DRAFT August 2004 (REFTEST202.xls)

REFERENCE FILE (.REF) EVALUATION SPREADSHEET

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Introduction.

An Excel spreadsheet to evaluate instrument performance using SRM 676 or SRM1976 corundum data formatted as a .REF file for ICDD Grant in Aid standard submission. An option is available to process data pasted from an ASCII file into the spreadsheet. Evaluation of results is based on values obtained in an Intensity Round Robin and limits set by ICDD for STAR quality patterns.
The spreadsheet employs macros to automate processing the data and operation has been checked with Excel V6 and later with macros enabled.

Aims.

The aim of this spreadsheet is to provide an instrument test routine that can be used in the workplace by anyone who submits powder diffraction data to ICDD and is based on certified reference materials supplied by NIST. By making the routine freely available, it is hoped that any instrumentation problems can be identified in the laboratory and corrected at an early stage and prevent the submission of poor quality data. The routine is of value to anyone who wants to check the performance of their diffractometer. Even corundum powder from sources other than NIST could be used on the basis that any test is better than no test.

Test Scan.

Scan from 5 – 145 degrees 2 theta, take 0.02 degree steps with 2 seconds per step using your usual power and slit settings.

Operation of Spreadsheet.

The Spreadsheet file REFTEST202.xls is the latest version of the evaluation routine and macros must be enabled for it to operate. A macro opens up a .ref file on your computer and loads information from that file into the spreadsheet for processing.

To load a file for evaluation - Hold down CTRL key and press the m key (CTRL + m)
An input box with 3 options is displayed:
Type in a number and click OK.

1. **Option 1** load a .REF file (ICDD ASCII file format).

A .REF file is chosen from a source on your computer via a windows file selection menu. Select the appropriate file and click open. A message is displayed giving the name and path of the file being opened with an option to cancel if the file is not appropriate. Click OK to continue or Cancel to restart with crtl +m. The required data is extracted from the .REF file you select and entered on the reftest spreadsheet. The calculation can take some time especially on older computers – a message indicating the progress is displayed in the toolbar at the bottom left of the spreadsheet.

2. **Option 2** manually enter data (not in the .REF file format)

   Directions are given to enter your data into the spreadsheet. In cells A1 to A26 you need to update the information after the = sign. It is important not to change anything in the cell before the = sign. Note the information in RED font is critical to the calculations and must be updated for the conditions used to gather your data. The information in green font will not affect the results but may be reproduced in the report sheet. All the angle values and corresponding intensity values are added down columns A and B starting at row 28. You can use your commercial software or a freeware file conversion routine such as ConvX.exe to convert your proprietary raw data files into an ASCII text file. The CCP14 site has several file conversion utilities. For Bruker users a spreadsheet

(scanplot101.xls) for displaying .RAW files submitted by Steve Norval is available at http://www.ccp14.ac.uk/ccp/web-mirrors/bca-spreadsheets/ just cut and paste the x-y data. A text file can be opened in Excel via the import wizard and the data cut and pasted into the REFTEST sheet. Once the data has been transferred you can save it as a .REF file using the macro "exprtref" (ctrl+w) to make it available for future use.
Once all the data is in place use ctrl +m and select option 3 to process the file.

3. **Option 3** process manually entered data (or reprocess existing data).
This is useful to reprocess data if you want to try calculating the data for a different slit or standard combination or to process manually entered data.

As the macro proceeds with its extraction some notification messages will occur if file header information is missing from the REF file. You should make note of and correct these omissions in a text editor before submitting your .REF to ICDD. You will be prompted to input any missing information critical for the calculations. (e.g. Tube anode, Count time, Standard Number, Divergence slit)

A message is given if a different slit and standard combination would give improved intensity standard deviations than those picked up for the calculation. This is useful to identify when a variable divergence slit (VDS) system has been set in fixed slit mode for the measurement but the file header records the slit as VDS. Note you should always check that an instrument artifact isn't the cause by referring back to the original measurement conditions for the scan.
You are given an option to change the slit type and may be given an option to change the standard used during processing. If you are not given the option to change standard simply edit cell A1 after the = and include 1976 if using the plate or 676 for the powder. Then reprocess the data using option 3.

Once the processing is complete the routine opens up the Scan plot sheet ready for you to start reviewing the extracted data. Click each tab in turn to see the results.

Spreadsheet TABS

Scan plot - shows the full scan - look out for low angle background problems (humps and bumps due to scattering or high background values) and spikes in the data. Use your usual commercial plotting routine to study problem areas in more detail and ensure that contamination peaks are absent.

024peak - displays the single (024) corundum peak used for FWHM and FOM tests. This allows you to visually check the resolution of the Kα peaks and check for an unwanted Kβpeak (indicated by a small peak to the left of the main peak). If a primary beam monochromator is used then only one peak should be observed and the peak should be fairly symmetrical. If a secondary beam monochromator is used then you should see two peaks separated by a valley. The peak on the right should be half the intensity of the left hand peak.

Rel Int Plot – This is a plot of the intensity of each of your peaks compared to the certified values.The points should all fall within the two blue warning lines if the instrument is in control based on SRM1976 certified intensity values. The red limit lines allow more flexibility especially for powder specimens. Common problems you may see are patterns in the data showing increasing or decreasing slope to the intensity due to an instrumental problem. You should attempt to have these hardware problems corrected or as a last resort apply a mathematical correction to your intensity data.

Displacement – This is a plot of the distance each of your peaks is displaced from its theoretical angle. The points should all fall within the two red limit lines which are set for the data to achieve STAR quality pattern status. Common problems are: a simple shift over the whole angular range easily corrected with an offset; displacement that varies with angle often caused by a one to two theta alignment problem.

FOM Plot - This shows how your intensity data compares with the UK Intensity Round Robin data and should be used for comparison purposes only. This test is most useful to identify any major intensity changes over time or between data submissions. It can also be used to test the effect on intensity/resolution of any changes made to your diffractometer configuration.

Report – This gives a summary results from your extracted data and provides some comparative Round Robin data. A RED note indicates a PROBLEM and BLUE gives a WARNING. The first 20 rows of column A are header information and are followed by the results which are explained below.

counts/sec/mA and Pk/Bg
These are from the intensity round robin test 1 and allow you to see how your intensity and peak to background ratios compare with UK and USA averages.

Intensity FOM gives the value of the Jenkins & Schreiner FOM

$$FOM = \frac{MW}{\sqrt{(W(M+4B))}}$$ M = Net intensity in cps; B = average background; W = FWHM

This value is used in the FOMplot sheet which makes it easier to make comparison with the round robin data.

Ave Intensity This is the average of the relative intensities and should be 1.0 - any significant difference indicates a pattern or scatter in your intensity data

Int Std Deviation
This is the standards deviation of the intensities from all the peaks the value should be low and within the round robin ranges. High values indicate scatter or a pattern in the data.

Point Intensities
This identifies if intensity falls outside the error bars and should read "OK" if not check out the Intensity plot.

Alpha2/Alpha1
This is the intensity ratio of the K∝1 and K∝2 and should be 0.50 unless a primary beam monochromator is used.

Valley/A2 this is the ratio of the intensity of the K∝2 peak to that in the valley between the K∝1 and K∝2 peak. The higher the value the better the resolution but beware too high could mean the receiving slit is smaller than the image from the divergence slit.

Ave Displacement Takes an average of the angular displacements of all the peaks and ideally should be zero.

Displacement gives a performance message and should read "OK" if not check out the displacement plot.

FWHM This is the full width at half maximum of the 52.56 degree 2 theta peak (Cu Kα)

COMMENT This gives the standard and slit combination used in the calculation and suggests a better combination if there is one.

Reftest - this is the spreadsheet that holds the working data. It is processed and displayed on the other sheets and the only changes you should make ever make are in columns A and B to manually input data.

KNOWN INCONVENIENCES

Only Cu, Co or Cr radiation supported
SRM 676 high angle intensities are not certified. Values are taken from calculated data!

Report errors or suggest improvements

Report errors or suggest improvements to Dave Taylor by e-mail djtaylor@lineone.net