



# 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 and later, some recent Linux and Unix distributions
  - Installers are available for Windows & OS X



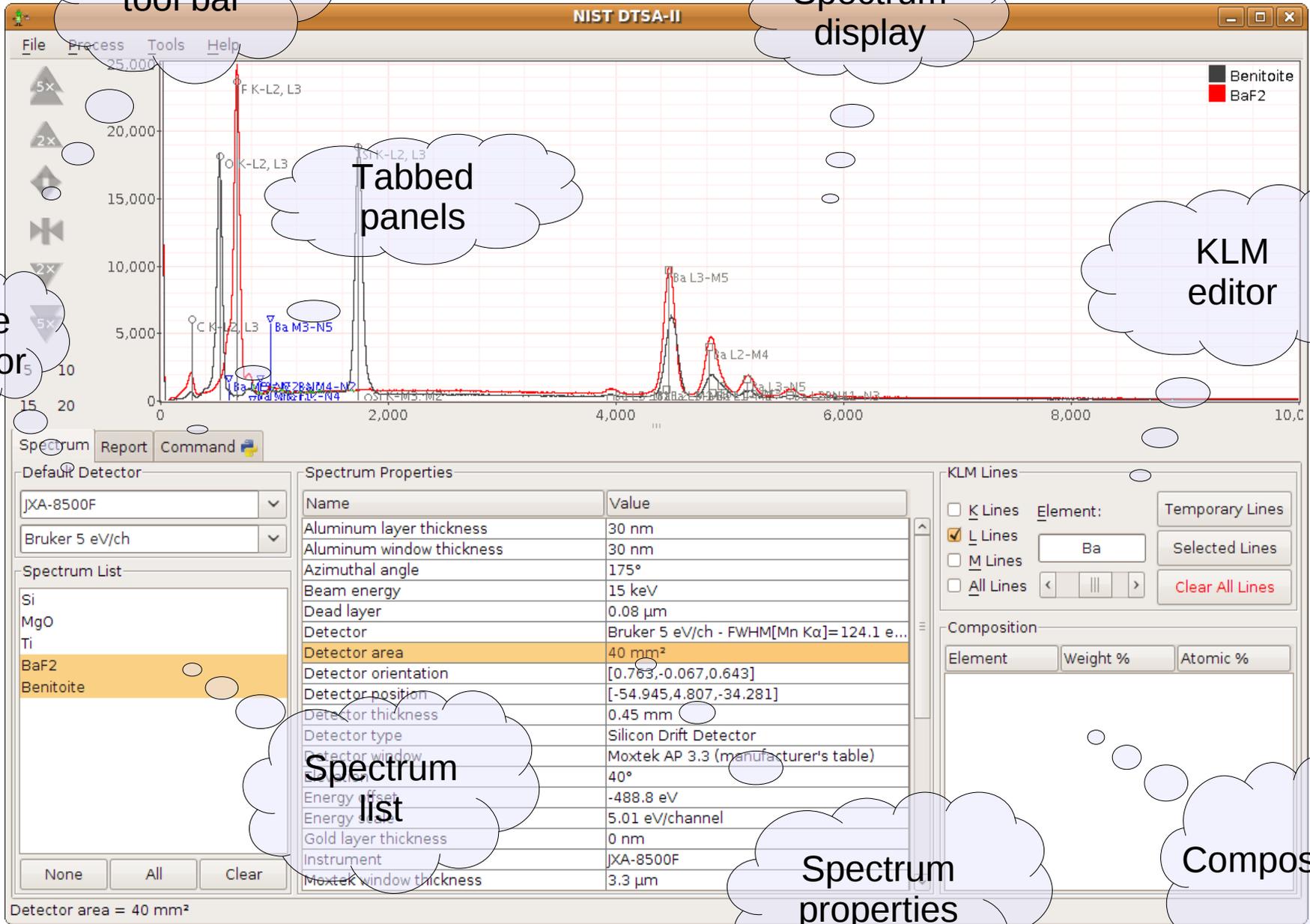
Spectrum tool bar

Spectrum display

Tabbed panels

KLM editor

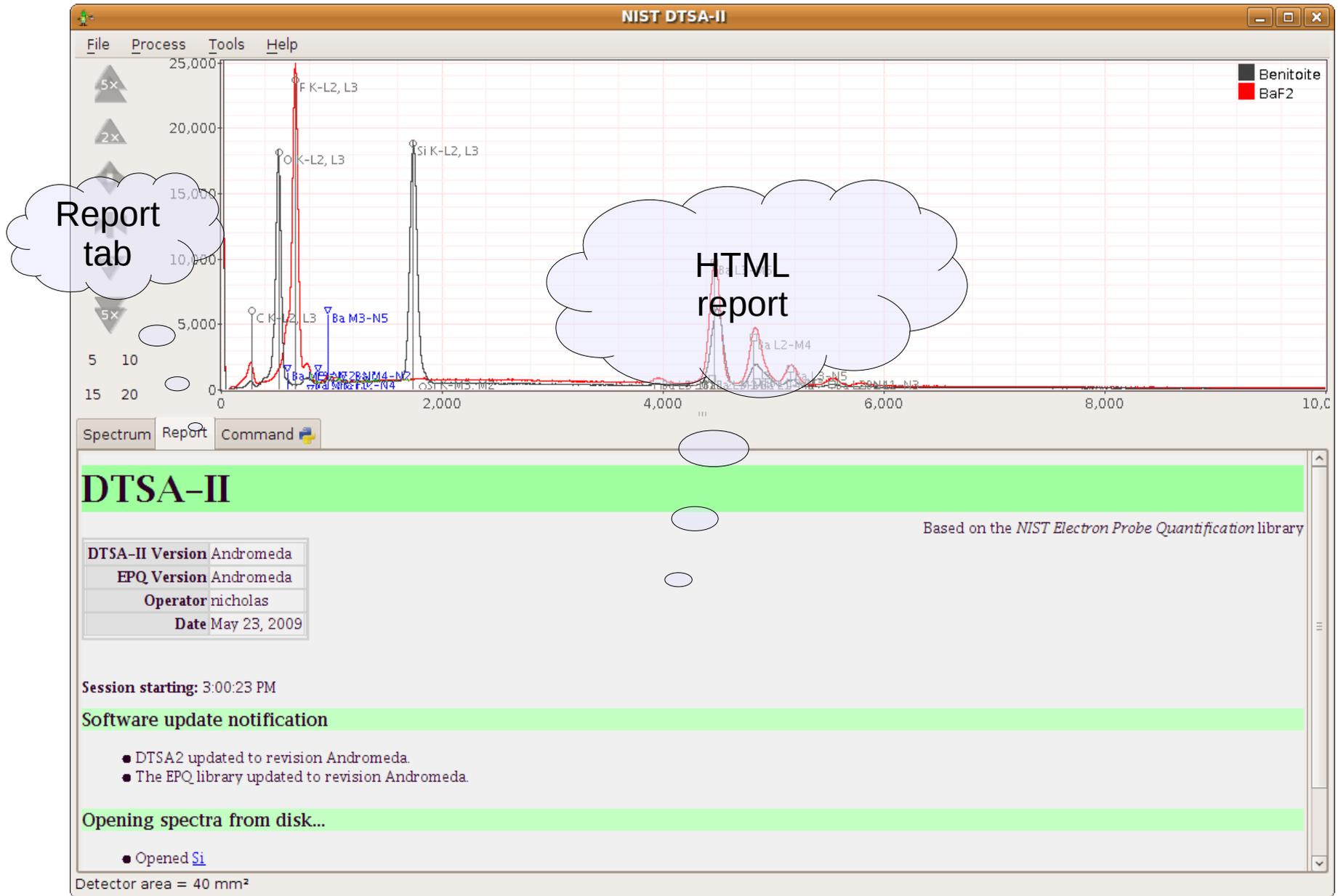
Active detector



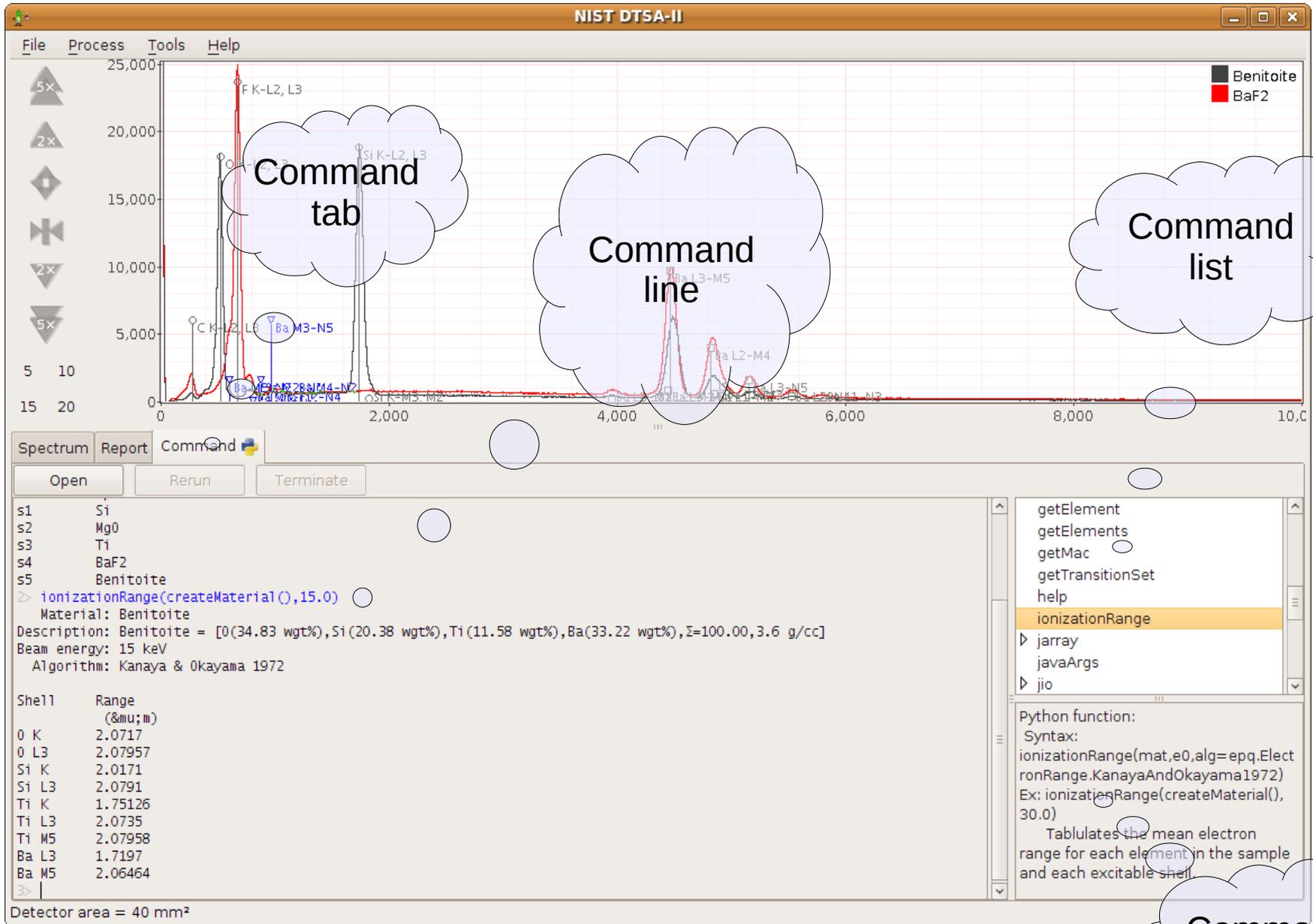
Spectrum list

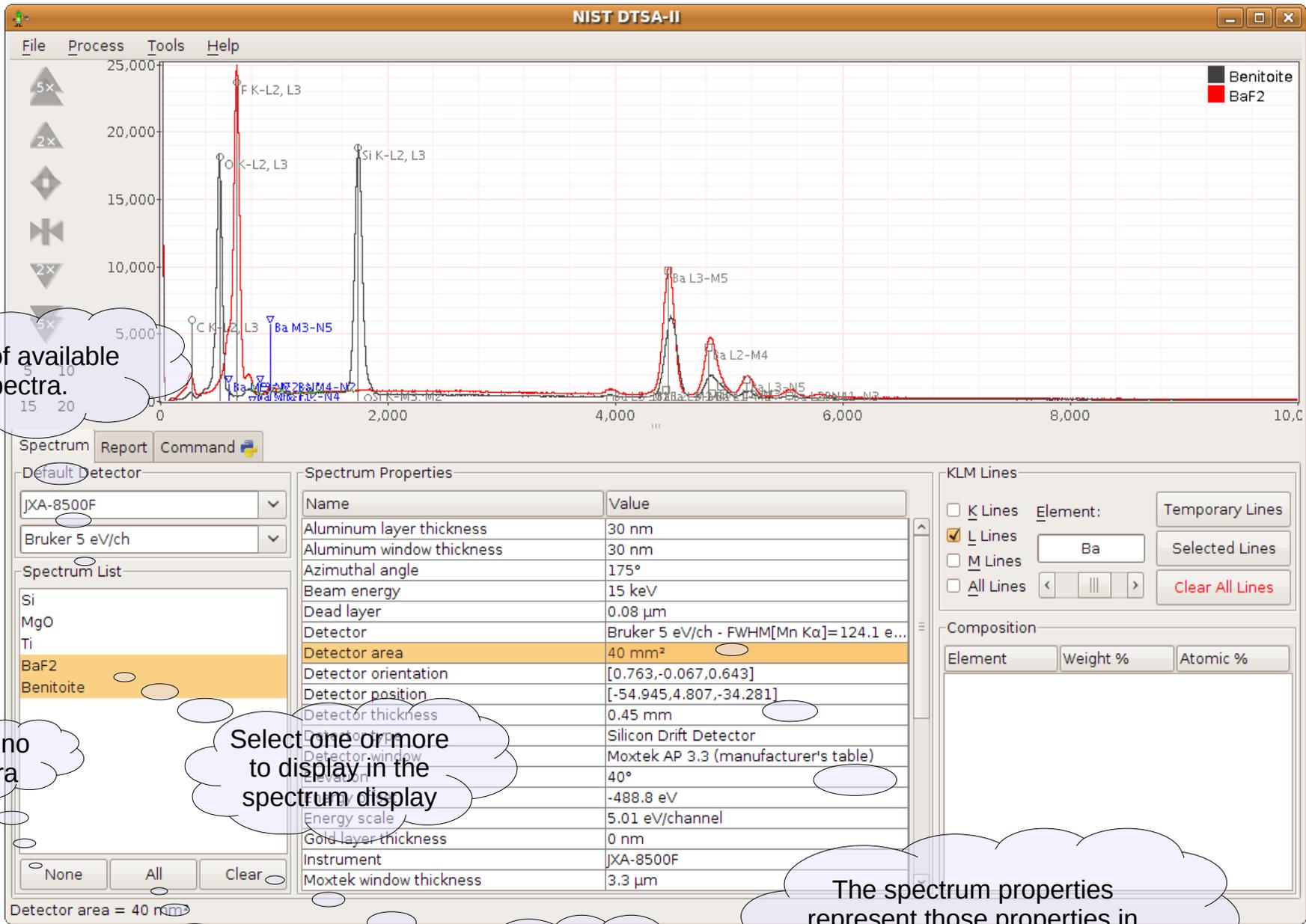
Spectrum properties

Composition



You can review old reports in a web browser...





A list of available spectra.

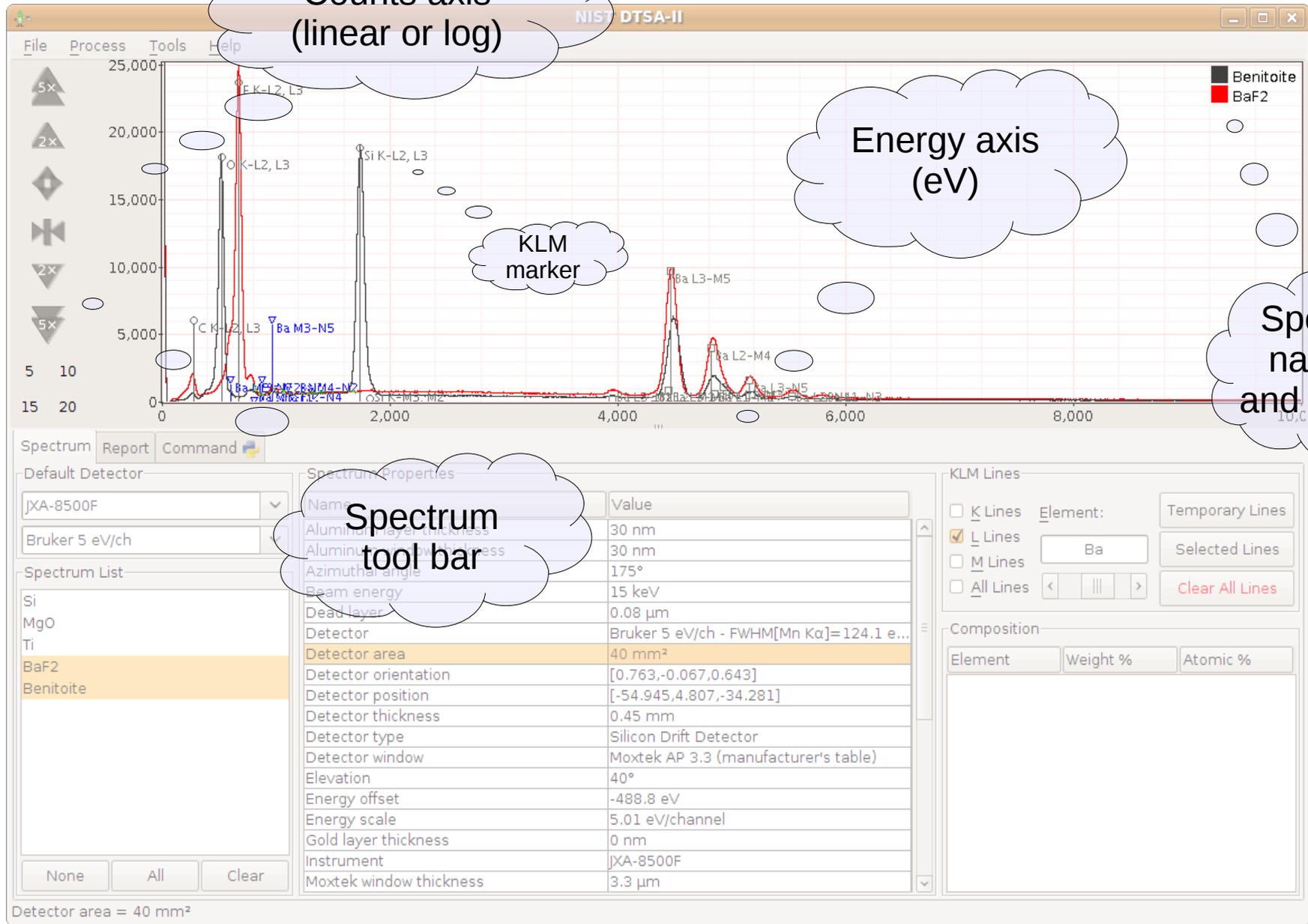
Select no spectra

Select one or more to display in the spectrum display

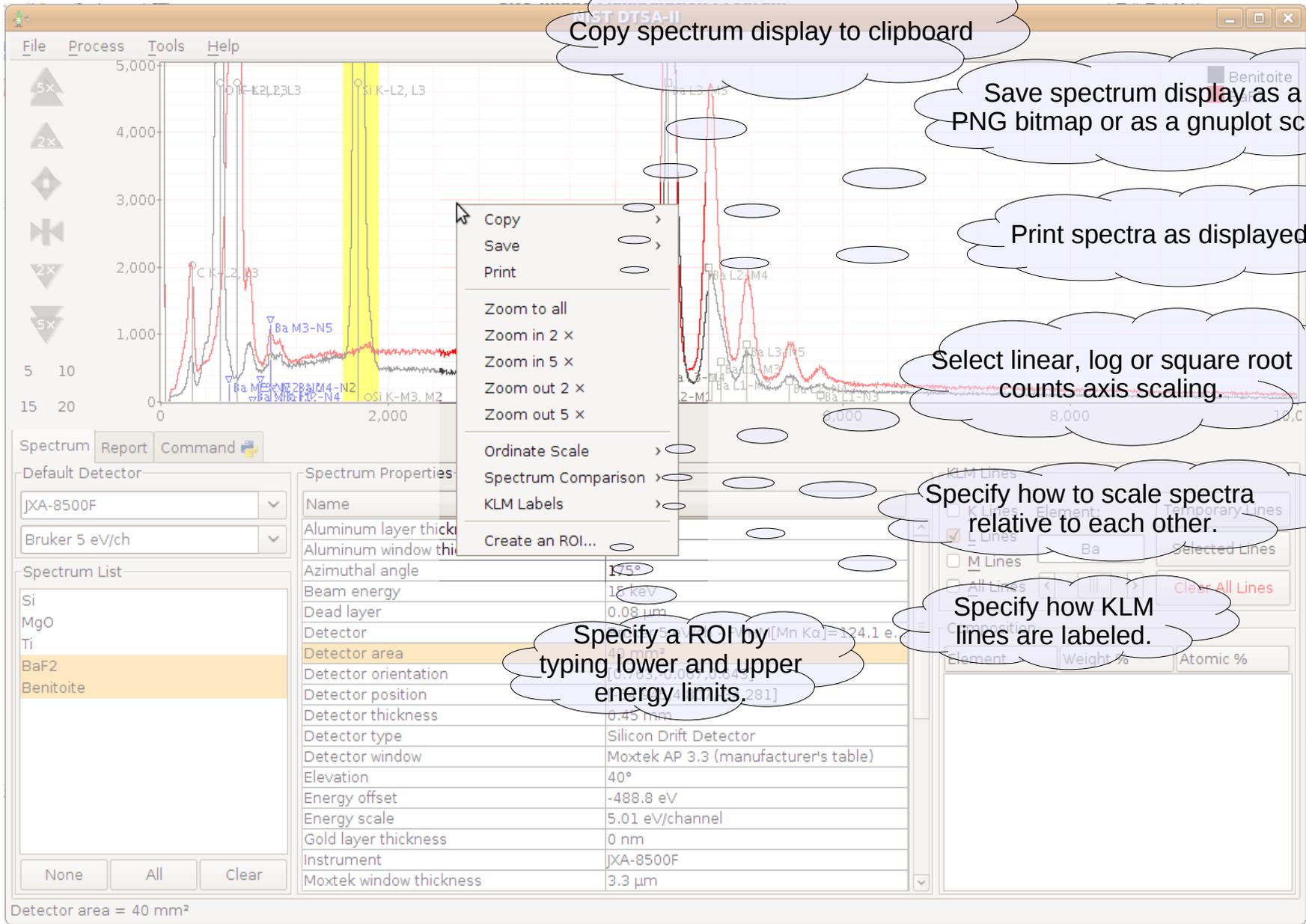
Select all spectra

Clear selected spectra from memory

The spectrum properties represent those properties in common to all selected spectra.



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



Copy spectrum display to clipboard

Save spectrum display as a PNG bitmap or as a gnuplot script

Print spectra as displayed

Select linear, log or square root counts axis scaling.

Specify how to scale spectra relative to each other.

Specify a ROI by typing lower and upper energy limits.

Specify how KLM lines are labeled.



Vertical zoom in by 5

Vertical zoom in by 2

Restore defaults

Zoom to ROI

Vertical zoom out by 2

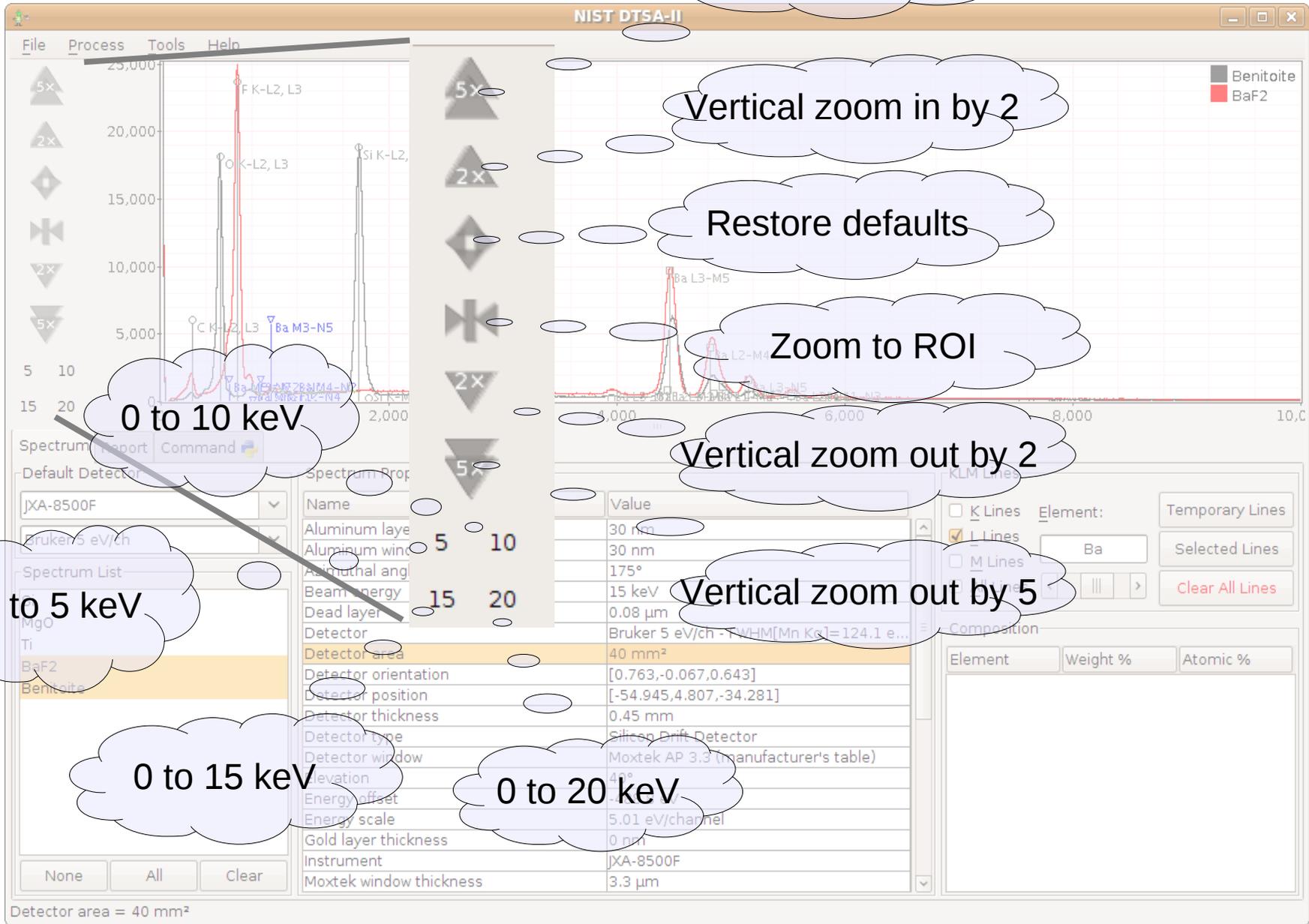
Vertical zoom out by 5

0 to 10 keV

0 to 5 keV

0 to 15 keV

0 to 20 keV





Specify the default instrument to associate with spectra.

The screenshot shows the NISX D software interface. At the top, a menu bar includes File, Process, Tools, and Help. Below the menu is a spectrum plot with a y-axis from 0 to 25,000 and an x-axis from 0 to 10,000. The plot shows several peaks labeled with element lines: F K-L2, L3; O K-L2, L3; Si K-L2, L3; Ba M3-N5; and Ba M4-N7. A legend in the top right corner identifies Benitoite (grey) and BaF2 (red). A 'Default Detector' dialog box is overlaid on the plot, showing 'JXA-8500F' in the instrument dropdown and 'Bruker 5 eV/ch' in the energy scale dropdown. Below the plot are three panels: 'Default Detector' (repeating the dropdowns), 'Spectrum Properties' (a table of parameters), and 'Spectrum List' (a list of elements). The 'Spectrum Properties' table is as follows:

Name	Value
Aluminum layer thickness	30 nm
Aluminum window thickness	30 nm
Azimuthal angle	175°
Beam energy	15 keV
Dead layer	0.08 μm
Detector	Bruker 5 eV/ch - FWHM[Mn Ka] = 124.1 e...
Detector area	40 mm <sup>2</sup>
Detector orientation	[0.763,-0.067,0.643]
Detector position	[-54.945,4.807,-34.281]
Detector thickness	0.45 mm
Detector type	Silicon Drift Detector
Detector window	Moxtek AP 3.3 (manufacturer's table)
Elevation	40°
Energy offset	-488.8 eV
Energy scale	5.01 eV/channel
Gold layer thickness	0 nm
Instrument	JXA-8500F
Moxtek window thickness	3.3 μm

The 'Spectrum List' panel shows a list of elements: Si, MgO, Ti, BaF2, and Benitoite. The 'Default Detector' panel shows 'JXA-8500F' and 'Bruker 5 eV/ch'. The 'Spectrum Properties' panel shows various parameters including 'Detector area = 40 mm<sup>2</sup>'.

Specify the default detector to associate with spectra.

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



ROI properties

Sum counts in the ROI

Click-and-drag to select a region-of-interest (ROI)



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



**NIST DTSA-II**

File Process Tools Help

5,000  
4,000  
3,000  
2,000  
1,000  
0

5x  
2x  
5x

5 10  
15 20

0 2000 4000 6000 8000 10,000

Benitoite  
BaF2

Zoom to full spectrum

Eliminate all defined ROIs.

Perform a background corrected peak integration on all defined ROIs

Zoom the energy axis to display the energies covered by the ROIs

Copy the spectrum data in the ROIs to the clipboard as text.

Clear regions  
Count events  
Zoom to region  
Zoom to all  
Copy Region(s) To Clipboard

Spectrum Report Command

Default Detector: JXA-8500F  
Bruker 5

Spectrum List:  
Si  
MgO  
Ti  
BaF2  
Benitoite

Spectrum Properties

Name	Value
Aluminum layer thickness	30 nm
Aluminum window thickness	30 nm
Azimuth angle	175°
Beam energy	15 keV
Dead layer	0.08 μm
Detector	Bruker 5 eV/ch - FWHM[Mn Kα]=124.1 e...
Detector area	40 mm <sup>2</sup>
Detector orientation	[0.763,-0.067,0.643]
Detector position	[-54.945,4.807,-34.281]
Detector thickness	0.45 mm
Detector type	Silicon Drift Detector
Detector window	Moxtek AP 3.3 (manufacturer's table)
Elevation	40°
Energy offset	-488.8 eV
Energy scale	5.01 eV/channel
Gold layer thickness	0 nm
Instrument	JXA-8500F
Moxtek window thickness	3.3 μm

Composition

Element	Weight %	Atomic %
---------	----------	----------

Detector area = 40 mm<sup>2</sup>



Open a spectrum from a disk file

Reopen a report from disk

Save the selected spectra to EMSA or text format

Edit application preferences including detectors

The screenshot shows the NIST DTSA-II software interface. The main window displays a spectrum plot with two traces: a black trace for Benitoite and a red trace for BaF2. The x-axis represents energy in eV, ranging from 0 to 10,000. The y-axis represents intensity. Several peaks are labeled with their corresponding element and shell, such as Si K-L2, L3, Ba M3-N5, and Ba L2, L3, M4. A yellow vertical bar highlights a region around 2,000 eV. The File menu is open, showing options like Open, Open Report, Save As, Bulk rename, Import from CSV, Batch export, Import into database, Search database, Print, Preferences, and Exit. The Spectrum Properties panel is also visible, showing various parameters such as Name, Aluminum layer thickness, Aluminum window thickness, Azimuthal angle, Beam energy, Dead layer, Detector, Detector area, Detector orientation, Detector position, Detector thickness, Detector type, Detector window, Elevation, Energy offset, Energy scale, Gold layer thickness, Instrument, and Moxtek window thickness. The KLM Lines panel shows checkboxes for K, L, M, and All Lines, with L Lines selected. The Composition panel shows a table for Element, Weight %, and Atomic %.

Name	Value
Aluminum layer thickness	30 nm
Aluminum window thickness	30 nm
Azimuthal angle	175°
Beam energy	15 keV
Dead layer	0.08 μm
Detector	Bruker 5 eV/ch - FWHM[Mn Kα]= 124.1 e...
Detector area	40 mm <sup>2</sup>
Detector orientation	[0.763,-0.067,0.643]
Detector position	[-54.945,4.807,-34.281]
Detector thickness	0.45 mm
Detector type	Silicon Drift Detector
Detector window	Moxtek AP 3.3 (manufacturer's table)
Elevation	40°
Energy offset	-488.8 eV
Energy scale	5.01 eV/channel
Gold layer thickness	0 nm
Instrument	JXA-8500F
Moxtek window thickness	3.3 μm

Default Detector: JXA-8500F  
Bruker 5 eV/ch

Spectrum List:  
Si  
MgO  
Ti  
BaF2  
Benitoite

KLM Lines:  
 K Lines  
 L Lines  
 M Lines  
 All Lines

Element: Ba  
Temporary Lines  
Selected Lines  
Clear All Lines

Composition:  
Element Weight % Atomic %

Detector area = 40 mm<sup>2</sup>

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



\* Not all files of all types are supported

The screenshot displays the NIST DTSA-II software interface. An 'Open spectrum files...' dialog box is open, showing a file list with 'Benitoite.msa' selected. The main window shows a spectrum plot with a legend for 'Benitoite' and 'BaF2'. The plot shows several peaks, with the highest peaks around 1,000 and 2,000 eV. The x-axis is labeled from 0 to 20,000 eV, and the y-axis is labeled from 0 to 15,000. The 'Spectrum preview window' is highlighted with a blue cloud callout.

Spectrum preview window

File open dialog



Fit the Bremsstrahlung background

Smooth the spectrum data (don't do this...)

Identify ranges of channels with peaks

**Process** menu options:  
Sub-sample spectrum  
Fit background  
Linearize energy axis  
Smooth (Savitzky-Golay)  
Trim  
Peak search

**Spectrum Properties**

Name	Value
Aluminum layer thickness	30 nm
Aluminum window thickness	30 nm
Azimuthal angle	175°
Beam energy	15 keV
Dead layer	0.08 μm
Detector	Bruker 5 eV/ch - FWHM[Mn Kα]= 124.1 e...
Detector area	40 mm <sup>2</sup>
Detector orientation	[0.763,-0.067,0.643]
Detector position	[-54.945,4.807,-34.281]
Detector thickness	0.45 mm
Detector type	Silicon Drift Detector
Detector window	Moxtek AP 3.3 (manufacturer's table)
Elevation	40°
Energy offset	-488.8 eV
Energy scale	5.01 eV/channel
Gold layer thickness	0 nm
Instrument	JXA-8500F
Moxtek window thickness	3.3 μm

**KLM Lines**

K Lines    Element: Ba    Temporary Lines  
 L Lines    Selected Lines  
 M Lines  
 All Lines    Clear All Lines

**Composition**

Element	Weight %	Atomic %
---------	----------	----------

Detector area = 40 mm<sup>2</sup>



Edit the properties of the selected spectra

Specify the composition of the material from which the selected spectra were collected.

Quantify the selected spectra

Simulate a spectrum

Calibrate a detector

Add a text note to the end of the current report

NIST DTSA-II

File Process Tools Help

- Edit spectrum properties
- Assign material
- Quantification alien
- Simulation alien
- Calibration alien
- Report note

5x, 2x, 5x zoom controls

Command

Spectrum Properties

Name	Value
Aluminum layer thickness	30 nm
Aluminum window thickness	30 nm
Azimuthal angle	175°
Beam energy	15 keV
Dead layer	0.08 μm
Detector	Bruker 5 eV/ch - FWHM[Mn Kα]= 124.1 e...
Detector area	40 mm <sup>2</sup>
Detector orientation	[0.763,-0.067,0.643]
Detector position	[-54.945,4.807,-34.281]
Detector thickness	0.45 mm
Detector type	Silicon Drift Detector
Detector window	Moxtek AP 3.3 (manufacturer's table)
Elevation	40°
Energy offset	-488.8 eV
Energy scale	5.01 eV/channel
Gold layer thickness	0 nm
Instrument	JXA-8500F
Moxtek window thickness	3.3 μm

KLM Lines

K Lines    Element: Ba    Temporary Lines

L Lines    Selected Lines

M Lines    Clear All Lines

All Lines

Composition

Element	Weight %	Atomic %

Detector area = 40 mm<sup>2</sup>





**NIST DTSA-II**

File Process Tools Help

5x 2x 5x 2x

3,000 2,000 1,000

5 10 15 20

0 2,000

0 8,000 10,000

Benitoite

**Assign material**

Material

Name

Density  g/cm<sup>3</sup> (optional)

Mode

Weight Fractions  Atomic Proportions

Your elements will appear here

Element:  Quantity:

**Spectrum Properties**

Acquisition time	
Aluminum layer thickness	
Aluminum window thickness	
Azimuthal angle	
Beam energy	
Dead layer	
Detector	
Detector area	
Detector orientation	
Detector position	[-54.945,4.807,-34.281]
Detector thickness	0.45 mm
Detector type	Silicon Drift Detector
Detector window	Moxtek AP 3.3 (manufacturer's table)
Display name	Benitoite
Durometer	4.92 keV
Elevation	40°
Energy offset	-488.8 eV
Energy scale	5.01 eV/channel

**Spectrum List**

Si	
MgO	
Ti	
Ba	
Benitoite	

None All Clear

Detector area = 40 mm<sup>2</sup>

**KLM Lines**

K Lines  L Lines  M Lines  All Lines

Temporary Lines

Ba Selected Lines

**Composition**

Element	Weight %	Atomic %
---------	----------	----------

Enter element names or abbreviations here

Specify a name for the material

Search the database or parse a chemical formula

Specify whether quantities are in weight fraction or atomic fraction

Specify a density in grams per cubic centimeter

Elements and amounts will display here

Enter amounts of elements here

Clear the element list

Add an element and quantity

Delete the elements selected in the list



### Mode 1: Parse a chemical formula

Material

Name BaTiSi3O9

Density 5 g/cm<sup>3</sup> (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%

Add Delete Clear

Ok Cancel

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

The search button.

### Mode 2: Search the material database

Material

Name Benitoite

Density 3.6 g/cm<sup>3</sup> (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%

Add Delete Clear

Ok Cancel

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

### Mode 3: Manual entry

Material

Name Benitoite

Density 3.6 g/cm<sup>3</sup> (optional)

Mode

Weight Fractions  Atomic Proportions

Ba 33.21% by weight

Element: Ti Quantity: 11.58

Add Delete Clear

Ok Cancel

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.
  - ✓ Otherwise the standard can act as a reference



**NIST DTSA-II**

File Process **Tools** Help

- Edit spectrum properties
- Assign material
- Quantification alien**
- Simulation alien
- Calibration alien
- Report note

**Step 2: From the Tools menu, select the "Quantification Alien"**

**Step 1: Highlight the spectrum or spectra you want to quantify**

5x  
2x  
15  
10,000  
5,000  
5 10  
15 20  
0 2,000 4,000 6,000 8,000 10,000 12,000 14,000 16,000 18,000 20,000

Spectrum Report Command

Default Detector: JXA-8500F  
Bruker 5 eV/ch

Spectrum List: Benitoite

None All Clear

**Spectrum Properties**

Name	Value
Acquisition time	7/29/08 11:14 AM
Aluminum layer thickness	30 nm
Aluminum window thickness	30 nm
Azimuthal angle	175°
Beam energy	15 keV
Dead layer	0.08 μm
Detector	Bruker 5 eV/ch - FWHM[Mn Kα]=124.1 ...
Detector area	40 mm <sup>2</sup>
Detector orientation	[0.763,-0.067,0.643]
Detector position	[-54.945,4.807,-34.281]
Detector thickness	0.45 mm
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

**KLM Lines**

K Lines Element: Temporary Lines  
 L Lines H Selected Lines  
 M Lines  
 All Lines < ||| > Clear All Lines

**Microanalytical Composition**

Element	Weight %	Atomic %
Oxygen	35.26 ± 0.06	64.82 ± 0.16
Barium	34.40 ± 0.14	7.37 ± 0.03
Silicon	19.72 ± 0.03	20.65 ± 0.05
Titanium	11.66 ± 0.07	7.16 ± 0.04

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**NIST DTSA-II**

File Process Tools Help

5x 2x 5x 2x 5x

15 20

Spectrum Report Command

Default Detector

JXA-8500F

Bruker 5 eV/channel

Spectrum List

Benitoite

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

**Quantification Alien**

First page

### Select a quantification mode

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. 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	7.16 ± 0.04

ent: Temporary Lines

H Selected Lines

Clear All Lines

Composition

000 18,000 20,000

Benitoite

**Step 3:** Select "Determine the composition of an 'unknown' spectrum by MLLSQ fitting to standards"

Click 'Next' to proceed to the next step.



**NIST DTSA-II**

File Process Tools Help

5x 2x 10x 5x

15 20

Spectrum Report Command

Default Detector

JXA-8500F

Bruker 5 eV/ch

Spectrum List

Benitoite

None All Clear

15,000

10,000

5,000

0

0 18,000 20,000

Benitoite

**Quantification Alien**

Specify the instrument

Previous: **calibration** are read from the spectrum you selected in step 0.

Next: Specify standard spectra

Instrument

Acquired on the JXA-8500F

Detector

using the Bruker 5 eV/ch

with calibration FWHM[Mn K $\alpha$ ]=124.1 eV - Jul 2...

Setting

at a beam energy of 15.0 keV.

Message:

More...

Back Next Finish Cancel

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	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

Welcome to NIST DTSA-II - Andromeda A revision

The beam energy is read from the spectrum you selected in step 0.

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



**NIST DTSA-II**

File Process Tools Help

5x  
2x  
5x  
5x  
5x

15 10  
15 20

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

None All Clear

15,000  
10,000  
5,000  
0

Benitoite

**Quantification Alien**

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

**Specify standard spectra**

Spectrum Elements Probe (nA) Live time Composition

File.. Database.. Remove Clear Properties

Message: Specify standard spectra and the associated elements and compo... More...

Back Next Finish Cancel

Weight %	Atomic %
5.26 ± 0.06	64.82 ± 0.16
3.40 ± 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 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

Welcome to NIST DTSA-II - Andromeda A revision

Spectrum from a disk file

Spectrum from a database (Advanced)

Remove selected standards

Clear all standards

Edit the spectrum properties of the selected standard spectra.



**NIST DTSA-II**

File Process Tools Help

### Open a spectrum

New Folder Delete File Rename File

/home/nicholas/Spectra/JXA-8500F/GMIIIA 15 keV/Extra stds

Select one or more of the standard spectra.

**Folders**

- /
- ./

**Files**

- NaCl.msa
- NaF.msa
- Pb.msa
- Si.msa
- Ti.msa
- Zn.msa

Selection: /home/nicholas/Spectra/JXA-8500F/GMIIIA 15 keV/Extra stds

BaF2.msa

Filter: All Files

Legend:

- BaF2
- MgO
- Si
- Ti

Atomic %

4.82 ± 0.16
.37 ± 0.03
0.65 ± 0.05
.16 ± 0.04

OK Cancel

None All Clear Gold layer thickness 0 nm

Welcome to NIST DTSA-II - Andromeda A revision



**NIST DTSA-II**

File Process Tools Help

5x  
2x  
5x

15,000  
10,000  
5,000  
0

5 10  
15 20

Spectrum Report Command

Default Detector

JXA-8500F

Bruker 5 eV/ch

Spectrum List

Benitoite

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

**Quantification Alien**

Previous: Specify the instrument

**Specify standard spectra**

Select the element(s) for which Si is a reference.

Silicon - Selected

Elements

File.. Database.. Remove Clear Properties

Message: Specify standard spectra and the associated elements and composition... More...

Back Next Finish Cancel

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: 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

Benitoite

Temporary Lines  
Selected Lines  
Clear All Lines

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

0 18,000 20,000

The file name of the spectrum file.

The element or elements with which to associate this spectrum.



**NIST DTSA-II**

File Process Tools Help

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

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

**Quantification Alien**

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

**Specify standard 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	BaSO4

File.. Database.. Remove Clear Properties

Message: More...

Back Next Finish Cancel

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 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

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

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**

File Process Tools Help

5x 2x 10x 5x

15 20

Spectrum Report Command

Default Detector: JXA-8500F

Detector: Bruker 5 eV/ch

Spectrum List: Benitoite

None All Clear

15,000 10,000 5,000 0

0 18,000 20,000

Benitoite

**Quantification Alien**

Previous: Specify the instrument

**Specify standard spectra**

Next: Specify unmeasured elements

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	BaSO4

File... Database... Remove Clear Properties More...

Message:

Back Next Finish Cancel

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
11.66 ± 0.07	7.18 ± 0.04

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

Welcome to NIST DTSA-II - Andromeda A revision

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.



NIST DTSA-II

File Process Tools Help

5x  
2x  
5x

15 20

Spectrum Report Command

Default Detector

JXA-8500F

Bruker 5 eV/ch

Spectrum List

Benitoite

None All Clear

15,000  
10,000  
5,000  
0

0 18,000 20,000

Benitoite

Quantification Alien

Previous: Specify starting material

Next: Specify the reference spectra

### Specify unmeasured elements

No extra element

Element by difference

Oxygen by stoichiometry

Element Cation Anion As

Add waters of hydration

Message: Specify how to handle unmeasured elements

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	7.16 ± 0.04

Welcome to NIST DTSA-II - Andromeda A revision

No extra element means that we've provided standards for all elements in the material.

Element by difference means assume all missing mass is from the specified element.

Assume that oxygen is associated with the measured elements according to the specified cation/anion ratios.



NIST DTSA-II

File Process Tools Help

■ Benitoite

Quantification Alien

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

### Specify the reference 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

Message: Specify reference spectra (as necessary)

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: 488.7 m

Energy offset: 5.01 eV/channel

Energy scale: 5.01 eV/channel

Gold layer thickness: 0.1 μm

ent: Temporary Lines

H Selected Lines

Clear All Lines

Composition

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

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

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.

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



**NIST DTSA-II**

File Process Tools Help

5x 2x 5x 15 20

Spectrum Report Command

Default Detector: JXA-8500F, Bruker 5 eV/ch

Spectrum List: Benitoite

Quantification Alien

Previous: Specify the reference spectra

Next: The results

**Specify unknown spectra**

Name	Live Time	Probe (nA)	Shape
Benitoite	59.4	2.500	Bulk

Buttons: Add file, Remove, Properties, Sample Shape

Message: Specify the unknown spectra

Buttons: Back, Next, Finish, Cancel, More...

Detector type: Silicon Drift Detector

Detector window: 14.00 keV

Manufacturer: 9.9 (manufacturer's table)

Energy offset: -478.8 eV

Energy scale: 5.01 eV/channel

Gold layer thickness: 0 nm

Buttons: None, All, Clear

Buttons: Temporary Lines, Selected Lines, Clear All Lines

Weight %	Atomic %
5.26 ± 0.06	84.82 ± 0.16
4.40 ± 0.14	7.37 ± 0.03
9.72 ± 0.03	20.65 ± 0.05

Welcome to NIST DTSA-II - Andromeda A revision

This is the spectrum you selected in step 0.

You may specify additional spectrum to quantify here.

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

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



**NIST DTSA-II**

File Process Tools Help

5x 2x 10x 5x

15 20

Spectrum Report Command

Default Detector

JXA-8500F

Bruker 5 eV/ch

Spectrum List

Benitoite

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

Quantification Alert

Previous: Specify unknown spectra

### The results

Finish

Normalization

Weight percent  Normalized weight percent  Atomic percent

Spectrum	Sum	O	Si	Ti	Ba
Benitoite	101.04 ± 0.17	35.26 ± 0.06	19.72 ± 0.03	11.66 ± 0.07	34.40 ± 0.14

Message: More...

Back Next Finish Cancel

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
11.66 ± 0.07	7.16 ± 0.04

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

ent: Temporary Lines  
H Selected Lines  
Clear All Lines

Composition

Results are tabulated as they are computed and can be displayed in various different forms.

Select Finish to view the tabulated results.



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)]	<a href="#">MgO</a>	Yes	2.500	59.3
Si	Pure silicon = [Si(100.00 wgt%),2.4 g/cc]	<a href="#">Si</a>	Yes	2.500	59.4
Ti	Pure titanium = [Ti(100.00 wgt%),4.5 g/cc]	<a href="#">Ti</a>	Yes	2.500	59.4
Ba	BaF2 = [F(0.67 atoms),Ba(0.33 atoms)]	<a href="#">BaF2</a>	Yes	2.500	59.4

#### Results

Spectrum	Quantity	O			Si			Ti			Ba			Sum
	Line	O All			Si All			Ti K-family			Ba L-family			
<a href="#">Benitoite</a>	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	
<b>Bulk</b>	k-ratios	0.6392 ±	0.0010	0.1547 ±	0.0002	0.1090 ±	0.0006	0.3673 ±	0.0015					
	weight %	<b>35.26</b> ±	0.06	<b>19.72</b> ±	0.03	<b>11.66</b> ±	0.07	<b>34.40</b> ±	0.14	101.04				
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	<a href="#">/home/nicholas/DTSA-II Reports/2009/May/24-May-2009/residual2361900809694010741.msa</a>													

**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

### 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)]	<a href="#">MgO</a>	Yes	2.500	59.3
Si	Pure silicon = [Si(100.00 wgt%),2.4 g/cc]	<a href="#">Si</a>	Yes	2.500	59.4
Ti	Pure titanium = [Ti(100.00 wgt%),4.5 g/cc]	<a href="#">Ti</a>	Yes	2.500	59.4
Ba	BaF2 = [F(0.67 atoms),Ba(0.33 atoms)]	<a href="#">BaF2</a>	Yes	2.500	59.4

#### Results

**Table:**Quantitative results (uncertainties are statistical-only, 1  $\sigma$ )

Spectrum	Quantity	O			Si			Ti			Ba			Sum
<b>Benitoite Bulk</b>	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	±	0.0010	0.1547	±	0.0002	0.1090	±	0.0006	0.3673	±	0.0015	
	weight %	<b>35.26</b>	±	0.06	<b>19.72</b>	±	0.03	<b>11.66</b>	±	0.07	<b>34.40</b>	±	0.14	101.04
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		<a href="#">/home/nicholas/DTSA-II Reports/2009/May/24-May-2009/residual2361900809694010741.msa</a>												



## Results

**Table:**Quantitative results (uncertainties are statistical-only, 1  $\sigma$ )

Spectrum	Quantity	O			Si			Ti			Ba			Sum
<b>Benitoite Bulk</b>	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	± 0.0010		0.1547	± 0.0002		0.1090	± 0.0006		0.3673	± 0.0015		
	weight %	<b>35.26</b>	± 0.06		<b>19.72</b>	± 0.03		<b>11.66</b>	± 0.07		<b>34.40</b>	± 0.14		101.04
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	<a href="#">/home/nicholas/D TSA-II Reports/2009/May/24-May-2009/residual2361900809694010741.msa</a>													

Results reported as:

**Weight Percent:**

**Normalize weight percent:**

**Atomic percent:**

Normally we report results like this

Used to report particle results

Relative to atom count rather than weight fraction

Also reported:

**Line:**

**Z·A·F terms:**

**k-ratios:**

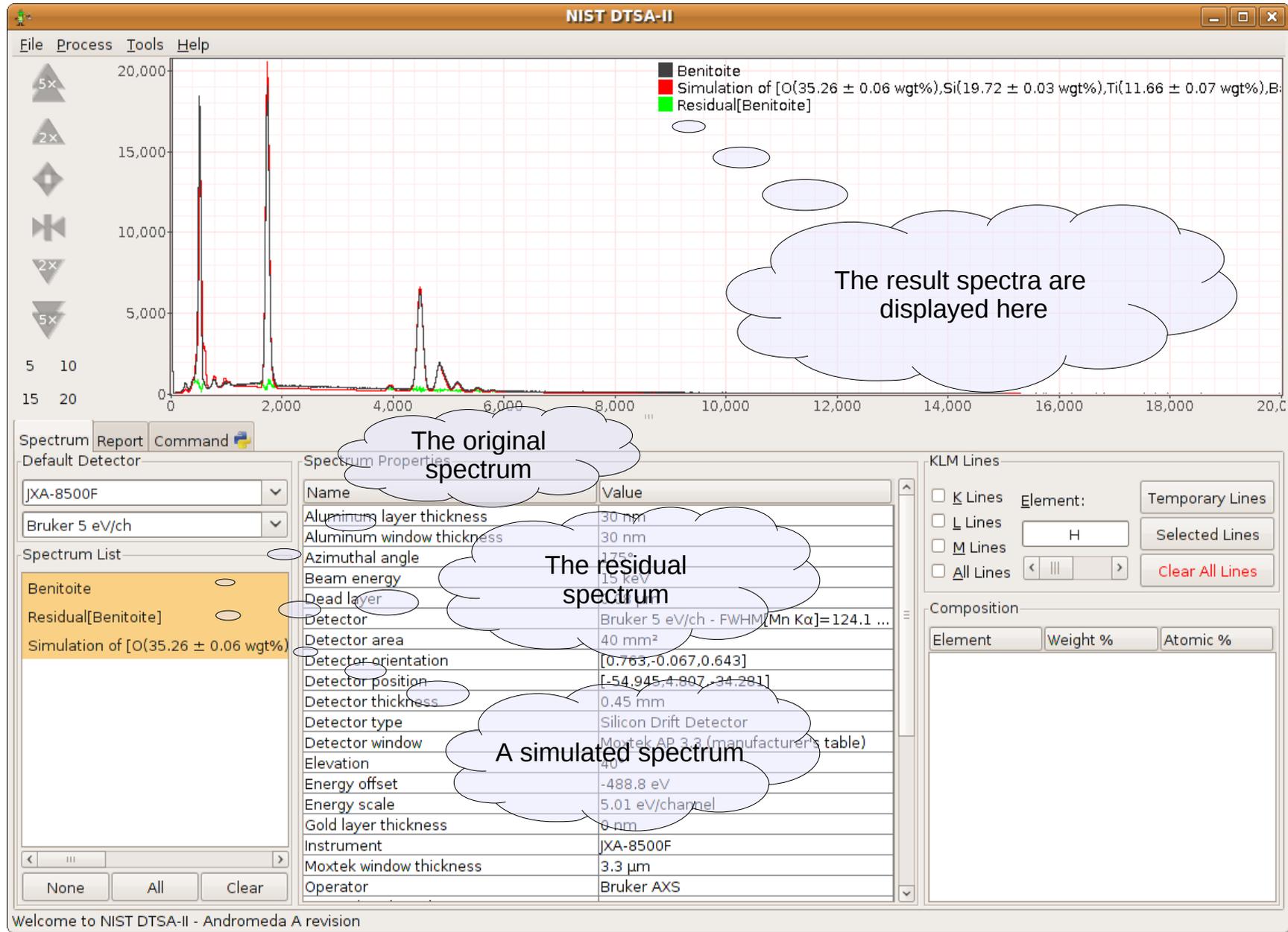
**Sum:**

The x-ray line family used to calculate the composition

The quantitative correction factors

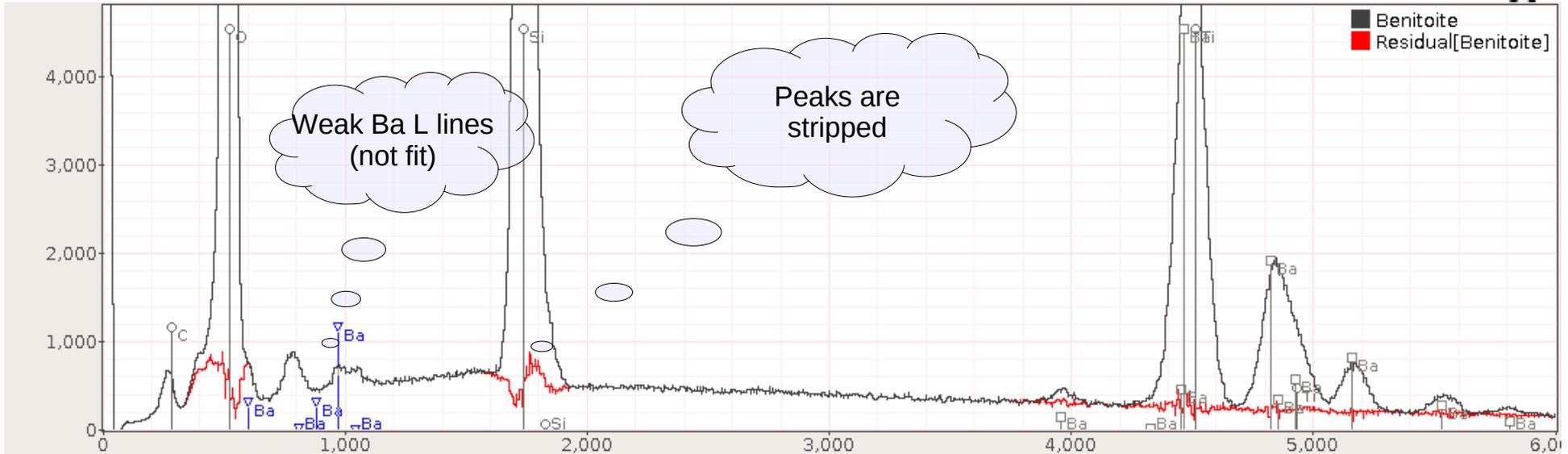
The raw ratio between unknown and standard

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

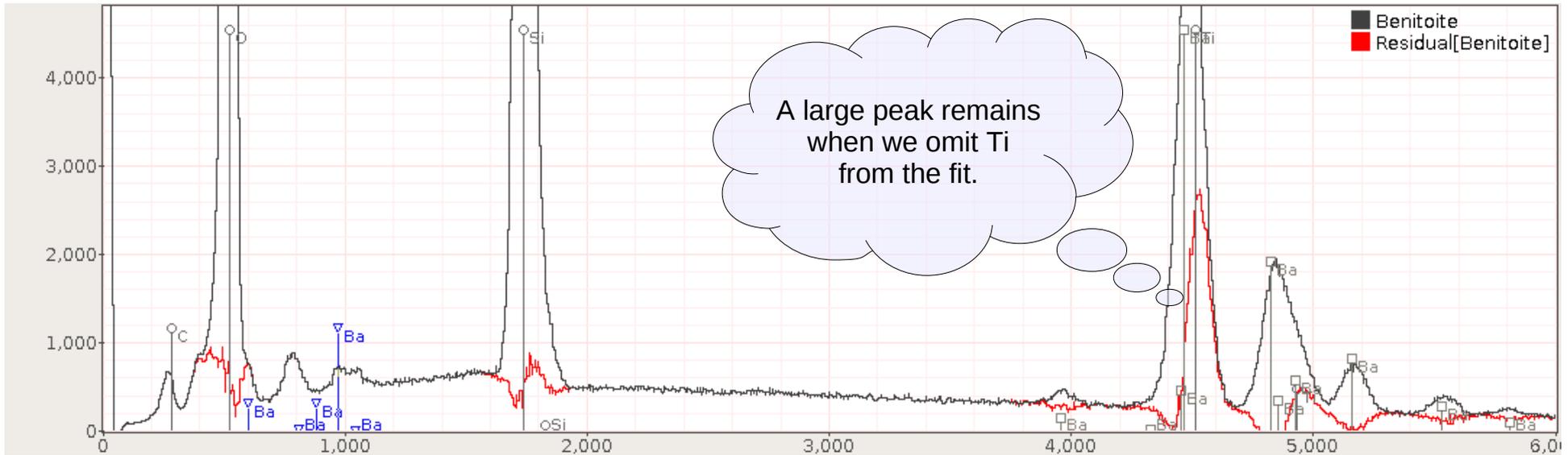




# A good residual spectrum



# 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



NIST DTSA-II

Select the Preferences item from the File menu (Windows & Linux) or the Application menu (OS X)

File Process Tools Help

- Open
- Open Report
- Save As
- Bulk rename
- Import from CSV
- Batch export >
- Import into database
- Search database
- Print >
- Preferences**
- Exit

15 20 0 1,000 2,000 3,000 4,000 5,000 6,000

Spectrum Report Command

Default Detector: JXA-8500F, Bruker 5 eV/ch

Spectrum List: None, All, Clear

Spectrum Properties

Name	Value
------	-------

KLM Lines

K Lines Element: Ba Temporary Lines

L Lines Selected Lines

M Lines

All Lines < ||| > Clear All Lines

Microanalytical Composition

Element	Weight %	Atomic %
---------	----------	----------

Welcome to NIST DTSA-II - Andromeda A revision



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

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

The screenshot shows the NIST DTSA-II software interface. A dialog box titled "Instruments and Detectors" is open. On the left, a tree view shows the following structure:

- 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

The main area of the dialog displays an "Overview" section with the following text:

D TSA-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

At the bottom of the dialog is an "Add" button. Below the dialog, the main software window has buttons for "None", "All", and "Clear".

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



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

The screenshot shows the NIST DTSA-II software interface. A window titled "DTSA-II - Preferences" is open, displaying a tree view on the left with "Probe" selected under "Instruments and Detectors". The main area shows "Instrument name" set to "Probe" and three buttons: "Add Si(Li) detector", "Add SDD detector", and "Add microcalorimeter". A status bar at the bottom reads "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  
2x  
5  
5x  
5 10  
15 20 0

Spectrum Rep  
Default Detect  
JXA-8500F  
Bruker 5 eV/c  
Spectrum List

None All Clear

DTSA-II - Preferences

Detector - Basic SDD

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

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

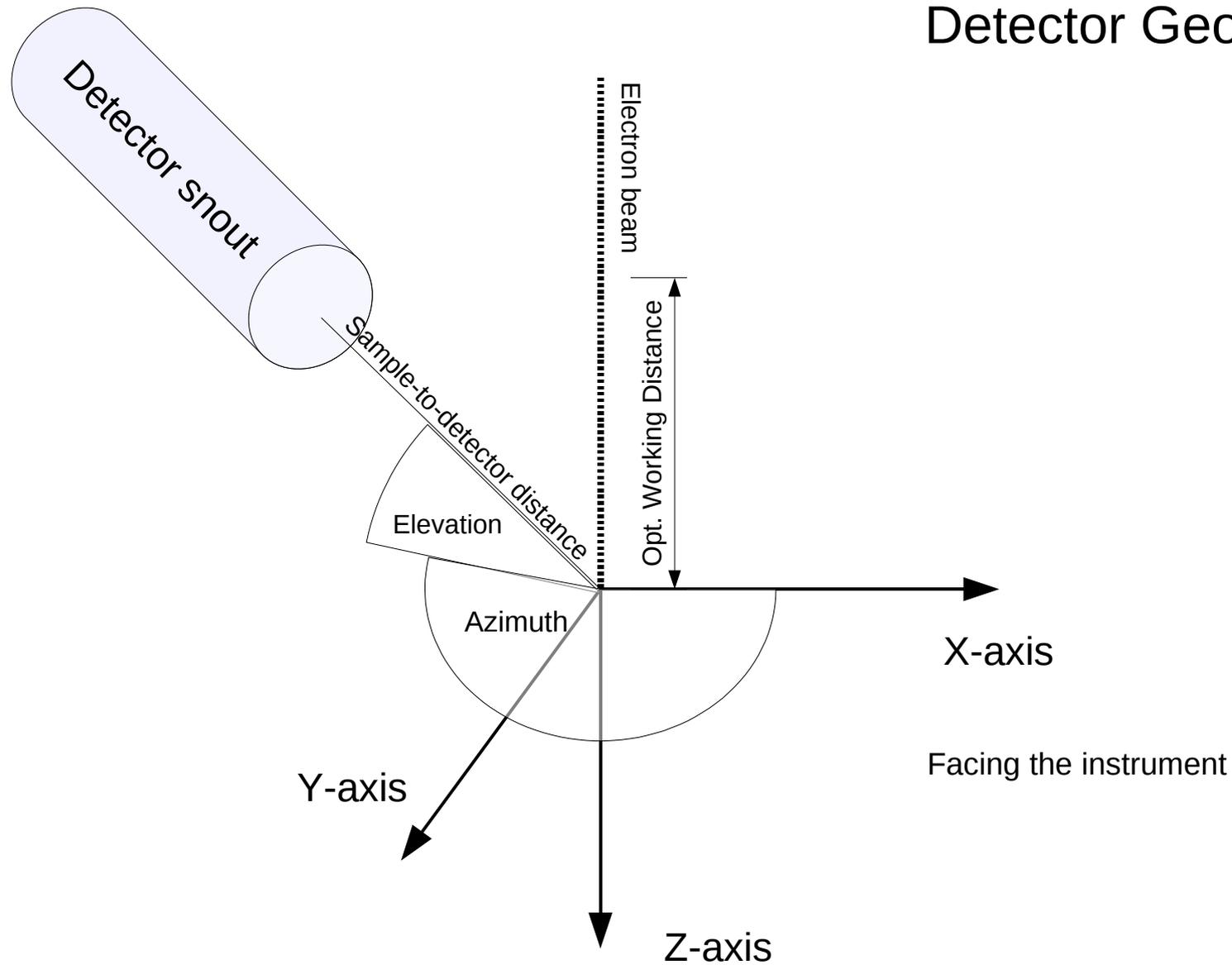
Temporary Lines  
Selected Lines  
Clear All Lines  
Atomic %

6,01

Welcome to NIST DTSA-II - Andromeda A revision

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	Enable detector <input checked="" type="checkbox"/>
Name	Detector name <input type="text" value="Basic SDD"/>
Import	Import from spectrum <input type="button" value="Import"/>
Window	Window <input type="text" value="No window"/>
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"/> mm <sup>2</sup> 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  $K\alpha$  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 2x 5x 2x 5x

15 10 5 0

5 10 15 20 0 2,000 4,000

Spectrum Report Command

Default Detector

Probe

Basic SDD

Spectrum List

None All Clear

Element Weight % Atomic %

20,0

Now the new detector type is available.

Default Detector

Probe

Basic SDD

Important Information

When spectra are loaded from disk, the program applies the properties from the default detector to the spectrum. This will overwrite the instrument and detector related parameters contained within the spectrum file but does not overwrite the live time, probe current and other spectrum specify data. If you don't want this behavior, select "--None--" for the instrument and detector. Use the "--None--" option with care as the instrument and detector data in spectrum files is often wrong.



**NIST DTSA-II**

File Process **Tools** Help

- Edit spectrum properties
- Assign material
- Quantification alien
- Simulation alien
- Calibration alien**
- Report note

5x 15  
2x  
10  
5x  
5 10  
15 20  
0 2,000 4,000 6,000 8,000 10,000 12,000 14,000 16,000 18,000 20,000

Spectrum Report Command

Default Detector: Probe, Basic SDD

Spectrum List: None, All, Clear

Spectrum Properties

KLM Lines:  K Lines,  L Lines,  M Lines,  All Lines. Element: Ba. Buttons: Temporary Lines, Selected Lines, Clear All Lines.

Composition: Element, Weight %, Atomic %

Welcome to NIST DTSA-II - Andromeda A revision

The Calibration Alien is used to specify the current calibration. Each detector can be associated with multiple calibrations. Calibrations are identified by date. When a spectrum is read from disk, it is associated with the last calibration performed before the spectrum was measured.



**NIST DTSA-II**

File Process Tools Help

5x 15  
2x  
10  
5x  
5 10  
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**

First page  
Next: Calibration method

**Select a detector**

Instrument and Detector

Instrument: Probe  
Detector: Basic SDD - FWHM[Mn K $\alpha$ ]= 124.0 eV - initial

Message: Select a detector to calibrate

More...

Back Next Finish Cancel

ent: Temporary Lines  
Ba Selected Lines  
Clear All Lines  
Weight % Atomic %

First, specify which detector to calibrate.



**NIST DTSA-II**

File Process Tools Help

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

Spectrum Report Command  
Default Detector  
Probe  
Basic SDD  
Spectrum List

0 2,000

00 18,000 20,000

ent: Temporary Lines  
Ba Selected Lines  
Clear All Lines  
Weight % Atomic %

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

**Calibrate an EDS detector**

Previous: *Select a detector*

Next: *Measured spectrum*

**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

Back Next Finish Cancel

More...

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



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

**Calibrate an EDS detector**

Previous: *Calibration method*      Next: *Fit Results*

**Measured spectrum**

Specify the material  
Material:

Specify a spectrum  
Spectrum:

Live time:  sec.      Probe current:  nA

Specify an effective date  
Effective date:

Message: The reference beam energy is 15.0 keV.

Specify the material from which the spectrum was collected.

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



**NIST DTSA-II**

File Process Tools Help

5x 2x 10x 5x

15 10 5 0

5 10 15 20 0 2,000

Spectrum Report Command

Default Detector

Probe

Basic SDD

Spectrum List

Mn  
Fit[Mn,Mn standard]

None All Clear

Welcome to NIST DTSA-II - Andromeda revision

**Calibrate an EDS detector**

Previous: Measured spectrum

### Fit Results

Next: Fit results

Progress

Message: More...

Back Next Finish Cancel

Weight % Atomic %

Temporary Lines  
Selected Lines  
Clear All Lines

Ba

The program will take a few seconds to fit the spectrum.



**NIST DTSA-II**

File Process Tools Help

5x 2x 5x 2x 5x

15 10 5 0

5 10 15 20 0 2,000

Spectrum Report Command

Default Detector

JXA-8500F

Bruker 5 eV/ch

Spectrum List

Mn

Fit[Mn,Mn standard]

None All Clear

Welcome to NIST DTSA-II - Andromeda revision

**Calibrate an EDS detector**

Previous: Fit Results

**Fit results**

Finish

Energy calibration

Fano factor  $0.1122 \pm 0.0006$

Noise  $4.67 \pm 0.06$  eV

FWHM  $124.56 \pm 0.40$  eV

Resolution calibration

Zero offset  $-467.00 \pm 0.34$  eV

Channel width  $4.9944 \pm 0.0003$  eV/channel

Message: More...

Back Next Finish Cancel

ent: Temporary Lines

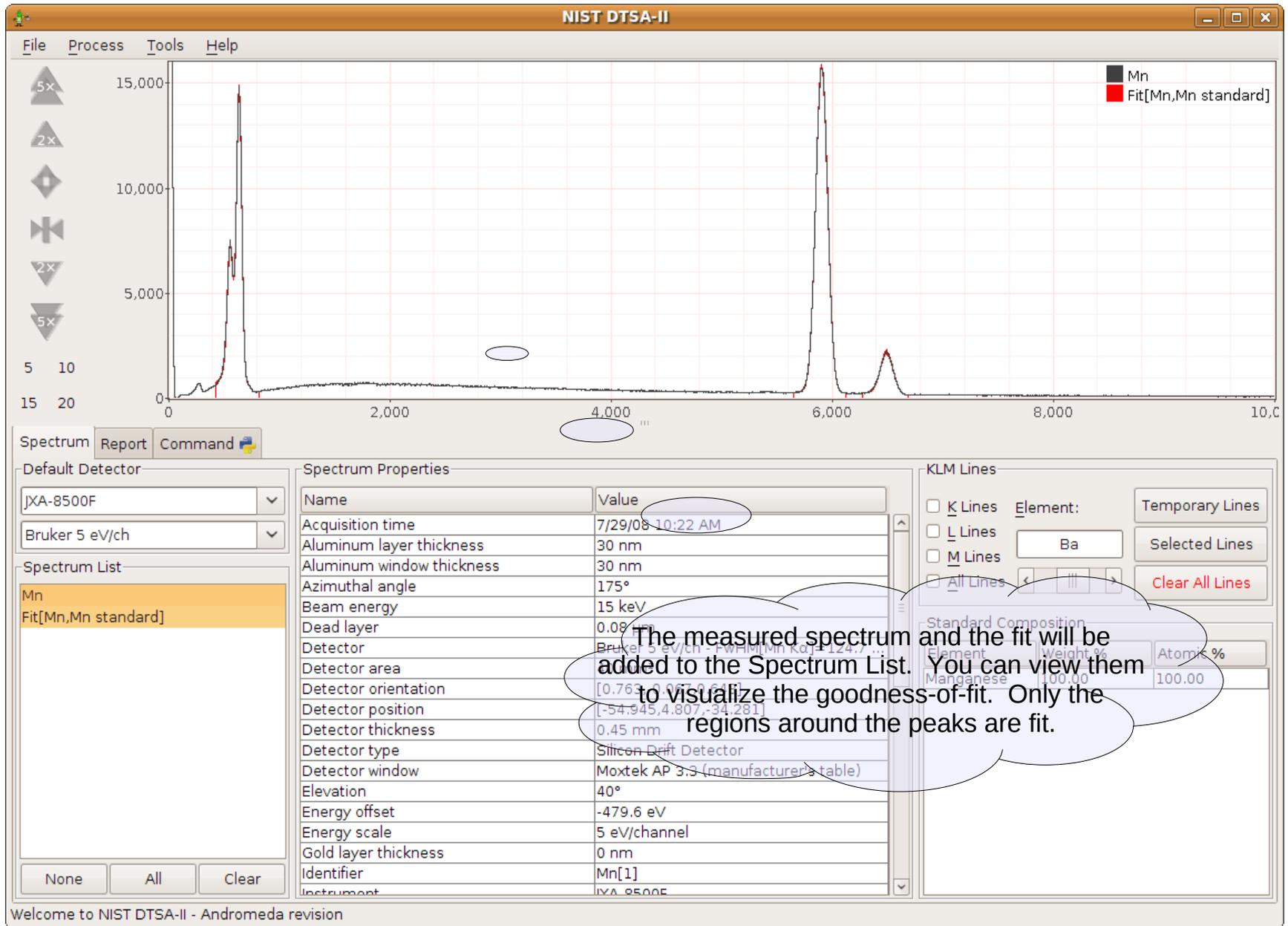
Ba Selected Lines

Clear All Lines

sition

Weight % Atomic %

A summary of the fit parameters is shown. Check that the measured parameters make sense and the click the "Finish" button to add the calibration to the database. Clicking "Cancel" won't add the calibration to the database.





### 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 $\alpha$	414962 ± 644
Manganese	K $\beta$	59911 ± 245
Manganese	L-family	251707 ± 502
Manganese	L $\alpha$	6 ± 2
Manganese	L $\beta$	177315 ± 421
Manganese	L $\gamma$	11 ± 3

### Energy Calibration

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

### Bremsstrahlung

Linear	Quadratic
317607.9 ± 11358.5	136.0 ± 0.0

### Detector Resolution

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

The Report also contains summary information from the calibration fit.



**NIST DTSA-II**

File Process **Tools** Help

- Edit spectrum properties
- Assign material
- Quantification alien
- Simulation alien**
- Calibration alien
- Report note

Select the "Simulation Alien" to compute simulated x-ray spectra from bulk, thin-film or particulate geometries.

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

0 2,000 4,000 6,000 8,000 10,000 12,000 14,000 16,000 18,000 20,000

Spectrum Report Command

Default Detector: JXA-8500F  
Bruker 5 eV/ch

Spectrum List

None All Clear

Spectrum Properties

KLM Lines

K Lines  
 L Lines  
 M Lines  
 All Lines

Element: Ba

Temporary Lines  
Selected Lines  
Clear All Lines

Composition

Element	Weight %	Atomic %
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Welcome to NIST DTSA-II - Andromeda A revision



**NIST DTSA-II**

File Process Tools Help

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

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

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

**Spectrum simulation**

Simulation Mode

Next: Configure sample

Analytical Simulation

- Analytical model of a bulk, homogeneous material

Monte Carlo Simulation

- Monte Carlo model of a bulk, homogeneous material
- Monte Carlo model of a film on a bulk, homogeneous substrate
- Monte Carlo model of a sphere on a bulk, homogeneous substrate
- Monte Carlo model of a cube on a bulk, homogeneous substrate
- Monte Carlo model of an inclusion in a bulk, homogeneous substrate

Message: Select the type of spectrum simulation to perform.

Back Next

Weight % Atomic %

Temporary Lines  
Selected Lines  
Clear All Lines

00 18,000 20,000

Analytical simulations are based on a  $\phi(\rho z)$  model x-ray production. They are quick to calculate but limited in geometry.

Monte Carlo models simulate the trajectory of electrons in a material. They are slower to calculate but based on fundamental physics and can simulate complex geometries.



**NIST DTSA-II**

File Process Tools Help

5x 15  
2x  
5x

5 10  
15 20 0 2,000

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

None All Clear

Previous: *Simulation Mode*  
Next: *Instrument configuration*

### Spectrum simulation

## Configure sample

Materials and Scale

Substrate material Carbon Edit None

Sphere material Albite Edit

Sphere diameter 1.2  $\mu\text{m}$

Overscan particle

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

Back Next Finish Cancel

Weight % Atomic %

Temporary Lines  
Selected Lines  
Clear All Lines

Weight % Atomic %

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

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.

Specify the scale of the particle in microns.



**NIST DTSA-II**

File Process Tools Help

5x 15  
2x  
10  
5x

5 10  
15 20 0 2,000

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

None All Clear

Welcome to NIST DTSA-II - Andromeda A revision

**Spectrum simulation**

Previous: *Configure sample*  
Next: *Other options*

**Instrument configuration**

Instrument Parameters

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

Message: More...

Back Next Finish Cancel

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

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

If the detector is configured correctly, the simulated spectrum will approximately match a measured spectrum collected with the same probe dose. Probe dose is (probe current) · (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.

The screenshot shows the NIST DTSA-II software interface. The main window is titled "NIST DTSA-II" and contains a large grid area. A "Spectrum simulation" dialog box is open, showing "Other options" for the simulation. The dialog box has a title bar with a close button and a small mascot icon. It includes navigation buttons for "Previous: Instrument configuration" and "Next: Perform Simulation". The "Noise parameters" section has a checked checkbox for "Apply simulated count statistics" and an "Instance count" input field set to "1". The "Extended output" section has a checked checkbox for "X-ray generation images (takes a little longer)" and a dropdown menu set to "Run the default number of electron trajectories". At the bottom of the dialog are "Back", "Next", "Finish", and "Cancel" buttons. The background window shows a menu bar with "File", "Process", "Tools", and "Help", and a status bar at the bottom that reads "Welcome to NIST DTSA-II - Andromeda revision".



**NIST DTSA-II**

File Process Tools Help

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

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

None All Clear

Welcome to NIST DTSA-II - Andromeda revision

**Spectrum simulation**

Previous: Other options

**Perform Simulation** Finish

Progress

Message: Computing the requested spectra... More...

Back Next Finish Cancel

ent: Temporary Lines  
Ba Selected Lines  
Clear All Lines  
Weight % Atomic %

The simulation will take tens of seconds to minutes to run depending on the speed of your computer.



**NIST DTSA-II**

File Process Tools Help

Noisy[MC simulation of a 1.200  $\mu\text{m}$  diameter sphere of Albite on Carbon (overscan)]

The simulated spectrum will be added to the spectrum list and displayed in the spectrum display. You can manipulate it just like you would a measured spectrum.

5x  
2x  
5x  
2x  
5x

5 10  
15 20

0 1,000 2,000 3,000 4,000 5,000

Spectrum Report Command

Default Detector  
JXA-8500F  
Bruker 5 eV/ch

Spectrum List  
Noisy[MC simulation of a 1.200  $\mu\text{m}$  d...

Spectrum Properties

Name	Value
Acquisition time	5/26/09 6:02 AM
Aluminum layer thickness	30 nm
Aluminum window thickness	30 nm
Azimuthal angle	175°
Beam energy	15 keV
Dead layer	0.08 $\mu\text{m}$
Detector	Bruker 5 eV/ch - FWHM[Mn K $\alpha$ ]=124.7 ...
Detector area	40 mm <sup>2</sup>
Detector orientation	[0.763,-0.067,0.643]
Detector position	[-54.945,4.807,-34.281]
Detector thickness	0.45 mm
Detector type	Silicon Drift Detector
Detector window	Moxtek AP 3.3 (manufacturer's table)
Display name	Noisy[MC simulation of a 1.200 $\mu\text{m}$ di...
Elevation	40°
Energy offset	-479.6 eV
Energy scale	5 eV/channel
Gold layer thickness	0 nm
Instrument	JXA-8500F

KLM Lines

K Lines Element: Temporary Lines  
 L Lines Selected Lines  
 M Lines  
 All Lines Clear All Lines

Composition

Element	Weight %	Atomic %
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Welcome to NIST DTSA-II - Andromeda revision

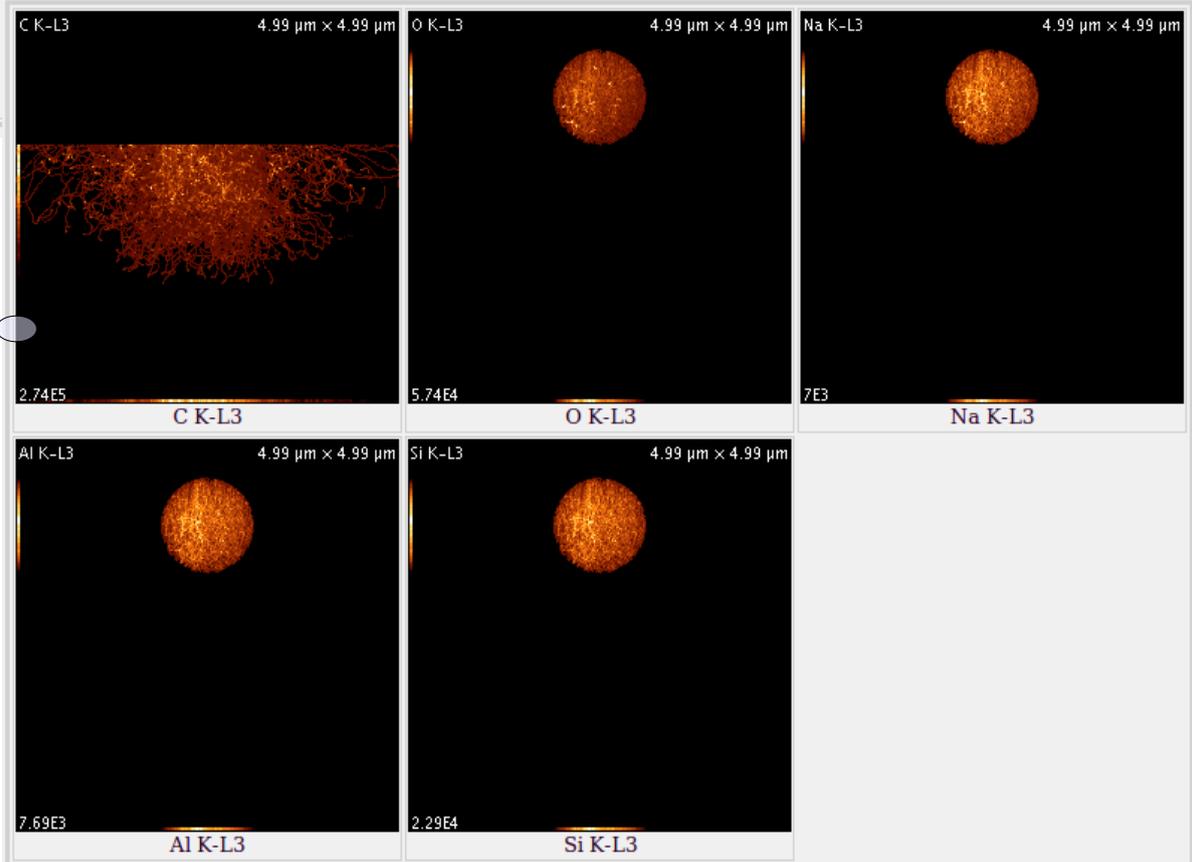


## Spectrum Simulation

<b>Simulation mode</b>	Monte Carlo model of a spherical sample			
<b>Substrate material</b>	Carbon = [C(100.00 wgt%),3 g/cc]			
<b>Object Material</b>	Albite = [O(48.81 wgt%),Na(8.77 wgt%),Al(10.29 wgt%),Si(32.13 wgt%),2.6 g/cc]			
<b>Sphere diameter</b>	1.200 $\mu\text{m}$			
<b>Beam energy</b>	15.000 keV			
<b>Probe dose</b>	120.000 nA·s			
<b>Instrument</b>	JXA-8500F			
<b>Detector</b>	Bruker 5 eV/ch			
<b>Calibration</b>	FWHM[Mn K $\alpha$ ]=124.7 eV - Sep 2, 2008 12:00:01 AM			
<b>Overscan</b>	true			
<b>Replicas (with Poisson noise)</b>	1			
<b>Result 1</b>	<a href="#">Noisy[MC simulation of a 1.200 <math>\mu\text{m}</math> diameter sphere of Albite on Carbon (overscan)] #1</a>			
<b>Trajectory view</b>	<a href="#">/home/nicholas/DTSA-II Reports/2009/May/26-May-2009/vrml3916995151658125043.vrml</a>			
<b>Intensity data</b>	<b>Transition</b>	<b>Generated 1/msR</b>	<b>Emitted 1/msR</b>	<b>Ratio (%)</b>
	<b>C K-L3</b>	177171.3	91265.3	51.5%
	<b>O K-L3</b>	33396.0	22585.6	67.6%
	<b>Na K-L3</b>	5738.9	4414.0	76.9%
	<b>Al K-L3</b>	6056.7	5319.8	87.8%
	<b>Si K-L3</b>	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.