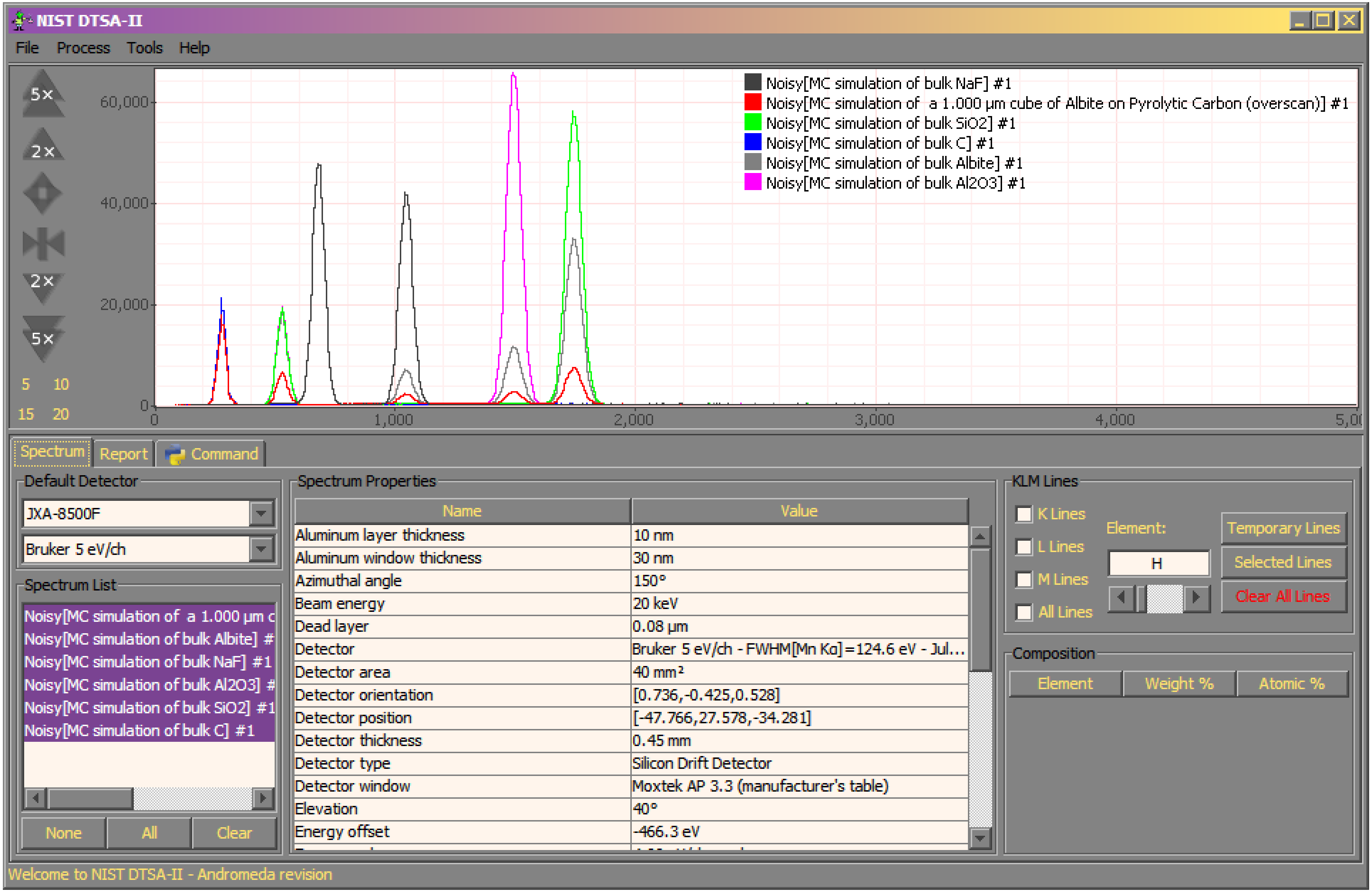


Using DTSA-II for Particle Analysis

Nicholas W. M. Ritchie
MAS Microanalysis of Particles 2009
21-Apr-2009

DTSA-II

- Software for electron probe microanalysis
  - Visualization / manipulation of spectra
  - Simulation of measured spectra
  - Quantification of measured spectra
- Available from the NIST web site
  - http://nist.gov/dtsa
- Available for
  - MS Windows, Apple OS X, Linux

With DTSA-II,

I. Simulation works

With DTSA-II,

I. Simulation works

II. Basic simulation is easy

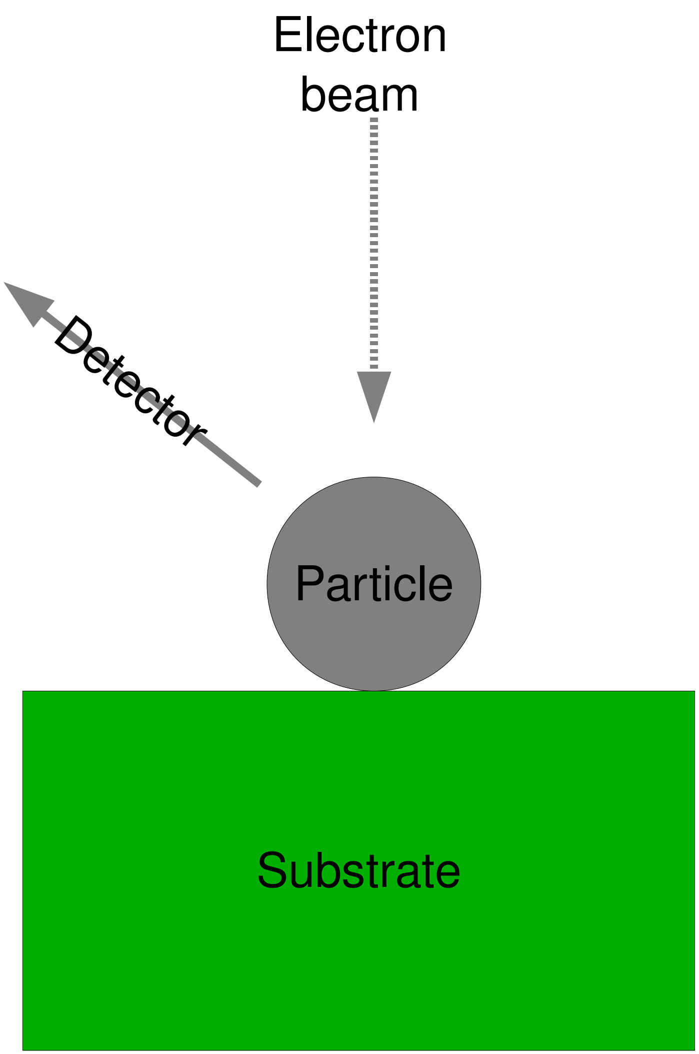
With DTSA-II,

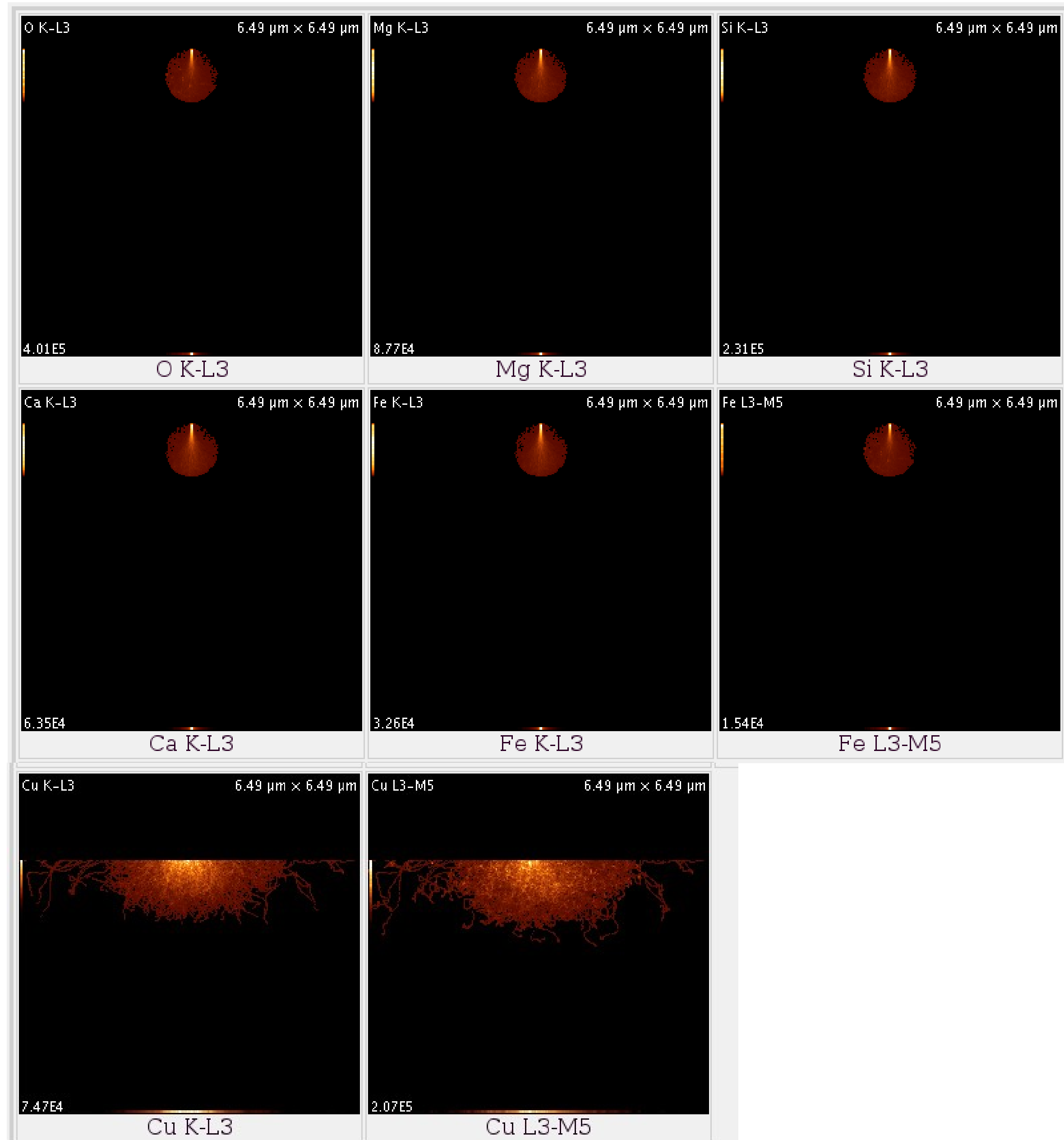
I. Simulation works
II. Basic simulation is easy
III. EDS particle quant is easy

With DTSA-II,

I. Simulation works
II. Basic simulation is easy
III. EDS particle quant is easy

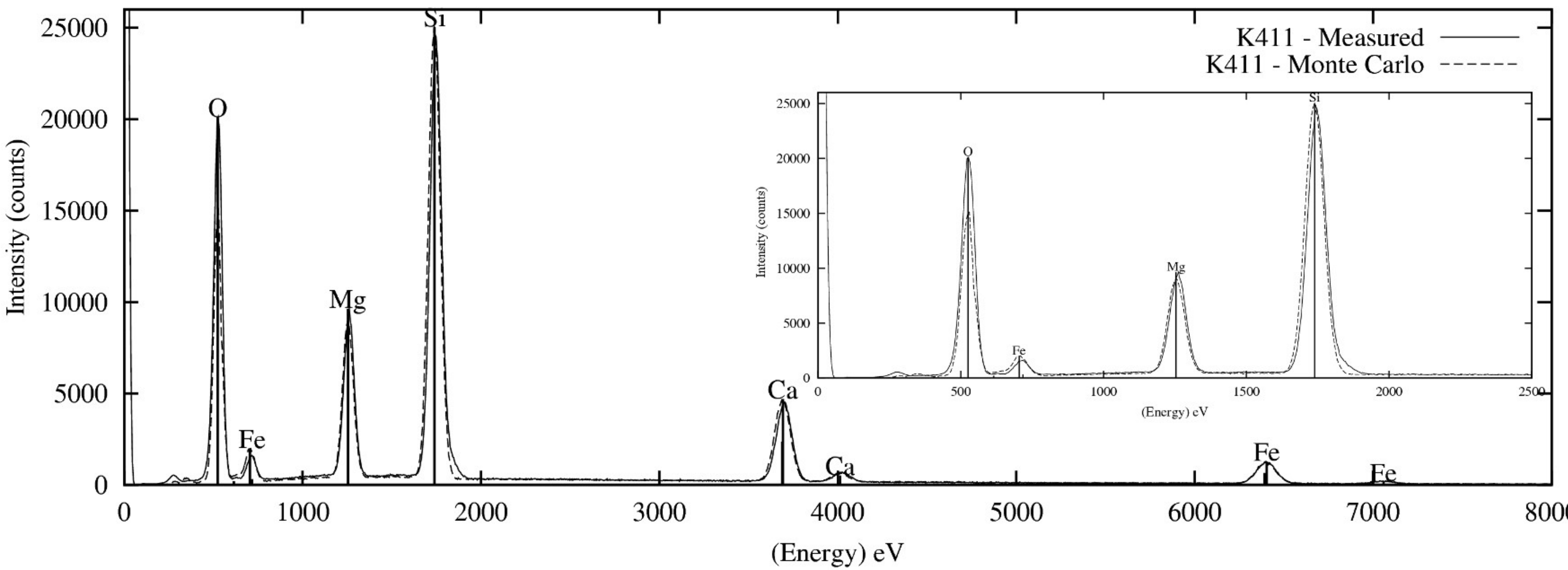
Getting the right answer still requires skill...





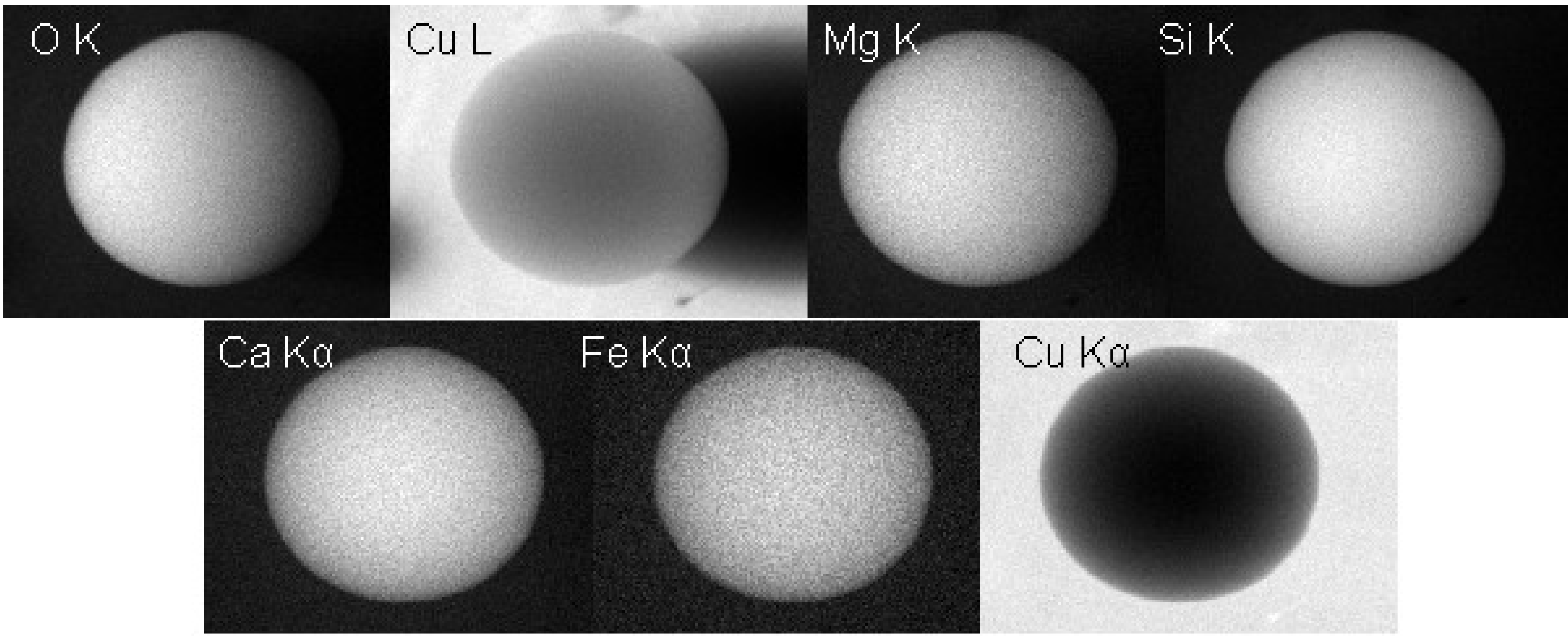
1 µm sphere at 20 keV

Simulating bulk K411 glass

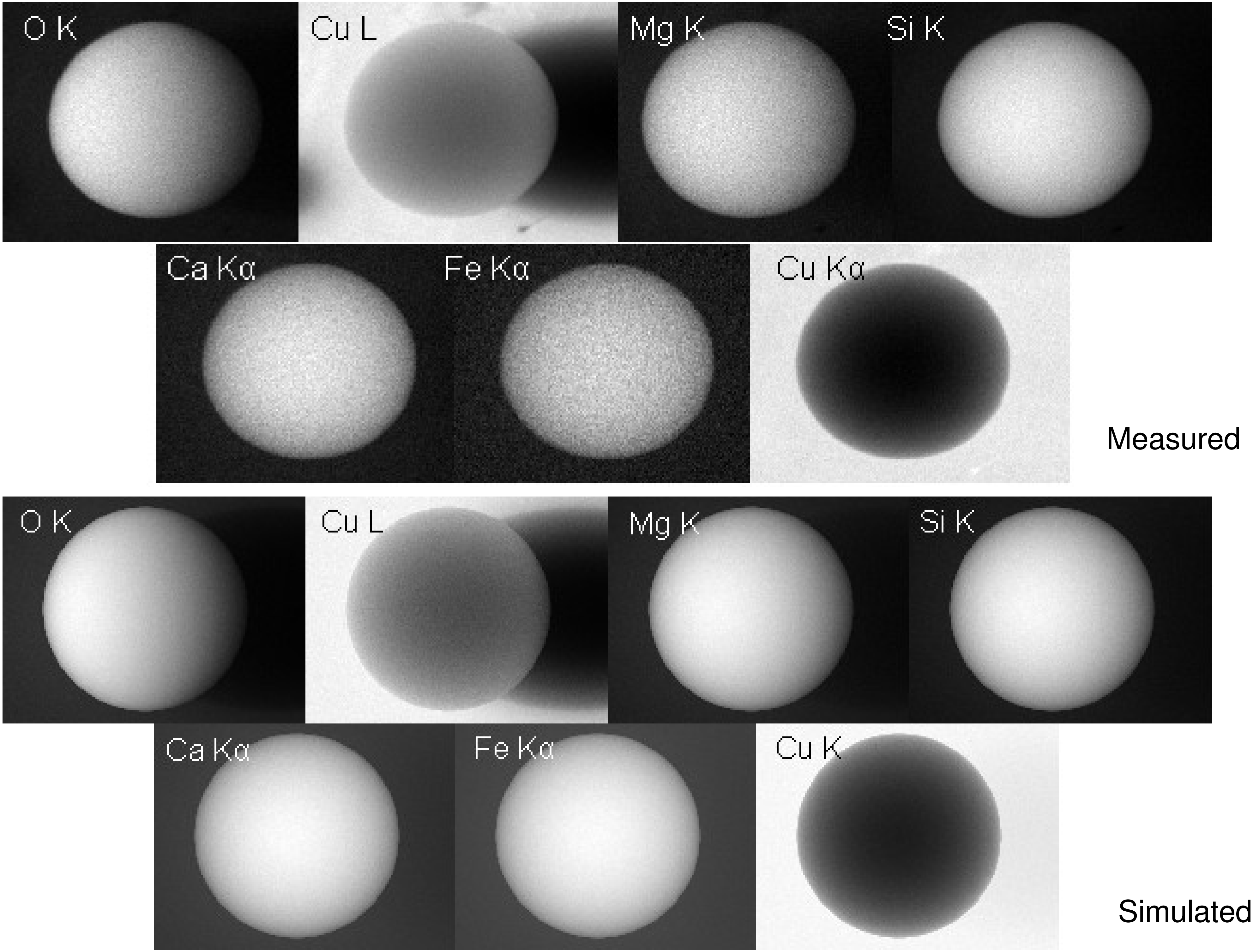


Element	SRM-2066 value (weight fraction)	Bulk Glass value (weight fraction)
Silicon	0.256 ± 0.017	0.2538 ± 0.0090
Calcium	0.112 ± 0.023	0.1106 ± 0.0014
Magnesium	0.092 ± 0.014	0.0885 ± 0.0012
Iron	0.112 ± 0.023	0.1121 ± 0.0016
Oxygen	0.429 ± 0.012	0.4236 ± 0.0024

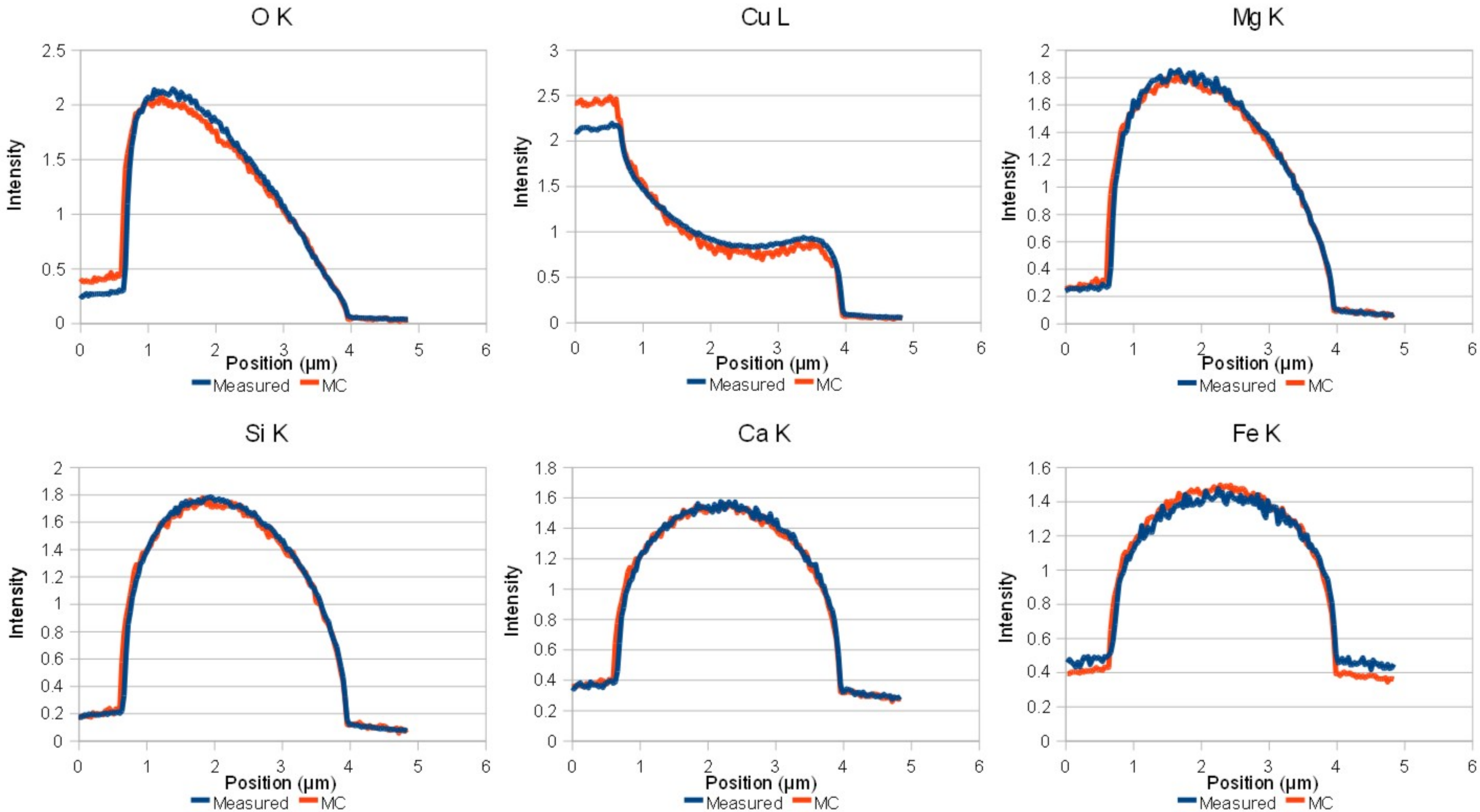
SRM-2066 - K411 microsphere

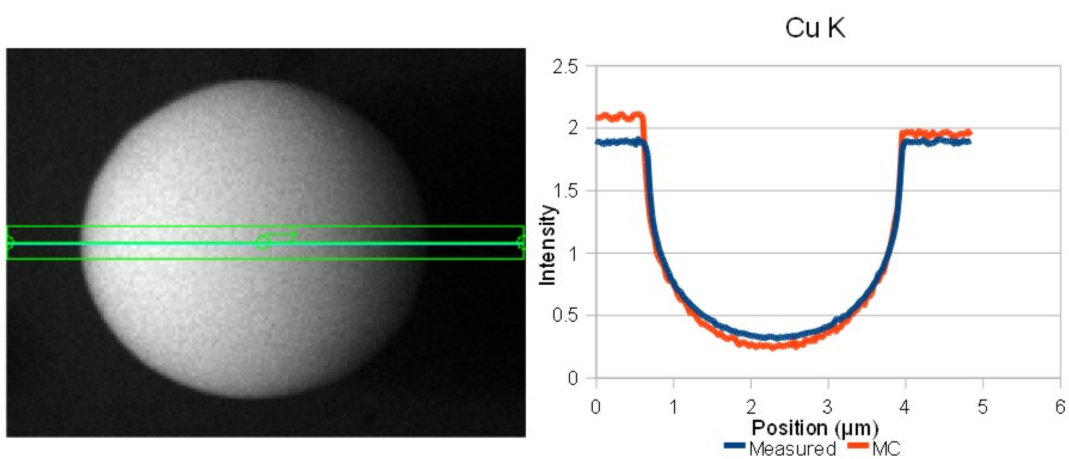


Data cube analyzed with LISPIX



O K
Cu L
Mg K
Si K
Ca Kα
Fe Kα
Cu Kα
Measured
O K
Cu L
Mg K
Si K
Ca Kα
Fe Kα
Cu K
Simulated



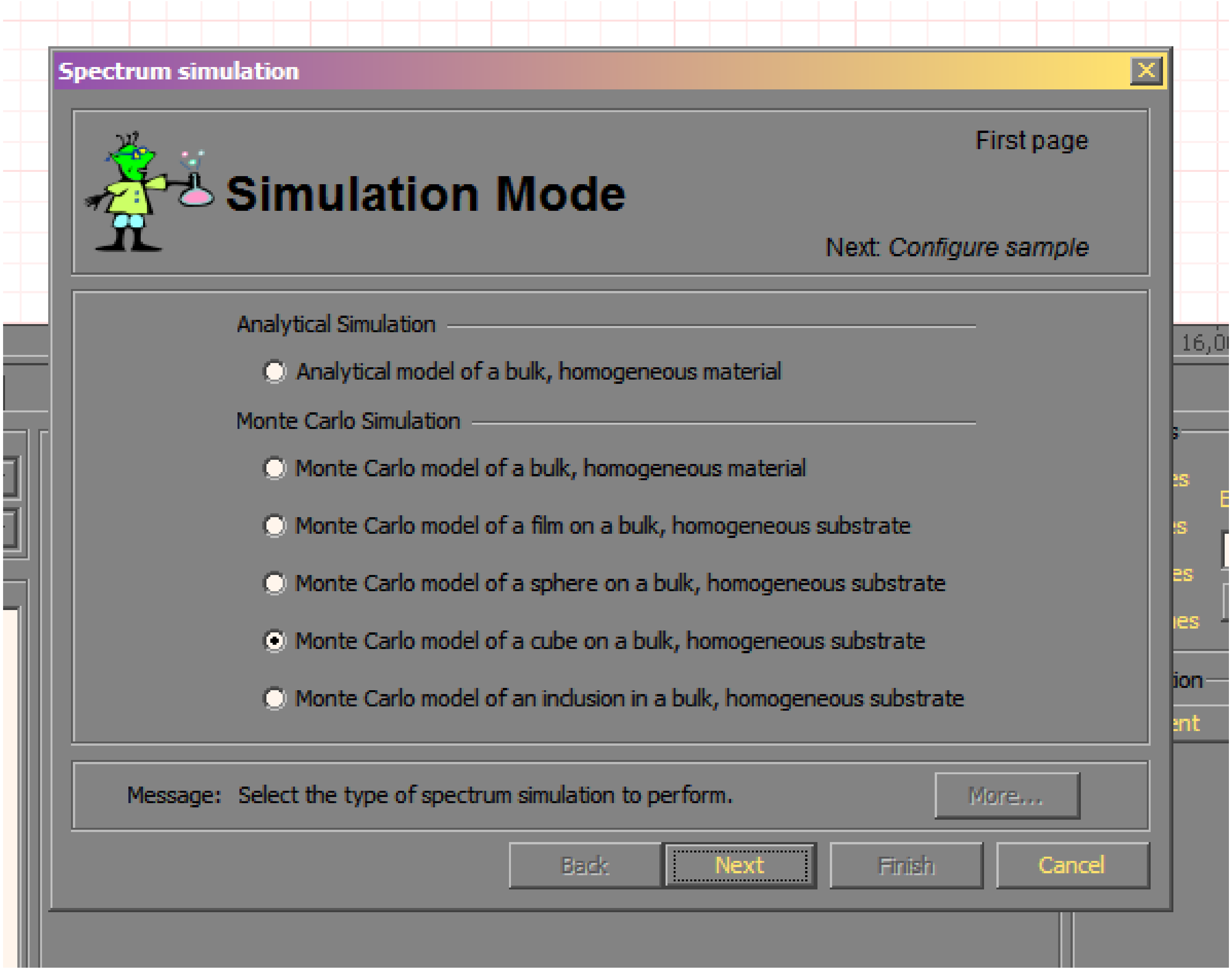


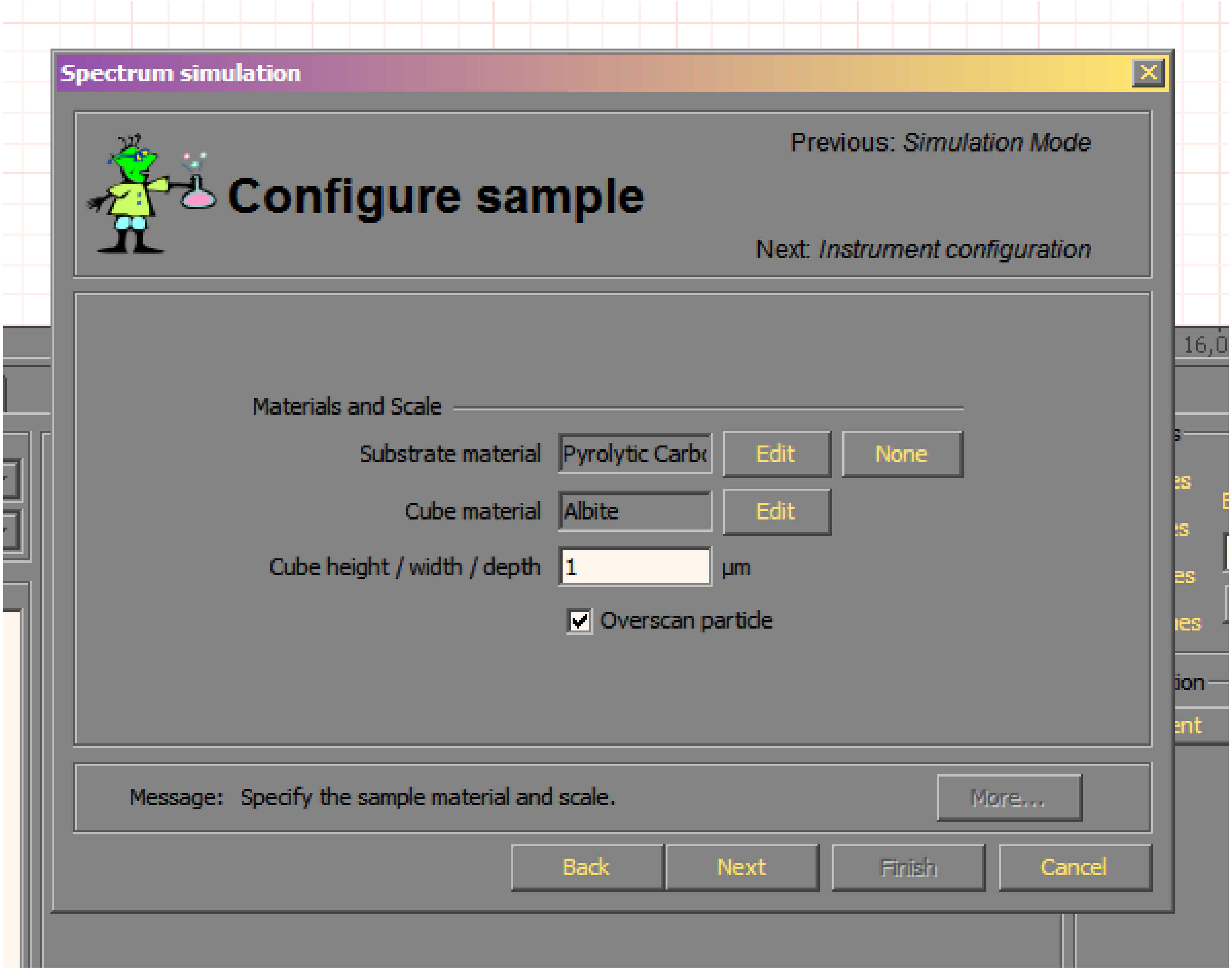
Scaled to average of all data.

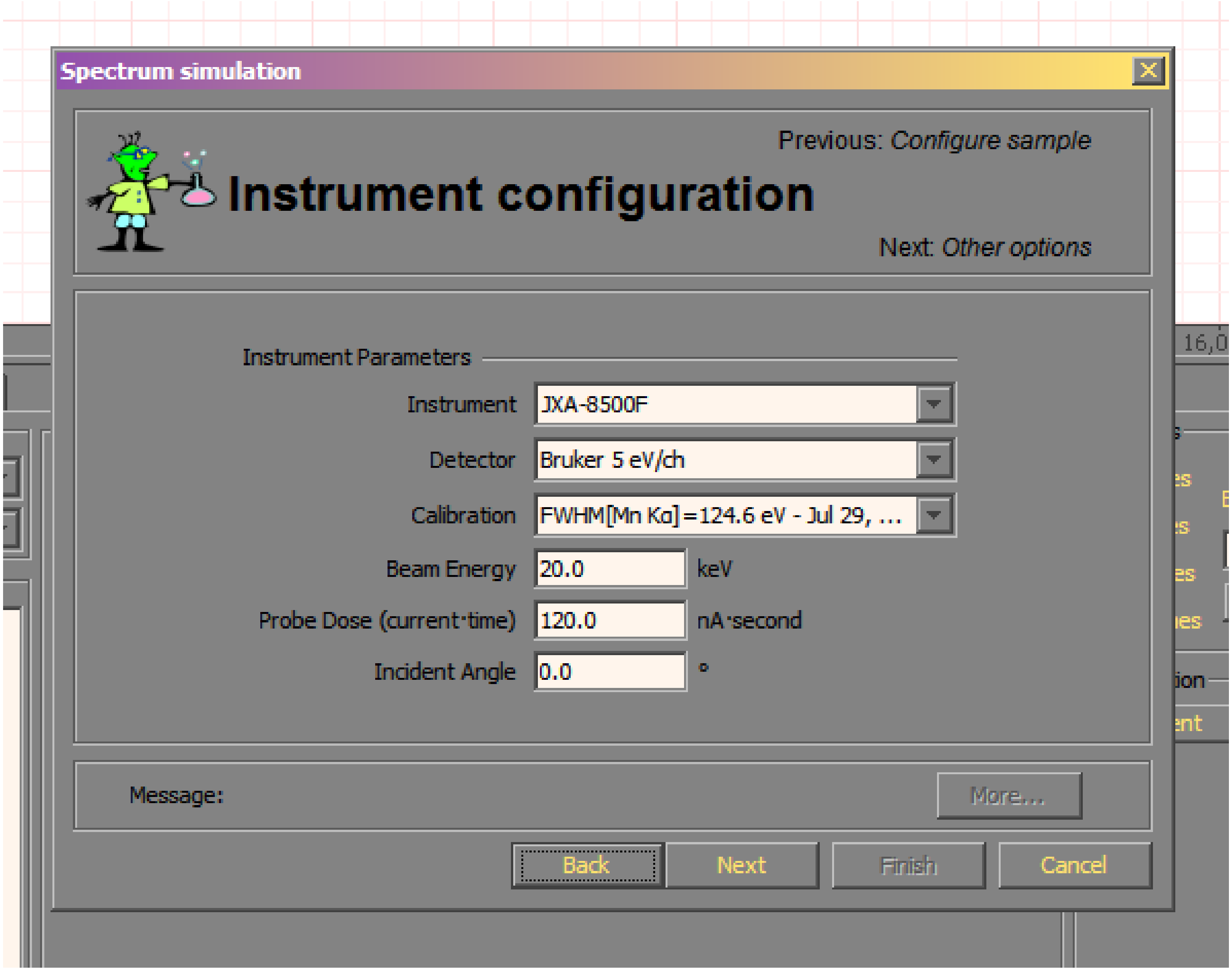
The hard way....

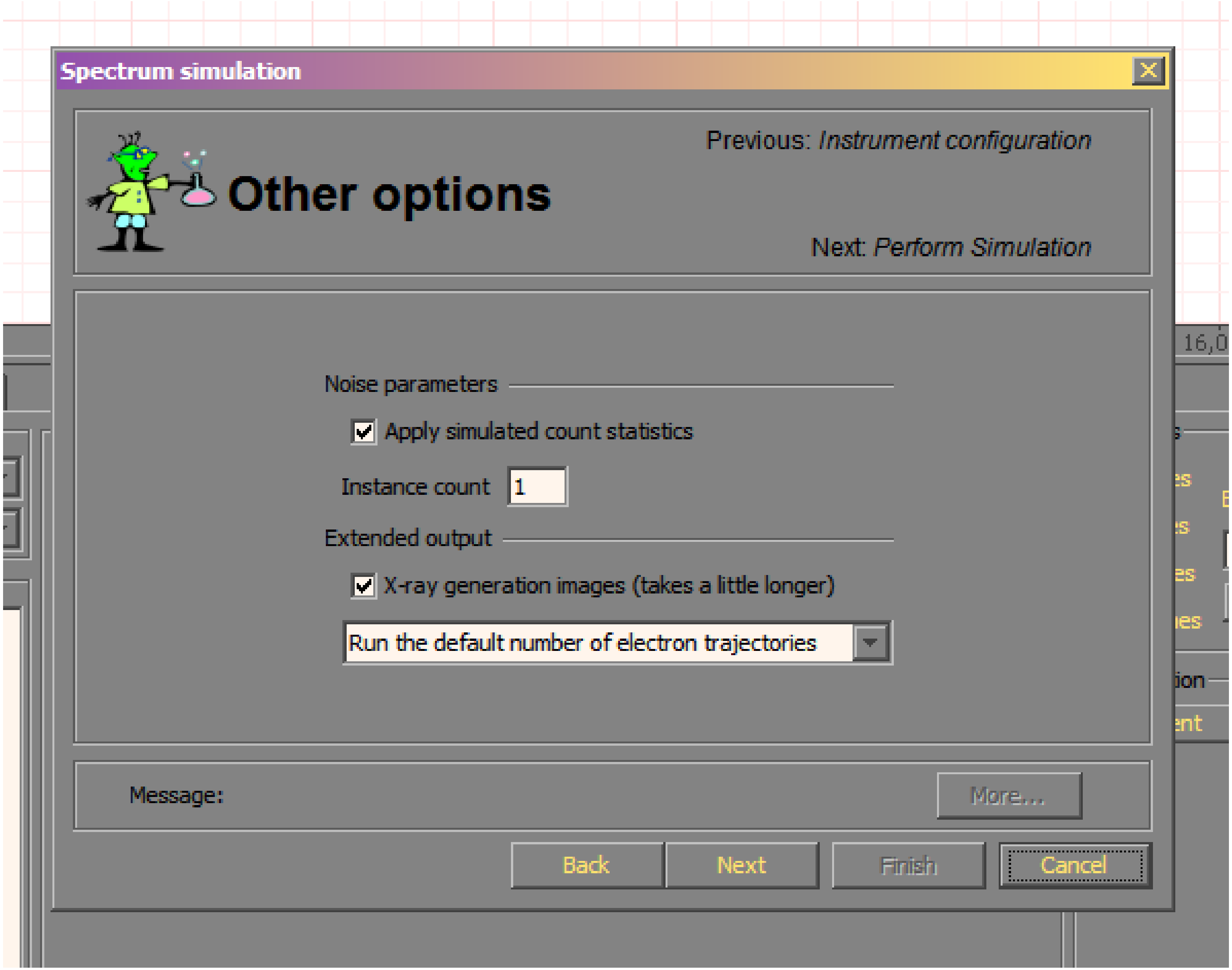
```
k411 = epq.Material(epq.Composition([epq.Element.Ca,epq.Element.Fe,epq.Element.O,
                                     epq.Element.Mg, epq.Element.Si],
                                    [0.112, 0.112, 0.429, 0.092, 0.256] ),
                                    epq.ToSI.gPerCC(2.946))
k411.setName("SRM-2066")
c = epq.MaterialFactory.createPureElement(epq.Element.Cu)

# Place the sample at the optimal location for the detector
det = findDetector("Bruker 10")
origin = epu.Math2.multiply(1.0e-3, epq.SpectrumUtils.getSamplePosition(det.getProperties()))

e0=25.0                  # beam energy (keV)
partDiameter = 3.3e-6    # particle size (meters)
size =  [ 160, 120 ]     # map dimensions
center = [ 76, 59 ]      # particle center (pixels)
stride = [ 4, 2 ]        # split the task up to run on multiple CPUs

# Bookkeeping
r = 0.5*partDiameter     # particle radius
pixSize = (2*r)/108.3    # edge length of a single pixel (meters)
dose = 150.0e-9          # A*sec
nTraj = 1000             # electrons
# Output files
path="%s/%s [%d,%d].%s" % ( DefaultOutput,k411,offset % stride[0], offset / stride[0],"%s")
res = ept.RippleFile(size[0],size[1],depth,ept.RippleFile.SIGNED,4,ept.RippleFile.DONT_CARE_ENDIAN,
            path % "rpl", path % "raw")
# Iterate over each pixel in the spectrum image
for x in range(offset % stride[0], size[0], stride[0]):
   if terminated:
      break
   for y in range(offset / stride[0], size[1], stride[1]):
      if terminated:
         break
      det.reset()        # clear the detector accumulator
      monte = nm.MonteCarloSS()
      gun=nm.GaussianBeam(1.0e-9)
      gun.setCenter([(x-center[0])*pixSize,(center[1]-y)*pixSize,-0.01])
      monte.setElectronGun(gun)
      monte.setBeamEnergy(epq.ToSI.keV(e0))
      # Create a sphere of k411
      center = epu.Math2.plus(origin,[0.0,0.0,r])
      monte.addSubRegion(monte.getChamber(),k411,nm.Sphere(center,r))
      # Place it on a carbon substrate
      monte.addSubRegion(monte.getChamber(), c, nm.MultiPlaneShape.createSubstrate([0.0,0.0,-1.0],
         epu.Math2.plus(origin,[0.0,0.0,2*r])) )
      # Add event listeners to model characteristic and Bremsstrahlung radiation
      xrel=nm.XRayEventListener2(monte,det)
      monte.addActionListener(xrel)
      brem=nm.BremsstrahlungEventListener(monte,det)
      monte.addActionListener(brem)
      # Run the MC
      monte.runMultipleTrajectories(nTraj)
      # Get the spectrum and assign properties
      spec=det.getSpectrum(dose / (nTraj * epq.PhysicalConstants.ElectronCharge))
      res.seek(y, x)
      res.write(epq.SpectrumUtils.toIntArray(spec)[0:2048])
      # Write the spectrum to disk and display
      fos = jio.FileOutputStream("%s/%s[%d,%d].msa" % (DefaultOutput, k411, x, y))
      ept.WriteSpectrumAsEMSA1_0.write(spec,fos,0)
      fos.close()
res.close()              # close the ripple file
```

http://dtsa2.svnrepository.com/DTSA2_Scripts/trac.cgi

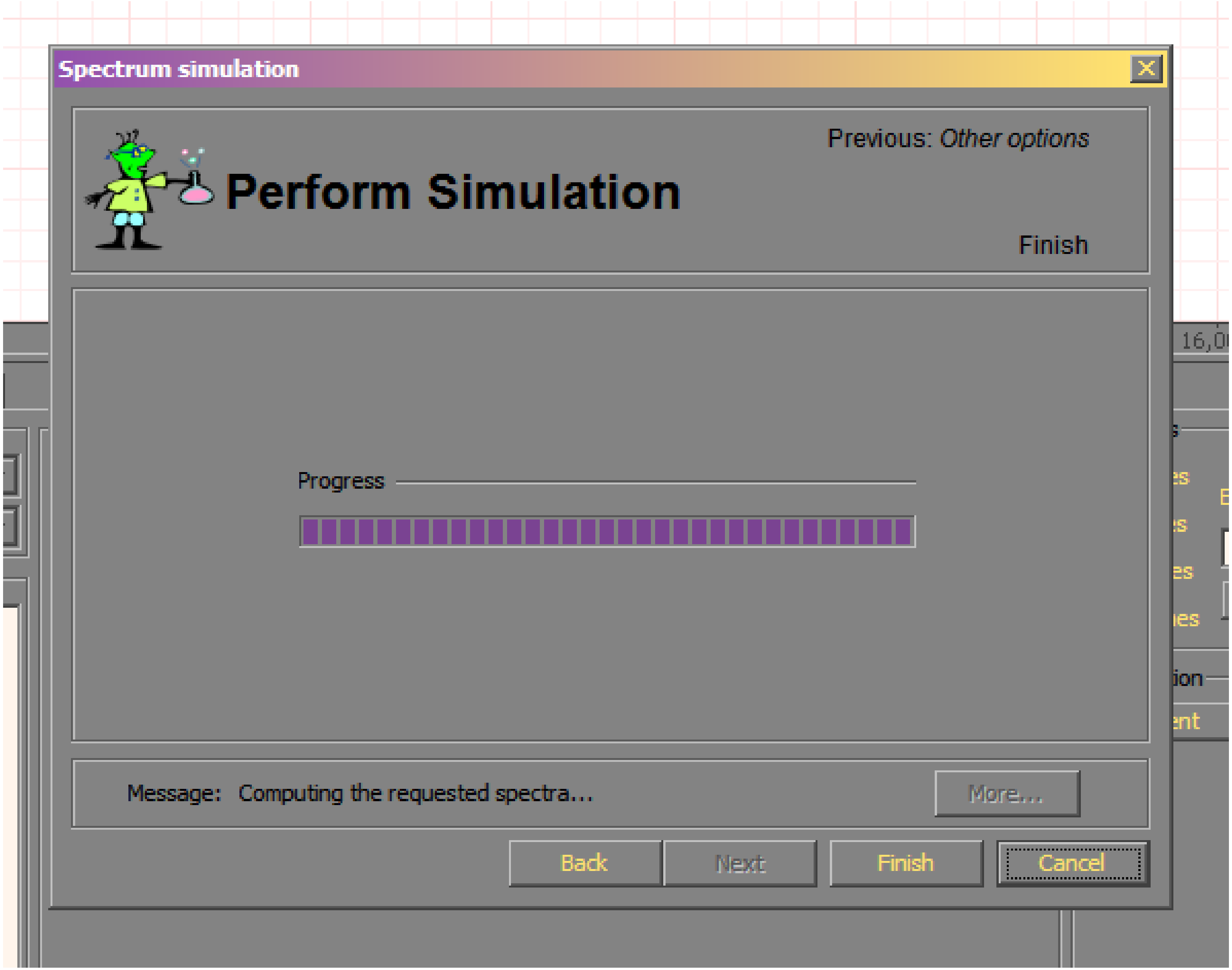

Spectrum simulation
First page
Simulation Mode
Next: Configure sample
Analytical Simulation
Analytical model of a bulk, homogeneous material
Monte Carlo Simulation
Monte Carlo model of a bulk, homogeneous material
Monte Carlo model of a film on a bulk, homogeneous substrate
Monte Carlo model of a sphere 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.
More...
Back
Next
Finish
Cancel

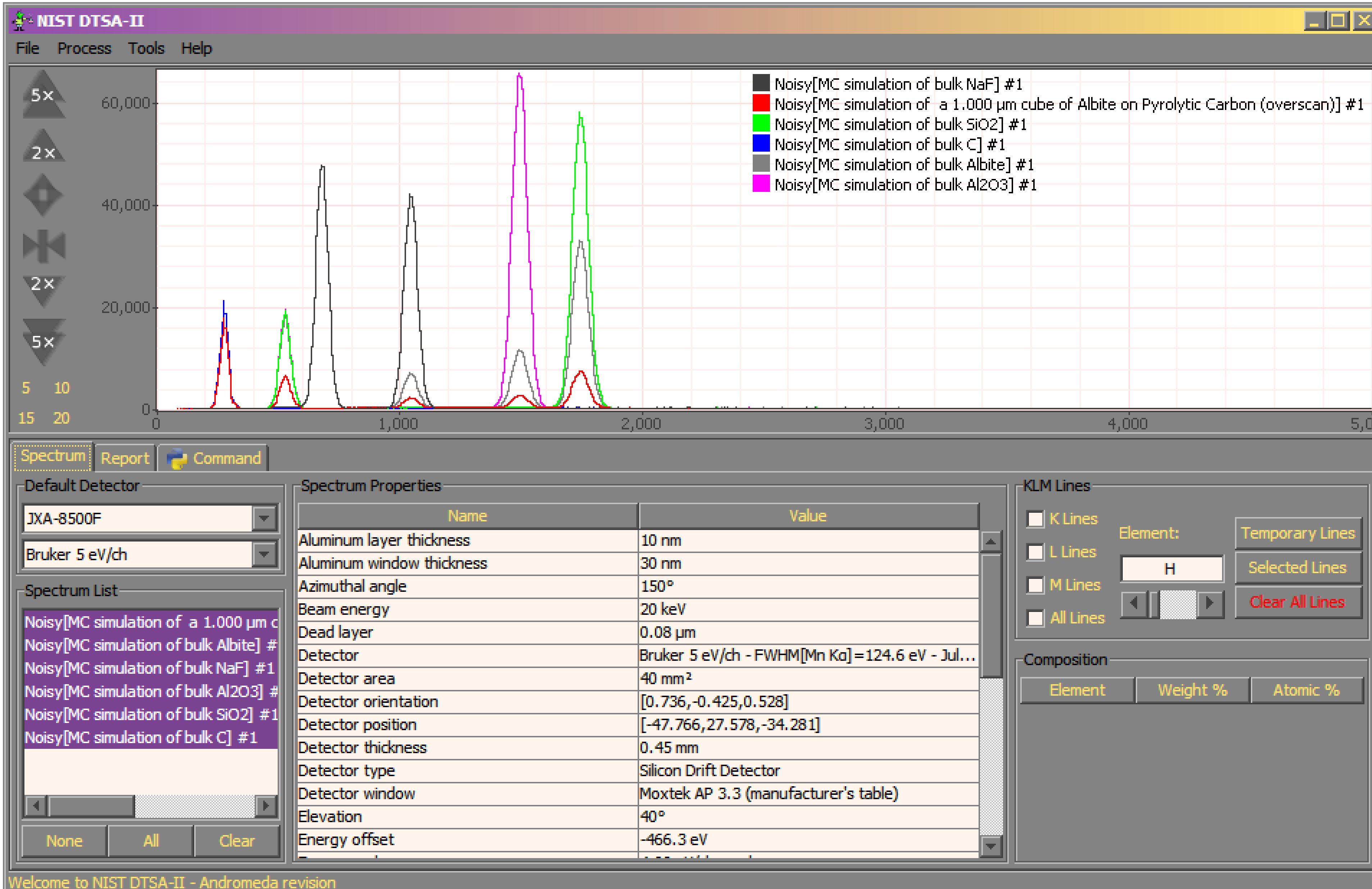

Spectrum simulation
Previous: Simulation Mode
Configure sample
Next: Instrument configuration
Materials and Scale
Substrate material
Pyrolytic Carb
Edit
None
Cube material
Albite
Edit
Cube height / width / depth
1
µm
Overscan particle
Message: Specify the sample material and scale.
More...
Back
Next
Finish
Cancel


Spectrum simulation
Previous: Configure sample
Instrument configuration
Next: Other options
Instrument Parameters
Instrument
JXA-8500F
Detector
Bruker 5 eV/ch
Calibration
FWHM[Mn Kα]=124.6 eV - Jul 29, ...
Beam Energy
20.0
keV
Probe Dose (current·time)
120.0
nA·second
Incident Angle
0.0
°
Message:
More...
Back
Next
Finish
Cancel



Spectrum simulation
Previous: Instrument configuration
Other options
Next: Perform Simulation
Noise parameters
Apply simulated count statistics
Instance count
1
Extended output
X-ray generation images (takes a little longer)
Run the default number of electron trajectories
Message:
More...
Back
Next
Finish
Cancel


Spectrum simulation
Previous: Other options
Perform Simulation
Finish
Progress
Message: Computing the requested spectra...
More...
Back
Next
Finish
Cancel



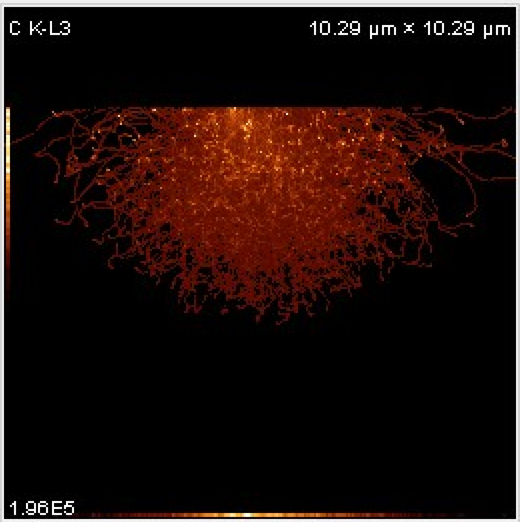
NIST DTSA-II
File
Process
Tools
Help
5×
2×
2×
5×
5
10
15
20
60,000
40,000
20,000
0
0
1,000
2,000
3,000
4,000
Noisy[MC simulation of bulk NaF] #1
Noisy[MC simulation of a 1.000 μm cube of Albite on Pyrolytic Carbon (overscan)] #1
Noisy[MC simulation of bulk SiO2] #1
Noisy[MC simulation of bulk C] #1
Noisy[MC simulation of bulk Albite] #1
Noisy[MC simulation of bulk Al2O3] #1
Spectrum
Report
Command
Default Detector
JXA-8500F
Bruker 5 eV/ch
Spectrum List
Noisy[MC simulation of a 1.000 μm c
Noisy[MC simulation of bulk Albite] #
Noisy[MC simulation of bulk NaF] #1
Noisy[MC simulation of bulk Al2O3] #
Noisy[MC simulation of bulk SiO2] #1
Noisy[MC simulation of bulk C] #1
None
All
Clear
Spectrum Properties
Name
Value
Aluminum layer thickness
10 nm
Aluminum window thickness
30 nm
Azimuthal angle
150°
Beam energy
20 keV
Dead layer
0.08 μm
Detector
Bruker 5 eV/ch - FWHM[Mn Kα]=124.6 eV - Jul...
Detector area
40 mm²
Detector orientation
[0.736,-0.425,0.528]
Detector position
[-47.766,27.578,-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
-466.3 eV
KLM Lines
K Lines
L Lines
M Lines
All Lines
Element:
H
Temporary Lines
Selected Lines
Clear All Lines
Composition
Element
Weight %
Atomic %
Welcome to NIST DTSA-II - Andromeda revision

Spectrum Simulation

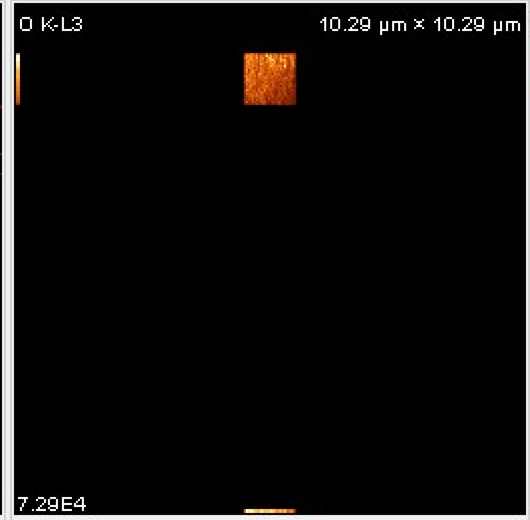
Simulation mode	Monte Carlo model of a cube sample
Substrate material	Pyrolytic Carbon = [C(100.00 wgt%),2.1 g/cc]
Object Material	Albite = [O(48.81 wgt%),Na(8.77 wgt%),Al(10.29 wgt%),Si(32.13 wgt%),2.6 g/cc]
Edge length	1.000 µm
Beam energy	20.000 keV
Probe dose	120.000 nA·s
Instrument	JXA-8500F
Detector	Bruker 5 eV/ch
Calibration	FWHM[Mn Kα]=124.6 eV - Jul 29, 2008 12:00:01 AM
Overscan	true
Replicas (with Poisson noise)	1
Result 1	Noisy[MC simulation of a 1.000 µm cube of Albite on Pyrolytic Carbon (overscan)] #1
Trajectory view	C:\Documents and Settings\nritchie.SMSD0\My Documents\DTSA-II Reports\2009\April\15-Apr-2009\vrml4008355382166422620.wrl
Intensity data	(see table below)

Transition	Generated 1/msR	Emitted 1/msR	Ratio (%)
C K-L3	293014.8	105491.0	36.0%
O K-L3	25961.8	17610.1	67.8%
Na K-L3	4668.2	3589.5	76.9%
Al K-L3	5081.4	4457.8	87.7%
Si K-L3	15471.5	13724.4	88.7%

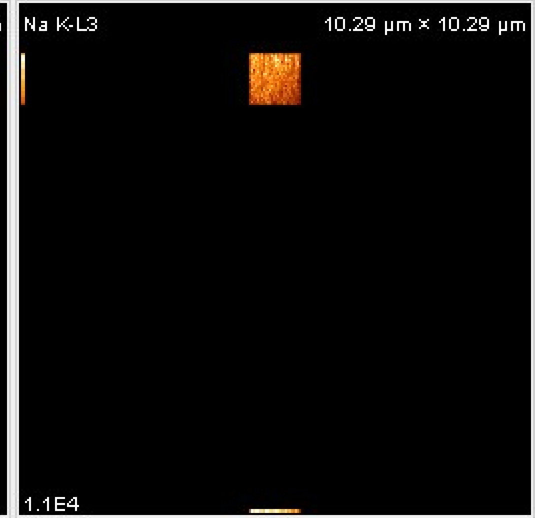
Emission Images



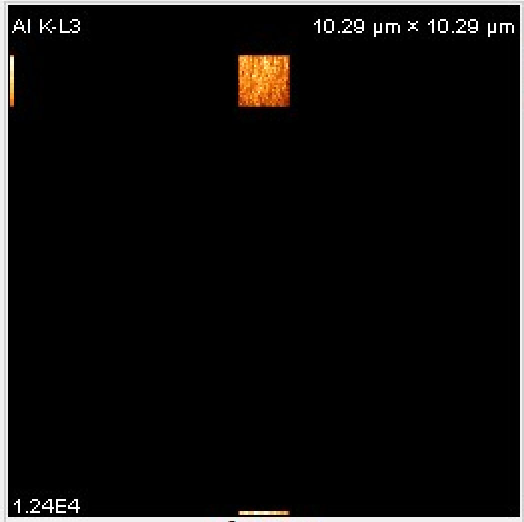
C K-L3



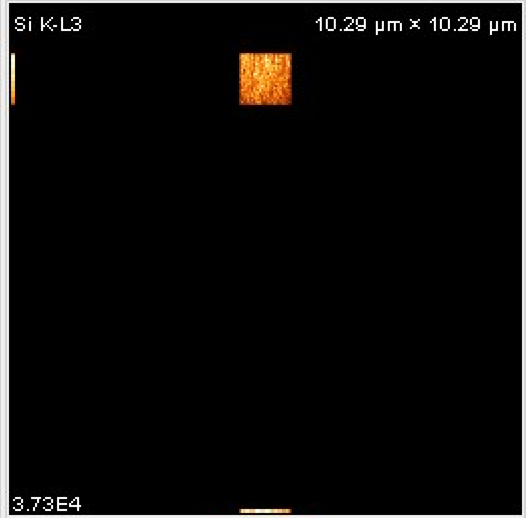
O K-L3



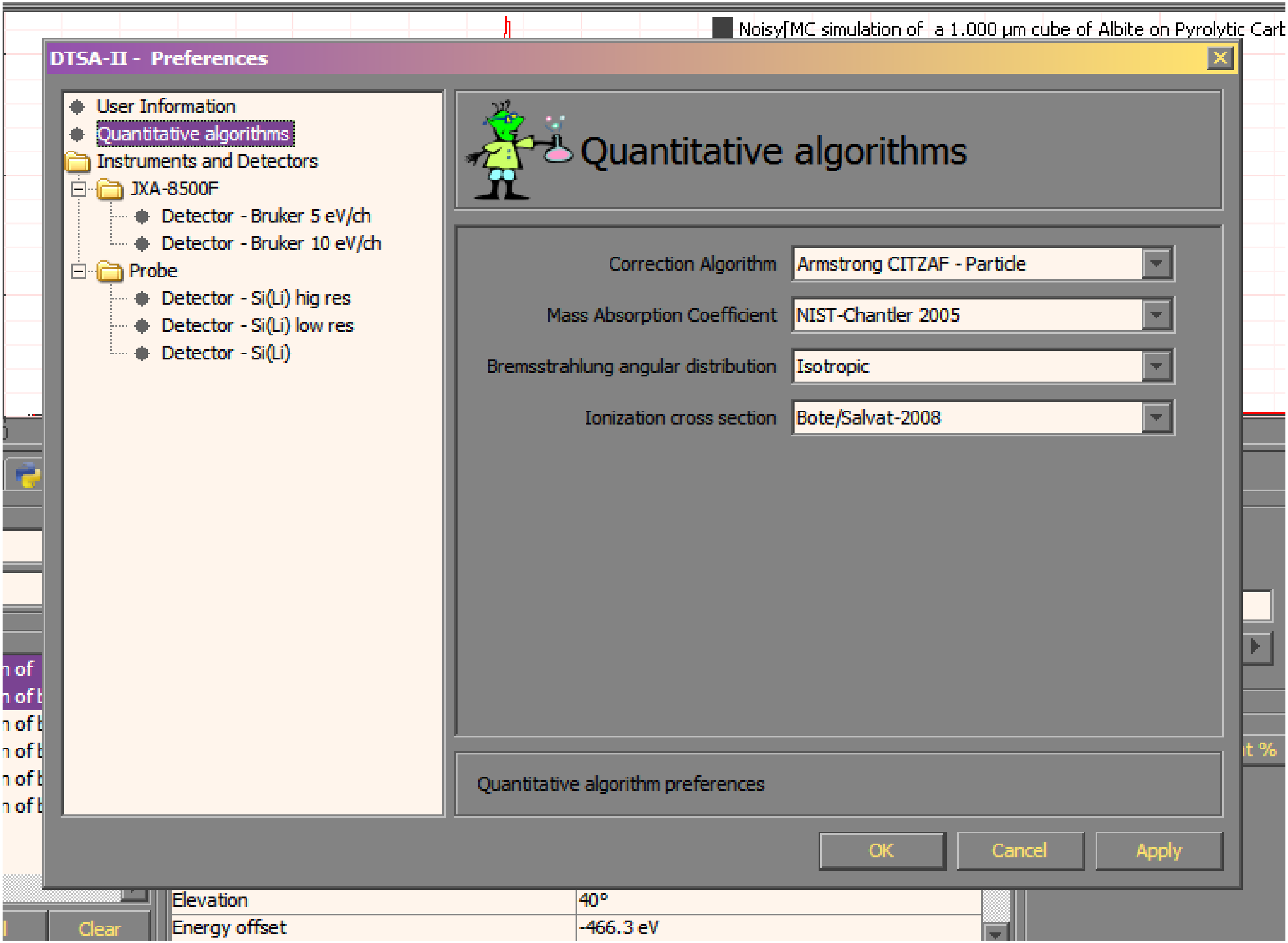
Na K-L3



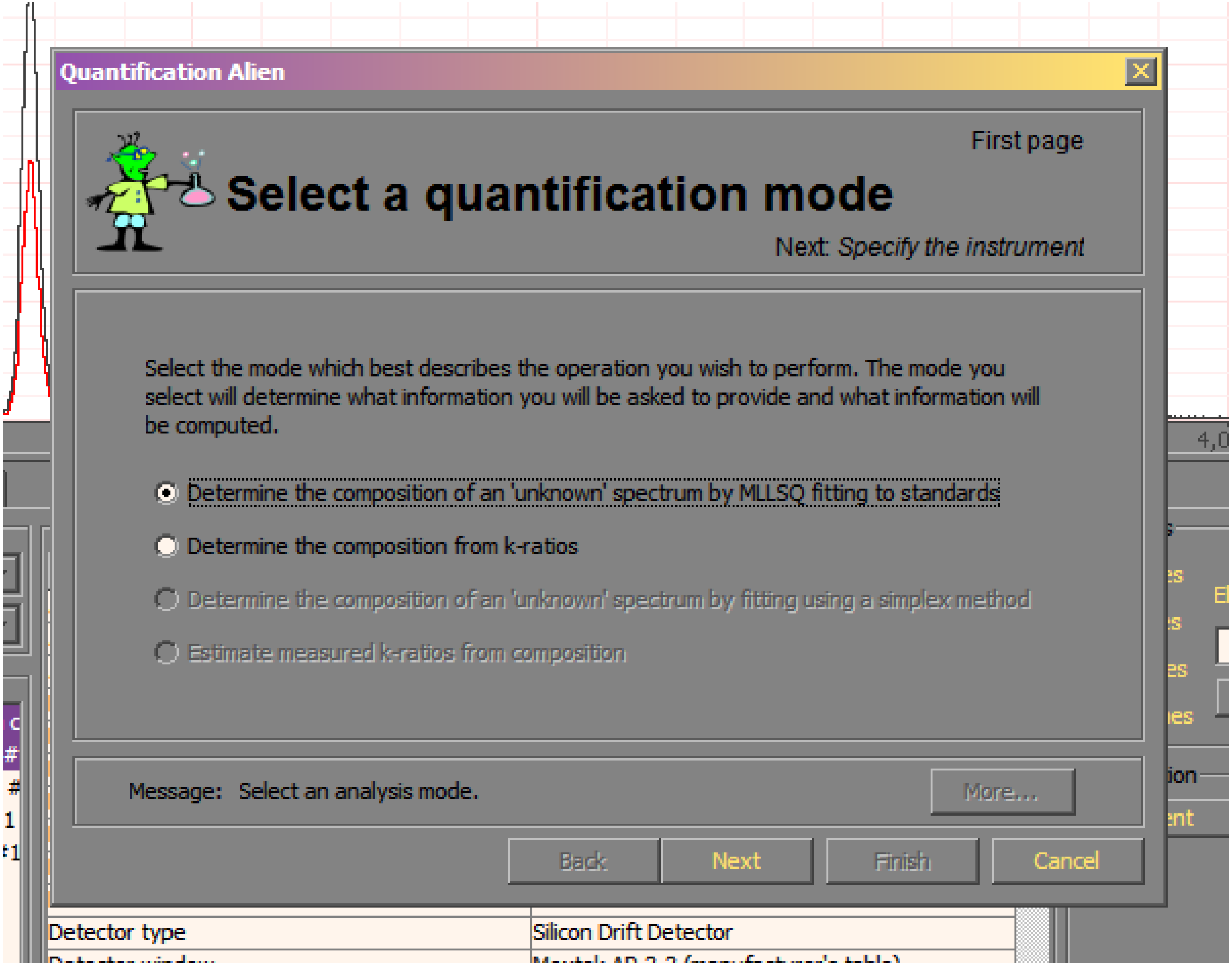
Al K-L3

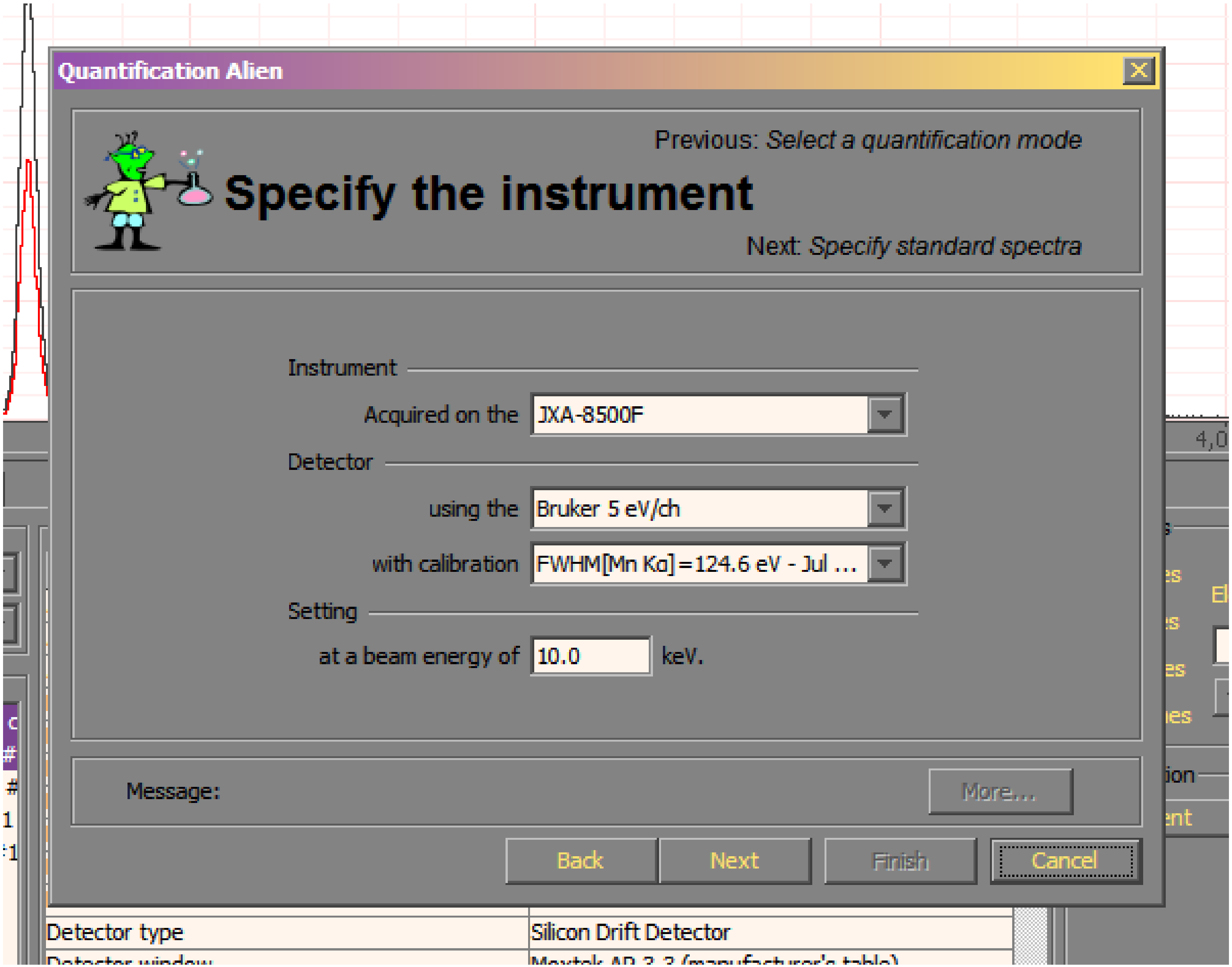


Si K-L3

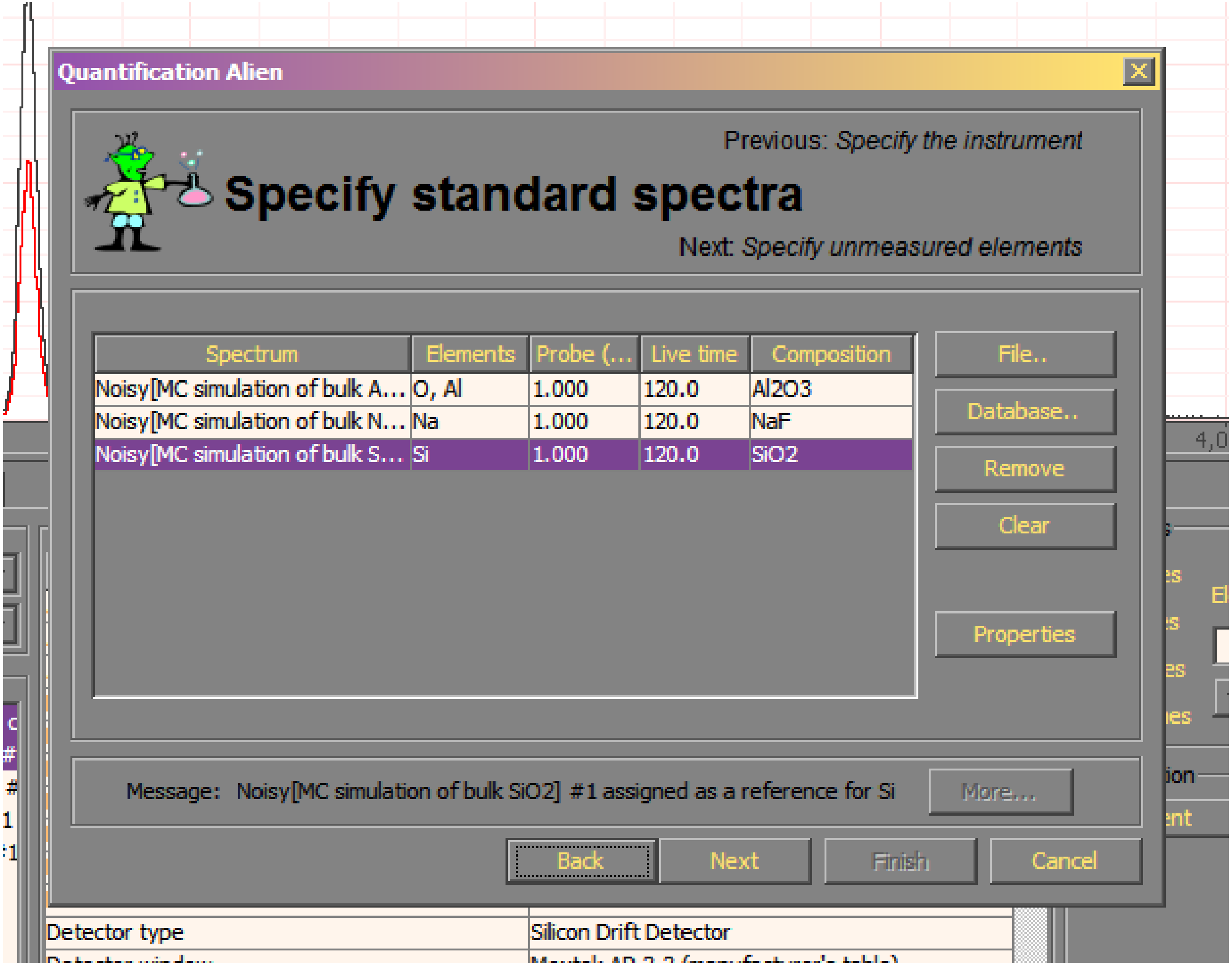


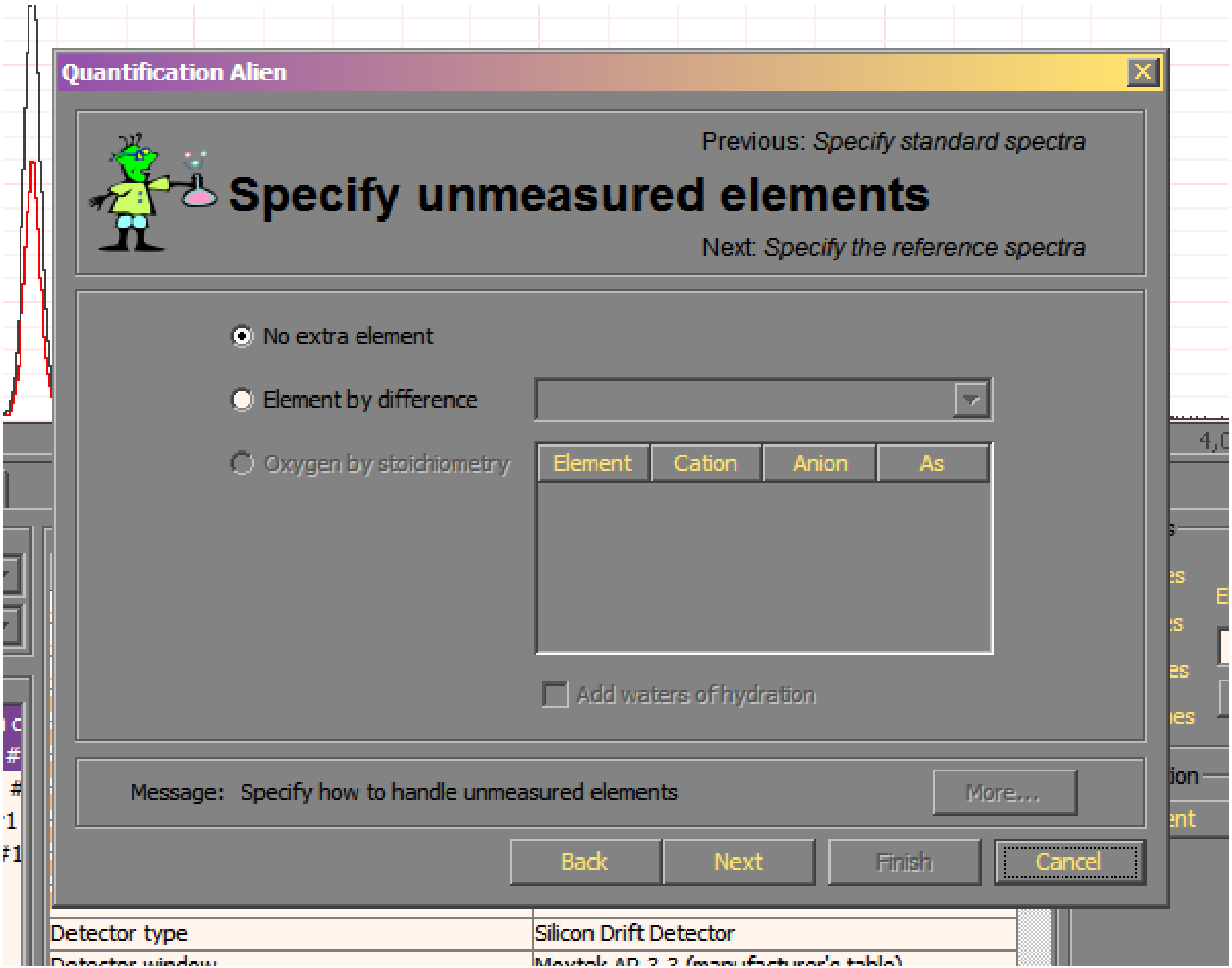
Noisy[MC simulation of a 1.000 µm cube of Albite on Pyrolytic Cart
DTSA-II - Preferences
User Information
Quantitative algorithms
Instruments and Detectors
JXA-8500F
Detector - Bruker 5 eV/ch
Detector - Bruker 10 eV/ch
Probe
Detector - Si(Li) hig res
Detector - Si(Li) low res
Detector - Si(Li)
Quantitative algorithms
Correction Algorithm
Armstrong CITZAF - Particle
Mass Absorption Coefficient
NIST-Chantler 2005
Bremsstrahlung angular distribution
Isotropic
Ionization cross section
Bote/Salvat-2008
Quantitative algorithm preferences
OK
Cancel
Apply
Clear
Elevation
40°
Energy offset
-466.3 eV

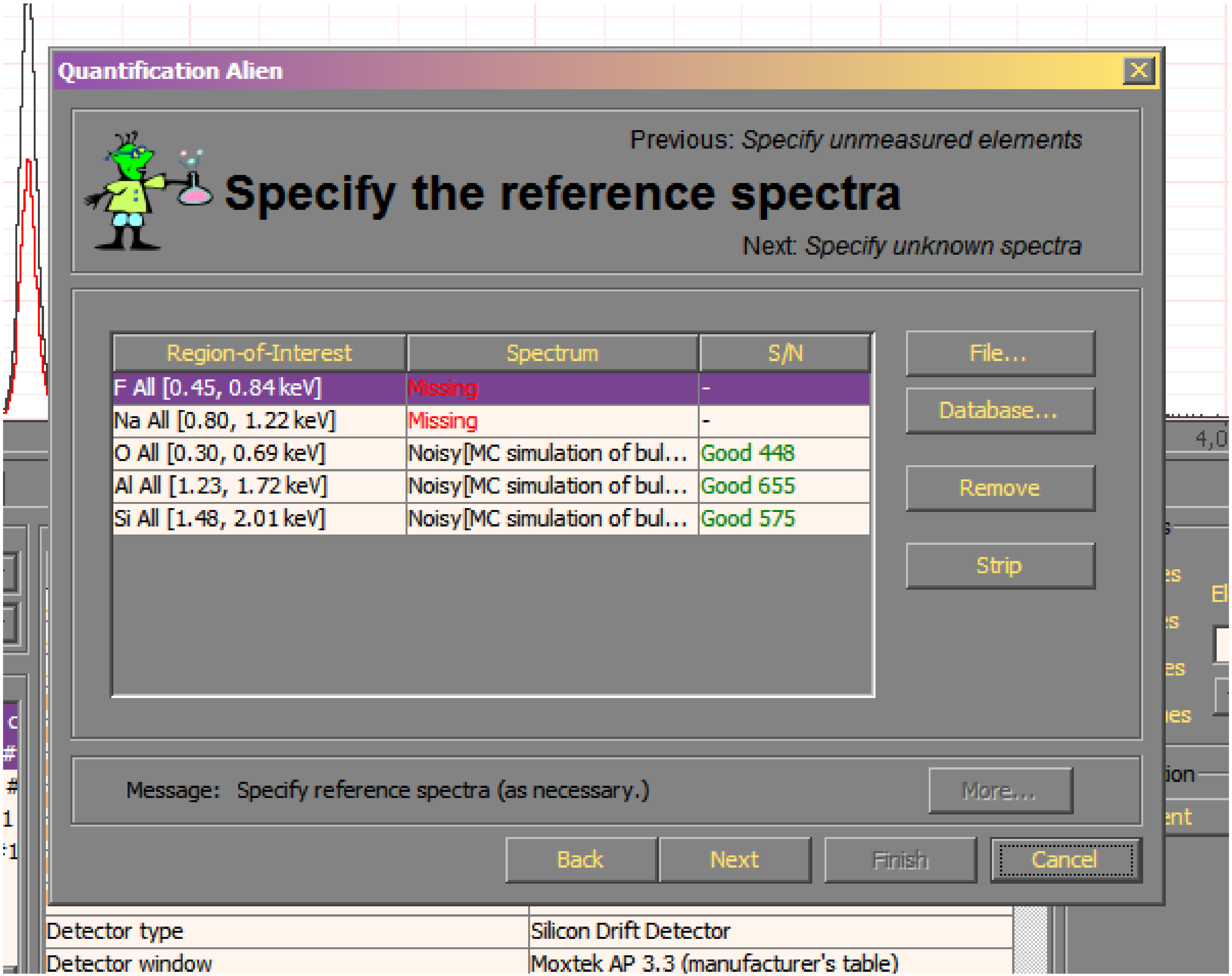

Quantification Alien
First page
Select a quantification mode
Next: Specify the instrument
Select the mode which best describes the operation you wish to perform. The mode you select will determine what information you will be asked to provide and what information will be computed.
Determine the composition 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

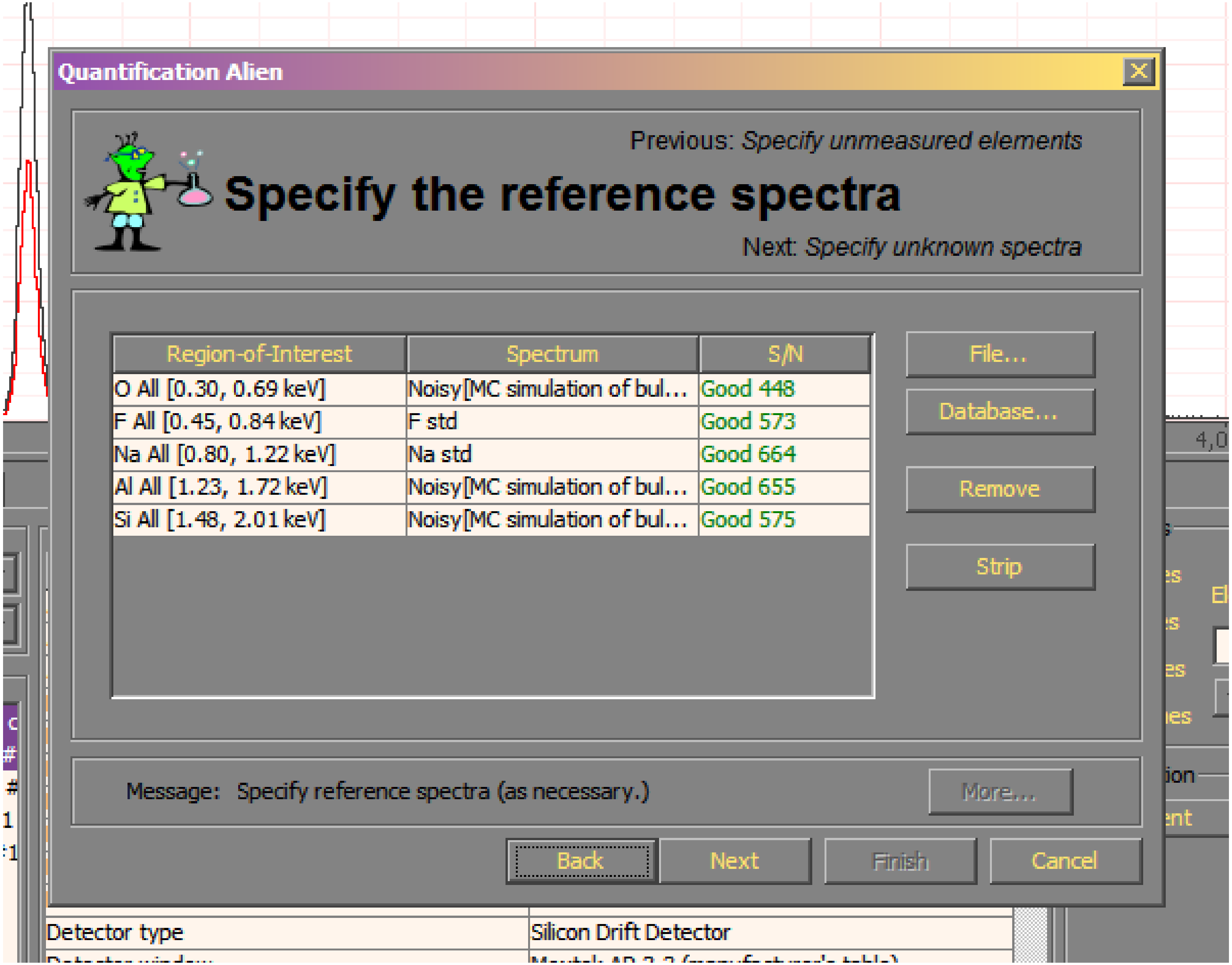


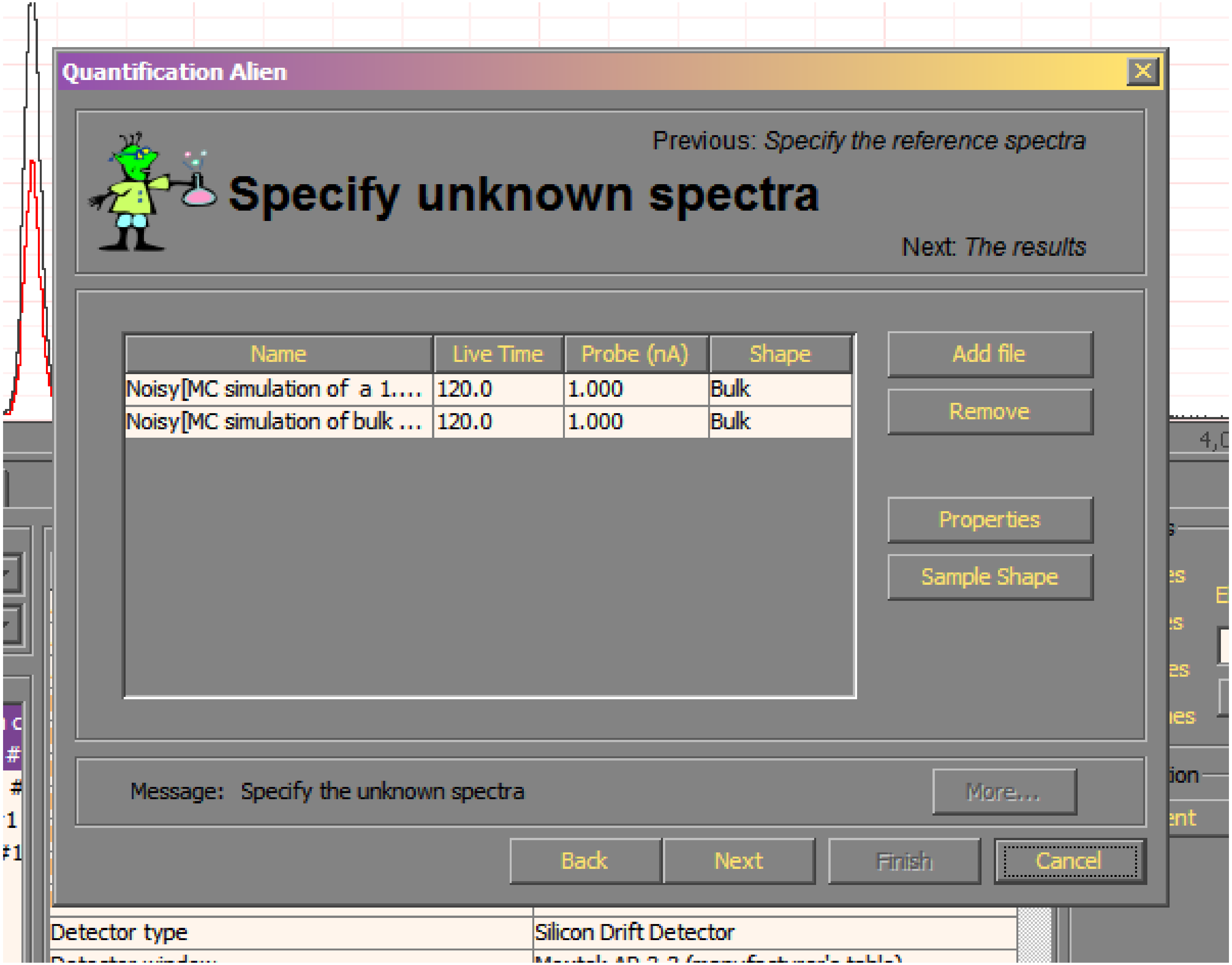
Quantification Alien
Previous: Select a quantification mode
Specify the instrument
Next: Specify standard spectra
Instrument
Acquired on the
JXA-8500F
Detector
using the
Bruker 5 eV/ch
with calibration
FWHM[Mn Kα]=124.6 eV - Jul ...
Setting
at a beam energy of
10.0
keV.
Message:
More...
Back
Next
Finish
Cancel
Detector type
Silicon Drift Detector

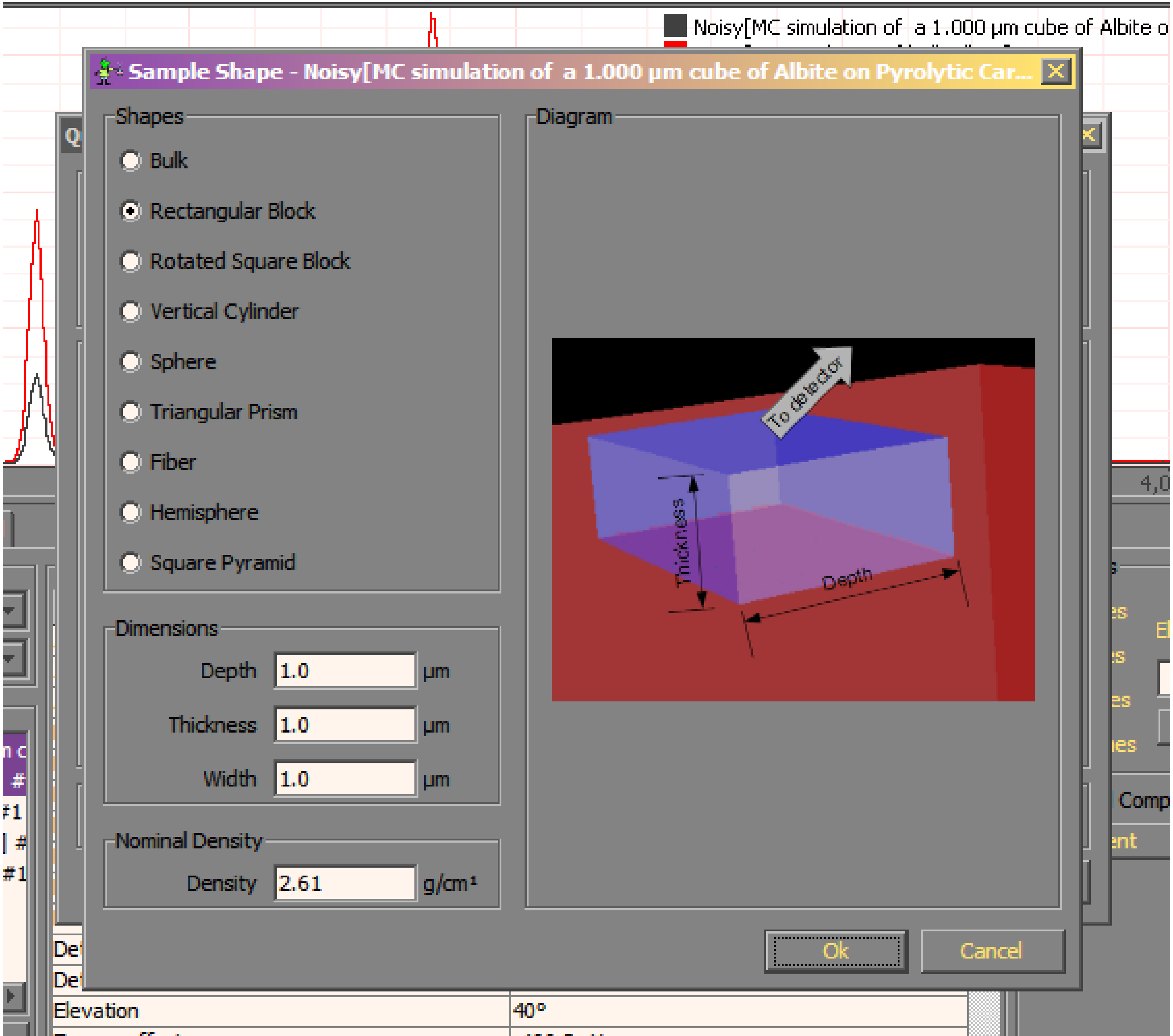

Quantification Alien
Previous: Specify the instrument
Specify standard spectra
Next: Specify unmeasured elements
Spectrum
Elements
Probe (...
Live time
Composition
Noisy[MC simulation of bulk A... O, Al 1.000 120.0 Al2O3
Noisy[MC simulation of bulk N... Na 1.000 120.0 NaF
Noisy[MC simulation of bulk S... Si 1.000 120.0 SiO2
File..
Database..
Remove
Clear
Properties
Message: Noisy[MC simulation of bulk SiO2] #1 assigned as a reference for Si
More...
Back
Next
Finish
Cancel
Detector type
Silicon Drift Detector

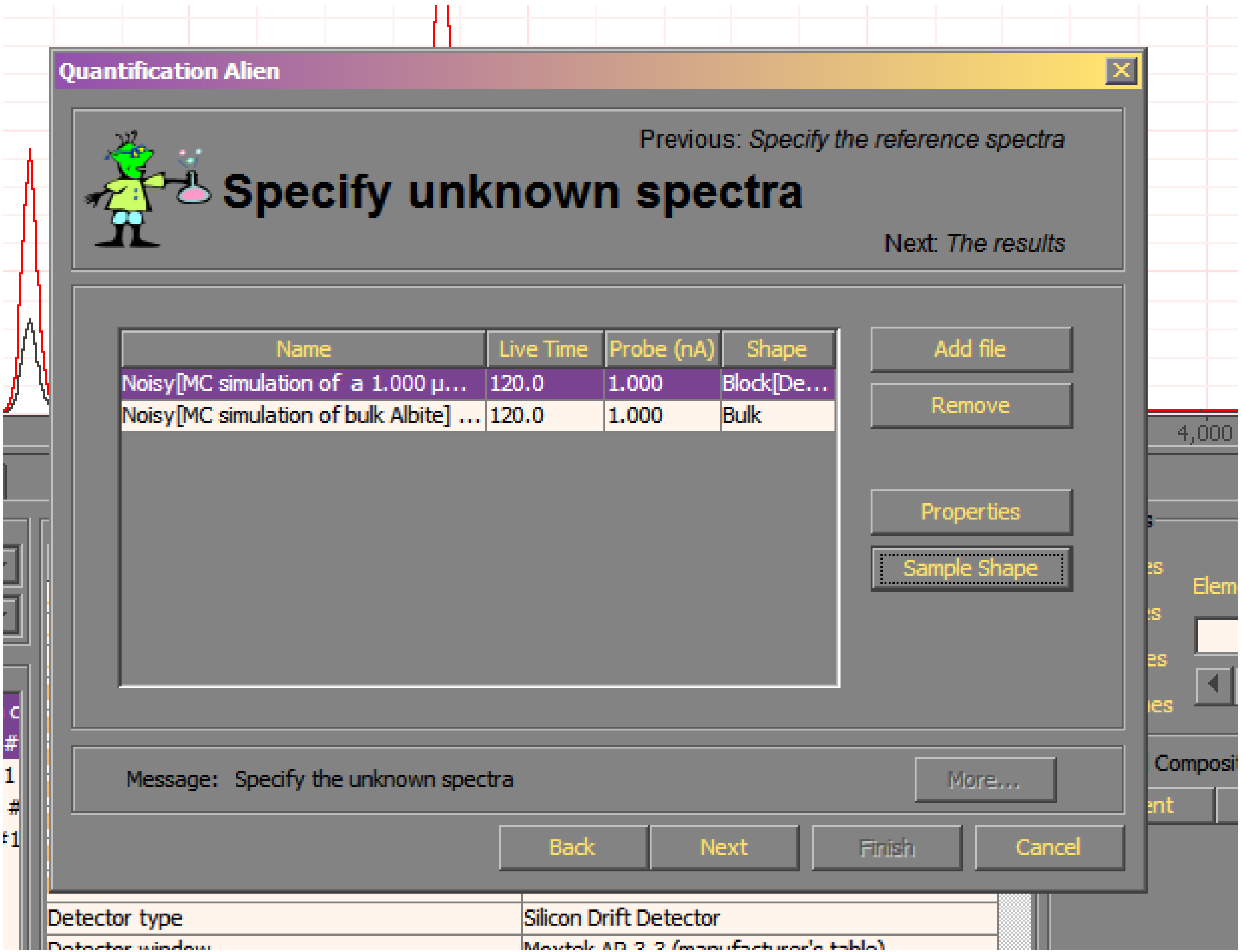

Quantification Alien
Previous: Specify standard spectra
Specify unmeasured elements
Next: Specify the reference spectra
No extra element
Element by difference
Oxygen by stoichiometry
Element
Cation
Anion
As
Add waters of hydration
Message: Specify how to handle unmeasured elements
More...
Back
Next
Finish
Cancel
Detector type
Silicon Drift Detector

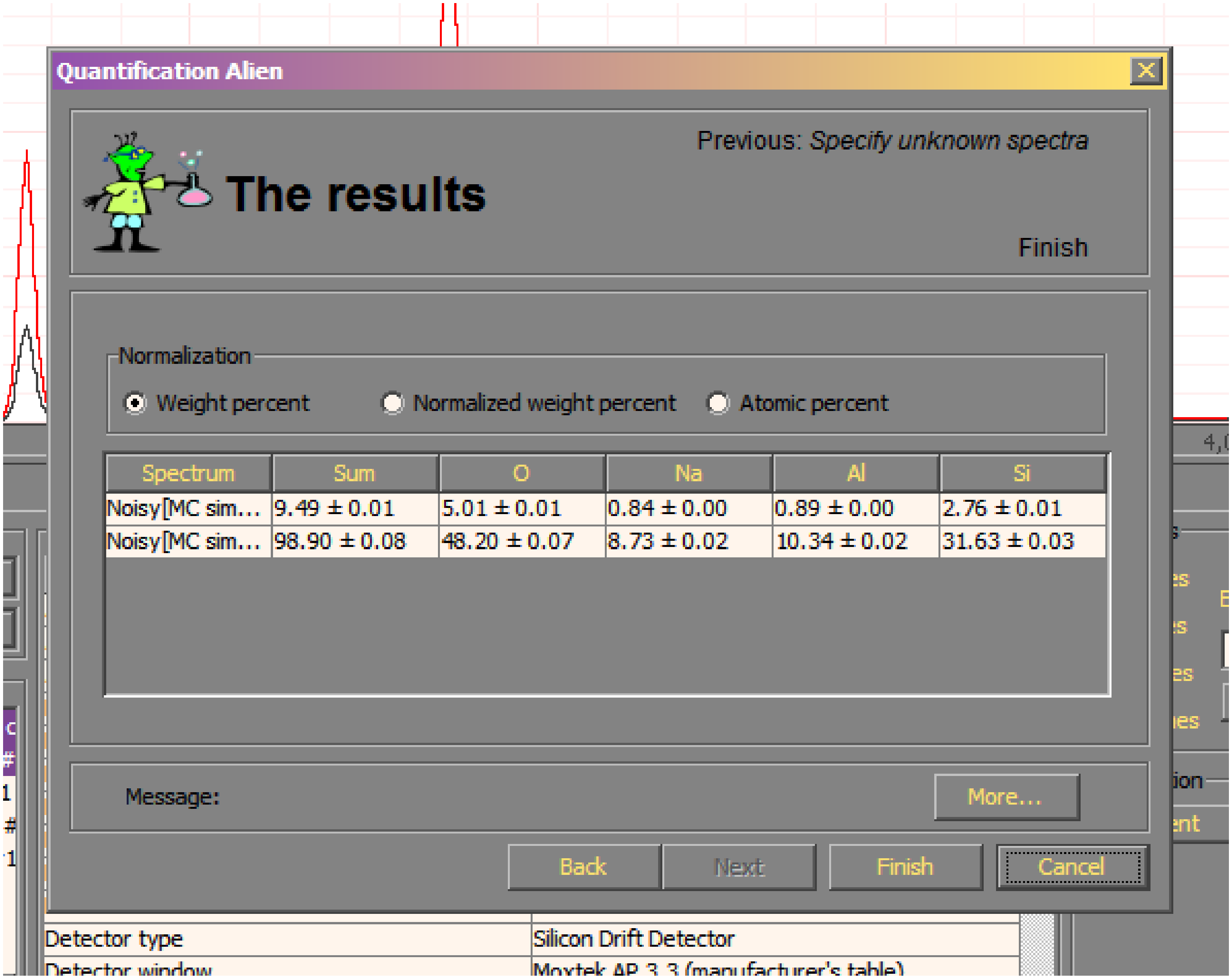

Quantification Alien
Previous: Specify unmeasured elements
Specify the reference spectra
Next: Specify unknown spectra
Region-of-Interest
Spectrum
S/N
F All [0.45, 0.84 keV]
Missing
-
Na All [0.80, 1.22 keV]
Missing
-
O All [0.30, 0.69 keV]
Noisy[MC simulation of bul...
Good 448
Al All [1.23, 1.72 keV]
Noisy[MC simulation of bul...
Good 655
Si All [1.48, 2.01 keV]
Noisy[MC simulation of bul...
Good 575
File...
Database...
Remove
Strip
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)

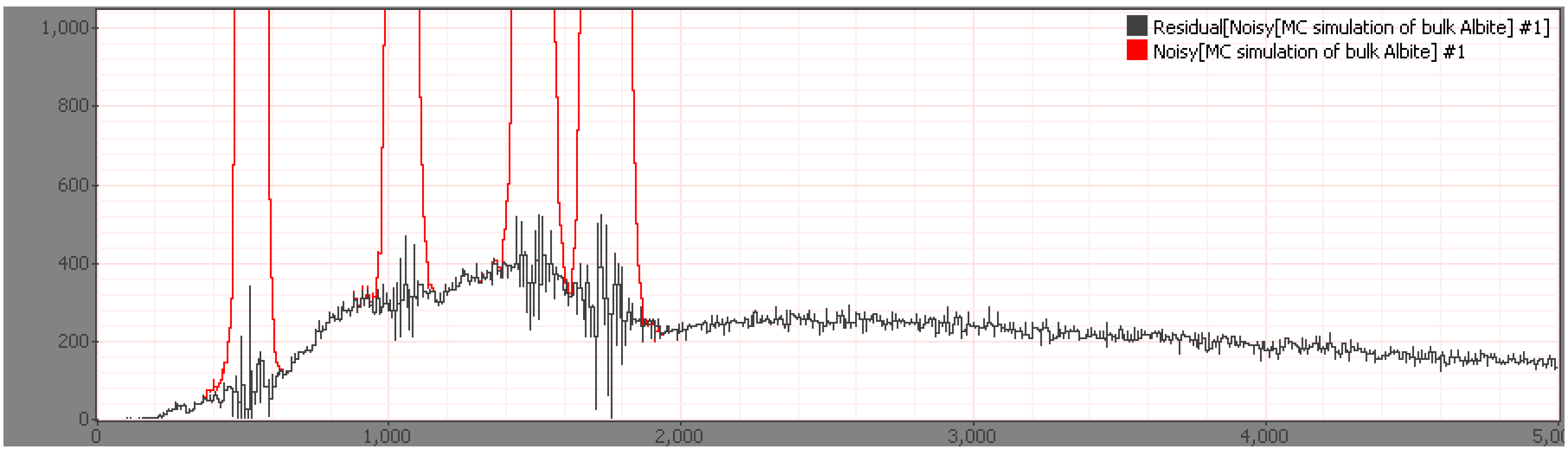

Quantification Alien
Previous: Specify unmeasured elements
Specify the reference spectra
Next: Specify unknown spectra
Region-of-Interest
Spectrum
S/N
O All [0.30, 0.69 keV]
Noisy[MC simulation of bul...
Good 448
F All [0.45, 0.84 keV]
F std
Good 573
Na All [0.80, 1.22 keV]
Na std
Good 664
Al All [1.23, 1.72 keV]
Noisy[MC simulation of bul...
Good 655
Si All [1.48, 2.01 keV]
Noisy[MC simulation of bul...
Good 575
File...
Database...
Remove
Strip
Message: Specify reference spectra (as necessary.)
More...
Back
Next
Finish
Cancel
Detector type
Silicon Drift Detector


Quantification Alien
Previous: Specify the reference spectra
Specify unknown spectra
Next: The results
Name
Live Time
Probe (nA)
Shape
Noisy[MC simulation of a 1....
120.0
1.000
Bulk
Noisy[MC simulation of bulk ...
120.0
1.000
Bulk
Add file
Remove
Properties
Sample Shape
Message: Specify the unknown spectra
More...
Back
Next
Finish
Cancel
Detector type
Silicon Drift Detector


Noisy[MC simulation of a 1.000 µm cube of Albite o
Sample Shape - Noisy[MC simulation of a 1.000 µm cube of Albite on Pyrolytic Car...
Shapes
Bulk
Rectangular Block
Rotated Square Block
Vertical Cylinder
Sphere
Triangular Prism
Fiber
Hemisphere
Square Pyramid
Dimensions
Depth
1.0
µm
Thickness
1.0
µm
Width
1.0
µm
Nominal Density
Density
2.61
g/cm³
Diagram
To detector
Thickness
Depth
Ok
Cancel
Elevation
40°
Energy offset
-466.3 eV


Quantification Alien
Previous: Specify the reference spectra
Specify unknown spectra
Next: The results
Name
Live Time
Probe (nA)
Shape
Noisy[MC simulation of a 1.000 µ... 120.0 1.000 Block[De...
Noisy[MC simulation of bulk Albite] ... 120.0 1.000 Bulk
Add file
Remove
Properties
Sample Shape
Message: Specify the unknown spectra
More...
Back
Next
Finish
Cancel
4,000
Detector type
Silicon Drift Detector


Quantification Alien
Previous: Specify unknown spectra
The results
Finish
Normalization
Weight percent
Normalized weight percent
Atomic percent
Spectrum
Sum
O
Na
Al
Si
Noisy[MC sim… 9.49 ± 0.01 5.01 ± 0.01 0.84 ± 0.00 0.89 ± 0.00 2.76 ± 0.01
Noisy[MC sim… 98.90 ± 0.08 48.20 ± 0.07 8.73 ± 0.02 10.34 ± 0.02 31.63 ± 0.03
Message:
More…
Back
Next
Finish
Cancel
Detector type
Silicon Drift Detector
Detector window



Residual[Noisy[MC simulation of bulk Albite] #1]
Noisy[MC simulation of bulk Albite] #1
1,000
800
600
400
200
0
0
1,000
2,000
3,000
4,000
5,00

Conditions

Item	Value
Instrument	JXA-8500F
Detector	Bruker 5 eV/ch
Beam Energy	10.0 keV
Correction Algorithm	Armstrong CITZAF - Particle
Mass Absorption Coefficient	NIST-Chantler 2005

Standards

Element	Material	Spectrum	Ref?	Probe (nA)	Live Time (s)
O	Al2O3 = [O(0.6 atoms),Al(0.4 atoms)]	Bulk Al2O3	Yes	1.000	120.0
Na	NaCl = [Na(0.5 atoms),Cl(0.5 atoms)]	Bulk NaCl	Yes	1.000	120.0
Al	Al2O3 = [O(0.6 atoms),Al(0.4 atoms)]	Bulk Al2O3	Yes	1.000	120.0
Si	SiO2 = [O(0.67 atoms),Si(0.33 atoms)]	Bulk SiO2	Yes	1.000	120.0

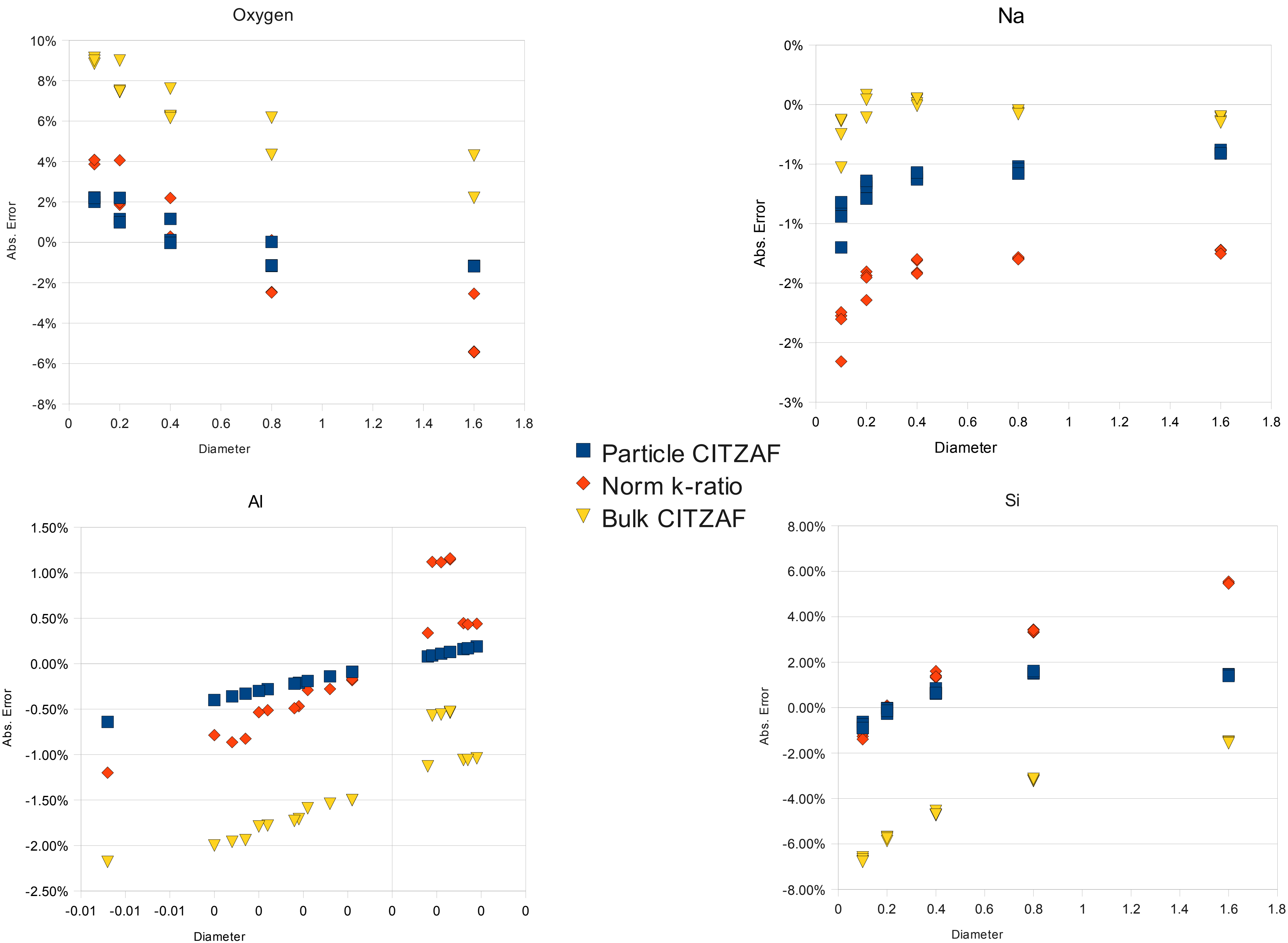
Results

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

Spectrum	Quantity	O			Na			Al			Si			Sum
Noisy[MC simulation of a 1.000 µm cube of Albite on C (overscan)] #1 **Block[Depth=1.0 µm,Width = 1.0 µm,Height=1.0 µm]**	Line	O All			Na All			Al All			Si All			
	Z · A · F	0.99	1.12	1.00	0.89	1.03	1.00	0.99	1.00	1.00	1.01	0.97	1.00	
	k-ratios	0.5510	±	0.0011	0.0950	±	0.0004	0.0923	±	0.0004	0.3263	±	0.0007	
	weight %	23.34	±	0.04	4.07	±	0.02	4.95	±	0.02	15.56	±	0.03	47.92
I = 1.000 nA	norm(wgt %)	**48.71**	±	0.09	**8.49**	±	0.03	**10.33**	±	0.04	**32.47**	±	0.07	-
LT = 120.0 s	atomic %	61.47			7.46			7.73			23.34			
Residual	/home/nicholas/DTSA-II Reports/2009/April/18-Apr-2009/residual8001149227620179364.msa													
Noisy[MC simulation of bulk Albite] #1 **Bulk**	Line	O All			Na All			Al All			Si All			
	Z · A · F	0.99	0.97	1.00	0.89	0.94	1.00	0.99	0.96	1.00	1.01	0.94	1.00	
	k-ratios	1.0058	±	0.0014	0.1848	±	0.0005	0.1876	±	0.0005	0.6526	±	0.0010	
	weight %	**49.38**	±	0.07	**8.69**	±	0.02	**10.44**	±	0.03	**32.17**	±	0.05	100.68
I = 1.000 nA	norm(wgt %)	49.05	±	0.07	8.63	±	0.02	10.37	±	0.03	31.95	±	0.05	-
LT = 120.0 s	atomic %	61.77			7.56			7.74			22.92			
Residual	/home/nicholas/DTSA-II Reports/2009/April/18-Apr-2009/residual5040019642226267730.msa													

Practical example

- Albite spheres – $NaAlSi_3O_8$
- Diameters = 0.1, 0.2, 0.4, 0.8, 1.6 µm
- Substrate – Carbon
- Procedure
  - Simulate using Monte Carlo
  - Quantify using bulk ZAF, normalized k-ratios, CITZAF particle ZAF
  - Compare


Oxygen
10%
8%
6%
4%
2%
0%
-2%
-4%
-6%
-8%
Abs. Error
0
0.2
0.4
0.6
0.8
1
1.2
1.4
1.6
1.8
Diameter
Na
0%
0%
-1%
-1%
-2%
-2%
-3%
Abs. Error
0
0.2
0.4
0.6
0.8
1
1.2
1.4
1.6
1.8
Diameter
Particle CITZAF
Norm k-ratio
Bulk CITZAF
Al
1.50%
1.00%
0.50%
0.00%
-0.50%
-1.00%
-1.50%
-2.00%
-2.50%
Abs. Error
-0.01
-0.01
-0.01
0
0
0
0
0
0
0
0
Diameter
Si
8.00%
6.00%
4.00%
2.00%
0.00%
-2.00%
-4.00%
-6.00%
-8.00%
Abs. Error
0
0.2
0.4
0.6
0.8
1
1.2
1.4
1.6
1.8
Diameter

http://nist.gov/dtsa