



What is DTSA-II?

- Power tools for microanalysis...
- Tools for
 - Quantification of almost any vendors EDS spectra
 - Simulation of both simple and complex x-ray microanalysis experiments
 - Spectrum manipulation
 - Generation of publication quality graphics
- Freely available (with source code)



Why would I use DTSA-II?

My EDS vendor already provides software

- Power tools for microanalysis
 - Access to lower level tools for spectrum analysis and manipulation
- Simulation
 - Few vendors provide simulation tools. None can handle the range of samples
- Standards-based quantification
 - Vendors tools are increasingly focused on standard-less analysis. Make standards-based analysis more difficult or impossible
- Quantify spectra from multiple vendors tools using only one tool
- Algorithms are documented.
- Works nicely on Macs!! and Linux!!!



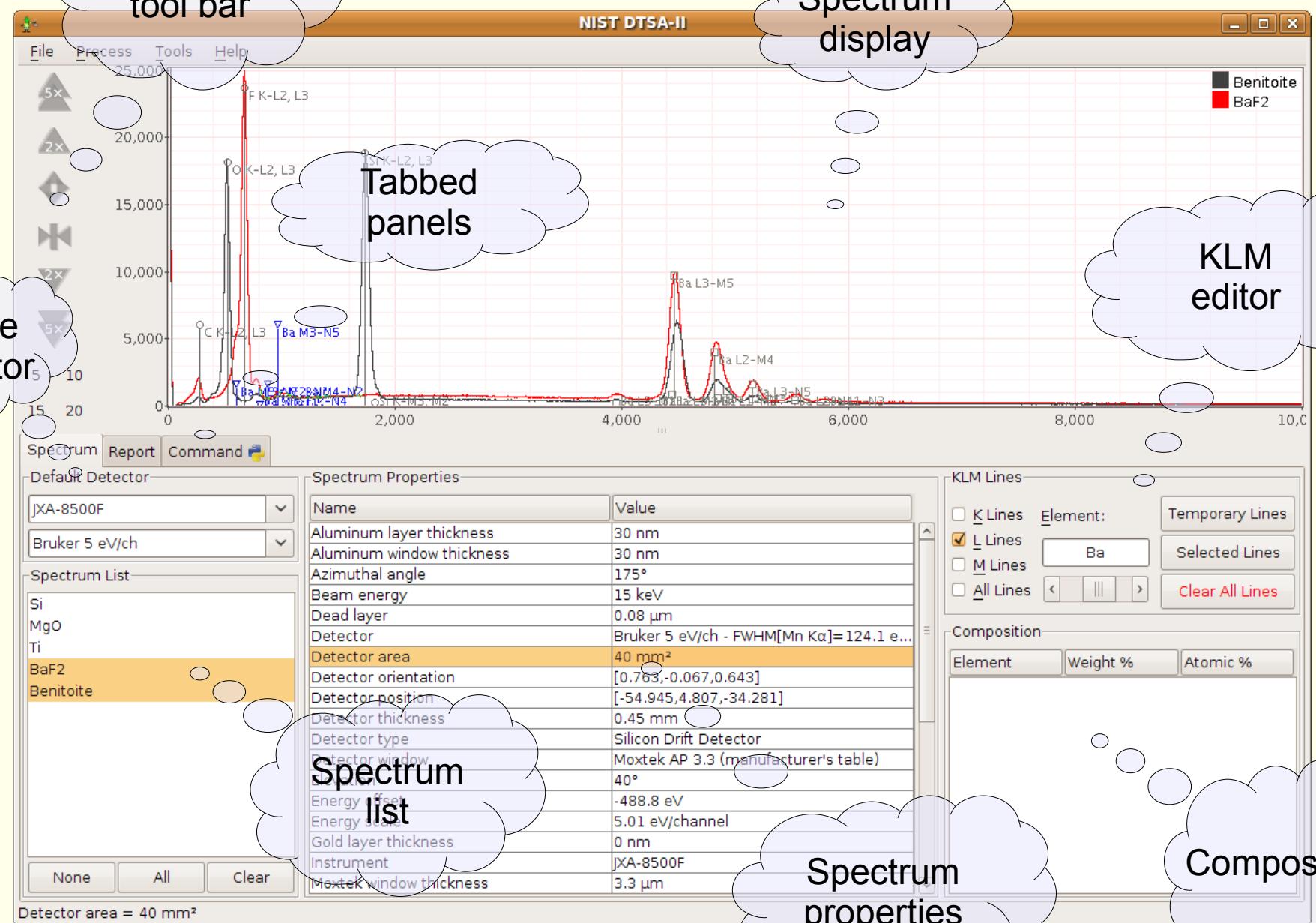
Getting started with NIST DTSA-II

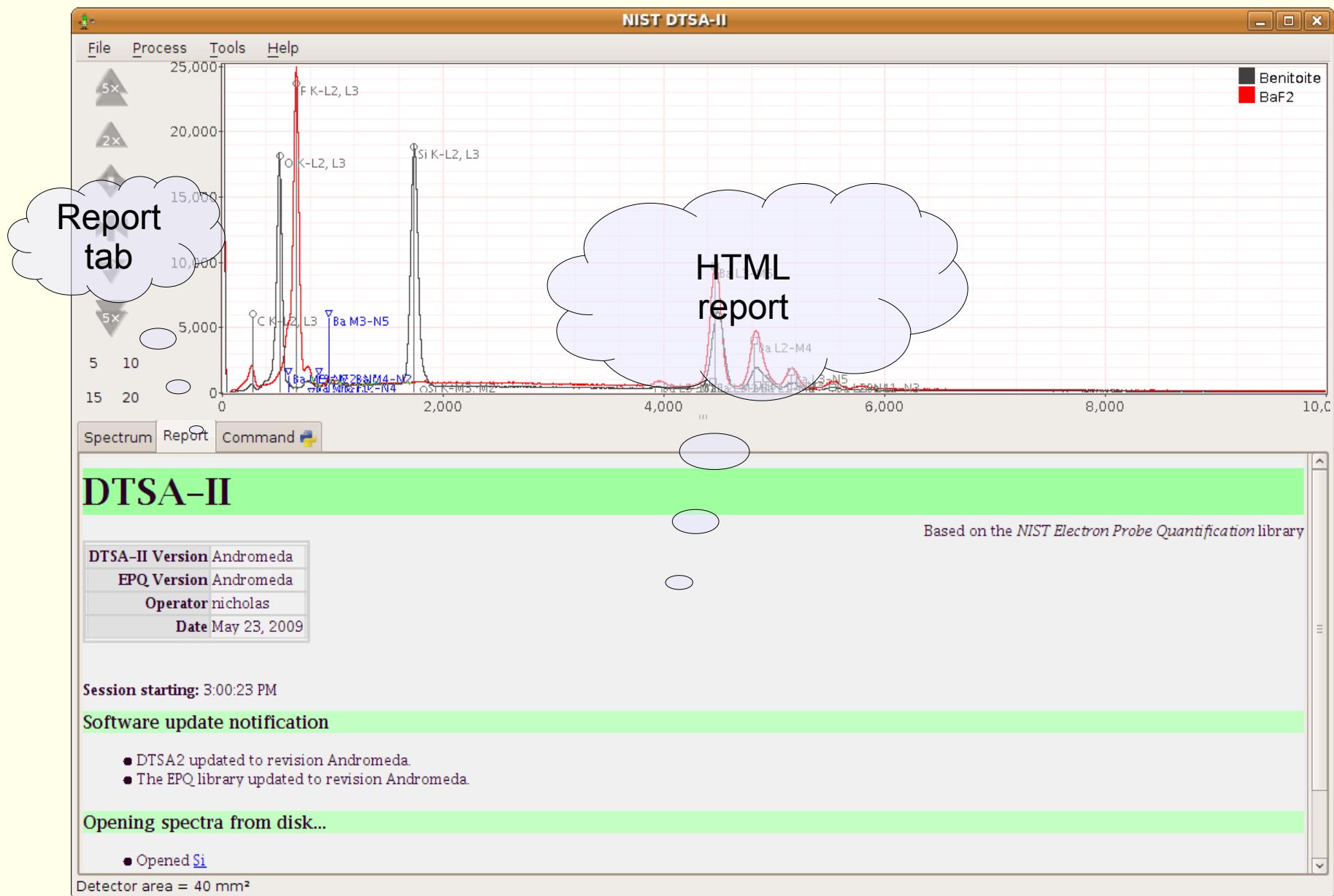
- Installing Java & DTSA-II
- Viewing and manipulating spectra
 - Viewing reports
- Performing standards-based quantification
- Performing spectrum simulation
- Creating and calibrating a detector

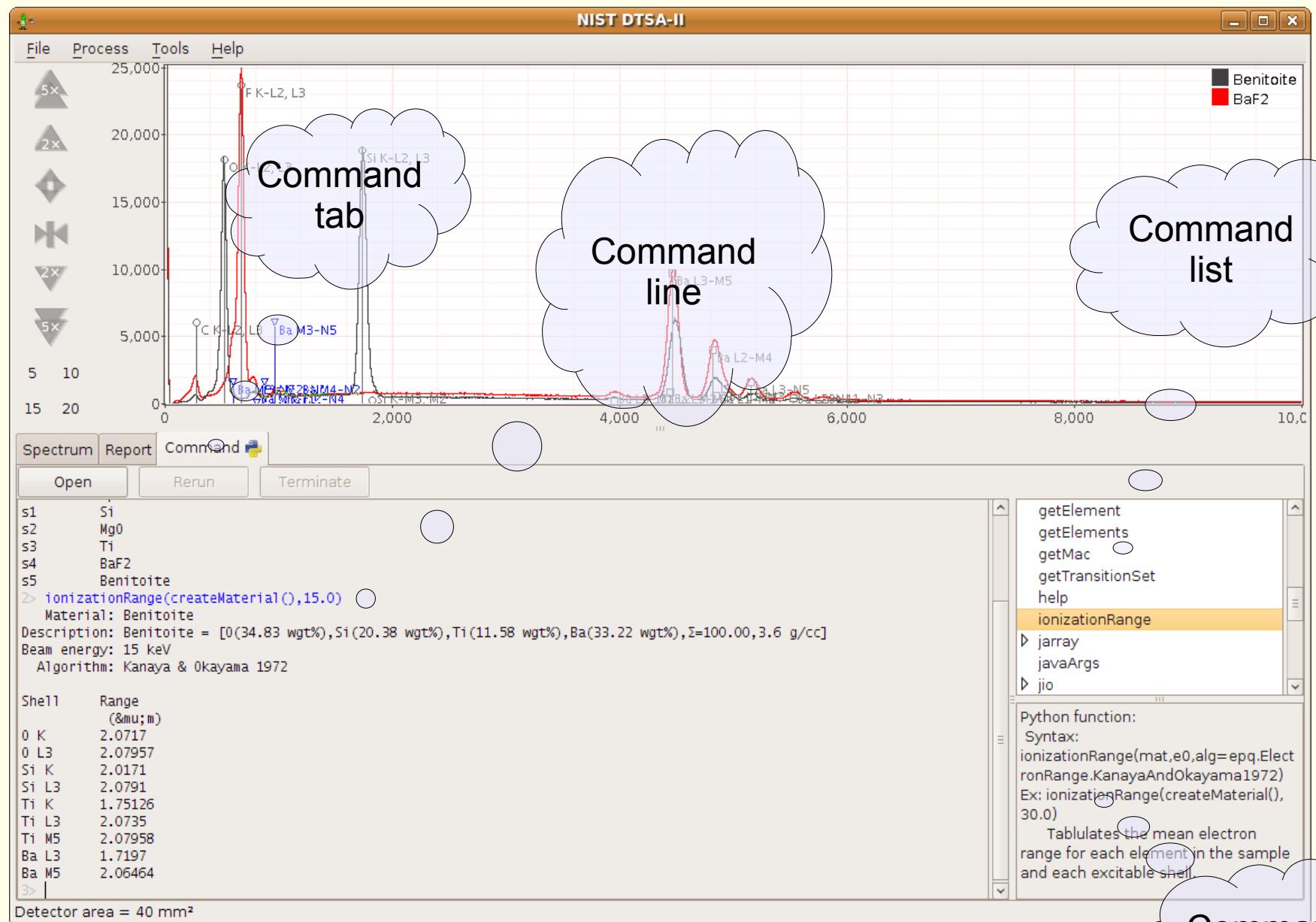


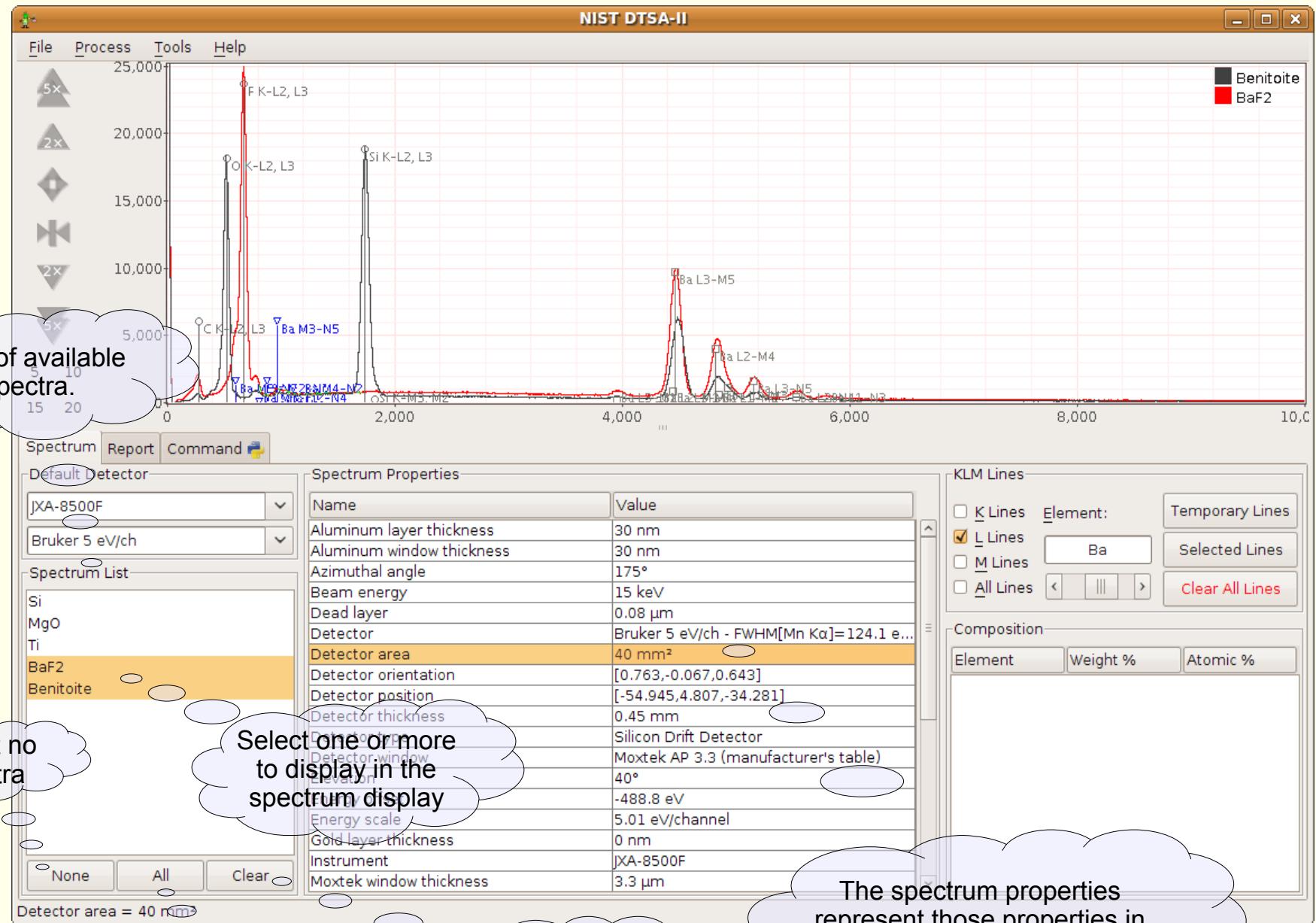
Installing DTSA-II

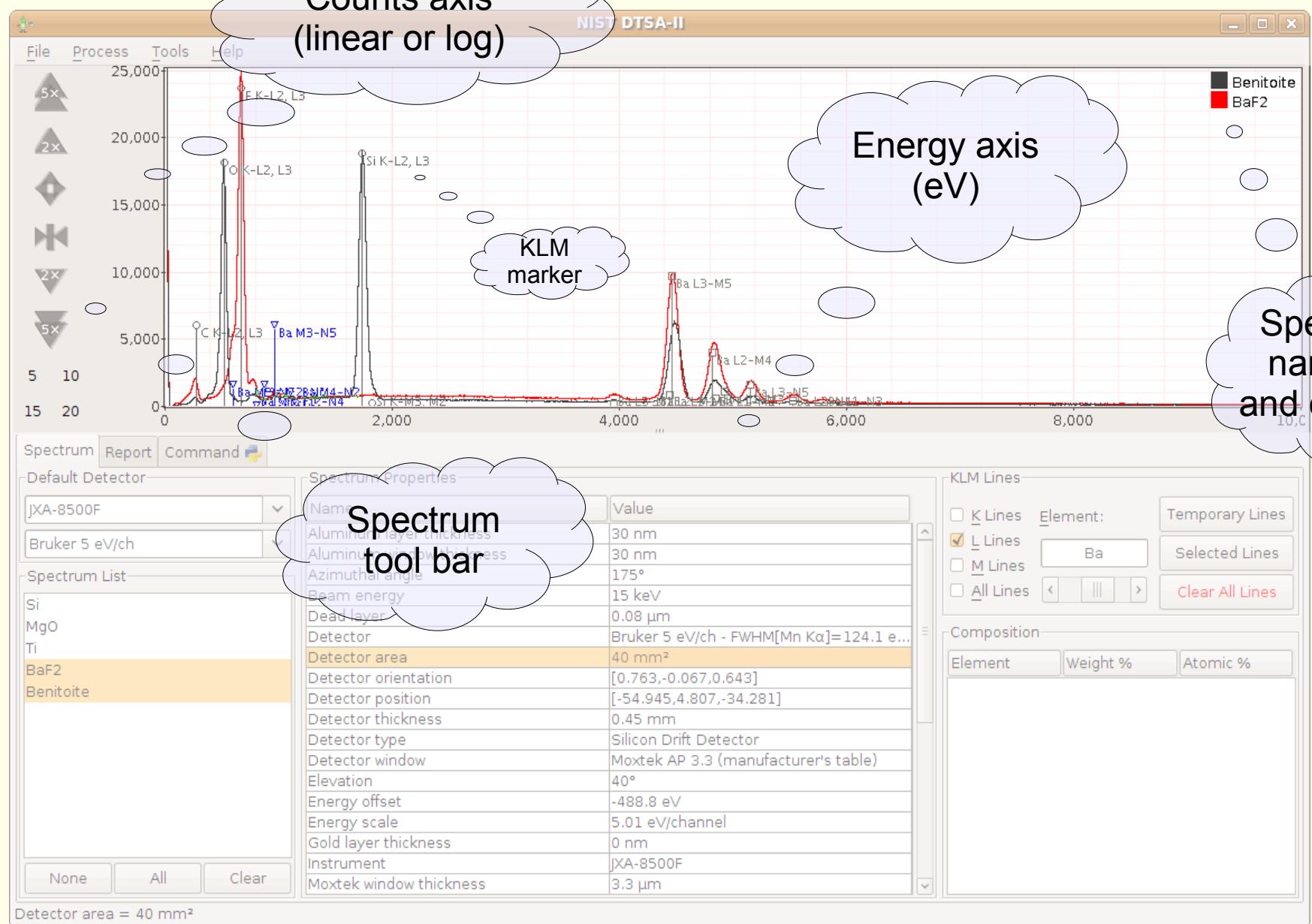
- Install Java 1.6 or later (sorry, 1.5 doesn't work!)
 - Download from <http://java.com>
- Download DTSA-II from <http://nist.gov/dtsa>
 - DTSA-II works on Windows XP and later, Apple OS-X 10.5, 10.6+, most Linux and Unix distributions
 - Installers are available for Windows & OS X



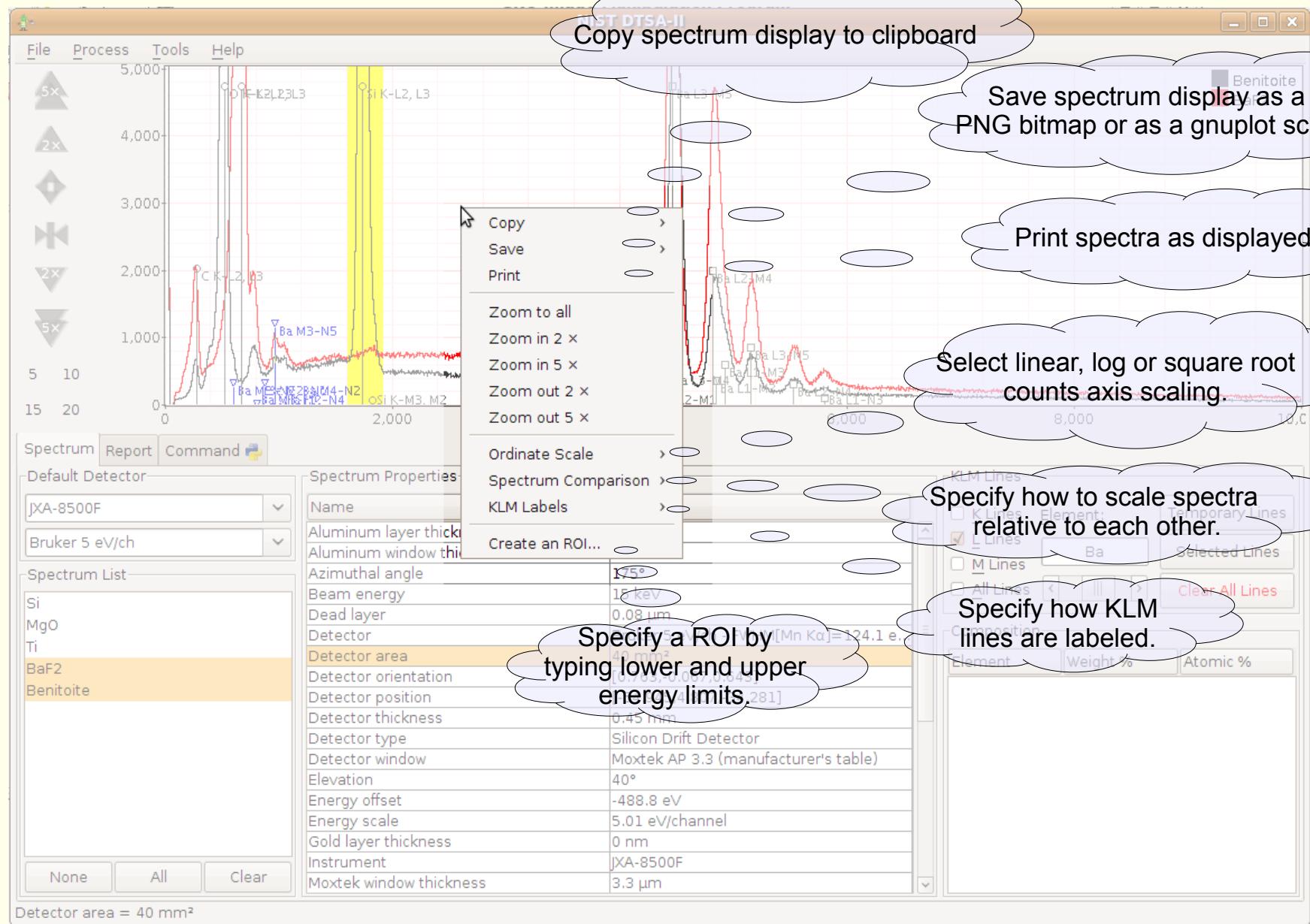








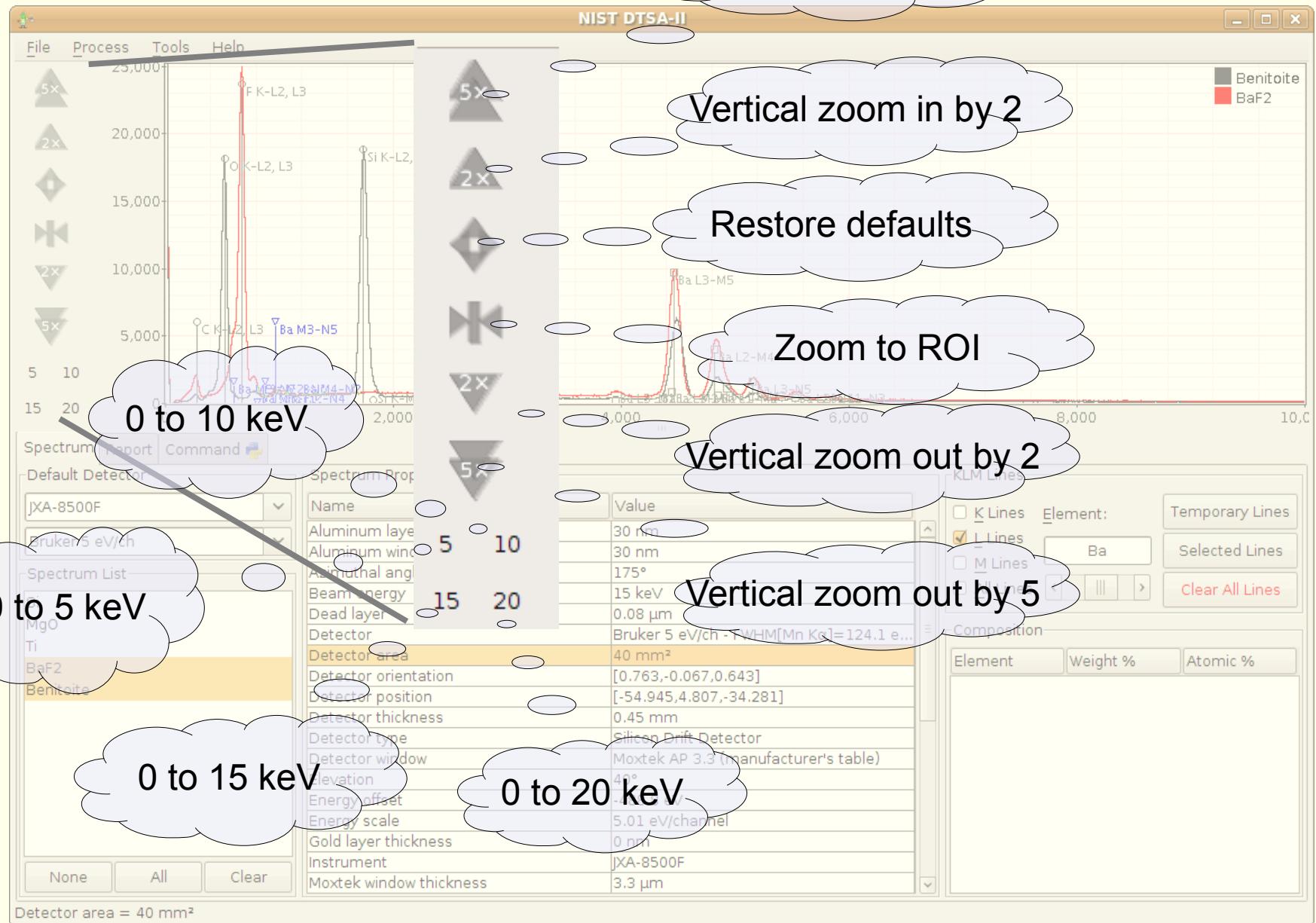
Right click in the spectrum window to display a popup menu.



Right click over the spectrum to display a popup menu.



Vertical zoom in by 5



Vertical zoom in by 2

Restore defaults

Zoom to ROI

Vertical zoom out by 2

Vertical zoom out by 5

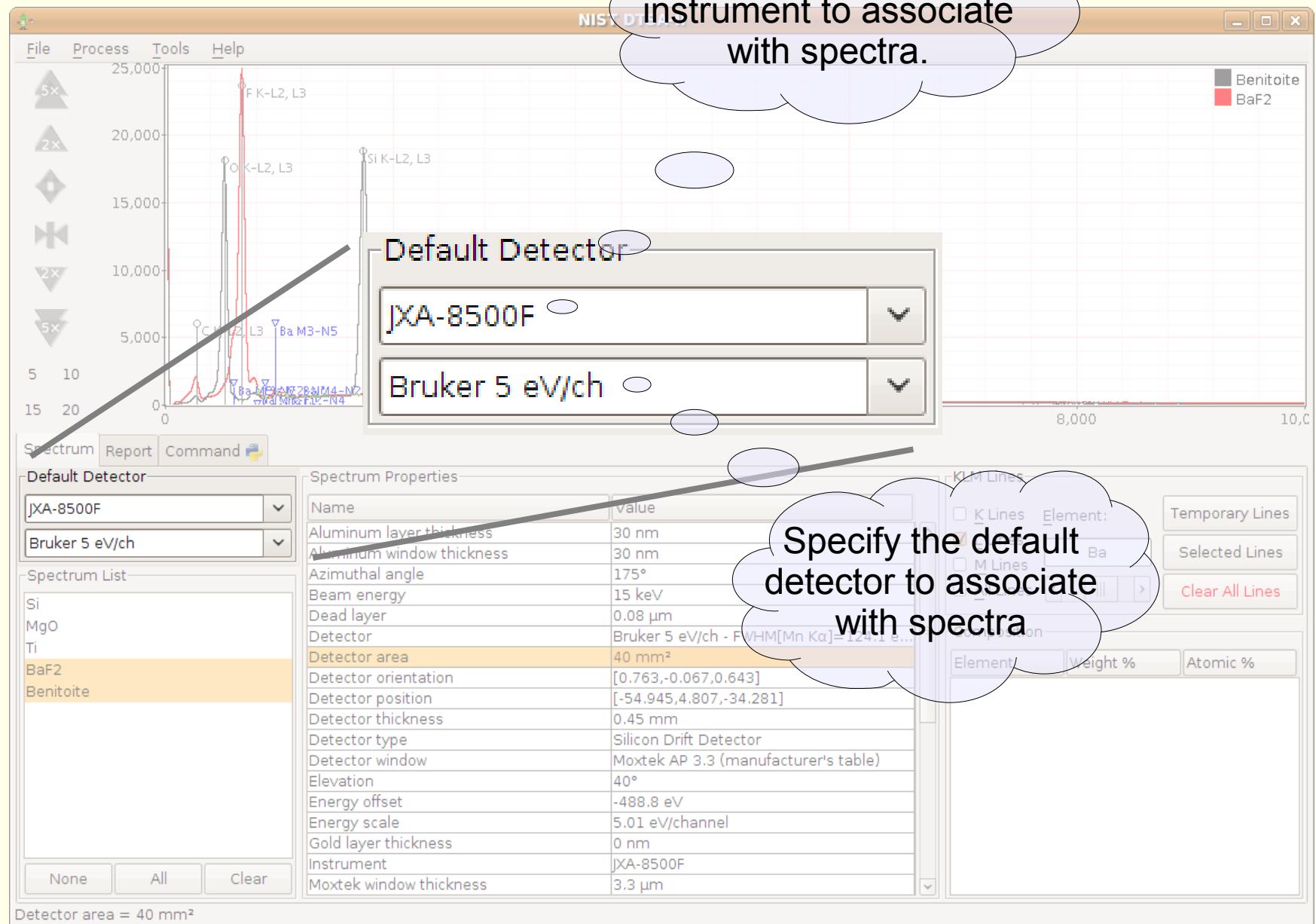
0 to 5 keV

0 to 15 keV

0 to 20 keV



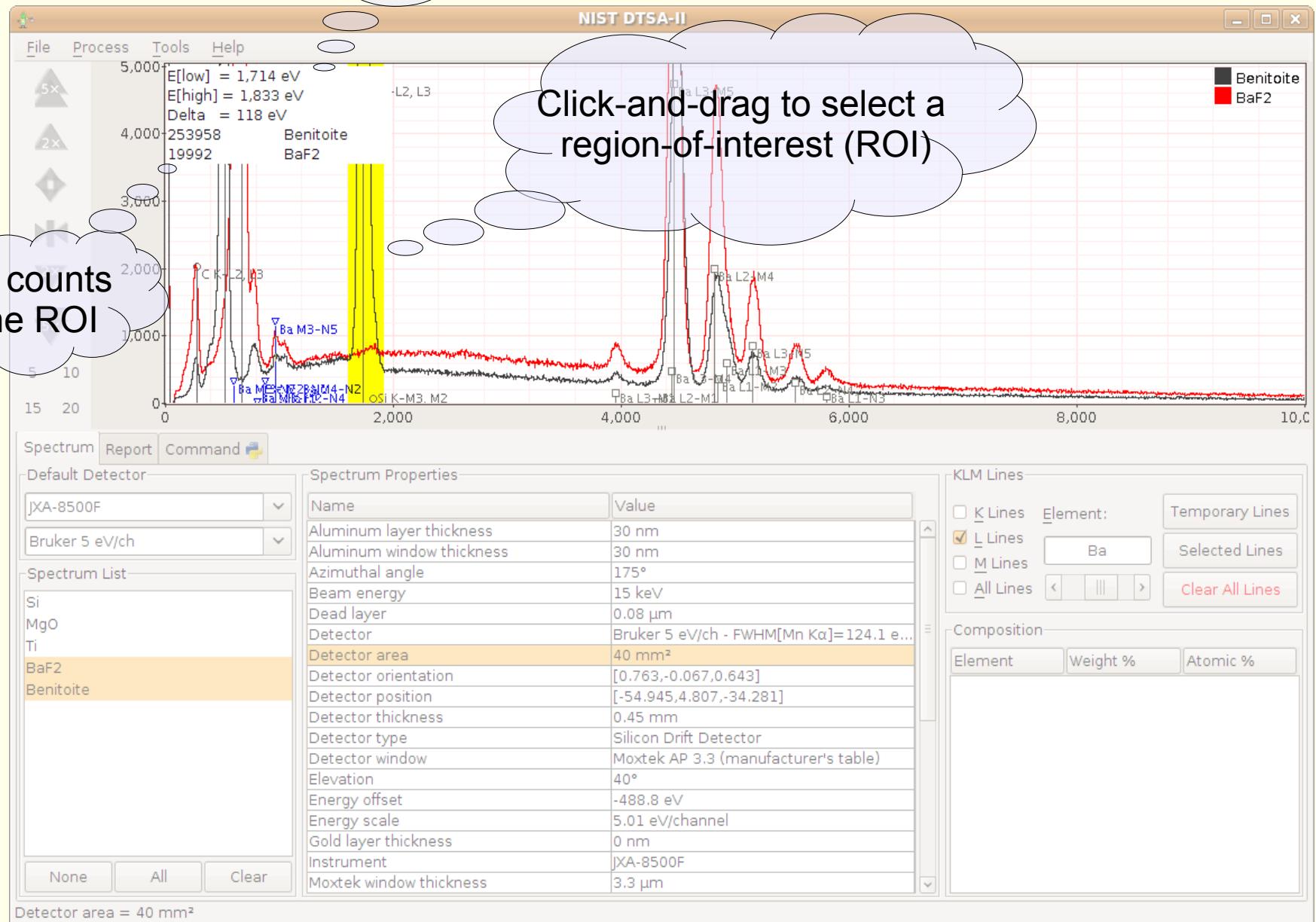
Specify the default instrument to associate with spectra.



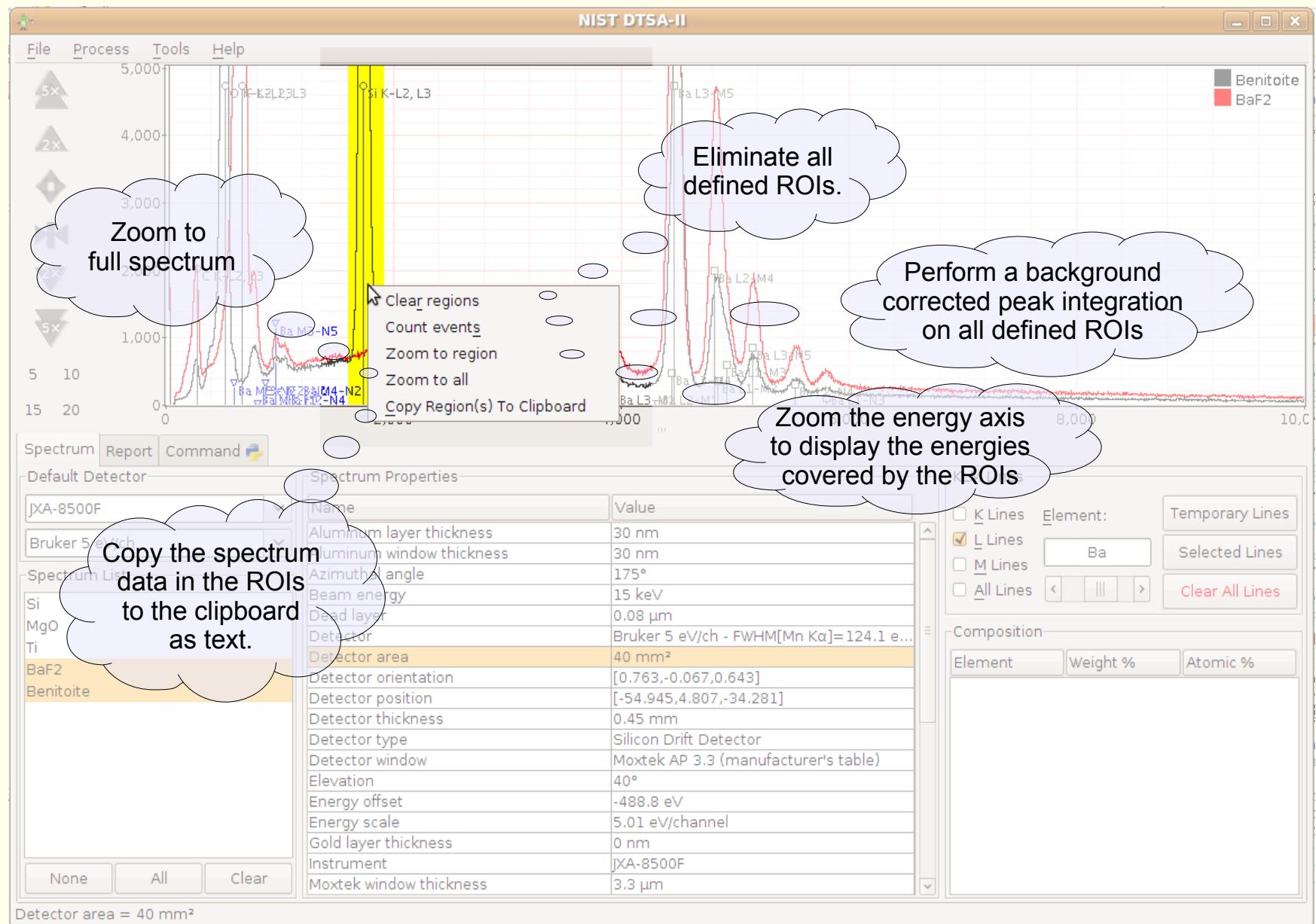
When spectra are read from disk, they are assumed to have been measured on the default detector.

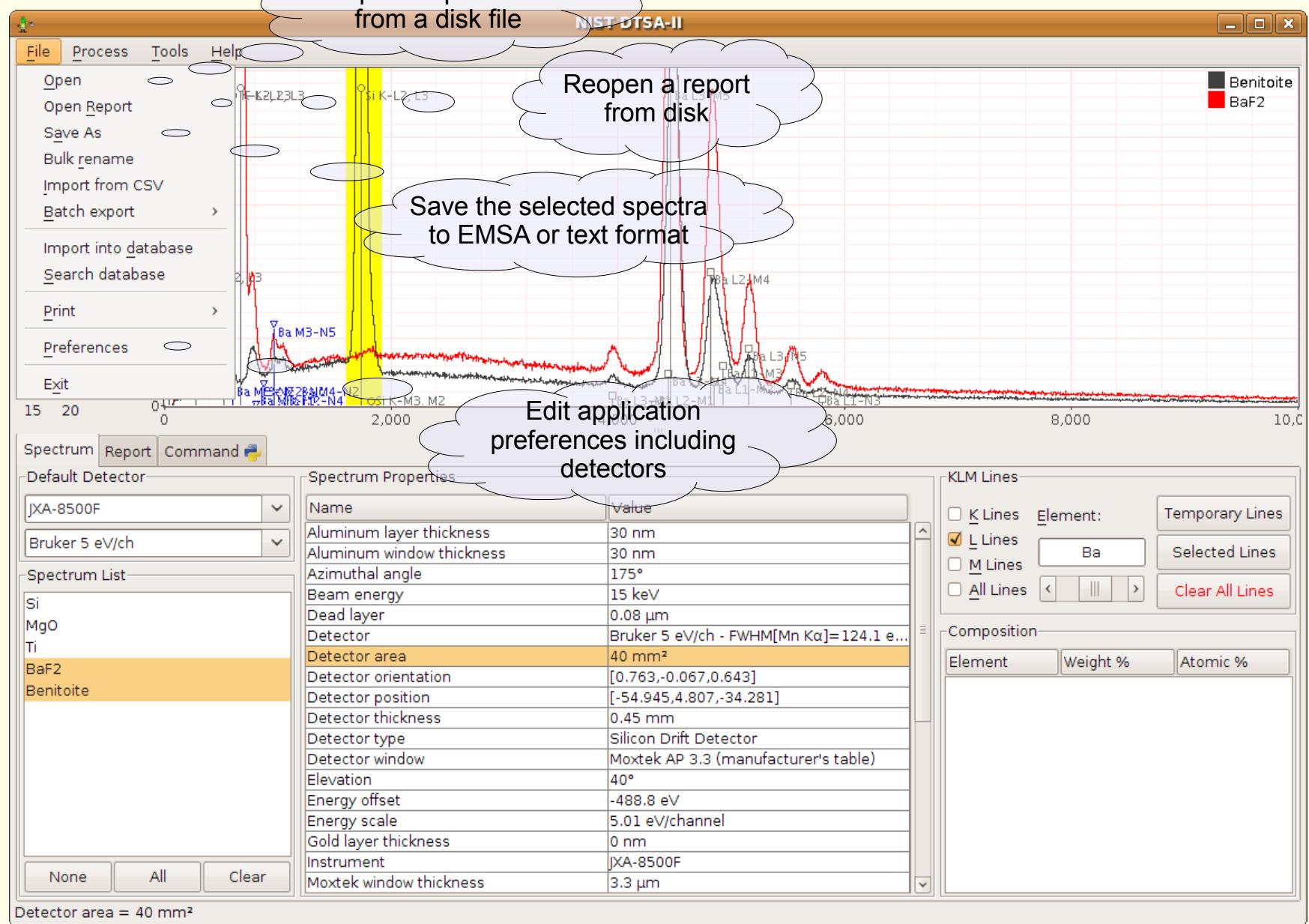


ROI properties



Right click in the ROI properties window to copy the text to the clipboard.

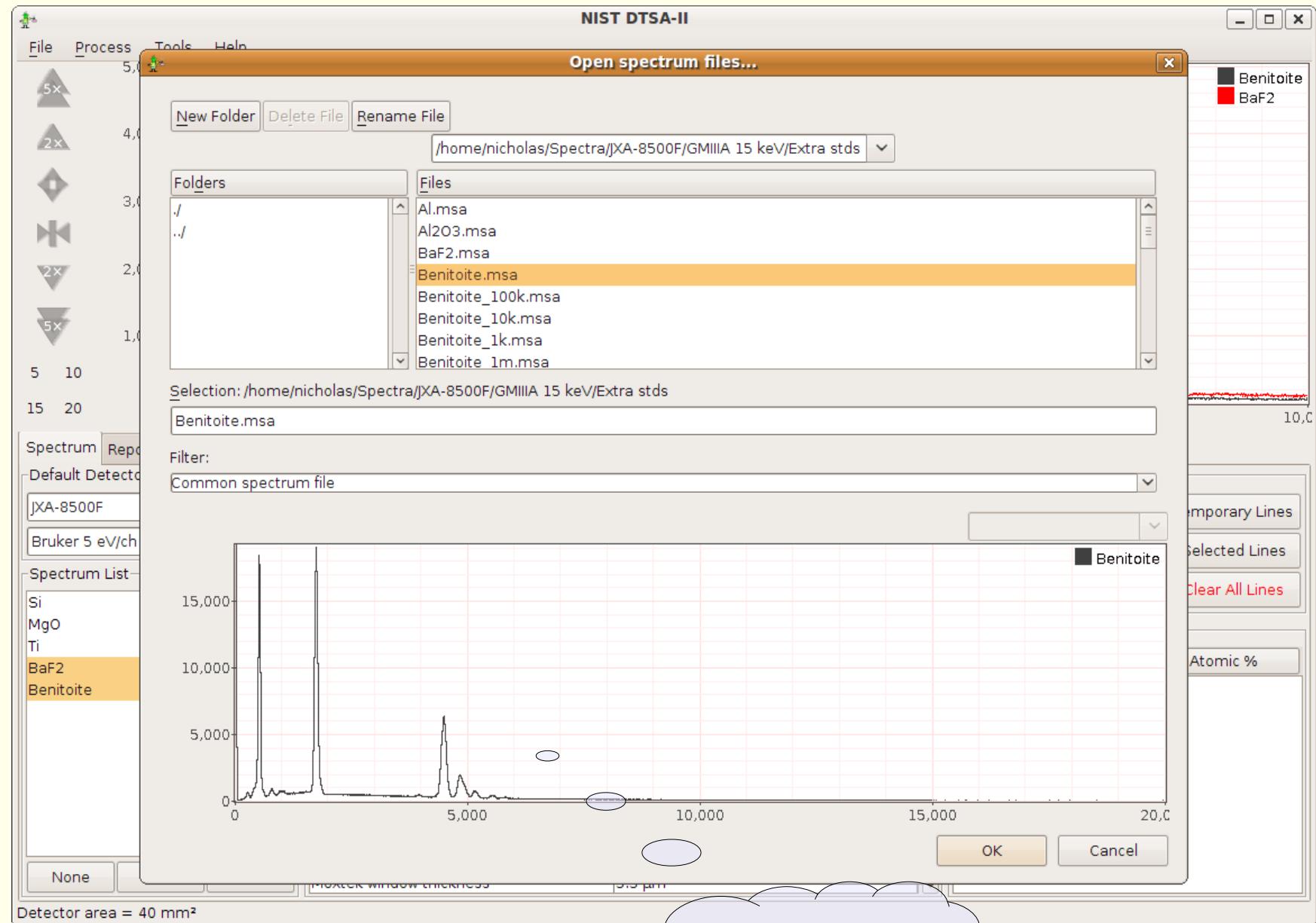






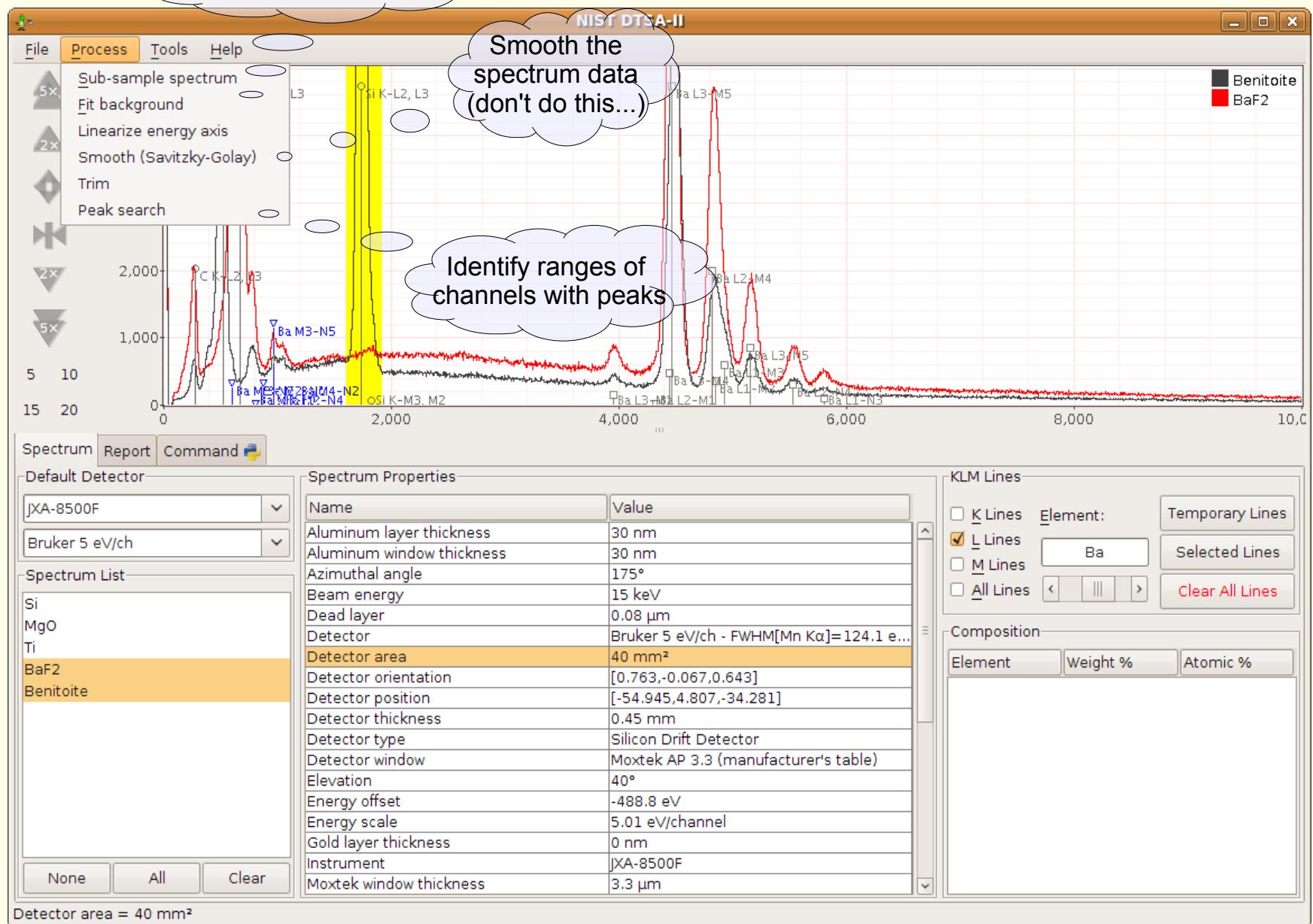
Supports EMSA 1.0, old DTSA, IXRF, Emispec, ASPEX TIFF, Radiant and EDAX spectra files*

* Not all files of all types are supported



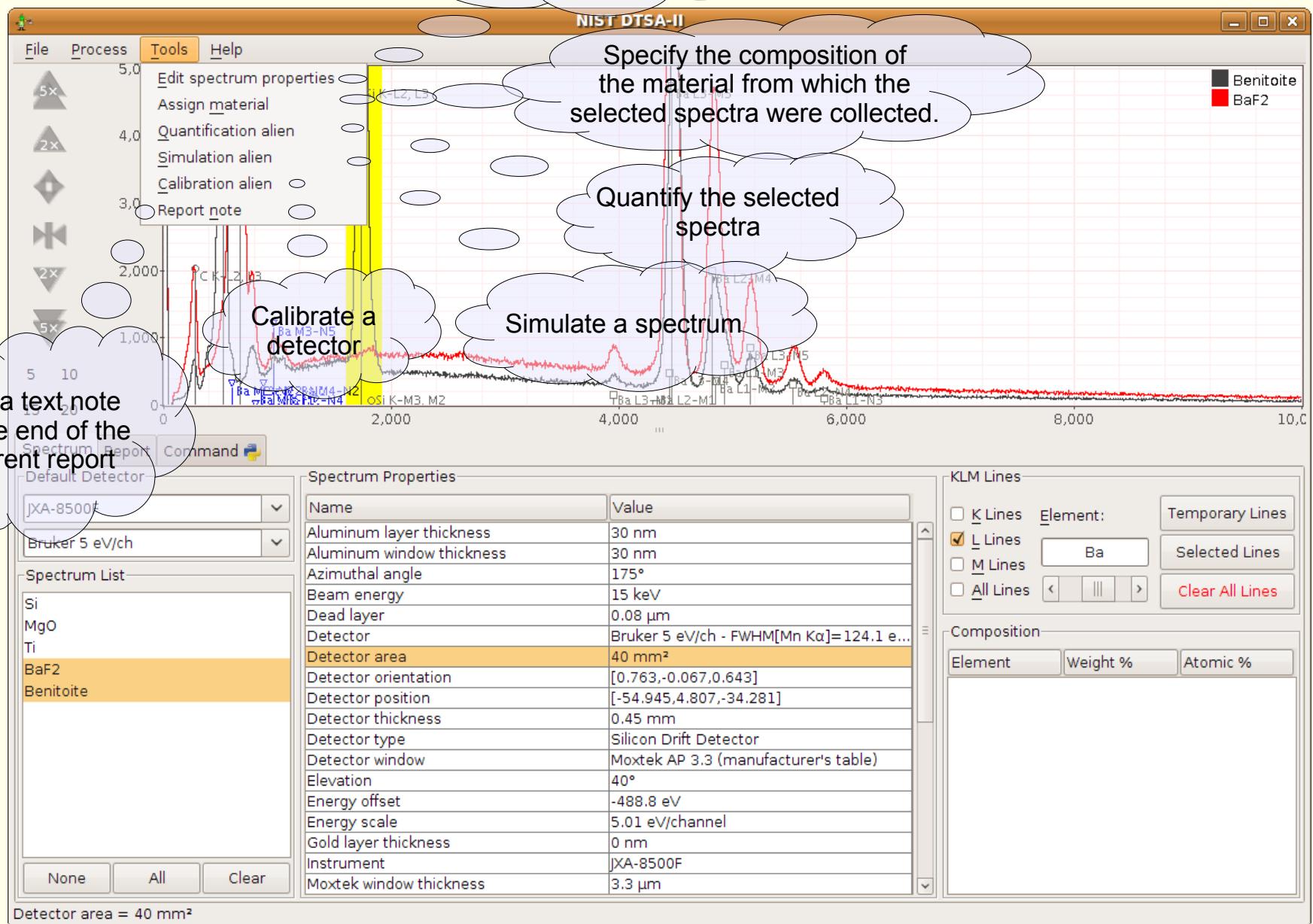


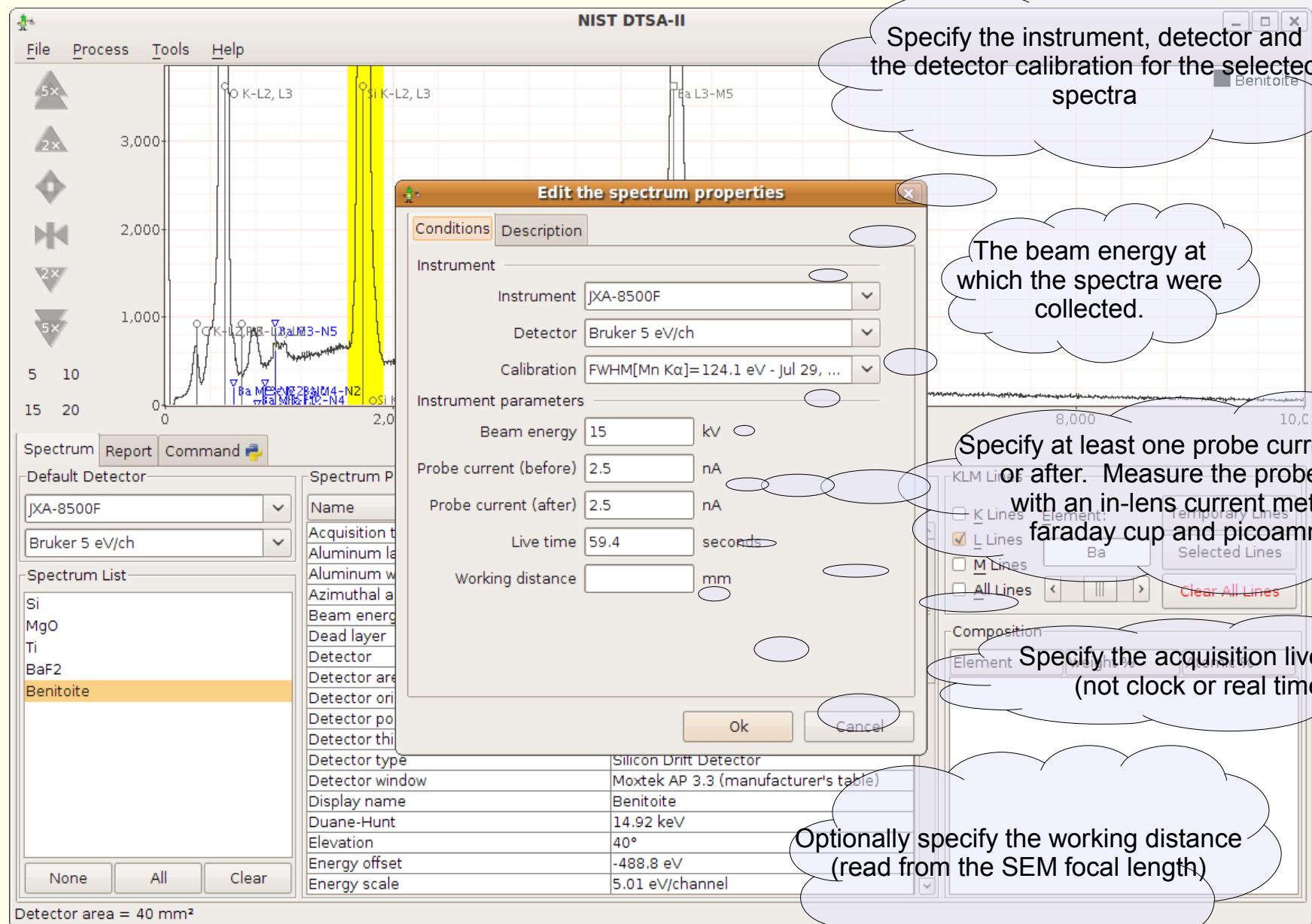
Fit the Bremsstrahlung background

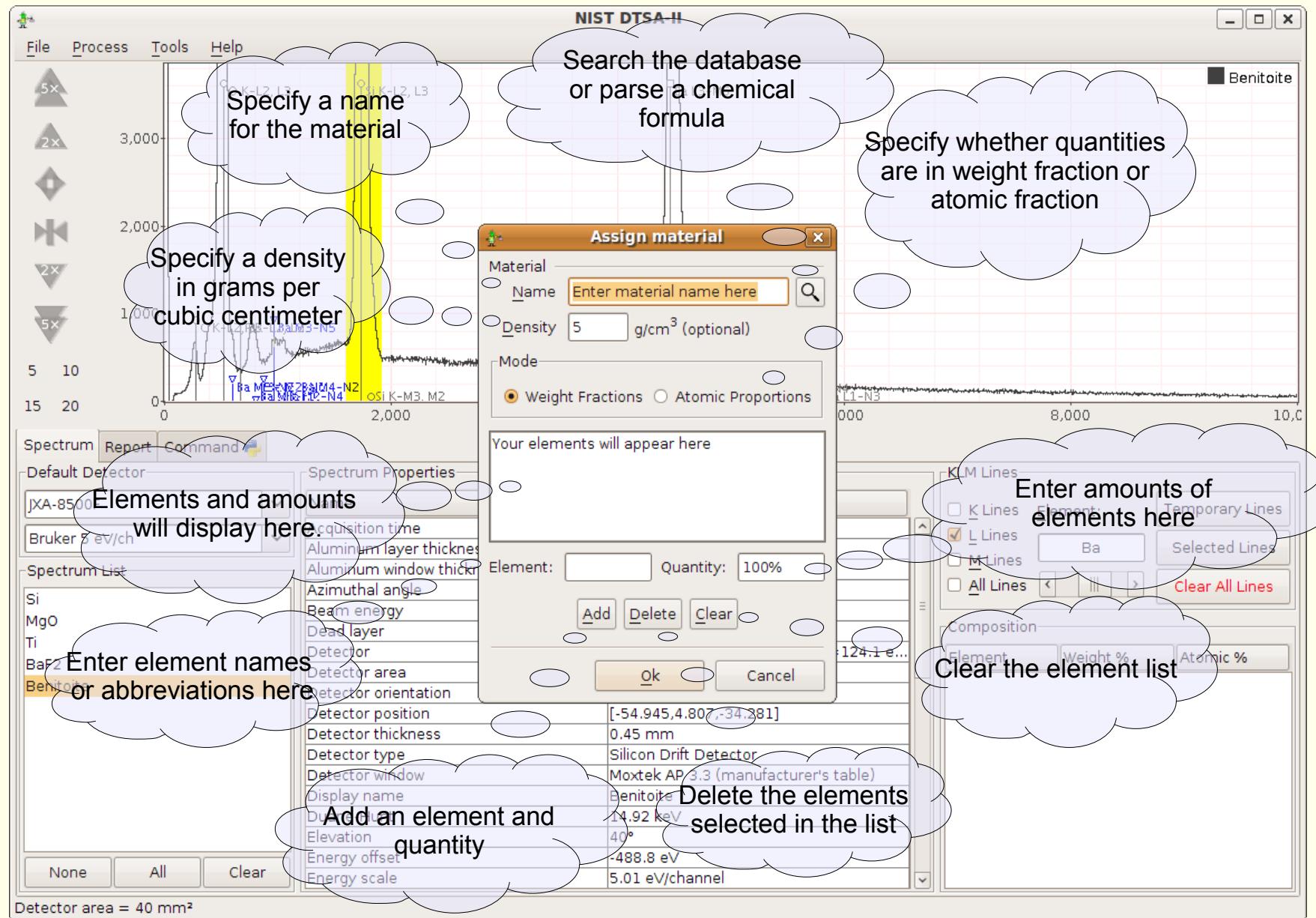




Edit the properties of the selected spectra









Mode 1: Parse a chemical formula

Assign material

Material

Name

Density g/cm³ (optional)

Mode

Weight Fractions Atomic Proportions

O 34.828% by weight
Si 20.3791% by weight
Ti 11.5776% by weight
Ba 33.2153% by weight

Element: Quantity: -0%

Enter a simple or complex chemical formula and press the search button.

The search button.

Mode 2: Search the material database

Assign material

Material

Name

Density g/cm³ (optional)

Mode

Weight Fractions Atomic Proportions

O 34.828% by weight
Si 20.3791% by weight
Ti 11.5776% by weight
Ba 33.2153% by weight

Element: Quantity: -0%

- Enter a name and press the search button
- User entered compositions are automatically added to the material database.

Mode 3: Manual entry

Assign material

Material

Name

Density g/cm³ (optional)

Mode

Weight Fractions Atomic Proportions

Ba 33.21% by weight

Element: Quantity: 11.58

Add each element as weight fractions or atomic proportions using the Element and Quantity edit boxes and the Add button.



Measuring Composition

- What you need...
 - ✓ A spectrum collected from the unknown material
 - ✓ A list of elements in the unknown
 - ✓ A standard spectrum for each element in the unknown (one standard can represent multiple elements)
- What you might need...
 - ✓ A reference spectrum for certain elements in the unknown



Measuring Composition

- What you need to know...
 - For the unknown spectrum and each standard
 - ✓ The probe current (in pA or nA)
 - ✓ The acquisition live time (in seconds)
 - ✓ The beam energy (in eV or keV)
 - For the standards
 - ✓ The composition of the standard material



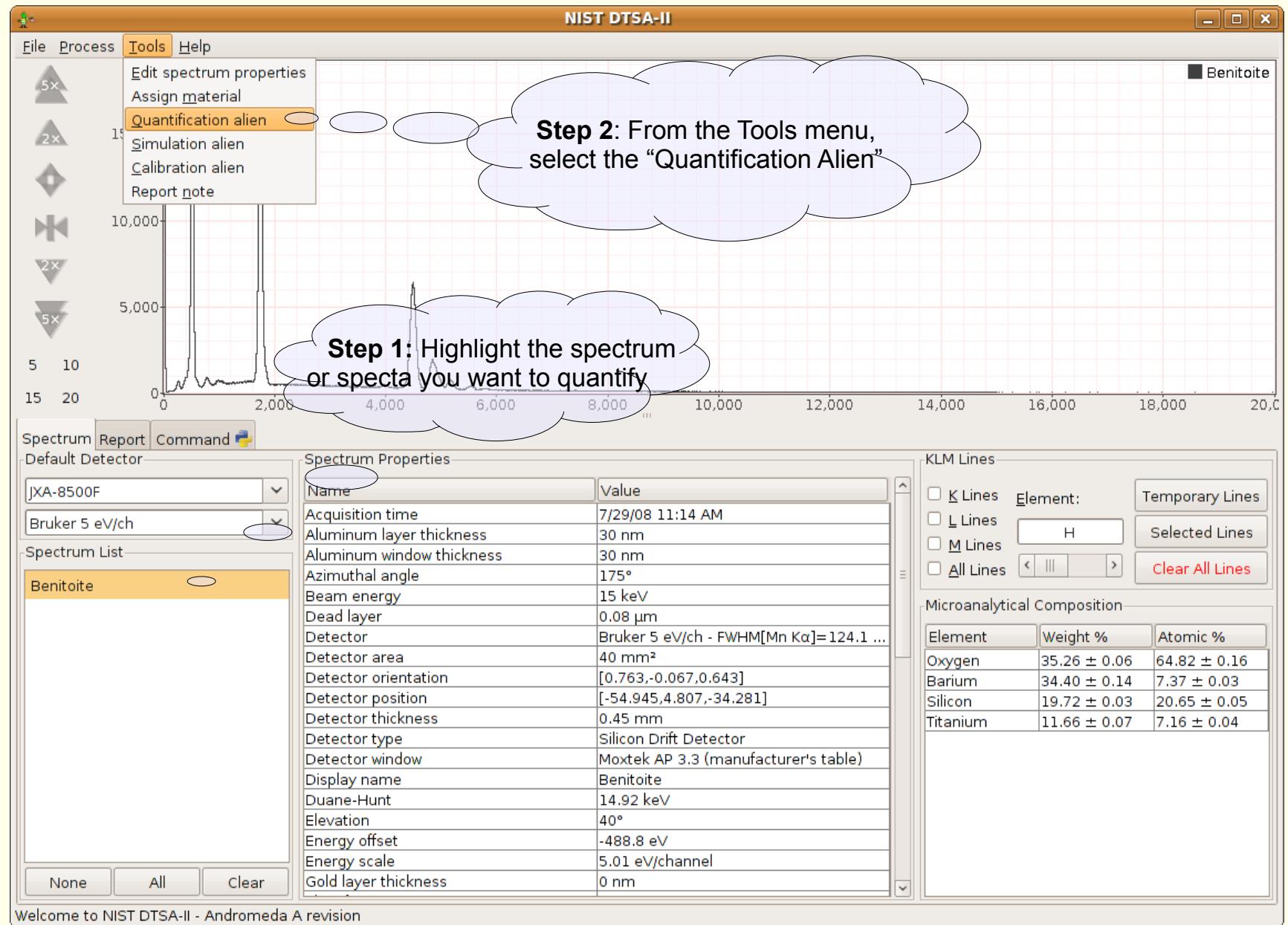
What is a standard?

- A standard spectrum is a spectrum collected from a material of known composition
 - Must be collected under the same measurement conditions as the unknown
 - ✓ Beam energy
 - ✓ Working distance
 - ✓ Detector parameters
 - Should be a high quality spectrum (many counts)
- The ideal standard is of similar composition to the unknown
 - ✓ Likely to provide the most accurate results



What is a reference?

- A reference spectrum is a spectrum that provides the unobstructed shape information about a set of characteristic x-ray lines for an element
 - ✓ A reference should be collected under similar conditions as the unknown but references are less susceptible to moderate changes in conditions
 - ✓ Should be a high quality spectrum (many counts)
- References are required when the characteristic lines from one element represented in a standard spectrum are obstructed by lines from an other element.





NIST DTSA-II

File Process Tools Help

5x
2x
Diamond
2x
5x
5 10
15 20

15,000
10,000
5,000
0

Spectrum Report Command

Default Detector

JXA-8500F
Bruker 5 eV/ch

Spectrum List

Benitoite

Quantification Alien

Select a quantification mode

First page

Next: Specify the instrument

Select the mode which best describes the operation you wish to perform. The mode you select will determine what information you will be asked to provide and what information will be computed.

Determine the composition of an 'unknown' spectrum by MLLSQ fitting to standards
 Determine the composition from k-ratios
 Determine the composition of an 'unknown' spectrum by fitting using a simplex method
 Estimate measured k-ratios from composition

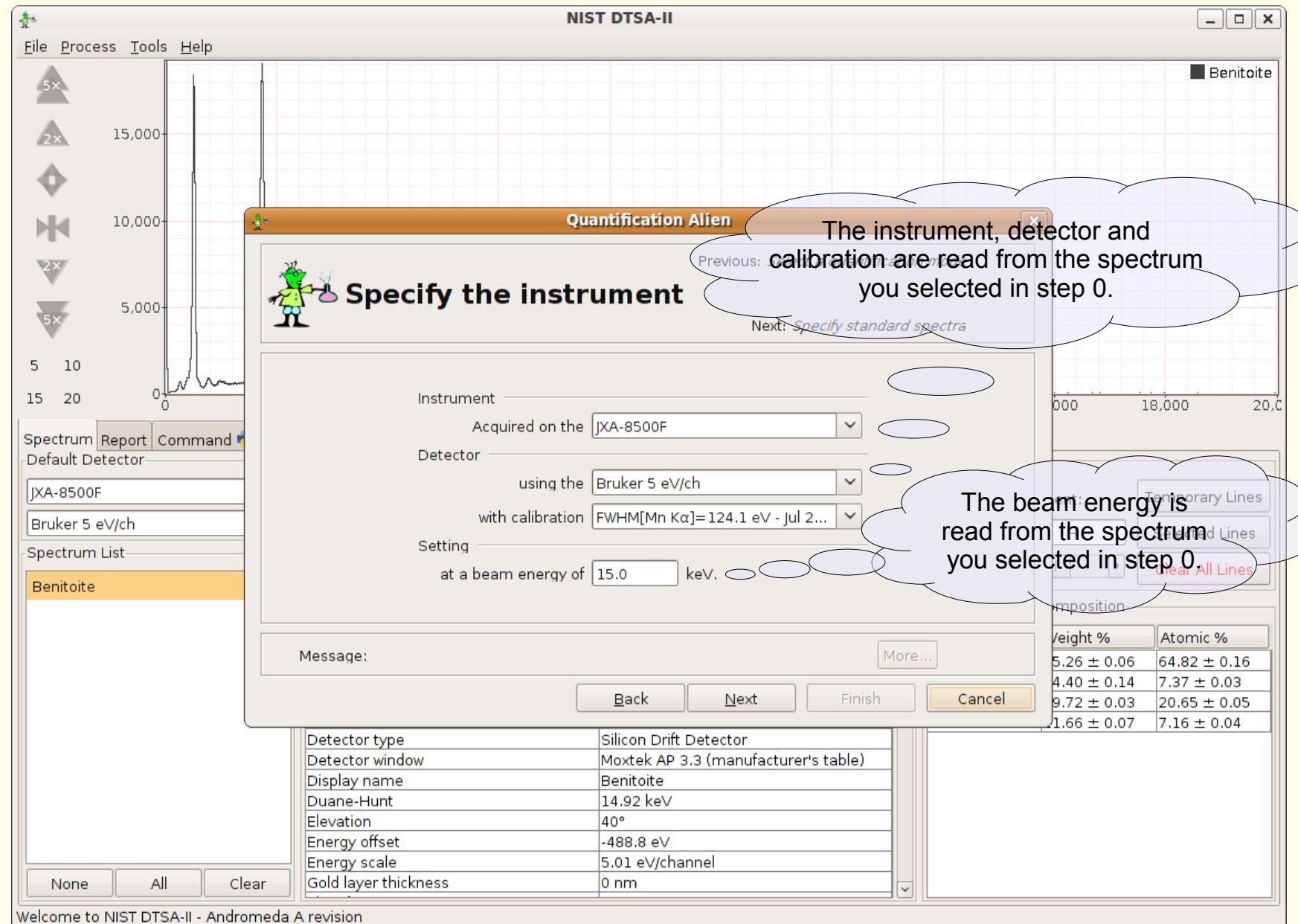
Message: Select an analysis mode.

Back Next Finish Cancel

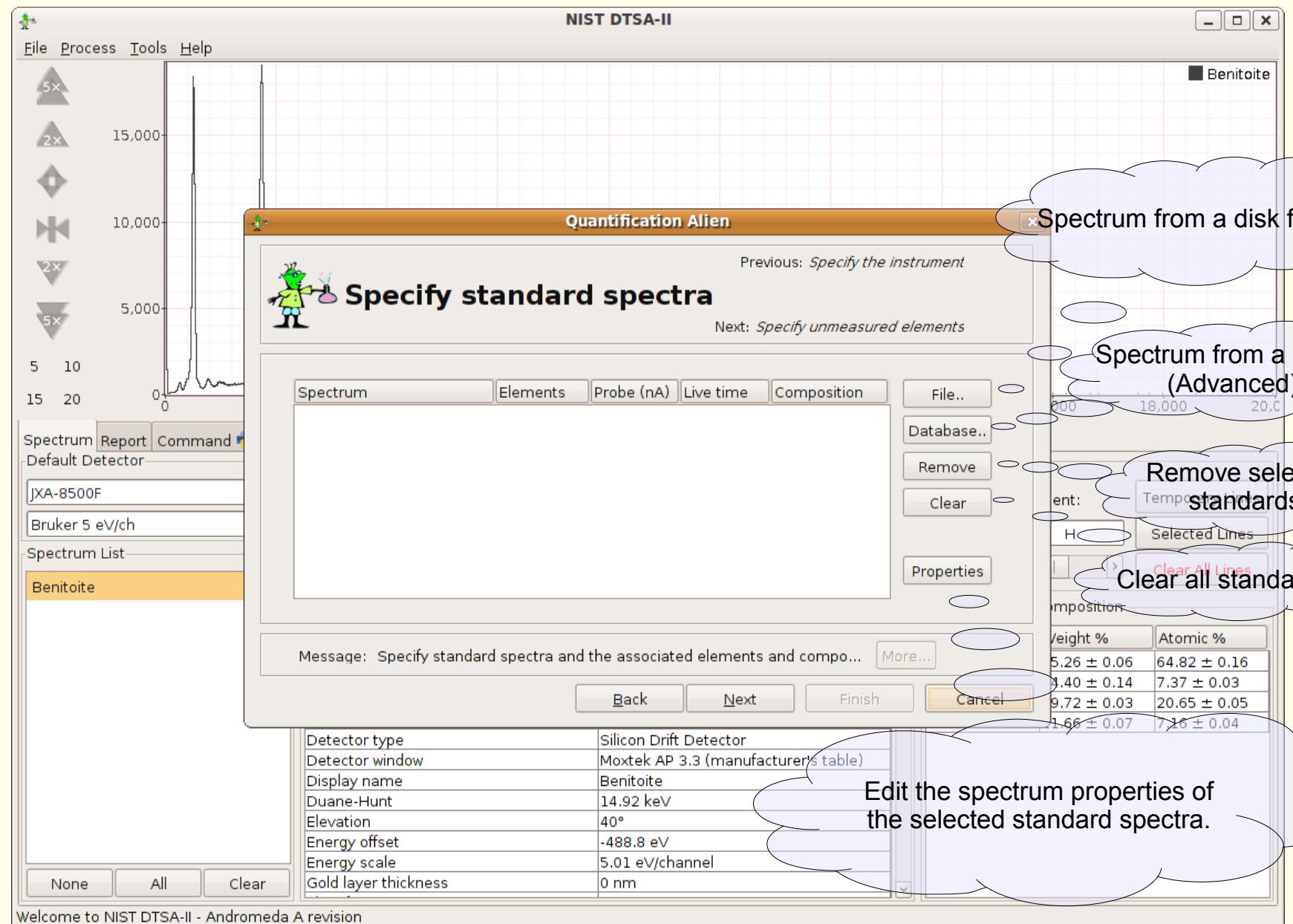
Detector type: Silicon Drift Detector
Detector window: Moxtek AP 3.3 (manufacturer's table)
Display name: Benitoite
Duane-Hunt: 14.92 keV
Elevation: 40°
Energy offset: -488.8 eV
Energy scale: 5.01 eV/channel
Gold layer thickness: 0 nm

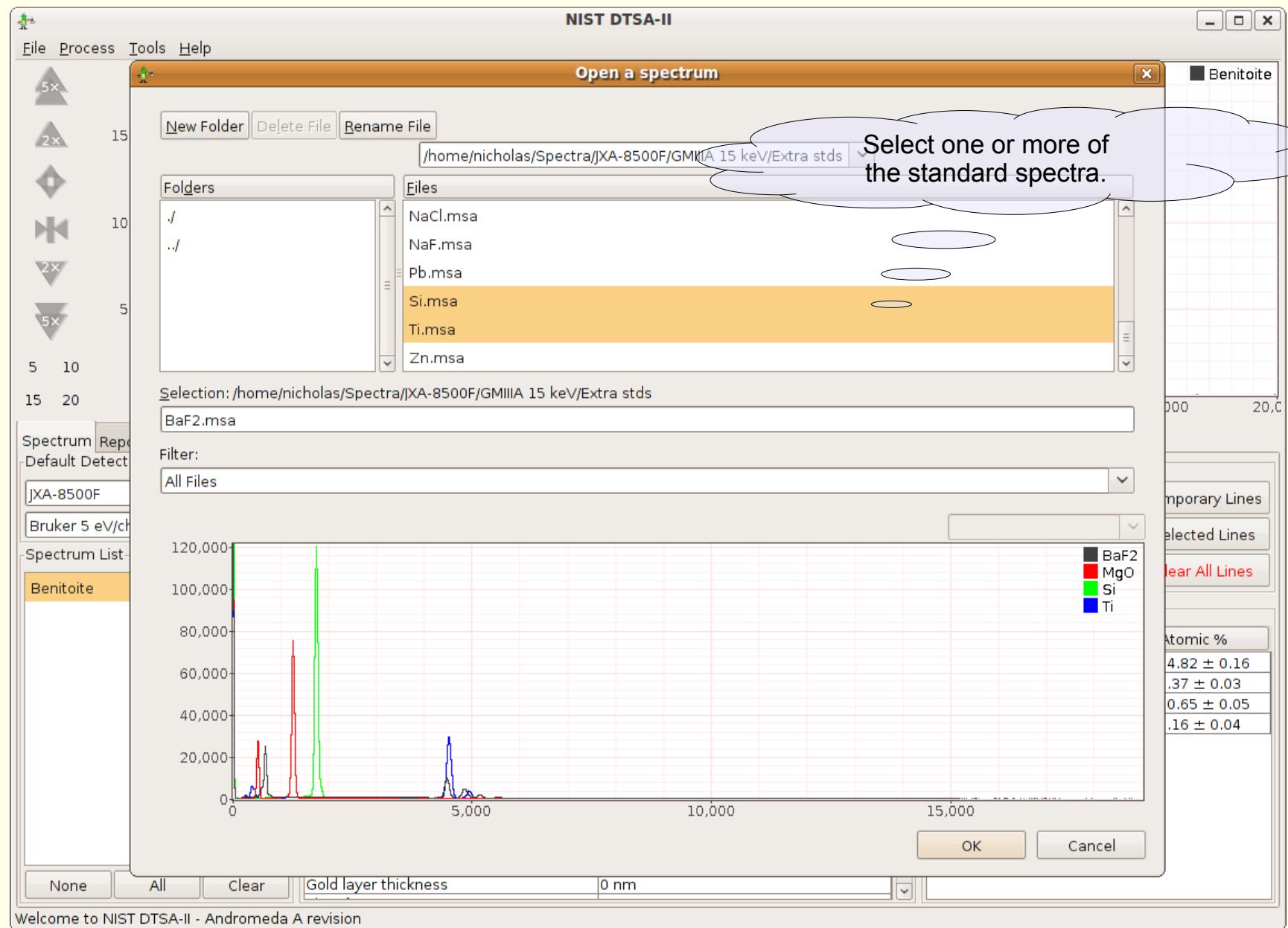
Click 'Next' to proceed to the next step.

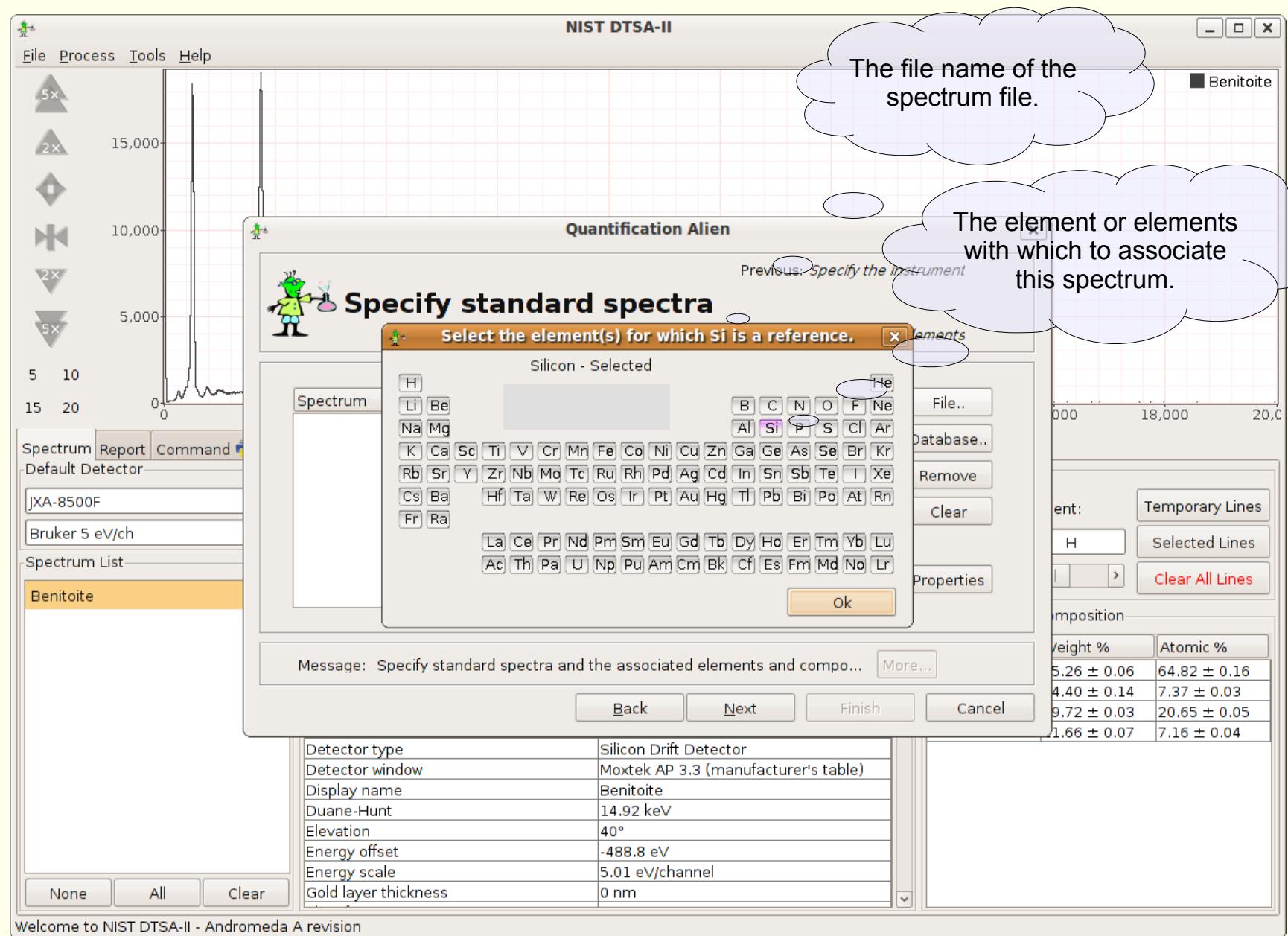
Welcome to NIST DTSA-II - Andromeda A revision



You may change these values but it is unlikely that it is a good idea.







For each spectrum, you will be asked to specify which elements are associated with which spectrum. A spectrum may act as a standard for multiple elements.



The name of the spectrum

The element or elements with which this spectrum is associated.

The composition of the material from which this standard spectrum was collected. This is guessed from the contents of the "Elements" column.

The composition of "BaF2" was guessed incorrectly. We'll need to fix this!

The probe current and live time for this standard spectrum.

NIST DTSA-II

Quantification Alien

Specify standard spectra

Spectrum Report Command

Default Detector

JXA-8500F

Bruker 5 eV/ch

Spectrum List

Benitoite

File Process Tools Help

5x
2x
15.000
10,000
5,000
0
5 10
15 20

Si Si 2.500 59.4 Pure silicon

MgO O 2.500 59.3 MgO

Ti Ti 2.500 59.4 Pure titanium

BaF2 Ba 2.500 59.4 BaSO₄

File.. Database.. Remove Clear Properties

Weight % Atomic %

5.26 ± 0.06 64.82 ± 0.16

4.40 ± 0.14 7.37 ± 0.03

9.72 ± 0.03 20.65 ± 0.05

1.66 ± 0.07 7.16 ± 0.04

Detector type Silicon Drift Detector

Detector window Motek AP 3.3 (manufacturer's table)

Display name Benitoite

Duane-Hunt 14.92 keV

Elevation 40°

Energy offset -488.8 eV

Energy scale 5.01 eV/channel

Gold layer thickness 0 nm

Back Next Finish Cancel

None All Clear

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You may also be asked to specify the probe current or the live time if this information is not specified in the spectrum file.



NIST DTSA-II

File Process Tools Help

5x
2x
15,000
10,000
5,000
0

15 20

Spectrum Report Command

Default Detector

JXA-8500F

Bruker 5 eV/ch

Spectrum List

Benitoite

5x
2x
15,000
10,000
5,000
0

15 20

Quantification Alien

Specify standard spectra

Previous: *Specify the instrument*
Next: *Specify unmeasured spectra*

Spectrum	Elements	Probe (nA)	Live time	Composition
Si	Si	2.500	59.4	Pure silicon
MgO	O	2.500	59.3	MgO
Ti	Ti	2.500	59.4	Pure titanium
BaF2	Ba	2.500	59.4	BaSO ₄

File.. Database... Remove Clear Properties

Message: More... Back Next Finish Cancel

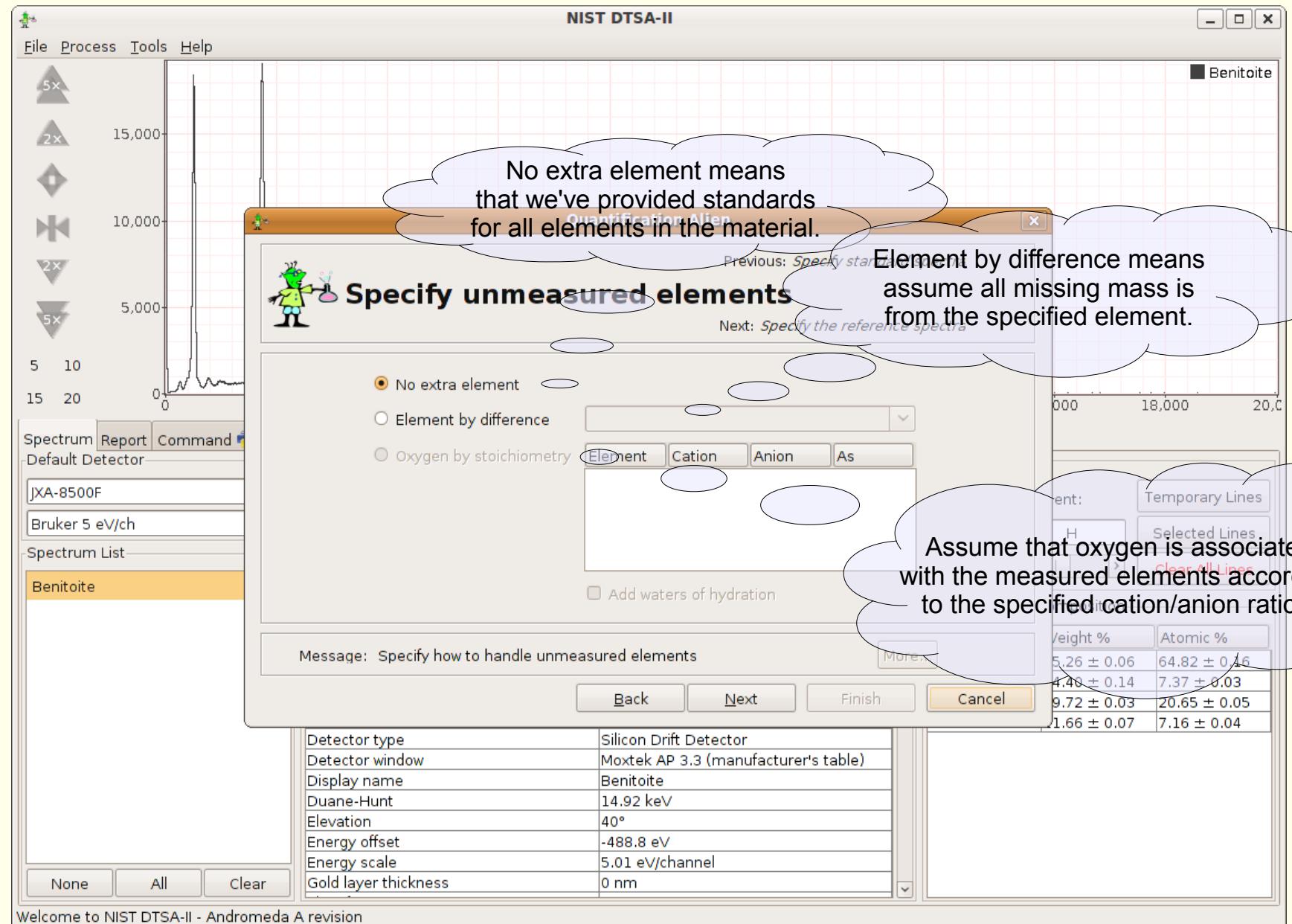
Detector type: Silicon Drift Detector
Detector window: Moxtek AP 3.3 (manufacturer's table)
Display name: Benitoite
Duane-Hunt: 14.92 keV
Elevation: 40°
Energy offset: -488.8 eV
Energy scale: 5.01 eV/channel
Gold layer thickness: 0 nm

Weight % Atomic %
5.26 ± 0.06 64.82 ± 0.16
4.40 ± 0.14 7.37 ± 0.03
9.72 ± 0.03 20.65 ± 0.05
1.66 ± 0.07 7.10 ± 0.04

The composition items are drop-down lists. Select the correct composition from the list or select "New material" if the correct composition is not listed.

You can update the probe current or live time using the properties button.

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Extra elements are elements which you don't want to measure directly but rather infer from the other elements we did measure.



NIST DTSA-II

File Process Tools Help

5x
2x
Diamond
2x
5x
5 10
15 20

15,000
10,000
5,000
0

Spectrum Report Command
Default Detector
JXA-8500F
Bruker 5 eV/ch
Spectrum List
Benitoite

Quantification Alien

We don't need to specify references because in this case the standards can act as references.

Specify the reference spectra

Previous: *Specify unmeasured elements*
Next: *Specify unknown spectra*

Region-of-Interest Spectrum S/N

Ti L-family [0.19, 0.67 keV]	Ti	Good 321
O All [0.30, 0.68 keV]	MgO	Good 532
Si All [1.48, 2.01 keV]	Si	Good 1341
Ba L-family [3.69, 6.19 keV]	BaF2	Good 336
Ti K-family [4.19, 5.17 keV]	Ti	Good 805

File... Database... Remove Strip

ent: Temporary Lines
H Selected Lines
I > Clear All Lines

Message: Specify reference spectra (as necessary)

More... Back Next Finish Cancel

Detector type: Silicon Drift Detector
Detector window: Moxtek AP 3.3 (manufacturer's table)
Display name: Benitoite
Duane-Hunt: 14.92 keV
Elevation:
Energy offset:
Energy scale: 5.01 eV/channel
Gold layer thickness: 10 nm

Weight % Atomic %

5.26 ± 0.06	64.82 ± 0.16
4.40 ± 0.14	7.37 ± 0.03
9.72 ± 0.02	20.65 ± 0.05
1.66 ± 0.07	7.16 ± 0.04

You may specify the reference from a file or from the database (advanced.)

A region of interest for which you must specify a reference will be marked as **missing** in the S/N (signal-to-noise) column.

You may specify an element to fit but which will not be included in the final composition.

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Standards can act as references when the relevant regions of interest in the standard are not close to x-ray lines from other elements.



This is the spectrum you selected in step 0.

You may specify addition spectrum to quantify here.

If the current correction algorithm supports complex geometries, you can specify the sample shape using this button. (Citzaf supports particles)

You can update the live time or probe current using the properties button.

NIST DTSA-II

Quantification Alien

Specify unknown spectra

Previous: [Specify the reference spectra](#)

Next: [The results](#)

Name: Benitoite

Live Time: 59.4

Probe (nA): 2.500

Shape: Bulk

Add file

Remove

Properties

Sample Shape

Weight %

Atomic %

5.26 ± 0.06

4.40 ± 0.14

9.72 ± 0.03

64.82 ± 0.16

7.37 ± 0.03

20.65 ± 0.05

Detector type: Silicon Drift Detector

Detector window: Benitoite A (333 (manufacturer's table))

Sample thickness: 14.02 kV

Elevation: 40

Energy offset: -478.8 eV

Energy scale: 5.01 eV/channel

Gold layer thickness: 0 nm

Spectrum Report Command

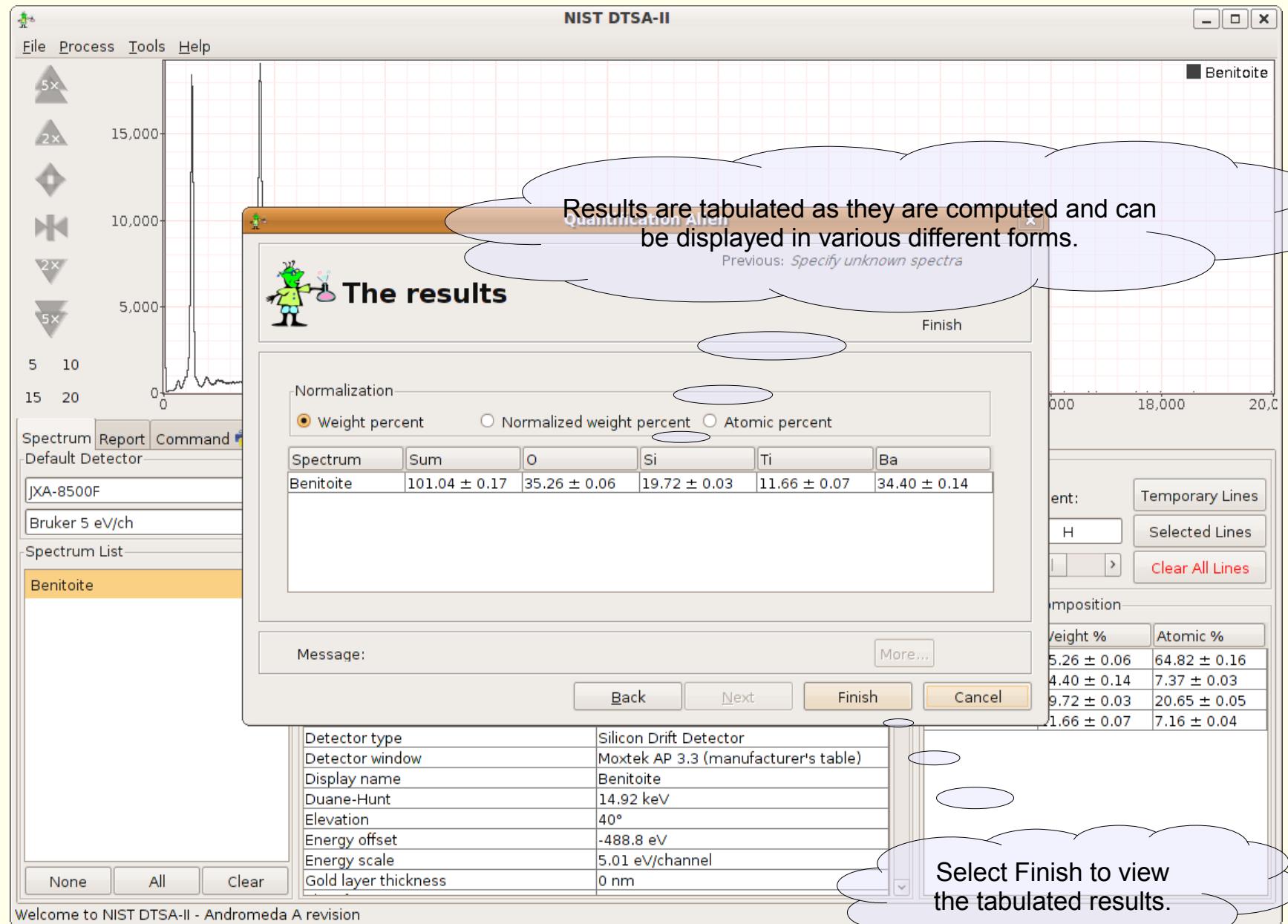
Default Detector: JXA-8500F

Spectrum List: Benitoite

None All Clear

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The File – Preferences dialog allows you to specify the quantification algorithm. (XPP is recommended for bulk samples, Citzaf for particles.)





Select the report tab

Spectrum Report Command

Composition from MLLSQ fit to standards

Conditions

Item	Value
Instrument	JXA-8500F
Detector	Bruker 5 eV/ch
Beam Energy	15.0 keV
Correction Algorithm	XPP - Pouchou & Pichoir Simplified (Non-normal)
Mass Absorption Coefficient	NIST-Chantler 2005

Standards

Element	Material	Spectrum	Ref?	Probe (nA)	Live Time (s)
O	MgO = [O(0.5 atoms),Mg(0.5 atoms)]	MgO	Yes	2.500	59.3
Si	Pure silicon = [Si(100.00 wgt%),2.4 g/cc]	Si	Yes	2.500	59.4
Ti	Pure titanium = [Ti(100.00 wgt%),4.5 g/cc]	Ti	Yes	2.500	59.4
Ba	BaF2 = [F(0.67 atoms),Ba(0.33 atoms)]	BaF2	Yes	2.500	59.4

Results

Spectrum	Quantity	O			Si			Ti			Ba			Sum
		Line	O All		Si All		Ti K-family		Ba L-family					
Benitoite	Z · A · F	1.14	0.63	1.00	1.09	0.72	1.00	0.95	0.98	1.01	0.82	1.02	1.00	
Bulk	k-ratios	0.6392	±	0.0010	0.1547	±	0.0002	0.1090	±	0.0006	0.3673	±	0.0015	
	weight %	35.26	±	0.06	19.72	±	0.03	11.66	±	0.07	34.40	±	0.14	
I = 2.500 nA	norm(wgt %)	34.90	±	0.06	19.52	±	0.03	11.54	±	0.07	34.05	±	0.14	
LT = 59.4 s	atomic %	64.82			20.65			7.16			7.37			
Residual		/home/nicholas/DTSA-II Reports/2009/May/24-May-2009/residual2361900809694010741.msa												

Table: Quantitative results (uncertainties are statistical-only, 1 σ)

Conditions and configuration

Standards spectra and parameters

Results



DTSA-II Report - May 24, 2009

File Edit View Go Bookmarks Tools Tabs Help

file:///home/nicholas/DTSA-II%20Reports/2009/May/24-May-2009/index1.html Go

Composition from MLLSQ fit to standards

Conditions

Item	Value
Instrument	JXA-8500F
Detector	Bruker 5 eV/ch
Beam Energy	15.0 keV
Correction Algorithm	XPP - Pouchou & Pichoir Simplified (Non-normal)
Mass Absorption Coefficient	NIST-Chantler 2005

Standards

Element	Material	Spectrum	Ref?	Probe (nA)	Live Time (s)
O	MgO = [O(0.5 atoms),Mg(0.5 atoms)]	MgO	Yes	2.500	59.3
Si	Pure silicon = [Si(100.00 wgt%),2.4 g/cc]	Si	Yes	2.500	59.4
Ti	Pure titanium = [Ti(100.00 wgt%),4.5 g/cc]	Ti	Yes	2.500	59.4
Ba	BaF2 = [F(0.67 atoms),Ba(0.33 atoms)]	BaF2	Yes	2.500	59.4

Results

Table: Quantitative results (uncertainties are statistical-only, 1σ)

Spectrum	Quantity	O	Si	Ti	Ba	Sum			
Benitoite Bulk	Line	O All	Si All	Ti K-family	Ba L-family				
	Z · A · F	1.14	0.63	1.00	0.95	0.98	1.01		
	k-ratios	0.6392	\pm 0.0010	0.1547	\pm 0.0002	0.1090	\pm 0.0006		
weight %	35.26	\pm 0.06	19.72	\pm 0.03	11.66	\pm 0.07	34.40	\pm 0.14	101.04
I = 2.500 nA norm(wgt %)	34.90	\pm 0.06	19.52	\pm 0.03	11.54	\pm 0.07	34.05	\pm 0.14	-
LT = 59.4 s atomic %	64.82		20.65		7.16		7.37		
Residual	/home/nicholas/DTSA-II Reports/2009/May/24-May-2009/residual2361900809694010741.msa								



Results

Table: Quantitative results (uncertainties are statistical-only, 1σ)

Spectrum	Quantity	O			Si			Ti			Ba			Sum
<u>Benitoite</u> Bulk	Line	O All			Si All			Ti K-family			Ba L-family			
	Z · A · F	1.14	0.63	1.00	1.09	0.72	1.00	0.95	0.98	1.01	0.82	1.02	1.00	
	k-ratios	0.6392	\pm	0.0010	0.1547	\pm	0.0002	0.1090	\pm	0.0006	0.3673	\pm	0.0015	
	weight %	35.26	\pm	0.06	19.72	\pm	0.03	11.66	\pm	0.07	34.40	\pm	0.14	101.04
I = 2.500 nA	norm(wgt %)	34.90	\pm	0.06	19.52	\pm	0.03	11.54	\pm	0.07	34.05	\pm	0.14	-
LT = 59.4 s	atomic %	64.82			20.65			7.16			7.37			
Residual	/home/nicholas/DTSA-II Reports/2009/May/24-May-2009/residual2361900809694010741.msa													

Results reported as:

Weight Percent:

Normally we report results like this

Normalize weight percent:

Used to report particle results

Atomic percent:

Relative to atom count rather than weight fraction

Also reported:

Line:

The x-ray line family used to calculate the composition

Z·A·F terms:

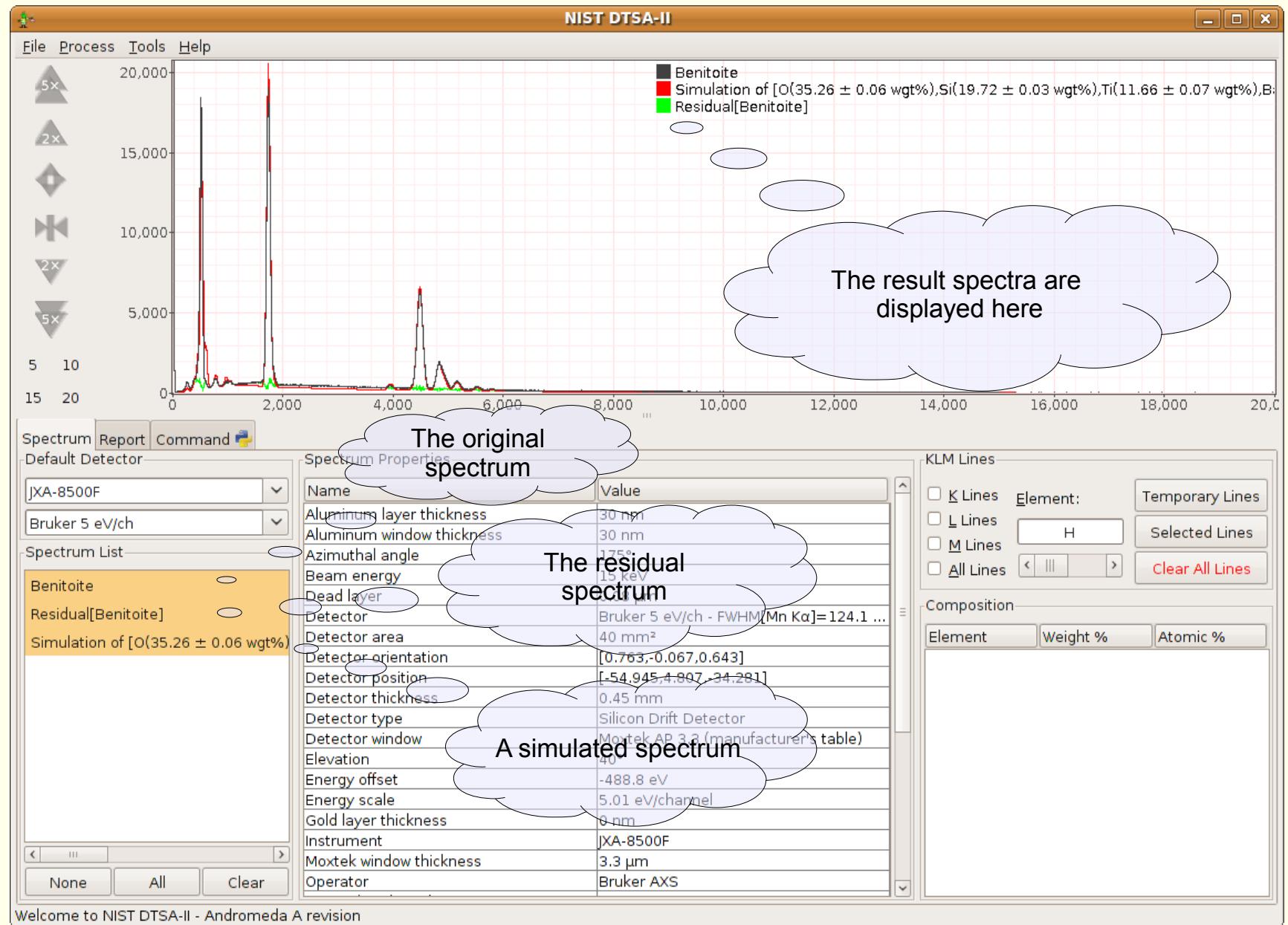
The quantitative correction factors

k-ratios:

The raw ratio between unknown and standard

Sum:

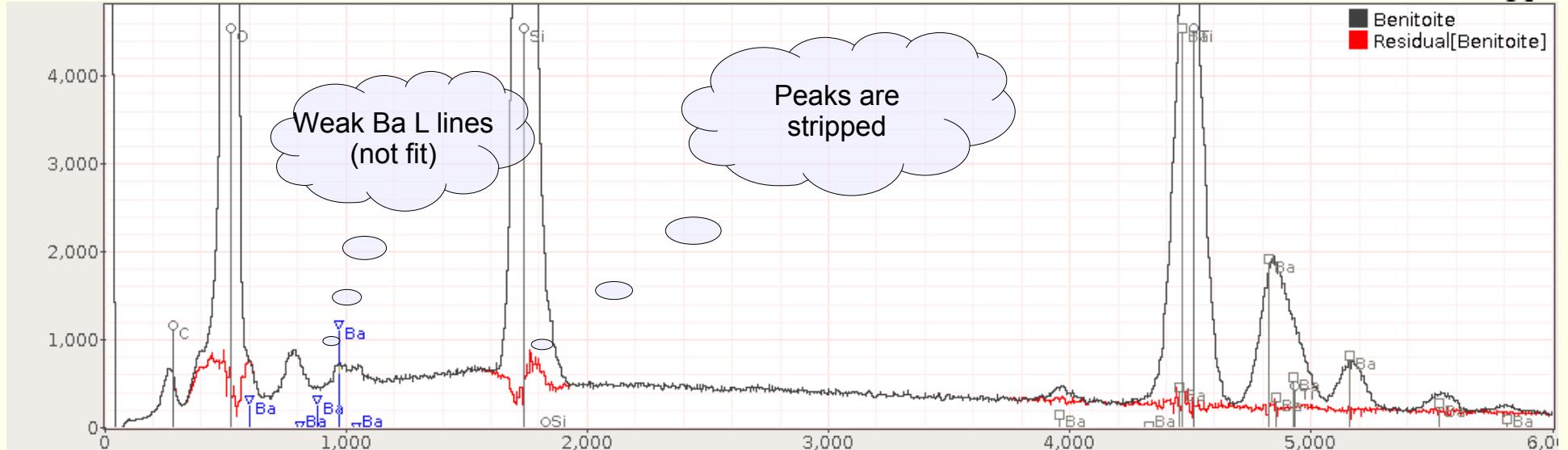
Also called the analytic total, this number should typically be close to 100 for good measurements of bulk materials.



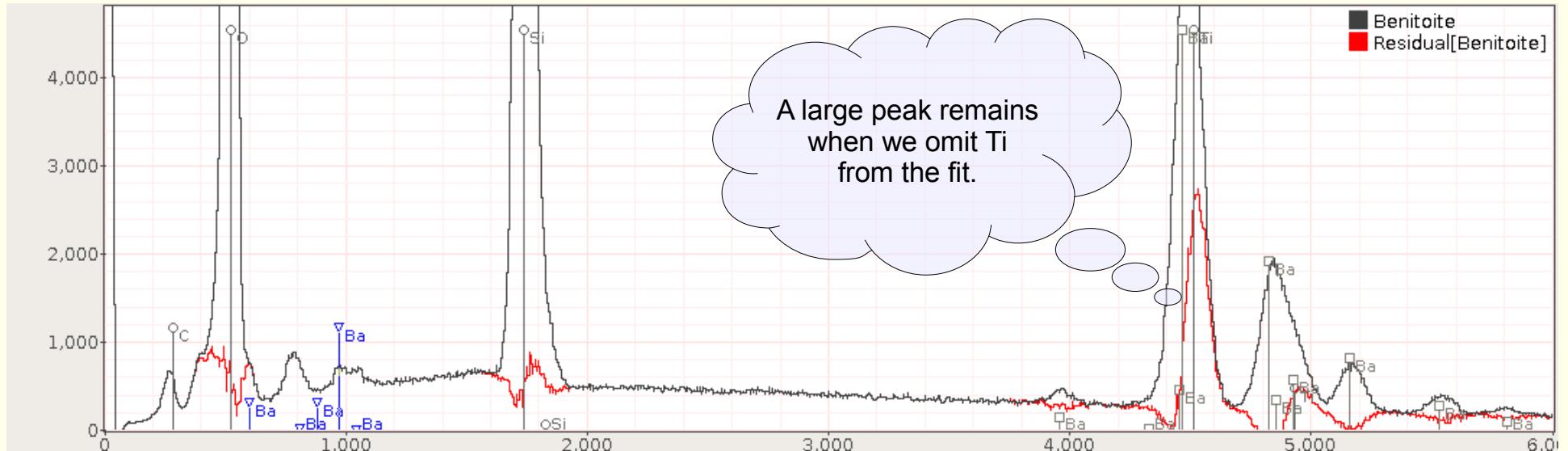
The residual spectrum is how you know you have not overlooked any elements.



A good residual spectrum



A bad residual spectrum

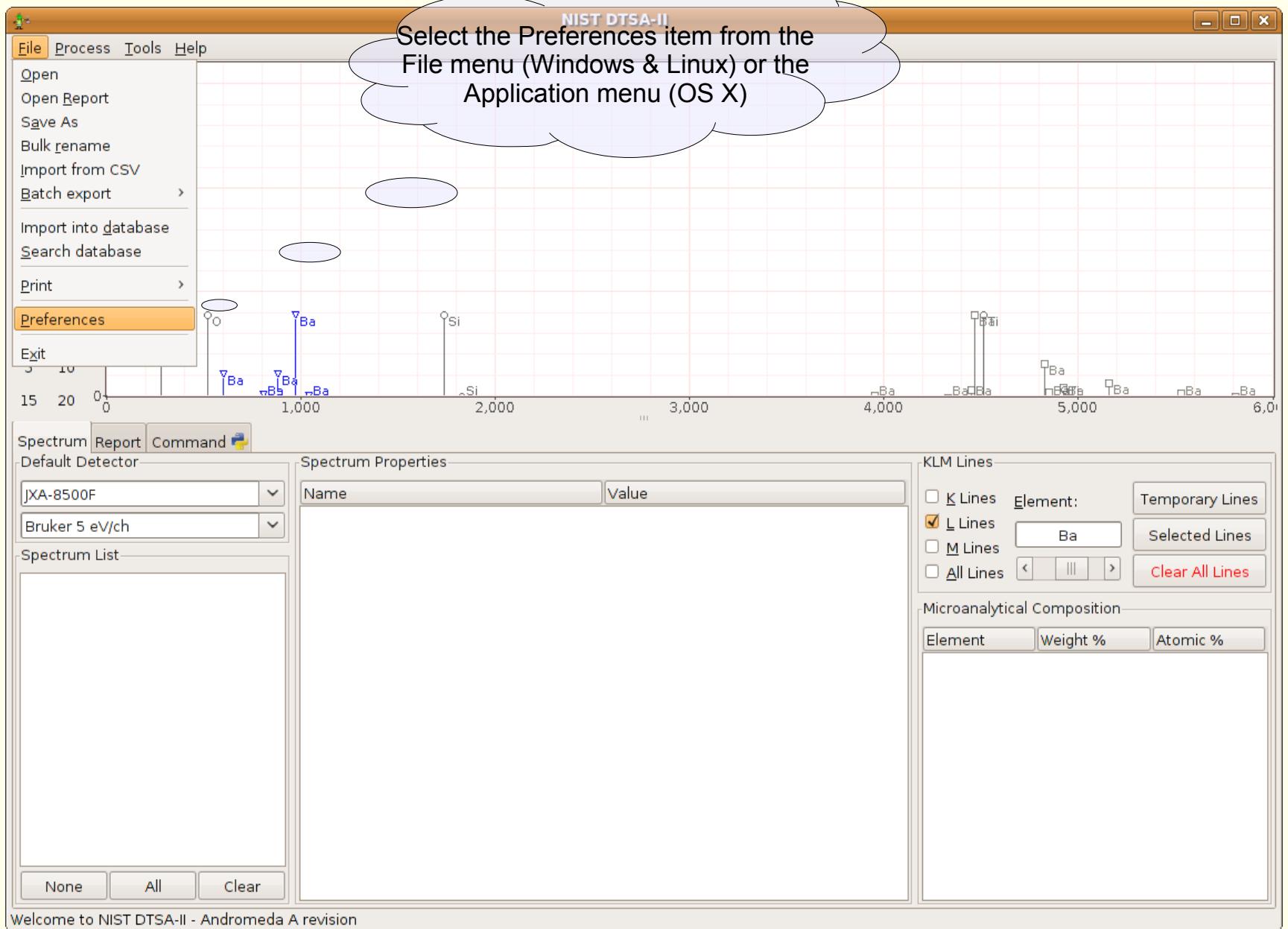


(The Sum (analytic total) was only 89% for the bad measurement.)



Defining detector models

- Define a unique detector model for each
 - eV/channel calibration;
 - resolution (pulse process time)
 - for each detector on each instrument
- Detector models define the
 - Energy calibration (eV/channel & eV offset);
 - Quantum efficiency as a function of energy;
 - Detector position & geometry





List of available instruments. In this case "Probe", "JXA-8500F" and "ASPEX"

NIST DTSA-II

List of defined detectors.
For the "JXA-8500F", the detectors
"Bruker 5 eV/ch" and "Bruker 10 eV/ch"
have been defined.

Spectrum Report Default Detector JXA-8500F Bruker 5 eV/ch Spectrum List

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

User Information Quantitative algorithms Instruments and Detectors ▾ Probe Detector - Si(Li) ▾ JXA-8500F Detector - Bruker 5 eV/ch Detector - Bruker 10 eV/ch ▾ ASPEX Detector - T=12.8us

Instruments and Detectors

Overview

DTSA-II makes extensive use of user defined detectors to define the instrumentation on which spectra are collected or on which spectra are to be simulated. In this model, all detectors are associated with instruments. Each instrument may have zero or more detectors. Detectors definitions contain all the relevant information about the performance of the detector including geometry, physical make-up and calibration. When a detector is first constructed, you supply an approximate default calibration. This calibration may be updated at any time using the Calibration alien in the Tools menu. Multiple calibrations may exist for a single detector and the optimal calibration for a specific

Add

Add or remove e-beam instruments and detectors.

OK Cancel Apply

Temporary Lines Selected Lines Clear All Lines Atomic %

Use the Add button on this page to create a new instrument.



Select the instrument to which you want to add a detector.

NIST DTSA-II

DTSA-II - Preferences

User Information
Quantitative algorithms
Instruments and Detectors
Probe
Detector - Si(Li)
JXA-8500F
Detector - Bruker 5 eV/ch
Detector - Bruker 10 eV/ch
ASPEX
Detector - T=12.8us

Instrument name: Probe

Add Si(Li) detector Add SDD detector Add microcalorimeter

Edit the preferences associated with an e-beam instrument.

OK Apply

Spectrum Report Default Detect JXA-8500F Bruker 5 eV/ch Spectrum List

nBa - Ba 6,01

Temporary Lines Selected Lines Clear All Lines

Atomic %

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

Adds a detector with typical properties for a Si(Li) detector.

Adds a detector with typical properties for a silicon drift detector (SDD).



NIST DTSA-II

File Process Tools Help

5x 15
2x 10
5x 5
5x 5
5 10
15 20 0 0

Spectrum Rep Default Detect

JXA-8500F

Bruker 5 eV/c

Spectrum List

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

User Information
Quantitative algorithms
Instruments and Detectors
Probe
Detector - Si(Li)
Detector - Basic SDD
JXA-8500F
ASPEX

DTSA-II - Preferences

Detector - Basic SDD

Status
Enable detector

Name
Detector name **Basic SDD**

Import
Import from spectrum **Import**

Window
Window **No window**

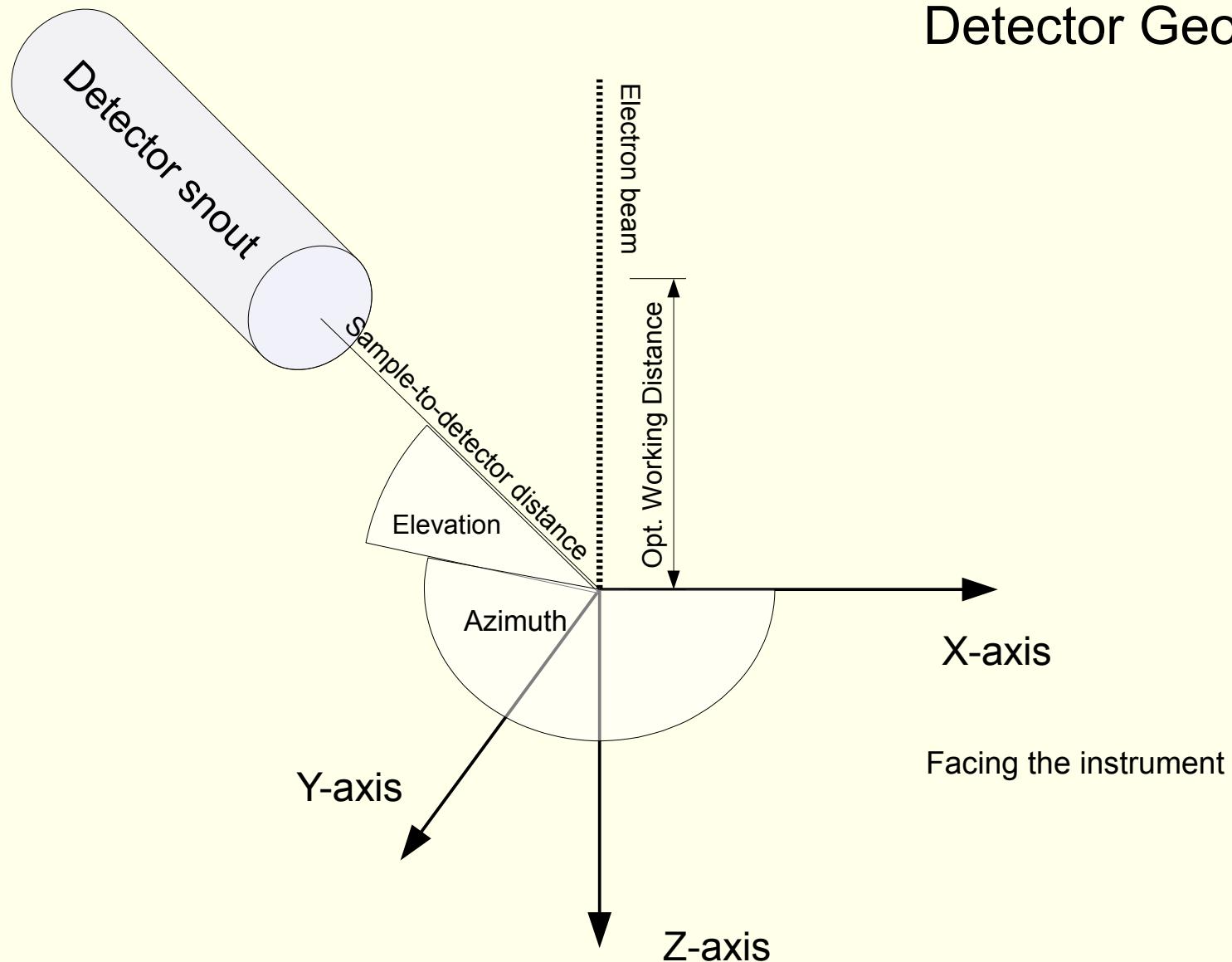
Position
Elevation angle **35.0 °**

Edit the properties of this detector.

OK Cancel Apply

Some properties of the detector can be imported from the tags in a spectrum file. This only works if the information is in the file and the information is correct.

Detector Geometry

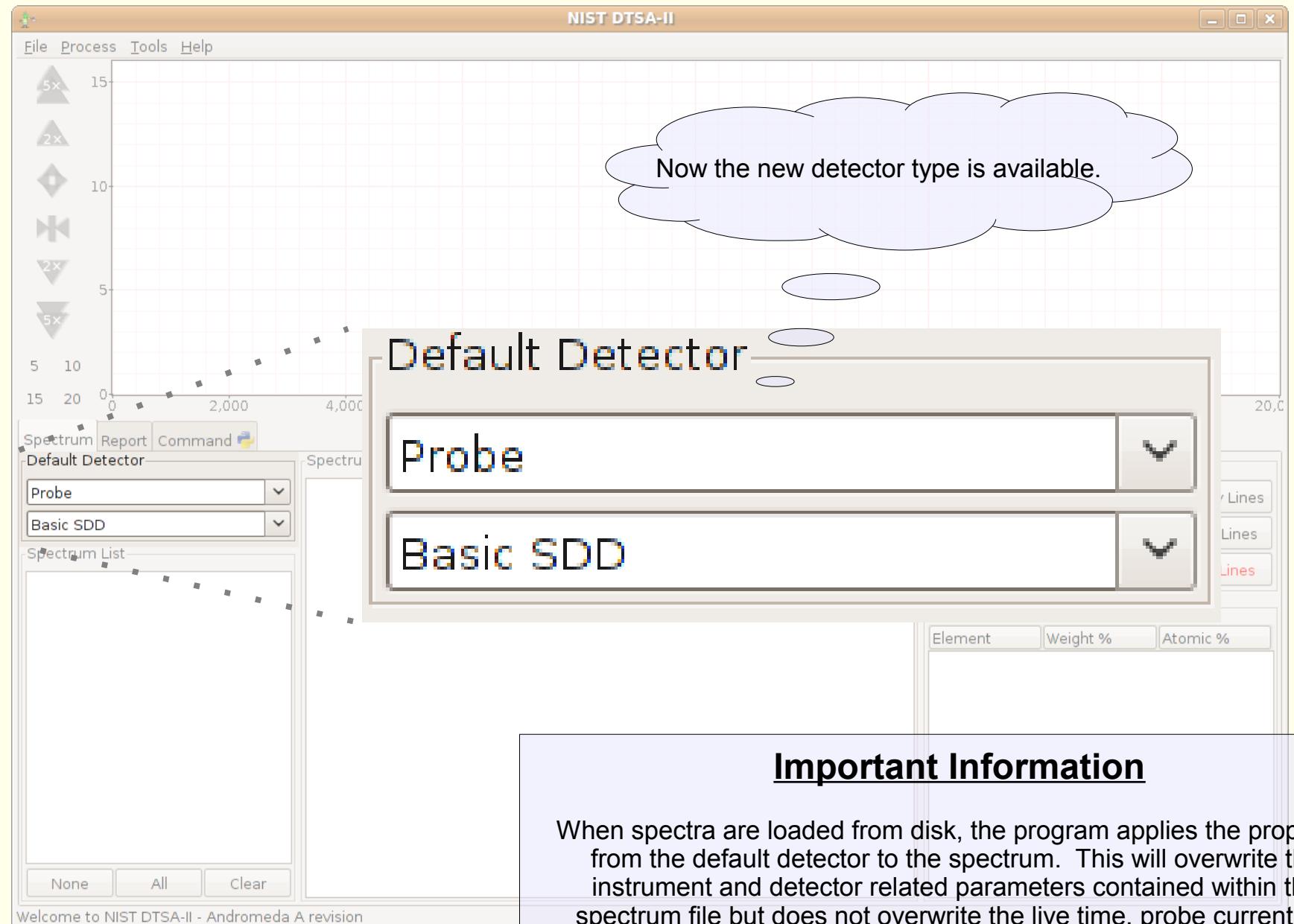


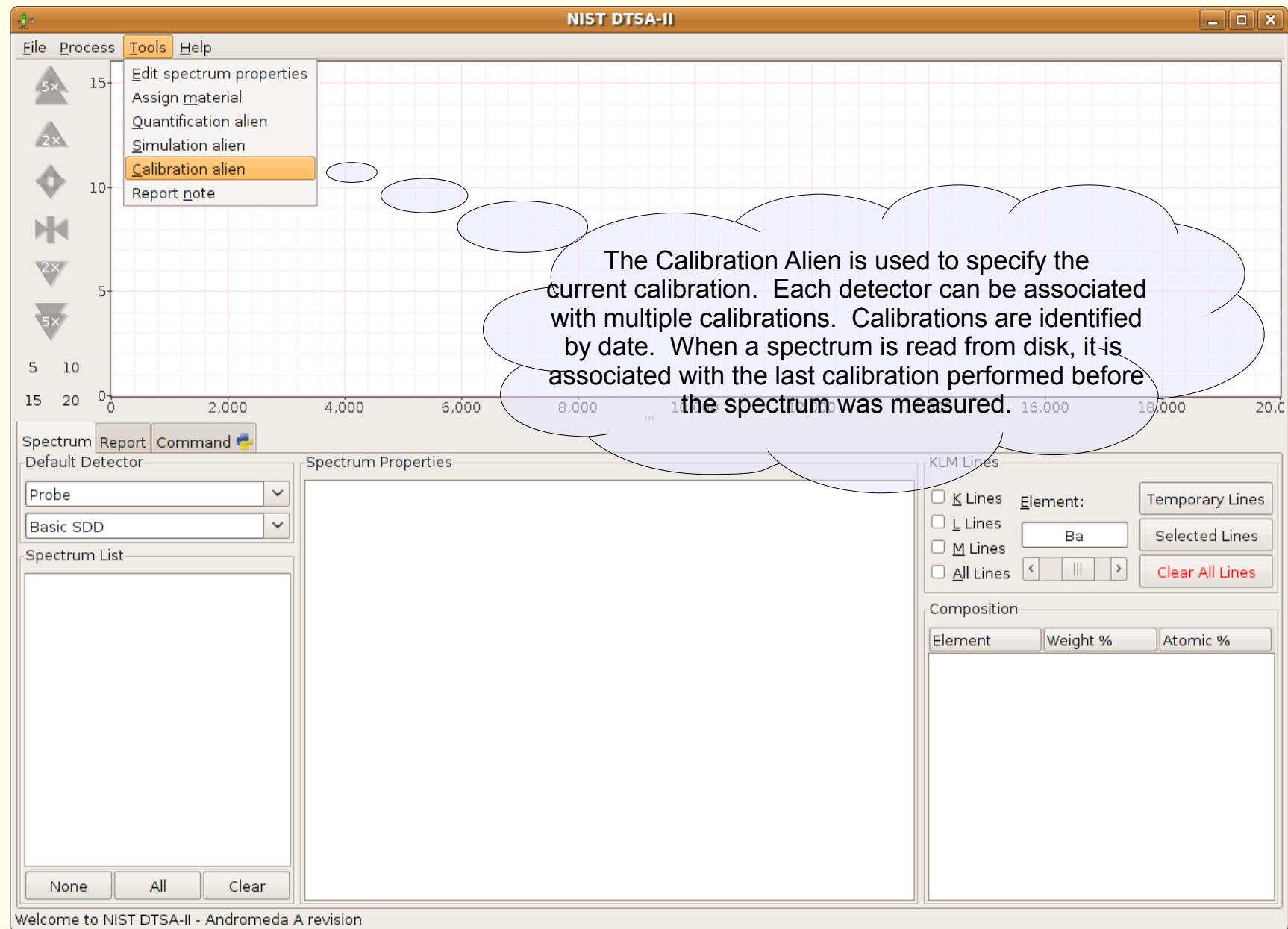
The sample-to-detector distance is measured from the sample position at the optimal working distance to the front face of the detector crystal. The optimal working distance is the distance through which a ray parallel to the detector axis intersects the electron beam axis. This is often the distance producing the highest x-ray flux.



Status	<input checked="" type="checkbox"/> Enable detector
Name	Detector name <input type="text" value="Basic SDD"/>
Import	Import from spectrum <input type="button" value="Import"/>
Window	Window <input type="button" value="No window"/> <input type="button" value=""/>
Position	Elevation angle <input type="text" value="35.0"/> ° Azimuthal angle <input type="text" value="0.0"/> ° Optimal working distance <input type="text" value="20.0"/> mm Sample-to-detector distance <input type="text" value="50.0"/> mm
Crystal parameters	Detector Area <input type="text" value="10.0"/> mm² Gold layer <input type="text" value="0.0"/> nm Aluminum layer <input type="text" value="10.0"/> nm Dead layer <input type="text" value="0.0"/> µm Thickness <input type="text" value="0.45"/> mm
Configuration	Number of channels <input type="text" value="4096"/> channels Zero strobe discriminator <input type="text" value="0.0"/> eV
Base Performance	Energy scale <input type="text" value="5.0"/> eV/channel Zero offset <input type="text" value="0.0"/> eV Resolution <input type="text" value="128.0"/> eV at Mn Ka

- ✓ Your EDS vendor can typically provide the window material. Beryllium windows typically can see K lines below about sodium. The Moxtek AP3.3 is a common modern polymer window.
- ✓ The elevation & azimuthal angle, optimal working distance and sample-to-detector distances are detailed on the previous slide.
- ✓ Your EDS vendor can typically provide nominal values for the crystal parameters. These nominal values are rarely precise.
- ✓ The number of channels is typically either 2048 for 10 eV/channel or 4096 for 5 eV/channel. Either of these choices provides a little over a 20 keV measurement range. Occasionally, detectors are configured for 4096 at 10 eV/channel for a range of energies exceeding 40 keV.
- ✓ Some detectors use a zero strobe to stabilize the detector offset. Some automatically remove this peak while others leave the peak visible. If it is visible, use this setting to trim it out of the spectrum when processing the spectrum.
- ✓ The energy scale and zero offset are very important parameters. Make certain they are approximately correct. You can refine these values using the “Calibration Alien” but if they are too far off the calibration alien will not work correctly.
- ✓ Specify the nominal resolution (full-width half-maximum) for a Manganese Ka x-ray. This is typically between about 123 eV for a modern high resolution detector and 150 eV for an older detector. The “Calibration Alien” allows you to refine these values using a measured spectrum.
- ✓ You won't be permitted to the base performance after you have created the detector.







NIST DTSA-II

File Process Tools Help

5x 15
2x 10
10x 5
2x 5
5x 0 15 20 0 2,000

Spectrum Report Command
Default Detector
Probe
Basic SDD
Spectrum List
None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

Calibrate an EDS detector

Select a detector

First page

Next: Calibration methods

Instrument and Detector

Instrument: Probe

Detector: Basic SDD - FWHM[Mn K α]=124.0 eV - initial

Message: Select a detector to calibrate

Back Next Finish Cancel

First, specify which detector to calibrate.

Temporary Lines
Selected Lines
Clear All Lines
Weight % Atomic %

Lehigh
Microscopy
School



NIST DTSA-II

File Process Tools Help

5x 15
2x 10
10x 5
2x 5
5x 15 20 0 0 2,000

Spectrum Report Command
Default Detector
Probe
Basic SDD
Spectrum List
None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

Calibrate an EDS detector

Calibration method

Specify a calibration method

Manually enter detector calibration

Calibrate using an elemental reference

Calibrate using the BAM EDS CRM

Administer calibrations

Review and administer available calibrations.

Message: Select a spectrum calibration method More...

Back Next Finish Cancel

Select "calibrate using an elemental reference" to extract the calibration precisely from a measured spectrum.

Previous: *Select a detector*

Next: *Measured spectrum*

Element: Temporary Lines
Ba Selected Lines
Clear All Lines

Weight % Atomic %



NIST DTSA-II

Specify a spectrum file collected from a material with both low energy (< 2 keV) lines and higher energy lines (>5 keV) lines.

2x
10x
5x
5x
10x
15x 20x 0 0 2,000

Spectrum Report Command
Default Detector
Probe
Basic SDD
Spectrum List

Measured spectrum

Previous: Calibration method
Next: Fit Results

Specify the material
Material Mn standard

Specify a spectrum
Spectrum Mn

Live time 59.34 sec. Probe current 2.500 nA

Specify an effective date
Effective date Jul 29, 2008

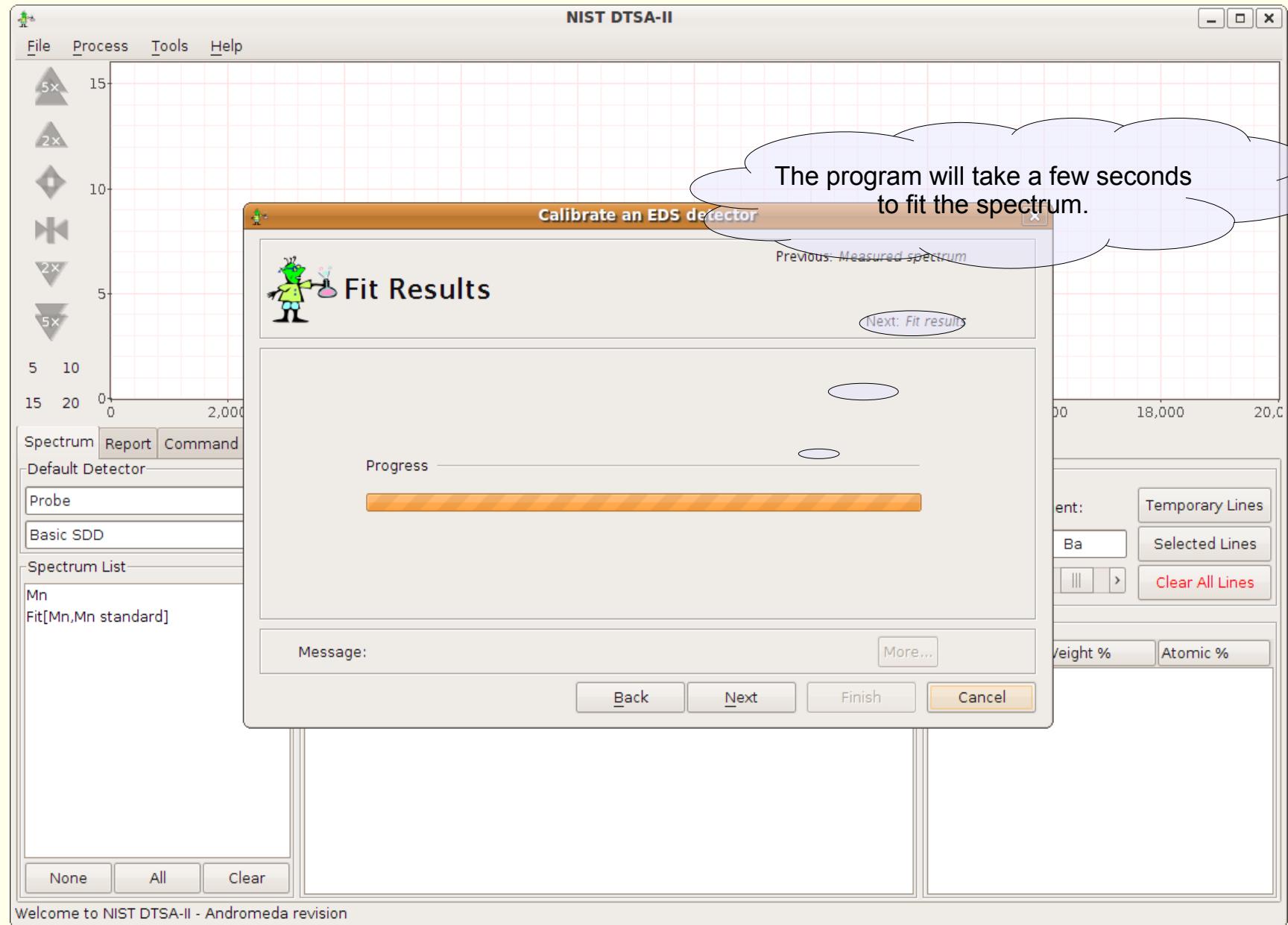
Message: The reference beam energy is 15.0 keV.

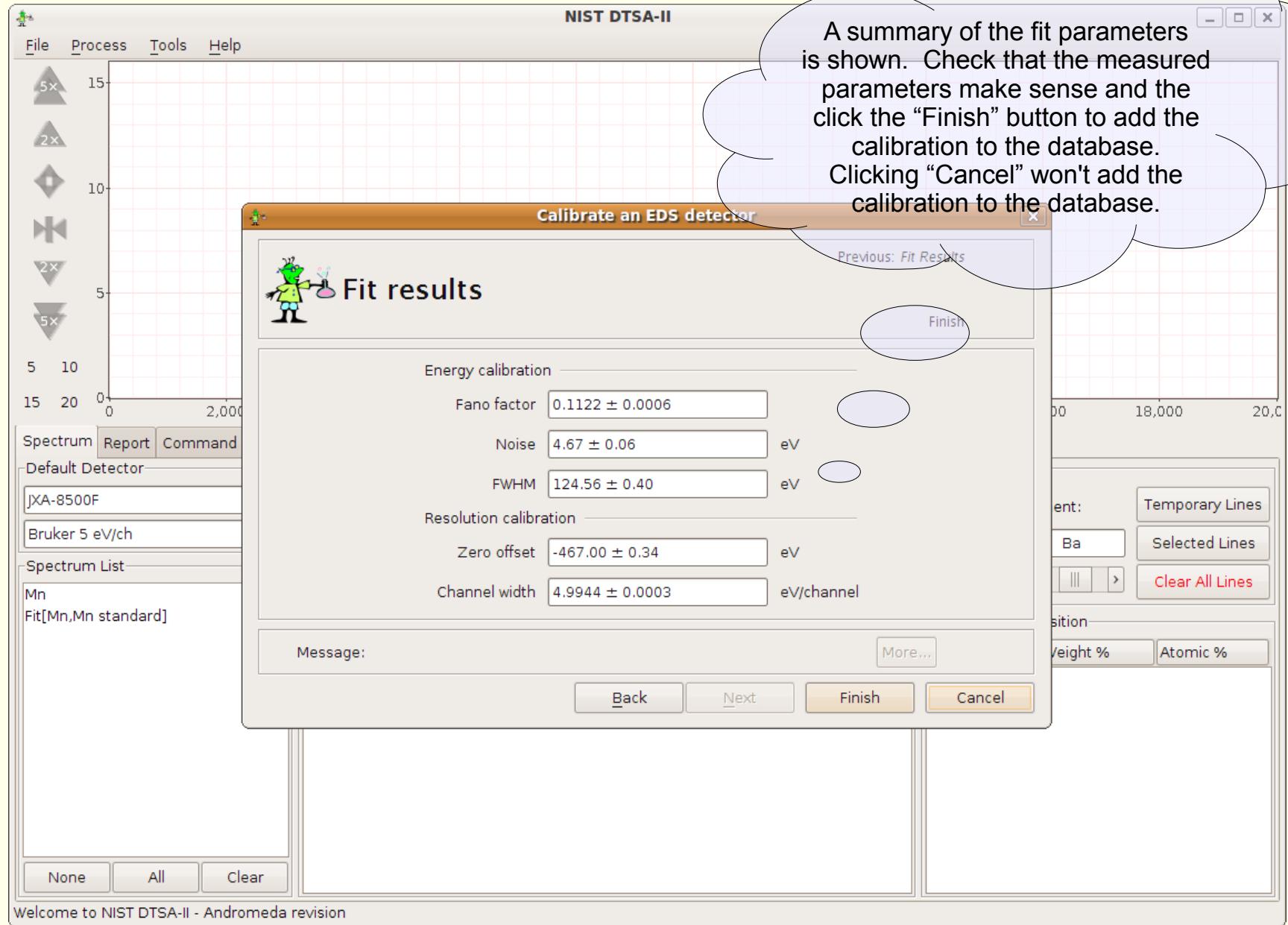
Back Next Finish Cancel

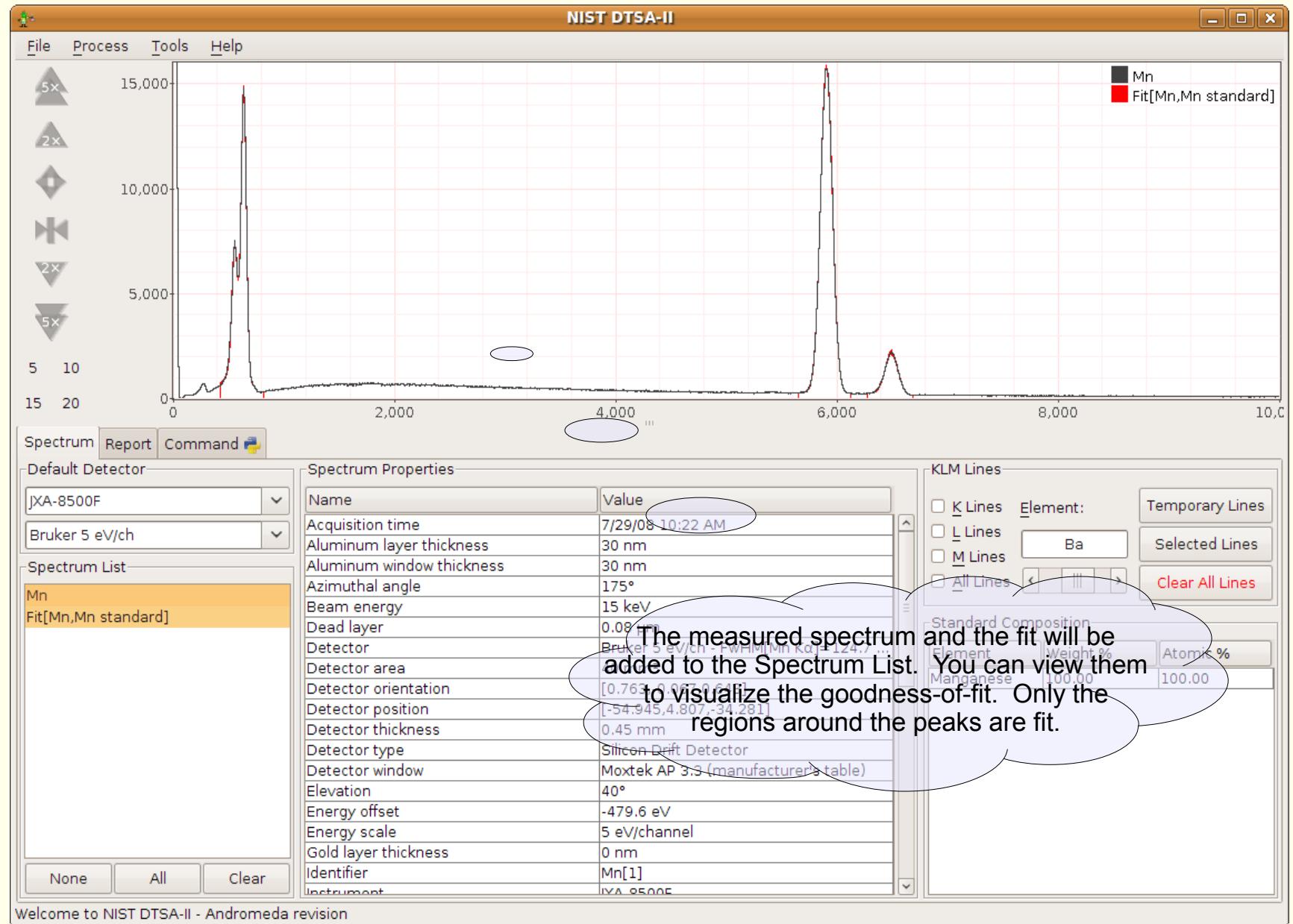
All spectra collected after 12:01 AM on the effective date but before 12:01 AM of the effective date of the next calibration will be associated with this calibration.

Welcome to NIST DTSA-II - Andromeda A revision

Specify the material from which the spectrum was collected.









Mn fit as Mn standard

Transition	Intensity	Gaussian Width (eV)	FWHM (eV)	Tail height	Tail Width	Channel	Energy (eV)
Mn K-L2	13 ± 0	52.9 ± 0.2	124.5 ± 0.4	0.0 ± 0.0	155 ± 69	1272.3 ± 0.1	5888
Mn K-L3	414949 ± 1103	52.9 ± 0.2	124.6 ± 0.4	135.5 ± 75.7	155 ± 69	1274.6 ± 0.1	5899
Mn K-M2	21163 ± 281	55.2 ± 0.2	130.0 ± 0.4	86.7 ± 11.6	198 ± 27	1393.0 ± 0.1	6490
Mn K-M3	36187 ± 821	55.2 ± 0.2	130.0 ± 0.4	148.3 ± 20.1	198 ± 27	1393.0 ± 0.1	6490
Mn K-M5	2562 ± 824	55.4 ± 0.2	130.4 ± 0.4	10.4 ± 3.6	198 ± 27	1401.3 ± 0.1	6532
Mn L1-M2	2150 ± 57	24.8 ± 0.1	58.3 ± 0.3	8.8 ± 1.8	61 ± 8	237.7 ± 0.1	720
Mn L1-M3	2799 ± 97	24.8 ± 0.1	58.3 ± 0.3	11.4 ± 2.4	61 ± 8	237.7 ± 0.1	720
Mn L2-M1	29530 ± 3949	23.4 ± 0.1	55.1 ± 0.3	127.6 ± 31.3	61 ± 8	207.1 ± 0.1	568
Mn L2-M3	4364 ± 2159	23.7 ± 0.1	55.9 ± 0.3	18.6 ± 10.0	61 ± 8	214.2 ± 0.1	603
Mn L2-M4	168002 ± 1089	24.1 ± 0.1	56.7 ± 0.3	704.6 ± 145.2	61 ± 8	222.5 ± 0.1	644
Mn L2-N1	11 ± 0	24.2 ± 0.1	56.9 ± 0.3	0.0 ± 0.0	61 ± 8	223.9 ± 0.1	651
Mn L3-M1	44846 ± 3295	23.3 ± 0.1	54.9 ± 0.3	195.5 ± 42.7	61 ± 8	204.9 ± 0.1	556
Mn L3-M4	3 ± 0	24.0 ± 0.1	56.5 ± 0.3	0.0 ± 0.0	61 ± 8	220.3 ± 0.1	633
Mn L3-M5	3 ± 0	24.0 ± 0.1	56.5 ± 0.3	0.0 ± 0.0	61 ± 8	220.3 ± 0.1	633

Element	Family	Intensity
Manganese	K-family	474874 ± 689
Manganese	K α	414962 ± 644
Manganese	K β	59911 ± 245
Manganese	L-family	251707 ± 502
Manganese	L α	6 ± 2
Manganese	L β	177315 ± 421
Manganese	L γ	11 ± 3

Energy Calibration

Zero Offset (eV)	Channel Width (eV/ch)	Quadratic Scale ($\times 10^{-6}$ eV/ch 2)
-467.0 ± 0.3	4.9944 ± 0.0003	0.0

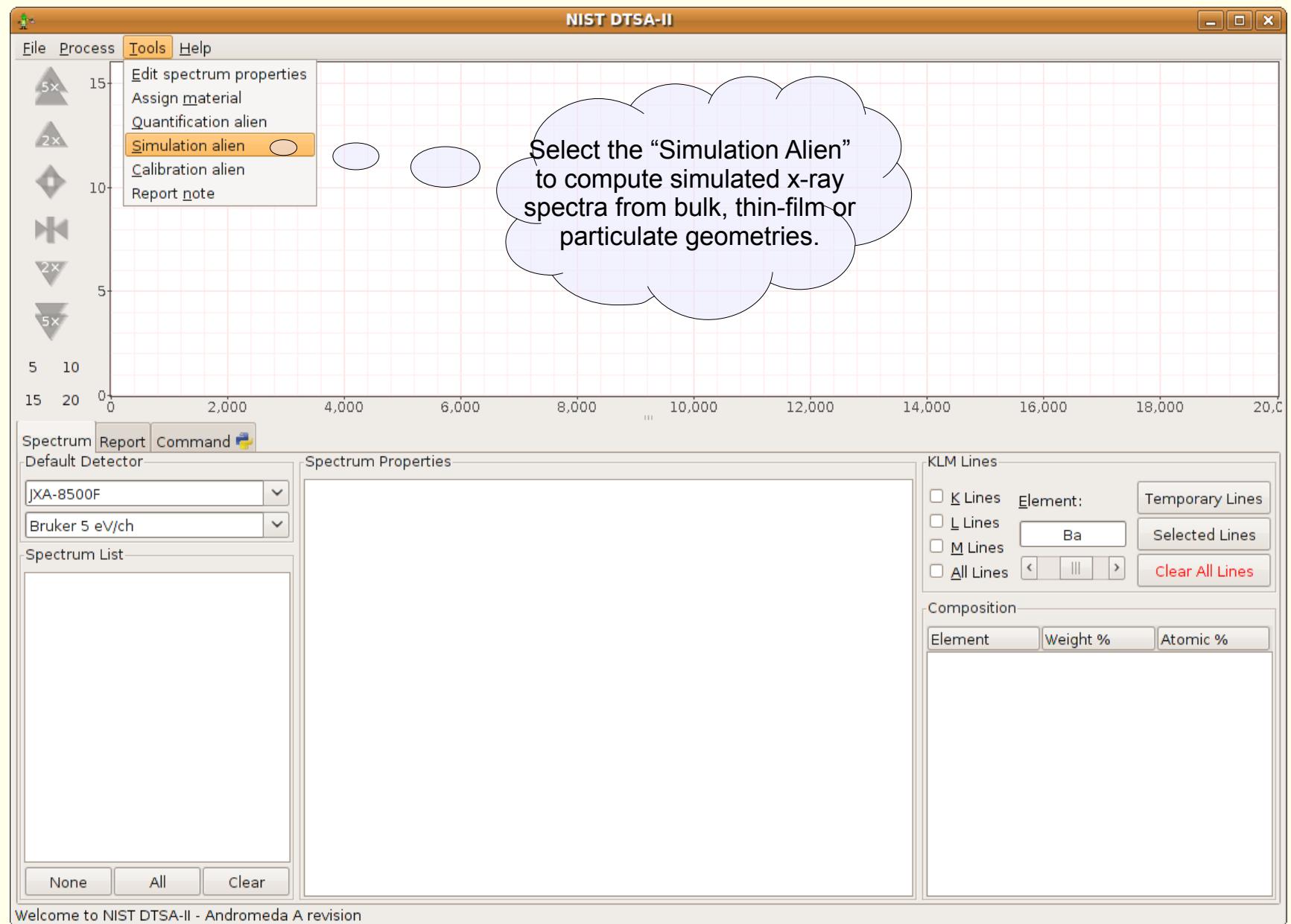
The Report also contains summary information from the calibration fit.

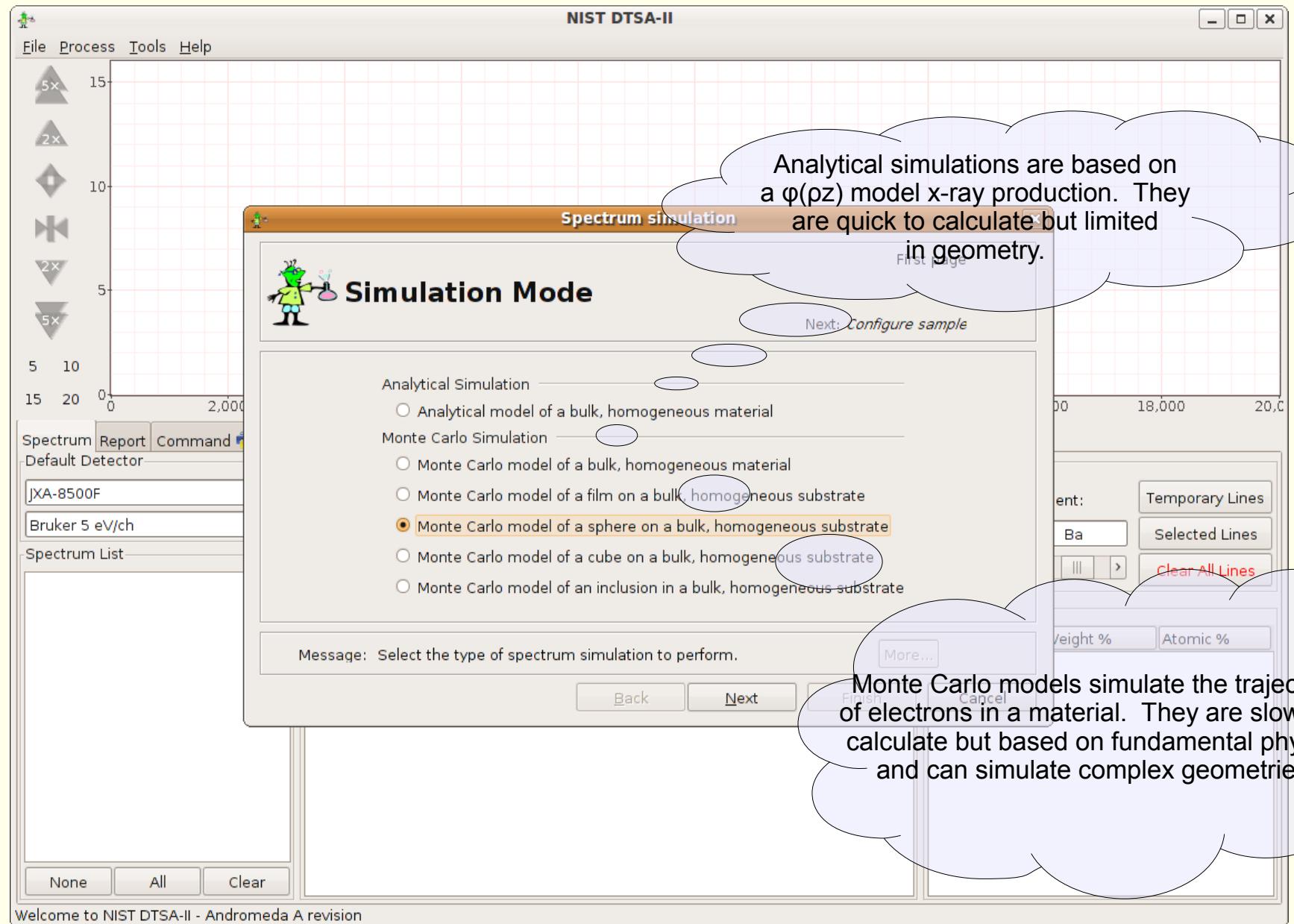
Bremsstrahlung

Linear	Quadratic
317607.9 ± 11358.5	136.0 ± 0.0

Detector Resolution

Fano Factor	Noise (eV)	FWHM at Mn K α (eV)
0.1122 ± 0.0006	4.6730 ± 0.0574	124.6 ± 0.4







NIST DTSA-II

File Process Tools Help

5x 15x 2x 5x 5x 10x 15x 20x 0x 2,000

Spectrum Report Command Default Detector Bruker 5 eV/chan Spectrum List

Configure sample

Spectrum simulation Previous: Simulation Mode Next: Instrument configuration

Materials and Scale

Substrate material Carbon Edit None

Sphere material Albite Edit

Sphere diameter 1.2 μm

Overscan particle

Message: Specify the sample material and scale. More...

Back Next Finish Cancel

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

Specify a material for the substrate and for the particle using the edit button and the material editor. The none button eliminates the substrate.

Specify the scale of the particle in microns.

The electron beam is usually placed at a single point at the center of the particle. Select this check box to raster the beam over the area of the particle.



NIST DTSA-II

File Process Tools Help

5x 15
2x 10
10x 5
2x 5
5x 0

Spectrum Report Command
Default Detector
JXA-8500F
Bruker 5 eV/ch
Spectrum List

Setting the incidence angle to a positive value tilts the sample towards the detector.

Spectrum simulation

Instrument configuration

Instrument Parameters

Instrument: JXA-8500F
Detector: Bruker 5 eV/ch
Calibration: FWHM[Mn K α]=124.7 eV - Sep 2, 2013
Beam Energy: 15 keV
Probe Dose (current-time): 120.0 nA·second
Incident Angle: 0.0 °

Previous: Configure sample
Next: Other options

Message: More... Back Next Finish Cancel

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

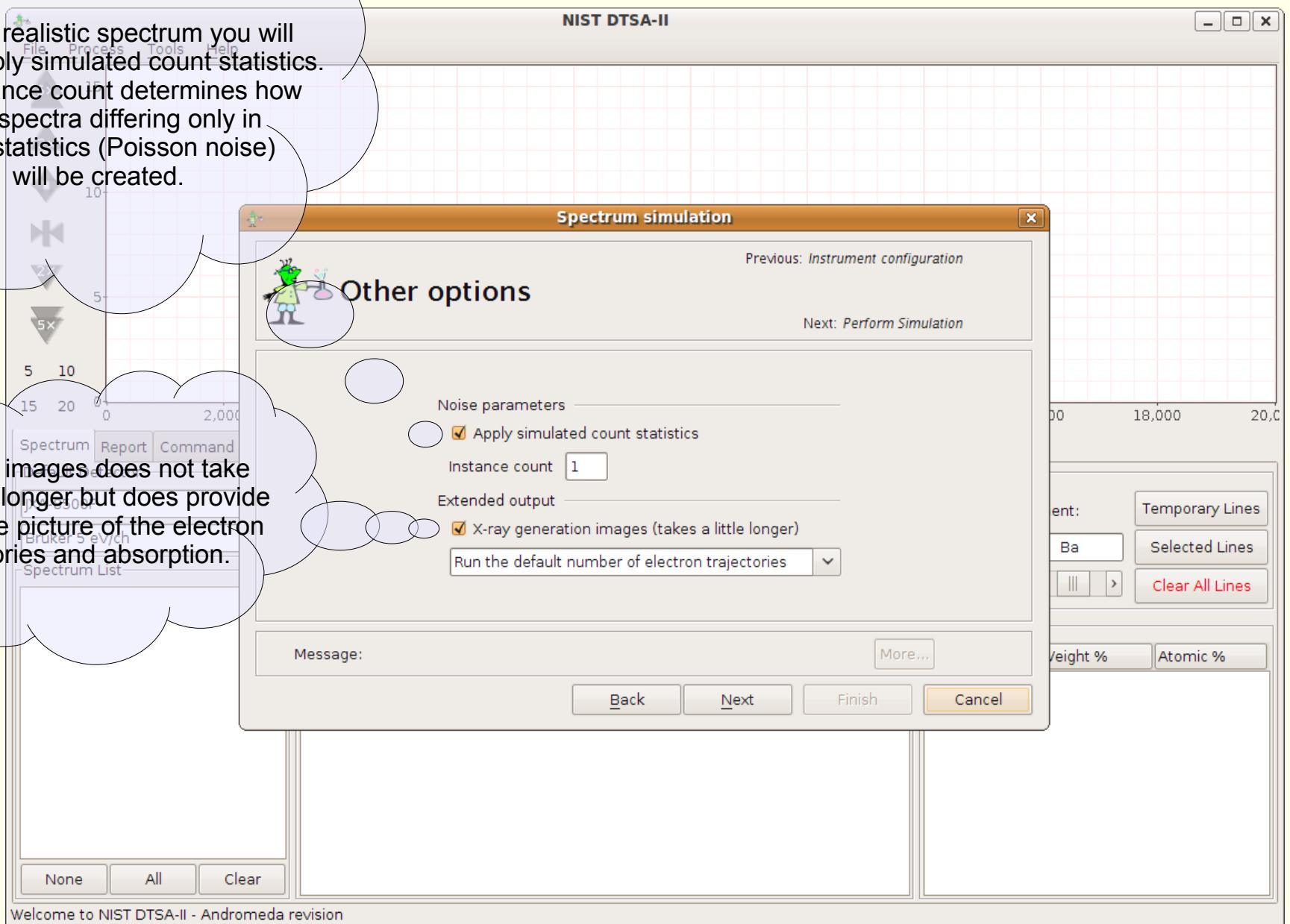
Simulation attempts to replicate the spectrum you would collect on your instrument with your detector. Specify these here.

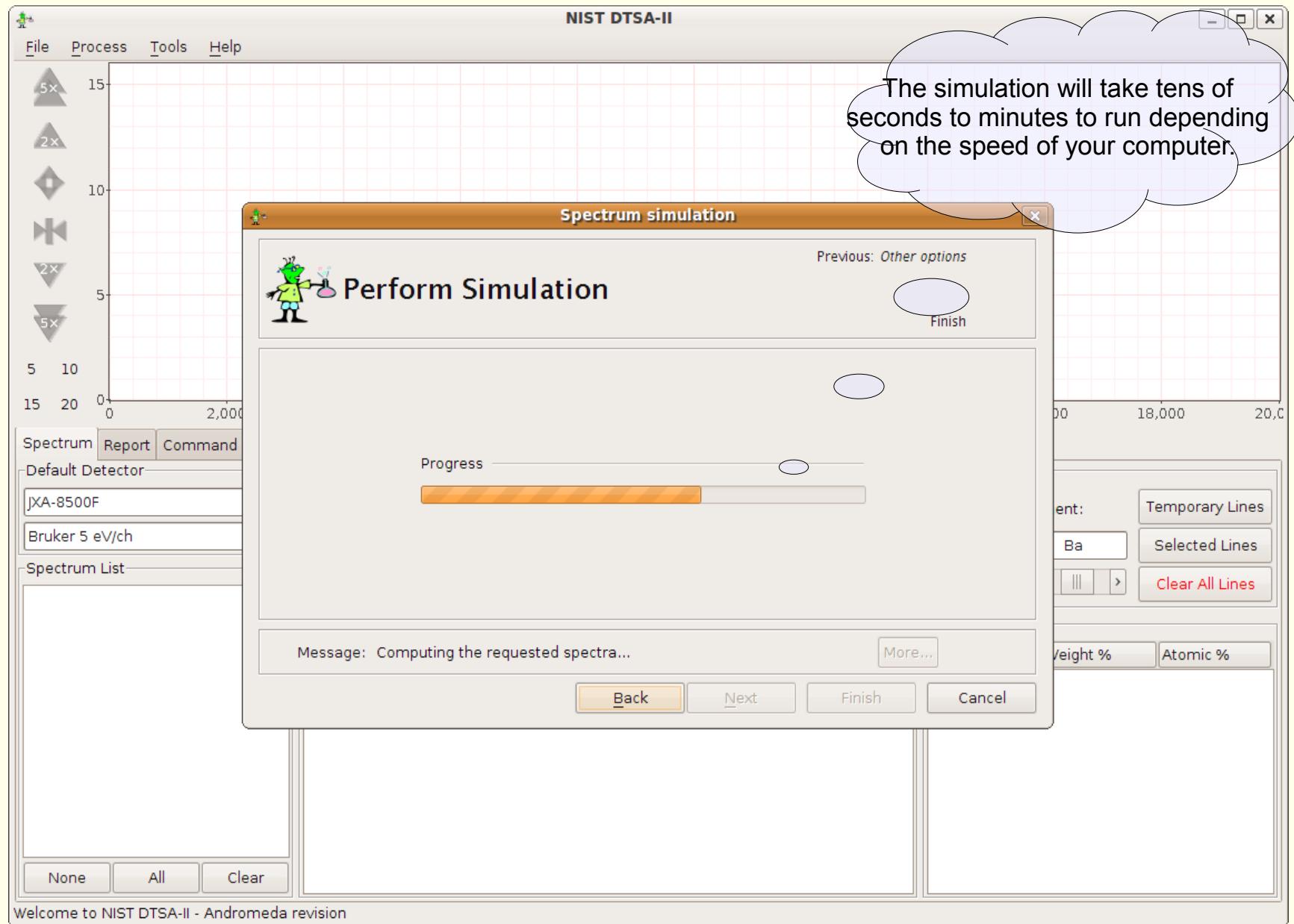
If the detector is configured correctly, the simulated spectrum will approximately match a measured spectrum collected with the same probe dose.
Probe dose is
 $(\text{probe current}) \cdot (\text{live time})$

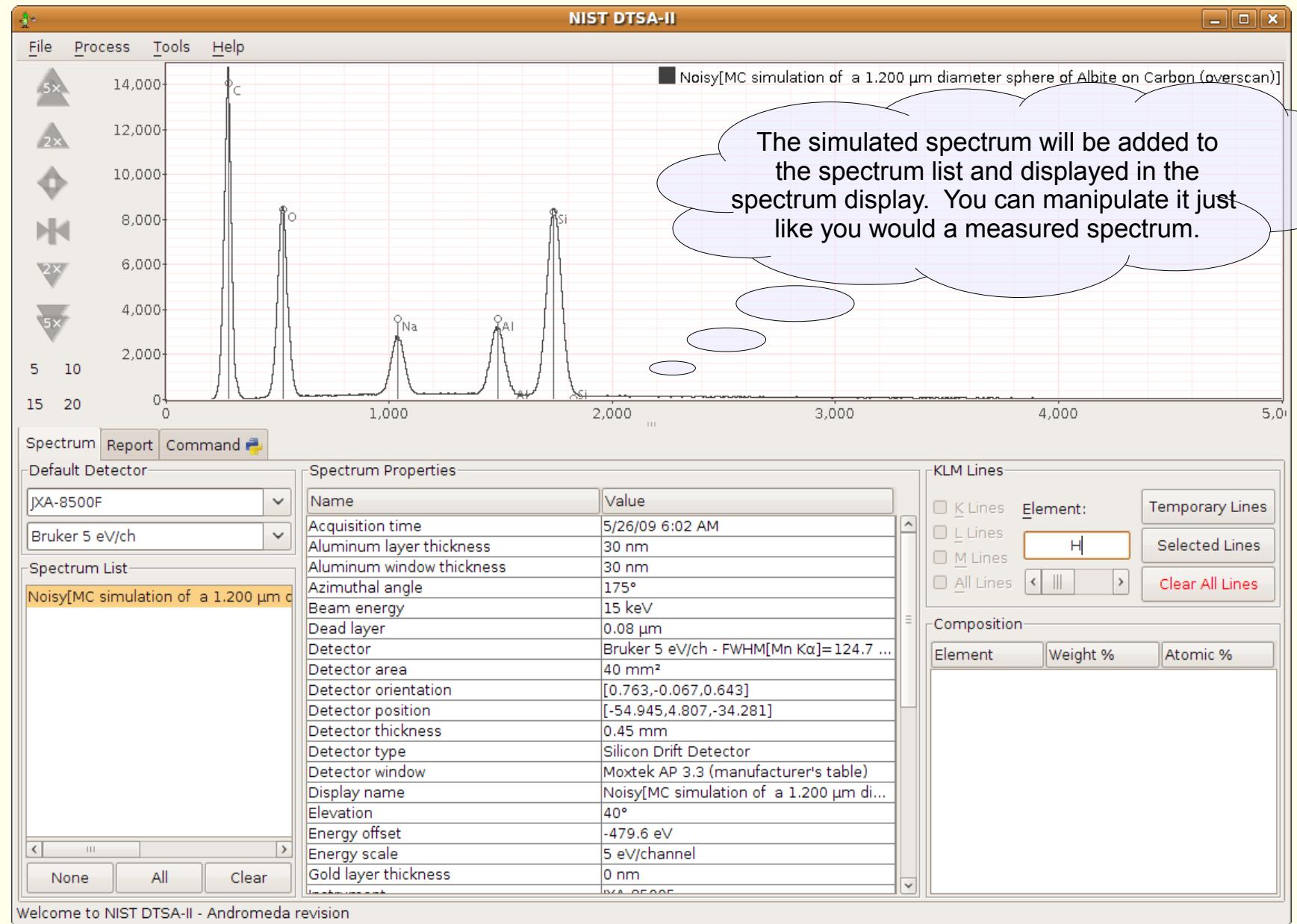


To get a realistic spectrum you will want to apply simulated count statistics. The instance count determines how many spectra differing only in count statistics (Poisson noise) will be created.

Creating images does not take that much longer but does provide an intuitive picture of the electron trajectories and absorption.







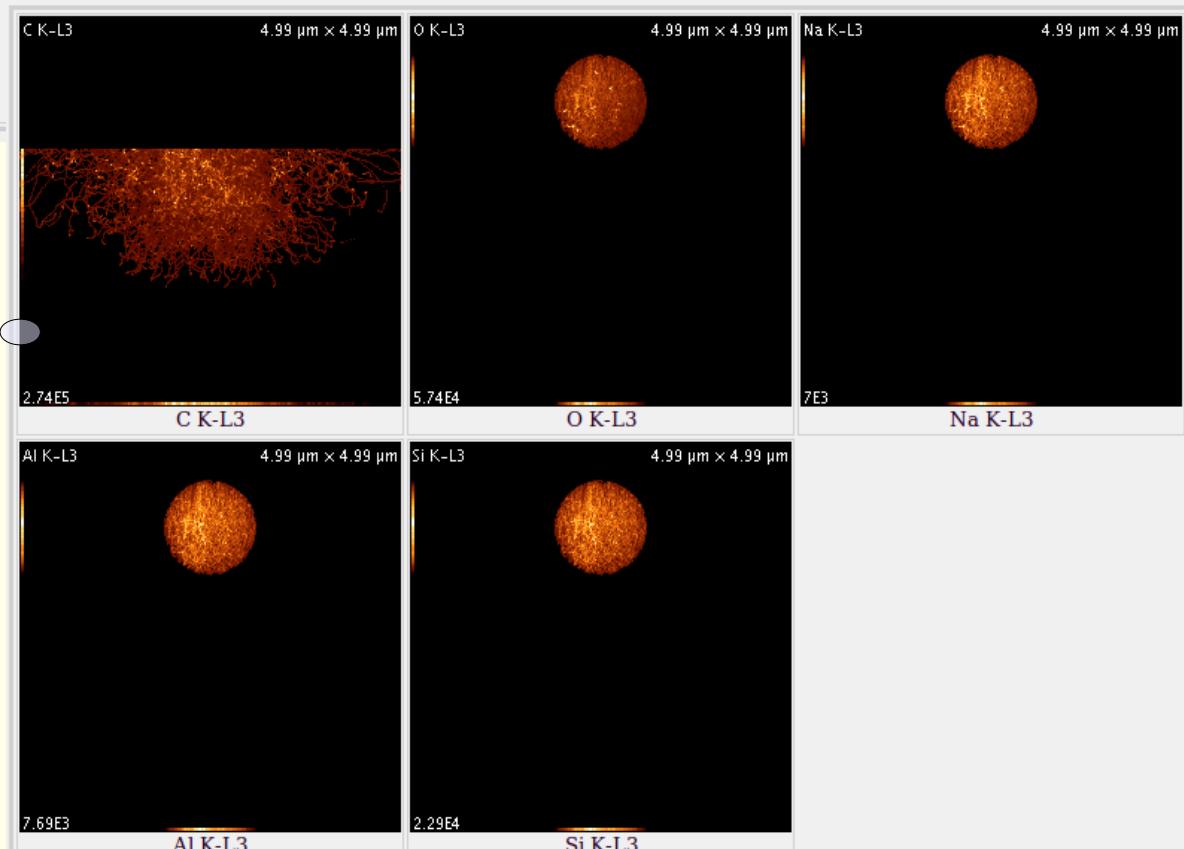


Spectrum Simulation

Simulation mode	Monte Carlo model of a spherical sample			
Substrate material	Carbon = [C(100.00 wgt%),3 g/cc]			
Object Material	Albite = [O(48.81 wgt%),Na(8.77 wgt%),Al(10.29 wgt%),Si(32.13 wgt%),2.6 g/cc]			
Sphere diameter	1.200 μm			
Beam energy	15.000 keV			
Probe dose	120.000 nA·s			
Instrument	JXA-8500F			
Detector	Bruker 5 eV/ch			
Calibration	FWHM[Mn K α]=124.7 eV - Sep 2, 2008 12:00:01 AM			
Overscan	true			
Replicas (with Poisson noise)	1			
Result 1	Noisy[MC simulation of a 1.200 μm diameter sphere of Albite on Carbon (overscan)] #1			
Trajectory view	/home/nicholas/DTSA-II Reports/2009/May/26-May-2009/vrm13916995151658125043.wrl			
Intensity data	Transition	Generated 1/msR	Emitted 1/msR	Ratio (%)
	C K-L3	177171.3	91265.3	51.5%
	O K-L3	33396.0	22585.6	67.6%
	Na K-L3	5738.9	4414.0	76.9%
	Al K-L3	6056.7	5319.8	87.8%
	Si K-L3	18095.6	16074.3	88.8%

The simulation configuration is summarized in a table for your records.

Emission Images

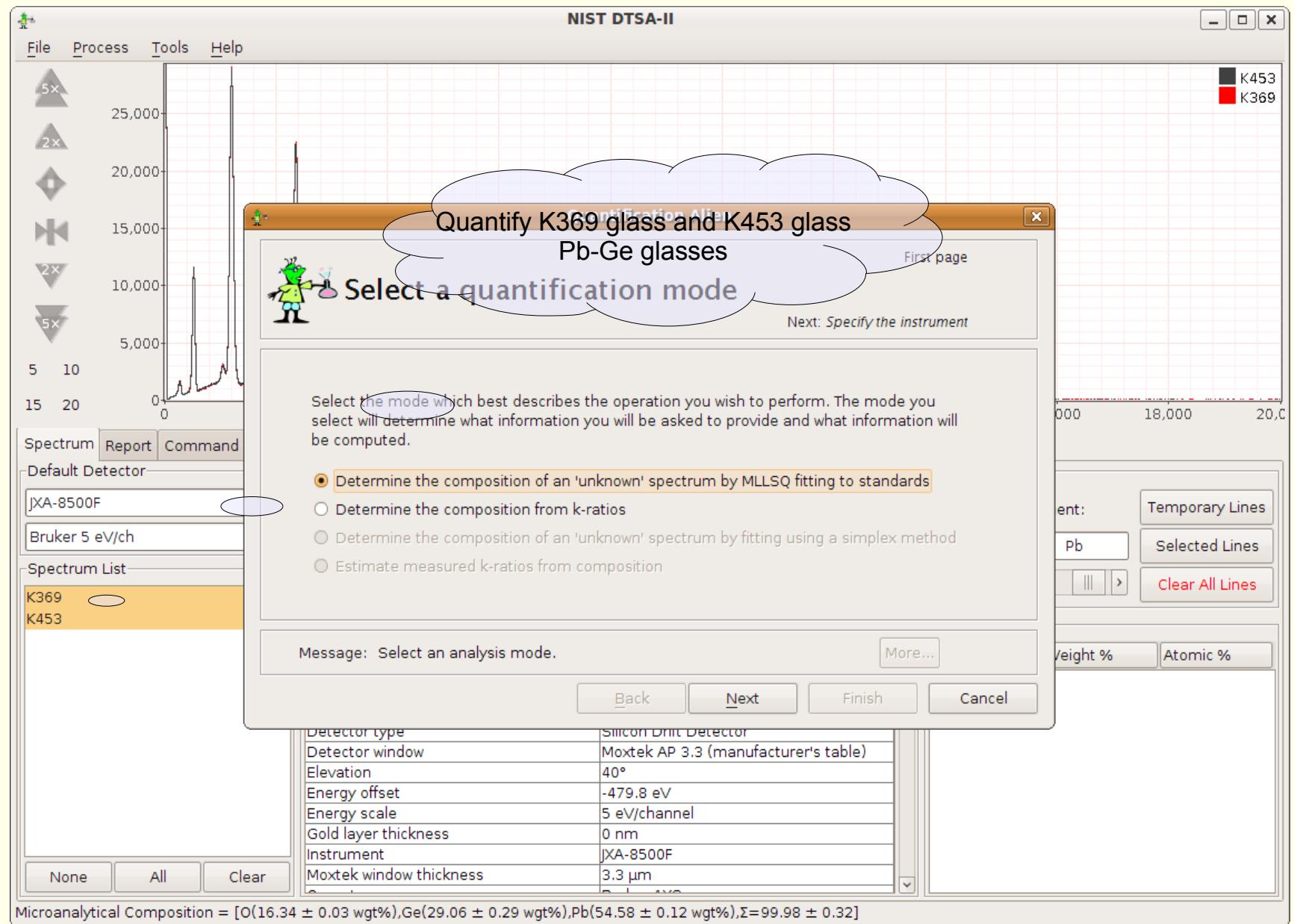


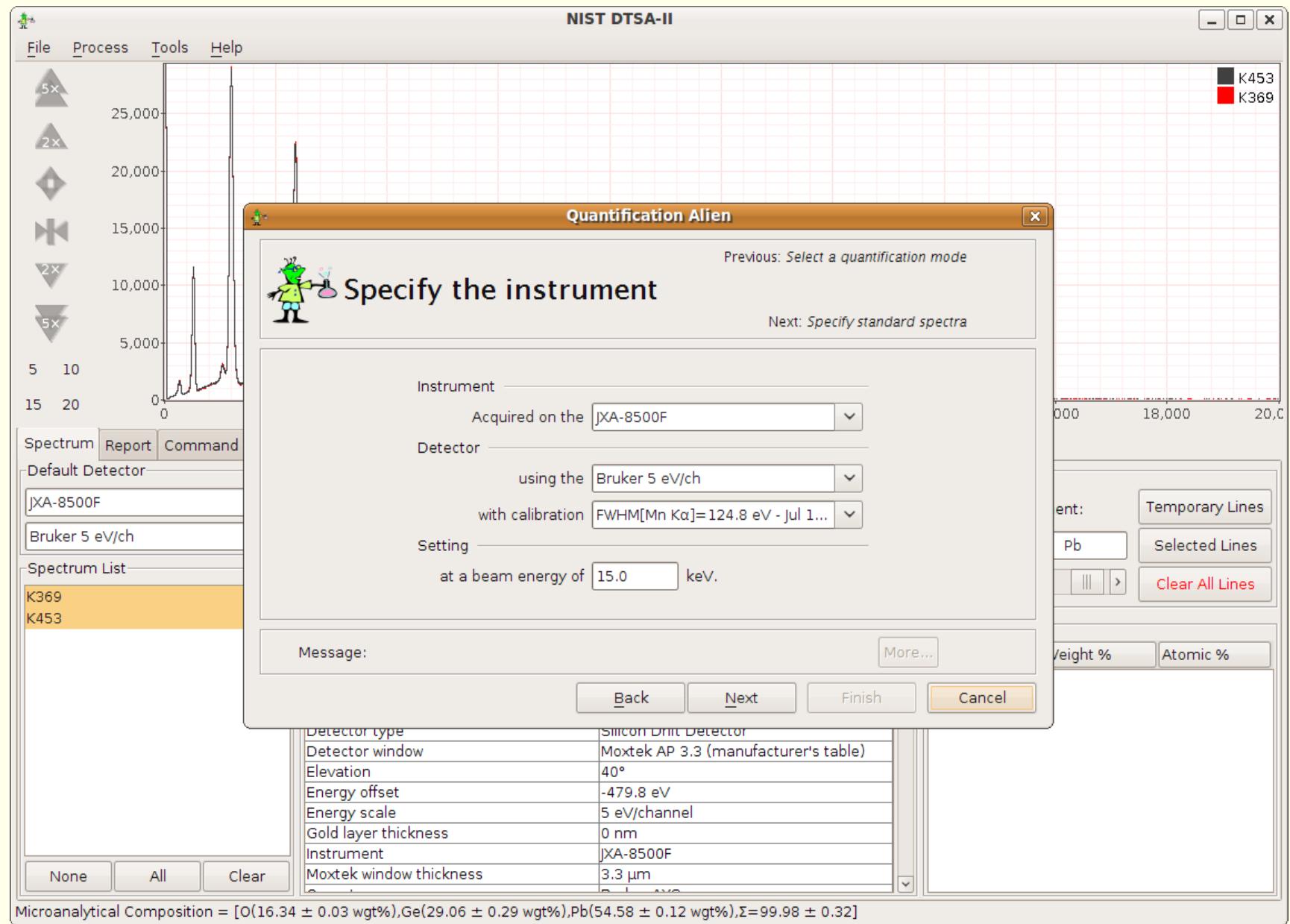
Images show where the emitted x-rays come from. In this case, the beam was rastered over the surface of the particle.

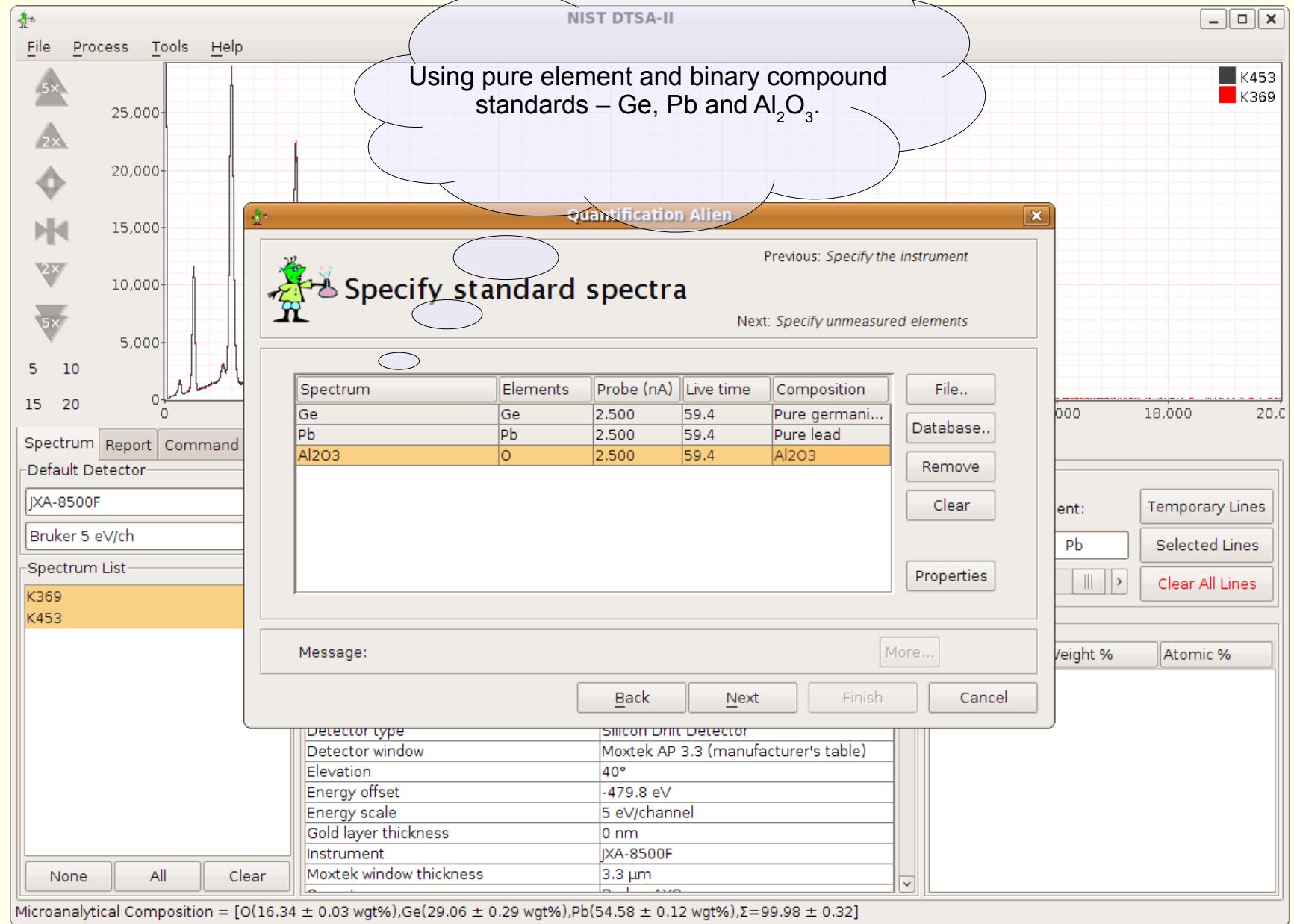


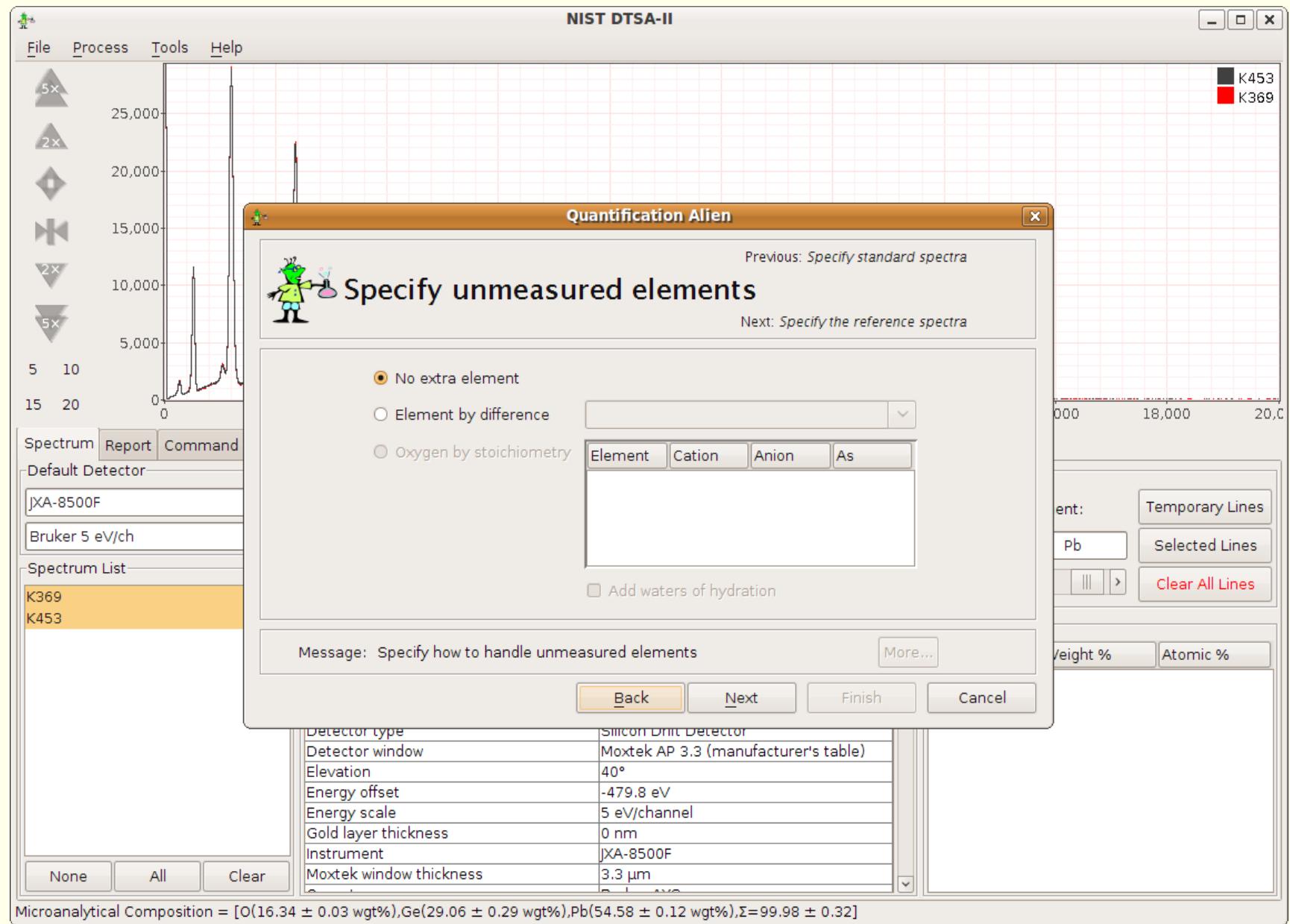
Example – Lead-Germanium glass

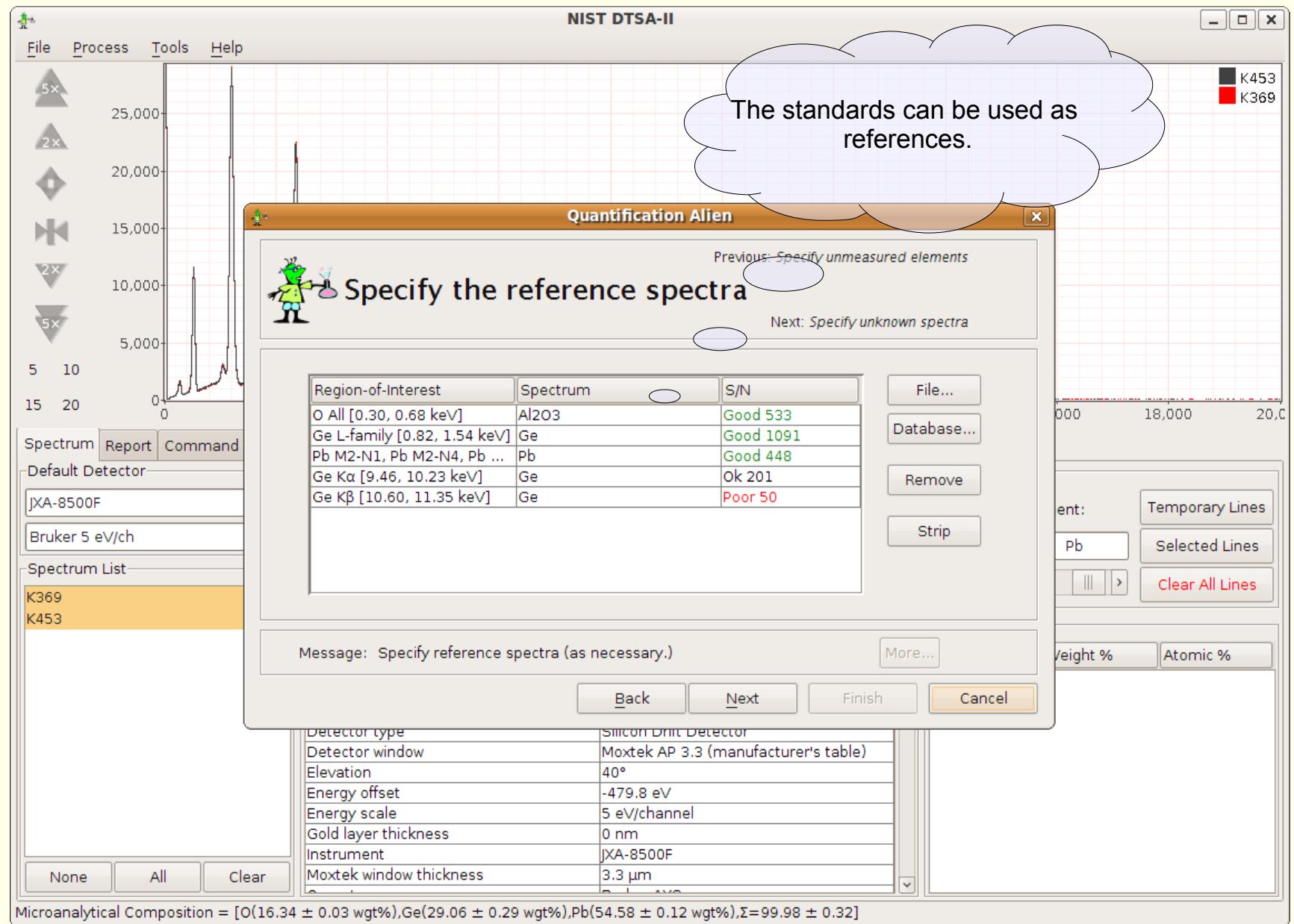
- Elemental standards
 - Pb, Ge, Al₂O₃
- Glass standards
 - K227
 - References for Pb, Si

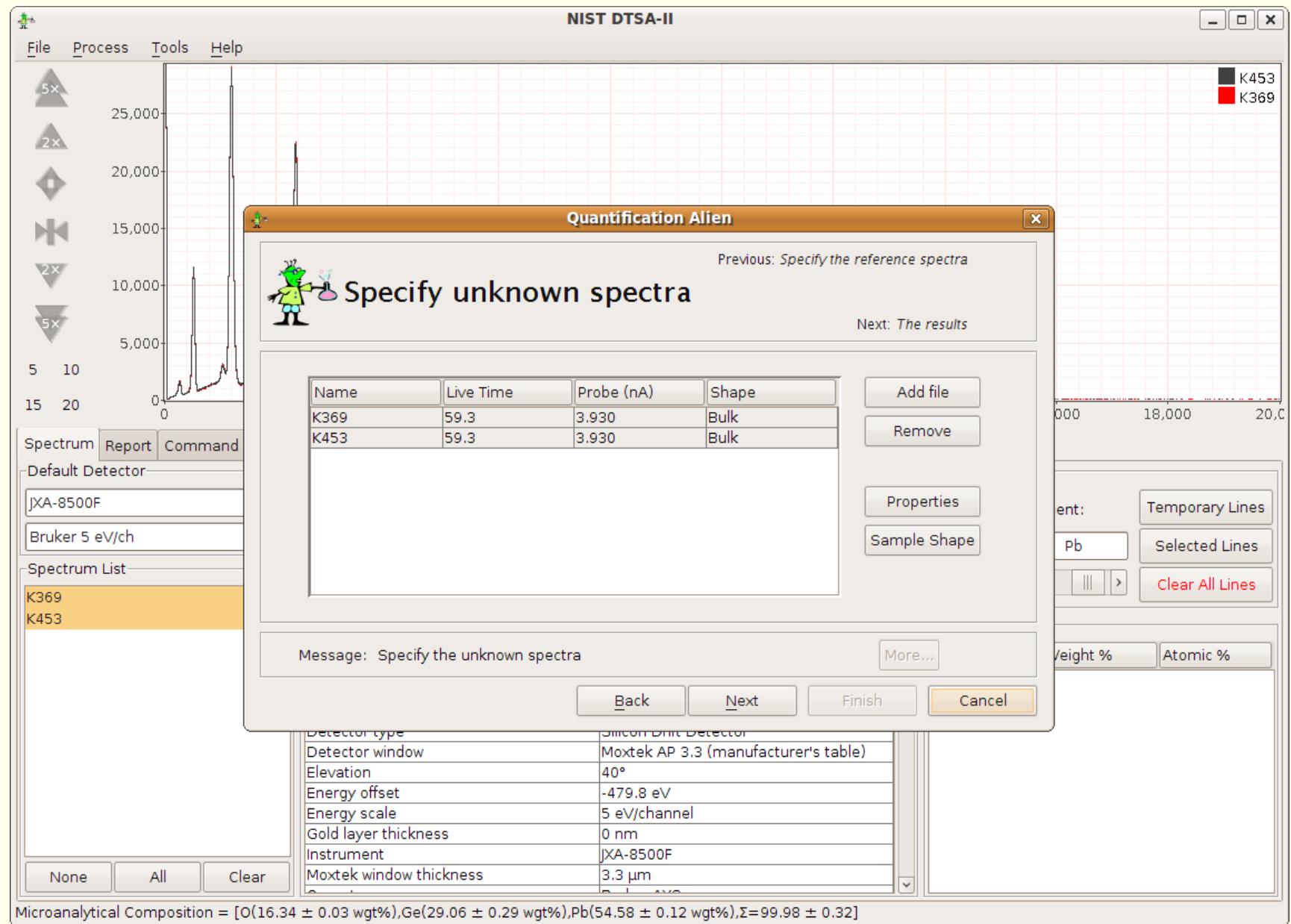


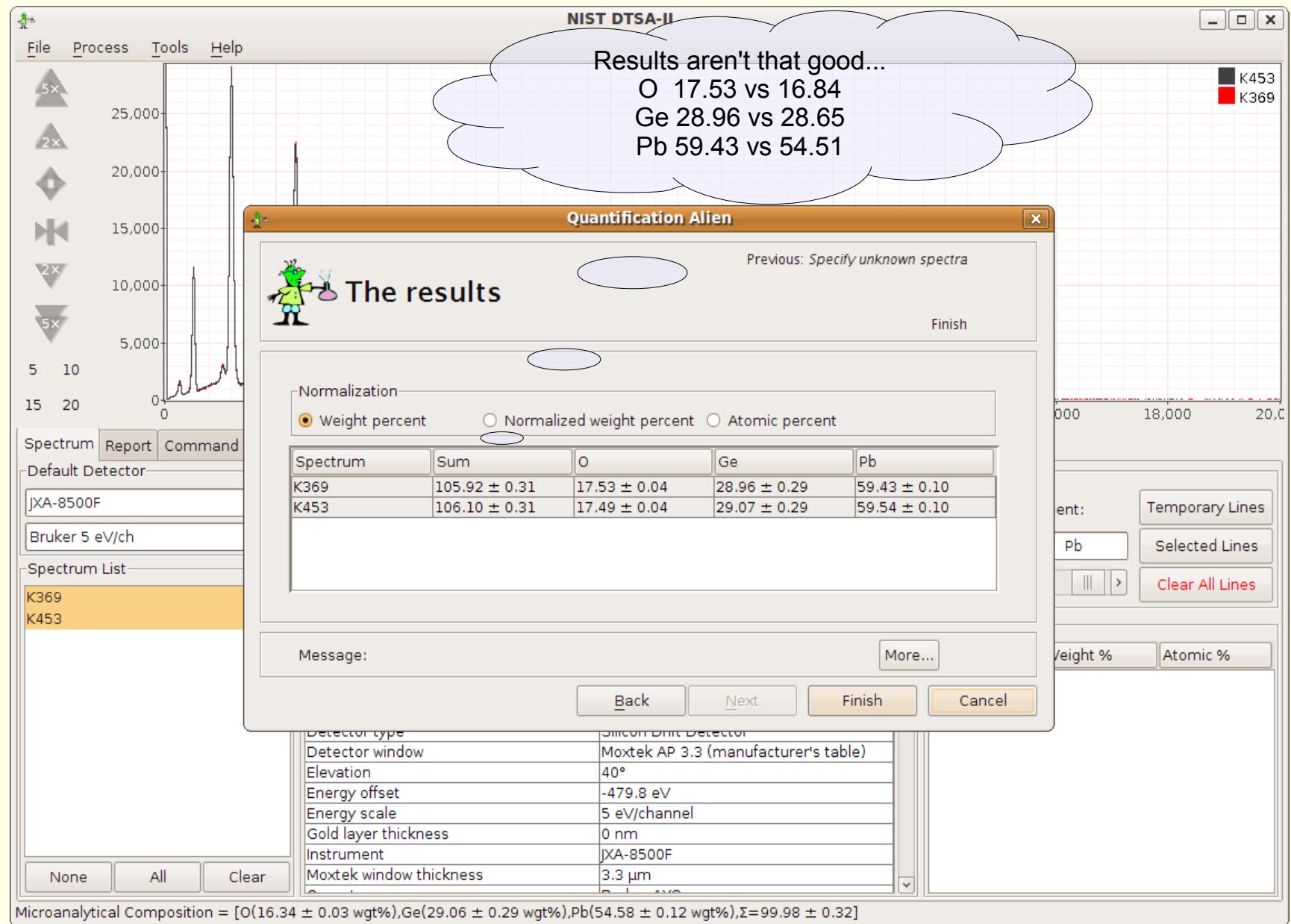


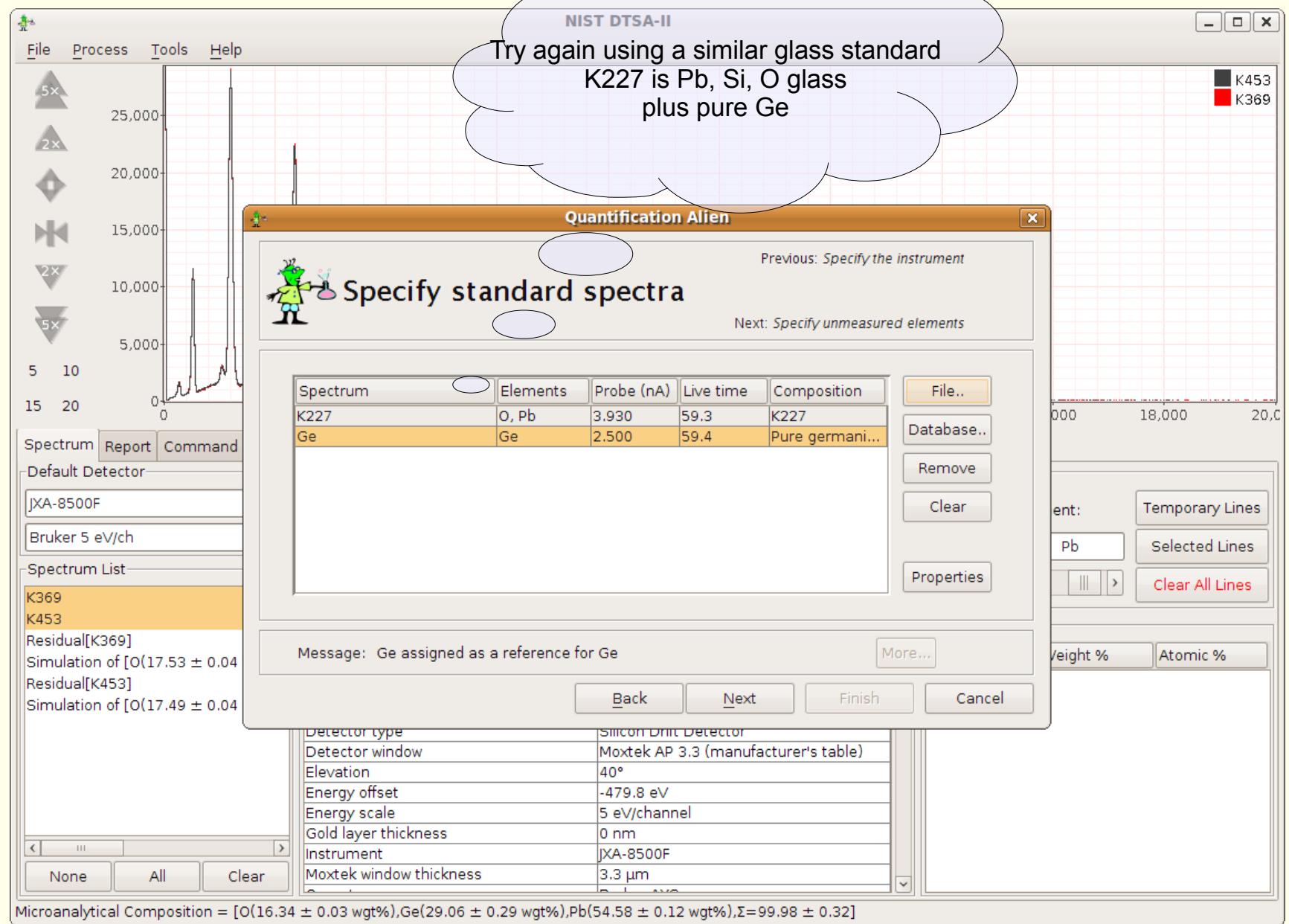


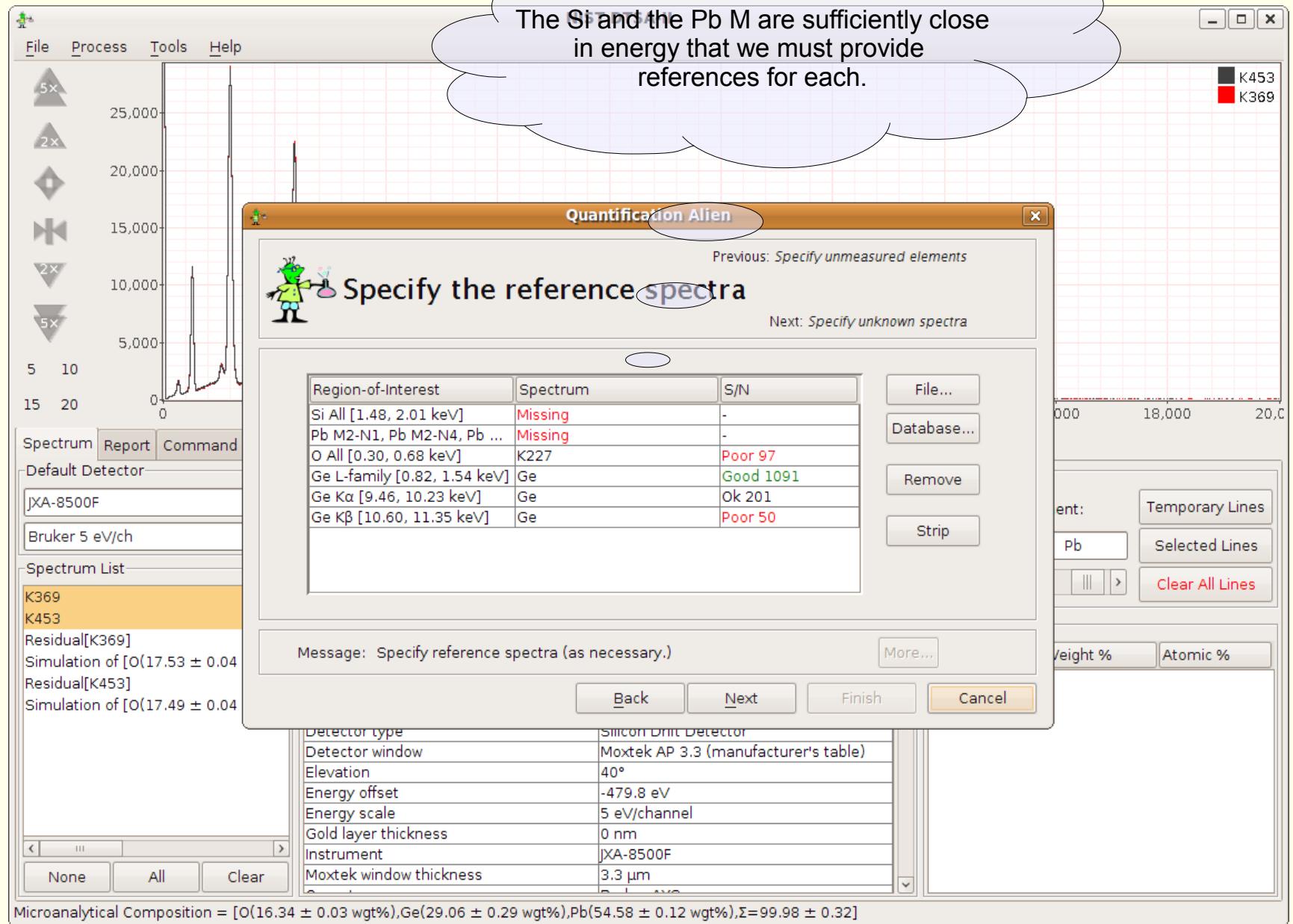


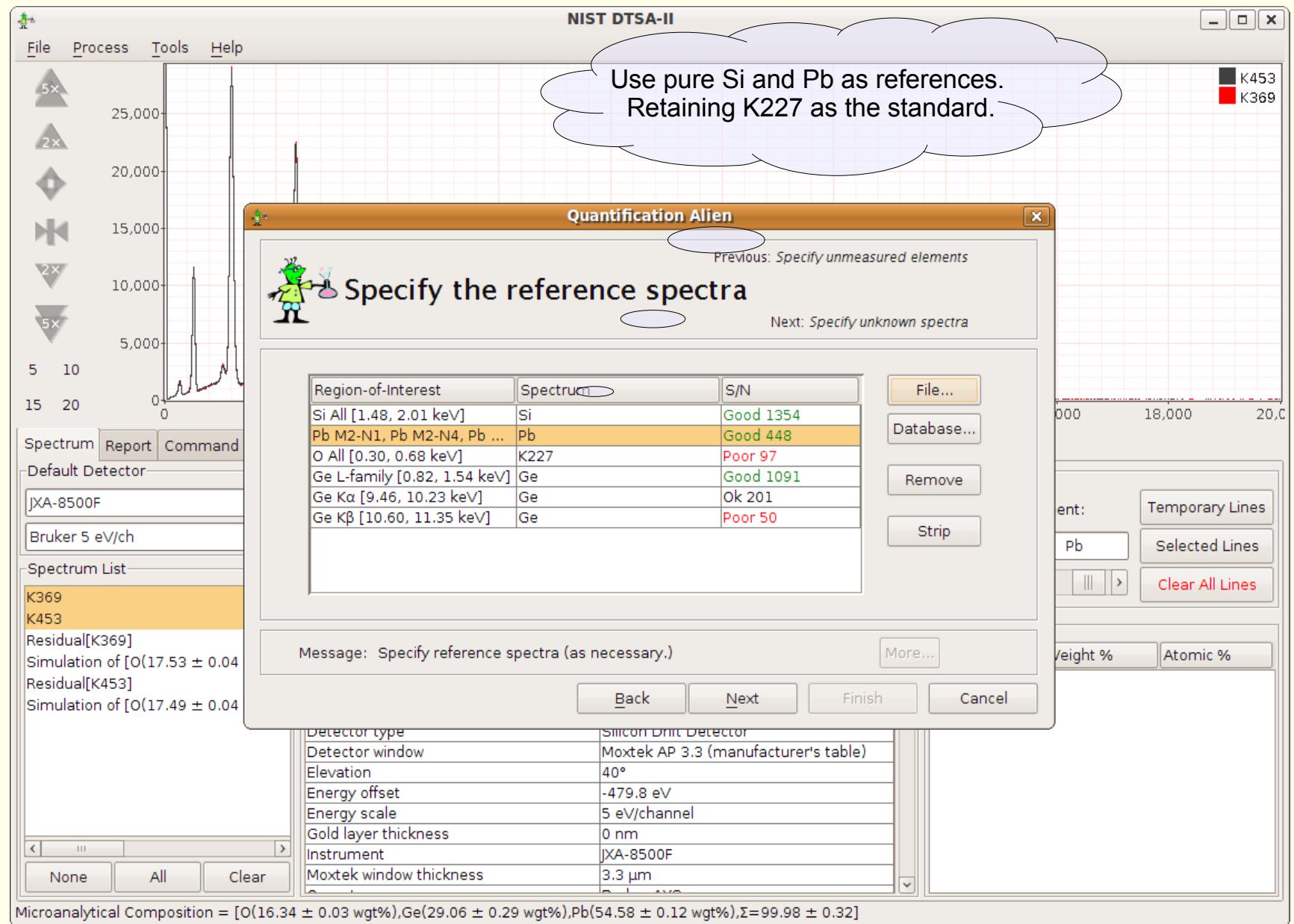


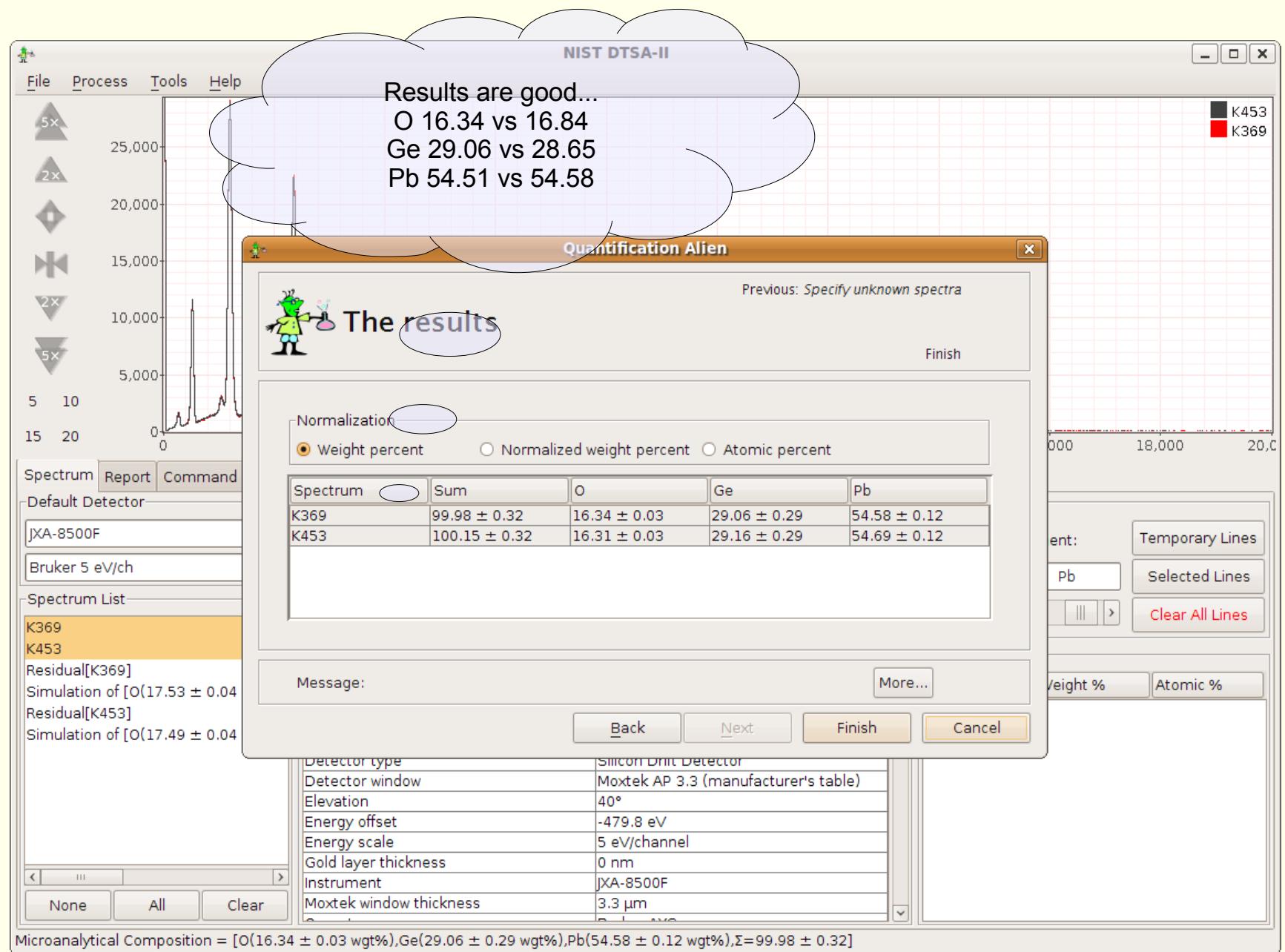












$$k_U = \frac{\beta_U C_U (Z \cdot A \cdot F)_U Q_U \omega_U P_U \varepsilon \frac{\Omega}{4\pi}}{\beta_{std} C_{std} (Z \cdot A \cdot F)_{std} Q_{std} \omega_{std} P_{std} \varepsilon \frac{\Omega}{4\pi}}$$

$$k_U = \frac{\beta_U C_U (Z \cdot A \cdot F)_U Q_U \omega_U P_U \varepsilon \frac{\Omega}{4\pi}}{\beta_{std} C_{std} (Z \cdot A \cdot F)_{std} Q_{std} \omega_{std} P_{std} \varepsilon \frac{\Omega}{4\pi}}$$

$$k_U = \frac{\beta_U C_U (Z \cdot A \cdot F)_U Q_U \omega_U P_U \varepsilon \frac{\Omega}{4\pi}}{\beta_{std} C_{std} (Z \cdot A \cdot F)_{std} Q_{std} \omega_{std} P_{std} \varepsilon \frac{\Omega}{4\pi}}$$

Standards-based

First principle
standard-less

Remote
Standards

EXAMPLE:

COMPARING QUANTIFICATION USING
SIMPLE COMPOUND STANDARDS
VS.
SIMILAR STANDARDS

FOR WHEN “GOOD ENOUGH” ISN’T...

REVIEW: UNCERTAINTY ANALYSIS

$$f = \frac{A}{B}$$

$$\left(\frac{\sigma_f}{f}\right)^2 = \left(\frac{\sigma_A}{A}\right)^2 + \left(\frac{\sigma_B}{B}\right)^2 - \frac{2\sigma_A\sigma_B}{AB} \rho_{AB}$$

$$A \pm \sigma_A$$

$$B \pm \sigma_B$$

ρ_{AB} is the correlation between A and B where $\rho_{AB} \in [0,1]$

$$k_U = \frac{\beta_U C_U (Z \cdot A \cdot F)_U Q_U \omega_U P_U \varepsilon \frac{\Omega}{4\pi}}{\beta_{std} C_{std} (Z \cdot A \cdot F)_{std} Q_{std} \omega_{std} P_{std} \varepsilon \frac{\Omega}{4\pi}}$$

First principle
standard-less

$$k_U = \frac{\beta_U C_U (Z \cdot A \cdot F)_U \cancel{Q_U} \omega_U P_U \varepsilon \frac{\Omega}{4\pi}}{\beta_{std} C_{std} (Z \cdot A \cdot F)_{std} \cancel{Q_{std}} \omega_{std} P_{std} \varepsilon \frac{\Omega}{4\pi}}$$

Remote standards

$$k_U = \frac{\beta_U C_U (Z \cdot A \cdot F)_U \cancel{Q_U} \omega_U \cancel{P_U} \varepsilon \frac{\Omega}{4\pi}}{\beta_{std} C_{std} (Z \cdot A \cdot F)_{std} \cancel{Q_{std}} \omega_{std} P_{std} \varepsilon \frac{\Omega}{4\pi}}$$

Standards-
based

$$k_U = \frac{\beta_U C_U (\cancel{Z \cdot A \cdot F})_U \cancel{Q_U} \omega_U P_U \varepsilon \frac{\Omega}{4\pi}}{\beta_{std} C_{std} (\cancel{Z \cdot A \cdot F})_{std} \cancel{Q_{std}} \omega_{std} P_{std} \varepsilon \frac{\Omega}{4\pi}}$$

Similar standards

K412 USING K411

K412 differs from K411 primarily in that K412 has Al and K411 doesn't.

Title:/home/nicholas/2010/SCSMM-2010.s

Creator:gnuplot 4.2 patchlevel 5

CreationDate:Tue Mar 9 10:29:49 2010

	Result	Certified	Difference
O	42.5	42.8	0.3
Mg	11.7	11.6	-0.1
Al	4.8	4.9	0.1
Si	21.2	21.2	0
Ca	10.9	10.9	0
Fe	7.8	7.7	-0.1

Using similar standards,
the accuracy can approach
the precision!

Quantification Alien

Previous: Specify unmeasured elements



Specify the reference spectra

Next: Specify lines to quantify.

Region-of-Interest	Spectrum	S/N
Fe L-family [0.40, 0.94 keV]	Missing	-
O All [0.30, 0.69 keV]	Al2O3 std	Good 481
Mg All [1.00, 1.45 keV]	K411 std	Good 277
Al All [1.23, 1.71 keV]	Al2O3 std	Good 938
Si All [1.47, 2.00 keV]	K411 std	Good 554
Ca K-family [3.38, 4.24 keV]	K411 std	Ok 246
Fe Ka [6.04, 6.70 keV]	K411 std	Ok 131
Fe Kβ [6.72, 7.34 keV]	K411 std	Poor 25

File...

Database...

Remove

Strip

Message: Specify reference spectra (as necessary.)

More...

Back

Next

Finish

Cancel



Quantification Alien

Previous: Specify unmeasured elements

Specify the reference spectra

Next: Specify lines to quantify.

Region-of-Interest	Spectrum	S/N
Fe L-family [0.40, 0.94 keV]	Fe std	Good 555
O All [0.30, 0.69 keV]	Al2O3 std	Good 481
Mg All [1.00, 1.45 keV]	K411 std	Good 277
Al All [1.23, 1.71 keV]	Al2O3 std	Good 938
Si All [1.47, 2.00 keV]	K411 std	Good 554
Ca K-family [3.38, 4.24 keV]	K411 std	Ok 246
Fe Ka [6.04, 6.70 keV]	K411 std	Ok 131
Fe Kβ [6.72, 7.34 keV]	K411 std	Poor 25

File...

Database...

Remove

Strip

Message: Specify reference spectra (as necessary.)

More...

Back

Next

Finish

Cancel



Quantification Alien

Previous: Specify the reference spectra

Next: Specify unknowns and properties

Element	Line Family
Oxygen	O All [0.30, 0.69 keV]
Magnesium	Mg All [1.00, 1.45 keV]
Aluminum	Al All [1.23, 1.71 keV]
Silicon	Si All [1.47, 2.00 keV]
Calcium	Ca K-family [3.38, 4.24 keV]
Iron	Auto



Message: Specify the line family used to quantify each element.

More...

Back

Next

Finish

Cancel

Quantification Alien

Previous: Specify lines to quantify.

Next: The results

Name	Live Time	Probe (nA)	Shape
K412 std	59.4	2.500	Bulk

Add file
Remove
Properties
Sample Shape

Message: Specify the unknown spectra

More...

Back

Next

Finish

Cancel

Title:/home/nicholas/Desktop/Fe std an
Creator:gnuplot 4.2 patchlevel 5
CreationDate:Sun Mar 7 17:34:16 2010

Title:/home/nicholas/Desktop/Si and br
Creator:gnuplot 4.2 patchlevel 5
CreationDate:Sun Mar 7 17:36:41 2010

Title:/home/nicholas/Desktop/CaF2 and
Creator:gnuplot 4.2 patchlevel 5
CreationDate:Sun Mar 7 17:37:26 2010

Title:/home/nicholas/Desktop/MgO and b

Creator:gnuplot 4.2 patchlevel 5
CreationDate:Sun Mar 7 17:37:06 2010

Title:/home/nicholas/Desktop/Al2O3 and
Creator:gnuplot 4.2 patchlevel 5
CreationDate:Sun Mar 7 17:36:14 2010

Conditions

Item	Value
Instrument	JXA-8500F
Detector	Bruker 5 eV
Beam Energy	15.0 keV
Correction Algorithm	XPP - Pouchou & Pichoir Simplified (Non-normal)
Mass Absorption Coefficient	NIST-Chantler 2005

Standards

Element	Material	Spectrum	Ref?	Probe (nA)	Live Time (s)
O	K411 = [O(42.86 wgt%),Mg(8.95 wgt%),Si(25.67 wgt%),Ca(11.18 wgt%),Fe(11.34 wgt%),5 g/cc]	K411 std	No	2.500	59.4
Mg	K411 = [O(42.86 wgt%),Mg(8.95 wgt%),Si(25.67 wgt%),Ca(11.18 wgt%),Fe(11.34 wgt%),5 g/cc]	K411 std	Yes	2.500	59.4
Al	Al2O3 = [O(47.07 wgt%),Al(52.93 wgt%),5 g/cc]	Al2O3 std	Yes	2.500	59.4
Si	K411 = [O(42.86 wgt%),Mg(8.95 wgt%),Si(25.67 wgt%),Ca(11.18 wgt%),Fe(11.34 wgt%),5 g/cc]	K411 std	Yes	2.500	59.4
Ca	K411 = [O(42.86 wgt%),Mg(8.95 wgt%),Si(25.67 wgt%),Ca(11.18 wgt%),Fe(11.34 wgt%),5 g/cc]	K411 std	Yes	2.500	59.4
Fe	K411 = [O(42.86 wgt%),Mg(8.95 wgt%),Si(25.67 wgt%),Ca(11.18 wgt%),Fe(11.34 wgt%),5 g/cc]	K411 std	Yes	2.500	59.4

References

Element/Lines	Spectrum
Fe L-family	/home/nicholas/Desktop/SCSMM-2010/K41x glasses/Fe std.msa

Preferred Characteristic Line Families

Element	Preferred ROI
Iron	Fe K α [6.04, 6.70 keV]

Results

Table: Quantitative results (uncertainties are statistical-only, 1 σ)

Spectrum	Quantity	O			Mg			Al			Si			Ca			Fe			Sum
K412 std	Line	O All			Mg All			Al All			Si All			Ca K-family			Fe K α			
	Z · A · F	1.00	1.01	1.00	1.00	1.04	1.00	1.01	0.80	1.01	1.00	0.96	1.00	1.00	1.00	1.00	0.99	1.00	1.00	
	k-ratios	1.0058	\pm	0.0020	1.3732	\pm	0.0026	0.0738	\pm	0.0003	0.7977	\pm	0.0011	0.9829	\pm	0.0029	0.6882	\pm	0.0056	
	weight %	42.50	\pm	0.08	11.70	\pm	0.02	4.79	\pm	0.02	21.24	\pm	0.03	10.93	\pm	0.03	7.75	\pm	0.06	98.91
	I = 2.500 nA norm(wgt %)	42.97	\pm	0.08	11.83	\pm	0.02	4.84	\pm	0.02	21.48	\pm	0.03	11.05	\pm	0.03	7.84	\pm	0.06	-
LT = 59.4 s atomic %		59.25			10.74			3.96			16.87			6.08			3.10			
Residual		/home/nicholas/DTSA-II Reports/2010/March/9-Mar-2010/residual6751533460246761921.msa																		

