based and ROM based peak scan parameters:

#### **Non-*ROM* scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

#### ***ROM* scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

#### ***ROM* Based Peaking Scan:**

- fine scan uses peaking time divided by 4 times 1.0
- coarse scan uses peaking time divided by 4 divided by 2.0
- fine scan uses peaking time divided by 4 times 1.5

## **Interval Halving**

Interval halving is a peak center procedure based on a peak crawl starting at the nominal peak position. The step size of the peak crawl is halved and the direction reversed whenever the count rate drops by more than a single standard deviation (square root of the intensity). The interval halving procedure is slow but extremely precise.

The initial peak step interval is always in a positive direction and the peak interval size is based on the Peaking Start Size as defined in the Peak/Scan Options dialog. Note that the default peaking start and stop sizes from the SCALERS.DAT file are modified by the following expression :

$$S_{actualstart} = S_{start} * \sqrt[3]{\frac{2d_{OnPeak}}{2d_{LiF200}}} (* 2) \sqrt{\frac{P_{HiLim}}{P_{OnPeak}}}$$

Where :

|                   |  |
|-------------------|--|
| $S_{actualstart}$ | is the actual calculated step size             |
| $S_{start}$       | is the spectrometer default start or stop size |
| $P_{HiLim}$       | is the spectrometer high limit                 |
| $P_{OnPeak}$      | is the spectrometer on-peak position           |
| $2 d_{OnPeak}$    | is the 2d spacing of the analyzing crystal     |
| $2 d_{LiF200}$    | is the 2d spacing for LiF (4.0267)             |

From this expression one can see that the actual start and stop sizes are unchanged at the spectrometer high limit when an LiF crystal is used, and are increased for lower spectrometer positions and larger crystal 2d spacings. The calculated start and stop sizes are also doubled when the crystal 2d spacing is larger than 30 angstroms (LDE crystals).

When the interval step size has been halved to a value less than the peaking stop size, the spectrometer is considered to be peak centered and the current position is saved as the new peak position.

If the number of intervals measured exceeds the "Maximum Peaking Cycles" as defined in the Peak/Scan Option dialog, the program terminates the peak center procedure with an error.

## **Parabolic Fit**

The parabolic peak center procedure is based on a moving three point triplet that attempts to fit an inverted parabola. The three positions measured are the middle, high and low positions. The middle position is initially the nominal peak position and the high and low positions are offset from the middle position based on the value of the Peaking Start Size, which is calculated using the process described in the interval peaking procedure above. Ideally the Peaking Start Size calculation should be adjusted to yield an offset which places the high and low positions at a position which produces approximately 1/2 the maximum intensity of the peak, for a more precise parabolic fit.

The peak center procedure then determines if the high and low position intensities are at least one standard deviation less than the middle position intensity. If the three intensities meet this criteria, the program calculates the centroid of a parabola based on the count-position data triplet. The results of the centroid peak fit can be viewed by placing the program into "Debug Mode" under the Output menu.

If the data cannot be fit to a parabola, the middle position is shifted by an amount equal to 1/2 of the Peaking Start Size and the middle, high and low position intensities are measured again.

If the number of count-position data triplets measured exceeds the "Maximum Peaking Cycles" as defined in the Peak/Scan Option dialog, the program terminates the peak center procedure with an error.

## **ROM Peaking**

The ROM peaking procedure is available for those interfaces that support an integrated ROM based peaking procedure. These are the JEOL 8900/8200/8500/8x30 and SX100/SXFive interface types. Note the following parameters apply to non-ROM based and ROM based peak scan parameters:

### **Non-*ROM* scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

### ***ROM* scan based**

1. Wavescan: # of points, wave count time
2. Quickscan: Spectro scan speed (%), quick count time

### ***ROM* Based Peaking Scan:**

- fine scan uses peaking time divided by 4 times 1.0
- coarse scan uses peaking time divided by 4 divided by 2.0
- fine scan uses peaking time divided by 4 times 1.5

The actual peaking procedure utilized by each interface is somewhat different but usually based on a parabolic fit of some variety. The program passes the "Peaking Start Size" to each interface specific ROM Peaking routine where it is modified if necessary.

Several ROM based peak center fit types are available. Applies to Cameca SX100/SXFive and JEOL 8200/8900/8500/8x30 microprobes.

- 0 = Internal (the instrument ROM peak method which applies only to Cameca)
- 1 = Parabolic (spectrometer scan data is fit to a parabolic fit)
- 2 = Maxima (spectrometer scan data is fit to Brent's Maxima function)
- 3 = Gaussian (spectrometer scan data is fit to a Gaussian fit)
- 4 = Dual ROM (Maxima for LiF crystals, Parabolic for other crystals)
- 5 = Dual ROM (Maxima for LiF and Gaussian for other crystals)
- 6 = Highest Intensity (using smoothed intensities)

To increase the default ROM scan width decrease the Peakscan Size (line 16) or increase the LiF Peaking Start Size (line 19) in the SCALERS.DAT file. Note that within the application, the ROM based peak scans are based on the Peaking Count Time divided by 4 and the number of Peak Scan Points. The ROM scan width is determined by the Peaking Start Size.

The internal ROM type is available only on Cameca instruments. The other options are available on JEOL 8200/8900/8500/8x30 and Cameca SX100/SXFive instruments. The parabolic and maxima fits require at least 3 data points above the threshold (see below). The parabolic and maxima methods have a user defined threshold that can be specified in the INI file and the program.

The ROM peaking thresholds are designed to allow the user to define the intensity values above which are used for one of the three ROM fitting methods (parabolic, maxima or Gaussian). The default is 0.33 which means that all intensity values 1/3 above the minimum to maximum intensity range are used in the fit.

**JEOL 8900/8200/8500/8x30 (InterfaceType=2)-** The "Peaking Start Size" is used to calculate the spectrometer scan width for the peak scan procedure. The coarse scan width is 3 times larger than the fine scan width.

$$S_{ScanWidth} = \frac{(P_{HiLim} - P_{LoLim})}{P_{factor}} \cdot \frac{P_{StartSize}}{P_{LiFStartSize}}$$

|         |                    |   |
|---------|--------------------|---|
| Where : | $S_{ScanWidth}$    | is the calculated peaking scan width                                      |
|         | $P_{HiLim}$        | is the spectrometer high limit (from MOTORS.DAT)                          |
|         | $P_{LoLim}$        | is the spectrometer low limit (from MOTORS.DAT)                           |
|         | $P_{factor}$       | is the peak scan size factor (from SCALERS.DAT)                           |
|         | $P_{StartSize}$    | is the crystal spectrometer peaking size (calculated for 2d and position) |
|         | $P_{LIFStartSize}$ | is the LIF spectrometer peaking size (from SCALERS.DAT)                   |

**SX100/SXFive (InterfaceType=5)-** The "Peaking Start Size" is modified to produce a number between 0 and 4 by utilizing the following expression :

$$pw = (M_{high} - M_{low}) * P / 3000000$$

where    pw      is the SX100/SXFive peak center width  
           M<sub>high</sub>    is the motor high limit  
           M<sub>low</sub>     is the motor low limit  
           P          is the Peaking Start Size

### **Manual Peaking**

The Manual method peak center is very simple. First a wavelength pre-scan is performed, after which the user is allowed to select the actual peak position. This is followed by a peak to background check, after which a wavelength post-scan is performed at a finer scale to allow the user to confirm the selected peak position. The user is then once again given the chance to adjust, confirm or cancel the peak center process.

Note that the final wavelength post-scan data will be saved to the Plot Peak Center array (see the Peaking dialog) for subsequent plotting or output by the user. Note that the post-scan range is 5% (+/- 2.5%) of the pre-scan range.

### **Final Peak-To-Background Measurements**

After a successful peak center procedure the peak-to-background is re-calculated by simply measuring the on-peak count rate at the new peak position. This result can be compared with the initial peak-to-background to evaluate the peak center procedure performance.

### **Post Scan Peak Confirmation**

This option gives the operator maximum control over the automated determination of the peak "center". Here the peak center found by one of the 3 methods can be verified, by a slow step across the top of the centroid. Optimal centering is left to the discretion of the user, but one strategy is to look for the small flat plateau that normally is present at the peak centroid, or looking at the centroid at 99% of maximum intensity. The values used are, respectively, the time value of the "peaking" in Count Times and the step width using 1/20 (5% or +/- 2.5%) of the width for peakscan limits, in Peak/Scan options.

### **Automation Actions**

The automation "actions" desired to be run are to be selected here. The program will execute each automation action for all selected samples in the order listed. Any combination of automation actions may be selected by the user.

Use the Confirm actions to simply confirm the position coordinates of the selected samples and the Acquire actions to actually acquire the data for the selected samples. To confirm positions and/or acquire data for both standards and unknown position samples, select the All Samples option and select the standards and unknowns desired. If a confirm action was selected, the program will ignore the setting of the Use Confirm During Acquisition for the confirmation of positions.

Use the "Acquire Standard Samples (again)" if it is desired to run the standards AFTER the unknown or wavescan samples have been run; of course this assumes that the specified standard mounts will still be present in the sample holder inside the instrument.

To use the **Peak Spectrometers** action, click the "Peaking" button to select the elements to peak on. If the Calibrate on Assigned Standards check box is checked the program will only peak the element on the assigned standard. If the Calibrate on Assigned Standards check box is not checked the program will attempt to peak on the first standard that it finds a sufficient peak to background ratio until all elements have been peaked.

If the Peak Spectrometers action was checked, the program will cease the automation process if one or more of the elements selected for peaking did not peak properly rather than continue any selected acquisition actions.

## Automation Options

The following automation options are supported by Probe for EPMA. Some options may not be available depending on the interface type and microprobe hardware.

### **Peak On Assigned Standards**

This option forces the program to only attempt a peak center on a standard position sample if the standard is assigned as the primary standard for that element. If the element has no assigned standard, then the program will attempt to assign one automatically based on the highest concentration of the element present among the standards in the run. To add additional standards to the run use the Standard | Add/Remove Standards To/From Run menu.

It is a good idea to check the standard assignments in the Analyze! window before running a peak center with this option selected to insure that the correct standard position samples are selected for peak centering.

### **Use "Quick" Standards**

Although it is generally recommended to acquire a complete analysis (acquire all elements based on the last unknown sample setup) for all selected standards, Probe for EPMA includes a feature which allows the acquisition of "quick" standardizations to save time. In this mode the program will, for each standard acquisition, acquire only those elements that are actually assigned to that standard. This includes both the primary standard assignments and the interference standard assignments (see the Standard Assignments button in the Analyze! Window), and also any MAN (mean atomic number) standard assignments for background fitting (See the MAN Fits menu under the Analytical menu).

### **Considerations in the Use of "Quick" Standards**

Although the use of "quick" standardizations can save considerable time, it is important to remember, that a complete acquisition (and hence analysis) of a standard enables the analyst to note the accuracy (Ziebold, 1967) of secondary standard elements and to observe on-peak and off-peak interferences. In addition, the complete acquisition (Use Quick Standards not checked) of data on standards used for the quantitative interference corrections is essential for this feature to work correctly if they have not been previously assigned.

However, sometimes for well characterized samples, it is feasible to acquire standard data only for those channels that are used as the assigned standard element for that channel. In these "quick" standardizations, Probe for EPMA will skip elements that do not contain any assigned elements for that standard.

In a typical run, one would normally acquire standard samples without the use of the "Quick" standards option. The resulting data can then be checked for interferences and background anomalies. Once these concerns have been addressed, then subsequent standardizations can be acquired using the "quick" standards option to save time.

### **Use of "Quick" Standards**

"Quick" Standards can only be acquired from the Automate! window and are based on the standard assignments of the last unknown (or last standard, if there are no unknowns in the run) as long as the Use Last Unknown (or Standard) option is selected. If another option is selected, for example, the Use Digitized Sample Setups option, then the program will base the "quick" standard on the standard assignments of the digitized sample setup.

For each selected standard in the position list, (in order from top to bottom), the program will check the primary standard, interference, and MAN standard assignments to determine whether an element should be acquired for a quick standard. If an element in the sample is actually utilized by the "quick" standard being acquired, then the program will retain it for the acquisition of intensities, otherwise the element will be skipped during the acquisition. Note that if no elements in the sample for a standard are assigned for any of these purposes (a true secondary standard), the program will acquire all the elements in the current sample setup for that standard.

Note that the program may warn the user that the element acquisition order has been changed to user defined to internally facilitate the acquisition of "quick" standards. However, the program will not change the actual order that the elements are being acquired.

### ***Use Filament Standby Afterwards***

This option (if supported by the hardware interface) is used to perform a filament shutdown after the automation actions have been completed. This is most useful for extended acquisitions overnight or over the weekend when it is desirable that the filament life be conserved as much as possible.

### ***Use Confirm During Acquisition***

This option will allow the user to perform a stage X, Y and Z adjustment just before the sample data is acquired on the first point of each position sample. The default confirm delay time is specified in the PROBEWIN.INI file. This option is ignored for Confirm automation actions.

### ***Use Beam Deflection For Position***

This option is available if an imaging interface is supported by the hardware interface (see the ImageInterfacePresent and ImageInterfaceType parameters in PROBEWIN.INI file). If this option is unchecked then normal stage motion is used to position the sample for acquisition. If this option is checked, then the program will utilize beam deflection for positioning the sample (beam position) for acquisition. This option affects only unknown and wavescan position samples (not standards).

This option is primarily intended for situations where very small beam diameters (low overvoltage) are used for precise positioning and acquisition at scales finer than the stage mechanism is capable of. Note that care must be taken to digitize positions that are all within the range of the current beam scan parameters and that a high enough magnification is utilized so that no spectrometer defocusing occurs.

For JEOL 8900/8200 instruments there is an option to suppress the stage backlash during the acquisition using beam deflection. This is for extremely high resolution analysis of single samples where it is desired that the stage not move at all.

### ***Confirm All Positions In Sample***

This option forces the program to move to EACH position in the selected position sample and allow the user to confirm the coordinates of that single point. This applies both to the explicit confirm action and to acquisition actions when the Use Confirm During Acquisition option is checked.

Note that even if this option is selected, only the first point in a position sample will be used for a peak calibration procedure. In addition, any coordinates for a standard position sample that are "automatically generated" during the acquisition process will be ignored for the explicit confirm action even if the Confirm All Positions In Sample option is checked.

### ***Combine Multiple Sample Setups***

This option allows the user to either run, for each position sample, each specified multiple sample setup as a separate sample (unchecked) or as a single combined sample (checked). This option only affects position samples that have been assigned multiple sample setups.

Specifically, if this checkbox is not checked, a separate sample based on each setup will be acquired for each position sample based on each setup specified in the multiple setup dialog. If it is checked then all specified multiple setups will be combined into a "combined condition" sample (each element can have different column condition parameters) and the

software will adjust the column conditions as necessary to acquire the elements as defined. Note that these defined multiple setups are just sample setups that could contain single column conditions or could even be multiple column conditions sample setups themselves.

### **Use ROM Auto Focus**

This option (if supported by the hardware interface) is used to perform an automatic ROM based automatic focus on the sample surface. This option supports the following types of auto focus during an automated acquisition:

- 1 = auto focus on a new position sample only
- 2 = auto focus on every point in each sample
- 3 = auto focus using the digitized auto focus flags (or imported from a .POS file)
- 4 = auto focus using the specified auto focus interval

### **Standard Points To Acquire**

This parameter specifies the number of standard point acquisitions to acquire on each standard. If fewer than this many points are actually digitized in the position database, the program will automatically increment the X stage axis by the amount specified in the Standard X Increment field.

Since Probe for EPMA automatically uses the average of the sample count data for the calibration of quantitative intensities, it is recommended to acquire at least 5 to 15 points on each standard for accurate quantitative analysis.

### **Automate Confirm Delay**

This is the time in seconds that the program will utilize for the Confirm action. To change the default confirm delay, edit the parameter in the PROBEWIN.INI file.

### **Standard X Increment**

This is the distance in microns that the program will automatically increment the X stage axis for each standard coordinate acquisition, if the number of points specified in Standard Points To Acquire is less than the number of standard coordinate positions actually contained in the position database. This value can be set to any distance from -100 to 100 microns.

### **Re-Standard Y Increment**

This is the distance in microns that the program will automatically increment the Y stage axis for each re-standardization. This parameter is used to avoid analyzing the same sample spot on the standard and should be set to a value somewhat larger than the typical beam size of the microprobe. This value can be set to any distance from -100 to 100 microns.

### **Re-Standard Interval (hrs)**

This parameter will (if box is checked) automatically re-run the selected standard samples after the specified interval of time (in hours). Upon completion of the re-standardization, the program will automatically re-start the interrupted unknown sample where it was previously left off.

### **Run Selected Samples**

This button starts the selected automation actions based on the selected samples. The program will confirm the number of standard, unknown and wavescan samples to be acquired during the automation.

The automation can acquire sample positions based on five different methods :

### **Use Last Unknown Sample**

The default method the program uses to acquire automated samples is to base the acquisition on the last unknown sample setup. If no unknown samples exist, the program will base the acquisition on the last standard sample setup.

If no unknown or standard samples exist, the program will not be able to run automated samples. Always create a default sample from the Acquire! window using the New sample button before attempting to run automated samples.

### ***Use Digitized Conditions***

If the microprobe interface supports computer control of the kilovolts, beam current and beam size then the user can specify that the program base the acquisition on the last unknown sample setup with these parameters based on the sample conditions specified when the sample positions were digitized. See the Conditions button above for more details on using this feature.

### ***Use Digitized Sample Setups***

Another method that can be used to acquire automated samples is to base the acquisition on sample setups that were specified when the sample positions were digitized. In this way, one can automatically data for different sample positions using different sample setups for tremendous flexibility. See the Sample Setups button above for more details on using this feature.

### ***Use Digitized File Setups***

Yet another method that can be used to acquire automated samples is to base the acquisition on file name setups that were specified when the sample positions were digitized. In this way, one can automatically data for different sample positions using different previously created file setups for tremendous flexibility. See the File Setups button above for more details on using this feature. Note that standard intensity data from the file setup will be automatically loaded (if it has not been already) unless the option has been turned off from the Acquisition Options window.

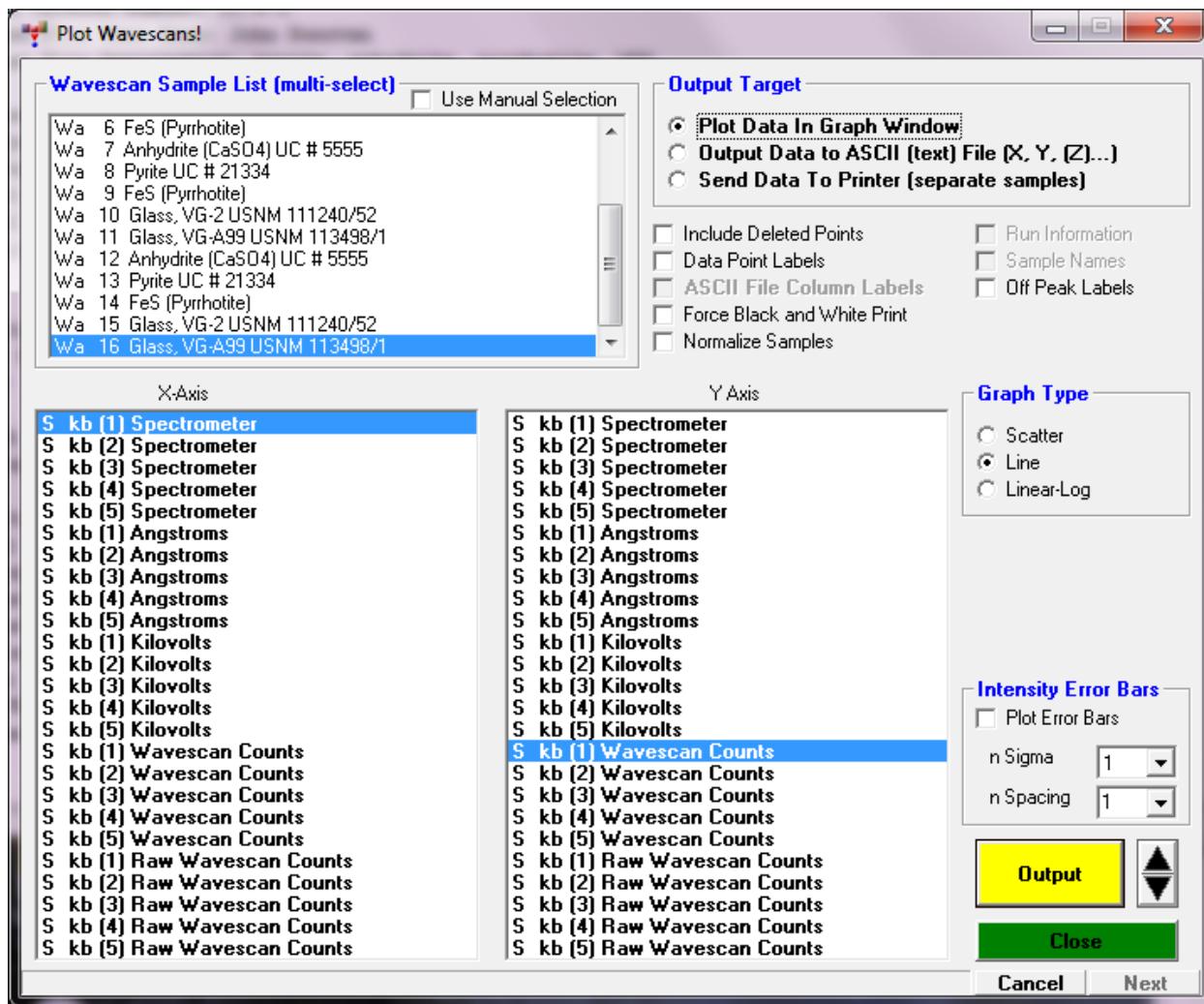
### ***Use Digitized Multiple Setups***

Finally as a last option one can acquire automated samples based on multiple sample setups, specified when the samples were digitized. This is of course similar to the sample Setups option above except that one can acquire more than one sample setup for each position sample. See the Multiple Setups button above for more details on using the feature.

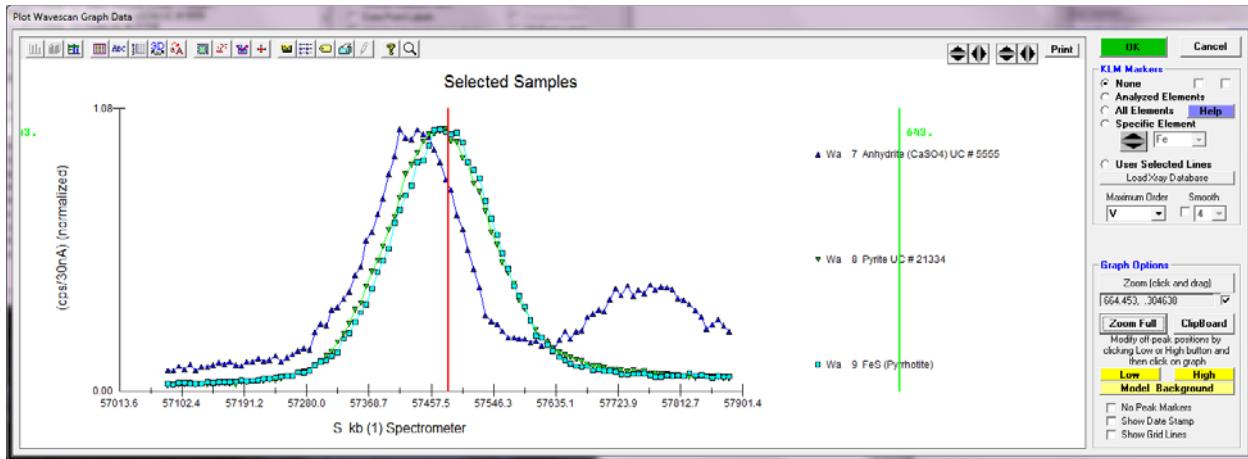
## Plot Waves cans!

The Plot Waves cans! modeless windows provides graphical plotting of spectrometer wavelength scans in Probe for EPMA. Standard, unknown and waves can sample data may be displayed from the Output | Plot Standards and Unknowns menu. Waves can samples can display KLM markers from the NIST database based on the analyzed elements in the range, all elements in the range or a specific element in the range.

Note that one can configure Probe for EPMA to specify elements that should never show up in the KLM markers. The default forbidden elements are Tc, Pm, Po, At, Rn, Fr, Ra, and Ac. See the [Plot] section of the PROBEWIN.INI file for instructions on adding or deleting elements from this list. Note that x-ray lines do not exist for H and He so they are automatically excluded from the KLM database.



Background types (linear, high only, low only, average, etc.) can be modeled based on existing waves can samples, evaluated and updated for both standard and unknown samples using the Model Background dialog.



## Output Target

The user may select either output to the Graph window or to a tab delimited ASCII file or to have each sample plot data automatically output to the default printer. If ASCII file output is selected, the program will ask for the output filename (\*.DAT extension) after calculation of all data has been performed. The default is to send data to the graph window for viewing.

### **Include Disabled Points**

This option, if selected, will output data from samples even if the data has been previously flagged as disabled. This option has no effect on quantitative sample output, which means that the disabled points will be output to the graph or output file, but the calculated values will be replaced with a small number (NotAnalyzedValue! = 0.00000001) equal to  $10^{-7}$ .

### **Data Point labels**

This option will display a line number for each value plotted in the graphical display mode. This option has no effect if ASCII file output is selected.

### **ASCII File Column Labels**

This option will output a line of data column labels for each column of data in the ASCII file. The labels will be enclosed in double quotes. If the elements being output change during the output process, the program will automatically write a new column label line. This option has no effect if Graph output is selected.

### **Run Information**

This option will output a header to the ASCII file including the user, title and description of the current run. In addition the current time and date will be displayed also. This option has no effect if Graph output is selected.

### **Sample Names**

This option will output a line containing the sample name in double quotes before the data for that sample is output to the ASCII file. This option has no effect on data sent to the graph window.

### **Force Black and White Print**

This option will force a color printer to print graphs in black and white instead of in color. This option applies to both the Print button in the Graph window as well as the Send To Printer option in the Plot! Window. In color mode monochrome printers (laser printers) will print grayscale and color printers will print color. In the Force Black and White mode, both types of printers will print black and white only. This option saves toner for monochrome printers, because the lightly colored background is not printed at all.

### **Off Peak Labels**

This option will add off-peak labels to the off-peak markers. Note that both actual and current off-peak markers and labels are shown. Actual off-peak markers are the positions of the off peaks when the sample was acquired while the current off-peak markers are the off-peak positions that are currently specified.

The on-peak marker and label is shown in light blue, the current off-peak markers and labels in bright green and the original off-peak markers and labels are shown in magenta.

### **SURFER .BAS File**

If the SURFER .BAS File option is selected, Probe for EPMA, will, in addition to the actual data file, create a script file for use with SURFER (from Golden Software) for automatically generating contour and surface plots of your data. This assumes that the sample data is applicable to this type of processing.

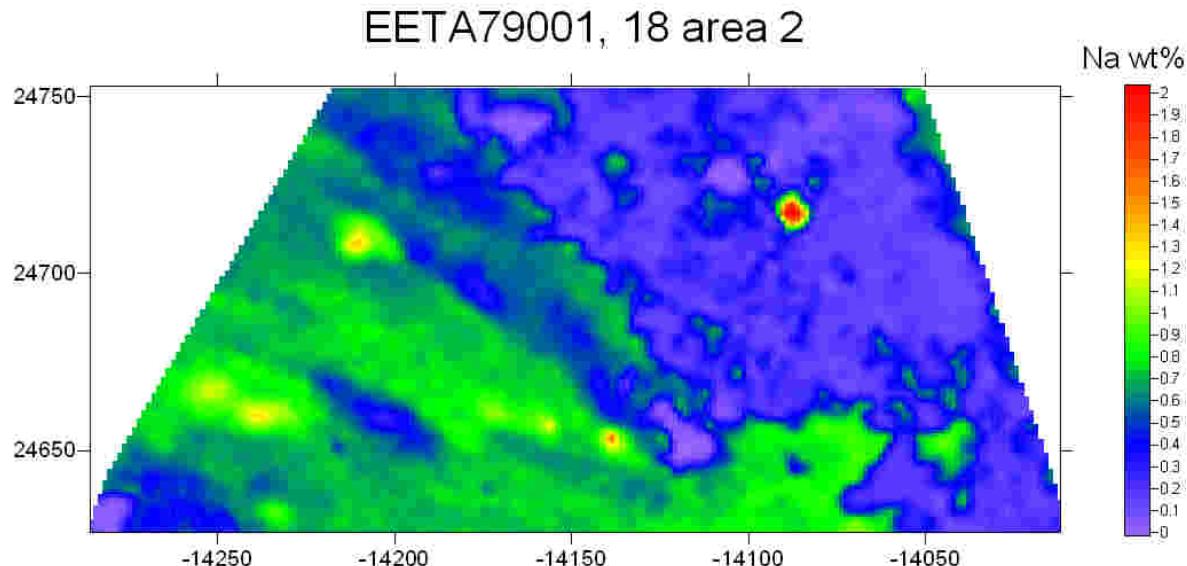
Be sure that the SurferOutputVersionNumber parameter is correctly specified in the PROBWIN.INI file when using this option. Probe for EPMA assumes Surfer v. 6.0, unless the parameter is specifically indicated. Due to changes in the file formats expected by Surfer v. 6 and Surfer v. 7, the correct script format must be output or the script will not run properly.

If the sample was a digitized polygon area grid, then the program will also automatically output a .BLN file for use with the SURFER surface and contour plot boundary feature. Notice that the digitized polygon grid coordinates are saved automatically and can be viewed, once the data is acquired, using the "plot" window to display digitized samples.

*Be aware that SURFER expects X and Y coordinate data in the first two columns of the .DAT file, followed by multiple columns of Z data for plotting contours or surfaces. To accomplish this, be sure to select the 3-D graph type and select the X coordinate data in the x-axis list box, the Y coordinate data in the y-axis list box and the Z data (weight percents, etc.) in the z-axis list box using multiple selection.*

The creation of the SURFER .BAS file requires that the GRIDBB.BAS to be present in the program data folder (**usually C:\ProgramData\Probe Software\Probe for EPMA**).

An example of the type of output that is obtained when polygon (or rectangular) grid quant data is exported from the Plot! Window using the “SURFER .BAS File” checkbox (first click the “Send Data To ASCII File” option) and the resulting .BAS script is run from the Golden Software Scripter applet is shown here:



## ***SURFER Instructions***

Probe for EPMA can create a .BAS basic script file containing OLE code for creating contour and surface plots, or just screen display for testing purposes. This .BAS basic file is only to be used with the GS Scripter utility that comes with SURFER v. 6.0 or higher.

To run the .BAS script, double click the GS Scripter icon, select File, Open and double click the correct .BAS file. Then click Run, Start to begin the automated plotting.

**IMPORTANT!** Note that the default output mode of the script file is "TEST", which will only output the plots to the screen and will produce a log of the plotting operations. To produce output to the default printer, comment out the line : OutputType\$ = "TEST" by inserting a single quote in front of the line like this : 'OutputType\$ = "TEST", and Uncomment the line : OutputType\$ = "SURF" by removing the single quote in front of it.

Remember, the last uncommented "OutputType\$" line will select the output. If necessary, the "GridMethodType%" can also be changed in the same fashion to change the gridding method used by the grid module. Note also, that any element column plot can be skipped by simply commenting out the appropriate "ZLabel\$" lines, by inserting a single quote in front of the line.

## ***Normalize Y Sets***

This option will normalize data from different Y (axis) data sets for both Graphing and ASCII output. Only data from the Y-axis list will be normalized. This might be useful to compare trends between different elements.

## ***Normalize Samples (Y Sets)***

This option will treat each Y-axis data separately, but will normalize Y data sets between different selected samples. This can be useful when comparing wavescan data from two separate wavescans. Note that the Normalize Y Sets and the Normalize Samples (Y Sets) options can be combined.

## ***Graph Type***

The various graph types are selected here. The default graph type may be set from the PROBEWIN.INI file. Currently the following graph types are supported :

- Scatter (x and y data as points)
- Line (x and y data as points connected by lines, suitable for wavescan samples)
- Lin-Log (x data on a linear axis and y data on a log axis)
- 3-D (x, y and z data as points)

## ***Analyzed Elements Only***

This option will re-load the axis lists to display either the analyzed elements only, or both the analyzed and specified elements. This option is used to allow the user to graph or output those elements calculated by difference or stoichiometry.

This option can also be utilized in order to output all elements in a run, even if the last sample does not contain elements analyzed and/or specified in other samples. Consider that because the elements listed in the axis lists are based on the last sample selected, if a selected sample prior to the last selected sample contains additional elements not in the last selected sample, they will not be output simply because they cannot be selected. Therefore, if it is desirable that these additional elements be output, follow these directions:

- From the Analyze! window sample list, select the last sample that will be output from the Plot! window.
- Click the Elements/Cations button.
- Add, as specified elements (no x-ray symbol), all additional elements (found in previous samples) that are desired to be output, that are not already in the last sample.

- Click OK and return to the Plot! window and uncheck the Acquired Only button. This will allow the program to display all elements in the last selected sample, including the specified elements just added.
- Select all elements to be output. The program will output the selected elements whether they are analyzed or specified.

### **Average Only**

This option will output only the data average of each sample to the plot window or the output ASCII data file. Note, if data point labels are selected for the plot window, instead of line numbers the program will use sample numbers.

### **Minimum Total**

This option (if selected) will output only those data lines whose totals are at least the value specified. This option must be used with at least one column of quantitative data selected for graph or file output.

### **Error Bars**

This option will allow the program to calculate one sigma error bars for certain data types, specifically the Elemental, Oxide Percents and On Peak Counts data types only.

#### **Plot Error Bars**

This option will allow the program to plot one sigma error bars for each data point in each data set. The bars will be centered on the data point.

#### **Plot Nth Bars**

This option will allow the program to plot N sigma error bars for each data point in each data set as above, based on the user selected number of sigma values (1,2 or 3) and data point spacing.

#### **Error Bar Calculations**

The formula for these error calculations are as specified:

##### **On Peak Counts Errors:**

$$\Delta I = \sqrt{I_p}$$

Where:  $I_p$  = On-peak counts (including background, de-normalized for deadtime, beam current and time)

Note that the result is normalized to deadtime, beam current and count time after the square root is calculated and before it is displayed.

##### **K-Ratio Errors:**

$$\Delta K = \frac{\sqrt{I_u}}{(I_s * ZAF_s)}$$

Where:  $I_u$  = Unknown on peak counts (background corrected, de-normalized for deadtime, beam and time)

$I_s$  = Standard on peak counts (background corrected, de-normalized for deadtime, beam and time)

$ZAF_s$  = ZAF correction for standard counts (to obtain pure element counts)

Note that the result is normalized to deadtime, beam current and count time after the square root is calculated and before it is displayed.

### Elemental (or Oxide) Weight Percent Errors:

$$\Delta C = \Delta K * ZAF$$

Where:  $\Delta K$  = K-ratio error (from above)  
 $ZAF$  = unknown ZAF correction factors

### Output

This button will start the output process selected based on the Output Target type (graph window or ASCII file) selected by the user.

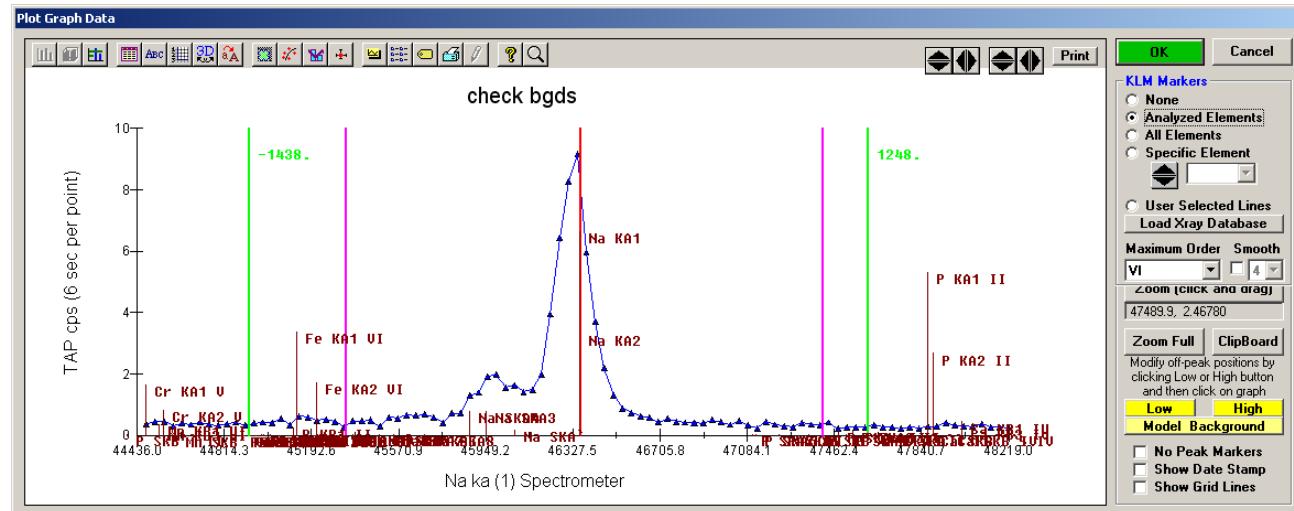
### Graph Data

The Graph Data window is used in two modes. The first to plot x-ray intensity or compositional data from standard and unknown samples, and the second to plot x-ray intensity and spectrometer position data for wavescan samples. Multiple Y data is automatically plotted and listed in a color coded legend if specified in the Plot! window.

Note that for wavescan plots if the on and off-peak positions are plotted, the markers for the on-peak position is cyan, the current off-peaks are green and the original off-peaks are magenta.

Digitized x and y position data from polygon boundary acquisitions may also be plotted in this window if this data exists in the current probe database file.

Numerous plot properties may be modified using the tool bar buttons at the top of the graph. A print button has also been added to facilitate printing but other print and output options can be accessed using the System tool bar button (printer icon).



### ASCII File Output

If ASCII file output was selected, the program will calculate data for the specified samples and then prompt the user for a path and filename to save the data to. All data files will be <tab> delimited and will normally have a .DAT extension.

### Plotting Standard and Unknown Data

Both x-ray and compositional data may be plotted in the Graph Data window. Click and drag any portion of the graph for zooming into a region of the graph.

Click the Zoom On button to use "Hot Hit" mode which allows the user to click near a specific symbol to get the exact x and y data values. In this mode the click and drag zoom feature is disabled. Click the "Hot Hit" button to restore click and drag zooming.

### **Plotting Wavescan Data**

When wavescan data is plotted in the Graph Data window additional options for wavelength dispersive data are enabled.

The on-peak marker and label is shown in light blue, the current off-peak markers and labels in bright green and the original off-peak markers and labels are shown in magenta.

### **KLM Markers**

- None: No KLM markers (default).
- Acquired only: All KLM markers for all elements in the wavescan sample (acquired and specified).
- All Elements: All KLM markers for all elements in range.
- User Selected Lines: Only lines selected by the user in the Load Xray Database window.
- Specific Element Only lines for the element selected in the list.

*Note that the emission line wavelengths are read from the XLINE.DAT file which are self-consistent with the XEDGE.DAT x-ray edge energies for quantification, but may vary slightly from the values from the NIST KLM x-ray database. Note also that all KLM wavelengths are corrected for refractive index values before display (based on the analyzing crystal used for the acquisition), but all KLM lines are systematically shifted so the 1<sup>st</sup> order KLM lines coincide with the emission line wavelength from the XLINE.DAT file.*

Note that one can configure Probe for EPMA to specify elements that should never show up in the KLM markers. The default forbidden elements are Tc, Pm, Po, At, Rn, Fr, Ra, and Ac. See the [Plot] section of the PROBEWIN.INI file for instructions on adding or deleting elements from this list. Note that x-ray lines do not exist for H and He so they are automatically excluded from the KLM database.

### **Load X-ray Database**

Allow the user to see a list of all files in the graph range. If the User Selected Lines option is clicked, the users can select specific lines for graphing.

**Xray Database**

**NIST Xray Lines (multi-select)**

| Xray Line                 | Angstroms      | Energy         | Intensity      | Reference |
|---------------------------|----------------|----------------|----------------|-----------|
| Dy LA1                    | 1.90926        | 6.49390        | 100.000        | ES        |
| Mn KB3                    | 1.91067        | 6.48910        | 6.80000        | ES        |
| Mn KB1                    | 1.91067        | 6.48910        | 11.8850        | ES        |
| Dy SLA3 <sup>~Z</sup>     | 1.91114        | 6.48750        | 1.00000        | ES        |
| <b>Mn SKB<sup>~</sup></b> | <b>1.91403</b> | <b>6.47770</b> | <b>1.00000</b> | <b>ES</b> |
| Dy LA2                    | 1.92017        | 6.45700        | 11.3400        | ES        |
| Eu LB1                    | 1.92077        | 6.45500        | 43.9040        | ES        |
| Eu LB4                    | 1.92587        | 6.43790        | 8.74000        | ES        |
| Fe SKA3 <sup>~</sup>      | 1.92617        | 6.43690        | 1.00000        | ES        |
| Fe SKA4 <sup>~</sup>      | 1.92676        | 6.43490        | 1.00000        | ES        |
| Fe SKA3                   | 1.92787        | 6.43120        | 1.00000        | ES        |
| Pm SLB2 <sup>~1</sup>     | 1.92844        | 6.42930        | 1.00000        | ES        |
| Fe SKA3 <sup>~</sup>      | 1.92886        | 6.42790        | 1.00000        | ES        |
| Fe SKA <sup>~</sup>       | 1.92997        | 6.42420        | 1.00000        | ES        |
| Fe SKA <sup>~</sup>       | 1.93344        | 6.41270        | 1.00000        | ES        |
| Fe KA1                    | 1.93636        | 6.40300        | 100.000        | ES        |
| Pm SLB2 <sup>~2</sup>     | 1.93636        | 6.40300        | 1.00000        | ES        |
| <b>Mn SKBN</b>            | <b>1.94012</b> | <b>6.39060</b> | <b>1.00000</b> | <b>ES</b> |
| Fe KA2                    | 1.94027        | 6.39010        | 51.2300        | ES        |
| Pm LB10                   | 1.94389        | 6.37820        | .540000        | ES        |
| Pr SLG1 <sup>~</sup>      | 1.94621        | 6.37060        | 1.00000        | ES        |
| Sm LB6                    | 1.94661        | 6.36930        | 1.40000        | ES        |
| Tm LI                     | 1.95527        | 6.34110        | 4.88900        | ES        |
| Ce LG3                    | 1.95557        | 6.34010        | 3.24000        | ES        |
| Pm LB2                    | 1.95628        | 6.33780        | 19.6000        | ES        |
| Pr LG1                    | 1.96148        | 6.32100        | 7.68700        | ES        |
| Sm LB3                    | 1.96276        | 6.31690        | 12.6690        | ES        |

Note that angstrom values in the above list with Bragg reflection orders higher than one (Roman Numeral "I") are NOT corrected for refraction index corrections. However, KLM markers displayed in the Graph Data wavescan plot dialog are corrected using the equation  $A' = A * (1 - (K / N^2))$

**Copy to Clipboard**    **Copy Selected to Clipboard**

To change the range enter the start and stop (ending) angstroms limits and click the Load New Range button. The keV parameter will limit the appearance of higher order lines that cannot be excited below the specified voltage. The Graph Selected button is only active if the User Selected Lines option is selected in the Graph dialog. The reference indicates the source. Specifically, ES is Eric Steele tabulated the first order lines, the higher order lines were calculated by JD (John Donovan).

Note that angstrom values in the x-ray database list with Bragg reflection orders higher than one (Roman Numeral "I") are NOT corrected for refraction index corrections. However, KLM markers displayed in the Graph Data wavescan plot dialog are corrected using the equation  $A' = A * (1 - (K / N^2))$  where A is the uncorrected angstrom position, K is the refractive index from the CRYSTALS.DAT file and N is the Bragg reflection order. From the expression  $\sin\theta = N * \lambda / (2d * (1.0 - K/N^2))$ .

The Maximum Order determines the maximum Bragg order that will be displayed that is less than the current operating voltage (not that some emission lines near the operating voltage that are displayed will not be generated since the actual energy required is always greater than the edge energy of the line in question). The default x-ray database XRAY.MDB displays up to 10<sup>th</sup> order lines. Additional x-ray databases are available with up to 20<sup>th</sup> order lines.

Note that one can configure Probe for EPMA to specify elements that should never show up in the KLM markers. The default forbidden elements are Tc, Pm, Po, At, Rn, Fr, Ra, and Ac. See the [Plot] section of the PROBEWIN.INI file for instructions on adding or deleting elements from this list. Note that x-ray lines do not exist for H and He so they are automatically excluded from the KLM database.

### Low and High Off Peak Positions

To move an off-peak (background) position for the next standard or unknown acquisition, click either the Low or High off-peak button. Position the cursor over the graph (note that the cursor changes to a cross-hair) and click where it is desired that the new off-peak position be located.

The on-peak marker and label is shown in light blue, the current off-peak markers and labels in bright green and the original off-peak markers and labels are shown in magenta.

If the current sample contains no data, the off-peak position is updated for that sample, otherwise a warning is given that the off-peak position selected will be applied to the next new sample that is created.

The new off-peak position is shown in yellow, while the original off-peak position for the displayed sample is shown in purple.

## Model Background

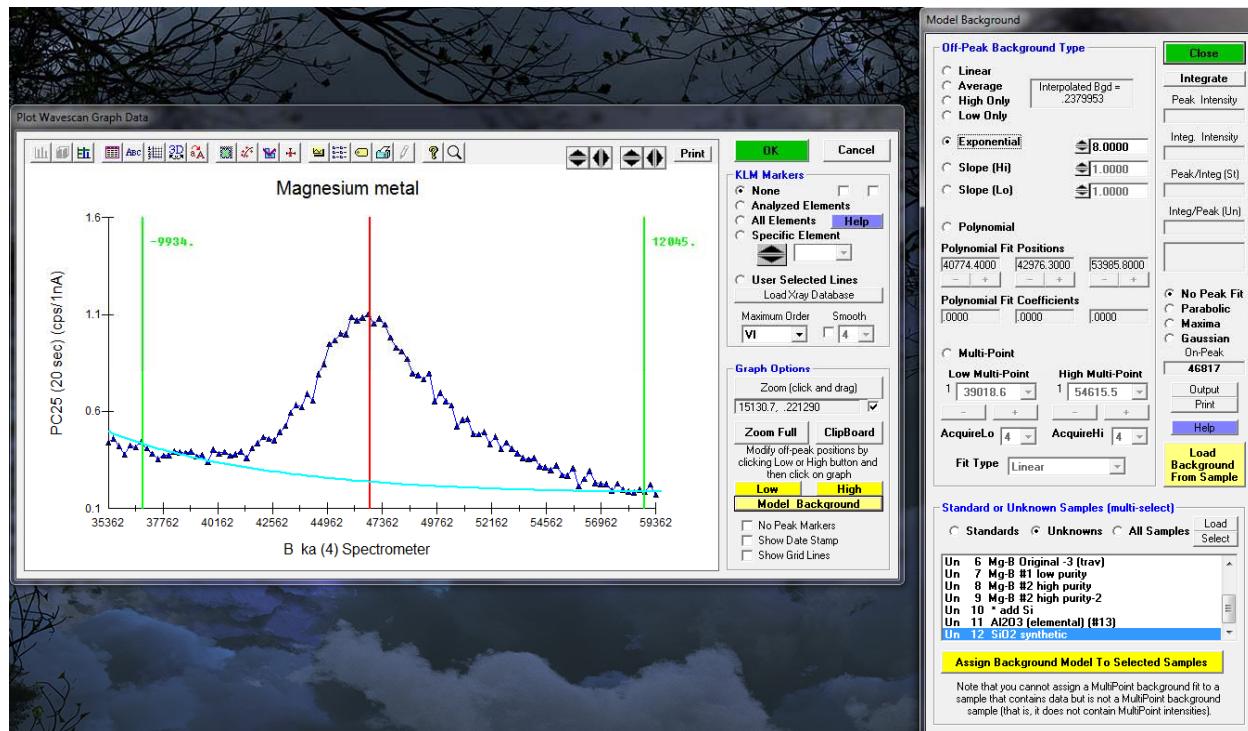
To model the various off-peak background correction types, click the Model Background button. This option is only available when a wavescan sample is displayed.

To see the background model, click the specific background type and observe the plotted background curve in the Graph window.

For the exponential, slope and polynomial off-peak background types coefficients are required. The exponential, slope-high and slope-low coefficients are entered by hand. The exponential exponent may be -6 to +6. For high or low only background types, a slope of one (1.) will produce exactly the same result as the high-only or low-only off-peak background type. In situations where there is some curvature and exponential cannot be used, examination of backgrounds on similar phases that lack the element of interest can give an appropriate "slope" factor to yield a proper background at the peak position. For example, if the high-only offset had a value of 10 and the slope factor entered was 1.3, the on peak background used would be 13.

The polynomial background fit coefficients are calculated automatically as the three different fit positions are moved by clicking the minus (-) or plus (+) buttons. The slope or polynomial coefficients can be applied to selected standard or unknown samples for use with a sample specific slope or polynomial off-peak background correction. That is, the actual slope or polynomial background correction will be calculated for each sample based on the actual off-peak data for that sample using the fit coefficients from the fit model.

To make the assignment to the selected unknown samples, be sure to click Ok in the Model Background dialog AND ALSO in the Graph Data dialog.



### **Calculating Specified or Compound APF Ratios**

To calculate specified or binary Peak/Integrated and Integrated/Peak ratios for use with the specified or compound Area Peak Factor (APF) correction methods, one might be required to perform careful high precision wavescans and calculate the peak and integrated intensities of the analytical peak in question to correct for peak shift and/or shape changes. This is usually the case for new compounds or other binary systems where prior measurements by Bastin are not found in the literature.

The first point is that whether you will utilize the Peak/Integrated intensity or the Integrated/Peak intensity depends on whether this wavescan is the "std" material or the "unk" material for the APF calculation. That is, use the P/I ratio from your standard material wavescan (the same as the primary quant standard) and use the I/P ratio from your unknown material wavescan (the same as your unknown sample).

By using the Model Background dialog one can utilize the Integrate button to perform most of these intensity calculations quickly and easily. An example for Boron Ka in Mg compounds is provided.

But first an important consideration: depending on whether you keep the peak position constant for the different standard and unknown materials (and thereby incorporate the peak shift effect in the APF calculation) or use a different peak position for each material (and minimize the peak shift and essentially only correct for peak shape changes, in addition to improving your counting statistics by using optimized peak positions), how you go about calculating the APF is slightly different for the two cases.

After loading a wavescan (on the standard or unknown material) to be used for the APF calculation, into the Graph dialog, click the Model Background button. If one now clicks the Integrate button the program calculates the Peak and Integrated intensities and also the P/I and I/P intensities based on the on-peak position of the wavescan sample. By default, the Probe for EPMA program uses the peak position recorded for each wavescan sample. Now if you are using the same peak position for all samples (and correcting for both peak shift and peak shape changes) that is fine. Multiply your P/I and I/P ratio from your two materials, edit your EMPAPF.DAT file and you are done.

Now in the case of sulfur the change between, say pyrite and anhydrite is almost all peak shift, so you probably wouldn't want to use the same spectrometer position for both materials though you could if you were consistently using the same peak position for both the wavescans and the standard and unknown samples. Also if the spectral change between the two materials is mostly peak shape them you probably would also use the same spectrometer positions and just correct for changes in peak shape.

But if you want to minimize the peak shift effect and use the optimum peak position for each material (and optimize your counting statistics), and only correct for peak shape changes, you should either make sure that each wavescan has the correct on-peak position specified before acquisition. Or you can click one of the peak fit options in the Model Background dialog (usually Maxima or Highest). Then click the Integrate button again and the program will recalculate the P/I and I/P values based on the newly fitted peak position.

Of course you must use the same optimized peak positions for your actual quantitative standard and unknown acquisitions if you use APFs based on these fitted peak positions.

### **Multi-Point Backgrounds**

Adjustment of multi-point backgrounds can also be performed graphically using the wavescan plot Model Background dialog. Select the High or Low side multi-point background and use the plus and minus buttons to graphically adjust the position on the wavescan. The number of background points on each side of the peak to acquire can also be specified here.

The number of points to iterate to and the background fit type (Linear or Polynomial) can be specified before or after the background intensity data has been acquired. Note that the sum of all multi-point intensities is automatically calculated and saved to the normal off-peak intensity arrays to so that normal off-peak background calculations can also be performed.

### **Area Peak Factor Calculations**

The Model Background dialog will also calculate the background corrected peak and integrated areas using a cubic spline fit to the intensity data. These are useful for Area Peak Factor (APF) calculations. The program will calculate both the P/I (Peak/Integrated, normally the “standard” or reference compound) and the I/P (Integrated/Peak, normally the “unknown” compound) values. The one that is ultimately utilized for an APF calculation by the user depends on whether the wavescan represents the primary standard or the secondary standard. See the section of APF calculations in the Analytical menu section for details on how these values are used for the calculation of APFs.

The APF calculations are in units of counts per second for the peak intensities and area counts per second divided by spectrometer range (between the low off-peak and high off-peak positions) for the integrated intensities (counts per second per spectrometer unit). Both the peak and integrated intensities are background corrected using the currently selected background fit type.

### *Centroid Calculations*

The Model Background dialog will also calculate the centroid of the displayed wavescan. The program will fit the wavescan intensities that are within the currently displayed x-axis zoom range (min to max) of the Graph dialog to a parabola (2<sup>nd</sup> order polynomial), maxima or Gaussian centroid. Therefore the user should zoom the wavescan to display only the top portion of the wavescan peak for best results. The parabolic peak centroid is calculated using the following equation:

$$P_C = \frac{2A}{-B}$$

Where:  $P_C$  is the centroid of the peak  
 $A$  is the first fit coefficient (intercept)  
 $B$  is the second fit coefficient (slope)

### **Close**

This button will close the Plot! Window, although since it is a modeless dialog it can be left open if enough screen space is available.

# Probe Menu Details

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## File

The File menu can be used to create new Probe for EPMA runs, open an existing run, save the current run to another name, close the current run, display the current probe database file information, print the current log window contents or log window selection or exit the program.

### New

The New menu opens a dialog to select a **new** Probe for EPMA database file. If the file already exists, the program will prompt the user whether to overwrite the existing file. After the new file has been created the program will display a file information dialog to allow the user the personalize the run title and description.

The program will automatically append a .MDB extension to the file name if one is not supplied by the user. The .MDB file name indicates Microsoft Database and is a valid Microsoft Access v. 1.1 database file.

### Open

The Open menu allows the user to select an **existing** Probe for EPMA database file to process previously acquired data or to append additional acquisition data. The program will check that the specified file is of the correct format and warn the user if not. Probe for EPMA generally uses a file extension of .MDB for its database file extension.

Probe for EPMA performs a general initialization of the ZAF correction factors when it opens a Probe for EPMA database. If a x-ray initialization error occurs during this procedure, the program will allow the user to continue to open the file anyway or not. If an error does occur, for example an x-ray overvoltage condition, the user should take whatever steps are necessary to correct the anomalous condition.

### Save As

The Save As menu allows the user to save the currently open Probe for EPMA database file to another file name.

This option allows the user considerable flexibility in how new runs are created and utilized. For example, one possibility is to create a number of generic Probe for EPMA runs that contains only a standardization for certain elements (FELDSPAR.MDB for example). Then, at a later time, the user could open that run and use the Save As option to save the run to another file name. Doing so, will create a new run with only the previously acquired standardization, to which unknown can then be acquired without altering the original run containing only the standardization.

Of course, whether this procedure is applicable or not depends greatly on both the reproducibility of the spectrometers and the accuracy required by the user.

## **Close**

The Close menu allows the user to close the currently open Probe for EPMA database file. This can be used to close the current file before opening a different Probe for EPMA database file without re-starting the program. The program will ask the user to confirm the close operation and all parameters are automatically saved.

## **Find File**

This menu allows the user to open an existing run by browsing the available runs on disk. This is useful when the filename itself is unknown to the user, but the run itself, might be recognized on another basis such as user name or title.

This dialog has a directory browser that can be used to load Probe run files from various sub-directories if desired. For example, each user could utilize a separate sub-directory or their own or the different types of analysis could be used to define a sub-directory structure to hold files.

## **File Information**

This menu displays and allows the user to change the run title and/or description fields for the currently open run. The title and user fields are limited to 64 characters and the description field is limited to 255 characters.

Normally, the user should completely fill out these fields since the text is saved not only to the user's run database, but also the user log database (see USERWIN.EXE). It is often helpful to use the description field to list the sample numbers or designations. Click the Enter <cr> button to make a new line in the description field.

## **Compact**

The Compact menu allows the user to compact the current Probe database file. This allows the database to be sorted for maximum efficiency and the indexes updated. If the database has become very fragmented this process can also save a significant amount of disk space for large database files.

## **Export Probe Configuration Files...**

Use this menu to export internally stored Probe for EPMA configuration files to the probe database file. This may be useful for running in "demo" mode off-line or simply to see the instrument configuration on another computer.

## **Import Probe Configuration Files...**

This menu is used to store a set of Probe for EPMA configuration files. Multiple copies of the instrument configuration files may be stored in each probe data file.

## **Print Log**

This menu allows the user to print the entire contents of the log window or selected portions of the log window. Be sure to click the Selection field if only a text selection is to be printed, otherwise the program will print the entire log window text.

## **Print Setup**

This menu allows the user to change the default printer or printer setup options for the currently selected printer.

## **User Wizard**

This menu pops up a guided pictorial introduction to starting and setting up a new Probe for EPMA database run for acquisition, processing and data output.

## **Probe for EPMA Quick Start Guide**

Opens the Probe for EPMA Quick Start Guide as a PDF file.

## **Probe for EPMA Frequently Asked Questions Guide**

Opens the Probe for EPMA Frequently Asked Questions PDF file. This document provides answers to commonly asked questions.

### **Exit**

This menu exits the program. The program will ask the user to confirm the exit procedure if an analytical run is open.

---

## **Edit**

### **Cut**

This menu cuts the selected text from the log window to the Windows Clipboard.

### **Copy**

This menu copies the selected text from the log window to the Windows Clipboard in plain text.

### **Copy Special**

The menu copies the selected text from the log window to the Windows Clipboard in “rich field text”. This method preserves font, style and color information.

### **Paste**

This menu pastes plain text from the Windows Clipboard to the log window.

### **Paste Special**

The menu pastes “rich field text” from the Windows Clipboard to the log window. This method preserves font, style and color information.

### **Select All**

This menu selects all text in the log window.

### **Clear All**

This menu clears all text from the log window.

---

## **Standard**

### **Standard Database**

This menu invokes a shell process to launch the Standard database application (STANDARD.EXE) as an asynchronous process. The standard database is used to maintain the database of standard compositions and also includes a number of features for exploring analytical conditions based on various standard compositions.

Since the shell process runs independently, it can be accessed during an automation or analysis procedure.

## Evaluate Standards

This menu runs the Evaluate program asynchronously (as a separate process) for independent evaluation of standard compositions for a selected probe database. The user selects an element, x-ray, crystal and spectrometer condition and the program will plot the relative (or absolute) compositional differences to other standards in that run containing the same element under the same analytical conditions. From this standard comparison one can evaluate the relative accuracy of a suite of standards.

The program also contains a method for plotting variation plots of unknown or standard samples. See the Evaluate application Window | Log Window menu for more details.

## Select Standard Database

This menu allows the user to load a non-standard database for various analytical situations. For example, if another microprobe laboratory sends you a probe database file that you would like to process but it is based on a different standard database than your default database, one can simply open the other laboratory's standard database by browsing to it using this menu which then temporarily assigns that database for the subsequent probe database operations.

Or perhaps you would like to process the same probe database with slightly different standard compositions to evaluate the effect of such differences on the same probe database intensities.

To return to using your default standard database select your default standard database using this menu or simply restart the program.

## Edit Standard Coating Parameters

This menu allows the user to specify the type and thickness of the conductive coating for each (or all) of the standards. This information can be used for documentation purposes or to correct for electron energy loss and/or x-ray absorption.

Note that the coating parameters for standards and unknowns are ignored in the quantitative calculations until the electron absorption and x-ray transmission coating correction flags are explicitly turned on from the Analytical | Analysis Options menu dialog.

## Add/Remove Standards To/From Run

This menu allows the user to specify additional standards to be used in the currently open Probe for EPMA run.

Note that if the run is closed before any data is acquired for these newly added standards, then they will have to be added again, if the run is re-opened later to acquire data for them unless they are saved as the probe database is closed by clicking Yes when asked whether to save them for the next session. This is because Probe for EPMA updates the list of standards in the run each time a database file is opened based on the standard samples that have been actually created.

All standards that need to be digitized, acquired or assigned, must be first added to the run using this window dialog.

Standards may also be removed from the standard list with some reservations. First, a standard cannot be removed from the standard list if it references a standard sample already created in the current probe database. Second, even if a standard does not reference a standard sample, it may still itself be referenced by a standard, interference standard or MAN (mean atomic number) background standard assignment, especially if a sample setup was loaded from another probe database using the Load File Setup option. It is strongly suggested that the user check that all assignments do not reference the standard number that is to be removed.

In the case of MAN standard assignments, it may be useful to use the Clear All MAN Assignments menu item under the Analytical menu to remove all standard references. These assignments can be easily reloaded by default the next time the MAN Fits menu is selected.

---

# X-Ray

## X-Ray Database

This menu allows the user to view specified ranges of the NIST x-ray wavelength database. This database covers the wavelength range from approximately 0.5 to 100.0 angstroms and includes higher order reflections as well. Higher order reflections are reduced in intensity by 25% for each subsequent order to simulate the effect of PHA analysis.

The x-ray list range (Start Angstroms and Stop Angstroms) and minimum search intensity (Minimum Intensity) of the x-ray list can be specified by the user. Note that since the display is limited to about 1000 items, the program will automatically increase the minimum intensity until the returned list of x-ray lines is less than 1000. To view x-ray lines of lesser intensity, simply reduce the x-ray range and click the Re-Load button.

Note that angstrom values in the x-ray database list with Bragg reflection orders higher than one (Roman Numeral "I") are NOT corrected for refraction index corrections. However, KLM markers displayed in the Graph Data wavescan plot dialog are corrected using the equation  $A' = A * (1 - (K - (K / N^2)))$  where A is the uncorrected angstrom position, K is the refractive index from the CRYSTALS.DAT file and N is the Bragg reflection order.

### **Description of the X-ray Database (adapted from NIST documentation by C. Fiori)**

The NIST x-ray database is based on 4985 (1st order) entries and includes all the measurable x-ray lines, satellites and absorption edges from under 100 eV to over 120 keV. Additionally, most of the x-ray lines and satellites are assigned a relative intensity (relative to the alpha-1 line in each family). The data base was assembled primarily from four sources:

- 1.) B.L. Doyle, W.F. Chambers, T.M. Christensen, J.M. Hall and G.H. Pepper "SINE THETA SETTINGS FOR X-RAY SPECTROMETERS", Atomic Data and Nuclear Data Tables Vol. 24, No 5, 1979.
- 2.) E.W. White, G.V. Gibbs, G.G. Johnson Jr. and G.R. Zechman "X-RAY WAVELENGTHS AND CRYSTAL INTERCHANGE SETTINGS FOR WAVELENGTH GEARED CURVED CRYSTAL SPECTROMETERS" Report of the Pennsylvania State Univ., 1964.
- 3.) J.A. Bearden "X-RAY WAVELENGTHS AND X-RAY ATOMIC ENERGY LEVELS" Rev. Mod. Phys., Vol. 39, No. 78, 1967.
- 4.) J.A. Bearden and A.F. Burr, "REEVALUATION OF X-RAY ATOMIC ENERGY LEVELS", Rev. Mod. Phys., Vol. 31, No. 1, 1967.

Each x-ray line or edge series as a function of atomic number was fit to a fourth degree polynomial. The fit was subtracted from the appropriate data and the residuals plotted and examined. In this way rogue entries could be identified and corrected. The resulting data base is considered to be sufficiently accurate for any application involving the Si (Li) x-ray detector and single crystal wavelength spectrometers.

Note that the last entry in the x-ray database window gives a code for the source of the entry. If the column is blank the source is reference 2. If the column contains the letter "C" the source is reference 1. If the letters "BB" appear, the source is reference 4. The letters "W,F" mean that reference 2 was used but the relative transition probability has been adjusted by Fiori. Reference 3 was used as a check since it is the source of many of the entries of reference 1.

In column 3 the notation KA1,2 means the entry is the weighted sum of the KA1 and KA2 in the ratio 2 to 1. For low atomic number the entries are not self consistent since the data is from different sources. If the column begins with the capital letter S then the entry is a satellite line due to doubly ionized atoms. The relative transition values for these entries are only valid for electron excited specimens, and are, at best, estimates.

The following are Siegbahn to shell-transition notation conversions:

$$\text{KA} = \text{KA1+KA2+KA3}$$

```

KA1,2 = (2*KA1+KA2)/3
KA1 = K-L3
KA2 = K-L2
KA3 = K-L1
KB = SUM(KBn)
KBX = Metal
KB1 = K-M3
KB1' = KB1+KB3+KB5
KB2 = (K-N3)+(K-N2)
KB2' = K-N3
KB2'' = K-N2
KB3 = K-M2
KB4 = (K-N4)+(K-N5)
KB5 = (K-M4)+(K-M5)
KB5' = K-M5
KB5'' = K-M4
Kd1 = K-O3
Kd2 = K-O2
LA = LA1+LA2
LA1 = L3-M5
LA2 = L3-M4
LB1 = L2-M4
LB10 = L1-M4
LB15 = L3-N4
LB17 = L2-M3
LB2 = L3-N5
LB3 = L1-M3
LB4 = L1-M2
LB5 = (L3-O4)+(L3-O5)
LB6 = L3-N1
LB7 = L3-O1
LB9 = L1-M5
LG1 = L2-N4
LG11 = L1-N5
LG2 = L1-N2
LG3 = L1-N3
LG4 = L1-O3
LG4' = L1-O2
LG6 = L2-O4
LG8 = L2-O1
L1 = L3-M1
Ln = L2-M1
Ls = L3-M3
Lt = L3-M2
Lu = (L3-N6)+(L3-N7)
Lv = L2-N6
MA1 = M5-N7
MA2 = M5-N6
MB = M4-N6
MG = M3-N5
MG2 = M3-N4
MZ1 = M5-N3
MZ2 = M4-N2
Md = M2-N4
Me = M3-O5

```

## **Spectrometer Table**

This menu item outputs a table of spectrometer positions for all elements based on the crystals configured for the current microprobe setup in the SCALERS.DAT file in the Probe for EPMA sub directory.

Both the alpha and beta series lines are listed. This table is especially useful when printed out and used as a reference when setting up an analytical setup to determine which spectrometers and crystal combinations can be used for the elements of interest.

## **Emission Table**

This menu lists a table of emission energies (or angstroms) for all elements and their major analytical x-ray lines. Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

## **Edge Table**

This menu lists a table of absorption edge energies (or angstroms) for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

## **Fluorescent Yield Table**

This menu lists a table of fluorescent yield fractions for all elements and their major analytical x-ray lines.

Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

## **MAC Table**

This menu lists a table of MACs (mass absorption coefficients) for the specified element and it's major analytical x-ray lines. Note that these values are stored in a binary data file. Although not recommended, it is possible to edit individual data entries use the CalcZAF program.

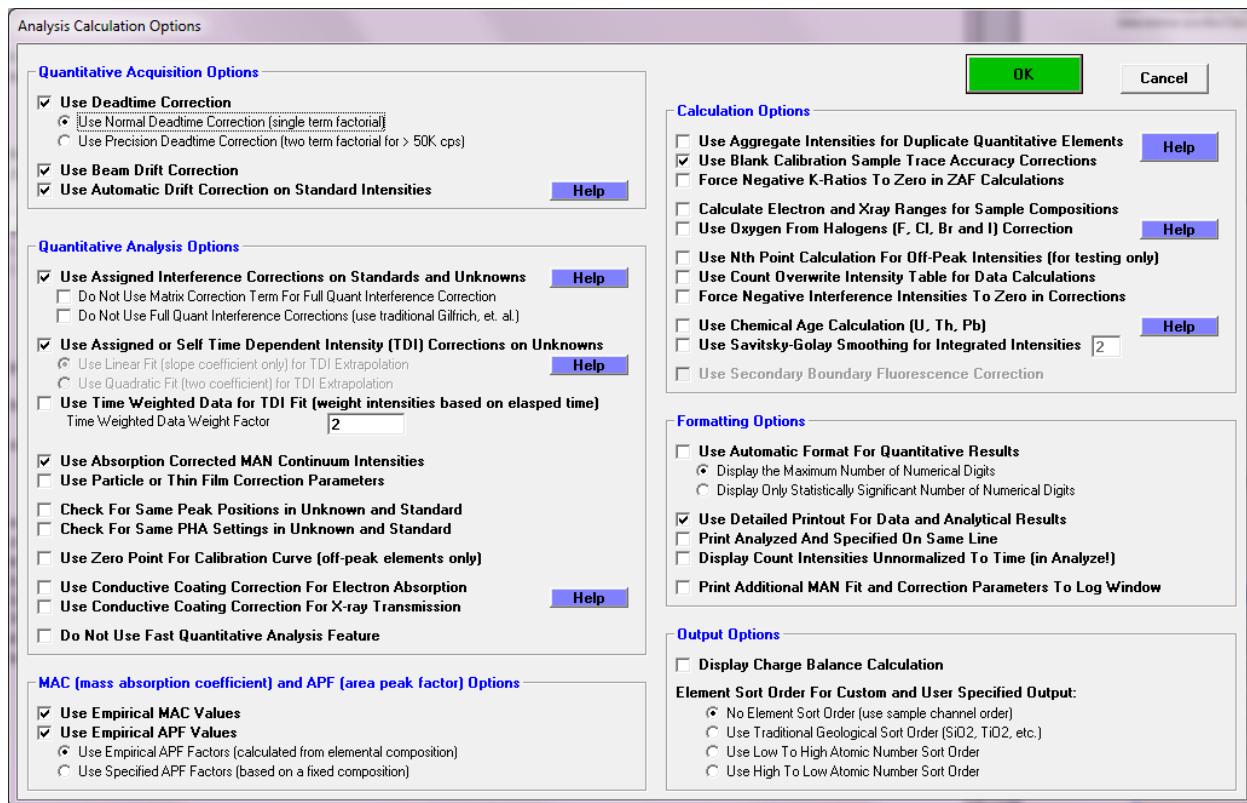
The program will automatically load the values based on the selected default MAC table. See the Analytical | ZAF Selection section for a description of the MAC table choices.

---

# **Analytical**

## **Analysis Options**

The Analysis Options dialog allows the user to turn on or off a number of analytical options. This allows for greater flexibility in the quantitative analysis of unusual samples or to pinpoint problems in the analysis.



## Use Deadtime Correction

The deadtime correction can be disabled for use in certain unusual analytical situations. The program will type a warning to the log window during a quantitative analysis if the deadtime correction is disabled. Two deadtime correction options are available, the normal expression shown here :

$$I = \frac{i}{1.0 - i\tau}$$

Expression # 1 :

Where :       $i$       is the raw measured counts in cps  
                    $\tau$       is the deadtime constant in seconds  
                    $I$       is the deadtime corrected counts in cps

and a high precision expression (Willis, 1993) for use with very high count rates (> 50K cps) shown here :

$$I = \frac{i}{1.0 - \left( i\tau + i^2 \frac{\tau^2}{2} \right)}$$

Expression #2 :

This high precision expression differs from the normal expression only at very high count rates. The following table shows the difference between the two expressions at various count rates :

| CPS | #1 @ 1 us | #2 @ 1 us | #1 @ 2 us | #2 @ 2 us | #1 @ 4 us | #2 @ 4 us |
|-----|-----------|-----------|-----------|-----------|-----------|-----------|
| 5K  | .995      | .9950125  | .99       | .99005    | .98       | .9802     |
| 10K | .99       | .99005    | .98       | .9802     | .96       | .9608     |

|      |     |        |    |      |    |     |
|------|-----|--------|----|------|----|-----|
| 50K  | .95 | .95125 | .9 | .905 | .8 | .82 |
| 100K | .9  | .905   | .8 | .82  | .6 | .68 |
| 200K | .8  | .82    | .6 | .68  | .2 | .52 |
| 400K | .6  | .68    | .2 | .52  | -- | --  |

### **Use Beam Drift Correction**

The beam drift correction can be disabled for use in certain unusual analytical situations. The program will type a warning to the log window if the beam drift correction is disabled.

The beam drift correction calculation is shown here (where beam integration time is taken into account) :

$$I_C = I_U \frac{B_N \frac{T_U}{T_N}}{B_U}$$

Where :  $I_C$  is the beam drift corrected unknown intensity

$I_U$  is the uncorrected unknown intensity

$B_N$  is the nominal beam current

$T_U$  is the count time for the unknown intensity

$T_N$  is the count time for the nominal beam current measurement

$B_U$  is the beam current for the unknown intensity

However, since Probe for EPMA stores and utilizes x-ray intensities in counts per second (already normalized to time), the actual beam drift correction, for direct reading faraday cup micropipes, is quite a bit more simple than the rigorous example above, and is instead shown here:

$$I_C = I_U \frac{B_N}{B_U}$$

Where :  $I_C$  is the beam drift corrected unknown intensity

$I_U$  is the uncorrected unknown intensity

$B_N$  is the nominal beam current

$B_U$  is the beam current for the unknown intensity

This means that if the actual measured beam current for your unknown decreases over time, the value of the fraction

$\frac{B_N}{B_U}$ , increases and therefore the corrected x-ray intensity is adjusted upwards. If on the other hand, the actual measured beam current for your unknown increases over time, then the value of the fraction  $\frac{B_N}{B_U}$ , decreases and therefore the

corrected x-ray intensity is adjusted downwards.

The beam drift correction essentially normalizes the x-ray intensities to the x-ray intensity that would be expected at the nominal beam current. For example, if most of the x-ray measurements are made at a beam current of 20 nA, then setting the nominal beam current to 20 nA will ensure that the x-ray intensities will appear scaled to readings taken at 20 nA (even if the measurement is taken at a different beam current). In an alternative usage, the x-ray intensities may be expressed in counts per second per nA by setting the nominal beam current to 1.0. See the Count Times dialog in the Acquire! Window to manually change the nominal beam current.

## **Use Automatic Drift Correction on Standard Intensities**

Probe for EPMA uses a sophisticated automatic drift correction to correct the counts on the standard, interference and MAN standard intensities. This standard drift correction should not be confused with the beam drift correction described previously. With the automatic standard drift correction, when Probe for EPMA loads the unknown count data to analyze a sample data point, the program will automatically check each element to see if the standard for that channel was measured both before and after the data on the unknown. If there exist sufficient standard sets, the program will automatically compute a linear interpolated drift corrected analysis.

If there are only a single standard data set, the program will simply use the set of counts on that standard which occurred closest in time prior to the unknown. Thus it is possible to perform analyses of unknowns as they are acquired, although they will not be corrected for drift. Later, when the standards have been run again, the program can recalculate all the analyses, and they will be corrected for standard count drift in real time.

The user may go back and acquire each standard up to 20 times during a run (20 "sets" of counts on the standard) and the program will determine which two sets, if any, were made closest, before and after, the counts on the unknown. The form of the standard drift correction is shown below :

$$I_s = I'_s + (I''_s - I'_s) \frac{(T_u - T'_s)}{(T''_s - T'_s)}$$

Where :  $I_s$  is the drift corrected standard intensity

$I'_s$  is the standard intensity from the preceding standardization

$I''_s$  is the standard intensity from the following standardization

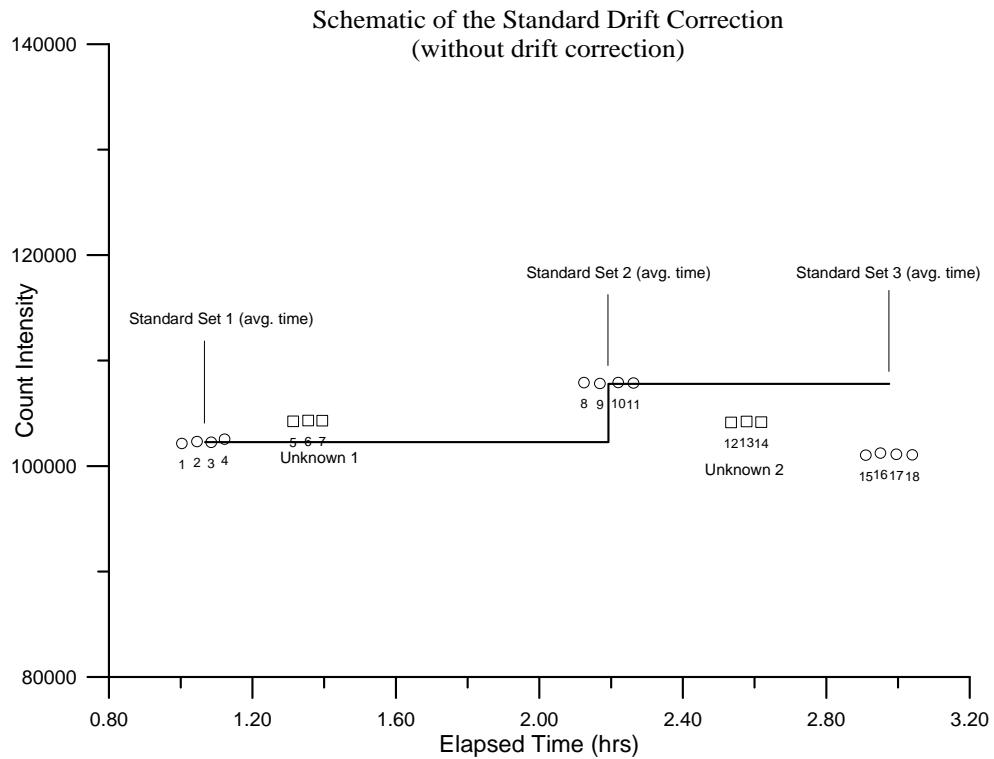
$T_u$  is the real time of the unknown analysis

$T'_s$  is the real time of the preceding standardization

$T''_s$  is the real time of the following standardization

The following explanation will demonstrate how the standard drift correction is actually implemented in Probe for EPMA. The example data shown here consists of four standard data points (set 1), followed by three unknown data points (points 5, 6 and 7), followed by four more standard data points (set 2), followed by three more unknown data points (points 12, 13, and 14), followed by a final four standard data points (set 3).

The first graph shows the standard intensities and how they are applied to unknown data **without** the use of a standard drift correction. The stepped horizontal lines drawn between the standard sets represents the standardization intensities that the program would calculate in the absence of a drift correction. In other words, the standard intensity is always based on the last standardization and remains constant until the next standardization.



Note that each unknown data point actually uses the average intensity of the previous standardization. In a situation where drift has occurred, significant errors will result for the unknown analyses without a drift correction for the standard intensity.

The second graph shows the standard intensities and how they are applied to unknown data **with** the use of a standard drift correction. The sloped horizontal lines drawn between the standard sets represents the standardization intensities that the program will calculate when a standard drift correction is used. Note that each unknown data point uses the **interpolated** average intensity of the previous **and** the subsequent standardizations. In a situation where linear drift has occurred, the standard drift correction allows for the adjustment of standard intensities for intervening unknown analyses.



Note that because all quantitative samples (standard and unknowns) are treated as unknowns for the purposes of quantitative analysis, the standard drift correction is also applied to the analysis of standard samples. However, it is useful to note that for a given primary (assigned) standard sample, standard data points acquired *just before* the average time of acquisition of that standard are interpolated between the previous standard and the standard itself, while standard data points acquired *just after* the average time of acquisition of that standard are interpolated between the standard itself and the subsequent standard. Therefore, for primary standards, the data points within that standard are normally analyzed using two different standard intensity data sets. Furthermore, even in the case of no standard drift correction, the program will still utilize two different standard intensities when analyzing those data points just before or just after the average time of acquisition of the standard.

### **Use Assigned Interference Corrections on Standards and Unknowns**

This option will either enable or disable the interference corrections for subsequent quantitative analysis. This option is automatically loaded if interference corrections are specified by the user. Use this option to easily determine the total effect of all of the interference corrections specified, on a quantitative analysis.

The “Do Not Use Full Quant On Interference Correction On Unknowns” option is for testing purposes only. It is should always be unchecked so that your interference corrections utilize the full matrix correction between the interference standard and the unknown sample.

The “Do Not Use Full Quant Interference Correction (use Gilfrich, et. al.)” is also for testing purposes to show what the interference correction would be using the traditional intensity ratio method.

### **Use Assigned or Self Time Dependent Intensity (TDI) Corrections on Unknowns**

This option will either enable or disable the Time Dependent Intensity (TDI) element corrections for subsequent quantitative analysis. This option is automatically loaded if Time Dependent Intensity (TDI) element corrections are specified by the user. Use this option to easily toggle the Time Dependent Intensity (TDI) element corrections on or off, for all quantitative analyses. Note the VOL%: line in the log window also shows the relative percent change in the intensity due to the Time Dependent Intensity (TDI) correction.

Both the assigned Time Dependent Intensity (TDI) and self Time Dependent Intensity (TDI) corrections are calculated based on the actual elapsed time of the calibration and the unknown intensity can be corrected for two different fit models:

- Use Linear Fit (slope coefficient only) for Time Dependent Intensity (TDI) Extrapolation
- Use Quadratic Fit (two coefficient) for Time Dependent Intensity (TDI) Extrapolation

The linear fit is a simple correction based on a straight line fit of the log intensities as a function of time. In other words the x data is elapsed time, the y data is the natural log of the count rate and the fit is a straight line fit in log space. The fit returned is of the  $y = a + bx$  form where  $b$  is the slope and  $a$  is the intercept. In the case of the linear fit, only the slope of the fit is applied to the unknown intensities as shown here. This is the normal correction and works well for situations where the intensity change is linearly exponential.

$$I_{corr} = e^{Ln(I_{raw}) - bx}$$

where:  $I_{raw}$  is the uncorrected intensity (cps)  
 $b$  is the slope of the fit to the natural log intensity versus time  
 $x$  is one half the total elapsed time of the measurement in seconds

The quadratic fit uses both the slope and curvature coefficients to the log intensity 2<sup>nd</sup> order polynomial fit to handle situations where the intensity change is non-linear. This non-linear intensity change can occur when the beam current is too high or the beam is too focused. In these cases where the relative percent change in intensity can exceed 100%, it is sometimes helpful to apply the quadratic fit.

$$I_{corr} = e^{Ln(I_{raw}) - bx - cx^2}$$

where:  $I_{raw}$  is the uncorrected intensity (cps)  
 $b$  is the slope of the fit to the natural log intensity versus time  
 $c$  is the curvature of the fit to the natural log intensity versus time  
 $x$  is one half the total elapsed time of the measurement in seconds

It is surmised that this quadratic non-linear or hyper exponential fit is the result of two separate exponential intensity change mechanisms with different time constants that are overlapping in time.

*Note that these options are disabled for current versions of the software since these options are now specified on an element by element basis in the Standard Assignments dialog from the Analyze! window.*

### **Use Time Weighted Data For TDI Fit**

This option will apply a weighting factor to the TDI intensity data for the TDI fit based on the inverse square of the weighting factor starting with the shortest elapsed time intensity. For example, if the weighting factor is 2, then the program will load the first TDI intensity for the regression fit two times, the second TDI intensity will be loaded as usual once and so on. If the weighting factor is 4 then the first TDI intensity will be loaded four times, the second TDI intensity two times and the third TDI intensity once as usual.

The purpose of this weighting is to give more emphasis to the first few TDI intensities which should be more representative of the zero time intensity.

### **Use Absorption Corrected MAN Continuum Intensities**

This option will either enable or disable the use of absorption corrected continuum intensities for calculation of the MAN background correction in subsequent quantitative analysis. For details on this procedure see Donovan and Tingle, 1996.

### **Use Particle and Thin Film Correction Parameters**

This is a global option to turn on or off the particle and thin film parameters specified on a sample by sample basis in the Calculation Options dialog.

### **Check For Same Peak Positions in Unknown and Standard**

Check this option to only use a standard intensity if the unknown and standard peak positions are within 0.005% of each other. For the JEOL this means the peak positions must be within 0.003 mm of each other at low Bragg angles and within 0.013 mm of each other at high Bragg angles. For Cameca instruments, the peak positions must be within 1 point at low Bragg angles and within 4 points at high Bragg angles.

This option is mainly used when multiple setups are being utilized in a single run and the setups have elements in common with the same keV, spectrometer, x-ray line and crystal, but not necessarily the same peak positions.

### **Check For Same PHA Settings in Unknown and Standard**

Check this option to only use a standard intensity if the unknown and standard PHA settings are within 0.5% of each other. This option checks the baselines, windows, gains, biases and Integral/Differential modes.

This option is mainly used when multiple setups are being utilized in a single run and the setups have elements in common with the same keV, spectrometer, x-ray line and crystal, but not necessarily the same PHA settings.

### **Use Zero Point For Calibration Curve (Off-Peak Elements Only)**

This analysis option is only applicable when using the Calibration Curve (multi-standard) option in the ZAF, Phi-Rho-Z, Alpha Factor and Calibration Curve Selections dialog. This option forces the program to utilize a synthetic zero intensity zero concentration data point for off-peak intensity data calibration curve calculations. It allows the use of a single standard for calibration curve calculations.

If this option is checked then the program will add an additional data point to the calibration curve fit of value 0,0. That is, zero counts and zero concentration. This option only applies to elements that have been acquired using off-peak backgrounds.

This option may be useful if some of the analyzed elements in the run have not been entered in the standard database with a concentration of zero and the user wishes to have the zero concentration data point included in the calibration curve fit.

### **Use Conductive Coating Correction for Beam Energy Loss**

Calculates the incident beam energy loss due to the conductive coating. Only affects low over-voltage x-rays. The parameters may be set independently for the standards (Standard menu) and the unknowns (Elements/Cations dialog). Only necessary if the standards and unknowns have different thickness or different elemental coatings.

Note that the coating parameters for standards and unknowns are ignored in the quantitative calculations until the electron absorption and x-ray transmission coating correction flags are explicitly turned on from the Analytical | Analysis Options menu dialog.

### **Use Conductive Coating Correction for X-ray Absorption**

Calculates the x-ray absorption effect due to the conductive coating. Only affects low energy x-rays. The parameters may be set independently for the standards (Standard menu) and the unknowns (Elements/Cations dialog). Only necessary if the standards and unknowns have different thickness or different elemental coatings.

Note that the coating parameters for standards and unknowns are ignored in the quantitative calculations until the electron absorption and x-ray transmission coating correction flags are explicitly turned on from the Analytical | Analysis Options menu dialog.

### **Do Not Use Fast Quantitative Analysis**

This checkbox turns off the “fast” quantitative analysis feature. When this feature is enabled the program will only re-load the standard drift arrays and re-calculate the standard k-factors if the same has changed its configuration to require a re-load and re-calculate (or if a standard data point has been added or disabled within the run).

### **Use Empirical MAC Values**

This option will either enable or disable the use of empirical MACs (mass absorption coefficients) for subsequent quantitative analysis. This option is automatically loaded if empirical MACs are specified by the user. Use this option to easily determine the total effect of all of the empirical MACs specified by the user, on a quantitative analysis.

### **Use Empirical APF Values**

This option will either enable or disable the use of empirical APFs (Area Peak Factors) for subsequent quantitative analysis to correct for spectral peak shape and shift. This option is automatically loaded if empirical APFs are specified by the user. Use this option to easily determine the total effect of all of the empirical APFs specified by the user, on a quantitative analysis. See the Analytical | Empirical APFs menu to select empirical APF factors from the default EMPAPF.DAT database (a user editable text file).

There are two options for Empirical APF factor:

- Use Empirical APF Factors (calculated from elemental composition)
- Use Specified APF Factors (based on a fixed composition)

### **Compound APFs**

The first option calculates a compound APF correction based on binary APF factors for an element in a binary standard (for example O ka in Cr<sub>2</sub>O<sub>3</sub>) and sums all binary APF factors based on the weight fraction of the emitting element to create a “compound” APF that applies to the specific analyzed composition. This is ideal in cases where all the elements affecting the emitting element have measured binary (end-member) APF factors. The calculation for compound APFs is shown here:

$$APF_{Comp} = \sum APF_j * \frac{W_j}{Sum_{Partial}}$$

Where:  $APF_j$  is the binary (end-member) APF for elements other than the affected element  
 $W_j$  is the weight fraction for elements other than the affected element

$Sum_{Partial}$  is the sum of all elements other than the affected element

A further normalization is performed on the compound APF to ensure that in situations where the partial sum is very low, the compound APF is not over applied to the affected (pure) element when only the trace elements are varying in precision. This calculation is show here:

$$APF_{Norm} = 1 + \frac{(APF_{Comp} - 1)}{\left( \frac{Sum_{Partial}}{W_{Affected}} \right)}$$

Note that this APF normalization correction is only applied when the absolute value of the scaling factor :

$$\frac{1}{\left( \frac{\text{Sum}_{\text{Partial}}}{W_{\text{Affected}}} \right)}$$

is greater than or equal to 1.0 to avoid problems with over applying the correction on major elements.

## Specified APFs

The second option is for more exotic absorbing systems when some elements have not been characterized for the binary APF (e.g., O ka in Na<sub>2</sub>O) and therefore it may be better to use a specified APF based on a measurement of the actual composition relative to the primary standard for the emitting element.

### Use Aggregated Intensities for Duplicate Quantitative Elements

If this option is checked and the samples contain duplicate elements (same element and x-ray line) for standards or unknown quantitative samples, then the program will sum the on and off peak intensities for the duplicated elements to the first instance of the duplicated element and set the duplicated element intensities to zero.

Normally this is utilized when extreme trace element analyses are required for very low concentration elements and there is a need for improving the statistics by aggregating the intensities from several spectrometers acquiring at the same time.

**CAUTION: It is important realize that measuring the same element more than once is a non-physical situation so far as regards the matrix correction. Obviously if one measures a major element on multiple spectrometers the resulting excessively high totals will make it clear that this analytical situation cannot be handled properly in the matrix correction.**

**In the case where a major or even minor element is measured as a duplicate element the onus is on the user to ensure that the matrix correction is properly calculated either by using the Aggregate Intensity option or by using the Disable Quant feature in the Elements/Cations dialog to have the element appear only once in the matrix correction.**

However, even in the case for duplicate trace elements, one must consider this effect also. Note for the unknown sample matrix, even though the change in the sample matrix correction is small, for the standard k-factor calculation a significant error can be introduced. Consider the following examples of measuring Ti on two spectrometers in a blank Quartz standard, where the Ti concentration is essentially zero.

In this calculation the program calculates the matrix correction for the quartz standard run as an unknown and the matrix correction of the sample is properly calculated as SiO<sub>2</sub>. However, the standard k-factor for Ti in TiO<sub>2</sub> should be 0.5547 at 15 keV, but is calculated as 0.5707 because the matrix is assumed to be Ti<sub>2</sub>O<sub>2</sub>.

Note that the program warns the user of the accuracy problem for duplicate elements in this first example of Ti as a duplicate element.

```
Un      2 std 14 SiO2
TakeOff = 40.0  KiloVolt = 15.0  Beam Current = 200.  Beam Size =    20
(Magnification (analytical) =     8000),           Beam Mode = Analog  Spot
(Magnification (default) =       600, Magnification (imaging) =    100)
Number of Data Lines:   5                 Number of 'Good' Data Lines:   5
First/Last Date-Time: 02/26/2007 01:35:59 PM to 02/26/2007 03:03:33 PM
WARNING- Using Exponential Off-Peak correction for ti ka
WARNING- Using Exponential Off-Peak correction for al ka
WARNING- Using Exponential Off-Peak correction for ti ka
```

WARNING- Using Alternating On and Off Peak Acquisition

|                            |        |                        |         |
|----------------------------|--------|------------------------|---------|
| Average Total Oxygen:      | 53.256 | Average Total Weight%: | 100.000 |
| Average Calculated Oxygen: | 53.256 | Average Atomic Number: | 10.805  |
| Average Excess Oxygen:     | .000   | Average Atomic Weight: | 20.029  |
| Average ZAF Iteration:     | 1.00   | Average Quant Iterate: | 2.00    |

Oxygen Calculated by Cation Stoichiometry and Included in the Matrix Correction

Element Si is Calculated by Difference from 100%

**WARNING- Duplicate analyzed elements are present in the sample matrix!!  
Use Aggregate Intensity option or Disable Quant feature for accurate matrix correction.**

#### Results in Elemental Weight Percents

| SPEC: | Si      | O       |        |         |        |                                |
|-------|---------|---------|--------|---------|--------|--------------------------------|
| TYPE: | DIFF    | CALC    |        |         |        |                                |
| AVER: | 46.740  | 53.256  |        |         |        |                                |
| SDEV: | .004    | .002    |        |         |        |                                |
| ELEM: | Ti      | Fe      | Al     | Ti      | K      |                                |
| BGDS: | EXP     | LIN     | EXP    | EXP     | LIN    |                                |
| TIME: | 600.00  | 600.00  | 600.00 | 600.00  | 599.92 |                                |
| ELEM: | Ti      | Fe      | Al     | Ti      | K      | SUM                            |
| XRAY: | (ka)    | (ka)    | (ka)   | (ka)    | (ka)   |                                |
| 110   | .000    | .002    | .000   | .000    | .000   | 100.000                        |
| 111   | .000    | .001    | .000   | .001    | .000   | 100.000                        |
| 112   | .000    | .002    | .005   | .000    | .006   | 100.000                        |
| 113   | .000    | .001    | .000   | .000    | .000   | 100.000                        |
| 114   | .000    | .001    | .000   | .001    | .001   | 100.000                        |
| AVER: | .000    | .001    | .001   | .000    | .002   | 100.000                        |
| SDEV: | .000    | .000    | .002   | .000    | .003   |                                |
| SERR: | .000    | .000    | .001   | .000    | .001   |                                |
| %RSD: | 223.4   | 34.3    | 223.6  | 82.6    | 163.1  |                                |
| STDS: | 22      | 395     | 374    | 22      | 374    |                                |
| STKF: | .5707   | .6779   | .0701  | .5707   | .1132  | < k-factor for TiO2 is wrong   |
| STCT: | 28957.5 | 14598.6 | 3841.1 | 10912.1 | 2424.3 |                                |
| UNKF: | .0000   | .0000   | .0000  | .0000   | .0000  |                                |
| UNCT: | -.2     | .2      | .3     | .1      | .3     |                                |
| UNBG: | 56.2    | 20.6    | 32.9   | 19.7    | 11.5   |                                |
| ZCOR: | 1.2024  | 1.2061  | 1.2286 | 1.2024  | 1.1696 | < matrix correction is correct |
| KRAW: | .0000   | .0000   | .0001  | .0000   | .0001  |                                |
| PKBG: | 1.00    | 1.01    | 1.01   | 1.00    | 1.03   |                                |

In the next example the aggregate intensity option (Analytical | Analysis Options menu) is turned on and the standard k-factor for the TiO2 standard is properly calculated because the x-ray counts from the two Ti channels are combined into a single channel.

Note that beam current and count time are **averaged** for the duplicate elements, since the calculation assumes that the aggregate intensity is simply increasing the geometric efficiency of the instrument. So two channels of Ti integrated at

600 seconds each is still displayed as at 600 seconds, but the total peak and off-peak counts are aggregated together to reflect the increased geometric efficiency.

```
Un      2 std 14 SiO2
TakeOff = 40.0 KiloVolt = 15.0 Beam Current = 200. Beam Size = 20
(Magnification (analytical) = 8000), Beam Mode = Analog Spot
(Magnification (default) = 600, Magnification (imaging) = 100)
Number of Data Lines: 5 Number of 'Good' Data Lines: 5
First/Last Date-Time: 02/26/2007 01:35:59 PM to 02/26/2007 03:03:33 PM
WARNING- Using Exponential Off-Peak correction for ti ka
WARNING- Using Exponential Off-Peak correction for al ka
WARNING- Using Exponential Off-Peak correction for ti ka
WARNING- Using Alternating On and Off Peak Acquisition
WARNING- Using Aggregate Intensities for Duplicate Elements
```

|                            |        |                        |         |
|----------------------------|--------|------------------------|---------|
| Average Total Oxygen:      | 53.256 | Average Total Weight%: | 100.000 |
| Average Calculated Oxygen: | 53.256 | Average Atomic Number: | 10.805  |
| Average Excess Oxygen:     | .000   | Average Atomic Weight: | 20.029  |
| Average ZAF Iteration:     | 1.00   | Average Quant Iterate: | 2.00    |

Oxygen Calculated by Cation Stoichiometry and Included in the Matrix Correction  
Element Si is Calculated by Difference from 100%

#### Results in Elemental Weight Percents

|       |      |      |
|-------|------|------|
| SPEC: | Si   | O    |
| TYPE: | DIFF | CALC |

|       |        |        |
|-------|--------|--------|
| AVER: | 46.740 | 53.256 |
| SDEV: | .004   | .002   |

|       |        |        |        |     |        |
|-------|--------|--------|--------|-----|--------|
| ELEM: | Ti     | Fe     | Al     | Ti  | K      |
| BGDS: | EXP    | LIN    | EXP    | EXP | LIN    |
| TIME: | 600.00 | 600.00 | 600.00 | .00 | 599.92 |

|       |      |      |      |      |      |         |
|-------|------|------|------|------|------|---------|
| ELEM: | Ti   | Fe   | Al   | Ti   | K    | SUM     |
| XRAY: | (ka) | (ka) | (ka) | (ka) | (ka) |         |
| 110   | .000 | .002 | .000 | .000 | .000 | 100.000 |
| 111   | .000 | .001 | .000 | .000 | .000 | 100.000 |
| 112   | .000 | .002 | .005 | .000 | .006 | 100.000 |
| 113   | .000 | .001 | .000 | .000 | .000 | 100.000 |
| 114   | .000 | .001 | .000 | .000 | .001 | 100.000 |

|       |       |      |       |      |       |         |
|-------|-------|------|-------|------|-------|---------|
| AVER: | .000  | .001 | .001  | .000 | .002  | 100.000 |
| SDEV: | .000  | .000 | .002  | .000 | .003  |         |
| SERR: | .000  | .000 | .001  | .000 | .001  |         |
| %RSD: | 213.9 | 34.3 | 223.6 | .0   | 163.1 |         |
| STDS: | 22    | 395  | 374   | 22   | 374   |         |

|       |         |         |        |       |        |                                |
|-------|---------|---------|--------|-------|--------|--------------------------------|
| STKF: | .5547   | .6779   | .0701  | .0000 | .1132  | < k-factor for TiO2 is correct |
| STCT: | 39869.6 | 14598.6 | 3841.1 | .0    | 2424.3 |                                |

|       |       |       |       |       |       |
|-------|-------|-------|-------|-------|-------|
| UNKF: | .0000 | .0000 | .0000 | .0000 | .0000 |
| UNCT: | -.2   | .2    | .3    | .0    | .3    |
| UNBG: | 75.9  | 20.6  | 32.9  | .0    | 11.5  |

|       |        |        |        |        |        |                                |
|-------|--------|--------|--------|--------|--------|--------------------------------|
| ZCOR: | 1.2024 | 1.2061 | 1.2286 | 1.0000 | 1.1696 | < matrix correction is correct |
|-------|--------|--------|--------|--------|--------|--------------------------------|

```

KRAW:    .0000   .0000   .0001   .0000   .0001
PKBG:    1.00     1.01     1.01     .00     1.03

```

In this last example the standard k-factor is properly calculated because the second Ti channel is disabled for quantification using the Disable Quant feature in the Elements/Cations dialog. Note that the first Ti channel could be disabled and the second Ti channel enabled to compare the two spectrometers. The duplicate element warning can be ignored in these cases.

```

Un      2 std 14 SiO2
TakeOff = 40.0 KiloVolt = 15.0 Beam Current = 200. Beam Size = 20
(Magnification (analytical) = 8000), Beam Mode = Analog Spot
(Magnification (default) = 600, Magnification (imaging) = 100)
Number of Data Lines: 5 Number of 'Good' Data Lines: 5
First/Last Date-Time: 02/26/2007 01:35:59 PM to 02/26/2007 03:03:33 PM
WARNING- Using Exponential Off-Peak correction for ti ka
WARNING- Using Exponential Off-Peak correction for al ka
WARNING- Using Exponential Off-Peak correction for ti ka
WARNING- Using Alternating On and Off Peak Acquisition
WARNING- Quantitation is Disabled For ti ka

```

```

Average Total Oxygen:      53.256      Average Total Weight%: 100.000
Average Calculated Oxygen: 53.256      Average Atomic Number: 10.805
Average Excess Oxygen:     .000        Average Atomic Weight: 20.029
Average ZAF Iteration:     1.00       Average Quant Iterate: 2.00

```

Oxygen Calculated by Cation Stoichiometry and Included in the Matrix Correction  
Element Si is Calculated by Difference from 100%  
WARNING- Duplicate analyzed elements are present in the sample matrix!!  
Use Aggregate Intensity option or Disable Quant feature for accurate matrix  
correction.

#### Results in Elemental Weight Percents

```

SPEC:      Si      O
TYPE:      DIFF    CALC

AVER:     46.740  53.256
SDEV:     .004    .002

ELEM:      Ti      Fe      Al      Ti      K
BGDS:      EXP     LIN     EXP     EXP     LIN
TIME:     600.00  600.00  600.00  600.00  599.92

ELEM:      Ti-D    Fe      Al      Ti      K    SUM
XRAY:      (ka)   (ka)   (ka)   (ka)   (ka)
  110     .000    .002    .000    .000    .000 100.000
  111     .000    .001    .000    .001    .000 100.000
  112     .000    .002    .005    .000    .006 100.000
  113     .000    .001    .000    .000    .000 100.000
  114     .000    .001    .000    .001    .001 100.000

AVER:     .000    .001    .001    .000    .002 100.000
SDEV:     .000    .000    .002    .000    .003
SERR:     .000    .000    .001    .000    .001
%RSD:     .0     34.3   223.6   82.6   163.1
STDS:     22      395    374     22     374

```

```

STKF:   .0000   .6779   .0701   .5547   .1132 < k-factor for TiO2 is correct
STCT: 28957.5 14598.6 3841.1 10912.1 2424.3

UNKF:   .0000   .0000   .0000   .0000   .0000
UNCT:   .0       .2       .3       .1       .3
UNBG:   .0       20.6    32.9    19.7    11.5

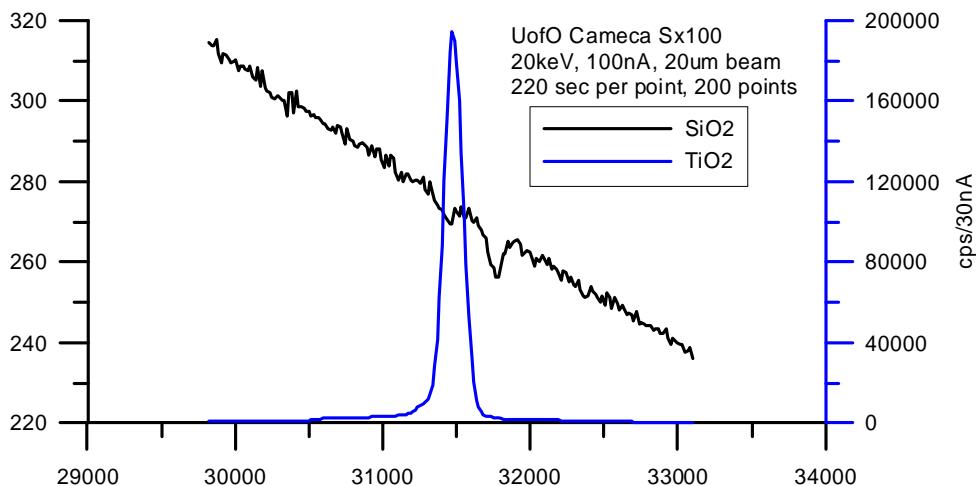
ZCOR:   1.0000  1.2061  1.2286  1.2024  1.1696 < matrix correction is correct
KRAW:   .0000   .0000   .0001   .0000   .0001
PKBG:   .00     1.01    1.01    1.00    1.03

```

### **Use Blank Calibration Sample Trace Element Accuracy Corrections**

This option will globally turn on or off the assigned blank correction parameters (see Standard Assignments dialog under the Analyze! window) to quickly evaluate the effect of the quantitative iterated blank correction.

The blank correction is a powerful method to improve the accuracy of trace elements (and because it is fully matrix corrected can even be applied in the case of oxygen analyses for water by excess oxygen). The following example demonstrates the application of the blank correction to a Ti in quartz analysis.



For example, spectrometer analyzing crystals have secondary lattice reflection planes which will produce small artifacts (“holes”) in the spectrum, some directly underneath the analytical peak (depending on the mounting orientation). This produces a systematic accuracy error in trace element analyses that can easily exceed the precision of the measurement even with moderate beam current and count times.

| ELEM:  | Ti      | Ti      | Ti      | Ti      | Ti      | Si      | O       | SUM     |
|--------|---------|---------|---------|---------|---------|---------|---------|---------|
| XRAY:  | (ka)    | (ka)    | (ka)    | (ka)    | (ka)    | ()      | ()      |         |
| 428    | -.00023 | .00029  | .00005  | .00076  | -.00061 | 46.7430 | 53.2572 | 100.000 |
| 429    | -.00023 | -.00003 | -.00001 | -.00009 | .00022  | 46.7430 | 53.2569 | 99.9998 |
| 430    | -.00010 | .00002  | -.00011 | -.00003 | .00045  | 46.7430 | 53.2572 | 100.000 |
| 431    | .00016  | -.00007 | -.00009 | -.00035 | .00037  | 46.7430 | 53.2570 | 100.000 |
| 432    | .00019  | -.00029 | .00002  | -.00016 | -.00037 | 46.7430 | 53.2566 | 99.9990 |
| AVER:  | -.00004 | -.00002 | -.00003 | .00003  | .00001  | 46.7430 | 53.2570 | 99.9999 |
| SDEV:  | .00020  | .00021  | .00007  | .00043  | .00047  | .00000  | .00024  |         |
| UNCT:  | -.1     | .0      | -.1     | .0      | .0      | .0      | .0      |         |
| KRAW:  | .00000  | .00000  | .00000  | .00000  | .00000  | .00000  | .00000  |         |
| PKBG:  | .99857  | .99949  | .99907  | 1.00219 | 1.00050 | .00000  | .00000  |         |
| BLNKL: | .000000 | .000000 | .000000 | .000000 | .000000 | ----    | ----    |         |
| BLNKV: | -.00043 | -.00009 | -.00053 | .000039 | .000704 | ----    | ----    |         |

By measuring the systematic offset (blank value line “BLNKV”) on a “blank” standard which contains a zero, or known non-zero level (“BLNKL”) for the element of interest in a similar matrix one can subtract the corresponding intensity from the sample during the matrix iteration for a rigorous treatment of all data including the k-ratio and P/B calculations.

### **Force Negative K-ratios to Zero In ZAF Calculations**

This option will force any intensity k-ratios to zero during the matrix iteration since a negative intensity is not normally a physically real situation. However, in the case of trace element analysis near zero concentrations one should generally calculate the slightly negative intensities around zero so that the average intensity or compositional value is correctly calculated.

### **Calculate Electron and X-ray Ranges For Sample Compositions**

This option will cause the program to calculate both the electron range Kanaya-Okayama incident electron scattering distance radii and effective x-ray transmission for a range of densities and thicknesses based on the actual sample composition at the current sample conditions (keV).

The expression:

$$\text{electron radius} = \frac{0.0276 \cdot A \cdot E^{1.67}}{\rho \cdot Z^{0.89}}$$

is used for the Kanaya-Okayama calculation and assumes, for compound specimens, that the average atomic number and average atomic weight are calculated based on the weight fractions of the elements in the specimen.

The expression:

$$\text{transmission fraction} = e^{-\mu \rho t}$$

is used to calculate the effective x-ray transmission, where  $\mu$  is the mass absorption coefficient,  $\rho$  is the density and  $t$  is the thickness and assumes, for compound specimens, that the average mass absorption coefficient is calculated based on the weight fractions of the elements in the specimen.

### **Use Oxygen From Halogens (F, Cl, Br and I) Correction**

This option will allow the program to calculate the equivalent oxygen from the measured or specified halogen concentrations (F, Cl, Br and I) and subtract that amount from the amount of calculated stoichiometric oxygen.

During the calculation, since it requires two halogen atoms to replace one oxygen atom, one-half (by atom) of the halogens present are converted to equivalent oxygen and that amount is subtracted during the compositional iteration procedure. The adjustment is iterated along with the modified matrix correction factors, due to the change in overall composition. This option is applicable for ZAF/phi-rho(z), Bence-Albee and calibration curve matrix correction calculations. This option applies only to samples where oxygen is calculated by stoichiometry and contain acquired or specified F, Cl, Br or I.

If this option is NOT used for samples where oxygen is calculated by stoichiometry and halogens are present, the software will simply report the oxygen equivalent of the halogens without subtracting the calculated amount. In this case, the user may then manually subtract the oxygen equivalent from the stoichiometric calculated oxygen; however, since the matrix correction is not adjusted for the change in oxygen concentration, the calculation of the other elements (especially F, due to its large correction factor in the presence of oxygen) will be slightly in error.

Finally it should be noted that to be internally consistent in the matrix calculations, all oxide standard compositions used in halogen analyses should reflect the same adjustment for equivalent oxygen in the standard database as is used for the unknown (or standard) analysis in Probe for EPMA.

For example, the following standard composition is entered with the assumption that all cations have a full complement of stoichiometric oxygen (and no correction for F or Cl):

```

St 112 biotite #3
TakeOff = 40 KiloVolts = 15
Oxide and Elemental Composition

Average Total Oxygen: 40.474      Average Total Weight%: 101.488
Average Calculated Oxygen: 40.474  Average Atomic Number: 13.413
Average Excess Oxygen: .000       Average Atomic Weight: 21.358
Oxygen Equiv. from Halogen: 1.697

ELEM: SiO2 Al2O3 FeO MgO CaO Na2O K2O TiO2
XRAY: ka ka ka ka ka ka ka ka
OXWT: 38.622 10.721 18.131 14.011 .020 .690 9.210 2.290
ELWT: 18.053 5.674 14.093 8.449 .014 .512 7.646 1.373
KFAC: .1368 .0382 .1205 .0528 .0001 .0024 .0689 .0119
ZCOR: 1.3201 1.4841 1.1692 1.6008 1.0860 2.1594 1.1093 1.1506
ATWT: 13.527 4.426 5.311 7.316 .007 .469 4.115 .603

ELEM: MnO BaO Rb2O Cl F H2O O
XRAY: ka la la ka ka ka ka
OXWT: .950 .111 .030 .020 4.020 2.663 .000
ELWT: .736 .099 .027 .020 4.020 .298 40.474
KFAC: .0062 .0008 .0002 .0002 .0112 .0030 .1826
ZCOR: 1.1891 1.3161 1.4008 1.2064 3.6053 .0000 2.2167
ATWT: .282 .015 .007 .012 4.453 6.222 53.236

```

Note that the total for the above composition is actually greater than 100% due to the fact that in reality the fluorine and chlorine actually replace some of the cation oxygen in this mineral. Note also, the oxygen equivalent from all halogens (F, Cl, Br and I) is reported (in the header), but not subtracted from the stoichiometric oxygen (oxygen from cations).

Calculations using this standard composition and an adjustment for equivalent oxygen from halogens in the matrix correction procedure will be slightly in error due to the fact that the standard k-factor calculation will not reflect the proper reduction of stoichiometric oxygen due to the presence of halogens.

Since the default mode of this analysis option (unchecked) is to only display the equivalent oxygen and not actually utilize it in the matrix corrections, then results calculated using typical standard compositions will at least be internally consistent.

However, if it is desired to use this analysis option by reducing the calculated stoichiometric oxygen in the matrix correction then for internally consistent results, the user should make an adjustment (reduction) in the amount of stoichiometric oxygen in the standard composition. This is easily done by noting the actual amount of stoichiometric oxygen (adjusted) in the Standard Composition dialog (see menu Standard | Modify) and entering that value for the concentration of oxygen as seen here:

```

St 112 biotite #3
TakeOff = 40 KiloVolts = 15
Oxide and Elemental Composition

Average Total Oxygen: 38.777      Average Total Weight%: 99.791
Average Calculated Oxygen: 40.474  Average Atomic Number: 13.505
Average Excess Oxygen: -1.697    Average Atomic Weight: 21.480
Oxygen Equiv. from Halogen: 1.697

ELEM: SiO2 Al2O3 FeO MgO CaO Na2O K2O TiO2

```

|       |        |        |        |        |        |        |        |        |
|-------|--------|--------|--------|--------|--------|--------|--------|--------|
| XRAY: | ka     |
| OXWT: | 38.622 | 10.721 | 18.131 | 14.011 | .020   | .690   | 9.210  | 2.290  |
| ELWT: | 18.053 | 5.674  | 14.093 | 8.449  | .014   | .512   | 7.646  | 1.373  |
| KFAC: | .1367  | .0382  | .1207  | .0529  | .0001  | .0024  | .0689  | .0119  |
| ZCOR: | 1.3208 | 1.4841 | 1.1681 | 1.5982 | 1.0858 | 2.1546 | 1.1091 | 1.1499 |
| ATWT: | 13.836 | 4.527  | 5.432  | 7.483  | .008   | .479   | 4.209  | .617   |
| ELEM: | MnO    | BaO    | Rb2O   | Cl     | F      | H2O    | O      |        |
| XRAY: | ka     | la     | la     | ka     | ka     |        | ka     |        |
| OXWT: | .950   | .111   | .030   | .020   | 4.020  | 2.663  | -1.697 |        |
| ELWT: | .736   | .099   | .027   | .020   | 4.020  | .298   | 38.777 |        |
| KFAC: | .0062  | .0008  | .0002  | .0002  | .0113  | .0030  | .1731  |        |
| ZCOR: | 1.1881 | 1.3153 | 1.4016 | 1.2073 | 3.5626 | .0000  | 2.2398 |        |
| ATWT: | .288   | .016   | .007   | .012   | 4.555  | 6.364  | 52.168 |        |

Note that if excess oxygen from Fe or another variable valence oxide is also present and reported, then that concentration needs to be added to the actual oxygen concentration (in Elements/Cations stoichiometry, e.g. Fe:O of 2:3 rather than 1:1) after subtraction of the oxygen equivalent from the halogens.

### **Use Nth Point Calculation For Off-Peak Intensities (for testing purposes only)**

This menu is for demonstration purposes only and will force all points in a sample to use the off-peak intensity measurements from the first point in the sample for all points in the sample. It is intended only for demonstrating the possible errors arising from using the Nth Point Acquisition option in the Acquisition Options dialog under Acquire!

### **Use Count Overwrite Table Intensity Table for Data Calculations**

This option will allow the program to utilize any non-zero “count overwrite” values to temporarily overwrite the actual measured x-ray intensities for the on-peak, high off-peak or low off-peak acquisition table values. This provides a convenient method to toggle between various “what-if” scenarios especially for use with various third party off-line background regression curves.

To learn the key stroke combination for adding overwrite intensity values to the CountOverwrite table please contact Probe Software directly. This information is not released generally to prevent users from accidentally adding overwrite values to the table.

### **Use Automatic Format For Results**

This option will allow the program to use maximum precision or only the statistically significant digits when formatting numbers for output rather than fixed format (F8.3). This option is useful when printing out trace element concentrations when more significant digits to the right of the decimal point are desired or when it is desirable to easily ascertain exactly how sensitive the quantitative analysis is, based on the counting statistics.

If the statistically significant automatic format output option is selected, then the program will calculate the percent error (precision) and the detection limit for each element and use this information to first format the number so that only statistically significant digits are displayed and finally it will check the value against the calculated detection limit and set the value to "n.d." (not detected) if the value is below the detection limit. This is scaled for each data type output (elemental, oxide, atomic percent and formula).

The calculation for significant digits is based on the percent analytical error as follows:

| Percent Analytical Error | Number of Significant Digits |
|--------------------------|------------------------------|
| > 10                     | 1                            |
| > 1                      | 2                            |
| > 0.1                    | 3                            |
| > 0.01                   | 4                            |

|          |   |
|----------|---|
| > 0.001  | 5 |
| > 0.0001 | 6 |

### **Use Detailed Printout for Data and Analytical Results**

This option will disable the output of some of the printed output to the log window for a quantitative analysis. The output options not seen if this option is enabled include the standard and unknown k-factors and ZAF correction factors.

### **Print Analyzed (Acquired) and Specified On Same Line**

This option will allow both analyzed (acquired or measured) and specified elements to be printed out on the same line in the log window. Normally the default is to print specified elements on a separate line from the analyzed elements. This causes the log window and Analyze! grid results to be more comparable in appearance.

### **Display Count Intensities Unnormalized To Time (in Analyze!)**

This option displays the x-ray intensities in the Analyze! window un-normalized to count time. In other words it displays the raw intensities. The log window intensities will still be displayed normalized to count time.

### **Display Charge Balance Calculation**

This option will output the calculated charge balance to the log window for the analyzed composition based on the Charge parameter as specified in the Elements/Cations dialog for each element.

### **Elemental Output Sort Order**

These options allow the user to specify the sort order for various output using either the Sort Stat and Data Grids In Geological or Atomic Number Order button in the Analyze! window and also several of the custom output formats.

The choices are traditional geological sort order based on cation oxidation ratio, low to high atomic number and high to low atomic number order.

## **Assign MAN Fits**

This menu allows the user to display or modify the MAN (mean atomic number) background assignments and fits used for the background correction of elements in the currently open Probe for EPMA database. The user has the option of using absorption corrected intensities and constant, linear or polynomial fits. An interactive graphical display is provided to facilitate the assignment process.

These MAN assignments are based on the element, x-ray, spectrometer number and crystal (and kilovolts and take-off angle) conditions and are applied globally to all quantitative samples in the run. If an element is analyzed on more than one set of x-ray, spectrometer number or crystal conditions, then the user is required to assign MAN fits for each additional set of conditions.

**Be sure to click the Update Fit button if any changes are made to the MAN assignments. This must be done after the changes to the MAN assignments to each element are made.**

MAN background corrections can be utilized when the P/B is sufficiently high (accuracy limit is ~100-200 PPM in most oxides and silicates) unless the “blank” correction is also applied.

The MAN Fits menu allows the user to enter which MAN (mean atomic number) background samples will be used to calculate the background fit for the polynomial MAN iteration of an analyzed sample. The MAN background correction scheme is valid, because the primary factor of background or continuum variation (aside from trace contamination and peak over-lap interferences) is the average atomic number of the sample. If a number of pure samples of known atomic number are measured, then a function can be fit to the data and applied to the unknown sample. However, since the atomic number depends on the composition of the unknown and that is not known, then the MAN background must be applied during the ZAF iteration calculation.

Again, remember that MAN backgrounds can be used whenever the ratio of x-ray peak to background is high (>10:1), and this is usually the case with WDS microanalysis. When measuring trace quantities be sure to use the off-peak measurements although the MAN correction can be utilized if a suitable matrix matched “blank” standard is available.

### **MAN Backgrounds**

There are three methods for performing a background measurement acquisition for WDS x-ray intensity data. The method most familiar to many microprobe users is the Off-Peak background method. Probe for EPMA uses this method, also a new Multi-Point off-peak background correction and another called the Mean Atomic Number (MAN) background correction.



This latter method is based on the fact that most of the background (continuum) production in a sample is directly proportional to the average atomic number of the sample. If the average atomic number of the sample is low (for example, silicates) and the peak to background ratio is high, then the MAN background correction method works quite well down to minor element concentrations, since the total background is often less than one standard deviation of the peak intensity of the characteristic x-ray signal.

The advantage of the MAN method is that it requires only a simple calibration of the analyzing channel over a range of atomic number that includes the atomic number range of the standards and samples. If many samples are to be measured for their major and even minor element concentrations, then substantial time may be saved using the MAN method.

However, if measuring high average atomic number samples (greater than, for example, silicates) and/or trace concentrations, one will need to use the off-peak background correction method. See below for more on off-peak background corrections.

The Mean Atomic Number (MAN) background correction is an empirical calibration curve correction for background. Probe for EPMA uses an iterated linear or polynomial fit of up to 16 background measurements to provide quantitative correction over a wide range of sample atomic numbers.

The technique is based on the fact that background is essentially a function of the average atomic number of a sample often referred to as "Z-bar". You can determine the Z-bar of any composition by using the "data" command in program Standard to print out a standard composition. You can of course also create a dummy composition for reference purposes and delete it later, or simply view the average atomic number values for standards with a similar composition to the one of interest in Standard.

The program uses a graphical display of the measured counts on the background standards versus their Z-bars for each analyzed element to allow the user to select individual points for the calibration curve. Once the curves are fit the program can then calculate the background of any sample during the ZAF iteration based on the composition of the sample for a quantitative background correction.

### **Selecting Suitable Standards for the MAN Calibration**

To start with you should have some idea of the range of Z-bar of your unknown samples. For silicates this will normally be in the 10.5 to 14 or 15 range, for oxides 10 or 12 to 20 or 22, for other samples use the Standard program to calculate the ranges or just look at a copy of the periodic table and estimate in your head (average atomic number is approximately calculated using the sum of the weight fractions of the atomic numbers in a compound). You then select standards (usually simple synthetic oxides or metals of high purity that will cover the range of Z-bar for both the standards and unknowns that you will be analyzing).

For example, in a silicate analysis of olivine the range of Z-bar will be from forsterite (10.6) to fayalite (18.7). Therefore you would select several standards in that range to use as MAN backgrounds including for instance MgO (Z-bar=10.4), Al<sub>2</sub>O<sub>3</sub> (Z-bar=10.6), SiO<sub>2</sub> (Z-bar=10.8), TiO<sub>2</sub> (Z-bar=16.4) and NiO (Z-bar=21.2). These standards adequately cover the range of Z-bar for olivine analysis. Other pure oxides of similar Z-bar could, of course, be used also.

To begin manually collecting data on a MAN background (let's say MgO, which was given a standard number of 12 in the standard database), create a new unknown sample from the Acquire! window. Click OK and the sample will display in the log window.

Note that "Set 1" means the first time you have collected data for this standard sample in this run. You can now manually start the counters, and collect a series of lines or points of data. Each data line or point of data represents a single unit of counting time. These lines or points are grouped together into samples each containing 100 or fewer "lines" (usually 3 to 5). All lines are numbered sequentially, and these line numbers are used frequently for reference purposes.

The line number, "status" of each line, beam counts and the x-ray counts on each channel are typed on the screen as the data is collected. The status of every line is initially "G" for good. If you see a bad line (epoxy, etc.) you can disable it at any time from the Analyze! window. This will change the status of that line to "B" for bad, and it will be ignored in all calculations. It may later be enabled if you change your mind.

When you have collected a sufficient number (5-10) of good lines for this background sample, proceed to the next one. Note that as each point is acquired, the program will print out a summary of the current sample. The summary includes average counts, the standard deviation and the one sigma (predicted standard deviation) of the average counts on successive lines. If the scatter in the counts is due totally to counting randomness, the standard deviation of the number of counts should equal to or less than the one sigma values. If the standard deviation is much larger it indicates scatter due to other causes, such as sample inhomogeneity etc.

Continue collecting data on the MAN background samples until you are done. Although the background due to the continuum usually drifts very little, the program will automatically correct the MAN background correction for drift, if more than one set of MAN backgrounds are acquired. If it is suspected that the background is drifting, possibly due to a change in the P-10 gas purity for instance, simply acquire an additional set of MAN background samples and the program will automatically calculate a linear drift correction before each quantitative analysis. For a description on the form of the drift correction see the section above titled "Automatic Standard Drift Correction".

### **MAN Background Assignments**

Once you have collected all the MAN (Mean Atomic Number) background samples you must decide which MAN backgrounds to use on each channel. MAN background corrections are made by assuming that background counts are a

linear function of the mean atomic number of the sample. For each channel you may give the program up to 10 different backgrounds to fit. It will compute a second order polynomial (default) or force a straight line fit between background counts and MAN for these samples. These coefficients are then used to determine background counts on your unknowns, based on iterative approximations of the composition of the unknowns.

**Be sure to click the Update Fit button if any changes are made to the MAN assignments. This must be done after the changes to the MAN assignments to each element are made.**

You will find that the MAN background corrections in PROBE work quite well down to approximately 200-300 ppm (0.02-0.03 weight percent) levels in silicate samples ( $\bar{Z}$  ~10 - 15) using 10 to 20 second count times. Higher atomic number samples will of course be more difficult and may require longer count times or the use of off-peak background measurements.

### ***Correction for Absorption of the Continuum***

Probe for EPMA will also automatically perform an absorption correction for the continuum background counts if it is specified. This correction is made using the ZAF or Phi-Rho-Z absorption correction selected by the user. Although it is not absolutely correct to use the absorption correction for characteristic lines to correct absorption of the continuum, it is a good approximation and since the background correction itself is a second order effect the difference is negligible (Donovan, 1996).

The correction for absorption is made by simply multiplying the absorption correction for each analyzed element in each MAN background standard times the measured counts before fitting to the calibration curve. This results in a MAN background calibration curve corrected for absorption. Then, before subtracting the background correction for the standards or unknowns, the program uncorrects the calculated background counts obtained from the calibration curve based on the absorption correction for that specific standard or unknown. This effect is usually very small, on the order of 100 ppm, however if a large absorption edge is present it could improve the MAN background correction somewhat.

Note that when comparing the background fit with the actual calculated background counts, be aware that because the MAN background fit is based on counts corrected for absorption, the calculated background for a particular sample is automatically uncorrected for the absorption in the sample, by dividing the calculated background from the fit by the absorption correction term, before subtracting the background counts from the measured raw intensity. This of course only applies when using the correction for absorption on the MAN fits.

### ***Graphical Plot of Background vs. Z-bar***

To tell the program which MAN backgrounds you want to use on which channels, click the MAN Fits under the Analytical menu. The program will load the standard count data and graphically plot the acquired MAN background samples using a default assignment. After selecting or deselecting (hold down the <ctrl> key while clicking) standards in the standard list box click the Update Fit button to save the changes. The program will then calculate and display the updated fit. The intercept, slope and curvature coefficients and the average relative deviation in percent is also displayed on the graph. You should avoid using samples which contain the channel element or produce an interference. These samples will show up as high points on the graph.

Note that the default selection of MAN standards by Probe for EPMA is somewhat intelligent; if the standard database composition for the standard contains more than 0.01 weight percent of the element in question, then the program will not assign that standard in the initial plot. Of course, the user can still specify the standard explicitly by clicking it in, but remember, if the element is present then you are not actually measuring the background for that element. The "force linear fit" option can be used when you feel that the program is over-fitting the data.

You can assign the MAN fits as soon as you finish collecting data for the backgrounds, or just before you begin your first analysis. You can change the MAN assignments you have made at any time. If additional MAN backgrounds are necessary, add them to the run and acquire the additional data.

## **Clear All MAN Assignments**

This menu will clear the current probe database of all MAN (mean atomic number) background assignments so that the next time the Assign MAN Fits menu is selected the program will automatically load an appropriate set of MAN assignments based on the currently available standards. This is generally only necessary, when after loading in a setup from another run, the user significantly modifies the element/spectrometer setup resulting in "orphaned" MAN assignments. In this case it is sometimes easier to simply clear the MAN assignments loaded with the file setup and re-assign them using the Assign MAN Fits menu.

After clearing the existing MAN assignments, simply use the MAN Fits menu to load the default MAN assignments for the current element set in the run based on the loaded standards and then modify as necessary.

## **Use Off Peak Elements For MAN Fit**

This menu will allow the program to utilize intensity data from standards that were acquired using off-peak backgrounds, for the MAN fit calibration. This means that elements in unknown or standard samples that were acquired using the MAN background acquisition option (no off-peaks), can utilize intensity data from standards that were acquired using the off-peak background acquisition option.

This is possible because an off peak element acquisition already contains the on-peak intensity acquired along with the off-peak intensities. When this option is utilized, the off peak data for standard assigned for the MAN fit is NOT subtracted from the on peak intensities so the "uncorrected" on-peak intensity can be utilized the purposes of the MAN background assignments. Normally, only standards that have been acquired using the MAN background acquisition option are utilized for the MAN assignments.

To enable this option, one must click the Use Off Peak Elements For MAN Fit menu, then one must clear the MAN fits using the Clear All MAN Assignments menu, and then re-assign the MAN fits using the Assign MAN Fits menu.

## **Use MAN Correction For Off Peak Elements**

This menu will allow the user to perform MAN background corrections on element data that was originally acquired using off-peak background methods (assuming that the MAN calibration curves are properly assigned). This option may require the acquisition of MAN standards for the MAN calibration and for user to clear the MAN fits and re-assign the MAN assignments to make sure that the off-peak elements are assigned MAN calibration curves.

This option is very powerful in that the user can switch between MAN and off-peak background corrections on the same sample data set to see exactly what the difference between the two methods are. Once the off-peak elements have been properly assigned, there is no need to re-clear and re-assign the MAN assignments when subsequently using this menu option.

## **Empirical MACs**

This menu allows the user to selectively load empirical MACs (mass absorption coefficients) from a supplied ASCII file or data entry.

Empirical MAC (Mass Absorption Coefficients) are stored in a file called EMPPMAC.DAT in the Probe for EPMA program data directory (**usually C:\ProgramData\Probe Software\Probe for EPMA**). If the user obtains any new empirical MAC data the file can be edited using any text editor such as NotePad or TextPad. Be careful to avoid adding any <tab> characters when editing the file (use space or comma delimited values only). The values can then be accessed using from the Analytical | Empirical MACs menu for runs that contains a matching emitter and absorber binary. Any changes to the default MAC values will be stored in your .MDB database file. Note that the elements may be in any order although they have been sorted to facilitate editing.

The format of the EMPPMAC.DAT file is shown below :

|     |      |     |      |                  |
|-----|------|-----|------|------------------|
| "b" | "ka" | "b" | 3471 | "Pouchou (1992)" |
| "b" | "ka" | "c" | 6750 | "Pouchou (1992)" |

|     |      |      |       |                      |
|-----|------|------|-------|----------------------|
| "b" | "ka" | "n"  | 11000 | "Pouchou (1992)"     |
| "b" | "ka" | "o"  | 16500 | "Pouchou (1992)"     |
| "b" | "ka" | "al" | 64000 | "Pouchou (1992)"     |
| "b" | "ka" | "si" | 80000 | "Pouchou (1992)"     |
| "b" | "ka" | "ti" | 15000 | "Pouchou (1992)"     |
| "b" | "ka" | "v"  | 18000 | "Pouchou (1992)"     |
| "b" | "ka" | "cr" | 20700 | "Pouchou (1992)"     |
| "b" | "ka" | "fe" | 27800 | "Pouchou (1992)"     |
| "b" | "ka" | "co" | 32000 | "Pouchou (1992)"     |
| "b" | "ka" | "ni" | 37000 | "Pouchou (1992)"     |
| "b" | "ka" | "zr" | 4400  | "Pouchou (1992)"     |
| "b" | "ka" | "nb" | 4500  | "Pouchou (1992)"     |
| "b" | "ka" | "mo" | 4600  | "Pouchou (1992)"     |
| "b" | "ka" | "la" | 2500  | "Pouchou (1992)"     |
| "b" | "ka" | "ta" | 23000 | "Pouchou (1992)"     |
| "b" | "ka" | "w"  | 21000 | "Pouchou (1992)"     |
| "b" | "ka" | "u"  | 7400  | "Pouchou (1992)"     |
| "c" | "ka" | "b"  | 39000 | "Pouchou (1992)"     |
| "c" | "ka" | "c"  | 2170  | "Pouchou (1992)"     |
| "c" | "ka" | "si" | 35000 | "Pouchou (1992)"     |
| "c" | "ka" | "ti" | 8097  | "Pouchou (1992)"     |
| "c" | "ka" | "v"  | 8850  | "Pouchou (1992)"     |
| "c" | "ka" | "cr" | 10700 | "Pouchou (1992)"     |
| "c" | "ka" | "fe" | 13150 | "Pouchou (1992)"     |
| "c" | "ka" | "zr" | 25000 | "Pouchou (1992)"     |
| "c" | "ka" | "nb" | 24000 | "Pouchou (1992)"     |
| "c" | "ka" | "mo" | 20500 | "Pouchou (1992)"     |
| "c" | "ka" | "hf" | 18000 | "Pouchou (1992)"     |
| "c" | "ka" | "ta" | 17000 | "Pouchou (1992)"     |
| "c" | "ka" | "w"  | 18000 | "Pouchou (1992)"     |
| "n" | "ka" | "b"  | 15800 | "Pouchou (1992)"     |
| "n" | "ka" | "n"  | 1640  | "Pouchou (1992)"     |
| "n" | "ka" | "al" | 13800 | "Pouchou (1992)"     |
| "n" | "ka" | "si" | 17000 | "Pouchou (1992)"     |
| "n" | "ka" | "ti" | 4270  | "Pouchou (1992)"     |
| "n" | "ka" | "v"  | 4950  | "Pouchou (1992)"     |
| "n" | "ka" | "cr" | 5650  | "Pouchou (1992)"     |
| "n" | "ka" | "fe" | 7190  | "Pouchou (1992)"     |
| "n" | "ka" | "zr" | 24000 | "Pouchou (1992)"     |
| "n" | "ka" | "nb" | 25000 | "Pouchou (1992)"     |
| "n" | "ka" | "mo" | 25800 | "Pouchou (1992)"     |
| "n" | "ka" | "hf" | 14000 | "Pouchou (1992)"     |
| "n" | "ka" | "ta" | 15500 | "Pouchou (1992)"     |
| "o" | "ka" | "li" | 1600  | "Bastin (1992)"      |
| "o" | "ka" | "b"  | 8550  | "Bastin (1992)"      |
| "o" | "ka" | "b"  | 1200  | "Bastin (1992)"      |
| "o" | "ka" | "f"  | 1850  | "Love et al. (1974)" |
| "o" | "ka" | "ne" | 2750  | "Love et al. (1974)" |
| "o" | "ka" | "na" | 3630  | "Love et al. (1974)" |
| "o" | "ka" | "mg" | 5170  | "Bastin (1992)"      |
| "o" | "ka" | "al" | 6720  | "Bastin (1992)"      |
| "o" | "ka" | "si" | 8790  | "Bastin (1992)"      |
| "o" | "ka" | "p"  | 9820  | "Love et al. (1974)" |
| "o" | "ka" | "s"  | 12400 | "Love et al. (1974)" |
| "o" | "ka" | "cl" | 14300 | "Love et al. (1974)" |
| "o" | "ka" | "ar" | 16100 | "Love et al. (1974)" |
| "o" | "ka" | "k"  | 20500 | "Love et al. (1974)" |
| "o" | "ka" | "ca" | 24600 | "Love et al. (1974)" |
| "o" | "ka" | "sc" | 26800 | "Love et al. (1974)" |
| "o" | "ka" | "ti" | 19900 | "Bastin (1992)"      |
| "o" | "ka" | "cr" | 2900  | "Bastin (1992)"      |
| "o" | "ka" | "mn" | 3470  | "Bastin (1992)"      |
| "o" | "ka" | "fe" | 4000  | "Bastin (1992)"      |
| "o" | "ka" | "co" | 4500  | "Bastin (1992)"      |
| "o" | "ka" | "ni" | 5120  | "Bastin (1992)"      |
| "o" | "ka" | "cu" | 5920  | "Bastin (1992)"      |
| "o" | "ka" | "zn" | 6350  | "Bastin (1992)"      |
| "o" | "ka" | "ga" | 7090  | "Bastin (1992)"      |
| "o" | "ka" | "y"  | 15100 | "Bastin (1992)"      |
| "o" | "ka" | "zr" | 16200 | "Bastin (1992)"      |
| "o" | "ka" | "nb" | 17100 | "Bastin (1992)"      |

|      |      |      |       |                       |
|------|------|------|-------|-----------------------|
| "o"  | "ka" | "mo" | 18000 | "Bastin (1992)"       |
| "o"  | "ka" | "ru" | 19700 | "Bastin (1992)"       |
| "o"  | "ka" | "sn" | 15050 | "Bastin (1992)"       |
| "o"  | "ka" | "ba" | 4560  | "Bastin (1992)"       |
| "o"  | "ka" | "la" | 3600  | "Bastin (1992)"       |
| "o"  | "ka" | "ta" | 10600 | "Bastin (1992)"       |
| "o"  | "ka" | "w"  | 11300 | "Bastin (1992)"       |
| "o"  | "ka" | "pb" | 11000 | "Bastin (1992)"       |
| "o"  | "ka" | "bi" | 12100 | "Bastin (1992)"       |
| "f"  | "ka" | "xe" | 27300 | "Henke et al. (1982)" |
| "na" | "ka" | "zn" | 10500 | "Henke et al. (1982)" |
| "na" | "ka" | "pm" | 7920  | "Henke et al. (1982)" |
| "mg" | "ka" | "tb" | 8240  | "Henke et al. (1982)" |
| "al" | "ka" | "tm" | 4730  | "Henke et al. (1982)" |
| "cr" | "la" | "te" | 39500 | "Henke et al. (1982)" |
| "co" | "la" | "co" | 1920  | "Henke et al. (1982)" |
| "ge" | "ka" | "ta" | 265   | "Henke et al. (1982)" |
| "ge" | "la" | "gd" | 8500  | "Henke et al. (1982)" |
| "mo" | "la" | "hg" | 951   | "Henke et al. (1982)" |

The first column ("b" in the example equals Boron) is the atomic symbol of the emitting element. The second column ("ka" in the example equals K $\alpha$ ) is the x-ray line of the emitting element. The third column is the atomic symbol of the absorber element. The element and x-ray symbols must be enclosed in double quotes. The fourth column is the actual mass absorption coefficient (MAC) that has been experimentally measured. The last parameter is a comment string that may contain a comment, usually the experimenter or attribution. The comment string must be in double quotes.

The first and third columns (emitting atomic number and absorber atomic number) must be integer values from 1 to 100. The second column (x-ray line type) must be a valid x-ray line (ka, kb, la, lb, ma, or mb). The fourth column (MAC value) must be a real (or integer) number greater than zero.

See Appendix C below for a large set of empirical mass absorption coefficients from a number of sources that indicates some of the variation among MACs from various sources.

## Empirical APFs

This menu allows the user to selectively load empirical APFs (Area Peak Factors) from a supplied ASCII file (compound APFs) or data entry (specified APFs). Specified APFs are discussed elsewhere.

A powerful feature in Probe for EPMA allows the user to select an Area-Peak-Factor (APF) correction for use in correcting x-ray intensities for peak shift and shape changes between the standard and the unknown. This can occur especially with K $\alpha$  lines of the light elements such as oxygen, nitrogen, carbon and boron. With these elements, differences in the bond energies between say TiC and SiC will produce significantly different peak shapes resulting in systematic errors in the analysis of these materials when only the peak x-ray intensities are used.

**For example, when analyzing oxygen and using MgO or Fe<sub>2</sub>O<sub>3</sub> as an oxygen standard, analyses of SiO<sub>2</sub> will produce errors up to about 4.5%. This effect is independent of the matrix correction and can be corrected for only by the use of the appropriate APFs.**

The APF concept was proposed by Bastin and Heijligers (Bastin and Heijligers, 1986) to provide a simple correction scheme for this effect. It basically requires measuring the ratio of the peak intensity to the integrated intensity between a primary standard and a number of secondary standards on the light element spectrometer used. The wavelength scan feature in Probe for EPMA can be used to acquire the peak shape profiles. After correcting for background and removal of extraneous peaks from interfering lines (requires use of an off-line peak stripping program), the APF can be calculated. The calculation of the APF factor is shown below :

$$APF = \frac{I_U^I \cdot I_S^P}{I_U^P \cdot I_S^I}$$

Where :  $I_U^I$  is the integrated intensity of the secondary standard (unknown)  
 $I_S^P$  is the peak intensity of the primary standard  
 $I_U^P$  is the peak intensity of the secondary standard (unknown)  
 $I_S^I$  is the integrated intensity of the primary standard

The peak shape and shift of light element peaks can be easily demonstrated using MgO and SiO<sub>2</sub> standard samples. First select a suitable layered dispersive element (LDE) analyzing crystal for oxygen K $\alpha$  analysis. Tune the spectrometer using the MgO standard and acquire a standard sample for MgO. Assign MgO as the calibration standard for oxygen. Then move to the SiO<sub>2</sub> standard and acquire another quantitative standard sample on the SiO<sub>2</sub> sample. Note that the analysis of SiO<sub>2</sub> using MgO as a calibration standard will result in a low total for the SiO<sub>2</sub>, somewhere around 96-97 %. This is not an error in the mass absorption coefficient (MAC), and no attempt should be made to correct this problem by entering an arbitrary MAC value.

Instead, the proper APF correction will need to be applied as shown below. Although the values below are relative to Fe<sub>2</sub>O<sub>3</sub>, you will note that the APF for MgO is 1.000, which means that these correction factors apply equally well relative to MgO. In the Oxygen Area Peak Factor table below you will note that oxygen in the presence of SiO<sub>2</sub> needs to be corrected by a factor of 1.0444 when MgO or Fe<sub>2</sub>O<sub>3</sub> is used as a calibration standard. After entering this APF correction factor for the oxygen channel and re-analyzing, note that the total is now close to 100 %.

It is very important to note that the APF values selected or entered are always measured relative to some standard sample. For example, if measuring C K $\alpha$  and using Fe<sub>3</sub>C as the primary standard for carbon, then any C K $\alpha$  APF values used must be those measured relative to Fe<sub>3</sub>C. For the same reason, if using APF values for a particular (light) element and one decides to re-assign the standard for that element, the APF values for that element must also be changed to reflect the standard re-assignment. See the section above for information on re-calculating the APF values relative to another standard.

For the above reason it is usually most efficient to simply always use the same standard for each light element analyzed. Typically (in order to utilize the APF values in the supplied EMPAPF.DAT file) these will be :

- Oxygen : MgO or Fe<sub>2</sub>O<sub>3</sub>
- Nitrogen : AlN
- Carbon : Fe<sub>3</sub>C
- Boron : B metal

Note: The **specified** APF correction in **Probe for EPMA Xtreme** will also allow the user to enter empirical APF values in each run at the bottom of the Element dialog from the Elements/Cations button in the Analyze! window and is a separate correction from the compound APF correction being discussed here.

The user may enter one or more APF factors for each emitting element although they are generally applied to soft x-ray lines. Note however that even S K $\alpha$  exhibits peak shift and shape changes when comparing sulfide and sulfate peaks and there is well documented shifts and peak shape changes for Si K $\alpha$  and Al K $\alpha$  from metallic to oxides or silicate compounds. The APF for each absorber will be summed according to its weight fraction in the composition and applied to the emitting element counts during the ZAF or Phi-Rho-Z iteration.

If it is desired to correct a complete unknown for peak shape changes, simply perform a wavelength scan on the standard being used for the light element emitter (oxygen, carbon, etc.) and also on the unknown. Calculate the APF using the expression above and then from the Analytical | Empirical APFs menu, enter the same APF for all absorbers in the unknown. The program will then sum the APFs which will be the same as the single measured APF. Note that this APF may only apply to a single composition. One can perform additional wavelength scans on other samples to confirm this however. Alternatively one can enter the single empirical APF in the APF field in the Elements/Cations dialog as described above.

The APF correction values are defined in the EMPAPF.DAT file in the Probe for EPMA program data directory (**usually C:\ProgramData\Probe Software\Probe for EPMA**). The file contains some 50 or 60 values that may or may not be applicable, depending on the analyzing crystals and standards available. The user may edit the file using any ASCII text editor such as NotePad or TextPad to insert their own measurements. Be careful to avoid adding any <tab> characters when editing the file (use space or comma delimited values only). Note that the elements may be in any order although they have been sorted to facilitate editing.

The format of the EMPAPF.DAT file is shown below :

|     |      |      |        |                        |
|-----|------|------|--------|------------------------|
| "b" | "ka" | "c"  | 1.02   | "B4C/B/STE"            |
| "b" | "ka" | "n"  | 1.2    | "BN/B/STE"             |
| "b" | "ka" | "al" | 1.12   | "AlB2/B/STE"           |
| "b" | "ka" | "al" | 1.01   | "AlB12/B/STE"          |
| "b" | "ka" | "si" | 1      | "SiB3/B/STE"           |
| "b" | "ka" | "si" | .92    | "SiB6/B/STE"           |
| "b" | "ka" | "ti" | .75    | "TiB/B/STE"            |
| "b" | "ka" | "ti" | .88    | "TiB2/B/STE"           |
| "b" | "ka" | "v"  | 1      | "VB2/B/STE"            |
| "b" | "ka" | "cr" | .9     | "CrB/B/STE"            |
| "b" | "ka" | "cr" | 1.1    | "CrB2/B/STE"           |
| "b" | "ka" | "fe" | 1.1    | "FeB/B/STE"            |
| "b" | "ka" | "fe" | 1.25   | "Fe2B/B/STE"           |
| "b" | "ka" | "co" | 1.2    | "CoB/B/STE"            |
| "b" | "ka" | "co" | 1.02   | "Co2B/B/STE"           |
| "b" | "ka" | "ni" | 1.2    | "NiB/B/STE"            |
| "b" | "ka" | "ni" | 1.06   | "Ni2B/B/STE"           |
| "b" | "ka" | "ni" | .98    | "Ni3B/B/STE"           |
| "b" | "ka" | "zr" | .8     | "ZrB2/B/STE"           |
| "b" | "ka" | "nb" | .8     | "NbB/B/STE"            |
| "b" | "ka" | "nb" | .9     | "NbB2/B/STE"           |
| "b" | "ka" | "mo" | .94    | "MoB/B/STE"            |
| "b" | "ka" | "la" | .9     | "LaB6/B/STE"           |
| "b" | "ka" | "ta" | .88    | "TaB/B/STE"            |
| "b" | "ka" | "ta" | 1.1    | "TaB2/B/STE"           |
| "b" | "ka" | "w"  | .98    | "WB/B/STE"             |
| "b" | "ka" | "u"  | 1.04   | "UB4/B/STE"            |
| "c" | "ka" | "b"  | 1.01   | "B4C/Fe3C/WSi/59.8"    |
| "c" | "ka" | "si" | .933   | "SiC/Fe3C/WSi/59.8"    |
| "c" | "ka" | "ti" | .868   | "TiC/Fe3C/WSi/59.8"    |
| "c" | "ka" | "v"  | .873   | "V2C/Fe3C/WSi/59.8"    |
| "c" | "ka" | "v"  | .873   | "VC/Fe3C/WSi/59.8"     |
| "c" | "ka" | "cr" | .8     | "Cr7C3/Fe3C/STE"       |
| "c" | "ka" | "cr" | .83    | "Cr3C2/Fe3C/STE"       |
| "c" | "ka" | "cr" | .8     | "Cr23C6/Fe3C/STE"      |
| "c" | "ka" | "zr" | .88    | "ZrC/Fe3C/WSi/59.8"    |
| "c" | "ka" | "nb" | .79    | "NbC/Fe3C/STE"         |
| "c" | "ka" | "mo" | .82    | "Mo2C/Fe3C/STE"        |
| "c" | "ka" | "hf" | .83    | "HfC/Fe3C/STE"         |
| "c" | "ka" | "ta" | .96    | "TaC/Fe3C/STE"         |
| "c" | "ka" | "w"  | .97    | "WC/Fe3C/STE"          |
| "c" | "ka" | "w"  | 1.02   | "W2C/Fe3C/STE"         |
| "n" | "ka" | "si" | 1.103  | "Si3N4/AlN/WSi/59.8"   |
| "n" | "ka" | "ti" | .997   | "TiN/AlN/WSi/59.8"     |
| "n" | "ka" | "v"  | 1.0226 | "VN/AlN/WSi/59.8"      |
| "n" | "ka" | "cr" | 1.018  | "Cr2N/AlN/WSi/59.8"    |
| "n" | "ka" | "fe" | 1.012  | "Fe2N/AlN/WSi/59.8"    |
| "n" | "ka" | "zr" | .9952  | "ZrN/AlN/WSi/59.8"     |
| "n" | "ka" | "hf" | 1.002  | "HfN/AlN/WSi/59.8"     |
| "o" | "ka" | "b"  | 1.0628 | "B6O/Fe2O3/WSi/59.8"   |
| "o" | "ka" | "mg" | 1      | "MgO/Fe2O3/WSi/59.8"   |
| "o" | "ka" | "al" | 1.0213 | "Al2O3/Fe2O3/WSi/59.8" |
| "o" | "ka" | "si" | 1.0444 | "SiO2/Fe2O3/WSi/59.8"  |
| "o" | "ka" | "ti" | .9796  | "TiO2/Fe2O3/WSi/59.8"  |
| "o" | "ka" | "cr" | .993   | "Cr2O3/Fe2O3/WSi/59.8" |
| "o" | "ka" | "mn" | 1.0121 | "MnO/Fe2O3/WSi/59.8"   |
| "o" | "ka" | "fe" | .9962  | "Fe3O4/Fe2O3/WSi/59.8" |
| "o" | "ka" | "co" | 1.0133 | "CoO/Fe2O3/WSi/59.8"   |
| "o" | "ka" | "ni" | 1.0153 | "NiO/Fe2O3/WSi/59.8"   |

```

"o"      "ka"      "cu"      .9946      "Cu2O/Fe2O3/WSi/59.8"
"o"      "ka"      "cu"      .9943      "CuO/Fe2O3/WSi/59.8"
"o"      "ka"      "zn"      .9837      "ZnO/Fe2O3/WSi/59.8"
"o"      "ka"      "ga"      1          "Ga2O3/Fe2O3/WSi/59.8"
"o"      "ka"      "zr"      .9823      "Y3Fe5O12/Fe2O3/WSi/59.8"

```

The first column (in the example is "b" for Boron) is the atomic symbol of the emitting element. The second column ("ka" in the example equals K $\alpha$ ) is the x-ray line of the emitter. The third column is the atomic symbol of the absorber element. The fourth column is the actual area peak factor (APF) that has been experimentally measured. The last parameter is a string that contains the conditions under which the APF was measured. For example, the comment "B4C/B/STE" indicates that the peak and integrated intensities for the APF were measured on a B<sub>4</sub>C (boron carbide) sample, relative to a B (elemental Boron) primary standard, using a Pb Stearate analyzer. The reason that this information is needed is because the APF is a relative measurement (to a standard) and the value is dependent on the resolution (crystal type) used. If a LDE (layered dispersive element) analyzer is used, the 2d of the analyzer should be indicated also as seen in the example file above.

The first and third columns (emitting atomic symbol and absorber atomic symbol) must be valid element symbols. The second column (x-ray line) must be "ka", "kb", "la", "lb", "ma" or "mb". All element and x-ray symbols must be enclosed in double quotes. The fourth column (APF value) must be a real number greater than 0.0. The comment string must be enclosed in double quotes.

These values can then be loaded from the Analytical | Empirical APFs menu item in Probe for EPMA. Note that the comments listed after each APF value can be used to indicate the experimental conditions under which the APF was measured. For consistency sake, the formula of the compound measured is listed first, then the primary reference standard used and finally the analyzing crystal type (and 2d spacing if desired) used. Edit the EMPAPF.DAT file in the Probe for EPMA directory to add additional APFs from your own measurements.

The following is a short listing of some typical APFs taken from Bastin and Heijligers (1986). Because the factors are spectrometer and crystal dependent these should be used as a guide only.

### Oxygen Area Peak Factors (APF)

Relative to Fe<sub>2</sub>O<sub>3</sub> or MgO using W/Si LDE (2d = 59.8)

| Oxide  | Area-Peak Factor (APF) |
|--|------------------------|
| B <sub>6</sub> O                               | <b>1.0628</b>          |
| MgO  | <b>1.0000</b>          |
| Al <sub>2</sub> O <sub>3</sub>                 | <b>1.0213</b>          |
| SiO <sub>2</sub>                               | <b>1.0444</b>          |
| TiO <sub>2</sub>                               | <b>0.9796</b>          |
| Cr <sub>2</sub> O <sub>3</sub>                 | <b>0.9930</b>          |
| Mn <sub>3</sub> O <sub>4</sub>                 | <b>1.0121</b>          |
| Fe <sub>3</sub> O <sub>4</sub>                 | <b>0.9962</b>          |
| CoO  | <b>1.0133</b>          |
| NiO  | <b>1.0153</b>          |
| Cu <sub>2</sub> O                              | <b>0.9946</b>          |
| CuO  | <b>0.9943</b>          |
| ZnO  | <b>0.9837</b>          |
| Ga <sub>2</sub> O <sub>3</sub>                 | <b>1.0000</b>          |
| Y <sub>3</sub> Fe <sub>5</sub> O <sub>12</sub> | <b>0.9823</b>          |

## **Carbon Area-Peak Factors**

Relative to Fe<sub>3</sub>C using W/Si LDE (2d = 59.8):

| Carbide          | Area-Peak-Factor (APF) |
|------------------|------------------------|
| B <sub>4</sub> C | 1.010                  |
| SiC              | 0.933                  |
| TiC              | 0.868                  |
| V <sub>2</sub> C | 0.873                  |
| VC               | 0.873                  |
| ZrC              | 0.880                  |

## **Nitrogen Area-Peak Factors**

Relative to AlN using W/Si LDE (2d = 59.8):

| Carbide                        | Area-Peak-Factor (APF) |
|--------------------------------|------------------------|
| AlN                            | 1.000                  |
| Si <sub>3</sub> N <sub>4</sub> | 1.103                  |
| TiN                            | .997                   |
| VN                             | 1.0226                 |
| Cr <sub>2</sub> N              | 1.018                  |
| Fe <sub>2</sub> N              | 1.012                  |
| ZrN                            | .9952                  |
| HfN                            | 1.002                  |

## **Calculation of Compound APFs**

The program will automatically calculate the compound APF based the actual concentrations of the sample and the end-member APFs from the Empirical APF dialog. The calculation for compound APFs is shown here:

$$APF_{Comp} = \sum APF_j * \frac{W_j}{Sum_{Partial}}$$

Where:  $APF_j$  is the binary (end-member) APF for elements other than the affected element

$W_j$  is the weight fraction for elements other than the affected element

$Sum_{Partial}$  is the sum of all elements other than the affected element

A further normalization is performed on the compound APF to ensure that in situations where the partial sum is very low, the compound APF is not over applied to the affected (pure) element when only the trace elements are varying in precision. This calculation is show here:

$$APF_{Norm} = 1 + \frac{(APF_{Comp} - 1)}{\left( \frac{Sum_{Partial}}{W_{Affected}} \right)}$$

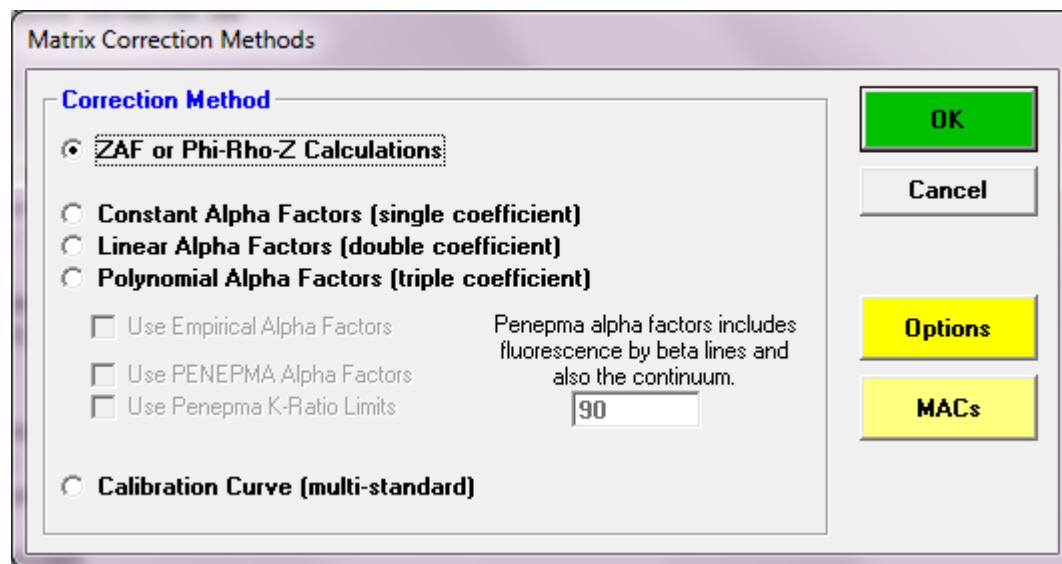
Note that this APF normalization correction is only applied when the absolute value of the scaling factor :

$$\left( \frac{Sum_{Partial}}{W_{Affected}} \right)$$

is greater than or equal to 1.0 to avoid problems with over applying the correction on major elements.

## ZAF, Phi-Rho-Z, Alpha Factor and Calibration Curve Selections

This menu allows the user to modify or display the ZAF or Phi-Rho-Z quantitative matrix correction options for all samples. The default selection is the Phi-Rho-Z correction optimized by John Armstrong for silicates. It is often the best choice for most compositions. The user will almost always find the default set of corrections satisfactory. Any changes to the default ZAF or Phi-Rho-Z selections will be saved along with any empirical MAC or APF data for the next time the run file is re-opened.



### **Alpha-Factors**

Alpha-Factor corrections based on ZAF or Phi-Rho-Z calculated k-ratio intensities and empirical a-factors from an ASCII disk file are also supported. There are two main reasons for this : first, alpha-factor corrections are very simple (once calculated for a run condition) and are therefore extremely fast. This is particularly useful when recalculating large (> 100-1000) data analyses (e.g., quantitative x-ray images).

By default the program calculates alpha factors based on the current ZAF or phi-rho-z and mass absorption coefficient table selections using the expression:

$$\alpha = (C / K - C) / (1 - C)$$

These binary alpha factors are combined into beta factors using the following expression (assuming a 2<sup>nd</sup> order polynomial fit):

$$\beta_B = \sum_1^{n_A} (\alpha_{1(B,A)} + C_A * \alpha_{2(B,A)} + C_A^2 * \alpha_{3(B,A)}) * C_A$$

Second, because it has been noted (Armstrong, 1988) that almost all reasonable ZAF or Phi-Rho-Z calculations will produce 2nd order polynomial fit alpha-factors with very similar slope and curvature terms (differing mainly in the intercept term), the use of a single binary standard empirically determined k-ratio allows the analyst to accurately construct an exact calibration curve for especially difficult matrices. This is easily accomplished by editing a single ASCII file (EMPFAC.DAT) for the empirically determined alpha-factor terms measured and fit by the user. The alpha-factor correction is summarized by the following expression :

$$C_A^u = \frac{C_A^s}{Beta_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)} Beta_{\lambda_A}^u$$

where :  $Beta_{\lambda_A}^u$  is the unknown beta-factor  
 $Beta_{\lambda_A}^s$  is the standard beta-factor

### **Use Empirical Alpha-Factors**

To utilize empirically determined Alpha-Factor terms, simply edit the EMPFAC.DAT file in the Probe for EPMA program data directory (**usually C:\ProgramData\Probe Software\Probe for EPMA**). If an Alpha-factor entry in the file matches the run conditions of the current run, it can be used to overwrite the previously internally calculated alpha-factor terms. The format of the EMPFAC.DAT file is described below :

|      |      |   |      |      |      |       |        |        |                 |         |
|------|------|---|------|------|------|-------|--------|--------|-----------------|---------|
| 52.5 | 20.0 | 1 | "au" | "la" | "ag" | 1.142 | 0.000  | 0.000  | "NBS Spec. Pub. | 260-28" |
| 52.5 | 20.0 | 1 | "ag" | "la" | "au" | 1.483 | 0.000  | 0.000  | "NBS Spec. Pub. | 260-28" |
| 52.5 | 20.0 | 1 | "au" | "la" | "cu" | 1.347 | 0.000  | 0.000  | "NBS Spec. Pub. | 260-28" |
| 52.5 | 20.0 | 1 | "cu" | "ka" | "au" | 0.758 | 0.000  | 0.000  | "NBS Spec. Pub. | 260-28" |
| 52.5 | 20.0 | 2 | "au" | "la" | "ag" | 1.159 | -0.056 | 0.000  | "NBS Spec. Pub. | 260-28" |
| 52.5 | 20.0 | 2 | "ag" | "la" | "au" | 1.505 | -0.071 | 0.000  | "NBS Spec. Pub. | 260-28" |
| 52.5 | 20.0 | 2 | "au" | "la" | "cu" | 1.347 | -0.014 | 0.000  | "NBS Spec. Pub. | 260-28" |
| 52.5 | 20.0 | 2 | "cu" | "ka" | "au" | 0.759 | -0.007 | 0.000  | "NBS Spec. Pub. | 260-28" |
| 52.5 | 20.0 | 3 | "au" | "la" | "ag" | 1.121 | 0.127  | -0.179 | "NBS Spec. Pub. | 260-28" |
| 52.5 | 20.0 | 3 | "ag" | "la" | "au" | 1.458 | 0.170  | -0.247 | "NBS Spec. Pub. | 260-28" |
| 52.5 | 20.0 | 3 | "au" | "la" | "cu" | 1.332 | 0.061  | -0.075 | "NBS Spec. Pub. | 260-28" |
| 52.5 | 20.0 | 3 | "cu" | "ka" | "au" | 0.740 | 0.089  | -0.096 | "NBS Spec. Pub. | 260-28" |

Each line contains a single empirical alpha-factors fit for half of a binary pair (one emitter-absorber system per line) for various take-off angles, operating voltage, elements, x-ray lines, etc.

Columns 1 and 2 contain the takeoff angle (degrees) and operating voltage (keV) that the alpha-factor was measured at. Column 3 is the fit flag (1 = constant expression, 2 = linear fit and 3 = polynomial fit). Columns 4 and 5 are the emitter element symbols and x-ray lines. Column 6 is the absorber element symbols. Note that Probe for EPMA only supports the use of elemental alpha-factors, i.e., referenced to elemental end-members as opposed to oxide end-members.

The three remaining columns are the alpha-factor terms. Note that constant alpha-factors have only one term, linear alpha-factors have two terms and non-linear or polynomial fit alpha-factors have three terms. For the non-linear alpha-factors, the first term is the intercept, the second term is the slope and the third term is the curvature term.

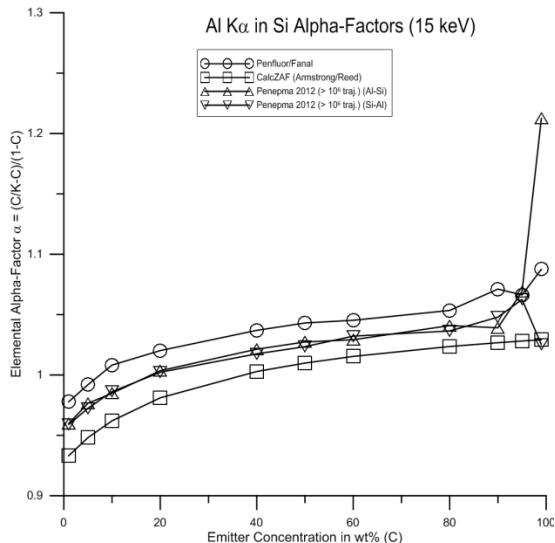
Empirical alpha-factors are calculated from experimentally measured k-ratios using a set of known binary (two element) standards covering a range of composition, by fitting the concentrations and intensity data to a plot of  $\alpha$  vs. C, where C represents the standard concentrations and  $\alpha$  is defined by the following expression :

$$\frac{\left(\frac{C}{K}\right) - C}{(1-C)} = \alpha$$

Each binary data set can be fit to any or all of the alpha-factor fits, that is, constant (single term at 50/50 composition), linear (two terms) or polynomial (three terms).

### **Use Penepma Alpha Factors**

This option allows the program to replace k-ratios calculated from the current ZAF or phi-rho-z method with k-ratios derived from Penepma (Penelope) Monte Carlo calculations. These k-ratios are stored in the matrix.mdb file and is updated periodically as further intensities are re-calculated using the latest Penepma version.



These Monte Carlo derived k-ratios provide a full physics treatment of all matrix effects including secondary fluorescence by characteristic lines (including beta and other emission lines) and also by the continuum.

### **Use Penepma K-ratio Limits**

This option can be utilized when the Use Penepma Alpha factors option is checked. It prevents the program from fitting to k-ratios greater than their specified weight percent of the emitting element. This is because as the concentration of the emitting element approaches the pure end-member of the binary, the calculation is especially sensitive to precision since one is subtracting two large numbers from each other in the alpha equation. On the other hand, the effect of the emitting element in itself approaches 1.0 in the beta equation

### **Calibration Curve**

In addition, Probe for EPMA offers a sophisticated calibration curve based on a 2nd order polynomial fit to multiple standard intensities. This option is best employed in special situations such as the analysis of trace carbon or nitrogen in metal alloys and when a suitable set of standards are available. Probe for EPMA will calculate a separate calibration curve for off-peak and MAN acquired data (MAN acquired data is not corrected for background with the Calibration Curve). If a standard is available that contains a zero concentration of the element, it will also be included in the Calibration Curve fit.

Generally, for best results, it is preferred to have a set of 4 or more standards with a similar matrix to the unknown and a range of the element of interest that brackets the unknown concentration. Probe for EPMA will construct a separate set of

calibration curves for both off-peak and MAN element data and fit a 2nd order polynomial expression that is used in the iteration procedure to determine the concentration of the element.

In the analysis printout the program will display the three polynomial fit coefficients for the standard intensity fit for each element (in cps/nominal beam units), "FIT1:", "FIT2:" and "FIT3:" and the average relative deviation in percent.

Elements calculated by difference or stoichiometry can be calculated also along with the calibration curve corrected elements. The program will print a warning if this correction method is selected.

## ZAF Matrix Corrections

ZAF matrix corrections (included here are also the newer Phi-Rho-Z corrections) are a fundamental calculation of the x-ray intensities based on physical models of the atomic and electron interaction.



It is the most accurate way available to correct for matrix effects without using an empirical calibration curve. It is also very calculation intensive, but for modern CPU's this is a non-issue, except possibly when analyzing extremely large numbers of elements or samples. The following expression summarizes the ZAF correction :

$$C_A^u = \frac{C_A^s}{[ZAF]_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)} [ZAF]_{\lambda_A}^u$$

$$\frac{C_A^s}{[ZAF]_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)}$$

where : is the unknown k-ratio and

$[ZAF]_{\lambda_A}^u$  is the ZAF correction factor of the unknown

and :  $I_A^u(\lambda_A)$  is the unknown intensity for element A at  $\lambda$

$I_A^s(\lambda_A)$  is the standard intensity for element A at  $\lambda$

$C_A^s$  is the concentration of the element in the standard

$[ZAF]_{\lambda_A}^s$  is the ZAF correction for the element in the standard

Probe for EPMA includes the most commonly used corrections schemes for ZAF, Phi-Rho-Z and PAP matrix corrections. All these options are available with a single mouse click allowing the user to switch quickly between correction schemes in order to compare the results obtained. The main choices are:

- 1 ... Select Individual Corrections
- 2 ... Armstrong/Love Scott (default)
- 3 ... Conventional Philibert/Duncumb-Reed
- 4 ... Heinrich/Duncumb-Reed
- 5 ... Love-Scott I
- 6 ... Love-Scott II
- 7 ... Packwood Phi(pz) (EPQ-91)
- 8 ... Bastin (original) Phi(pz)
- 9 ... Bastin PROZA Phi(pz) (EPQ-91)
- 10 ... PAP- Pouchou and Pichoir – Full (Original)
- 11 ... PAP- Pouchou and Pichoir – Simplified (XPP)

The default selection is the Armstrong/Love-Scott Phi-Rho-Z option. Note that depending on the actual mass absorption coefficients, atomic weights, x-ray emission and absorption edge energies and fluorescent yields used, the analytical results can vary slightly. The files used for these purposes are documented elsewhere (see CalcZAF.Exe) but are noted here as well:

|           |  |
|-----------|--|
| XLINE.DAT | Default x-ray line energies (copy XLINE1.DAT or XLINE2.DAT to this name) |
| XEDGE.DAT | Default x-ray edge energies (copy XEDGE1.DAT or XEDGE2.DAT to this name) |
| XFLUR.DAT | Default fluorescent yields (copy XFLUR1.DAT or XFLUR2.DAT to this name)  |

The following are the original files from two different sources that should be renamed to be utilized by the Probe for EPMA applications:

|            |  |
|------------|--|
| XLINE1.DAT | Johnson and White x-ray line energies (no beta energies)   |
| XEDGE1.DAT | Johnson and White x-ray edge energies (no beta energies)   |
| XFLUR1.DAT | Armstrong ELEMINFO.DAT Fluorescent yields (no beta yields) |
| XLINE2.DAT | Armstrong ELEMINFO.DAT x-ray line energies                 |
| XEDGE2.DAT | Armstrong ELEMINFO.DAT x-ray edge energies                 |
| XFLUR2.DAT | Armstrong ELEMINFO.DAT fluorescent yields (no beta yields) |

The "Select Individual Corrections" option may be used to specifically select individual correction procedures from a large array of options. Note that many correction procedures will only work with other specific choices. The following is taken from John Armstrong's CITZAF documentation on how to select among the various matrix correction options :

### **1. Choice of mean ionization potentials:**

- 1 ... Mean Ionization of Berger-Seltzer
- 2 ... Mean Ionization of Duncumb-Da Casa
- 3 ... Mean Ionization of Ruste and Zeller
- 4 ... Mean Ionization of Springer
- 5 ... Mean Ionization of Wilson
- 6 ... Mean Ionization of Heinrich
- 7 ... Mean Ionization of Bloch (Love-Scott)
- 8 ... Mean Ionization of Armstrong (Springer-Berger)
- 9 ... Mean Ionization of Joy (Wilson-Berger)

Calculation #2 should only be used if the Philibert (#1) absorption correction and Duncumb-Reed atomic number (#1) correction are employed. Selection #1 is the default.

## **2. Choice of backscatter coefficients:**

- 1 ... Backscatter Coefficient of Heinrich
- 2 ... Backscatter Coefficient of Love-Scott
- 3 ... Backscatter Coefficient of Pouchou and Pichoir
- 4 ... Backscatter Coefficient of Hungler-Kuchler (A-W Mod.)

The default selection for backscatter is #2.

## **3. Choice of absorption corrections:**

- 1 ... Absorption of Philibert (FRAME)
- 2 ... Absorption of Heinrich (Quadratic Anal. Chem.)
- 3 ... Absorption of Heinrich (Duplex 1989 MAS)
- 4 ... Absorption of Love-Scott (1978 J. Phys. D.)
- 5 ... Absorption of Sewell-Love-Scott (1985-I J. Phys. D.)
- 6 ... Absorption of Sewell-Love-Scott (1985-II J. Phys. D.)
- 7 ... Phi(pz) Absorption of Packwood-Brown 1982/XRS Alpha
- 8 ... Phi(pz) Absorption of Bastin 1984/XRS Alpha
- 9 ... Phi(pz) Absorption of Armstrong/Packwood-Brown 1981 MAS
- 10 ... Phi(pz) Absorption of Bastin 1986/Scanning
- 11 ... Phi(pz) Absorption of Riveros 1987/XRS
- 12 ... Phi(pz) Absorption of Pouchou and Pichoir-Full (Original)
- 13 ... Phi(pz) Absorption of Pouchou and Pichoir-Simplified (XPP)
- 14 ... Phi(pz) Absorption of Packwood (New)
- 15 ... Phi(pz) Absorption of Bastin Proza (EPQ-91)

The conventional ZAF correction is #1 which is also used in the FRAME program developed by Kurt Heinrich at NIST (NBS). The best results for silicates and other oxide samples are obtained with the Phi(Rho-Z) correction (#9) and with the original Love/Scott correction (#4) or the Full (Original) or Simplified (XPP) PAP correction. The default selection is #9.

## **4. Choice of Phi(0) expressions:**

(Not applicable if one of the ZAF corrections (#1-6) or PAP was selected for the absorption correction)

- 1 ... Phi(pz) Equation of Reuter
- 2 ... Phi(pz) Equation of Love-Scott
- 3 ... Phi(pz) Equation of Riveros
- 4 ... Phi(pz) Equation of Pouchou and Pichoir
- 5 ... Phi(pz) Equation of Karduck and Rehbach
- 6 ... Phi(pz) Equation of August and Wernisch
- 7 ... Phi(pz) Equation of Packwood

Both #1 and #2 give similar results. The default selection is #2.

## **5. Choice of stopping power corrections:**

- 1 ... Stopping Power of Duncumb-Reed (FRAME)
- 2 ... Stopping Power of Philibert and Tixier
- 3 ... Stopping Power (Numerical Integration)
- 4 ... Stopping Power of Love-Scott
- 5 ... Stopping Power of Pouchou and Pichoir
- 6 ... Stopping Power of Phi(pz) Integration

The conventional correction is #1. Corrections #1 and #4 give similar results. When using #4, also use the Love/Scott expressions for backscatter coefficients and Phi(0). When using #1, use the Heinrich backscatter coefficients and Reuter Phi(0). Correction #6 may only be employed when one of the Phi(Rho-Z) absorption corrections are being used. Even

then, it does not give as good results as #1 or #4 for silicates and oxides and it is seldom used. The default selection is #4.

### **6. Choice of backscatter loss corrections:**

- 0 ... No Backscatter (used for Packwood and Bastin original)
- 1 ... Backscatter of Duncumb-Reed (FRAME-I)
- 2 ... Backscatter of Duncumb-Reed (COR-II) and Heinrich
- 4 ... Backscatter of Duncumb-Reed (COR-II) and Heinrich
- 5 ... Backscatter of Love-Scott
- 6 ... Backscatter of Myklebust-I
- 7 ... Backscatter of Myklebust and Fiori (not implemented)
- 8 ... Backscatter of Pouchou and Pichoir
- 9 ... Backscatter of August, Razka and Wernisch
- 10 ... Backscatter of Springer
- 11 ... Backscatter of Donovan (Duncumb-Reed Mod.)

The conventional correction is #1. Corrections #1 and #4 give similar results. The default correction is #4. A new option is #11 which is the standard Duncumb/Reed correction with a modified backscatter loss calculation based on modified electron fractions as opposed to mass fractions. See Donovan and Westphal (2000). This modification is using a  $Z^{0.85}$  power scaling to compensate for nuclear screening effects. This modified calculation works especially well when the standard and unknown are very different in average Z.

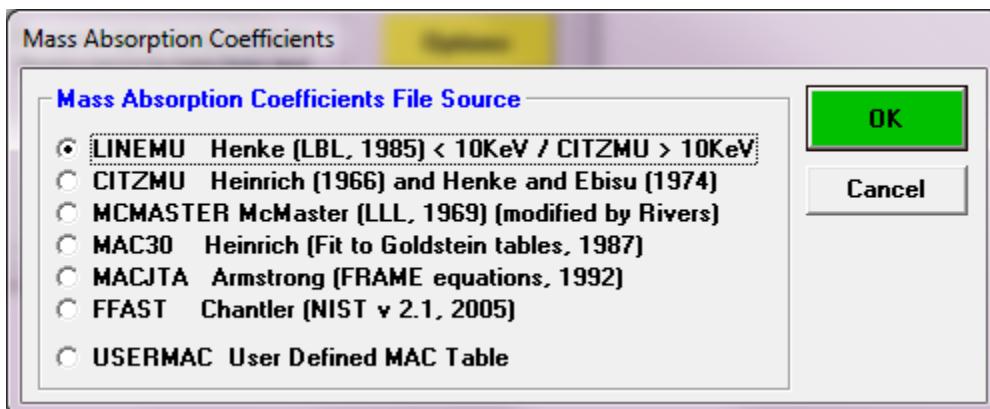
### **Fluorescence Correction**

Probe for EPMA uses as a default, a modified form of the REED/ARMSTRONG fluorescence from John Armstrong's CITZAF program. The two modifications are the addition of a fluorescence by M line correction, calibrated using data from Heinrich on Nb and U binary alloys and a modification of the fluorescence intensity (Armstrong, Microbeam Analysis, 1988). The analyst may also select the exact fluorescence correction from Armstrong's CITZAF code without the M line fluorescence or Armstrong ionization term. Also provided is a fluorescence correction with the fluorescence by M lines but without the Armstrong ionization term and a fluorescence correction with the Reed intensity expression.

- 1 ... Reed/JTA w/ M-Line Correction and JTA Intensity Mod.
- 2 ... Reed/JTA (CITZAF.BAS- original with no M-Line Correction)
- 3 ... Reed/JTA w/ M-Line Correction Only
- 4 ... Reed/JTA w/ M-Line Correction and Reed Intensity Mod.

### **Mass Absorption Coefficients**

Probe for EPMA provides seven automatic lookup tables of mass absorption coefficients for use in the analytical calculations. \



LINEMU.DAT is a compilation of Henke (unpublished, 1985) coefficients for energies less than 10 keV and Heinrich (1966) for energies greater than 10 keV.

CITZMU.DAT is the complete set of coefficients from Heinrich used by John Armstrong at CalTech/NIST which is from Heinrich (1966) and Henke and Ebisu for the light elements.

MCMASTER.DAT is a calculated set derived from equations (fits) from McMaster at Livermore National Laboratory (modified by Rivers).

MAC30.DAT is from Heinrich (1987), which is a fit to the Goldstein, et. al. MAC table.

MACJTA.DAT is from Armstrong's MACCALC program.

FFAST.DAT is a tabulation of the Chantler (et. Al.) 2005 MAC values provided by Nicholas Ritchie (NIST 2005). The original published NIST table is at: <http://physics.nist.gov/PhysRefData/FFast/Text/cover.html>

See: Chantler, C.T., Olsen, K., Dragoset, R.A., Kishore, A.R., Kotchigova, S.A., and Zucker, D.S. (2005), X-Ray Form Factor, Attenuation and Scattering Tables (version 2.1). [Online] Available: <http://physics.nist.gov/ffast> [Mar-2005]. National Institute of Standards and Technology, Gaithersburg, MD. Originally published as Chantler, C.T., J. Phys. Chem. Ref. Data 29(4), 597-1048 (2000); and Chantler, C.T., J. Phys. Chem. Ref. Data 24, 71-643 (1995).

USERMAC.DAT is a user defined table of mass absorption coefficients from any number of sources. It is not distributed by Probe Software but can be created by the user based on an existing selected MAC table and edited as desired.

**To obtain mass absorption coefficients for elements 93 and above, the USERMAC.DAT file should be created using the MAC30.DAT file as its "basis" and then updated using the USERMAC.TXT file from CalcZAF. The USERMAC.TXT file contains the database values from the Institute of Transuranium Elements. The reference is:**

**I. R. Farthing and C. T. Walker, Heinrich's Mass Absorption Coefficients for the K, L and M Lines, The European Commission, Institute for Transuranium Elements, Karlsruhe, Report K0290140, 1990**

The default mass absorption coefficient file is LINEMU.DAT. This default MAC file may be specified in the PROBEWIN.INI file. If one or more of the mass absorption coefficients in these tables are not acceptable, the user can select the use of other empirically derived values from the Analytical | Empirical MACs menu. Empirical mass absorption coefficients will override values specified from the MAC tables.

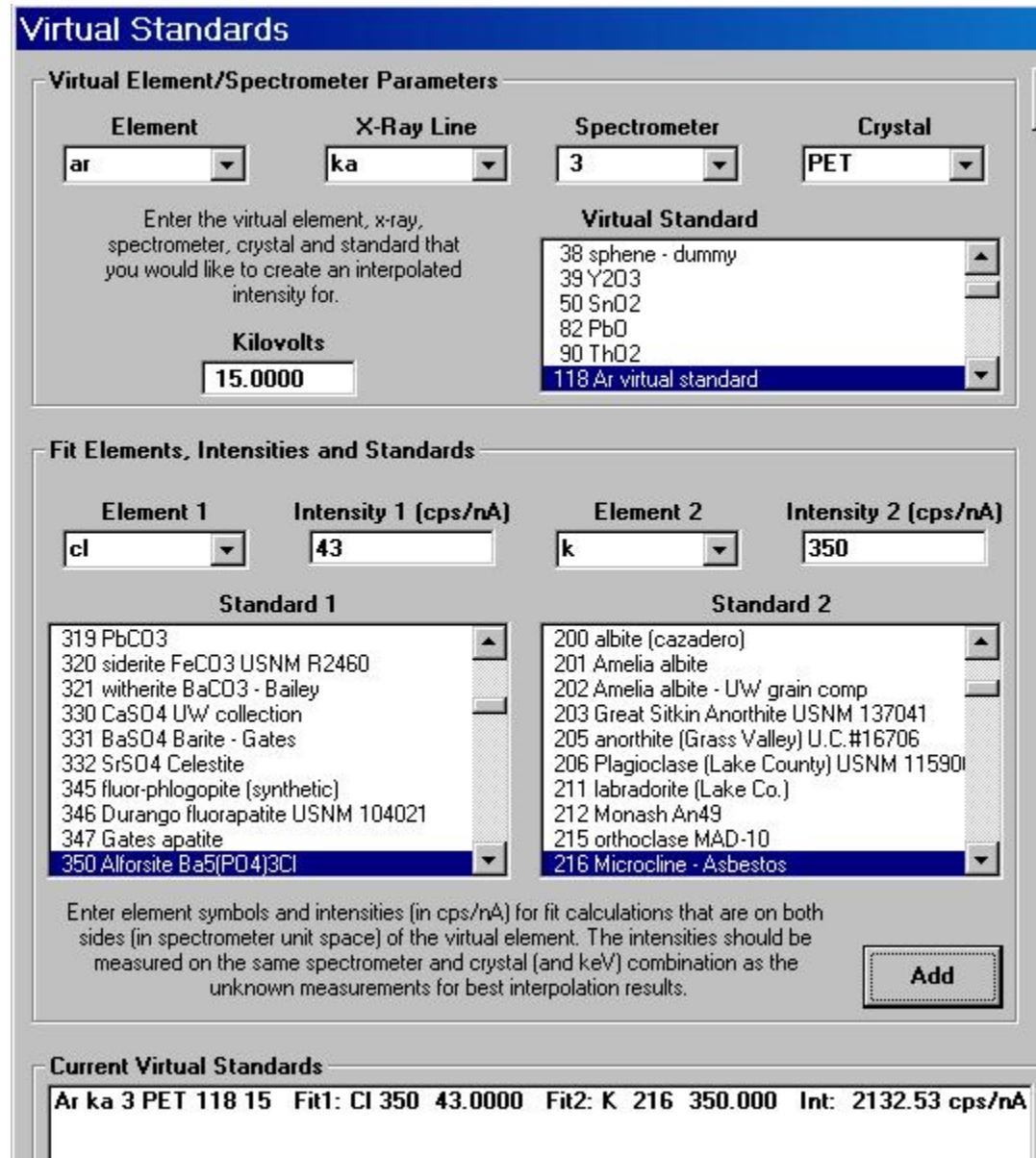
## Create Virtual Standard Intensity

A new option is the use of "virtual" standard intensities. These are calculated intensities based on interpolation and matrix corrected similar (atomic numbers) elements measured on the same crystal, spectrometer and voltage as the unknowns. This may be useful in cases where a physical standard is not available (e.g., Ar).

To calculate a virtual standard intensity, first decide on the element and create a "virtual" standard composition using the Standard database application. This will be a composition that does not physically exist in the microprobe laboratory (e.g., pure iodine). Then decide what x-ray line, spectrometer, crystal and kilovolts to use for the unknown measurement. These will be used for the actual measurements on the unknown (usually trace concentrations). If it is not possible to peak the spectrometer properly without a major concentration of the element in a standard, the use of a multiple peaks calibration procedure is suggested (see program StartWin for details) for getting the spectrometer position calibrated. Set the PHA parameters as for x-ray lines of similar energy.

Enter these parameters into the Virtual Element/Spectrometer Parameters frame. Then using the same x-ray line, spectrometer, crystal, and kilovolts, measure an available element on either side (in atomic number space) of the element that is used for the virtual standard intensity. For example if creating a virtual Ar intensity, it might be useful to measure Cl and K since they are close to Ar.

Enter the chosen fit elements, intensities and standards in the Fit Elements, Intensities and Standards dialog and click the Add button. The program will calculate the virtual intensity and store it in a table in the database file. To utilize this intensity for standardizing an unknown, go to the Analyze window and click the Standard Assignments button, click the virtual element and select the virtual standard from the standard list and check the Use Virtual Standard for Virtual Intensity Calculation checkbox.



## Update Deadtime Constants

This dialog allows the user to easily make changes to the deadtime constants for groups of samples. This feature allows the user to change the deadtime correction based on new or updated deadtime calibration measurements of the instrument.

## Student's "t" Table

This menu item provides a listing for typical confidence levels of Student's "t" values. William Gossett (1876-1937) worked for the Guinness Brewing Company on quality control issues. He developed a number of statistical tools for

situations where it was not always feasible to measure a large enough number of replicate samples. Student's "t" or test table is one of these. He was not allowed to use his own name so he published them under the name "Student". These values are used in many statistical calculations to this day. See Calculate Detection Limits and Homogeneity above for an example of this parameter in use.

The following is the printout from the Student's "t" table menu, where # pts is the number of data points, d.f. is the "degrees of freedom", 0.60 to 0.99 is the probability confidence level.:

| # pts | d.f. | 0.60   | 0.80   | 0.90   | 0.95    | 0.99    |
|-------|------|--------|--------|--------|---------|---------|
| 2     | 1    | 1.3764 | 3.0777 | 6.3138 | 12.7062 | 63.6567 |
| 3     | 2    | 1.0607 | 1.8856 | 2.9200 | 4.3027  | 9.9248  |
| 4     | 3    | .9785  | 1.6377 | 2.3534 | 3.1824  | 5.8409  |
| 5     | 4    | .9410  | 1.5332 | 2.1318 | 2.7764  | 4.6041  |
| 6     | 5    | .9195  | 1.4759 | 2.0151 | 2.5706  | 4.0321  |
| 7     | 6    | .9057  | 1.4398 | 1.9432 | 2.4469  | 3.7074  |
| 8     | 7    | .8961  | 1.4149 | 1.8946 | 2.3646  | 3.4995  |
| 9     | 8    | .8889  | 1.3968 | 1.8595 | 2.3061  | 3.3554  |
| 10    | 9    | .8834  | 1.3831 | 1.8331 | 2.2622  | 3.2498  |
| 11    | 10   | .8791  | 1.3722 | 1.8125 | 2.2281  | 3.1693  |
| 12    | 11   | .8755  | 1.3634 | 1.7959 | 2.2010  | 3.1058  |
| 13    | 12   | .8726  | 1.3562 | 1.7823 | 2.1788  | 3.0545  |
| 14    | 13   | .8702  | 1.3502 | 1.7709 | 2.1604  | 3.0123  |
| 15    | 14   | .8681  | 1.3451 | 1.7613 | 2.1448  | 2.9768  |
| 16    | 15   | .8662  | 1.3406 | 1.7531 | 2.1314  | 2.9467  |
| 17    | 16   | .8647  | 1.3368 | 1.7459 | 2.1199  | 2.9208  |
| 18    | 17   | .8633  | 1.3334 | 1.7396 | 2.1098  | 2.8982  |
| 19    | 18   | .8621  | 1.3304 | 1.7341 | 2.1009  | 2.8784  |
| 20    | 19   | .8610  | 1.3277 | 1.7291 | 2.0931  | 2.8609  |
| 21    | 20   | .8600  | 1.3253 | 1.7247 | 2.0860  | 2.8453  |
| 22    | 21   | .8591  | 1.3232 | 1.7207 | 2.0796  | 2.8314  |
| 23    | 22   | .8583  | 1.3212 | 1.7171 | 2.0739  | 2.8188  |
| 24    | 23   | .8575  | 1.3195 | 1.7139 | 2.0687  | 2.8073  |
| 25    | 24   | .8569  | 1.3178 | 1.7109 | 2.0639  | 2.7969  |
| 26    | 25   | .8562  | 1.3163 | 1.7081 | 2.0595  | 2.7874  |
| 27    | 26   | .8557  | 1.3150 | 1.7056 | 2.0555  | 2.7787  |
| 28    | 27   | .8551  | 1.3137 | 1.7033 | 2.0518  | 2.7707  |
| 29    | 28   | .8546  | 1.3125 | 1.7011 | 2.0484  | 2.7633  |
| 30    | 29   | .8542  | 1.3114 | 1.6991 | 2.0452  | 2.7564  |
| 31    | 30   | .8538  | 1.3104 | 1.6973 | 2.0423  | 2.7500  |
| 32    | 31   | .8534  | 1.3095 | 1.6955 | 2.0395  | 2.7441  |
| 33    | 32   | .8530  | 1.3086 | 1.6939 | 2.0369  | 2.7385  |
| 34    | 33   | .8526  | 1.3077 | 1.6924 | 2.0345  | 2.7333  |
| 35    | 34   | .8523  | 1.3069 | 1.6909 | 2.0322  | 2.7284  |
| 36    | 35   | .8521  | 1.3062 | 1.6896 | 2.0301  | 2.7238  |
| 37    | 36   | .8517  | 1.3055 | 1.6883 | 2.0281  | 2.7195  |
| 38    | 37   | .8514  | 1.3049 | 1.6871 | 2.0262  | 2.7154  |
| 39    | 38   | .8512  | 1.3042 | 1.6860 | 2.0244  | 2.7116  |
| 40    | 39   | .8509  | 1.3036 | 1.6849 | 2.0227  | 2.7079  |
| 41    | 40   | .8507  | 1.3031 | 1.6839 | 2.0211  | 2.7045  |
| 42    | 41   | .8505  | 1.3025 | 1.6829 | 2.0195  | 2.7012  |
| 43    | 42   | .8503  | 1.3021 | 1.6820 | 2.0181  | 2.6981  |
| 44    | 43   | .8501  | 1.3016 | 1.6811 | 2.0167  | 2.6951  |
| 45    | 44   | .8499  | 1.3011 | 1.6802 | 2.0154  | 2.6923  |
| 46    | 45   | .8497  | 1.3007 | 1.6794 | 2.0141  | 2.6896  |
| 47    | 46   | .8495  | 1.3002 | 1.6787 | 2.0129  | 2.6871  |
| 48    | 47   | .8493  | 1.2998 | 1.6779 | 2.0117  | 2.6846  |
| 49    | 48   | .8492  | 1.2994 | 1.6772 | 2.0106  | 2.6822  |

50        49        .8491    1.2991    1.6766    2.0096    2.6800

## **CalcZAF Calculations**

This menu runs the CalcZAF program asynchronously (as a separate process) for calculating arbitrary compositional k-ratios and modeling detection limits and x-ray and electron ranges.

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## **Window**

This menu lists a number of general utility functions for moving the stage and setting the column conditions

### **Stage**

This menu opens the Stage Map window which allow the user to move the stage using a variety of graphical images or drawings. See the [standard] section of the PROBEWIN.INI file for details on configuring this feature.

### **Move**

This menu opens the Move window which allows the user to move the stage to specific coordinates or set the spectrometer positions to specific element emission lines.

### **Ratemeter**

This menu displays the spectrometer digital rate meter which is useful for tuning the spectrometers manually. The red mark shows the highest intensity value attained.

### **Kiosk Display Mode**

This menu will display a modeless dialog that provides a large easily readable realtime display window that can be used to monitor the instrument activity from a distance.

### **Analytical Conditions**

This menu opens the Analytical Conditions dialog which allows the user to specify either analytical conditions or column conditions. Analytical conditions utilize the current column settings and only adjust the keV, beam current and beam size along with the beam mode (spot or scan). Column conditions utilize all column parameters including gun shift and tilt, etc.

### **Load Column Conditions From File**

This menu allows the user to select a previously saved column condition file and load those conditions to the instrument column. All column parameters such as gun shift, tilt, bias, and filament saturation (for W filaments) are restored.

### **Save Column Conditions To File**

This menu allows the user to save the current column conditions to a file on disk for later use. It saves all column parameters that are capable of being saved.

### **View Column Conditions File**

This menu allows the user to view the selected column condition file since the column conditions are stored as ASCII text values.

## **Filament Standby On**

This menu allows the user to set the filament to a standby mode (not available for FEG columns). In other words set the filament heat to the standby heat value (150 for Cameca SX100/SXFive and 90 for JEOL 8900/8200/8500/8x30)

## **Filament Standby Off**

This menu allows the user to turn off the filament standby mode (not available for FEG columns). In other words restore the filament heat to the last saturation value.

## **Turn Detector Bias Voltages Off**

This menu turns the WDS detector voltages to zero (useful for changing the P-10 gas bottle).

## **Turn Detector Bias Voltages On**

This menu restores the WDS detector voltages back to their previous values. One can also simply restart the software and the values will be automatically restored to the default values.

## **Reset Current Window Positions To Default**

This menu allows the current user to delete the current window position (and size) information saved for this user and restore the default window positions (usually screen centered). This may be useful if the user has copied the WINDOW.INI configuration file to another computer with a smaller screen resolution and some of the windows are no longer visible.

This operation only affects the current user information. Note that the current user name is the user's name given when opening a new probe database. However, if the user has not opened a probe database then the user name will be a generic name for that application (e.g., Probewin, Stage, Startwin, etc.).

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## **Run**

This menu will output a variety of run information in summary format to the log window. This information can then be cut and pasted to another application or simply selected and printed using the File | Print Log menu.

## **List Run Summary**

This menu will output a summary of all major run and sample parameters (based on the current sample) to the log window. This summary may be worth printing out for record keeping purposes.

## **List Rows, Names, Conditions**

This menu will output a summary of all standard, unknown and wavescan samples to the log window. Note that the first column of numbers listed here are the sample row numbers of the samples, which is also used as a pointer for saving and loading sample setups within the run.

This is followed by the sample names and sample acquisition conditions (beam mode, beam current, beam diameter).

## **List Anomalous Intensity Data for Standards or Unknowns**

List all data lines that contain at least one element intensity that is deviates from the mean intensity by more than 4 sigma (based on the predicted variance assuming counting statistics only). The user may click "Yes" to list unknown sample intensities or "No" to list standard intensities.

This feature is useful for finding data points which are statistically significantly different from the other data points in the sample due to holes, cracks, epoxy, etc.

Note that this feature can be used with the Analyze! window to list the intensities without output to the log window by checking the new “Do Not Output To Log Window” checkbox.

## **List Sample Calculation Options**

This menu will output a summary of all sample calculation options. This menu is useful to determine if all sample calculation options are appropriately assigned to the proper samples without having to perform analyses on all samples.

## **List Standard Counts**

This menu will output a summary of all standard counts in the order acquired to the log window. This includes all standards, whether or not they are actually assigned as a standard for primary, interference or MAN calibrations. The date and average time of acquisition is also listed in the output.

To see a listing of the standard intensities that are applicable to a specific quantitative sample, see the List Standard intensities in the Analyze! window.

## **List Standard Compositions**

This menu item will list the compositions of all standards in the current run based on the current values in the standard composition database.

## **List Fiducial Coordinate Sets**

This menu will list all fiducial coordinates sets stored in the current run.

## **List Current MACs**

This menu item will list the currently loaded MACs (mass absorption coefficients) from the last quantitative analysis. Note that any empirical MACs listed from this menu will be used to overwrite table based MACs automatically during calculations.

## **List Current APFs**

This menu item will list the currently loaded APFs (area peak factors) from the last quantitative analysis.

## **List Current Alpha Factors**

This menu will list the current alpha factor values and indicate any empirical and/or Penepma generated alpha factors for the current sample.

## **Display, Fit and Export Spectrometer Peak and PHA Scans**

This menu will display the Scan Data dialog. This dialog will display all PHA, bias and gain scans performed on elements in the current run. Note that scans performed from the Move window are not saved to the probe database.



Also peak and ROM scan data are also automatically saved to the scan data tables. All scan data displayed can be exported to ASCII files.

## Display, Annotate and Export Analog Signal Images

This dialog will display images acquired using the Digitize Image (manual or automated) or Imaging (manual) windows. The image signal type, size and sample name referenced is displayed. The stage coordinates of the image extents and the min and max of the intensity data is also displayed in the Image Parameters box. In addition any data points associated with the referenced sample can be displayed along with the line numbers of the data points.



The image data can be output to a BMP file (8 bit), TIF file (16 bit) or GRD file (32 bit). The GRD file is the file format used by Golden Software's Surfer program. When this format is saved, a second file with a .DAT extension will be saved with the X, Y and line numbers for creating "post data" on the Surfer image or plot which will display the data point coordinates and an optional line number.

The image can also be printed to the default printer and copied to the clipboard for pasting into another document application. Note that the clipboard and printer output includes the graphics methods for the data points, line numbers and scale bar.

## Display and Export EDS Spectra

This menu will display all EDS spectra acquired for all point analyses. The elements by EDS (for quantification) are specified in the Elements/Cations dialog.



## Display and Export CL Spectra

This menu will display all CL spectra acquired for all point analyses.

## Display Time Dependent (TDI) and Alternating On/Off Intensities

This menu will display plots of (self calibration) Time Dependent Intensity (TDI) and alternating on/off peak intensities versus time. To acquire self-calibration intensity data click the Special Options button in the Acquire window and select the self calibration sample acquisition type.

To acquire alternating on and off-peak data click the Acquisition Option button in the Acquire! Window and check the "Use Alternating On and Off Peak" option. Then click the Count Times button in the Acquire! Window and enter Unknown Count Factors greater than one. The program will automatically acquire on and off-peak data based on the count time and the unknown count factor. This is useful for trace element acquisition when it is desirable to know if the on and off-peak ratios are changing over time.

Note that the extrapolated x-ray intensity and percent change calculated and displayed in this dialog is based on the Y intercept. This is not the method used for the actual Time Dependent Intensity (TDI) element correction. Instead the Time Dependent Intensity (TDI) element correction utilizes a calculation using the log slope of the fitted data.

## Display Integrated Intensities

This menu will display plots of the standard and unknown integrated intensity acquisitions (see check box in the Elements/Cations dialog).

This data is then fitted to a cubic spline peak fit and the integrated intensity is calculated and subtracted from the fitted background and used for quantitative calculations where the peak shape and/or position changes due to changes valance bond or coordination. This is particularly true for low energy lines involving the outer x-ray shells.

All off-peak options may be used with the integrated intensity option. The reported integrated intensities are in units of counts per second times spectrometer units divided by the spectrometer unit range (cps per second per spectrometer unit).

If an element is acquired using the integrated intensity option, the standard intensities for that element must also be acquired using the integrated option and preferably using the same off-peak range.

This acquisition method is very slow and usually the APF method is preferred unless APF values are not available for the analyzed elements.

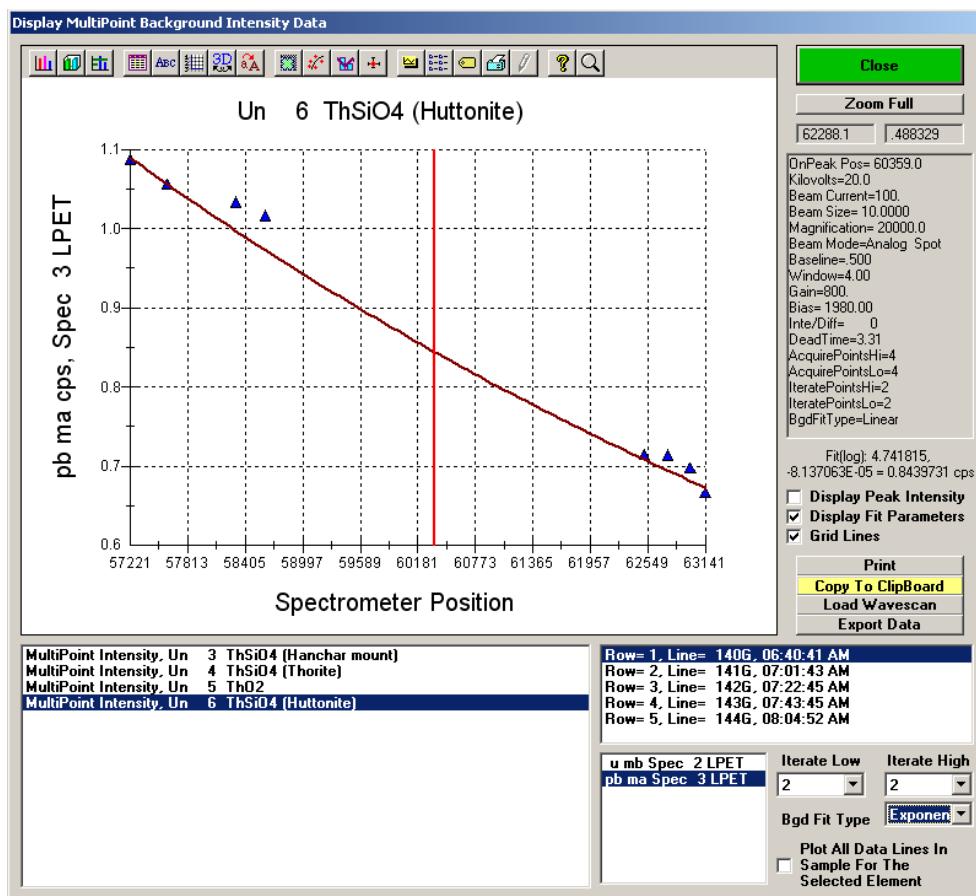
## Display Calibration Curve Intensities

This menu will display the calibration curve intensities for all standards for each selected unknown or standard sample and the interpolated unknown counts and concentrations for that sample. This is a multi-standard XRF type calibration correction and is extremely useful for certain types of difficult analyses provided that one has standards with appropriate concentrations in a matrix similar to the unknown to be measured. For example carbon in steel alloys.

The ZAF, Phi-Rho-Z, Alpha Factor and Calibration Curve Selections option for Calibration Curve must be selected from the Analytical menu first. Also at least two standards per element are required although only one standard is required if the Use Zero Point For Calibration Curve option is selected in the Analysis Options dialog available from the Analytical menu.

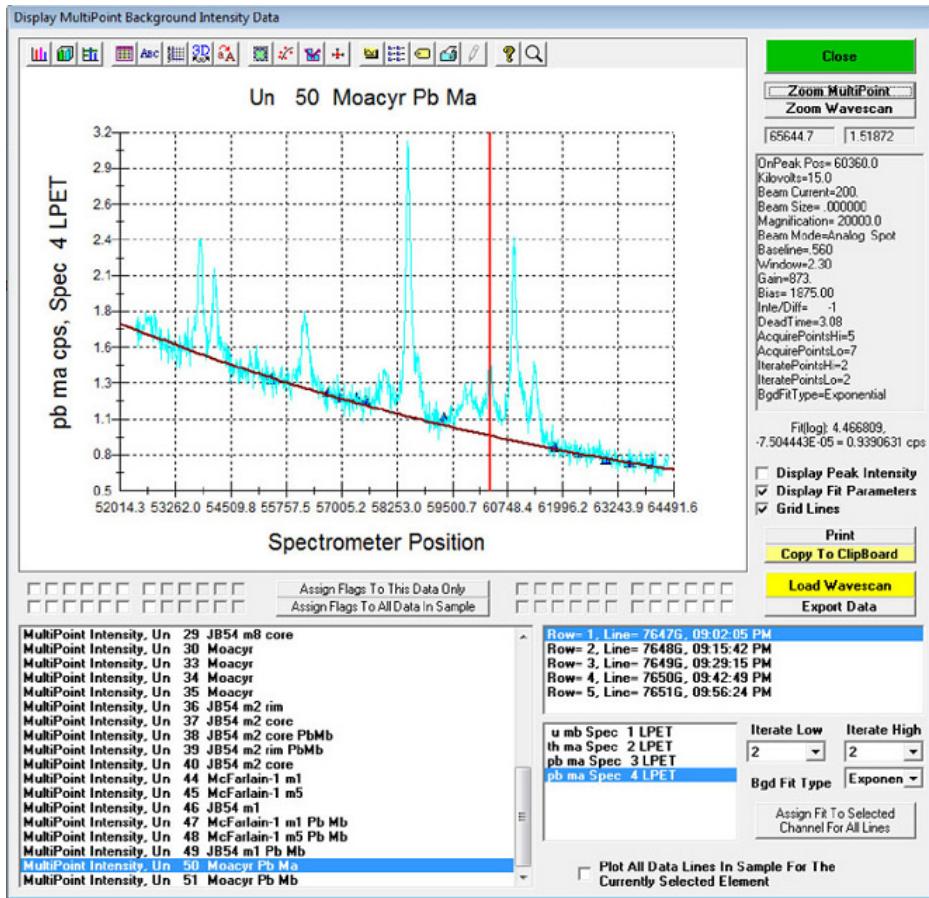
## Display Multi-Point Background Intensities

This dialog will allow the user to display previously acquired multi-point background intensities and calculate the best iterated fit based on the number of points to iterate to (see source code in Acquire! window documentation). The fit line and the fit coefficients are displayed.



### Load Wavescan

Use this button to load a wavescan for overlay on the multi-point backgrounds. The wavescan should have been acquired on a sample with a similar atomic number so the continuum background count rate is comparable to the multi-point sample acquisition as shown below:



## Display PictureSnap

This menu opens the PictureSnap! Window which allows the user to open any scanned image, for example, from a flatbed or slide scanner, and calibrate the image to the stage coordinates of the microprobe stage.

After the image is calibrated using two diagonal positions on the image for which the stage coordinates can be located, the image will display the current stage coordinate as a live floating cursor. One can also double-click any image position and the stage will move to that position. Based on tests using a 4000 dpi slide scanner, one can calibrate the image so that individual grains can be located to within 5 um depending on the accuracy with which the image is calibrated.

In addition the software allows the user to display currently digitized points by clicking the Display menu items (Real Time mode only) or to display previously analyzed positions (on-line and off-line mode). Use the menus in the PictureSnap window to display currently digitized position samples and use the Run menu items from the log window to display previously analyzed positions within the currently open probe database.

The displayed image plus the plotted stage positions (and floating scale bar) can be printed using the File | Print menu. To obtain a digital image use the File | Print Setup menu to select the Adobe Acrobat PDF printer. One can also use the Copy To Clipboard menus, though method 1 does not copy the plotted stage positions, while method 2 does, but not for the entire image area, just the portion visible in the scrollable image window.

Note that a live cursor is displayed in the window caption bar. It displays pixel (twip) coordinates in uncalibrated images and stage coordinates in calibrated images. Image calibrations are automatically saved to a .ACQ file which is automatically reloaded if the image is opened subsequently (on or off line).



Note the Save As GRD File menu under the File menu which allows the user to export the image to a Surfer GRD file format along with any positions (and position sample or line numbers) that are currently displayed, to a .DAT file for using as a “post data” file in Surfer. Creation of publication quality images showing exact analysis positions is easy with this feature.

## Display Acquired Standard Positions on PictureSnap

Click this menu item to display any previously analyzed standard positions on the calibrated image. Use the Display Line Labels on PictureSnap to show the line number labels.

## Display Acquired Unknown Positions on PictureSnap

Click this menu item to display any previously analyzed unknown positions on the calibrated image. Use the Display Line Labels on PictureSnap to show the line number labels.

## Display Acquired Wavescan Positions on PictureSnap

Click this menu item to display any previously analyzed wavescan positions on the calibrated image. Use the Display Line Labels on PictureSnap to show the line number labels.

## Display Line Labels on PictureSnap

Use this menu to show line numbers of the analyzed positions on the image.

## **Display Long Line Labels On PictureSnap**

Display analysis line numbers with each analysis position using the format ,sample number>-<line number>.

## **Display Short Line Labels On PictureSnap**

Display the analysis line numbers with each analysis position using only the analysis line number (no preceding sample number)

## **Skip Disabled Points On PictureSnap**

Checkable menu to display or not display disabled analyses. The default is to display disabled points.

## **Skip Duplicate Points on PictureSnap**

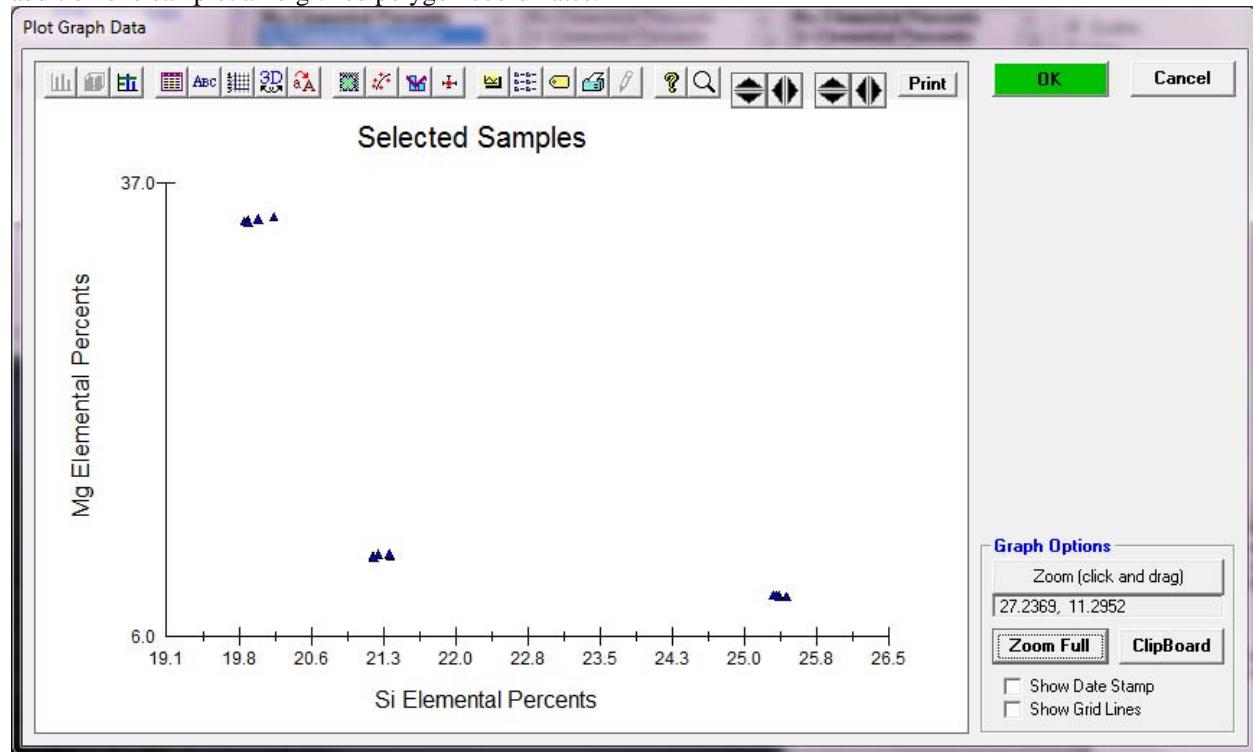
Checkable menu to display or not display analysis points that have identical stage positions. The default is to display points with duplicate stage positions.

---

# **Output**

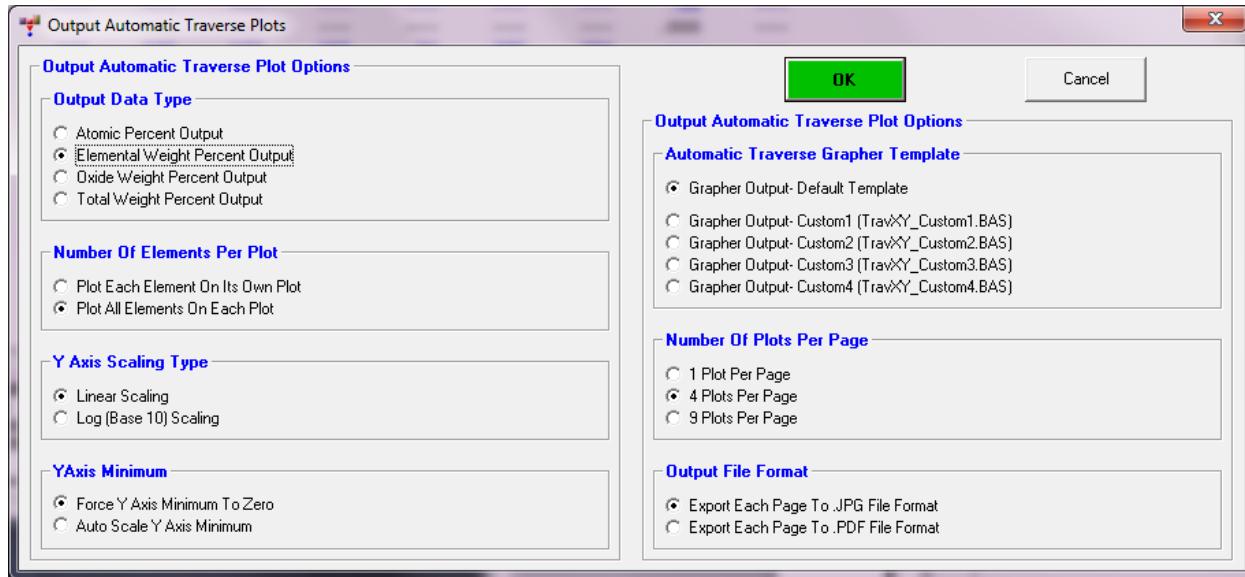
## **Output Standard and Unknown Plots**

This menu will allow the user to plot (or output) intensity and quantitative results for standard and unknown samples. In addition one can plot all digitized polygon coordinates.



## **Output Automatic Traverse Plots**

Use this menu to create automatic traverse plots for all samples.



## Output Automatic Ternary Plots

Use this menu to create automatic ternary plots for all samples.

## Save User Specified Format Output

This menu will allow the user to output specifically selected data types for the current run. These selections are automatically saved to the current probe run. The defaults for this dialog may be specified in the PROBEWIN.INI file in the [software] section. See Configuration Files section for more information.

The data types that may be saved are:

|             |   |
|-------------|---|
| SAMPLE      | Sample name (in double quotes)                                |
| NUMBER      | Sample number   |
| TO          | Analytical conditions (Takeoff)                               |
| KEV         | Analytical conditions (Kilovolts)                             |
| NA          | Analytical conditions (Beam current in nA)                    |
| UM          | Analytical conditions (Beam size in microns)                  |
| LINE        | Line number   |
| El WT%      | Elemental weight percent                                      |
| El (O2)     | Oxide weight percent  |
| El AT%      | Atomic weight percent (elemental mole percent)                |
| El OXMOL%   | Oxide mole percents   |
| TOTAL       | Total weight percent  |
| FORMULA     | Formula basis element (or none for sum of cations)            |
| BASIS       | Formula basis element number (or total cations)               |
| El FORMULA  | Formula basis calculation based on FORMULA and BASIS          |
| MINERAL     | Mineral end member basis (olivine, feldspar, etc)             |
| END-MEMBER1 | Value for end member 1  |
| END-MEMBER2 | Value for end member 2  |
| END-MEMBER3 | Value for end member 3  |
| END-MEMBER4 | Value for end member 4  |
| El CDL99    | Detection limit in elemental weight percent at 99% confidence |

|                        |  |
|------------------------|--|
| El %ERR                | Analytical error in relative percent (one sigma)   |
| X-POS                  | X stage position   |
| Y-POS                  | Y stage position   |
| Z-POS                  | Z stage position   |
| RELDIST                | Relative distance (in microns)   |
| BEAMCURR               | Beam or probe current (in nA) (array if combined conditions sample)  |
| ABSCURR                | Absorbed current (in nA) (array if combined conditions sample)   |
| BEAMCURR2              | Beam or probe current (in nA) (array if combined conditions sample)  |
| ABSCURR2               | Absorbed current (in nA) (array if combined conditions sample)   |
| El ONTIM               | On-peak counting time (includes x-ray line, spectro number and crystal)                                      |
| El HITIM               | High off-peak counting time (x-ray, spectro and crystal)   |
| El LOTIM               | Low off-peak counting time (x-ray, spectro and crystal)  |
| El ONCNT               | Raw On-peak count intensity (cps/nominal beam) (x-ray, spectro and crystal)                                  |
| El OFCNT               | Calculated Off-peak count intensity (cps/nominal beam) (x-ray, spectro and crystal)                          |
| El NECNT               | Net (off-peak, interference, etc. corrected) count intensity (cps/nominal beam) (x-ray, spectro and crystal) |
| El KRAT                | Elemental k-ratio (normalized to pure element) (x-ray, spectro and crystal)                                  |
| DATE-TIME              | Acquisition date and time (Excel format)   |
| Z-COR                  | Atomic number correction factor  |
| A-COR                  | Absorption correction factor   |
| F-COR                  | Fluorescence correction factor   |
| ZAF                    | Z*A*F correction factor  |
| MAC                    | Elemental Mass Absorption Coefficient  |
| El K-RAW               | Elemental raw k-ratio (unk counts/standard counts)   |
| STD_NUM                | Primary standard assignment number   |
| STD_NAM                | Primary standard assignment name   |
| TOTAL-WT%              | Total weight percent   |
| TOTAL-OXYGEN           | Total oxygen (excess plus calculated)  |
| TOTAL-CATIONS          | Total cations  |
| OXYGEN(Calc)           | The calculated oxygen (from stoichiometry)   |
| OXYGEN(Excess)         | The excess oxygen (specified or measured)  |
| Z-BAR                  | The average atomic number (weight fraction normalized)   |
| AVG ATWT               | The average atomic weight  |
| OXYGEN(Halogen Equiv.) | The oxygen equivalent of all halogens  |
| OXYGEN(Halogen Corr.)  | The halogen corrected stoichiometric oxygen  |
| CHARGE                 | The calculated total charge balance for all cations and anions   |
| FE-CHARGE              | The avenge calculated charge balance for Fe  |
| INTF%                  | Interference correction percent (relative)   |
| MANABSCOR%             | MAN continuum absorption correction percent (relative)   |
| APF                    | Area peak factor corrections (1.0 = none)  |

|          |   |
|----------|---|
| TDI_COR% | Time dependent intensity correction percents (relative)   |
| TDI_DEV% | Time dependent intensity correction fit average deviation |

## Save Custom Analysis Format #1 (C.G.S.)

This menu will automatically create a custom file format (C.G.S. format). This format is a fixed length field, comma delimited ASCII file that contains unknown weight percent data only. The data will be output to a different file based on the analytical setup used to create the sample (if one was used). The file naming convention is based on the file setup name used to create the sample. For example, samples created using a setup file called FELDSPAR.MDB, will be output to FELDSPAR.DAT, and so on.

The first column contains an eight character wide field containing the sample name for each data point. The final column contains the totals. All elements, analyzed and specified will be output. The column labels are not enclosed in double quotes but the sample names are. The oxide or elemental basis for calculating the data is based on the first sample of a particular setup file to ensure that the data type is consistent within a single output file.

This format is intended for use with the Canadian Geological Survey MREPORT mineral recalculation program set which has been published as an open file report. An short example is shown here:

```

SAMPLE      ,      BI,      SB,      TOTAL
"mabisb06", 99.7657, .207527, 99.9733
"mabisb06", 99.4798, .185703, 99.6655
"mabisb06", 99.1180, .298709, 99.4167
"mabisb75", 99.4394, .978249, 100.418
"mabisb75", 98.2293, 1.69337, 99.9227
"mkbisb06", 98.0328, 1.69929, 99.7321
"mkbisb06", 97.5753, 1.45358, 99.0289
"mkbisb06", 98.8127, 1.78520, 100.598
"msbisb25", 89.4619, 10.3681, 99.8300
"msbisb25", 99.6894, 1.03415, 100.724
"msbisb25", 82.0337, 18.5264, 100.560
"msbisb25", 80.8562, 17.6338, 98.4900
"msbisb25", 85.7570, 13.9368, 99.6939
"mabisb06", 99.4645, .463434, 99.9279
"mabisb06", 99.2842, .682973, 99.9671
"mabisb06", 99.4594, .567973, 100.027
"mabisb25", 26.0476, 73.0580, 99.1056
"msbisb25", 98.9069, .576286, 99.4832
"msbisb25", 98.9150, .624202, 99.5392
"msbisb25", 99.4395, .368290, 99.8078
"msbisb25", 94.9391, 4.22091, 99.1600
"mabisb75", 98.6824, .741002, 99.4234
"mabisb75", 100.201, .230280, 100.431
"mabisb75", 99.5045, .514928, 100.019
"mabisb75", 99.3453, .749748, 100.095
"mabisb75", 98.2343, .853120, 99.0874

```

## Save Custom Analysis Format #2 (H.T.)

This menu will automatically create a custom file format (H.T. format). This format is a tab delimited ASCII file and contains extensive information including elemental, oxide, atomic weight percents, raw, background and net counts, k-ratios and one sigma calculations for several data types, stage positions, count times, and ZAF correction factors.

A separate output file is created for each unknown based on each position sample. In addition, an option is available to have each output file automatically imported into an excel spreadsheet. This file format was specified by Haldor Topsoe Ltd. and cannot be modified for other applications.

A short explanation of the various column labels used for this custom output format is given here, but note that in reality, the symbol (El) will be replaced by the actual element symbol:

|          |   |
|----------|---|
| LINE     | Line number   |
| El AT%   | Atomic weight percent   |
| El WT%   | Elemental weight percent  |
| Ox WT%   | Oxide weight percent  |
| TOTAL    | Total weight percent  |
| El OX/CA | Oxide to cation ratio   |
| El ONTIM | On-peak counting time   |
| El HITIM | High off-peak counting time                                     |
| El LOTIM | Low off-peak counting time                                      |
| El DETIM | Deadtime correction factor                                      |
| X-POS    | X stage position  |
| Y-POS    | Y stage position  |
| Z-POS    | Z stage position  |
| RELDIST  | Relative distance (in microns)                                  |
| BEAMCUR  | Beam current  |
| El ONCNT | On-peak count intensity (cps/nominal beam)                      |
| El OFCNT | Off-peak count intensity (cps/nominal beam)                     |
| El NECNT | Net (off-peak corrected) count intensity (cps/nominal beam)     |
| El +-CNT | One sigma standard deviation count intensity (cps/nominal beam) |
| El KRAT  | Raw k-ratio   |
| El +-RAT | One sigma standard deviation raw k-ratio                        |
| El Z-COR | Atomic number correction factor                                 |
| El A-COR | Absorption correction factor                                    |
| El F-COR | Fluorescence correction factor                                  |
| El ZAF   | Z*A*F correction factor   |

The elemental raw k-ratios and x-ray intensities are corrected for deadtime, beam current and normalized to cps in addition to background, Time Dependent Intensity (TDI), area peak factors and spectral interference corrections.

### Save Custom Analysis Format #3 (J.H.)

This menu will automatically create a custom file format (J.H. format). This format is a tab delimited ASCII file and contains extensive information including elemental, oxide, atomic weight percents, detection limits, analytical error (percent error), raw k-ratios, raw and corrected x-ray intensities, background and net counts, count times, deadtimes, beam current, stage positions, relative distance (in microns) and standard counts.

This file format was specified by John Hanchar at George Washington University and cannot be modified for other applications. A short explanation of the various column labels used for this custom output format is given here, but note that in reality, the symbol (El) will be replaced by the actual element symbol:

|          |   |
|----------|---|
| LINE     | Line number   |
| El AT%   | Atomic weight percent   |
| El WT%   | Elemental weight percent                                      |
| El (O2)  | Oxide weight percent  |
| TOTAL    | Total weight percent  |
| El CDL99 | Detection limit in elemental weight percent at 99% confidence |
| El %ERR  | Analytical error in relative percent (one sigma)              |
| El K-RAW | Elemental raw k-ratio (unk counts/standard counts)            |
| El ONCNT | On-peak count intensity (cps/nominal beam)                    |
| El OFCNT | Off-peak count intensity (cps/nominal beam)                   |
| El NECNT | Net (off-peak corrected) count intensity (cps/nominal beam)   |
| El ONTIM | On-peak counting time   |
| El HITIM | High off-peak counting time                                   |
| El LOTIM | Low off-peak counting time                                    |

|           |                                      |
|-----------|--------------------------------------|
| El DETIM  | Deadtime correction factor (in secs) |
| BEAMCUR   | Beam current (in nA)                 |
| X-POS     | X stage position                     |
| Y-POS     | Y stage position                     |
| Z-POS     | Z stage position                     |
| RELDIST   | Relative distance (in microns)       |
| El STDCTS | Standard counts (cps/nominal beam)   |

The elemental raw k-ratios and x-ray intensities are corrected for deadtime, beam current and normalized to cps in addition to background, Time Dependent Intensity (TDI), area peak factors and spectral interference corrections.

### **Save Custom Analysis Format #4 (J.J.D.)**

This menu (J.J.D. format) will create an output file of standard or unknown average weight, oxide and atomic percents along with average detection and analytical sensitivity for each data point. The user can select averages only, standard deviations only, or averages and standard deviations (x.xxx +/- x.xxx) output.

|          |   |
|----------|---|
| SAMPLE   | Sample number and name (in double quotes)                           |
| El AT%   | Atomic percent  |
| El WT%   | Elemental weight percent  |
| Ox WT%   | Oxide weight percent  |
| TOTAL    | Total weight percent  |
| El CDL60 | Elemental t-test detection limit (60 percent confidence)            |
| El CDL80 | Elemental t-test detection limit (80 percent confidence)            |
| El CDL90 | Elemental t-test detection limit (90 percent confidence)            |
| El CDL95 | Elemental t-test detection limit (95 percent confidence)            |
| El CDL99 | Elemental t-test detection limit (99 percent confidence)            |
| El CAS60 | Elemental t-test analytical sensitivity (60% CI for major elements) |
| El CAS80 | Elemental t-test analytical sensitivity (80% CI for major elements) |
| El CAS90 | Elemental t-test analytical sensitivity (90% CI for major elements) |
| El CAS95 | Elemental t-test analytical sensitivity (95% CI for major elements) |
| El CAS99 | Elemental t-test analytical sensitivity (99% CI for major elements) |

This menu is useful for creating tables for publication. Note that values output as x.xxx +/- x.xxx will be interpreted by most programs as a string rather than a numerical value.

### **Save Custom Analysis Format #5 (J.J.D.-2)**

This menu (J.J.D.-2 format) will create an output file of standard or unknown x, y, z coordinates and relative distance in microns, elemental weight percents, along with single point detection and analytical sensitivity and raw k-ratios. Each sample in the data file will automatically be output to an ASCII file (\*.DAT extension) with a unique filename based on the probe database file name and the sample type, sample set and name. The directory will be the same one that the database file resides in.

|         |   |
|---------|---|
| LINE    | Data file line number   |
| X-POS   | X stage position  |
| Y-POS   | Y stage position  |
| Z-POS   | Z stage position  |
| RELDIST | Relative stage distance in microns  |
| WT%     | Elemental weight percent  |
| TOTAL   | Total weight percent  |
| CDL99   | Single line detection limit (99% confidence) in weight percent            |
| %ERR    | Relative percent error (one sigma)  |
| K-RAW   | Raw k-ratio (corrected) (includes x-ray line, spectro number and crystal) |

This format is especially useful when it is desired to plot multiple (or duplicate) datasets in a graph with similar or separate axes for each dataset. The elemental raw k-ratios and x-ray intensities are corrected for deadtime, beam current and normalized to cps in addition to background, Time Dependent Intensity (TDI), area peak factors and spectral interference corrections.

## Save Custom Analysis Format #6 (H.W.)

This menu (H.W. format) will create an output file of standard or unknown atomic, elemental and oxide weight percents, along with single point detection and analytical sensitivity and raw k-ratios, count times, beam current and x, y, z coordinates and relative distance in microns.

|          |  |
|----------|--|
|          | Sample description text (no column label)  |
| LINE     | Line number  |
| El AT%   | Atomic percent   |
| El WT%   | Elemental weight percent   |
| El (O2)  | Oxide weight percent   |
| TOTAL    | Total weight percent   |
| El CDL99 | Detection limit in elemental weight percent at 99% confidence  |
| El %ERR  | Analytical error in relative percent (one sigma)   |
| El K-RAW | Elemental raw k-ratio (unk counts/standard counts) (includes x-ray line, spectro number and crystal) |
| El ONTIM | On-peak counting time(includes x-ray line, spectro number and crystal)                               |
| El HITIM | High off-peak counting time(includes x-ray line, spectro number and crystal)                         |
| El LOTIM | Low off-peak counting time(includes x-ray line, spectro number and crystal)                          |
| BEAMCUR  | Beam current (in nA)   |
| X-POS    | X stage position   |
| Y-POS    | Y stage position   |
| Z-POS    | Z stage position   |
| RELDIST  | Relative distance (in microns)   |

This file format was specified by Heather Wright at the University of Oregon and cannot be modified for other applications. In addition the first column of tab delimited data will contain any sample description text as separate strings enclosed in double quotes. **This is the only output format in PROBE that will export the sample description field text.**

## Save Custom Analysis Format #7 (NIST)

This menu (NIST format) will create an output file of raw (uncorrected, un-normalized) intensities for standard or unknown samples and count times, deadtimes, beam current and x, y, z coordinates and relative distance in microns, raw k-ratios and corrected unknown and standard intensities. These last three items are corrected for deadtime, beam current and normalized to cps in addition to background, Time Dependent Intensity (TDI), area peak factors and spectral interference corrections.

Note that the x-ray intensities are output in total raw counts at the actual beam current of acquisition.

This file format was specified by The National Institute of Standards and Technology and cannot be modified for other applications.

|          |   |
|----------|---|
| LINE     | Data file line number                       |
| RAWONCNT | Raw on-peak intensity (no correction)       |
| RAWHICNT | Raw high off peak intensity (no correction) |
| RAWLOCNT | Raw low off peak intensity (no correction)  |
| RAWOFCNT | Raw background intensity (no correction)    |

|         |  |
|---------|--|
| ONTIM   | Actual on peak count time                    |
| HITIM   | Actual high off peak count time              |
| LOTIM   | Actual low off peak count time               |
| DETIM   | Deadtime in micro-seconds                    |
| BEAMCUR | Faraday or beam current reading in nano-amps |
| X-POS   | Stage x axis position                        |
| Y-POS   | Stage x axis position                        |
| Z-POS   | Stage x axis position                        |
| RELDIST | Stage distance in relative microns           |
| K-RAW   | Raw k-ratio (corrected)                      |
| UNCNT   | Unknown count intensity (corrected)          |
| STCNT   | Standard count intensity (corrected)         |

Note that RELDIST is the accumulated number of microns between each data point with the first point of each sample reset to zero.

### Save Custom Analysis Format #8 (MAN)

This output format (MAN or mean Atomic Number format) will allow the user to output the on-peak (not background corrected) intensities for the acquired standards and their "ideal" average atomic numbers from the STANDARD.MDB file. The ZAF factors are also output to the file along with the absorption corrected intensities. All points or just the averages may be output to the file.

The file format is as shown below:

|         |  |
|---------|--|
| LINE    | Data file line number                            |
| SNUM    | Sample number (standard number)                  |
| SNAM    | Sample name (standard name)                      |
| ZBAR    | Sample average atomic number                     |
| RAWCNT  | Raw count intensity                              |
| CORCNT  | Corrected count intensity (absorption corrected) |
| ZEDCOR  | Atomic number correction factor                  |
| ABSCOR  | Absorption correction factor                     |
| FLUCOR  | Fluorescence correction factor                   |
| ZAFCOR  | Total ZAF correction factor                      |
| ONTIM   | On-peak (MAN) counting time                      |
| BEAM    | Beam current in nA                               |
| BEAMCUR | Beam current (combined condition samples)        |

This format is intended for use by those wishing to create their own MAN plots of atomic number versus background intensities.

### Save Custom Analysis Format #9 (P.C.)

This menu (P.C. format) will create an output file of standard or unknown atomic, elemental and oxide weight percents, along with single point detection and analytical sensitivity and raw k-ratios, count times, beam current and x, y, z coordinates and relative distance in microns.

It will also output the formula atoms (based on the sum of 8 cations if no element basis is specified) and mineral end-members if they are specified by the user in the Calculation Options dialog (from the Analyze! window).

|                                  |  |
|----------------------------------|--|
| SAMPLE                           | Sample name (in double quotes)                     |
| LINE                             | Line number  |
| E <sub>1</sub> (O <sub>2</sub> ) | Oxide weight percent                               |
| E <sub>1</sub> WT%               | Elemental weight percent                           |
| TOTAL                            | Total weight percent                               |
| DAT/TIM                          | Acquisition date and time in system defined format |

|             |  |
|-------------|--|
| X-POS       | X stage position   |
| Y-POS       | Y stage position   |
| Z-POS       | Z stage position   |
| RELDIST     | Relative distance (in microns)   |
| BEAMCUR     | Beam or probe current (in nA) (array if combined conditions sample)  |
| ABSB CUR    | Sample or absorbed current (in nA)   |
| El AT%      | Atomic percent   |
| FORMULA     | Formula basis (number of atoms)  |
| BASIS       | Formula element basis (if blank, then uses sum of 8 cations)   |
| El FORMULA  | Formula atoms  |
| MINERAL     | Mineral end-member string if specified (one of the following):<br>Olivine (Fo,Fa)<br>Feldspar (Ab,An,Or)<br>Pyroxene (Wo,En,Fs)<br>Garnet (Gro,Pyr,Alm,Sp)<br>Garnet (Gro,And,Uva) |
| END-MEMBER1 | Mineral end-member abundance (%) (blank if not specified)  |
| END-MEMBER2 | Mineral end-member abundance (%) (blank if not specified)  |
| END-MEMBER3 | Mineral end-member abundance (%) (blank if not specified)  |
| END-MEMBER4 | Mineral end-member abundance (%) (blank if not specified)  |
| El CDL99    | Detection limit in elemental weight percent at 99% confidence  |
| El %ERR     | Analytical error in relative percent (one sigma)   |
| El ONTIM    | On-peak counting time  |
| El HITIM    | High off-peak counting time  |
| El LOTIM    | Low off-peak counting time   |
| El K-RAW    | Elemental raw k-ratio (unk counts/standard counts)   |
| STD_NUM     | Primary standard assignment number   |
| STD_NAM     | Primary standard assignment name   |

This file format was specified by Paul Carpenter at the NASA Marshall Flight Center and cannot be modified for other applications. **This is the only output format in PROBE that will export the mineral end-member fields.**

### Save Wavescan Samples (based on sample names)

Use this output format to export wavescan sample data to separate tab delimited files. This menu will also prompt to send the data to an Excel spreadsheet. The stage position and relative distance columns vary only if the wavescan stage increment option was utilized.

|         |   |
|---------|---|
| LINE    | Line number   |
| X-POS   | X stage position  |
| Y-POS   | Y stage position  |
| Z-POS   | Z stage position  |
| RELDIST | Relative distance (in microns)                              |
| On-Peak | On-peak position  |
| Hi-Peak | Hi-peak position  |
| Lo-Peak | Lo-peak position  |
| SPC     | Spectrometer position in spectrometer units                 |
| ANG     | Spectrometer angstroms in angstroms                         |
| CPS     | Intensity in counts per second                              |
| NCPS    | Intensity in normalized counts per second (to highest peak) |
| COUNTS  | Intensity in raw counts                                     |
| KEV     | Spectrometer position in keV (includes spectrometer offset) |

This is the only custom output format for exporting wavescan data.

This output option will also output the KLM markers to a .KLM file for each wavescan. This file can be used in conjunction with the supplied Grapher sample data files and template files to produce customized publication quality wavescan graphs:

|  |                         |
|--|-------------------------|
| Corning VWX Full Range WDS-Wa 40 Glass 95IRW S1-30.grf | ' Grapher plot template |
| Corning VWX Full Range WDS-Wa 40 Glass 95IRW S1-30.dat | ' Wavescan data file    |
| Corning VWX Full Range WDS-Wa 40 Glass 95IRW S1-30.klm | ' KLM data file         |

The column format of the file is:

|           |   |
|-----------|---|
| LINE      | Line number   |
| On-Peak   | On-peak position  |
| Hi-Peak   | Hi-peak position  |
| Lo-Peak   | Lo-peak position  |
| Hi-Wave   | High wavescan limit   |
| Lo-Wave   | Low wavescan limit  |
| Elm-Xray  | KLM markers for the wavescan element                          |
| Elm-Ord   | KLM line orders for the wavescan element                      |
| Elm-Spec  | KLM spec positions for the wavescan element                   |
| Elm-Ang   | KLM ang positions for the wavescan element                    |
| Elm-Int   | KLM relative intensities for the wavescan element             |
| Anal-Xray | KLM markers for the "analyzed" wavescan elements              |
| Anal-Ord  | KLM orders for the "analyzed" wavescan elements               |
| Anal-Spec | KLM spec positions for the "analyzed" wavescan elements       |
| Anal-Ang  | KLM ang positions for the "analyzed" wavescan elements        |
| Anal-Int  | KLM relative intensities for the "analyzed" wavescan elements |
| All-Xray  | KLM markers for all wavescan elements                         |
| All-Ord   | KLM line orders for all wavescan elements                     |
| All-Spec  | KLM spec positions for all wavescan elements                  |
| All-Ang   | KLM ang positions for all wavescan elements                   |
| All-Int   | KLM relative intensities for all wavescan elements            |

The On, Hi, Lo, Hi-Wave and Lo-Wave columns are the same for all rows. The "Elm-" columns are the KLM lines for the element tuned on that spectrometer only (no x-ray lines may appear in these columns if no lines for that element are in the x-ray range of the scan). The "Anal-" columns are the KLM lines for the analyzed (and specified) elements for the wavescan sample only (no x-ray lines may appear in these columns if no lines for the analyzed or specified elements are in the x-ray range of the scan). The "All-" columns are the KLM lines for all elements in the x-ray range for that wavescan.

## Save Wavescan Samples (based on spectro/element)

Use this menu to output each spectrometer/element pair to a separate file (Carpenter-Goemann output format).

## Save Wavescan Centroids

This menu will calculate the peak centroid for all elements in all wavescan samples using the parabolic, maxima and Gaussian fit routines and output them to files based on the wavescan sample name. Consecutive wavescan samples that have the same name will be output to the same file.

The parabolic, maxima and Gaussian threshold levels in the PROBEWIN.INI file (as displayed in the Peaking dialog) will be used for the fitting routines. The column labels are documented as follows:

|          |                       |
|----------|-----------------------|
| STG XPOS | Stage x-axis position |
|----------|-----------------------|

|                                      |   |
|--------------------------------------|---|
| STG YPOS                             | Stage y-axis position                           |
| STG ZPOS                             | Stage z-axis position                           |
| <element> PARABOLIC SP<spectrometer> | Parabolic centroid for element and spectrometer |
| <element> MAXIMA SP<spectrometer>    | Maxima centroid for element and spectrometer    |
| <element> GAUSSIAN SP<spectrometer>  | Gaussian centroid for element and spectrometer  |
| SAMPLE                               | Sample name in double quotes                    |

## Output Wavescan Spectrum Image (Lispix Format)

A new capability is the output of Lispix format spectrum image data files including the .RPL parameter file. Note that this feature will output each complete WDS sample (including “continued” samples) as a single pixel in the data hypercube. Therefore the program will output all wavescan samples in a single probe run as a complete spectrum image.

Because Lispix expects the data in a rectangular array, be sure that samples correspond to WDS data acquired from a rectangular array of gridded wavescan points. Because the user will need to specify the X and Y grid array coordinate dimensions it will be necessary for the number of data rows and columns be noted, for example in the sample name or description fields, and be sure that the “Stagger The Grid Points” option was NOT used for the digitization of the stage coordinates so that an orthogonal grid is produced.

Generally because each wavescan represents only a single pixel, it is generally preferred that the entire probe run be devoted to a single WDS spectrum image. With the current limit of 9999 samples per run that limits the Lispix spectrum image to 9999 wavescans or pixels or a grid of such wavescans of almost 100 x 100 pixels.

## Export Wavescan Samples for Feig Method Development Method Tool

Use this menu to export wavescan samples for import into the web based Feig Method Development Tool.

## Save Images To BMP Files

This menu will automatically export all images in the current database to separate BMP files, including the drawing layer (stage coordinates, labels, scale-bar, etc.) based on the user specified selections. Be sure to use the Run | Display and Export Images menu to first specify the drawing layer components before exporting them.

## Save MultiPoint Position and Intensity Data

This menu will output a tab delimited file of position and intensities for samples with multi-point background data.

Note that only samples with multi-point data will have columns of multi-point data. But all samples will have their normal high and low peak position and intensity data output.

|                     |  |
|---------------------|--|
| BACKTYP             | Background acquisition type, 0=off-peak, 1=MAN, 2=multi-point  |
| OFFTYP              | Background correction type, 0=linear, 1=average, 2=high only, 3=low only, 4=exponential, 5=slope hi, 6=slope lo, 7=polynomial, 8=multi-point |
| NECNT<br>corrected) | Corrected on-peak intensity (background, etc. interference   |
| OFCNT               | Calculated background intensity  |
| MACQHI              | MultiPoint parameters, number of acquire high side points  |
| MACQLO              | MultiPoint parameters, number of acquire low side points   |
| MUITHI              | MultiPoint parameters, number of high side points to iterate to  |
| MUITLO              | MultiPoint parameters, number of low side points to iterate to   |
| MULFIT              | MultiPoint parameters, multi-point fit type, 0=linear, 1=polynomial, 2=exponential   |
| MHPos               | MultiPoint high side positions   |
| MLPos               | MultiPoint low side positions  |

|         |  |
|---------|--|
| MHIint  | MultiPoint high side intensities                         |
| MLOint  | MultiPoint low side intensities                          |
| MHItim  | MultiPoint high side count times                         |
| MLOTIM  | MultiPoint low side count times                          |
| MHIflg  | MultiPoint high side manual override flags               |
| MLOflg  | MultiPoint high side manual override flags               |
| ONPOS   | On-peak positions  |
| HIPOS   | Hi-peak positions (averaged for multi-point)             |
| LOPOS   | Lo-peak positions (averaged for multi-point)             |
| ONCNT   | On-peak intensities                                      |
| HICNT   | Hi-peak intensities (average of multi-point intensities) |
| LOCNT   | Lo-peak intensities (average of multi-point intensities) |
| ONTIM   | On peak count times                                      |
| HITIM   | Hi peak count times                                      |
| LOTIM   | Lo peak count times                                      |
| DETIM   | Deadtime parameters                                      |
| BEAMCUR | Line beam current  |
| BEAM    | Element beam current (combined conditions only)          |

## Save Time Dependent Intensities (TDI)

This menu will output the Time Dependent Intensity (TDI) intensity arrays for each standard or unknown sample. Only elements that are Time Dependent Intensity (TDI) corrected will be output (only the first element of each spectrometer). It will output the assigned or self Time Dependent Intensity (TDI) intensities depending on the Time Dependent Intensity (TDI) assignment. Sample assigned to another sample are assigned Time Dependent Intensity (TDI) corrected, sample assigned to themselves are self Time Dependent Intensity (TDI) corrected.

|               |  |
|---------------|--|
| El TIME       | Elapsed time, element labels and raw TDI intensities   |
| El SEC        |  |
| El CPS        |  |
| El SEC        | Elapsed time, element labels and beam drift corrected TDI intensities                          |
| El CPS/nA     |  |
| El SEC        | Elapsed time, element labels and beam drift corrected and background corrected TDI intensities |
| El P-B_CPS/nA |  |
| El ABSCURR    | Absorbed current (in nA) for each TDI element.   |

Both raw cps, beam normalized (cps/nA) and background corrected intensities are output. Combined condition sample are beam drift corrected based on the arrays of measured beam current for each element. Note: Only off-peak Time Dependent Intensity (TDI) element intensities are background corrected, no background correction is applied to MAN background corrected Time Dependent Intensity (TDI) elements.

## Save All EDS Spectra To EMSA (Output all EDS spectra to EMSA format files)

This menu will export EDS spectra intensities to MSA EMSA format files automatically for all samples in the current run. The files will be named with the run name, sample name and line number. To export individual EDS spectra use the Export Spectrum (EMSA) button in the Assign EDS Spectral Elements dialog from the Calculation Options dialog from the Analyze! window.

## Save All CL Spectra to EMSA (Output all CL spectra to EMSA format files)

This menu will export EDS spectra intensities to MSA EMSA format files automatically for all samples in the current run. The files will be named with the run name, sample name and line number. To export individual EDS spectra use the Export Spectrum (EMSA) button in the Assign EDS Spectral Elements dialog from the Calculation Options dialog from the Analyze! window.

In the EMSA CL output the first column is the light spectrum and the second column is the dark spectrum.

## Save Trace Element Statistics

This menu will export statistical data especially useful for trace element analyses. Only a single line is output for each sample. The format is as follows:

|                  |  |
|------------------|--|
| SAMPLE           | Sample name (in double quotes)                                 |
| TOTAL            | Total weight percent (average for sample)                      |
| ONTIM            | Actual on peak count time (average for element)                |
| HITIM            | Actual high off peak count time (average for element)          |
| LOTIM            | Actual low off peak count time (average for element)           |
| DETIM            | Deadtime in micro-seconds                                      |
| BEAMCUR          | Faraday or beam current reading in nano-amps (average)         |
| El AVG WT%       | Elemental weight percent averages                              |
| El SDEV          | Elemental weight percent standard deviations                   |
| El SERR          | Elemental weight percent standard errors                       |
| El RELDEV        | Elemental relative deviations (stddev / average)               |
| El MIN           | Elemental weight percent minimums                              |
| El MAX           | Elemental weight percent maximums                              |
| El CDL99 (Pred.) | Elemental weight percent detection limits (3 sigma prediction) |
| El CDL99         | Elemental weight percent detection limits (average 3 sigma)    |
| El CDL99 (PPM)   | Elemental PPM detection limits (average 3 sigma)               |
| El %ERR          | Elemental analytical error in relative percent (one sigma)     |
| El CDL(60ci)     | Elemental t-test detection limit (60 percent confidence)       |
| El CDL(80ci)     | Elemental t-test detection limit (80 percent confidence)       |
| El CDL(90ci)     | Elemental t-test detection limit (90 percent confidence)       |
| El CDL(95ci)     | Elemental t-test detection limit (95 percent confidence)       |
| El CDL(99ci)     | Elemental t-test detection limit (99 percent confidence)       |
| El SEN(60ci)     | Elemental t-test analytical sensitivity (for major elements)   |
| El SEN(80ci)     | Elemental t-test analytical sensitivity (for major elements)   |
| El SEN(90ci)     | Elemental t-test analytical sensitivity (for major elements)   |
| El SEN(95ci)     | Elemental t-test analytical sensitivity (for major elements)   |
| El SEN(99ci)     | Elemental t-test analytical sensitivity (for major elements)   |

The expression used for the predicted detection limits utilizes both the statistics from the peak and background:

$$X_U = \frac{3\sqrt{I_U^B \cdot t_U^B \cdot B_U \cdot C_S}}{t_U^B \cdot B_U \cdot I_S^P}$$

where:  $I_U^B$  is the intensity of the background in cps/nA (divide the unknown counts by beam current)  
 $t_U^B$  is the count time used for the unknown background or peak measurement (must be equal)  
 $B_U$  is the beam current used for the unknown measurement in nA

- $C_S$  is the concentration of the element in the standard in weight percent (wt. %)  
 $I_S^P$  is the intensity of the standard in cps/nA (divide the standard intensity by standard beam current)  
 $X_U$  is the unknown concentration in weight percent (wt. %)

## Save Homogeneity Calculations

This menu (homogeneity output format) will create an output file of standard or unknown average weight, oxide and atomic percents along with homogeneity calculations and also the average detection and analytical sensitivity for each data point.

Please note that the HTO (homogeneity test of) is calculated yes, no or null at the 1% concentration level.

|           |  |
|-----------|--|
| SAMPLE    | Sample number and name (in double quotes)                              |
| El AT%    | Atomic percent   |
| El WT%    | Elemental weight percent   |
| Ox WT%    | Oxide weight percent   |
| TOTAL     | Total weight percent   |
| El HRO60  | Elemental t-test "homogeneity range of" (60 percent confidence)        |
| El HRO80  | Elemental t-test "homogeneity range of" (80 percent confidence)        |
| El HRO90  | Elemental t-test "homogeneity range of" (90 percent confidence)        |
| El HRO95  | Elemental t-test "homogeneity range of" (95 percent confidence)        |
| El HRO99  | Elemental t-test "homogeneity range of" (99 percent confidence)        |
| El HTO60  | Elemental t-test "homogeneity test of" (60 percent confidence)         |
| El HTO80  | Elemental t-test "homogeneity test of" (80 percent confidence)         |
| El HTO90  | Elemental t-test "homogeneity test of" (90 percent confidence)         |
| El HTO95  | Elemental t-test "homogeneity test of" (95 percent confidence)         |
| El HTO99  | Elemental t-test "homogeneity test of" (99 percent confidence)         |
| El HLO60  | Elemental t-test "homogeneity level of" (60 percent confidence)        |
| El HLO80  | Elemental t-test "homogeneity level of" (80 percent confidence)        |
| El HLO90  | Elemental t-test "homogeneity level of" (90 percent confidence)        |
| El HLO95  | Elemental t-test "homogeneity level of" (95 percent confidence)        |
| El HLO99  | Elemental t-test "homogeneity level of" (99 percent confidence)        |
| El HIRO60 | Elemental t-test "homogeneity ideal range of" (60 percent confidence)  |
| El HIRO80 | Elemental t-test "homogeneity ideal range of" (80 percent confidence)  |
| El HIRO90 | Elemental t-test "homogeneity ideal range of" (90 percent confidence)  |
| El HIRO95 | Elemental t-test "homogeneity ideal range of" (95 percent confidence)  |
| El HIRO99 | Elemental t-test "homogeneity ideal range of" (99 percent confidence)  |
| El HARO60 | Elemental t-test "homogeneity actual range of" (60 percent confidence) |
| El HARO80 | Elemental t-test "homogeneity actual range of" (80 percent confidence) |
| El HARO90 | Elemental t-test "homogeneity actual range of" (90 percent confidence) |
| El HARO95 | Elemental t-test "homogeneity actual range of" (95 percent confidence) |
| El HARO99 | Elemental t-test "homogeneity actual range of" (99 percent confidence) |
| El CDL60  | Elemental t-test detection limit (60 percent confidence)               |
| El CDL80  | Elemental t-test detection limit (80 percent confidence)               |

|          |   |
|----------|---|
| El CDL90 | Elemental t-test detection limit (90 percent confidence)            |
| El CDL95 | Elemental t-test detection limit (95 percent confidence)            |
| El CDL99 | Elemental t-test detection limit (99 percent confidence)            |
| El CAS60 | Elemental t-test analytical sensitivity (60% CI for major elements) |
| El CAS80 | Elemental t-test analytical sensitivity (80% CI for major elements) |
| El CAS90 | Elemental t-test analytical sensitivity (90% CI for major elements) |
| El CAS95 | Elemental t-test analytical sensitivity (95% CI for major elements) |
| El CAS99 | Elemental t-test analytical sensitivity (99% CI for major elements) |

See the section above entitled: Calculation Options | Calculate Detection Limits and Analytical Sensitivity for a detailed description of the homogeneity calculation output.

## Save U, Th, Pb Age Calculations

This menu (U, Th, Pb Age calculation format) will create an output file of standard or unknown samples using code based on Montel's age calculations written by Julien Allaz and John Donovan for monazite age calculations based on the uranium, thorium and lead concentrations (assuming all lead is radiogenic).

In addition the elemental and oxide weight percents, along with single point detection and analytical sensitivity and raw k-ratios, count times, beam current and x, y, z coordinates and relative distance in microns will also be output for all elements.

|             |   |
|-------------|---|
| SAMPLE      | Sample name (in double quotes)                                      |
| LINE        | Line number   |
| U WT%       | Elemental weight percent of uranium                                 |
| Th WT%      | Elemental weight percent of thorium                                 |
| Pb WT%      | Elemental weight percent of lead                                    |
| U PPM       | Elemental weight percent of uranium in PPM                          |
| Th PPM      | Elemental weight percent of thorium in PPM                          |
| Pb PPM      | Elemental weight percent of lead in PPM                             |
| Age[My]     | Calculated monazite age in millions of years                        |
| Calc Pb     | Pb calculated (predicted)   |
| %Pb(U       | Percent of Pb from radioactive decay of uranium                     |
| %Pb(Th)     | Percent of Pb from radioactive decay of thorium                     |
| <br>El (O2) | Oxide weight percent  |
| El WT%      | Elemental weight percent  |
| TOTAL       | Total weight percent  |
| DAT/TIM     | Acquisition date and time in system defined format                  |
| X-POS       | X stage position  |
| Y-POS       | Y stage position  |
| Z-POS       | Z stage position  |
| RELDIST     | Relative distance (in microns)                                      |
| BEAMCUR     | Beam or probe current (in nA) (array if combined conditions sample) |
| ABSCUR      | Sample or absorbed current (in nA)                                  |
| El AT%      | Atomic percent  |
| El CDL99    | Detection limit in elemental weight percent at 99% confidence       |
| El %ERR     | Analytical error in relative percent (one sigma)                    |
| El ONTIM    | On-peak counting time   |
| El HITIM    | High off-peak counting time   |
| El LOTIM    | Low off-peak counting time  |
| El K-RAW    | Elemental raw k-ratio (unk counts/standard counts)                  |
| <br>STD_NUM | Primary standard assignment number                                  |
| STD_NAM     | Primary standard assignment name                                    |

This file format was specified by Julien Allaz and Michael Jercinovic at the University of Massachusetts and cannot be modified for other applications.

## Save Hanchar-Montel Geochron Calculations

This menu will output Pb, Th and U concentrations (in PPM), Rel% Error (in percent) and Detection Limits (in PPM) for use with John Hanchar's monazite calculation spreadsheet. The format is as follows:

|             |   |
|-------------|---|
| SAMPLE      | Sample name (in double quotes)                                      |
| LINE        | Line number   |
| El Conc Pb  | Elemental weight percent Pb (in PPM)                                |
| Rel% Err Pb | Relative analytical error Pb (in percent)                           |
| CDL Pb      | Elemental detection limit Pb (in PPM)                               |
| El Conc Th  | Elemental weight percent Th (in PPM)                                |
| Rel% Err Th | Relative analytical error Th (in percent)                           |
| CDL Th      | Elemental detection limit TH (in PPM)                               |
| El Conc U   | Elemental weight percent U (in PPM)                                 |
| Rel% Err U  | Relative analytical error U (in percent)                            |
| CDL Pb      | Elemental detection limit U (in PPM)                                |
| TOTAL       | Total weight percent  |
| DAT/TIM     | Acquisition date and time in system defined format                  |
| X-POS       | X stage position  |
| Y-POS       | Y stage position  |
| Z-POS       | Z stage position  |
| RELDIST     | Relative distance (in microns)                                      |
| BEAMCUR     | Beam or probe current (in nA) (array if combined conditions sample) |
| ABSB CUR    | Sample or absorbed current (in nA)                                  |
| El ONTIM Pb | On-peak counting time   |
| El HITIM Pb | High off-peak counting time   |
| El LOTIM Pb | Low off-peak counting time  |
| El ONTIM Th | On-peak counting time   |
| El HITIM Th | High off-peak counting time   |
| El LOTIM Th | Low off-peak counting time  |
| El ONTIM U  | On-peak counting time   |
| El HITIM U  | High off-peak counting time   |
| El LOTIM U  | Low off-peak counting time  |

Note that the total number of lines output will be stored before the first line data output. Samples that do not contain all three elements will be skipped and a warning will be logged to the log window.

## Save CalcZAF Format

This menu will output x-ray intensity raw k-ratio data for all unknown samples in the current run to a CalcZAF compatible format. Each line in each sample will be output to the file. Import in CalcZAF.exe using the File | Open menu and click the Load Next Dataset From File button to load the next data line.

See CalcZAF section for documentation on the CalcZAF import format.

## Save CalcZAF "Standard" Format

This menu will export all standards in the current run to a specified file in the CalcZAF "Standard" format. This is a single line per standard format that also includes the published values of each standard for the purposes of evaluating the accuracy of standard analyses. It is essentially a numerical version of the Evaluate applet, but requires post processing in CalcZAF using the Analytical | Calculate Standard Concentrations menu.

The first column is "standard name" in double quotes.

The second column is the takeoff angle.

The third column is the kilovolts.

The fourth column is the oxide or elemental flag (1 = elemental, 2 = oxide).

The fifth column is the number of elements.

The next n columns are the element symbols (use double quotes).

The next n columns are the element x-ray lines (use double quotes) (leave empty for specified concentration, must be last element(s)).

The next n columns are the measured k-ratios for each element (blank for specified concentration).

The next n columns are the "published" weight percent for each element.

The next n columns are the primary standard number assignment for each element.

The next n columns are the number of cations per element.

The next n columns are the number of oxygens per element.

The following is an example of the output created using this menu. The first line has oxygen measured and the second line has oxygen specified using the value from the standard database

```
"Fayalite", 40.0, 15.0, 2, 3, "fe", "si", "o", "ka", "ka", "ka", .7372, .2523, 1.0086, 54.809,  
13.785, 31.407, 895, 14, 895, 1, 1, 1, 1, 2, 0  
"Fayalite", 40.0, 15.0, 2, 3, "fe", "si", "o", "ka", "ka", "", .7372, .2523, 0.0, 54.809, 13.785,  
31.407, 895, 14, 0, 1, 1, 1, 1, 2, 0
```

## Save StrataGem Format

This menu will open a dialog that allows the user to select samples of different voltage conditions for use in thin film calculations in the StrataGem application. The format includes the raw k-ratios for all samples and the standard compositions assigned in the last selected sample. The user has the choice of exporting all data in a sample or just the average of each sample.

Once the file is exported, from StrataGem select File | Import | StrataGem Import and the program will prompt the user whether to import or skip each k-ratio. The program will average all imported values for each element that have the same voltage along with the specimen description, if specified.

## Save Cluster Classification Format (for CalcImage)

This menu will export all standard or unknown sample quantitative results to a tab delimited text file for further statistical processing in CalcImage using various clustering methods including a modified Hartigan-Wong kmeans clustering technique.

The output format is as follows (note the first line has the total number of data rows repeated three times for compatibility with the CalcImage pixel clustering data format):

1st Line = (rows), (rows), (rows), (columns: NK + X + Y + NS + NL + ND + <data1>, <data2>...)

2nd Line = column labels

3rd Line = data values

1st column = NK (zero placeholder for classify cluster numbers)

2nd column = X stage coordinates

3rd column = Y stage coordinates

4th column = NS (Sample number)

5th column = NL (Line number)

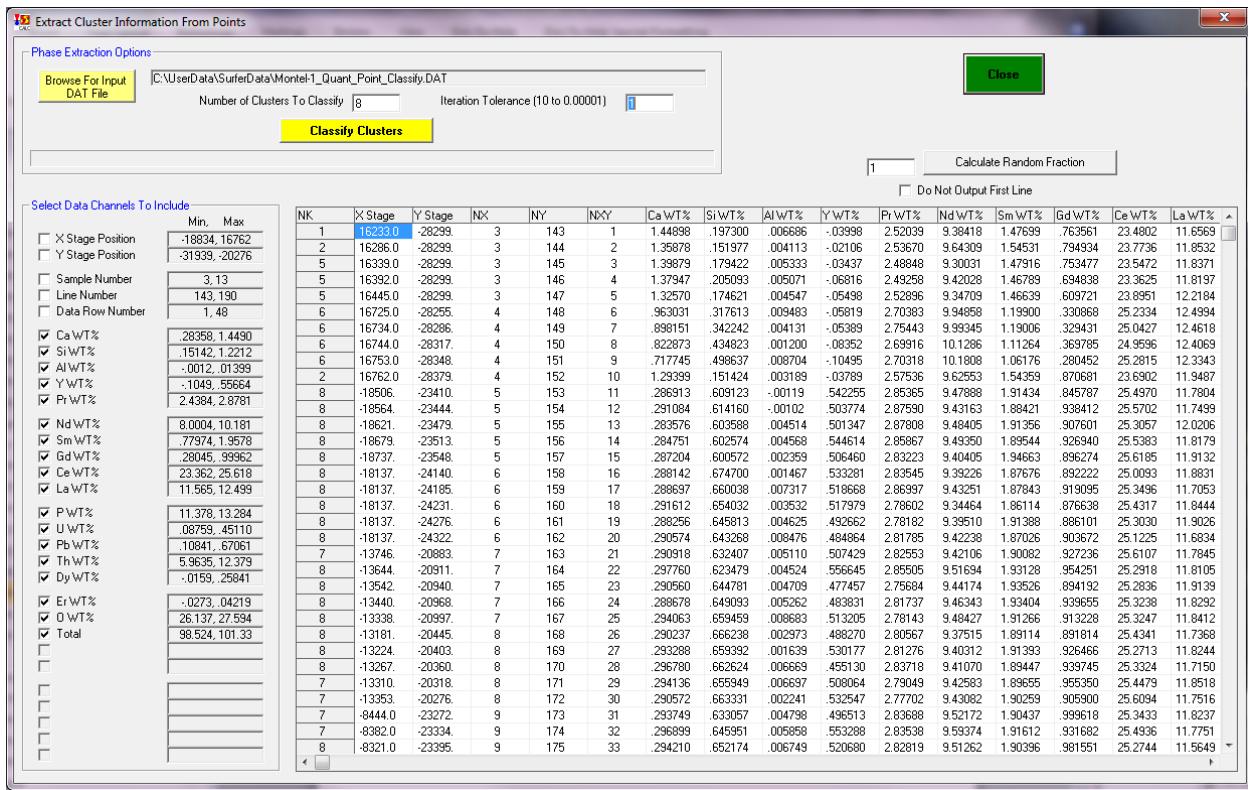
6th column = ND data row number index

7th column = channel 1 data values...

8th column = channel 2 data values...

9th column = channel 3 data values...

The program will prompt the user for whether all data should be exported toseparate files based on samples names and after importing and processing in CalcImage the first column (originally containing zeros, will be saved and displayed as shown here along with the centroid (average) compositions:



## Extended Format

This menu toggles the output option for printout to the log window of element data. If unchecked, the program automatically wraps element data output to 8 elements per line. Note that wavescan data is wrapped 4 elements per line. If this option is checked, the program does not wrap element data to the log window or disk file (if Save To Disk option is enabled), instead the data is written out as far as necessary to the right.

## Debug Mode

This menu toggles the program in and out of Debug mode. In Debug mode, the program generates an abundance of additional output to the log window for the purposes of debugging the program or viewing the analytical calculations in greater detail.

Debug mode is also useful to check the low level commands sent to the hardware interface. Note that in Debug mode the Form level events are not protected against unanticipated errors.

## Verbose Mode

This menu toggles the “verbose” output mode of the program. This will result in continuous detailed output to the log window. Normally only used for detailed debugging issues.

## Time Stamp Mode

Display the current time of day that each comment is sent to the log window. Useful for various timing experiments on the instrument interface.

## **Driver Logging Mode**

This menu will close the instrument interface, change the current driver logging mode and re-open the interface. If the current driver logging mode was none (zero), the logging mode will be changed to verbose (2) and visa versa.

## **Log Window Font**

This menu allows the user to modify the current log window font type, size and appearance using the font Common dialog. It is strongly recommended to use a non-proportional font, such a New Courier, so that numeric column data is properly aligned in the log window.

Note that the default font and size of the log window may be permanently defined in the PROBEWIN.INI file.

## **Save To Disk Log**

This menu toggles the output option to mirror all log window output to a user specified text file. This may be useful for importing text or data from the log window to another non-Windows program. The default extension is .OUT but another extension may be entered by the user.

If a large amount of output is to be generated, this option will capture all output to the log window so that it can be printed out afterwards. If the output file selected by the user already exists, the program offers the option to overwrite or append subsequent output to the already existing file.

## **View Disk Log**

This menu invokes the file viewer specified in the PROBEWIN.INI file (default file viewer is NotePad) to view the previously or currently opened disk log file.

If the file is currently open, the program will first close the disk log file before viewing it. Note that because the default file viewer (NotePad) is somewhat limited, it may be necessary to specify a more robust file viewer such as TextPad. See the section on editing the PROBEWIN.INI file above to change the default file viewer.

## **Open File Viewer**

This menu will open a blank file with the default text editor specified in the PROBEWIN.INI file.

## **Open Link To Excel**

This menu will create an OLE (Object Linking and Embedding) link to an Excel application (if it is available). This link is available to both the StartWin and ProbeWin applications for sending both intensity and quantitative data to an Excel spreadsheet. This feature requires Excel 8 (Office 97) or higher.

From StartWin, each time intensity data is acquired, it will be automatically sent to an Excel spreadsheet along with the column labels. From ProbeWin, the analysis line number and elemental weight percents and totals are sent to an Excel spreadsheet after an analysis is performed and the user clicks the ">>Excel" button in the Analyze! window. In the ProbeWin application the column labels will be sent if they differ from the previous sample.

## **Close Link To Excel**

This menu will close an open OLE (Object Linking and Embedding) link to an Excel application (if it is available).

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# Help

## About Probe for EPMA

This menu displays the copyright notice, acknowledgments and contact information for users of **Probe for EPMA Xtreme**.

## Help On Probe for EPMA

This menu opens this help file for hypertext help file viewing at the main table of contents. Note that both a searchable index and glossary are available for browsing by the user at any time during a run. Each help topic may be printed. To obtain a complete printout of the help file, ask for an updated copy of the PROBEWIN.PDF file, which is an Adobe Acrobat portable document file that may be viewed and/or printed using the free Acrobat Reader.

**In addition, context sensitive help (that is, help that is applicable to the current program context) is available for each window in all applications by simply pressing the F1 key. To get help on a menu item simply highlight with the mouse and hit the F1 key.**

## User Reference Manual

This menu opens the User Reference manual which describes each application function in detail with installation, configuration instructions, examples and references.

## Getting Started Manual

This menu opens the Getting Started manual which provides step by step instructions for the most common microanalysis tasks in Probe for EPMA. For example, entering standard compositional data, peaking the spectrometers, and automating sample acquisitions.

## Advanced Topics Manual

This menu provides step by step instructions for advanced procedures in Probe for EPMA. For example using multiple sample setups, specifying interference corrections and time dependent intensity corrections.

## Update Probe for EPMA

This menu will open a dialog that allows the user to update Probe for EPMA to the latest version. Note that after the download is completed you will be required to close the application and press a key to continue the update process so that all files are properly updated.

The download can be performed using secure FTP or HTTP. The FTP method is faster but some firewalls may not allow it. In this case please use the alternate HTTP download method which should work on all network systems (provided they are connected to the Internet).

## Display Program Information

Opens the VERSION.TXT as a text file in order to view changes to the software. To see the most recent changes to the software simply scroll down to the bottom of the file.

## Probe Software On The Web

This menu will open your default browser and go to the Probe Software web site, [www.probesoftware.com](http://www.probesoftware.com).

## **User Wizard**

This menu opens a pictorial guided tour of the software to open a new Probe database run and get started with data acquisition, processing and output.

## **Probe for EPMA Quick Start Guide**

Opens the Probe for EPMA Quick Start Guide PDF file. This document is useful as a quick and easy reference for starting a new Probe for EPMA run.

## **Probe for EPMA Frequently Asked Questions**

Opens the Probe for EPMA Frequently Asked Questions PDF file. This document provides answers to commonly asked questions.

# **Appendix A**

## **Upgrading from Probe for Windows 32 Bit to Probe for EPMA**

Probe for EPMA Xtreme Edition is almost fully compatible with Probe for Windows 32 bit. All configuration files from Probe for Windows 32 bit can be simply copied over to the Probe for EPMA Xtreme Edition, including the PROBEWIN.INI, SCALERS.DAT, MOTORS.DAT, DETECTORS.DAT, CRYSTALS.DAT, EMPMAC.DAT, EMPAPF.DAT, EMPFAC.DAT, EMPPHA.DAT, \*.POS, SETUP.MDB, USER.MDB, POSITION.MDB and STANDARD.MDB files.

The ELEMENTS.DAT and CHARGES.DAT files are not compatible since they have been extended for 100 elements but can be edited by the user if desired. The binary x-ray and mass absorption coefficient database files XLINE.DAT, XEDGE.DAT, XFLUR.DAT, LINEMU.DAT, CITZMU.DAT, etc. are also not compatible since they now support beta lines and 100 elements. The newly installed Xtreme Edition versions of these files will have to be used.

Finally some of the binary k-ratio files utilized by CalcZAF have been changed to support beta lines. These include NISTBIN.DAT and POUCHOU.DAT.

The probe database MDB files are fully backward compatible with Probe for EPMA Xtreme Edition though some new features may not be available if an older version data file is utilized for new acquisitions.

# Appendix B

## Problems with Light Element Analysis

The analysis of light elements (fluorine - beryllium) present special problems for the microprobe analyst. Some of these problems are technical in substance, relating to the measurement procedure, while others are inherent and are due to physical and chemical effects. The following is just an overview of these concerns. For a more complete treatment the reader is referred to (Bastin and Heijligers, 1986) and (Goldstein, et. al., 1981, and 1992).

### Physical and Chemical Effects

1. Low fluorescence yields from these k-lines, compounded by significant matrix absorption at "normal" accelerating voltages (15-20 keV), the relatively poor reflectivity of traditional analyzing crystals (e.g., STE), and the low sensitivity of detector systems, can result in extremely low count rates. Increasing the beam current can produce unacceptably high deadtime corrections for any metal lines being measured, although Probe for EPMA now can accommodate "combined conditions" where certain low intensity elements are run at high beam currents, and other high intensity elements are run at low beam currents. Count rates can be enhanced by use of layered dispersive element (LDE) reflectors as well as decreasing the absorption path length by running at lower operating voltages.
2. Errors in the estimation or accuracy of the takeoff angle or operating voltage of the microprobe. While slight variations in these parameters normally have a little effect on a typical analysis, due to the very high absorption corrections of low energy x-rays, the effect on the ZAF correction can be much larger for the light elements and can sometimes result in large systematic errors.
3. Errors in the mass absorption coefficients (or MACs) for the x-ray absorption cross sections for these elements are considerable. See Appendix C for a table of MACs for the light elements from a variety of sources. There is considerable disagreement among investigators. Probe for EPMA allows the user to enter MACs from any source for re-calculation purposes.
4. Chemical bonding effects can result in large peak shift and/or shape changes to the analytical x-ray lines. In fact the degree of shift and/or shape changes in light element K lines and transition metal L lines can sometimes be related to the degree of oxidation in the compound. But normally these effects are a nuisance to the analyst. In a few instances there has even been documented cases of particular lines being strongly enhanced ( $\text{Ni L}\alpha$  in NiAl) or reduced ( $\text{B K}\alpha$  in nickel borides) (Pouchou and Pichoir, 1988).
5. Mobilization toward or away from the beam ("volatilization") in some glasses and a few minerals of certain light elements such as fluorine and oxygen as well as alkali elements such as sodium and potassium to a lesser extent is often the case. This requires special attention, either by operating a lower current, defocusing the beam, using a cold stage (not common), or using a robust software correction. See the section on the Time Dependent Intensity (TDI) element correction in this document.
6. Crystallographic orientation can play a role for analysis of certain phases. This has been well documented for some crystalline borides, and also when measuring fluorine in apatites.

## Problems with the Measurement Techniques

1. Higher order interferences from metal lines are everywhere and must be avoided when possible or corrected for in software when not. The elements Ti, Cr, Mn, Zr and Nb are well known in this respect. See Donovan et al (1993). These interferences are especially severe for minor and trace level measurements. Use of pulse height analysis to reduce those interferences that are caused by high order interferences ( $>1$ ) is a logical choice; however it can introduce further problems if not used with care. In particular, narrow windows required for discrimination of second order peaks may be so narrow that a shift in pulse location ("pulse height depression", as pulse voltage drops with high count rate) will produce significant errors. Similarly, the pulse location may shift between the standard (pure element) and the unknown in extreme cases.
2. Carbon contamination is a serious problem for quantitative analysis of light elements, especially of course carbon, but also other light elements. Contamination is produced by the polymerization of hydrocarbons at the point of impact of the electron beam. Carbon compounds present may include mainly rotary but also some diffusion pump oils, polishing agents, cleaning solvents, and "canned air" propellants, among others. The use of an air jet and cold finger has been shown to dramatically reduce carbon contamination, though the use of a Freon chilled baffle at the top of the diffusion pump will completely eliminate back streaming of oil from a diffusion pump. A turbo pumped system is expensive but also eliminates oil contamination from the pumping system. It is suggested that the analyst perform measurements to ascertain the actual carbon contamination rate in their microprobe for each element being analyzed.
3. Trace analysis of light elements is particularly difficult, especially oxygen and carbon. Keeping a metallic sample surface from oxidizing is not a trivial matter. The use of an ion mill inside the probe to remove the very top layer of the sample, just prior to the analysis, if available, would be ideal. Note that the increase in temperature of non-thermally conducting samples can be significant enough to cause additional oxidation of the surface. Trace carbon analyses can be difficult without the use of an air jet to reduce the carbon contamination at the point of impact of the beam; however, it has been found to be counterproductive for oxygen analysis (i.e., it enhances oxidation under the beam).
4. Another problem which has not been adequately addressed is x-ray production in non-conductive samples. Effects on x-ray production are seen even when a conductive coating has been applied, possibly due to internal charge buildup below the conductive layer. This effect may also be implicated in the "Time Dependent Intensity (TDI) element" behavior observed in glasses and some minerals.
5. The selection of standards is also critical due to the already mentioned peak shift and/or shape changes for light element x-rays. The use of Area Peak Factors (APFs) (Bastin and Heijligers, 1986) can provide the analyst with a practical method for treatment of these effects. Probe for EPMA supports the use of APF factors for analysis of light elements. See the previous sections on using APFs for correcting for peak shape and shift effects when analyzing light elements.
- Many analysts use high purity metals as standards for various elements, without realizing that many metals develop oxide skins, which will reduce the actual number of x-rays of the particular element of interest (e.g., Al, Ti, Mn being among the worst). One either needs to repolish immediately prior to probing, or spend some time estimating the oxide content, or find an alloy that doesn't oxidize as quickly.
6. Be aware also that carbide, boride and nitride standards are often pressed and sintered powders and are not 100% dense, resulting in significant errors in the analysis. If single crystals, or arc-melted pieces (e.g., boron) can be obtained, so much the better.

# Appendix C

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## Use of Empirical MACs

The following is a small compilation of mass absorption coefficients (MACs) from a number of investigators. Some of these MACs may be selected for use in the quantitative calculations in Probe for EPMA, from the Analytical | Empirical MACs menu item. Up to MaxEmp% empirical mass absorption coefficients may be defined for each run. These MACs will be saved to the .MDB file for subsequent re-processing and can be added to, changed or deleted again.

## Empirical Mass Absorption Coefficients (MACs)

| <b>Emitter</b> | <b>Absorber</b> | <b>Henke Ebisu</b> | <b>Ruste</b> | <b>Henke et al.</b> | <b>Bastin Heijligers</b> | <b>Heinrich</b> | <b>Frazer</b> | <b>Pouchou Pichoir</b> |
|----------------|-----------------|--------------------|--------------|---------------------|--------------------------|-----------------|---------------|------------------------|
| B ka           | B               | 3353               |              | 3350                | 3500                     |                 |               | 3471                   |
| B ka           | C               | 6456               |              | 6350                | 6500                     |                 |               | 6750                   |
| B ka           | N               | 10570              |              | 11200               | 11200                    |                 |               | 11000                  |
| B ka           | O               |                    |              |                     |                          |                 |               | 16500                  |
| B ka           | Al              | 65170              |              | 64000               | 64000                    |                 |               | 64000                  |
| B ka           | Si              | 74180              |              | 84000               | 84000                    |                 |               | 80000                  |
| B ka           | Ti              | 15280              |              | 15300               | 14700                    |                 |               | 15000                  |
| B ka           | V               | 16710              |              | 16700               | 17700                    |                 |               | 18000                  |
| B ka           | Cr              | 20670              |              | 20700               | 20200                    |                 |               | 20700                  |
| B ka           | Fe              | 25780              |              | 27600               | 27300                    |                 |               | 27800                  |
| B ka           | Co              | 28340              |              | 30900               | 33400                    |                 |               | 32000                  |
| B ka           | Ni              | 33090              |              | 35700               | 42000                    |                 |               | 37000                  |
| B ka           | Zr              | 38410 <sup>1</sup> |              | 8270                | 4000                     |                 |               | 4400                   |
| B ka           | Nb              | 4417               |              | 6560                | 4600                     | 3822            |               | 4500                   |
| B ka           | Mo              | 4717               |              | 5610                | 4550                     | 4208            |               | 4600                   |
| B ka           | La              | 3826               |              | 3730                | 2500                     | 11654           |               | 2500                   |
| B ka           | Ta              | 20820              |              | 20800               | 22500                    |                 |               | 23000                  |
| B ka           | W               | 19660              |              | 19700               | 21400                    |                 |               | 21000                  |
| B ka           | U               | 2247               |              | 9020                | 8200                     |                 |               | 7400                   |
|                |                 |                    |              |                     |                          |                 |               |                        |
| C ka           | B               | 37020              | 37020        | 37000               | 41000                    |                 |               | 39000                  |
| C ka           | C               | 2373               | 2373         | 2350                | 2373                     | 2147            |               | 2170                   |
| C ka           | Si              | 36980              | 36980        | 36800               | 37000                    |                 |               | 35000                  |
| C ka           | Ti              | 8094               | 8094         | 8090                | 9400                     | 8290            |               | 8097                   |
| C ka           | V               | 8840               | 9236         | 8840                | 10100                    |                 |               | 8850                   |
| C ka           | Cr              | 10590              | 10482        | 10600               | 10950                    |                 |               | 10700                  |
| C ka           | Fe              | 13300              | 13300        | 13900               | 13500                    | 14103           |               | 13150                  |
| C ka           | Zr              | 31130              | 31130        | 21600               | 24000                    | 31304           |               | 25000                  |
| C ka           | Nb              | 33990 <sup>1</sup> | 24203        | 19400               | 23200                    | 29760           |               | 24000                  |
| C ka           | Mo              | 32420 <sup>1</sup> | 15500        | 16400               | 19200                    |                 |               | 20500                  |
| C ka           | Hf              |                    |              |                     |                          |                 |               | 18000                  |
| C ka           | Ta              | 18390              | 20000        | 18400               | 16000                    |                 |               | 17000                  |
| C ka           | W               | 18750              | 21580        | 18800               | 17000                    |                 |               | 18000                  |
|                |                 |                    |              |                     |                          |                 |               |                        |
| N ka           | B               |                    |              |                     |                          |                 |               | 15800                  |
| N ka           | N               |                    |              |                     |                          |                 |               | 1640                   |
| N ka           | Al              |                    |              |                     |                          |                 |               | 13800                  |
| N ka           | Si              |                    |              |                     |                          |                 |               | 17000                  |

|       |    |       |  |       |       |       |       |
|-------|----|-------|--|-------|-------|-------|-------|
| N ka  | Ti |       |  |       |       |       | 4270  |
| N ka  | V  |       |  |       |       |       | 4950  |
| N ka  | Cr |       |  |       |       |       | 5650  |
| N ka  | Fe |       |  |       |       |       | 7190  |
| N ka  | Zr |       |  |       |       |       | 24000 |
| N ka  | Nb |       |  |       |       |       | 25000 |
| N ka  | Mo | 23220 |  | 20200 |       | 26458 | 25800 |
| N ka  | Hf |       |  |       |       |       | 14000 |
| N ka  | Ta |       |  |       |       |       | 15500 |
| O ka  | Li | 1602  |  | 1600  | 1600  |       |       |
| O ka  | B  | 7416  |  | 7420  | 8550  |       |       |
| O ka  | O  | 1200  |  | 1200  | 1200  |       |       |
| O ka  | Mg | 5174  |  | 5170  | 5170  |       |       |
| O ka  | Al | 6715  |  | 6720  | 6720  |       |       |
| O ka  | Si | 8790  |  | 8790  | 8790  |       |       |
| O ka  | Ti | 22140 |  | 22100 | 19900 |       |       |
| O ka  | Cr | 3143  |  | 3140  | 2900  |       |       |
| O ka  | Mn | 3468  |  | 3470  | 3470  |       |       |
| O ka  | Fe | 4001  |  | 4000  | 4000  |       |       |
| O ka  | Co | 4407  |  | 4410  | 4500  |       |       |
| O ka  | Ni | 5245  |  | 5120  | 5120  |       |       |
| O ka  | Cu | 5920  |  | 5920  | 5920  |       |       |
| O ka  | Zn | 6548  |  | 6550  | 6350  |       |       |
| O ka  | Ga | 7086  |  | 7090  | 7090  |       |       |
| O ka  | Y  | 15140 |  | 15100 | 15100 |       |       |
| O ka  | Zr | 16140 |  | 14800 | 16200 |       |       |
| O ka  | Nb | 17850 |  | 15300 | 17100 |       |       |
| O ka  | Mo | 18660 |  | 16700 | 18000 |       |       |
| O ka  | Ru | 19680 |  | 19700 | 19700 |       |       |
| O ka  | Sn | 23090 |  | 23100 | 15050 |       |       |
| O ka  | Ba | 4560  |  | 4560  | 4560  |       |       |
| O ka  | La | 4690  |  | 4690  | 3600  |       |       |
| O ka  | Ta | 10560 |  | 10600 | 10600 |       |       |
| O ka  | W  | 10990 |  | 11000 | 11300 |       |       |
| O ka  | Pb | 12470 |  | 12500 | 11000 |       |       |
| O ka  | Bi | 12690 |  | 12700 | 12100 |       |       |
| Al ka | Al |       |  | 403   |       | 397   | 404   |
| Al ka | Cu |       |  | 4550  |       | 4764  | 5410  |
| Si Ka | Si |       |  | 350   |       | 347   | 332   |
|       |    |       |  |       |       |       | 356   |

|       |    |  |  |       |  |       |       |       |
|-------|----|--|--|-------|--|-------|-------|-------|
| Si ka | Ta |  |  | 3760  |  | 1698  |       | 1500  |
|       |    |  |  |       |  |       |       |       |
| S ka  | Au |  |  |       |  | 3380  | 3203  | 2200  |
| S ka  | Hg |  |  |       |  | 1499  | 932   | 850   |
|       |    |  |  |       |  |       |       |       |
| Sc la | Sc |  |  |       |  |       |       | 4750  |
| Ti la | Ti |  |  |       |  |       |       | 4550  |
| V la  | V  |  |  |       |  |       |       | 4370  |
| Cr la | Cr |  |  |       |  |       |       | 3850  |
| Mn la | Mn |  |  |       |  |       |       | 3340  |
| Fe la | Fe |  |  |       |  |       |       | 3350  |
| Co la | Co |  |  |       |  |       |       | 3260  |
| Ni la | Ni |  |  |       |  |       |       | 3560  |
| Cu la | Al |  |  | 1450  |  | 1421  | 1494  | 1464  |
| Cu la | Ni |  |  | 11700 |  | 11776 | 12106 | 11879 |
| Cu la | Cu |  |  |       |  |       |       | 1755  |
| Zn la | Zn |  |  | 1520  |  | 1485  | 1581  | 1500  |
| Ge la | Ge |  |  | 1260  |  | 1262  | 1278  | 1240  |
| As la | Ga |  |  |       |  |       |       | 7000  |
| Nb la | Nb |  |  | 726   |  | 718   | 726   | 779   |
| Mo la | Au |  |  | 3680  |  | 3437  |       | 2200  |
| W la  | W  |  |  |       |  | 1215  | 1458  | 1258  |
| Au ma | Au |  |  |       |  | 1061  | 1101  | 1103  |

<sup>1</sup> extrapolated across absorption edge

# Appendix D

## Problems with the Analysis

Quantitative analysis is always beset with many difficulties and it is often difficult to pinpoint the cause of "bad" analyses. What is a bad analysis? This is usually based upon one or two tests: is the weight percent elemental (or oxide) totals very close to 100%? And if the material should have a certain stoichiometry, does it?

More often than not, the effect is the sum of more than one problem. Several things need to be checked. First of all, start by examining the standards.

Are the standards really "good" standards (both on their own, and for your particular suite of unknowns). Have their compositions been determined by a reliable analytical method? Traditionally, major element concentrations of standards have been determined using a bulk chemical technique, commonly "wet chemistry". The greatest problem is with natural minerals and glasses, which may be somewhat heterogeneous, and may have a sample "split" that is of a slightly different composition compared to the "official" composition. It is in situations like this, the use of secondary standards can really be helpful in determining what is going on. If the primary and secondary standards do not agree with each other, there is a problem: which is correct?

Traditionally, analysts have run analyses of several standards, using another as the standard for a particular element, and then repeat with different standards, to see which gives the "most correct" values the most times. The Probe for EPMA "Evaluate" application takes this one step further and plots up all standards for one element against each other (stated composition vs. ZAF corrected intensity), giving a clear indication of which standards are "good" (consistent with each other) and which are not (i.e., off the 1:1 line).

Also, are the concentrations entered in the Standard.mdb database correct and without typographical errors? And were counts acquired on the actual intended standard? It is easy to get lost at 300-400 magnification when using a standard mount which contains many standards (EDS helps here).

Some other things to consider :

1. Is the operating voltage correctly specified? Is the correct x-ray line tuned for each element? Check the on-peak position offsets from the Peak/Scan Option dialog and see if they are reasonable. The program will usually type a warning if the actual and calculated peak positions are very different. Be sure that the spectrometer is not tuned on a nearby line of another element if using multi-element standards.
2. Be sure that no "bad" data points are in the standards samples used for the quantitative calibration. The best way to check for this is to analyze each standard and examine the results to look for points with obviously bad or low totals (epoxy, bad surface polish, bad carbon coat, etc.). If a bad point is found, one can disable it. Remember, one can always enable data later on. A disabled point is simply not used in calculating the analytical calculations but is still present. A good rule of thumb is to only disable points that have low totals since generally most of the problems mentioned above will result in fewer x-ray counts. Avoid disabling points just to get better agreement between the primary and secondary standards. Points that have high totals should not be

arbitrarily disabled. It may be necessary to look for other problems such as points with low totals in the primary standards.

3. Look for interferences on the analyzed elements. One easy way to do this is to use the Interferences menu item in the Standard program. One can also perform a wavelength scan and display possible interfering peak markers. If the element causing the interference is present in significant concentrations in your unknowns, and is not being analyzed for, it may be necessary to add the interfering element to the run by creating a new sample with the interfering element as an analyzed element. Be sure that the proper standards are available to use for the interference correction.

4. It would be useful to examine the precise choices for x-ray peaks; did the software/firmware routine choose the best peak position? Sometimes the algorithm doesn't. You want to use the center of the peak quasi-plateau where either any small spectrometer drift or peak/shape effects will be minimized. Probe for EPMA has a "post-scan" confirmation option where you can verify this. This issue would be for the main elements in a multi-element phase, and not that important for minor or trace elements where the determination of the background is the critical endeavor.

5. If none of the above suggestions seem to help, try acquiring the standards again. Probe for EPMA uses an automatic standard drift corrections which can make a significant difference in situations where one or more of your analytical channels is drifting. Note that since the program will perform an automatic drift correction not only on the standards, but also the interference standards and the MAN background standards, it might be also be necessary to run additional sets of those standards or MAN standards.

6. In the case of trace or minor elements, also check to see that none of the off-peak positions are interfered with by another peak. This can cause a reduction in the on-peak counts, sometimes enough to result in a negative k-ratio. Always run at least one wavelength scan on a sample, using the same count time as your quantitative analyses, and if a peak is seen interfering with the off-peak marker, use the Low and/or High buttons in the Graph Data window to select a new off-peak position that is not interfered with.

10. Detector electronics: Did you explicitly check/set the bias, gain, baseline? If operating in differential mode, did you verify the window width? Are you aware that high count rates on a standard will give you artificially low counts, as the pulse shifts to the left, potentially outside the lower window? To prevent this occurrence always check your PHA setting by running the Graph PHA Distribution button in the PHA setting dialog.

11. If you have high totals, have you considered the possibility of secondary fluorescence, if you are measuring small phases within a matrix or near another phase? Normally this is a small effect on the total but when measuring trace or minor elements it can be a source for large relative errors.

In addition, to solve problems with trace analysis, it is always useful to run at least one "blank" that is similar to your unknown sample, at least in terms of average atomic number, if not actual chemistry. For example, when analyzing trace elements in quartz, try to run an unknown analysis on a "pure" synthetic quartz sample, one that is known to contain known concentrations of the trace elements. The point is to try to determine how closely one can measure "zero" concentrations.

# Appendix E

## Problems with the carbon or conductive coating

There are two main problems caused by differences in the thickness (or absence) of the carbon or conductive coating that can affect the emitted intensity of the analyzed x-rays. The first is from differential absorption of low energy x-rays from different thicknesses of carbon coat. The second is from retardation of electrons from differences in thickness (or absence) of the carbon or conductive coating. The first is mainly a problem only for very low energy x-rays such as oxygen or nitrogen, the second is mainly a problem for high energy x-ray at low overvoltages,

In the first, soft x-rays emitted from the sample are absorbed by the coating. Hence if the absorption is significant enough, then differences in the thickness of the coating between the standard and the unknown will produce a difference in the intensity of the x-ray detected from the sample and standards. Due to the non-linear and complex nature of the absorption (absorption edges) it is not possible to make a general statement regarding the magnitude of the effect. The following table gives several examples for absorption of several commonly measured soft x-rays in carbon coats of three different thicknesses:

Percent x-ray transmission (assume density of carbon is 2.7 gm/cm<sup>3</sup>):

|                       | 10 nm (carbon) | 20 nm (carbon) | 40 nm (carbon) |
|-----------------------|----------------|----------------|----------------|
| Ti K $\alpha$ (19.76) | 0.99994        | 0.99989        | 0.99978        |
| Si K $\alpha$ (356.8) | 0.99903        | 0.99807        | 0.99615        |
| Al K $\alpha$ (557.2) | 0.99849        | 0.99699        | 0.99400        |
| Mg K $\alpha$ (904.8) | 0.99756        | 0.99512        | 0.99027        |
| Na K $\alpha$ (1534)  | 0.99586        | 0.99175        | 0.98356        |
| F K $\alpha$ (6366)   | 0.98295        | 0.96620        | 0.93355        |
| O K $\alpha$ (12,380) | 0.96712        | 0.93533        | 0.87484        |
| N K $\alpha$ (25,490) | 0.93349        | 0.87140        | 0.75935        |

Another way in which the carbon coat can affect the emitted intensities is due to the slowing down (retardation) of the primary beam electrons in the coating. This slowing down of the primary electrons results in an effective loss in ionization efficiency by the incident electrons. For x-rays with a moderate to high overvoltage this reduction in ionization efficiency is negligible, but for elements with an over voltage less than 1.5 (for example Ni K $\alpha$  excited at 10 keV), not accounting for the differences in the thickness (or absence) of a carbon or conductive coating could affect the generated intensity significantly.

# Glossary of Terms

## ALPHA-FACTOR

Early EPMA workers did not have the luxury of high speed computers, and the complex rigorous physics-based matrix corrections could not be easily performed in an applications-oriented probe lab. Alpha factors or coefficients for matrix correction in binary compounds (e.g., Fe on Ni Ka, Ni on Fe Ka) were initially conceived of by Castaing in his 1951 Ph.D. dissertation. Ziebold and Ogilvie (1964) revisited alpha factors in the popular scientific literature as the development of commercial electron microprobes took off. Shortly thereafter, efforts to consider phases of more than 2 elements spurred the development of beta-factors. This was applied to geological EPMA by Bence and Albee (1968) who developed oxide alpha factors; this paper is one of the most frequently cited references in geology (over 1800 citations between 1982-2001). Several decades later, it was recognized that the alpha-factor approach could be fine-tuned for improved quantitative results using additional fit coefficients (linear and polynomial) (Rivers, pers. comm., 1990, and Armstrong, 1991).

These factors are derived by fitting concentrations vs. alpha-factors, from calculated or measured k-ratios in a system of binary standards covering a range of composition. See also beta-factors. The expression for the calculation of a single alpha-factor is shown below:

$$\frac{\left(\frac{C}{K}\right) - C}{(1 - C)} = \alpha$$

where :            C        is the elemental weight fraction of the element  
                          K        is the calculated or measured k-ratio at that concentration

The calculation of the matrix correction for an unknown sample is shown below:

$$C_A^u = \frac{C_A^s}{Beta_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)} Beta_{\lambda_A}^u$$

where :            Beta <sub>$\lambda_A$</sub> <sup>u</sup>        is the unknown beta-factor  
                          Beta <sub>$\lambda_A$</sub> <sup>s</sup>        is the standard beta-factor

## ANALYTICAL ERROR

This calculation is for analytical sensitivity of each line (= one measurement), considering \_both\_ peak and background count rates (Love and Scott, 1974). It is a similar type of statistic as the 1 sigma counting precision figure, but it is presented as a percentage in the Probe for EPMA statistical printout.

$$\mathcal{E}_{P-B} = \frac{\sqrt{\frac{N_P}{t_P^2} + \frac{N_B}{t_B^2}}}{\left( \frac{N_P}{t_P} - \frac{N_B}{t_B} \right)}$$

Where :  $N_P$  is the total peak counts

$N_B$  is the total background counts

$t_P$  is the peak count time

$t_B$  is the background count time

## ANGSTROM

Unit of measure for x-ray wavelengths. Wavescan intensities and KLM markers may be displayed in spectrometer or angstrom units.

The conversion of spectrometer to angstrom units is by default based on the calculated spectrometer offset from the theoretical peak position compared to the actual or measured peak position using the following expression:

$$\Delta P = P_T - P_A$$

where:  $P_T$  = the theoretical spectrometer peak position

$P_A$  = the actual or measured spectrometer peak position

This assumes that any inaccuracy in spectrometer position from the exact theoretical position is a constant offset regardless of the spectrometer position. Usually this is sufficient for most purposes, where the spectrometers are not too far out of alignment. Of course, you will determine the exact peak positions in practice; the accuracy discussed here affects the initial locating of spectrometers and labeling of peaks in the Plot! displays.

For ultimate accuracy in initial spectrometer locating and peak labels in Plot!, use of the multiple peak calibration option in the StartWin application will produce a calibration table that will be used by Probe for EPMA in calculating a variable spectrometer offset based on the spectrometer position for each spectrometer and crystal combination and for each Ka, Kb, La, Lb, Ma or Mb family. See the PROBEWIN.INI keyword "UseMultiplePeakCalibrationOffset" for more information on the use of this variable spectrometer offset feature.

## APF (Area Peak Factor)

The APF values are used to correct light element intensities (e.g., O ka, N ka, C ka and B ka) for peak shape (and shift) effects that can affect quantitative analysis. Since the degree of shape and shift is to a first order dependent only on the element which is bonded to the light element (and to a lesser extent on the coordination or valence of the element) the binary effect (as measured on pure oxide, carbide, nitride or boride, etc. compounds) is first characterized using the following equation.

$$APF = \frac{I_U^I}{I_U^P} \cdot \frac{I_S^P}{I_S^I}$$

where:  $I_U^I$  is the integrated intensity for the “unknown”

$I_U^P$  is the peak intensity for the “unknown”

$I_S^P$  is the peak intensity for the “standard”

$I_S^I$  is the integrated intensity for the “standard”

The total peak shape/shift effect can be summed together to obtain an “average” APF for a complex compound by summing the weight fractions of the “binary” APF values.

The “binary” APF values are taken from the EMPAPF.DAT file and the default values were determined by Bastin on a Cameca microprobe. Since the values depend on the spectrometer and crystal resolution, each analyst should measure the values on their own instrument by performing careful wavelength scans on both “standard” reference peaks and “unknown” reference peaks. For example, if it necessary to correct for oxygen peak shape changes in an unknown compound containing both Si and Al bonded to oxygen (SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>), the user should measure a reference oxygen peak, usually Fe<sub>2</sub>O<sub>3</sub> (used by Bastin) or MgO (used by Donovan) and also the oxygen peaks on SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>.

In the example given, MgO or Fe<sub>2</sub>O<sub>3</sub> are the standard reference  $\frac{I_S^P}{I_S^I}$  and Al<sub>2</sub>O<sub>3</sub> and

SiO<sub>2</sub> are the binary references  $\frac{I_U^I}{I_U^P}$ .

The Model Backgrounds dialog provides a quick way to determine integrated peak intensities and provides the P/I and I/P values which can also be output to a text file, though more sophisticated applications will be necessary if other peaks are present (e.g., Igor).

## ATOMIC PERCENT

The atomic percent is calculated based of the fraction of the atomic weight of the element. Also called "elemental mole" percent. Since the calculation normalizes the data to a 100 % total, it is recommended that the calculation be only performed on samples which total close to 100 % (e.g., 98-102). The calculation is as follows :

$$A.P. = \frac{100}{\sum_{i=1}^n \left( \frac{W_i}{A_i} \right)} \cdot \frac{W_i}{A_i}$$

where :  $W_i$  is the elemental weight percent of the element  
 $A_i$  is the atomic weight of the element

## AVERAGE

Also known as the mean or arithmetic mean. The average is calculated by simply dividing the sum by the number of values. The calculation is shown below.

$$A = \frac{\sum_{i=1}^n X_i}{n}$$

where :  $X_i$  are the values to be averaged  
 $n$  is the number of values to be averaged

## AVERAGE ATOMIC NUMBER

Also known as Z-bar or Mean Atomic Number (MAN). The sum of the elemental weight or atomic fractions of the element atomic numbers of a sample.

The sum of the weight fractions of the atomic numbers of a sample is applicable for modeling the background continuum and is used in the MAN background correction iterated polynomial calculation by Probe for EPMA to approximate the average z-bar (electron fractions are actually more rigorous).

On the other hand, the sum of the atomic fractions of the atomic numbers of a sample is applicable to stopping power calculations and is used in Monte Carlo calculations of electron trajectories.

The weight fraction calculation is quite simply :

$$\bar{Z} = \sum_{i=1}^n \left( \frac{W_i}{\sum_{i=1}^n W_i} \right) \cdot Z_i$$

where :  $Z_i$  is the atomic number of the element  
 $W_i$  is the elemental weight or atomic percent of the element

## BETA-FACTOR

Beta-factors are the normalized sum of the weight fraction weighted alpha-factors for a given compound composition. These are utilized in the quantitative calculation of unknown compositions. See alpha-factor above.

## BRAGG'S LAW

In 1913, the father-son team of William H + William L Bragg built an X-ray spectrometer, using NaCl to resolve Pt X-rays. For this they received the Nobel Prize in 1915.

$$n\lambda = 2d \sin \phi$$

where :

$n$  is the order of the x-ray (1st, 2nd, etc.)

$\lambda$  is the wavelength in angstroms

$2d$  is the analyzing crystal 2d spacing in angstroms

$\phi$  is the angle of diffraction

There is a more precise form of Bragg's Law that allows for refraction of x-rays by the crystal or diffractor; this can be shown as a modification of the 2d such that  $d' = d[1-(k/n^2)]$  where  $n$  is the order of reflection and  $k$  is a constant for each crystal.



Plot from Julien Allaz

These constants range from  $6 \times 10^{-5}$  for LiF to  $2.2 \times 10^{-3}$  for TAP (Reed, 1993). They can cause higher order lines to "cross over" and appear on the opposite side of a peak, such as the 3rd order P Ka upon the F Ka for LDEs. Probe for EPMA automatically uses this precise calculation.

## CATION

The metal component of a chemical compound. For example, in the compound  $\text{SiO}_2$  the Si or silicon is the metal or cation and the  $\text{O}_2$  or oxygen is the anion. In this example, the stoichiometry (ratio) for Silicon to Oxygen is 1 to 2 and the subscript for Silicon is 1 and for oxygen is 2.

## CHARACTERISTIC X-RAY

Inner shell electrons can be knocked out by high energy electrons, with an electron from a higher shell taking its place. The excess energy of the second electron is emitted as a photon which either is converted internally into an Auger electron or an x-ray with a characteristic energy/wavelength. See Fluorescence Yield

## CONTINUUM

The continuum is the background radiation produced during the production of the characteristic x-rays. It is generated as the high energy electrons from the gun

interact with the nuclei of the target atoms, and their trajectories are altered by the positive charge of the nuclei. When the electron trajectory bends, x-ray radiation is emitted at an energy level that ranges on a continuous scale ( hence continuum) from a minimal low value, up to a maximum of the acceleration voltage value. Also known as Bremstrahlung (literally "braking radiation").

## CPS

Counts per second. X-ray count rates are always given in cps units to normalize for different count times. To get the actual counts multiply the displayed x-ray counts by the count time used (and be sure that the nominal current is set at the actual value used to obtain the unnormalized intensity data).

## DEADTIME

The interval between the arrival of an x-ray pulse signal and the time that the system (including detector and amplifier electronics) is ready to receive the next one is defined as the deadtime. The deadtime correction is only significant at count rates above 10K to 20K counts per second (cps). For example, if the deadtime is 1 us and the count rate is 10K cps, the count rate will be reduced by 1%.

The best way to determine the deadtime constant is to measure it by the function of count rate vs. beam current. The normal deadtime correction is given by the expression below:

$$I = \frac{i}{1.0 - i\tau}$$

Where :       $i$       is the raw measured counts in cps  
                    $\tau$       is the deadtime constant in seconds  
                    $I$       is the deadtime corrected counts in cps

An optional high precision expression is also available for very high count rates (> 50K cps) and is shown here :

$$I = \frac{i}{1.0 - \left( i\tau + i^2 \frac{\tau^2}{2} \right)}$$

Note that Probe for EPMA supports the use of a hardware "enforced" deadtime for rigorous correction of the deadtime effect if available on the microprobe (e.g. Cameca SX-50/51/100). In this situation, an arbitrary deadtime constant, that is larger than the largest deadtime measured on the spectrometer, is utilized by the amplifier electronics to gate off any pulses that are received by the amplifier after the initial pulse, within the value specified. This has the net result of masking the "intrinsic" deadtime of the system, which has been noted by several observers to be quite dependent upon the energy of the pulses received from the detector. Hence, the "enforced" deadtime value is used in software to correct the intensities without regard to any dependence upon pulse energy.

To facilitate the measurement of the intrinsic deadtime of your PHA amplifiers, an easy to use but rigorous Excel spreadsheet created by Paul Carpenter is available from Probe Software.

## DETECTION LIMIT

The lowest concentration that can reliably be detected above the level of the background. To achieve low (10-100 ppm) detection level requires a large number of counts which means running at maximum currents (hundreds of nA), long times (100s of seconds) and the highest accelerating voltage possible. It has been shown that ( $P^2/B$ ) is the "figure of merit" for trace element analysis, and it increases for many elements with increasing accelerating voltage.

Probe for EPMA offers two versions of the detection limit calculation. The first is for each line ("single line") which means that in a heterogeneous material, it is the detection limit for each measured spot. Alternatively, if many counts are acquired on a homogeneous material, then they can be averaged, yielding a lower detection limit. The software provides such a detection limit for an average sample at a variety of confidence levels (normal level is 99%) utilizing a T-test. (see T-test)

## EDS

Energy Dispersive Spectrometry. This method utilizes a solid state detector that operates similarly to the gas-filled detector in the electron microprobe spectrometer, with important distinctions. In the solid state detector, the x-rays knock out electrons (specifically the photoelectric effect), producing electron-hole pairs, each part migrating to + or - surface of the voltage biased Si(Li) or Ge crystal. The number of electron-hole pairs is directly proportional to the x-ray energy, and a small pulse is generated. This is immediately amplified in an adjacent FET (field effect transistor), and then the signal proceeds to a pre-amp, and then to the processing and counting electronics. The result is a histogram of energy levels, or spectrum.

Many electron microprobes also have EDS detectors that are used to "fingerprint" materials prior to WDS analysis. EDS is also combined with WDS on some instruments for increasing the number of elements that can be elementally mapped. Probe for EPMA offers additional options for both acquiring elements for quantitative analysis, as well as acquiring and storing EDS spectra when each WDS measurement is made (useful for off-line data processing).

## EPMA

Electron Probe Micro Analysis. Differentiated from SEM/EDS analysis by the use of high precision, high resolution, high sensitivity vertical WDS spectrometers.

## ESCAPE PEAK

In both the EDS detector and the WDS gas-filled proportional counter, there is the statistically probability that besides photoelectrons (and holes in EDS, and Ar cations in P10-filled WDS detector), there may be some secondary fluorescence. That is, the incident x-ray (let's say Fe Ka for example) could knock out an inner shell electron and produce another x-ray (Si Ka in typical Si-Li detector; Ar Ka in P10-filled detector). Most of these x-rays will be "consumed" within the detector, yielding photoelectrons, but some will find a means to escape the detector. Their characteristic energy will be lost to the detector for this one x-ray, resulting in one pulse that is at a lower energy level than is expected, which will then show up as a small peak at a lower voltage.

Taking the Fe Ka X-ray as an example, in the Si-Li EDS spectrum, there will be a small peak at 4.66 keV, 1.74 keV below the Fe Ka peak of 6.40 keV. In the WDS detector using P10 gas (90% Ar, 10% CH<sub>4</sub>), Ar-escape peaks will occur on the PHA

energy display at a proportionally lower value, i.e., if the Fe Ka pulse is centered at 2.5 volts, then there will be a small Ar-escape peak at around 1.3 volts, because Ar Ka = 2.96 keV, so the escape peak will be  $6.40 - 2.96 = 3.44$  keV;  $3.44/6.40 = 0.54$ ;  $0.54 * 2.5$  volts = 1.34 volts.

In WDS, sometimes it is impossible to acquire the complete Ar-escape peak, because the lower part is too low in energy and falls below the baseline cutoff. In that case, it is recommended that the baseline be raised to cut off all of the escape peak; either acquire all of it, or none of it. See section on PHA for more details.

## FIDUCIAL

Refers to the physical marking of a sample mount with a number of reference marks, so that all data can be referenced directly to those marks on the sample surface. This is used to provide a method of re-locating specific positions even after the sample has been removed from the sample holder. This method can also be used to relocate coordinates from an entirely different coordinate system such as a digitizing microscope stage. In the Probe for EPMA software package all samples using this feature must have 3 fiducial marks. This is because the program assumes that the positions are digitized in three dimensions (fixed to x, y and z).

## FLUORESCENCE YIELD

The fraction of inner shell ionizations that yield characteristic x-rays; the remaining ionizations yield Auger electrons of very low energy.

## GRID

(Grid File) A file format used by SURFER (Golden Software) to store single precision floating point data with 3 coordinates of information (X, Y and Z). The file is a binary file and may be read from VB, FORTRAN, C or Pascal. A grid file always has the extension ".GRD". The format (v. 6 Surfer) is as follows :

|                |  |
|----------------|--|
| cdum           | a 4 byte string containing the characters 'DSBB' (not null terminated) |
| ixgrid, iygrid | two integer*2 values containing the grid density                       |
| xmin, xmax     | two real*8 values containing the X data minimum and maximum            |
| ymin, ymax     | two real*8 values containing the Y data minimum and maximum            |
| zmin, zmax     | two real*8 values containing the Z data minimum and maximum            |
| data           | ixgrid * iygrid real*4 values containing the Z data values             |

Starting with Surfer 7, a new GRD file format is utilized which is also supported by Probe for EPMA and Probe Image (a full featured image acquisition and analysis application available from Probe Software). The older grid file format used by Surfer 6 and under is supported by Probe for EPMA for output but not by Probe Image. See the PROBEWIN.INI file parameter "SurferOutputVersionNumber" in the [software] section to define the version of the GRID file format for PFE.

## HI PEAK COUNTS

Hi Peak counts are one background (continuum) measurement usually above the peak position, although the "high" adjective is strictly relative to the "low peak", so in practice could be below the peak position, but higher than the "low peak" position.

In Probe for EPMA the "high" peak position is always measured first, following the on-peak measurement.

## INTERFERENCE

An analytical situation when a peak from another element overlaps a primary analytical line or background measurement position used in the data acquisition. Ignoring this interference will often result in an incorrect analysis. This is sometimes seen as a high total when the on-peak interference is large or as a negative raw k-ratio (KRAW) when the off-peak position is interfered with. However when the interfered element is only a minor or trace quantity, even a small interference can result in a large error on the interfered element without affecting the overall total significantly. These primary line interferences can be completely and quantitatively corrected for using Probe for EPMA. Be aware however, that interferences on your off-peak positions need to be checked for, before acquiring any off-peak sample data, especially for trace and minor element analyses.

In some cases where one has the choice between use of two diffracting crystals, the one with the lowest 2d will provide the maximum spectral resolution available for minimizing overlap of peaks.

## INTERFERENCE STANDARD

A standard used for the quantitative interference correction in Probe for EPMA. A standard used for the interference correction can also be used for the analytical calibration and need only be analyzed once, although additional interference standardizations can be used in the automatic drift corrections if they are available (this is done automatically if the interference is declared in the Analyze! | Standard Assignments). To use the quantitative interference corrections in Probe for EPMA you must acquire counts on both the interfered element (obviously) **and** the interfering element. To use a standard as an interference standard, it must contain a known concentration of the interfering element, **and** it must not contain the interfered element, nor can it contain any other elements that interfere with the interfered element. You can add additional standards to a Probe for EPMA run using the Standard | Add/Remove Standards To/From Run menu.

## ITERATION

A technique which is used to calculate a result when you have more unknowns than coefficients. Probe for EPMA used an iterative loop for the ZAF and Phi-Rho-Z matrix calculation and an additional iteration loop around that to calculate other compositionally dependent parameters such as the spectral interference, MAN background correction and the APF correction.

## K-FACTOR

Initial probe work by Castaing was metallurgical and the standards were pure elements, and so the K-ratio was defined relative to a pure element standards. In the situations where the standard is not the pure element, a means to use the normal form K-ratio is to normalize the standard, using the standard k-factor. This is the concentration of the element in the standard (as a fraction) divided by the ZAF correction of the element in the standard. See also raw k-ratio, unknown k-ratio and ZAF. The expression is :

$$K_{\text{std}} = \frac{C_A^S}{[ZAF]_{\lambda_A}^S}$$

where :  $C_A^S$  is the concentration of element A in the standard  
 $[ZAF]_{\lambda_A}^S$  is the ZAF correction for element A in the standard

## K-RATIO

See raw k-ratio, unknown k-ratio, k-factor and ZAF.

## LOW PEAK COUNTS

Low Peak counts are one background (continuum) measurement usually below the peak position, although the "low" adjective is strictly relative to the "hi peak" position, so could be above the peak position, but lower than the "hi peak" position.

In Probe for EPMA the "high" peak position is always measured first, following the on-peak measurement, followed by the "low" peak measurement.

## MAC

Mass Absorption Coefficient. A physical constant in units of  $\text{cm}^2/\text{gm}$ , which describes the amount of absorption of an x-ray of specific energy in a pure element matrix. Used by the ZAF and Phi-Rho-Z matrix corrections.

## MAN

Mean Atomic Number. The average atomic number of a sample used in the on-peak background correction. A calibration curve type of background correction in which the background count rate is measured while the spectrometer is tuned to the element of interest on samples of varying atomic number each not containing the element of interest. An additional option has been added whereby the background count rates at the characteristic peaks in samples containing the element of interest are found using the measured off-peak backgrounds, and similarly utilized.

A function is then fitted to a plot of counts vs. average atomic number and used in the compositional iteration to calculate a background correction. This method works quite well for major and even minor elements in low to moderate average atomic number matrices, since the background of a sample is almost entirely dependent upon atomic number. See the above section on MAN backgrounds.

## MATRIX CORRECTION

This is an algorithmic calculation to correct for the absorption, fluorescence and atomic number effects of other elements upon the element of interest in the material. In his 1951 Ph.D. thesis, Raymond Castaing laid out the two approaches that could be used to apply matrix corrections to the data: an empirical "alpha factor" correction for binary compounds, where each pair of elements has a pair of constant a-factors representing the effect that each element has upon the other for measured X-ray intensity; and a more rigorous physical model explicitly accounting for absorption and fluorescence in the specimen. This later approach also includes atomic number effects and became known as ZAF correction. Probe for EPMA supports the various

matrix corrections, i.e., alpha, Bence Albee, and several types of both ZAF and Phi-Rho-Z algorithms. See those terms.

## MULTI-SELECT

Multi-select means that the list box supports the selection of more than one item. To select a range of items click the first item in the list and then holding the <shift> key, select the last item to be selected. To select individual items, click while holding the <ctrl> key down.

## NOMINAL

Nominal is defined in the Oxford American Dictionary as "in name only" or "a token fee". However, in the Probe for EPMA software it is meant to indicate that the value referred to is not necessarily the actual value. This use of the word is also seen in the lumber trade, where a 2 by 4 is the nominal dimension, but the actual dimension is often less.

For example, in this sense, the *nominal beam current* is not the actual measured beam current but only a close approximation that is used to calibrate the beam drift correction. The beam drift correction only uses the nominal beam current to set the magnitude of the correction. If the nominal beam current is close to the actual measured beam current then the correction is close to 1.0 and the beam drift corrected counts displayed will be close in magnitude to the counts displayed on the screen scalers.

## OFF COUNTS

Off Counts are the values used for the background (continuum) at the characteristic peak position. They are the measured at the peak position (if MAN background) or calculated from one or two off-peak positions (if off peak method).

## ON COUNTS

On Counts are the net or background-corrected counts at the peak position, and result from the characteristic x-ray intensity minus the background intensity.

## ON PEAK COUNTS

On Peak Counts are the total or gross counts at the peak position, and are composed of x-rays of the continuum (background) upon which the characteristic x-ray intensity rides.

## ONE SIGMA

This is a statistical value giving precision based upon counting statistics, i.e. count rate and counting time and is calculated from the square root of the total counts. It is sometimes also referred to as a "standard deviation" even though one can calculate a value based upon only one measurement (see Poisson Statistics) Use the one sigma value to evaluate whether a given average count rate represents a homogeneous set of sample data.

Standard deviations (SDEV) larger than the one sigma value (1SIG), i.e., SIGR >1, indicate the possibility of actual compositional variation within the data set. The 1 sigma value is the predicted standard deviation based on the count rate and counting

time. It is calculated by dividing the square root of the count rate by the square root of the average counting time.

$$\sigma = \frac{\sqrt{i_{cps}}}{\sqrt{t_s}}$$

where :  $i_{cps}$  = intensity in counts per second  
 $t_s$  = count time in seconds

## OXIDE

Geologists and geochemists typically report their data in wt.% oxides, as oxygen is the dominant element in earth materials and many elements in nature are bonded with oxygen in crystal lattices or glass. Thus, silicon (Si) is reported as silicon dioxide or silica (SiO<sub>2</sub>), Ca as CaO, Na as Na<sub>2</sub>O, phosphorus as P<sub>2</sub>O<sub>5</sub>. These are discrete, stoichiometric ratios with charge balance.

Some elements can easily exist in different valence states (e.g. Fe, Cr) but for convenience sake are nominally assumed to exist in one form (e.g., FeO, Cr<sub>2</sub>O<sub>3</sub>). They can be easily "translated" to elemental weight values; simply create a fraction using atomic weights from the periodic table, with numerator the element wt. % and denominator the oxide wt.% summed up. Thus to determine Si from SiO<sub>2</sub>, one would divide 28.09 by (28.09 + [2 x 16.00]), yielding the fraction 0.4675. Thus, something at 100 wt.% SiO<sub>2</sub> is actually 46.75 wt.% Si and the balance oxygen by weight. And elemental wt.% values can be converted to oxide wt.% by the inverse. In a Probe for EPMA run, the sample compositions are reported as oxides if the Display As Oxides option is selected for that sample.

## P/B

Peak to Background Ratio. Strictly the total counts at the peak position (which will also include the continuum contribution in addition to the characteristic x-ray counts). A measure of the sensitivity of the analysis. To improve your detection limit, you will want to increase the peak to background ratio. This can be accomplished by using the analyzing crystal with the highest theta available (for example run K K<sub>α</sub> on LiF rather than PET) by using the highest accelerating voltage reasonably feasible, and using lower noise flow detectors rather than sealed detectors. The P/B is calculated as :

$$P / B = \frac{I_P + I_B}{I_B}$$

where :  $I_P$  is the background corrected peak intensity  
 $I_B$  is the background continuum intensity

## PHOTOELECTRIC EFFECT

The photoelectric effect was discovered in 1887: light (photon of energy > metals absorption edge) falling on metal surface ejects electrons from the metal. X-rays

hitting a metal, a semi-conductor, or a gas will similarly eject electrons from the materials, and the electrons are therefore called photoelectrons.

## PHOTOELECTRONS

The electrons knocked out of materials (e.g., SiLi detector, Ar molecules in P10 gas) are photoelectrons.

## POISSON

X-ray production is a random process, where each repeated measurement represents a sample of the same specimen volume (for a homogeneous specimen). The expected distribution can be described by Poisson statistics, which for large number of counts is closely approximated by the "normal" Gaussian distribution ("bell curve"). For these distributions, 1 sigma = square root of the counts, and 68.3% of the sampled counts should fall within  $\pm 1$  sigma, 95.4% within  $\pm 2$  sigma, and 99.7 within  $\pm 3$  sigma.

## PHA

Pulse Height Analysis (or Analyzer). The x-rays that satisfy Bragg's Law reach the detector inside the spectrometer housing, where they ionize the gas (produce photoelectrons and Ar cations) and create a small analog electronic signal. This signal passes to a pre-amp and then through an electronic circuit that shapes it into a digital pulse for input to the x-ray counter scalers. In a properly configured detection system, the bias in the detector is set so that the ionization occurs in the "proportional range", and higher order ( $n > 1$ ) diffractions that also satisfy Bragg's Law (and are discrete fractions of the wavelength of the desired element), will have discrete multiples of the pulse energy. Thus, if the first order peak has a peak at 2.0 volts, the 3rd order peak would have a peak at 6.0 volts. This provides one way to reduce or eliminate higher order interferences: setting a window so that the desired element's x-rays are passed through, but that the higher energy interfering x-rays are rejected. This is known as "differential" mode, whereas under "integral" mode there is no window. Also, a lower limit or baseline (e.g. 0.5 volts) is set to filter low energy electronic noise. Use of differential mode PHA should be used with care, as there are possible serious errors that can occur to the unwary (see discussion of energy shifts in the manual). Probe for EPMA offers a sophisticated interference correction which in many situation eliminates the need to use differential mode PHA (which can only reduce higher order interferences in any case).

## PHI-RHO-Z

In developing models of x-ray generation, analytical scientists decided that the easiest way was to consider "mass depth" or Rho-Z, the product of the sample density and the linear depth Z. The function that describes the relative intensity of x-ray generation with mass depth, is call Phi. The expression is often written as  $\rho\phi(z)$  Phi (Rho-z). At the surface there is a moderate amount of x-rays produced, but it rapidly increases as the electrons scatter laterally, and then quickly reaches a maximum, and trails off to zero. Some matrix corrections use this explicitly, combining the absorption and atomic number corrections. This is an alternative method to the ZAF matrix correction, which can be more easily verified using so called tracer and wedge experiments.

## POLYGON

An enclosed shape consisting of a number of line segments. **Probe for EPMA Xtreme** supports the automated acquisition of data from within user defined polygon boundaries. The digitized polygon coordinates are automatically saved to the run file as they are acquired and are automatically output when the Surfer .BAS option is selected.

## POLYNOMIAL

A mathematical expression of the form :  $Y = A + BX + CX^2 + \dots$ . The MAN background correction used in **Probe for EPMA Xtreme** uses an iteration of this expression to calculate for major element backgrounds without the need for off-peak measurements. The user may select a 1st order (straight line) or 2nd order (parabolic) fit to the MAN data. The parabolic fit is the default.

## PPM

Parts per million, can be either elemental or oxide. To convert wt. % to ppm, multiply by 10000. Thus 0.1 wt.% = 1000 ppm; 0.01 wt.% = 100 ppm; etc.

## PRIMARY STANDARD

An analytical standard which is *assigned* as the calibration standard for one or more analyzed elements in a quantitative run. Elements in standards not assigned as the primary calibration, but which contain an acquired element can be considered secondary standards.

## RAW K-RATIO

A k-ratio is the ratio of unknown intensity relative to a reference intensity. Several varieties of these are defined. The raw k-ratio is defined as the ratio of the unknown counts to the standard counts for an element. The intensities are usually corrected for beam drift, dead time, background and standard drift. See also unknown k-ratio, k-factor and ZAF. The expression is :

$$K_{\text{raw}} = \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)}$$

where :  $I_A^u(\lambda_A)$  is the unknown intensity for element A at  $\lambda$

$I_A^s(\lambda_A)$  is the standard intensity for element A at  $\lambda$

If the standard were a pure element, then the  $K(\text{raw})$  would be a fraction less than or equal to 1, and very roughly equivalent to the elemental fraction in the unknown. This approximation has become known as "Castaing's first approximation", after the founder of EPMA.

## RELATIONAL DATABASE

A data file storage and retrieval model based on a set of defined relationships between two dimensional tables consisting of fields (table items) and rows (data items). From both the programmer and end user point of view, these tables behave

like spreadsheets where each row is a record and various operations such as browsing and searching can be performed without knowledge of the actual physical file structure.

Unlike other EPMA software programs that write or store the user's data in "normal" computer files, Probe for EPMA uses a robust industry standard (Microsoft Access) file format based on transactional processing, guaranteeing the preservation of the data regardless of power failures, computer crashes, etc.

## SCANNING SPECTROMETER

A spectrometer that can be tuned to a range of analytical x-ray lines. Usually contains two to four analyzing crystals. Scanning spectrometers are required for off-peak background corrections. MAN background corrections can be used for scanning spectrometers to save time and improve precision.

## SECONDARY STANDARD

A standard which is not assigned as a primary standard for a given element (that is, not directly used in the quantitative calculation of unknowns). However, it is useful, if the secondary standard contains a known quantity of the analyzed element, to check the quality of the secondary analysis can be judged by observing the "% VAR" line when an analysis is performed. The "% VAR" is calculated as follows :

$$\% \text{ VAR} = \frac{W_{\text{AVER}} - W_{\text{PUBL}}}{W_{\text{PUBL}}} \bullet 100$$

where :  $W_{\text{PUBL}}$  is the published concentration of the element

$W_{\text{AVER}}$  is the measured average concentration of the element

## SIGMA RATIO

The sigma ratio is simply the measured variance (standard deviation) divided by the predicted variance (square root of the absolute intensity). Note that the predicted one sigma is scaled to the cps/nA specified by the user in Probe for EPMA.

Values for sigma ratio greater than 3 may indicate sample compositional heterogeneity or instrument reproducibility problems.

## SPECIFIED CONCENTRATION

A concentration which is used in the matrix correction calculation to provide a more accurate matrix correction. These concentrations are used in two ways in Probe for EPMA. Standards which contain elements which are not being measured, will have their concentrations automatically loaded by the program for possible use in analyzing a standard.

Second, if you are analyzing an unknown which contains elements that you are not measuring, but whose concentrations you know or can estimate, you can add these elements as specified elements and specify their concentrations. This will result in an improved matrix correction for those elements that you are analyzing for.

## SPECIFIED ELEMENT

An element included in sample matrix corrections that is present in the specimen but is not being measured (reasons: difficult or impossible to measure; concentration is known; stoichiometry is known). It is included to account for its effect on atomic number effects, absorption and fluorescence in the sample matrix, as well as to yield an analytical total more accurately representative of the sample.

## STANDARD DEVIATION

A calculation that essentially describes the error for a single point of a sample. It can only be rigorously calculated when more than one data point has been measured. The calculation is :

$$S.D. = \sqrt{\frac{\sum_{i=1}^n (X_i - \mu)^2}{n-1}}$$

where :  $X_i$  is the data observations  
 $\mu$  is the mean of the data  
 $n$  is the number of observations

When only a few points have been measured and it is necessary to know the confidence that one can assign to the precision, the use of the Student's "t" factor is often applied.

## STANDARD ERROR

A calculation that essentially describes the error for the average of a sample. It is calculated by dividing the standard deviation by the square root of the number of data points. The calculation is :

$$S.E. = \frac{\sqrt{\frac{\sum_{i=1}^n (X_i - \mu)^2}{n-1}}}{\sqrt{n}}$$

where :  $X_i$  is the data observations  
 $\mu$  is the mean of the data  
 $n$  is the number of observations

## STOICHIOMETRY

A fixed atomic ratio between elements. For example the mineral olivine has a stoichiometry of (Fe,Mg):Si:O of 2:1:4 (atomic). A test for a "good" analysis would be to calculate (Analyze!|Calculation Options) the stoichiometry on the basis of 4 oxygens. Values for Si of .997 and sum for Fe and Mg of 1.995 would be generally considered very good.

## STUDENT'S T

The general problem relevant to WDS EPMA of having some level of confidence that a measure is equivalent to another, or larger/smaller than another, is that the sample size is small and the population variance is unknown. This type problem was

first treated in 1905 by W.S. Gossett, who published his analysis under the pseudonym "Student". His employer, the Guinness Breweries of Ireland, had a policy of keeping all their research as proprietary secrets. The importance of his work argued for its being published, but it was felt that anonymity would protect the company.

In statistical evaluation of x-ray count data, an exacting criterion is that of calculating a precise range (in wt.%) and level (in %) of homogeneity. These calculations utilize the standard deviation of measured values and the degree of statistical confidence in the determination of the average.

The degree of confidence means that we wish to avoid a risk (alpha) of rejecting a good result a large per cent of the time (95 or 99%) of the time. Student's t distribution gives various confidence levels for evaluation of data, i.e. whether a particular value could be said to be within the expected range of a population -- or more likely, whether two similar compositions could be confidently said to be the same. The degree of confidence is given as 1- $\alpha$ , usually .95 or .99. This means we can define a range of homogeneity, in wt.%, where on the average only 5% or 1% of repeated random points would be outside this range.

## TIFF

(Tagged Image File Format) An image file format consisting of rasterized pixel color information. Probe for EPMA can also create an OLE script that will automatically create .GRD files that can be imported into CalcImage for quantitative mapping.

## TUNABLE SPECTROMETER

A spectrometer that can be tuned to a range of analytical x-ray lines. Usually contains two to four analyzing crystals. See SCANNING SPECTROMETER above.

## UNKNOWN K-RATIO

The unknown k-ratio is defined as the product of the raw k-ratio and the standard k-factor. It is always a fraction, since the pure element is 1.0. See also raw k-ratio, k-factor and ZAF. The expression is :

$$K_{\text{unk}} = \frac{C_A^s}{[\text{ZAF}]_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)}$$

where :  $I_A^u(\lambda_A)$  is the unknown intensity for element A at  $\lambda$   
 $I_A^s(\lambda_A)$  is the standard intensity for element A at  $\lambda$   
 $C_A^s$  is the concentration of the element in the  
 standard  
 $[\text{ZAF}]_{\lambda_A}^s$  is the ZAF correction for the element in the  
 standard

## WDS

(Wavelength Dispersive Spectrometry) X-ray analysis based on Bragg's law of x-ray diffraction.

$$n\lambda = 2d \sin \phi$$

where :

- $n$  is the order of the x-ray (1st, 2nd, etc.)
- $\lambda$  is the wavelength in angstroms
- $2d$  is the analyzing crystal 2d spacing in angstroms
- $\phi$  is the angle of diffraction

## ZAF

A method for the matrix correction of emitted (as opposed to generated) x-rays from a sample. The method calculates the effects of atomic number (Z), absorption (A) and characteristic (and sometimes continuum) fluorescence (F) separately, hence the name ZAF. To put all of this in perspective, here is the complete ZAF correction expression for x-ray intensities solved for the concentration of an unknown concentration :

$$C_A^u = \frac{C_A^s}{[ZAF]_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)} [ZAF]_{\lambda_A}^u$$

where :  $\frac{C_A^s}{[ZAF]_{\lambda_A}^s} \frac{I_A^u(\lambda_A)}{I_A^s(\lambda_A)}$  is the unknown k-ratio and

$[ZAF]_{\lambda_A}^u$  is the ZAF correction factor of the unknown

$I_A^u(\lambda_A)$  is the unknown intensity for element A at  $\lambda$

$I_A^s(\lambda_A)$  is the standard intensity for element A at  $\lambda$

$C_A^s$  is the concentration of the element in the standard

$[ZAF]_{\lambda_A}^s$  is the ZAF correction for the element in the standard

Because the ZAF correction factor for the unknown is exactly that : unknown, the calculation requires a iterative solution. See also Phi-Rho-Z.

## Z-BAR

See Average Atomic Number.

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