Latest advances in identifying mineral composition variation by the M4 TORNADO AMICS

Bruker Nano Analytics, Berlin, Germany
Webinar, June 15, 2017

Presenters

Samuel Scheller

Sr. Product Manager, Micro-XRF & Automated Mineralogy
Bruker Nano Analytics, Berlin, Germany

Gerda Gloy

Application Specialist, Natural Resources
Bruker Pty Ltd, Brisbane, Australia

Overview

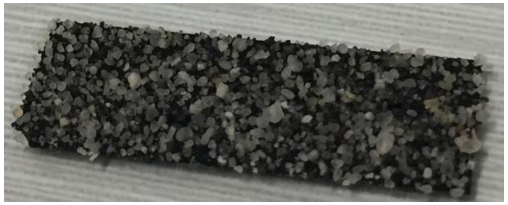
- Introduction to Micro-XRF
- What is AMICS
- Synthetic Spectra
- Spectrum Tree
- Demonstration
- Conclusion
- Questions

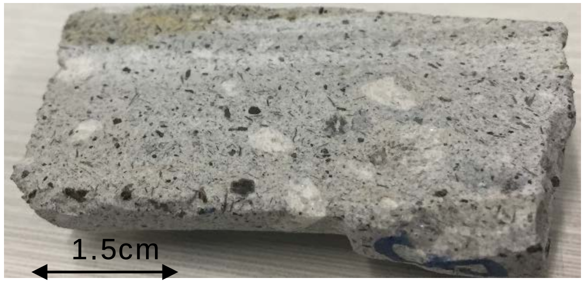
The M4 Tornado
Main advantages

- Rectangular chamber design which accommodates large samples of up to 200 x 160 x 120 mm(WxDxH)
- Pump down <2min allowing detection to Na
- Three cameras assist with sample view and positioning
- Fast 100 mm/s stage with and 4 µm resolution, mouse-controlled and autofocus
- Capillary optics < 20 µm spot size at Mo Kα and high excitation intensity
- Dual SDD in 30 or 60 mm^2 with < 145 eV @ Mn Kα

The M4 Tornado
Main advantages

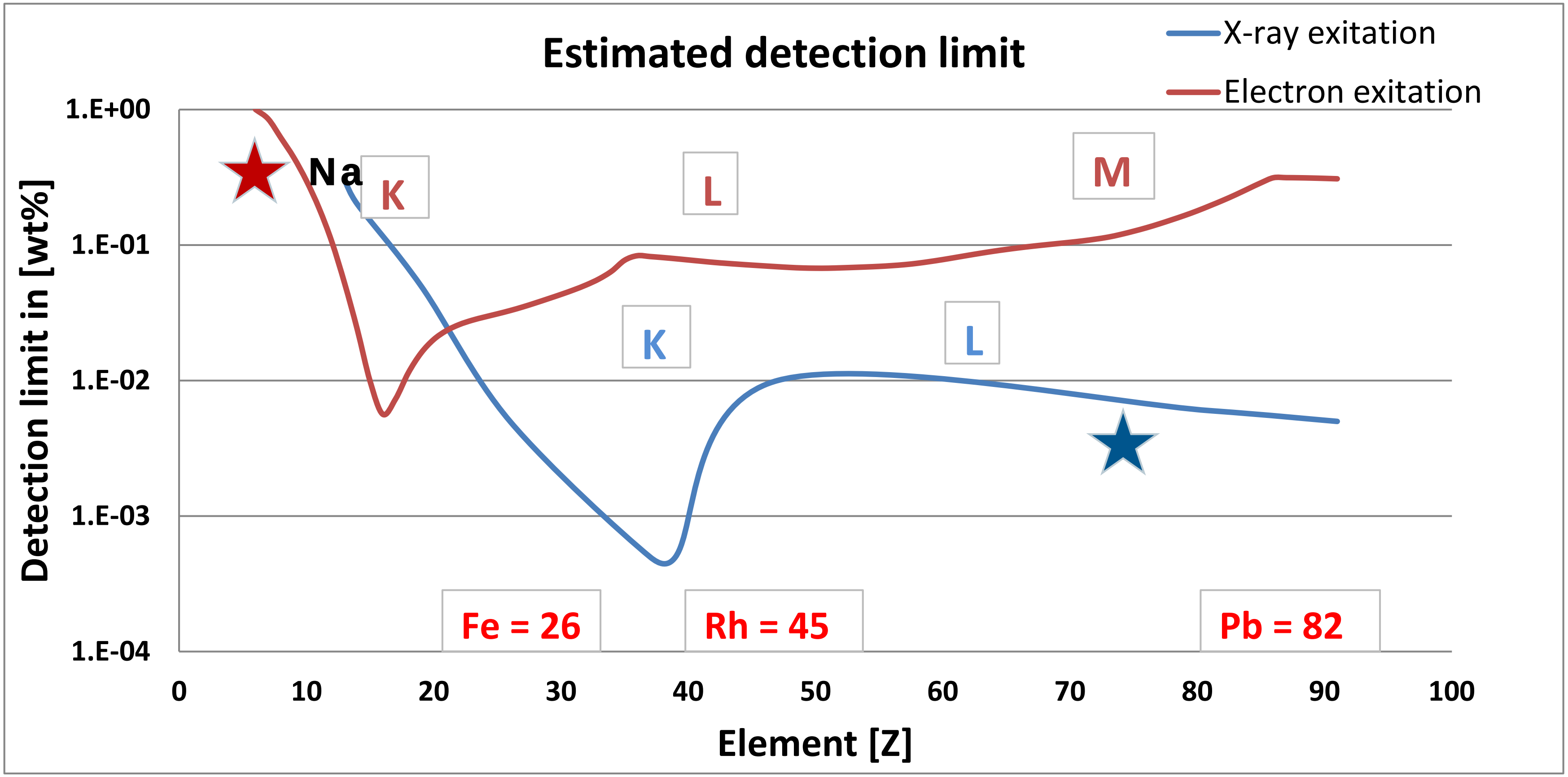
- Little/no sample preparation





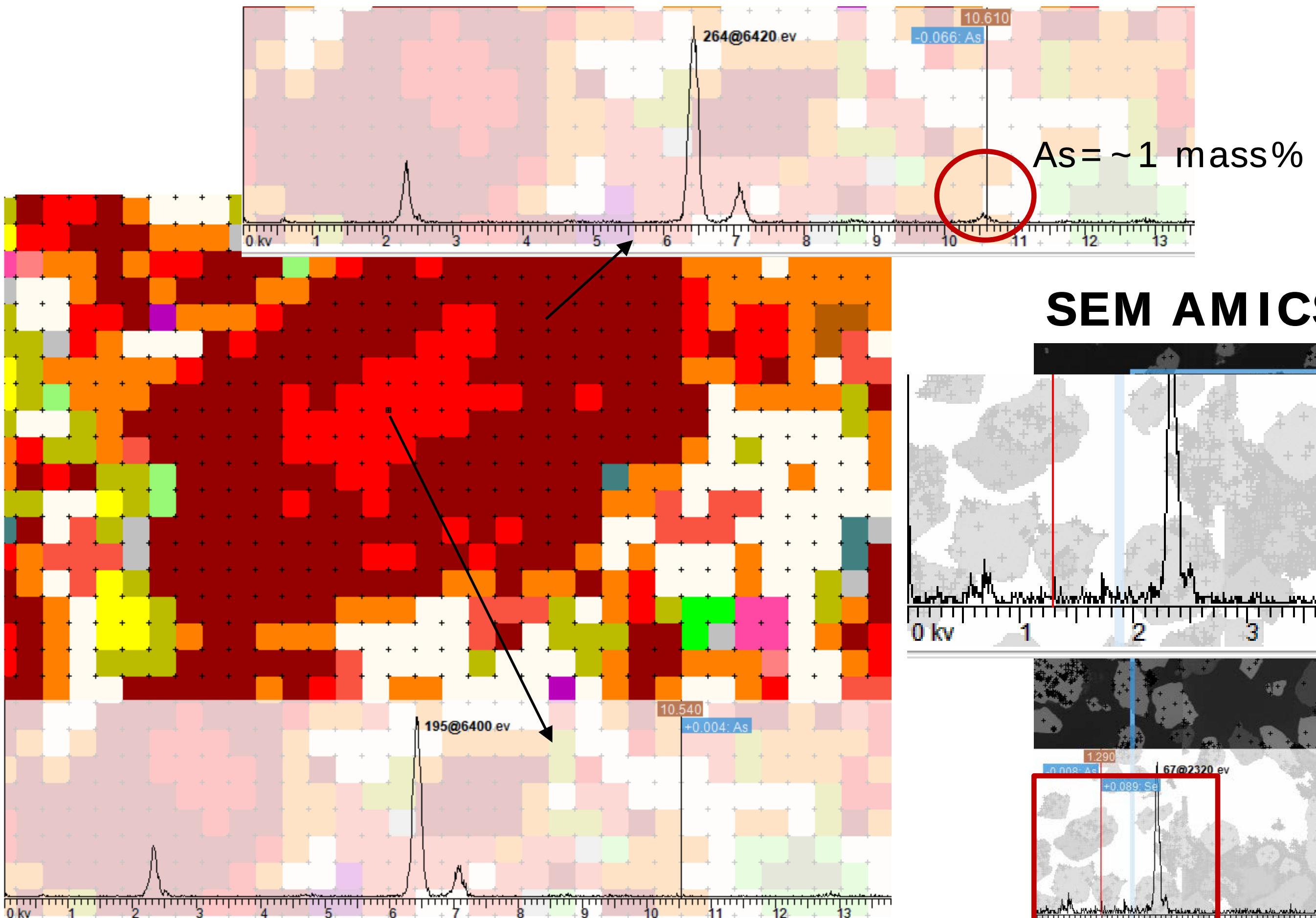
Estimated detection limits

Electron excitation vs. photon excitation

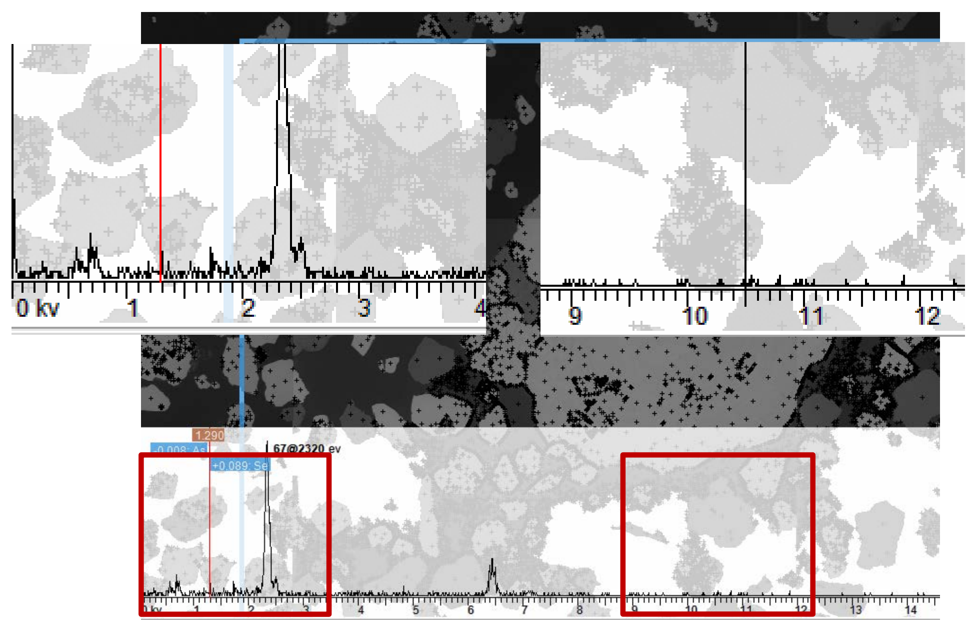


Estimated detection limits
Electron excitation vs. photon excitation

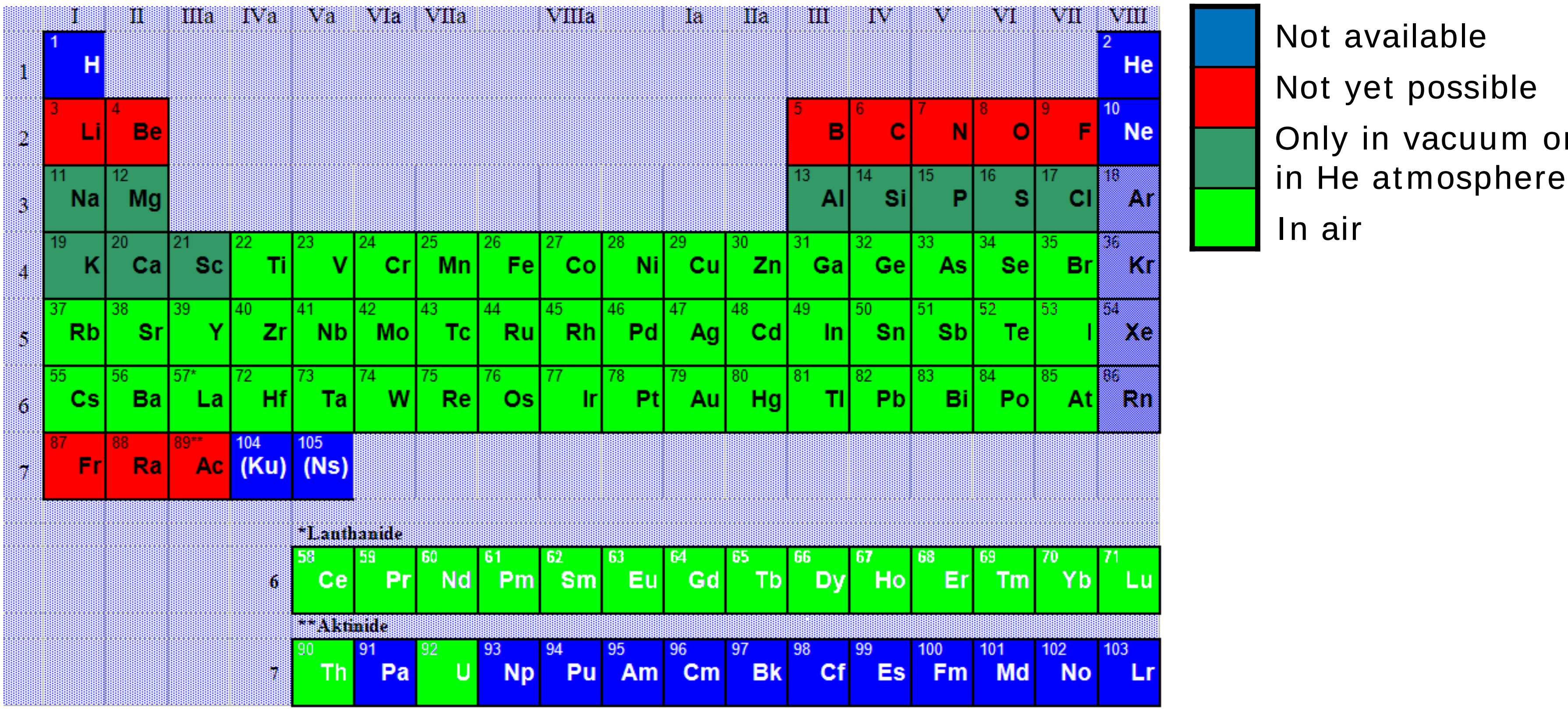
M4Tornado AMICS



SEM AMICS

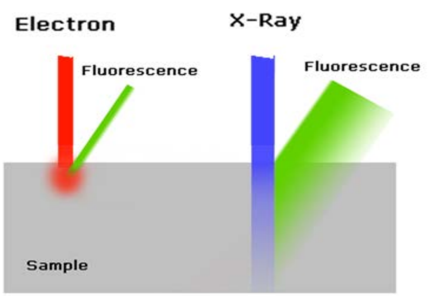


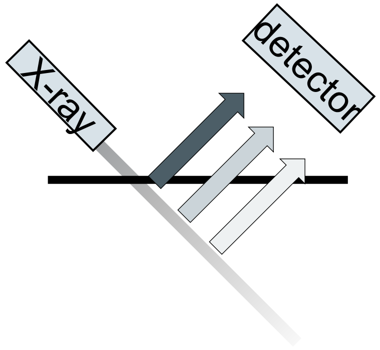
Element detection M4
Overview

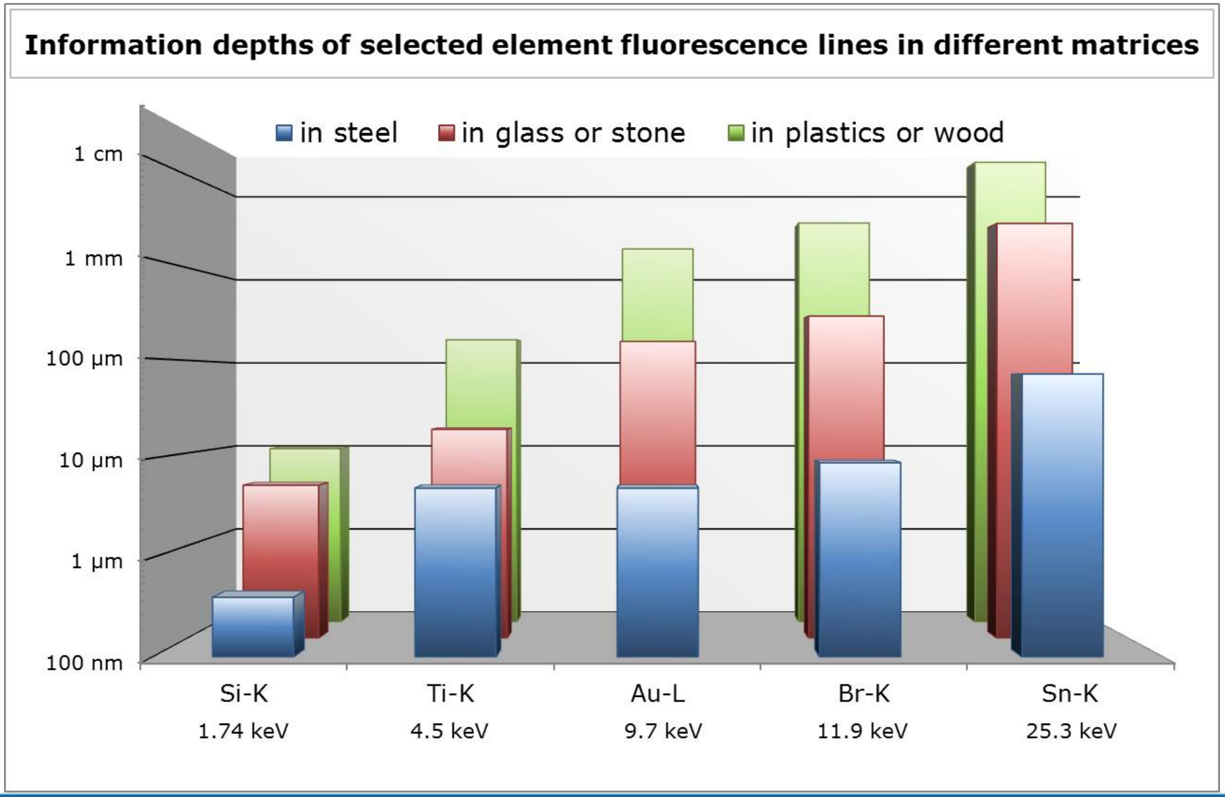


X-ray Interaction with sample
Information depth

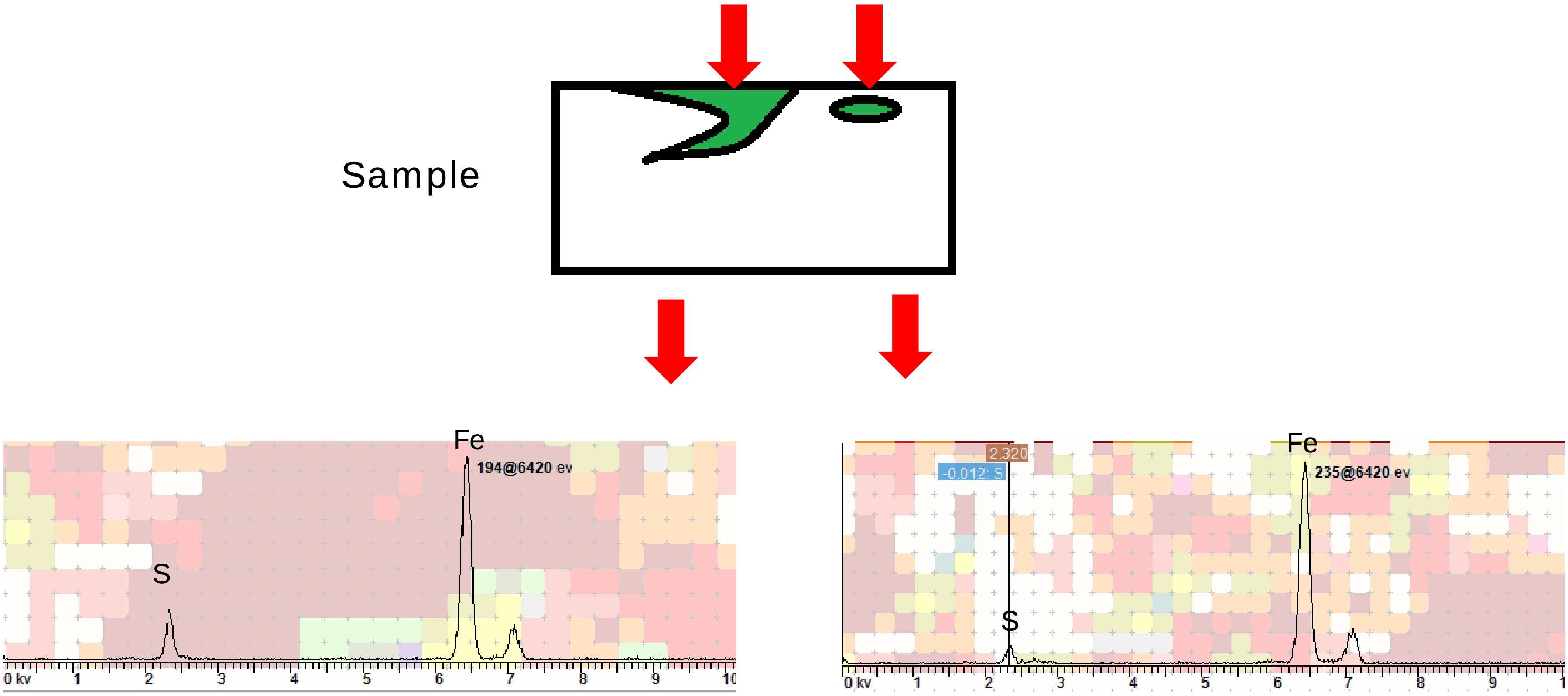
- Penetration depth: the depth that can still be excited
- Information depth: the depth from which fluorescence X-rays can still reach the detector







X-ray Interaction with sample
Example



Interaction with the sample
Scattering

- Inelastic scattering (Compton)
- Elastic Scattering (Rayleigh)
- Bragg Diffraction – scattering of X-rays from a crystal lattice. The position of this detected interference will depend on the orientation of the crystal and the angle of the detector

Advanced Micro-XRF
Electron vs. Photon excitation

	Electron	Photon
Detection	EDS	
Detection limit	100 ppm	**1 ppm****
Resolution	nm	**> 20 µm**
Optimal excitation	Z < Ca	**Z > Ca**
Highest line	Fe (15 kV) - Rb (30 kV)	**Ce, Nd @ ~ 40 keV**
Lowes Z	Be (Li..)	**Na (N…)**
Information depth	nm - < 5 µm	**µm-cm ****
Measurement artefacts	bremsstrahlung	Compton, Rayleigh, **Bragg****
Scan mode	beam/sample	**sample**
Sample visualization	fast -SE/BSE	**slow -total intensity**

X-ray analysis - µXRF-based vs. SEM-based

µXRF-based

Advantage

- No vacuum required
- Little sample preparation
- Large sample size
- Trace element sensitive (better detection limit)
- Higher excitation energy

Limitation

- Rel. large spot size / interaction volume
- Currently no light element detection below Na
- Spectrum artefacts

SEM-bassed

Advantage

- small interaction volume -> high resolution
- Light element detection

Limitation

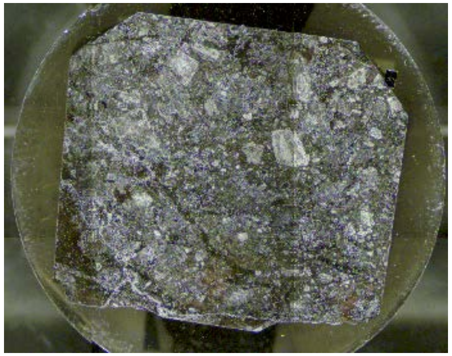
- Vacuum required
- Sample preparation
- Rel. small sample size

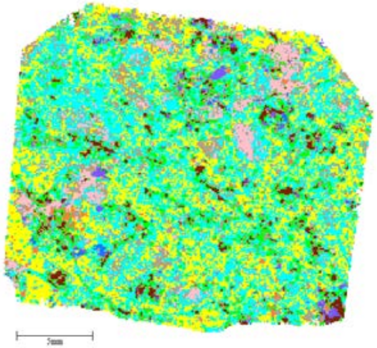
What is AMICS?

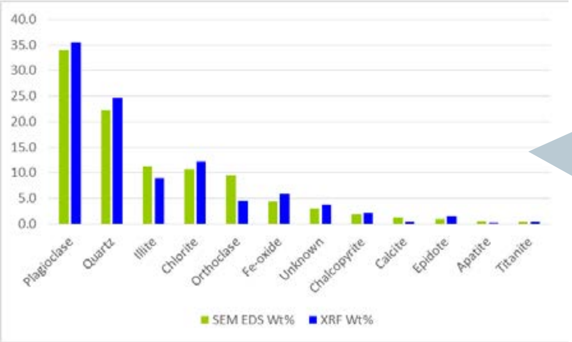
AMICS- Advanced **M**ineral **I**dentification and **C**haracterization **S**ystem

Is an **automated system** to

- Perform high speed, autonomous image and spectral analysis
- Provide statistical information on phases contained in the sample and spatial distribution

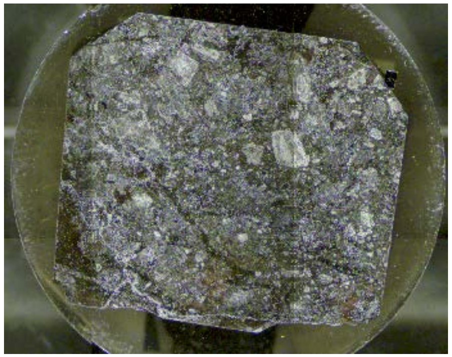




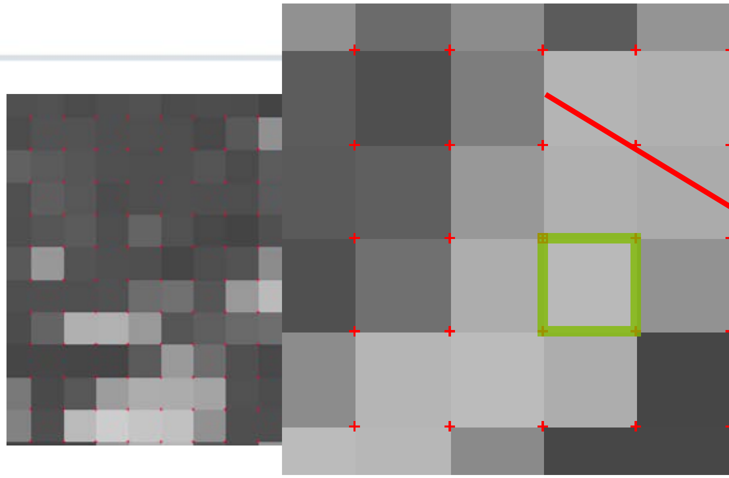


AMICS

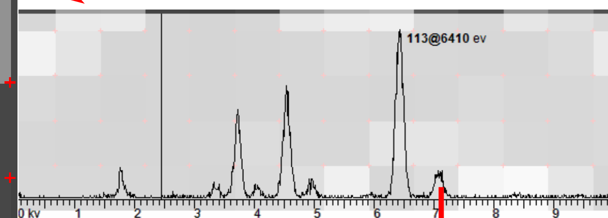
How Does AMICS for M4 Work



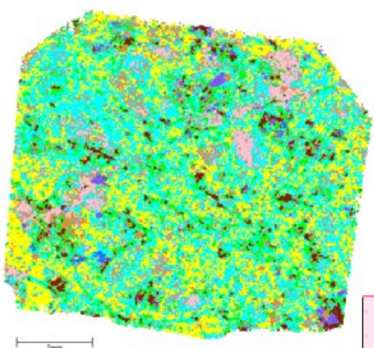
Sample



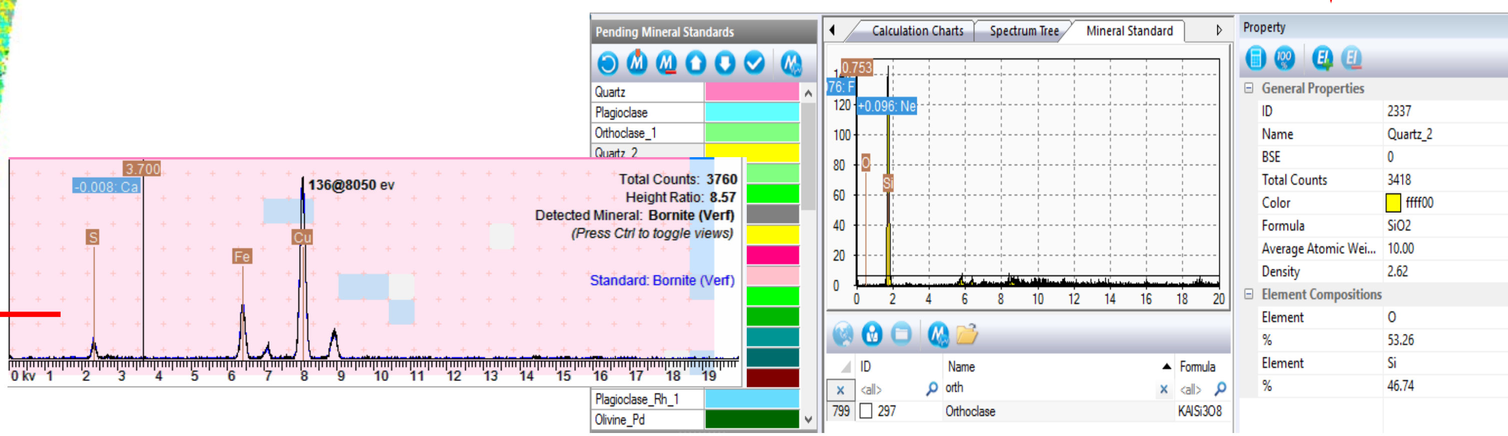
Measurement



Mineral identification
and
classification

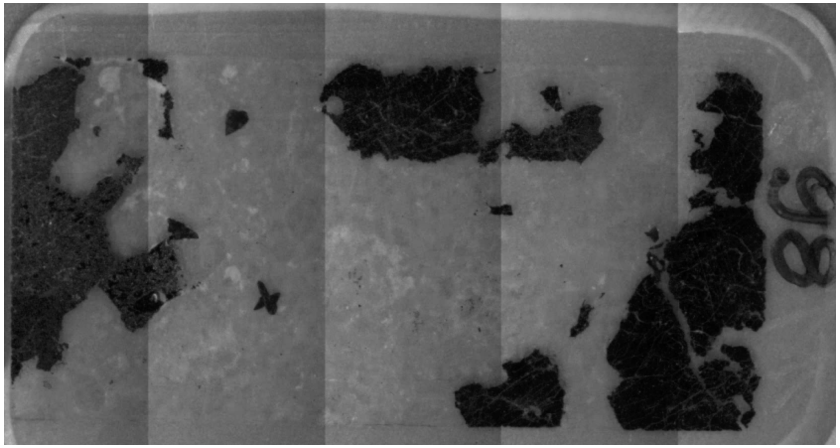


Mineral Map



AMICS
Result Reporting

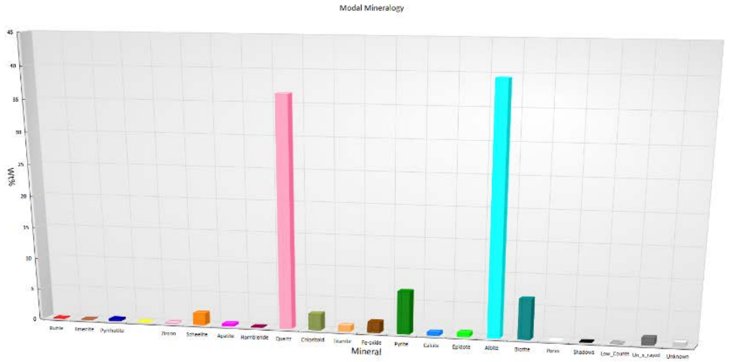
Map overview with mineral phases or table

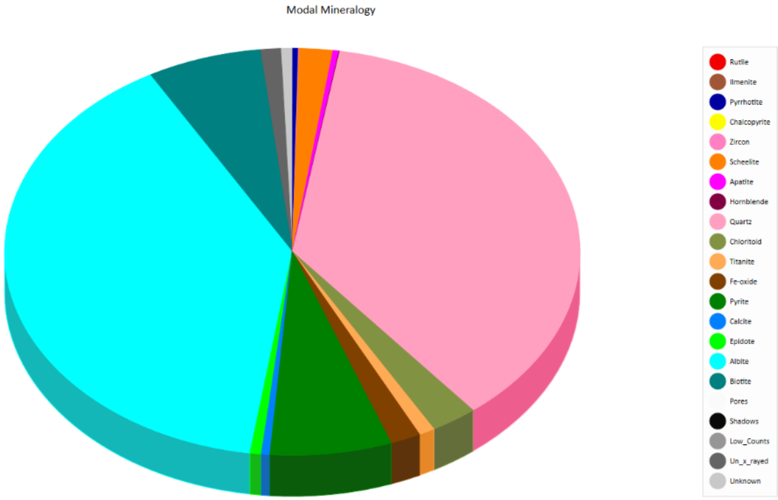


Modal Mineralogy

	Name	Wt%	Area%	Area (µ2)	Particle Num...	Grain Number	Relative Error
	<all>	<all>	<all>	<all>	<all>	<all>	<all>
1	Quartz	6.20	7.32	784575.10	1	931	1.87
2	Andesine	21.93	25.37	2721339.45	1	1067	1.87
3	Ferrohornble...	48.03	46.09	4942962.19	1	666	1.87
4	Biotite	9.97	9.74	1044473.43	1	2051	1.87
5	Ilmenite	7.24	4.74	508612.79	1	467	1.87
6	Apatite	4.58	4.44	476034.98	1	205	1.87
7	Epidote	0.66	0.60	63998.02	1	403	1.87
8	Chlorite	0.39	0.38	40686.61	1	547	1.87
9	Other	0.61	0.58	62555.11	1	712	1.87
10	Unknown	0.39	0.74	79395.40	8	2864	0.00

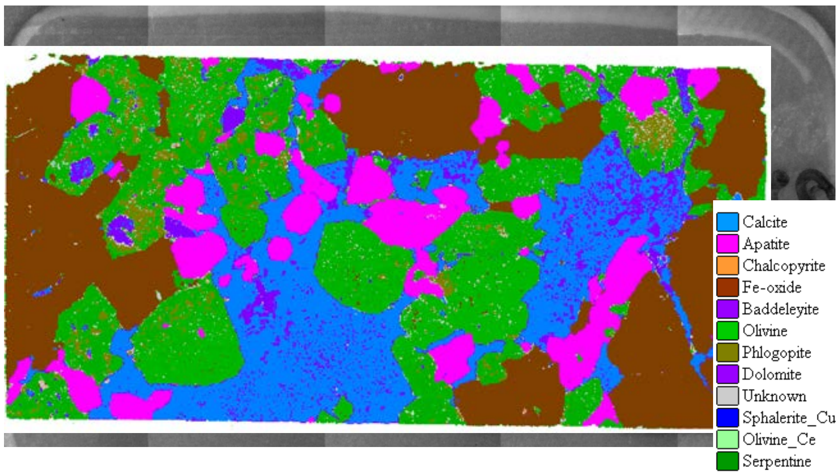
Minerals or elements in different chart diagrams





AMICS
Result Reporting

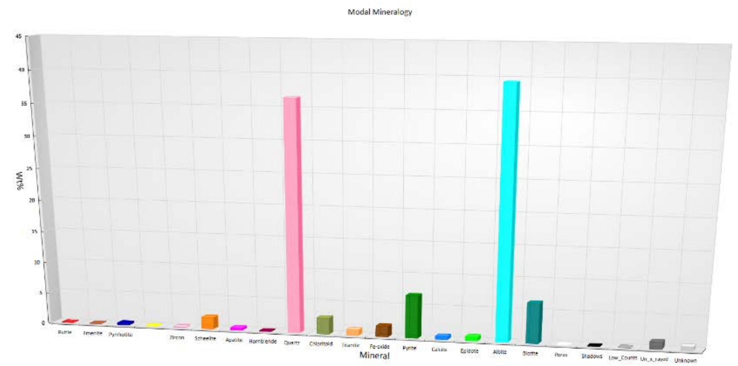
Map overview with mineral phases or table

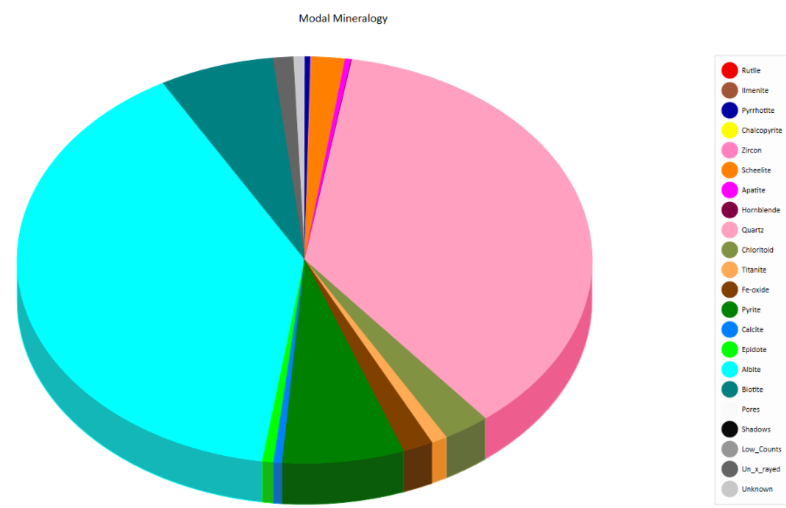


Modal Mineralogy

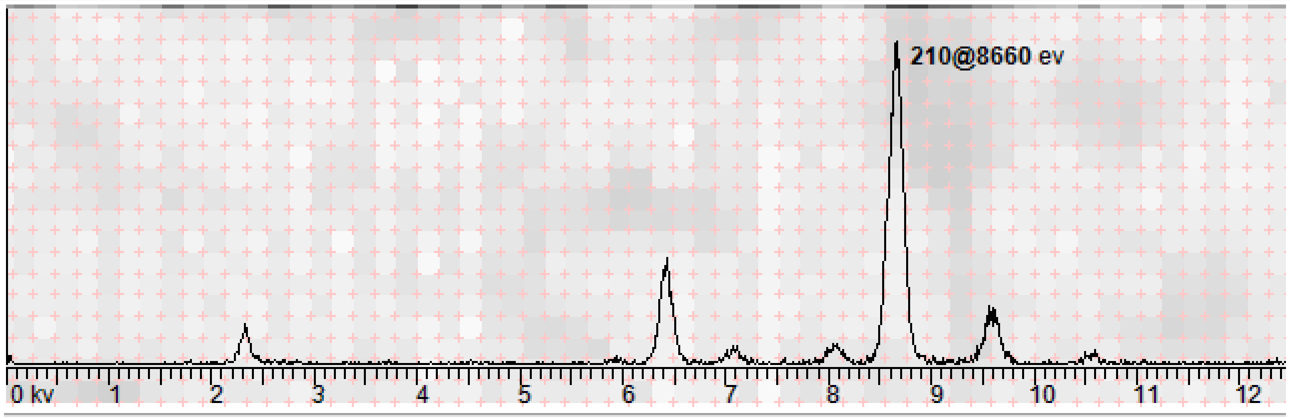
	Name	Wt%	Area%	Area (µ2)	Particle Num...	Grain Number	Relative Error
	<all>	<all>	<all>	<all>	<all>	<all>	<all>
1	Quartz	6.20	7.32	784575.10	1	931	1.87
2	Andesine	21.93	25.37	2721339.45	1	1067	1.87
3	Ferrohornble...	48.03	46.09	4942962.19	1	666	1.87
4	Biotite	9.97	9.74	1044473.43	1	2051	1.87
5	Ilmenite	7.24	4.74	508612.79	1	467	1.87
6	Apatite	4.58	4.44	476034.98	1	205	1.87
7	Epidote	0.66	0.60	63998.02	1	403	1.87
8	Chlorite	0.39	0.38	40686.61	1	547	1.87
9	Other	0.61	0.58	62555.11	1	712	1.87
10	Unknown	0.39	0.74	79395.40	8	2864	0.00

Minerals or elements in different chart diagrams



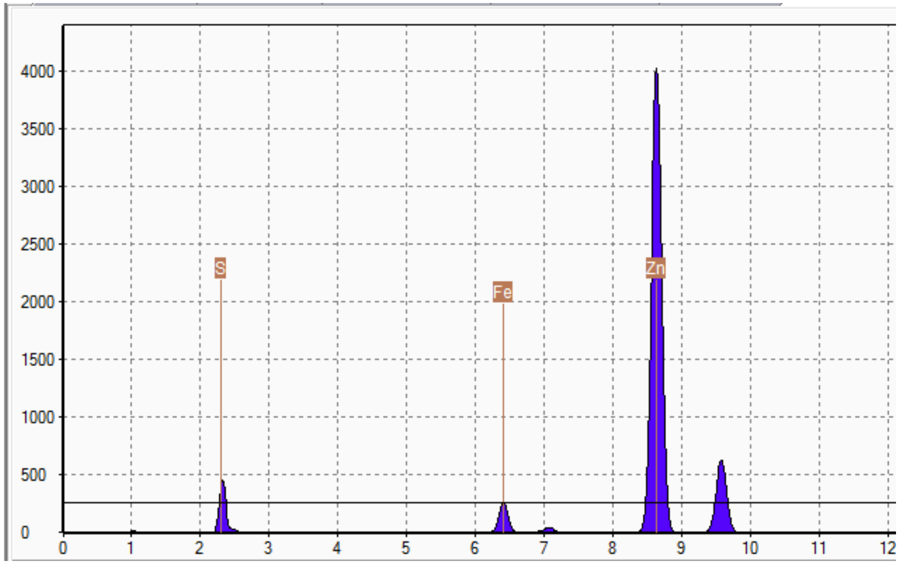


Mineral Classification
Global/Reference spectra



Measured Spectrum

Reference Spectrum



Mineral Classification
Synthetic Generated Spectra

- Patented software from Bruker
- Generate spectra from mineral composition
- Accounts for system specific characteristics
- Possible to generate standards for variable compositions such as solid solution series olivine, feldspar carbonates and even arsenian pyrite
- Mixed spectra

Mineral Classification
Current classification model

- Chi-square statistical method
- Not very sensitive for small peaks/minor elements
- Incorrect classifications/false positives
- Mixed Spectra – difficult to account for all possible mixes
- Artefact such as Bragg diffraction that can occur with X-ray generated technique

Spectrum Tree
Aim

- Control the mineral classification
- Ensure accuracy and repeatability
- Easily browse and search spectrum

Spectrum Tree

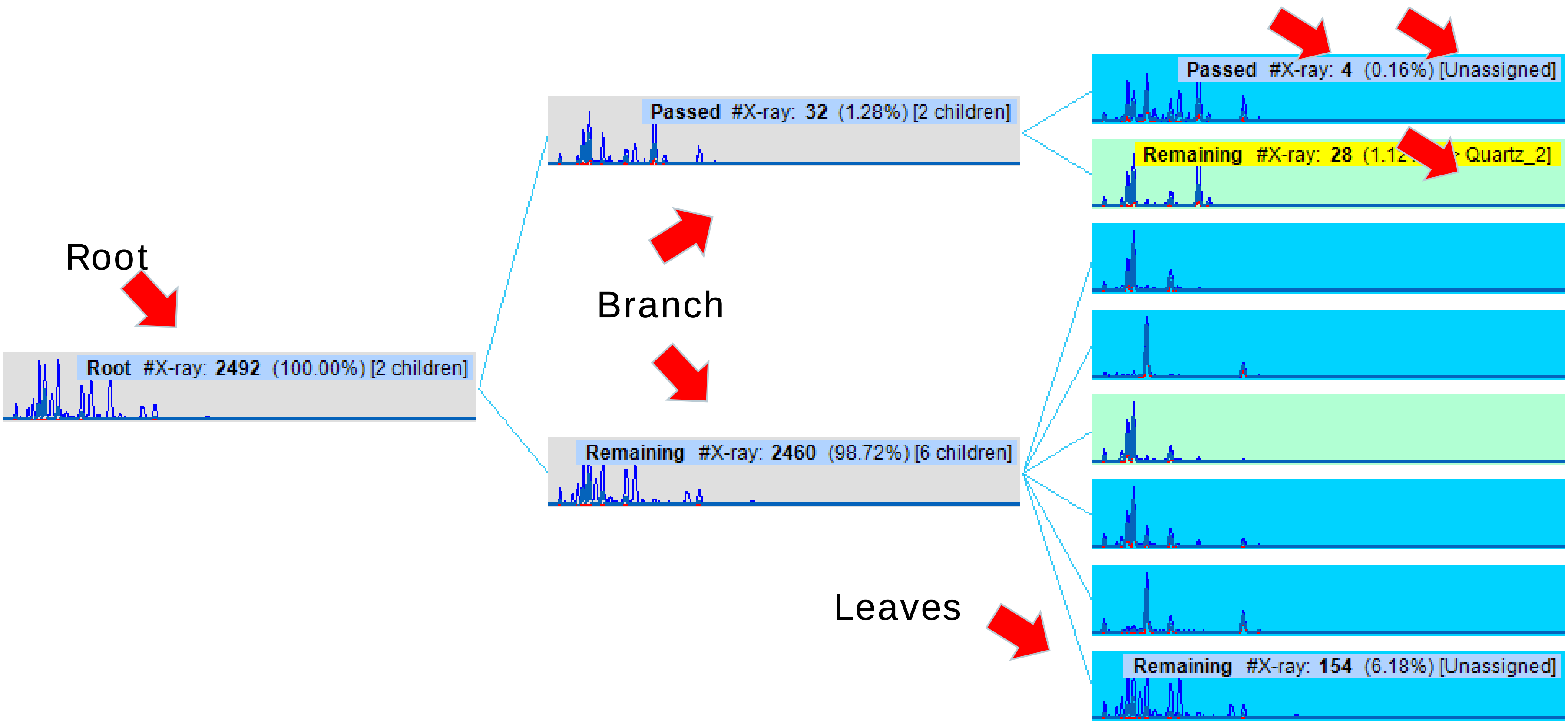
Motivations

- To handle corner cases of mineral classification (e.g. trace/low amounts)
- To take other information into account (e.g. BSE for Hematite/Magnetite)
- To inspect the measured spectrum for quality assurance
- More transparency on the accuracy of the classification process
- Ability to investigate the classification of any individual spectra

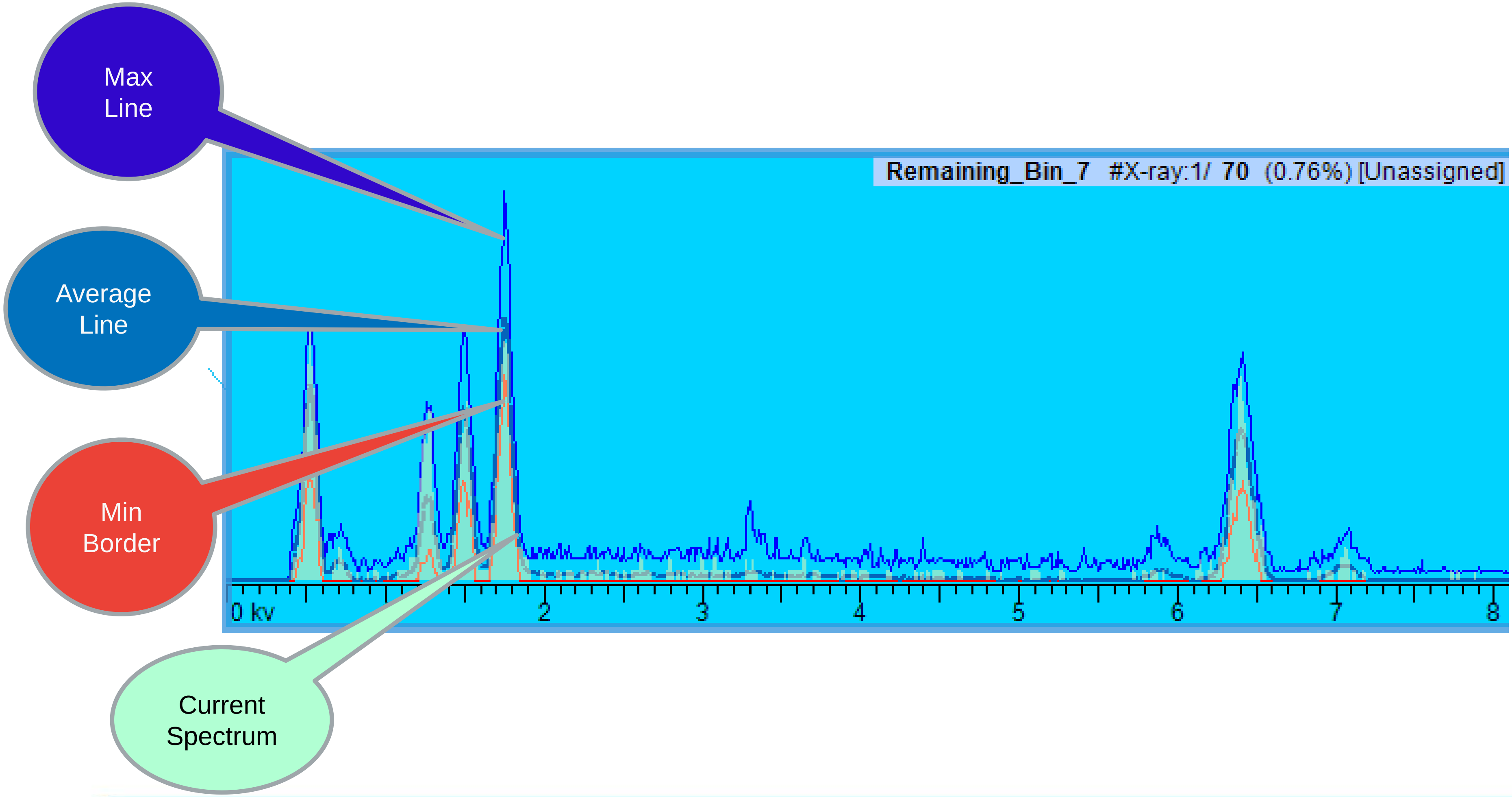
Spectrum Tree
Parameters

- Standard listing
- By energy filtering (energy regions)
- By BSE value
- By count clustering
- By manual clustering
  - Best automatic search
  - Selected spectrum
- By automatic clustering

Spectrum Tree
Layout

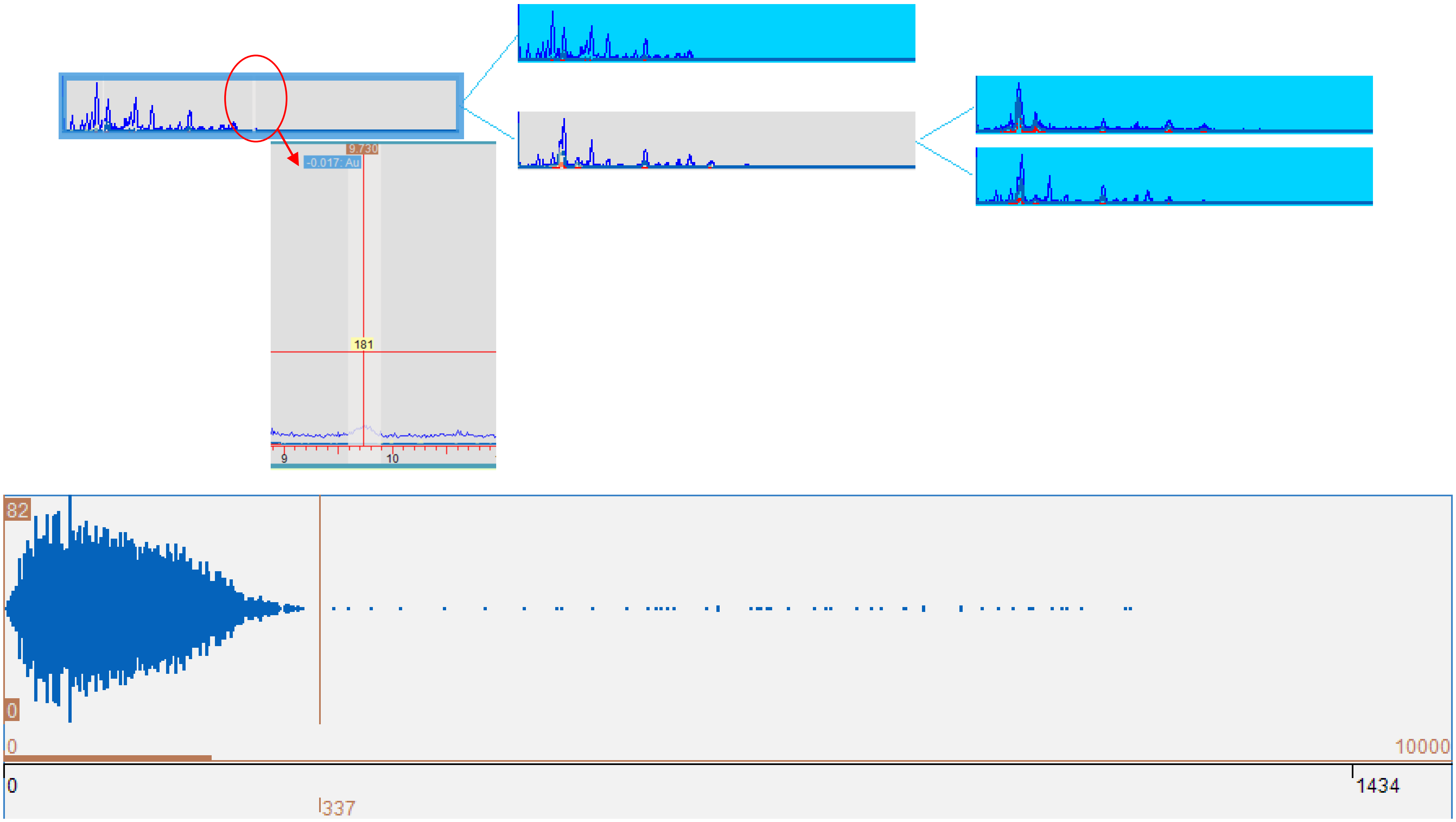


Spectrum Tree
Layout

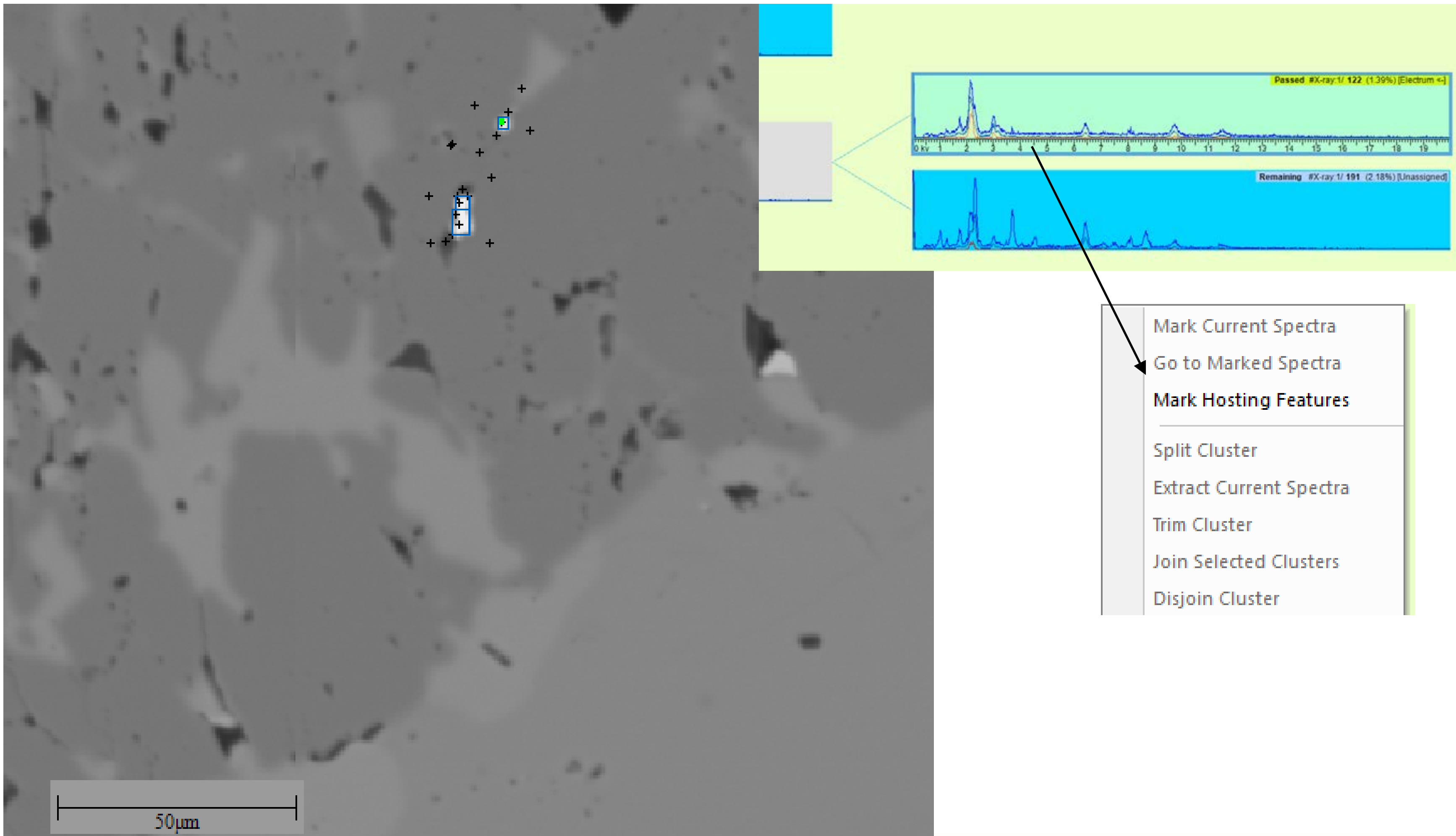


Spectrum Tree

Trace Elements: Gold



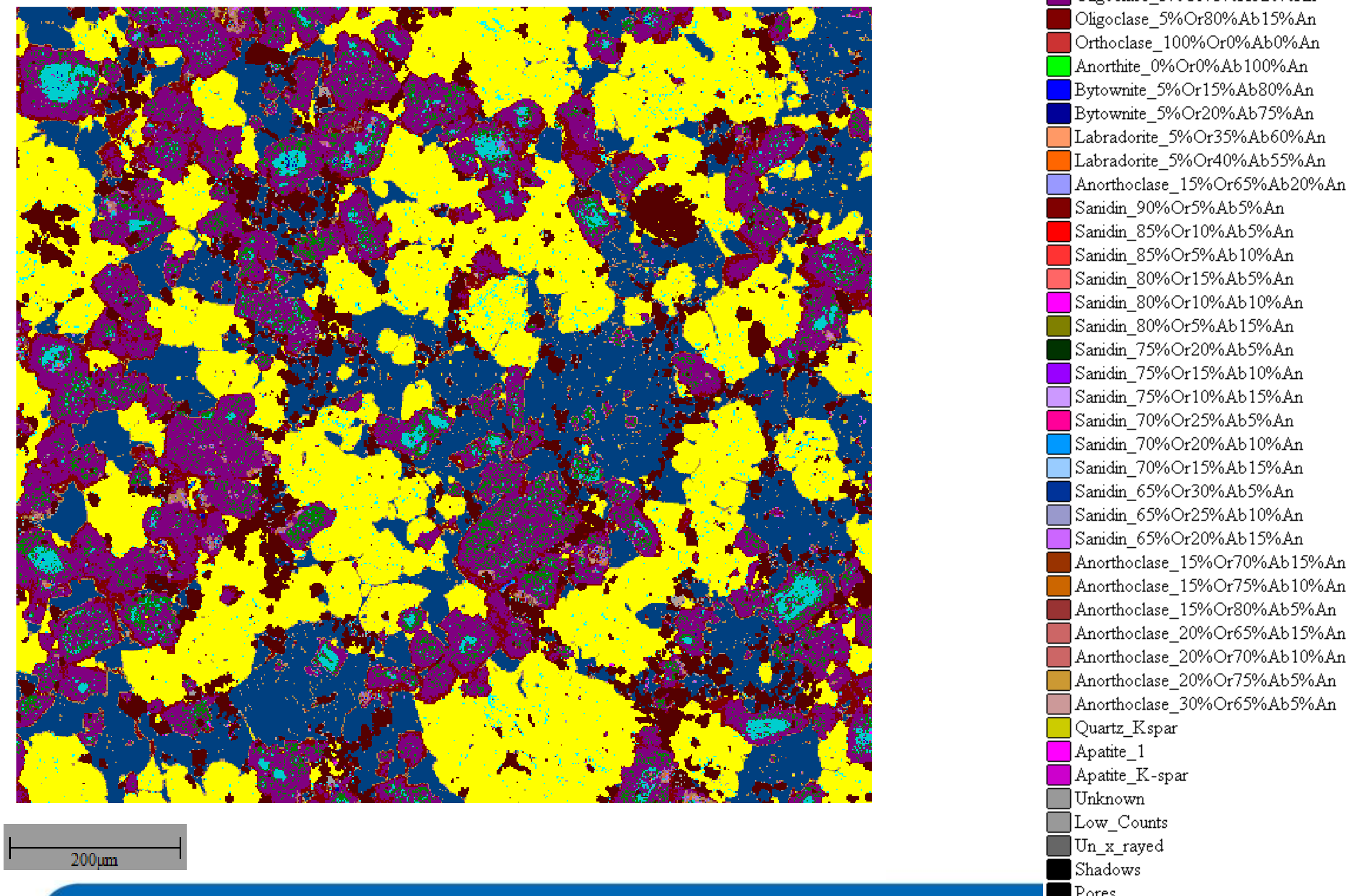
Spectrum Tree
Gold



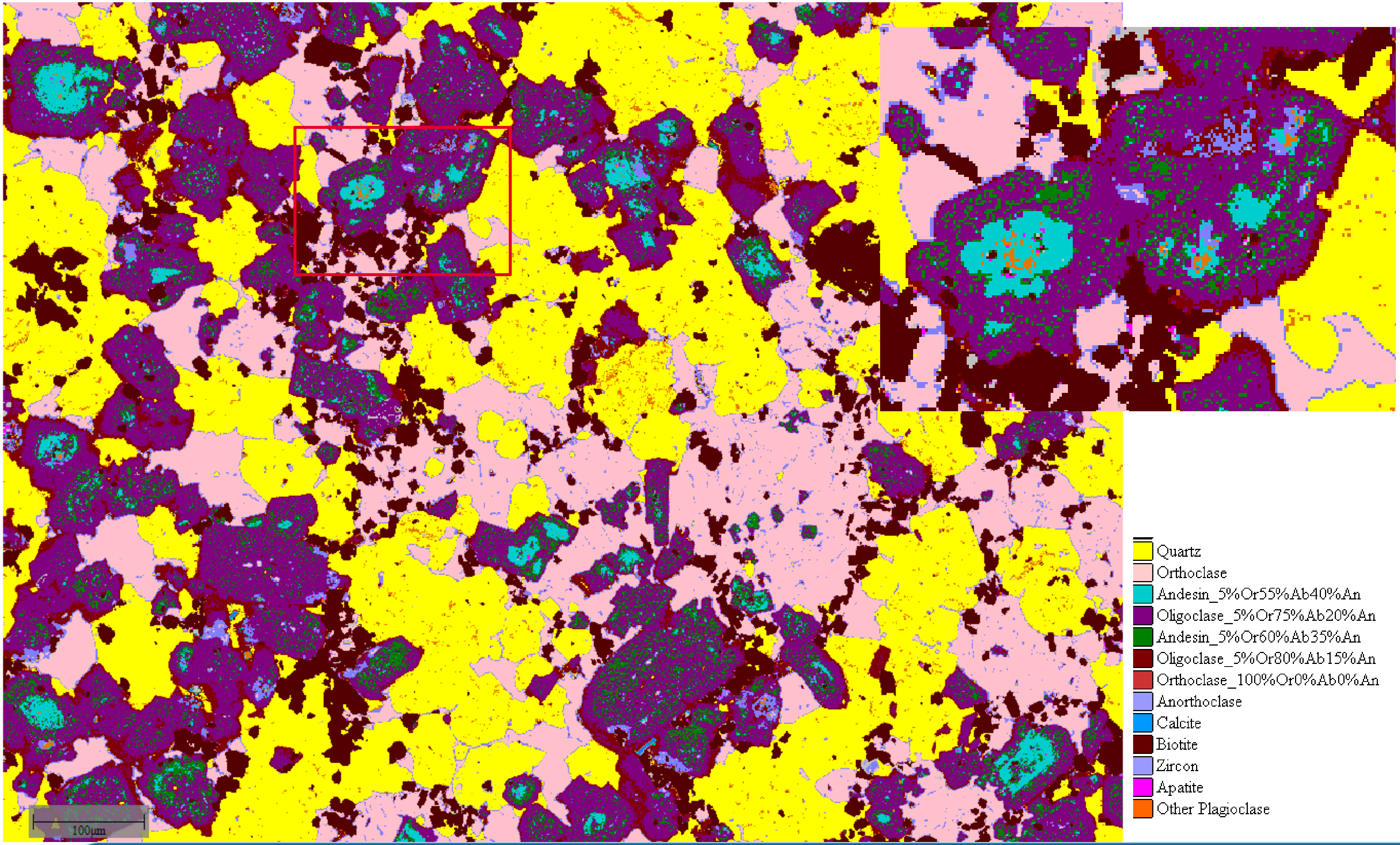
Spectrum Tree
Detailed Mineralogy

Spectrum Tree

Granite Zoned Plagioclase



Spectrum Tree
Granite Zoned Plagioclase



Demonstration

M4 TORNADO AMICS
Summary

- New synthetic spectra assist greatly with the creation of reference spectra and identification of minerals, and even compositional variations can be captured
- Spectrum Tree assist with evaluating and refining classification
- Better utilize the lower detection limit possible with the M4 using the classification parameters provided by the spectrum tree
- Excellent application for capturing information on large samples without any preparation needed or damage to sample
- Helps to make more informed decisions in selecting samples for time-consuming SEM-EDS analysis or even thin section selection for optical microscopy

Q&A

Are There Any Questions?

Please type in the questions you might have

in the Q&A box and press *Send*.

For more information, please contact us:

samual.scheller@bruker.com

gertrudia.gloy@bruker.com

info.bna@bruker.com

https://www.bruker.com/products/x-ray-diffraction-and-elemental-analysis/micro-xrf-and-txrf/m4-tornado/m4-tornado-amics.html

Innovation with Integrity