

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



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# Presenters



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# Overview



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

# The M4 Tornado

## Main advantages



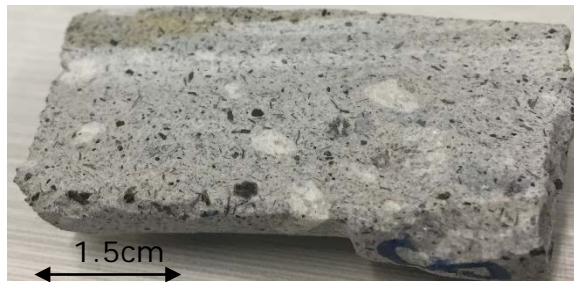
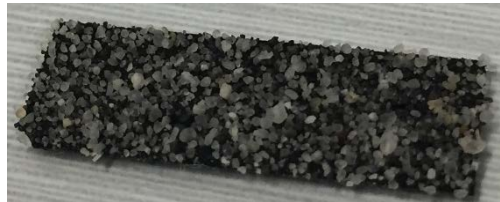
- Rectangular chamber design which accommodates large samples of up to 200 x 160 x 120 mm(WxDxH)
- Pump down <2min allowing detection to Na
- Three cameras assist with sample view and positioning
- Fast 100 mm/s stage with and 4  $\mu\text{m}$  resolution, mouse-controlled and autofocus
- Capillary optics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

# The M4 Tornado

## Main advantages

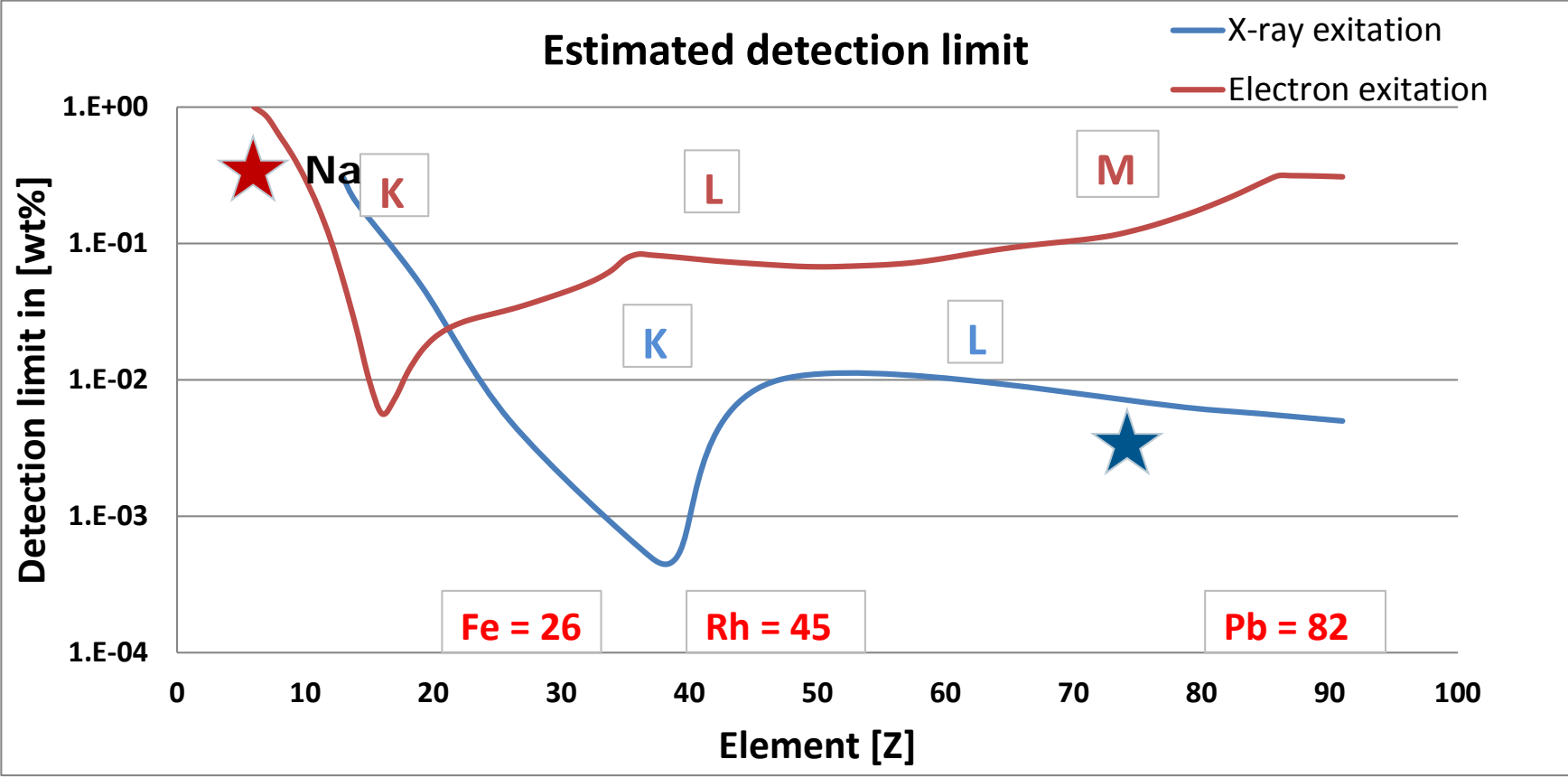


- Little/no sample preparation



# Estimated detection limits

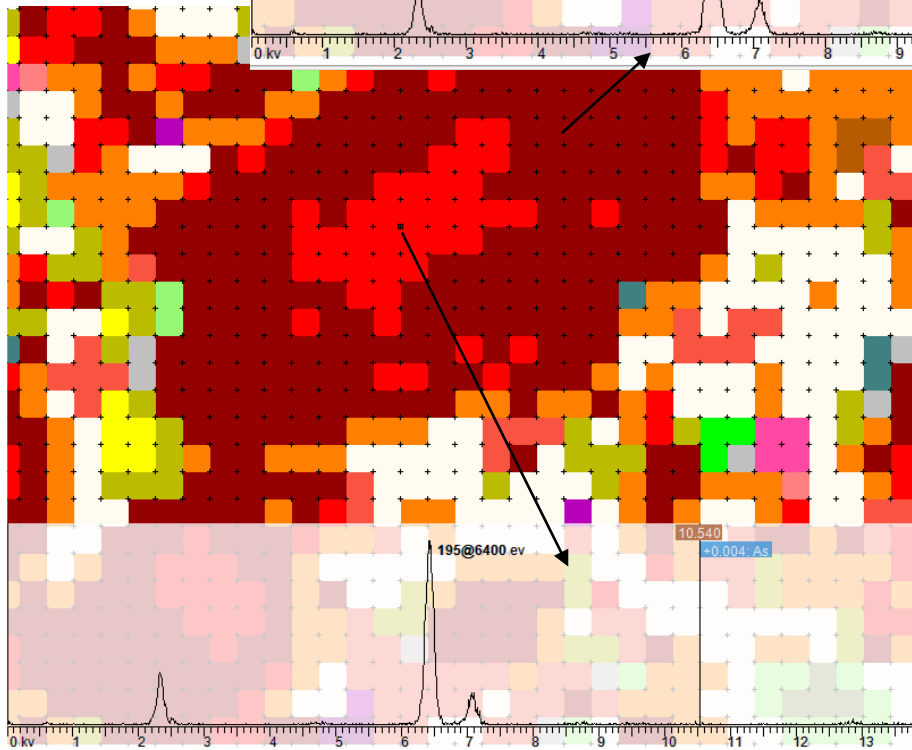
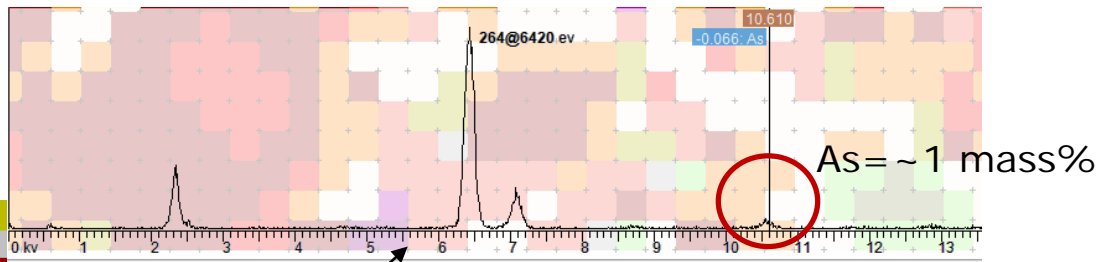
## Electron excitation vs. photon excitation



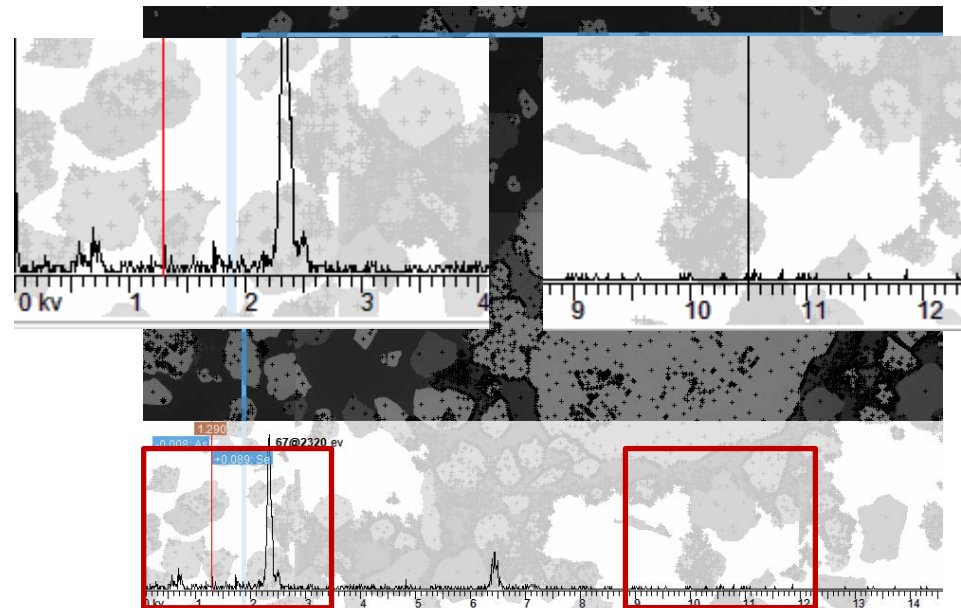
# Estimated detection limits Electron excitation vs. photon excitation



## M4Tornado AMICS



## SEM AMICS



# Element detection M4 Overview



	I	II	IIIa	IVa	Va	VIa	VIIa		VIIIa	Ia	IIa	III	IV	V	VI	VII	VIII		
1	1 H																2 He		
2	3 Li	4 Be										5 B	6 C	7 N	8 O	9 F	10 Ne		
3	11 Na	12 Mg										13 Al	14 Si	15 P	16 S	17 Cl	18 Ar		
4	19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr	
5	37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe	
6	55 Cs	56 Ba	57* La	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn	
7	87 Fr	88 Ra	89** Ac	104 (Ku)	105 (Ns)														
			*Lanthanide																
			58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu			
			**Aktinide																
			90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr			

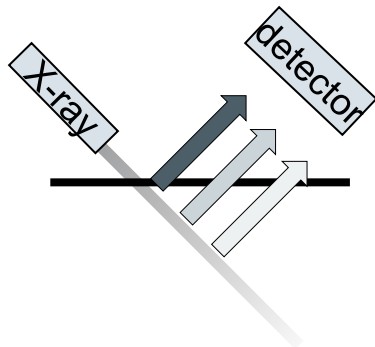
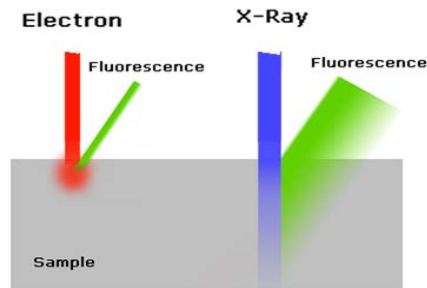
	Not available
	Not yet possible
	Only in vacuum or in He atmosphere
	In air



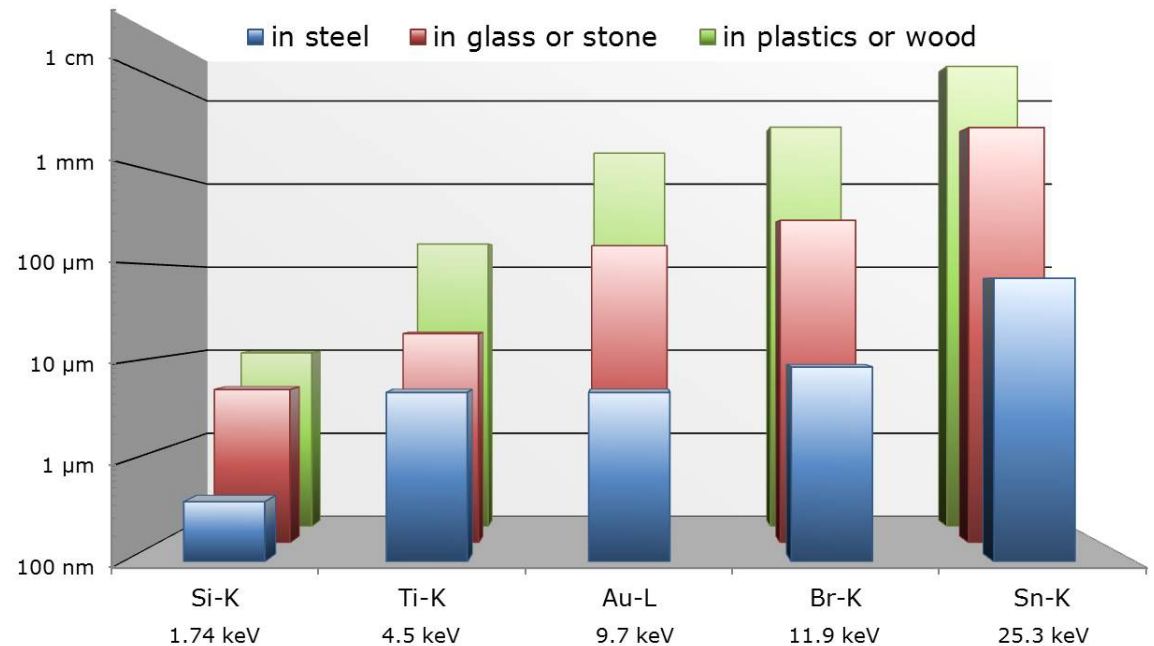
# X-ray Interaction with sample

## 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

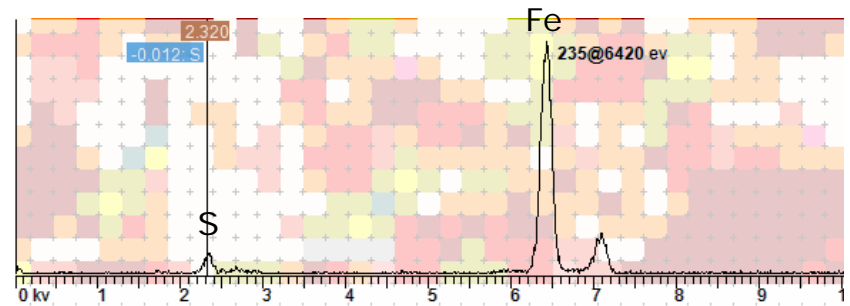
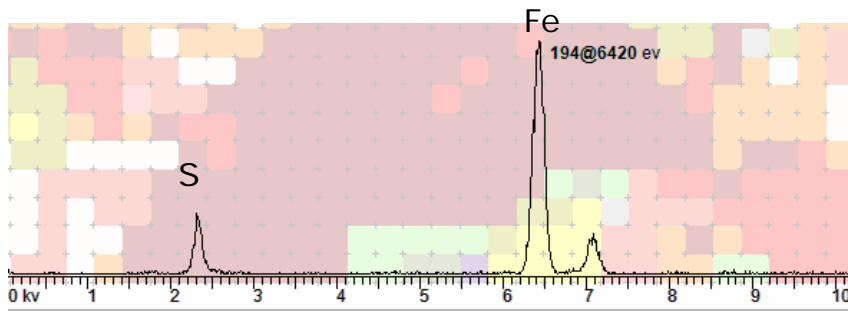
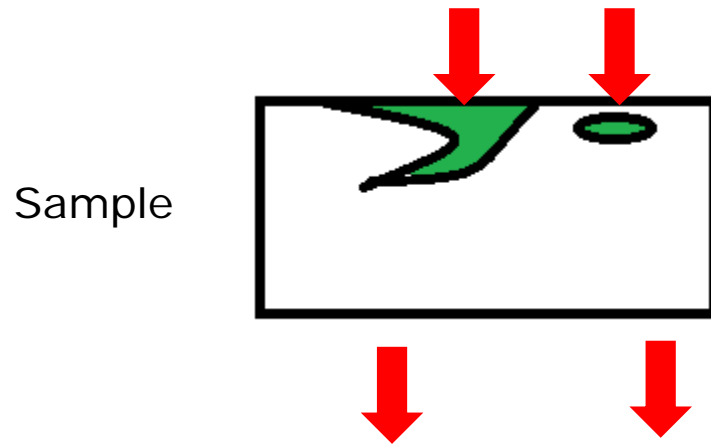


**Information depths of selected element fluorescence lines in different matrices**



# 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 - $\mu$ XRF-based vs. SEM-based



## $\mu$ XRF-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-based

### Advantage

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

### Limitation

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

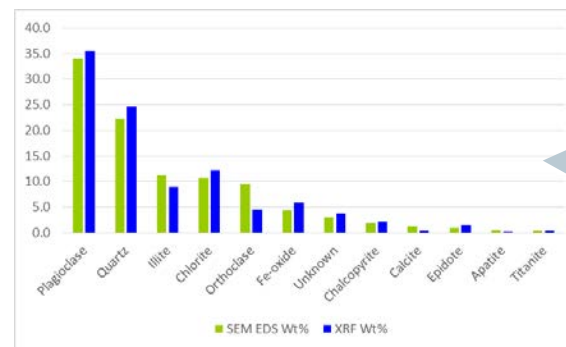
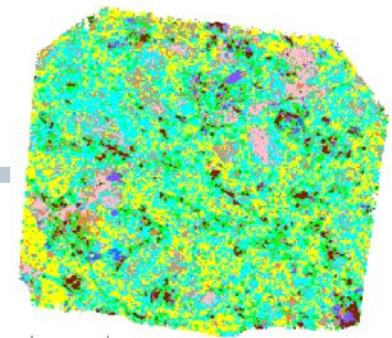
# What is AMICS?



## AMICS- Advanced Mineral Identification and Characterization System

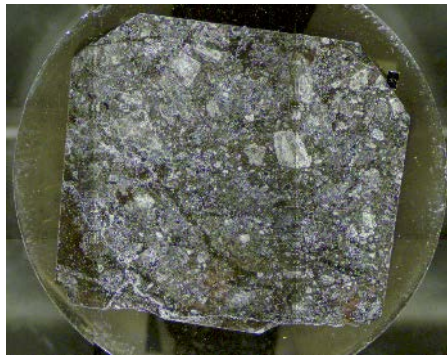
Is an **automated system** to

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

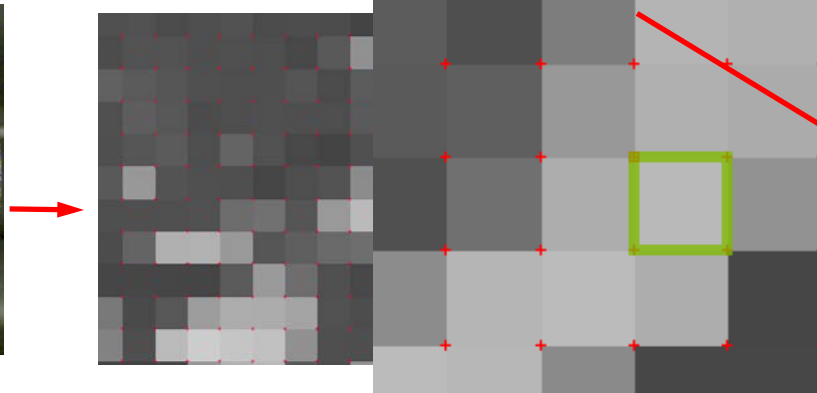


# AMICS

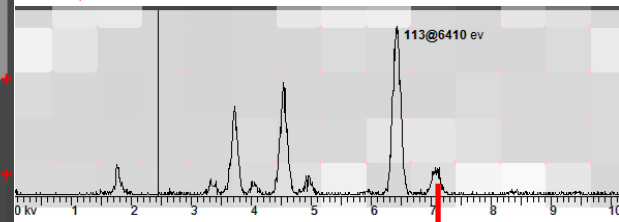
## How Does AMICS for M4 Work



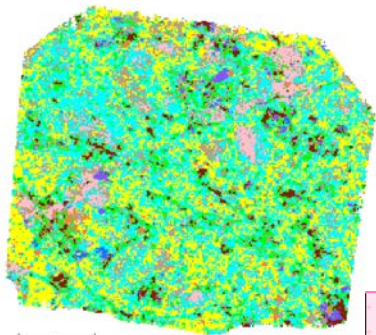
Sample



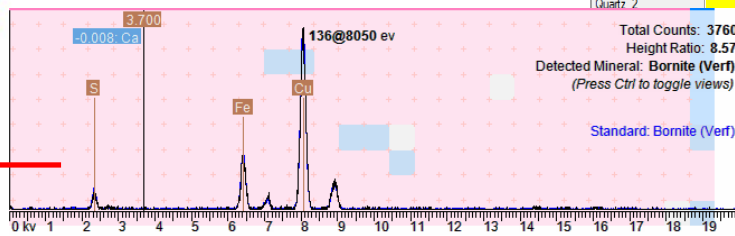
Measurement



Mineral identification and classification



Mineral Map



Software interface showing mineral identification results.

**Pending Mineral Standards**

Quartz	
Plagioclase	
Orthoclase_1	
Quartz_2	

**Property**

General Properties

ID	2337
Name	Quartz_2
BSE	0
Total Counts	3418
Color	ffff00
Formula	SiO2
Average Atomic Wei...	10.00
Density	2.62

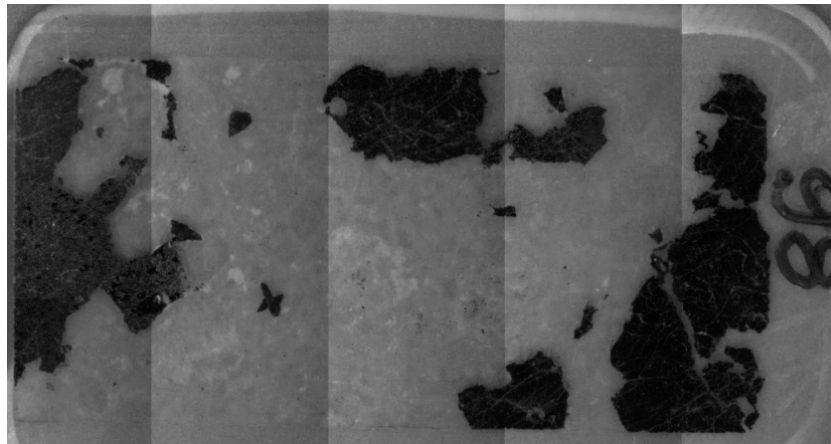
Element Compositions

Element	O	%	53.26
Element	Si	%	46.74

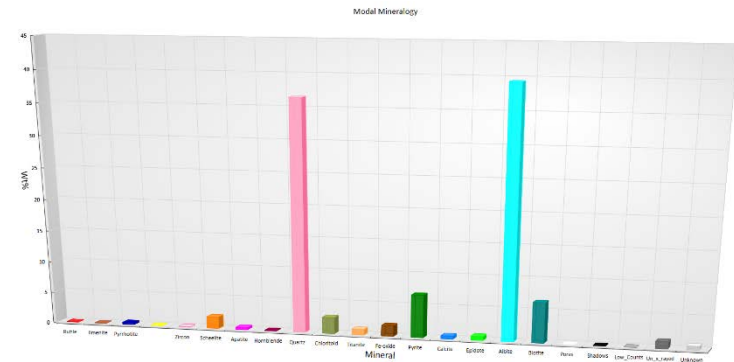
# AMICS Result Reporting



Map overview with mineral phases or table

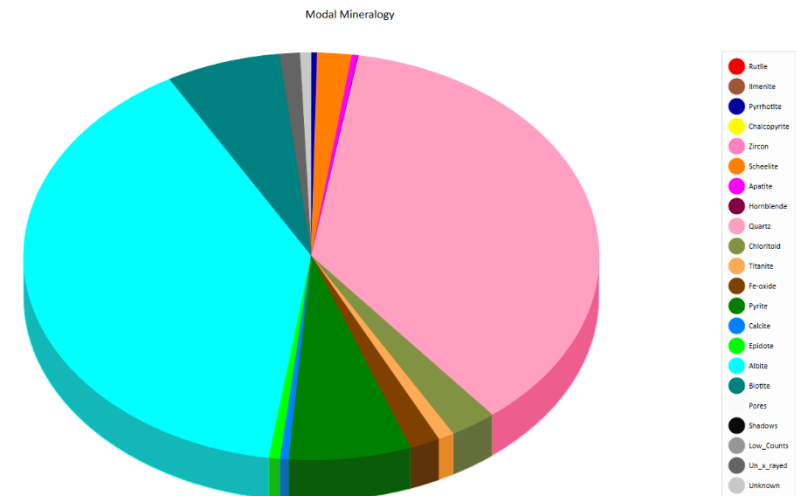


Minerals or elements in different chart diagrams



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 Ferromagnesian	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

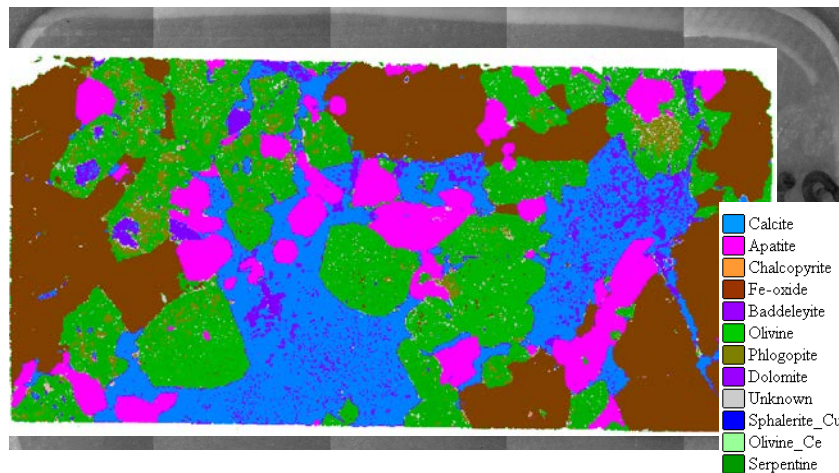




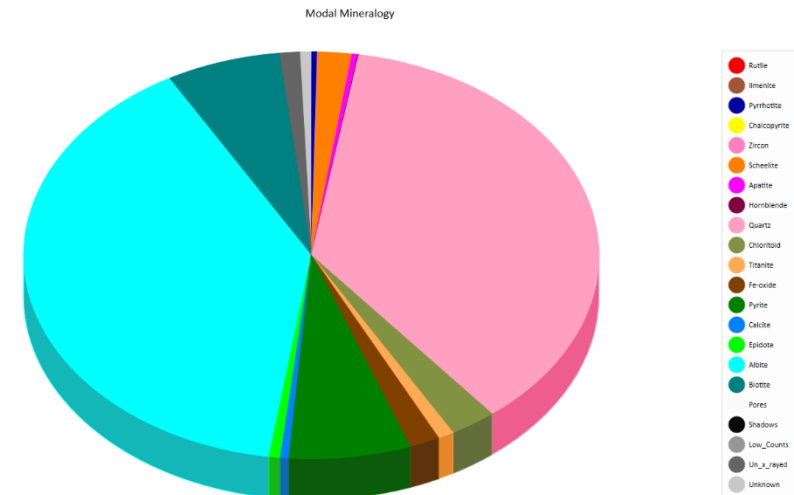
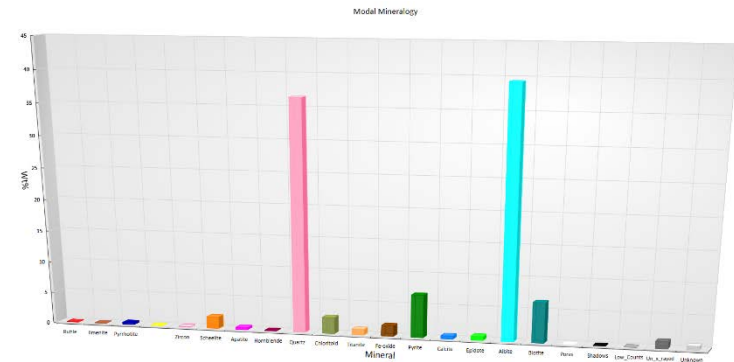
# AMICS Result Reporting



Map overview with mineral phases or table



Minerals or elements in different chart diagrams

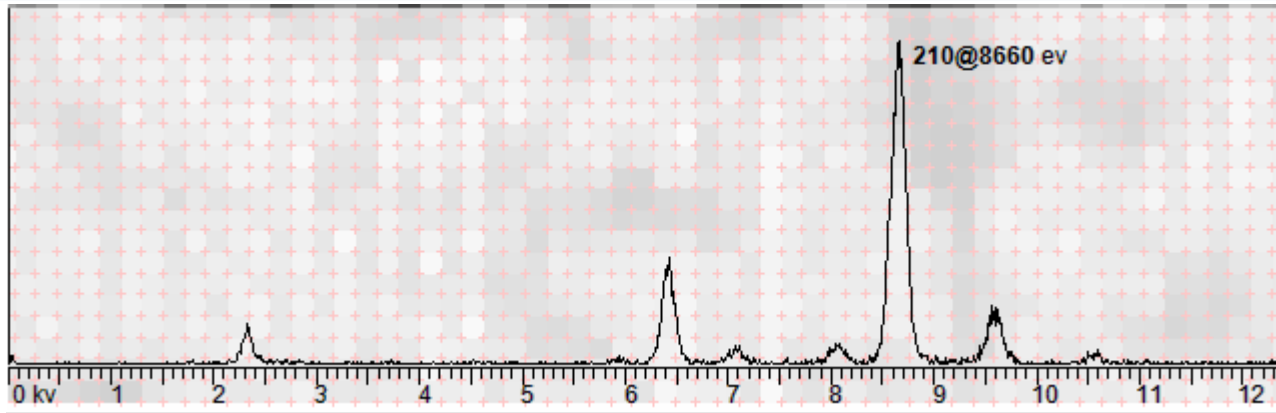


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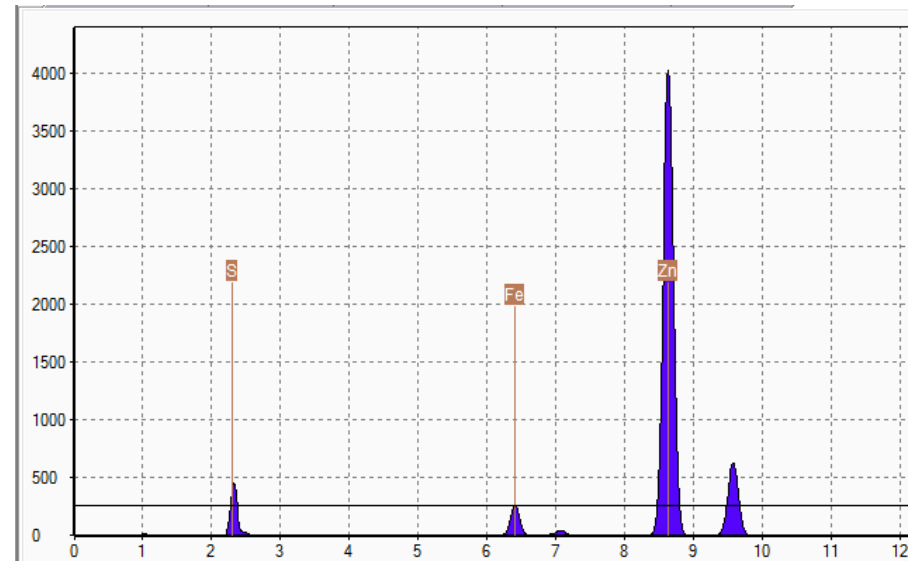
# Mineral Classification

## 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

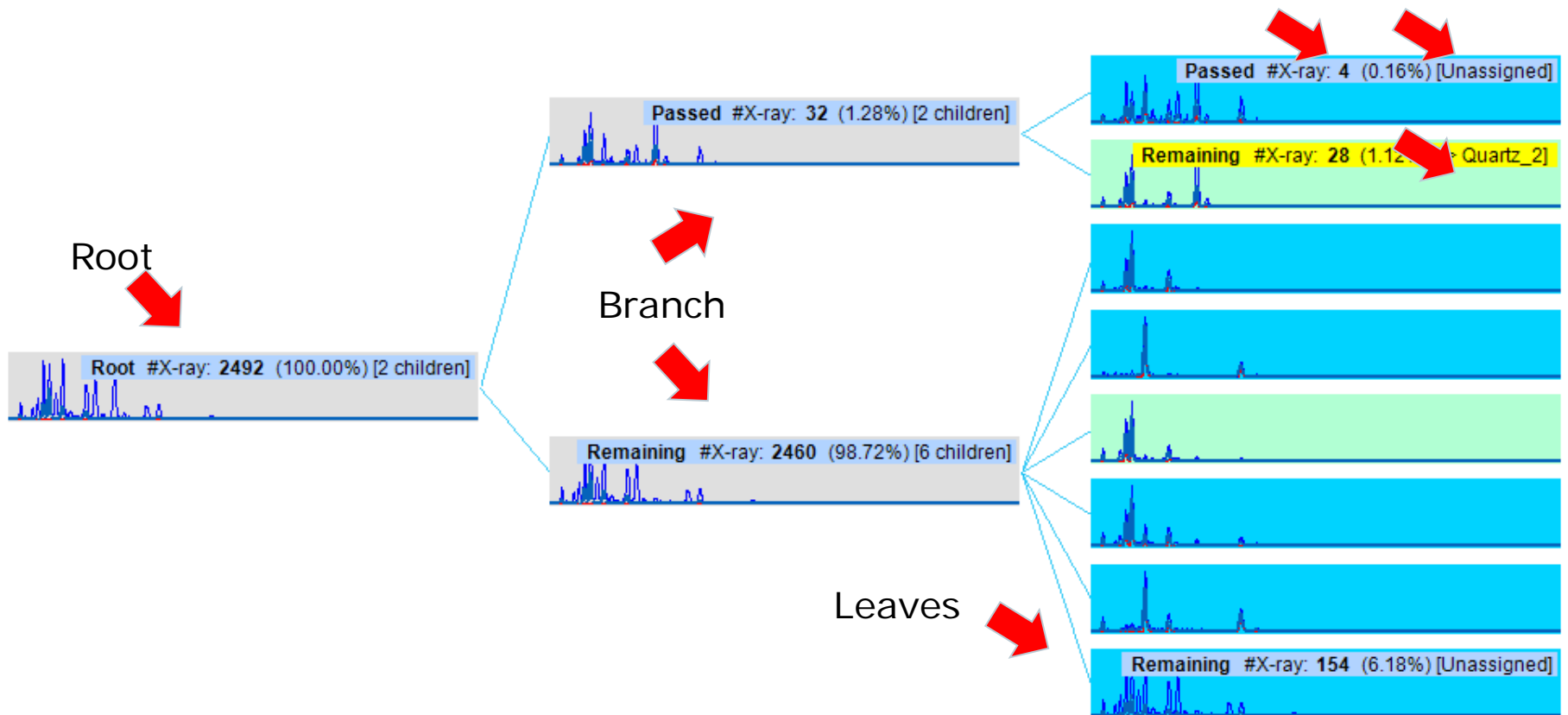
# Spectrum Tree Parameters



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

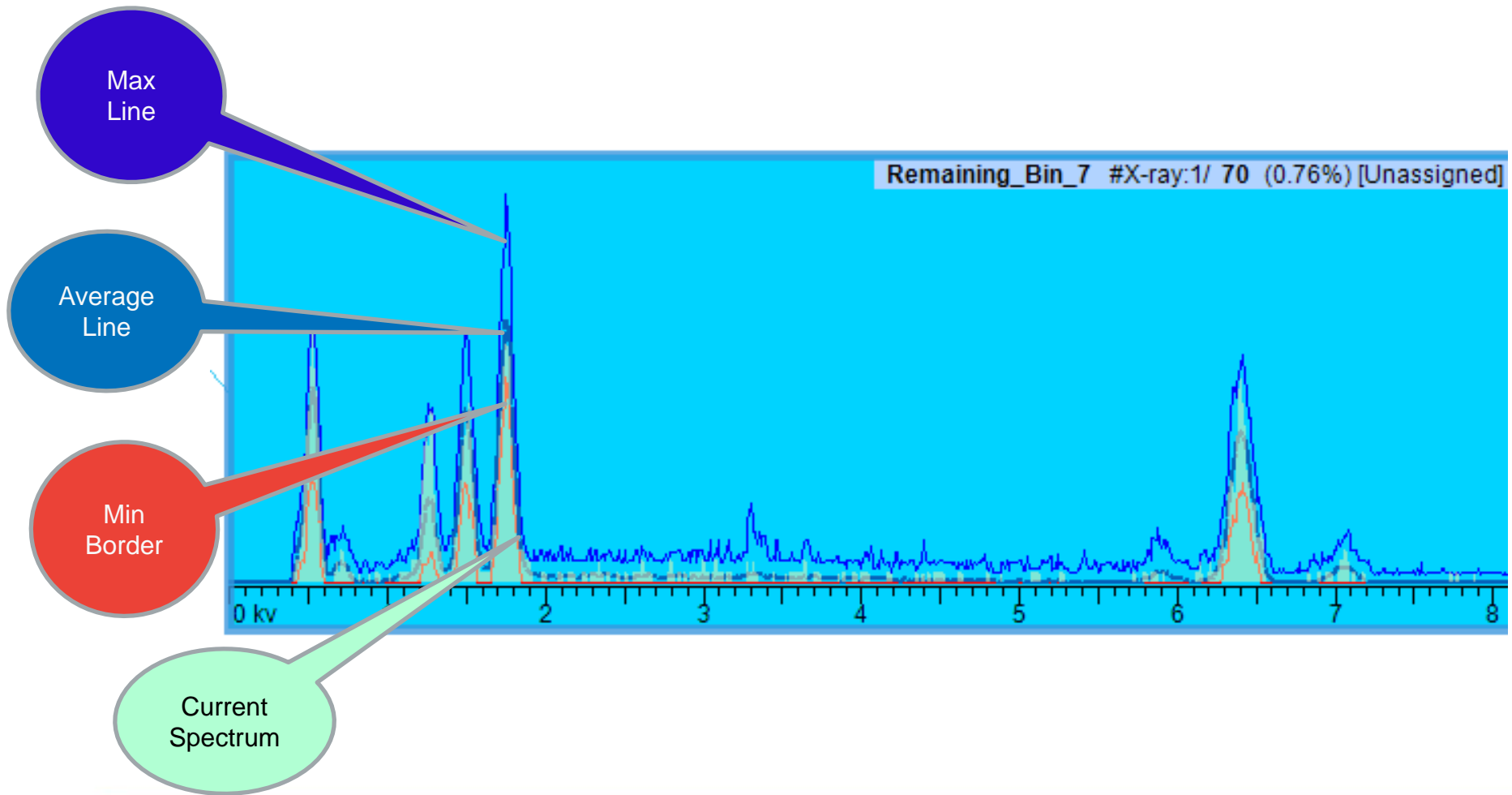


# Spectrum Tree Layout



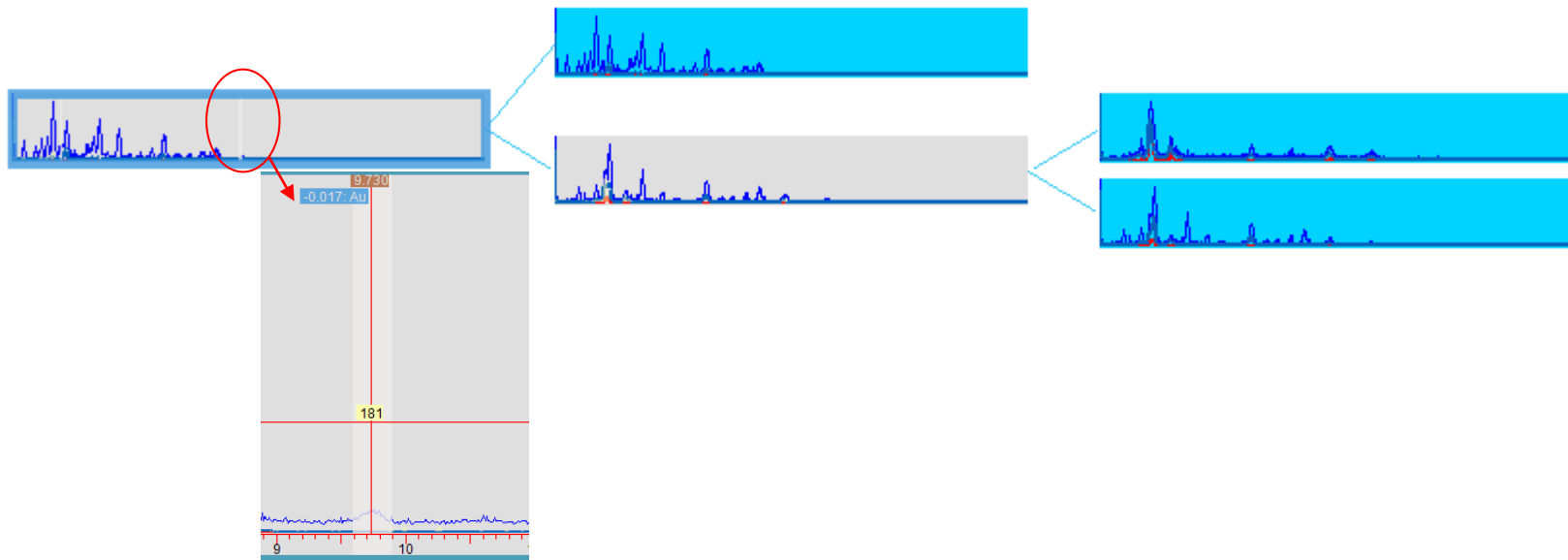


# Spectrum Tree Layout

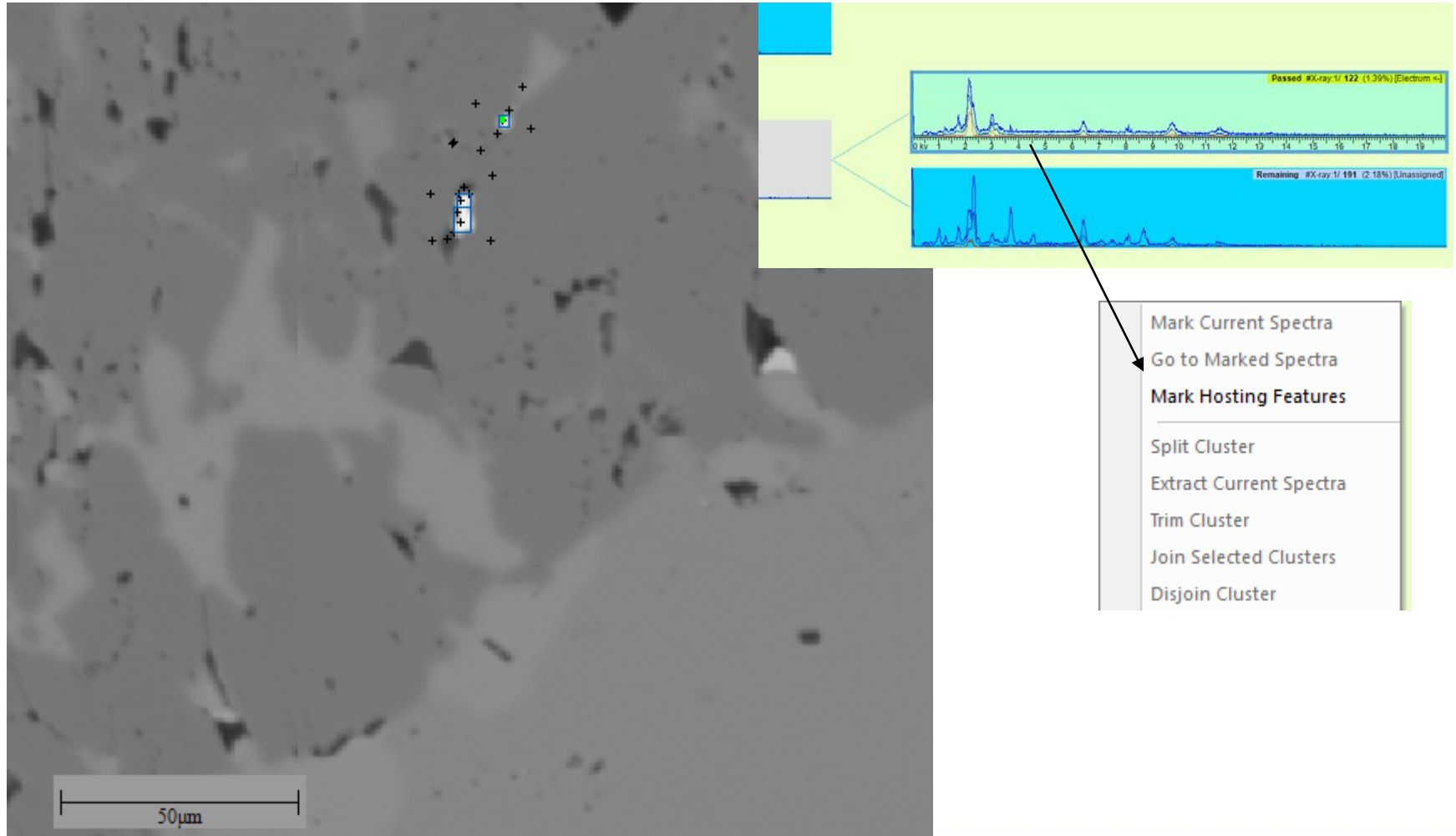


# Spectrum Tree

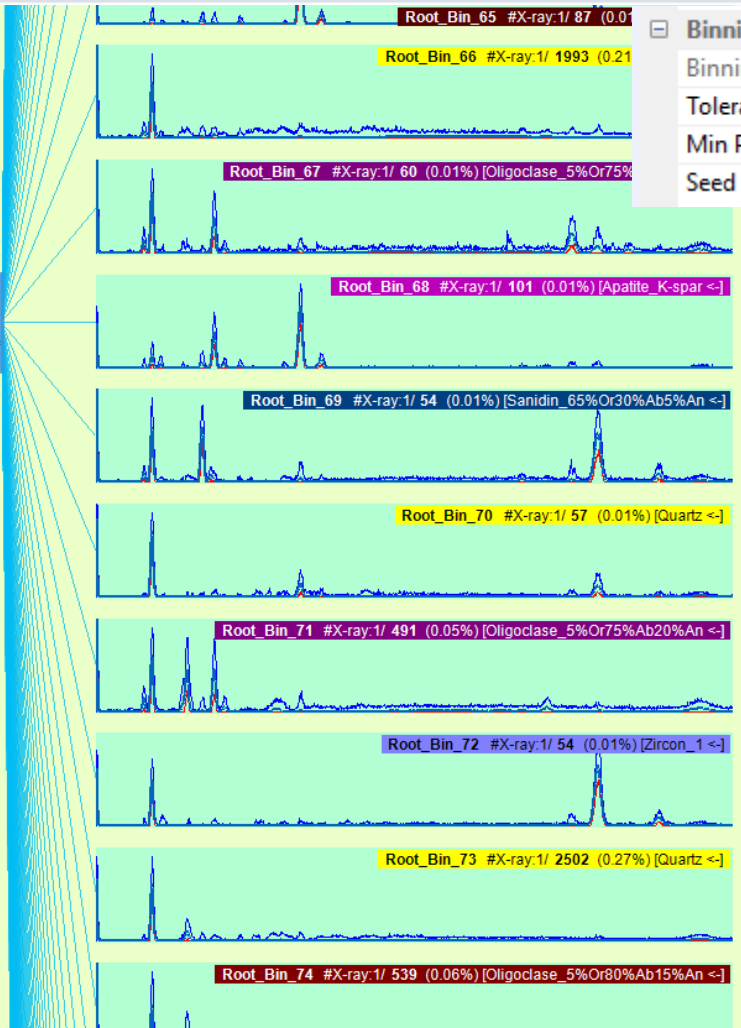
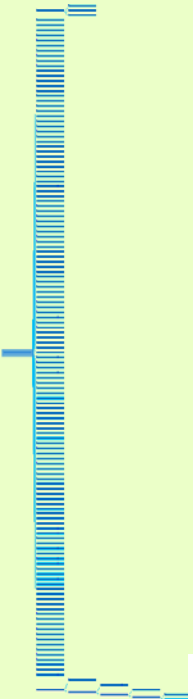
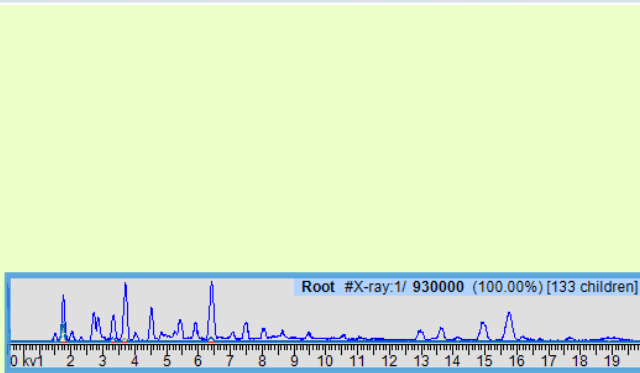
## Trace Elements: Gold



# Spectrum Tree Gold



# Spectrum Tree Detailed Mineralogy

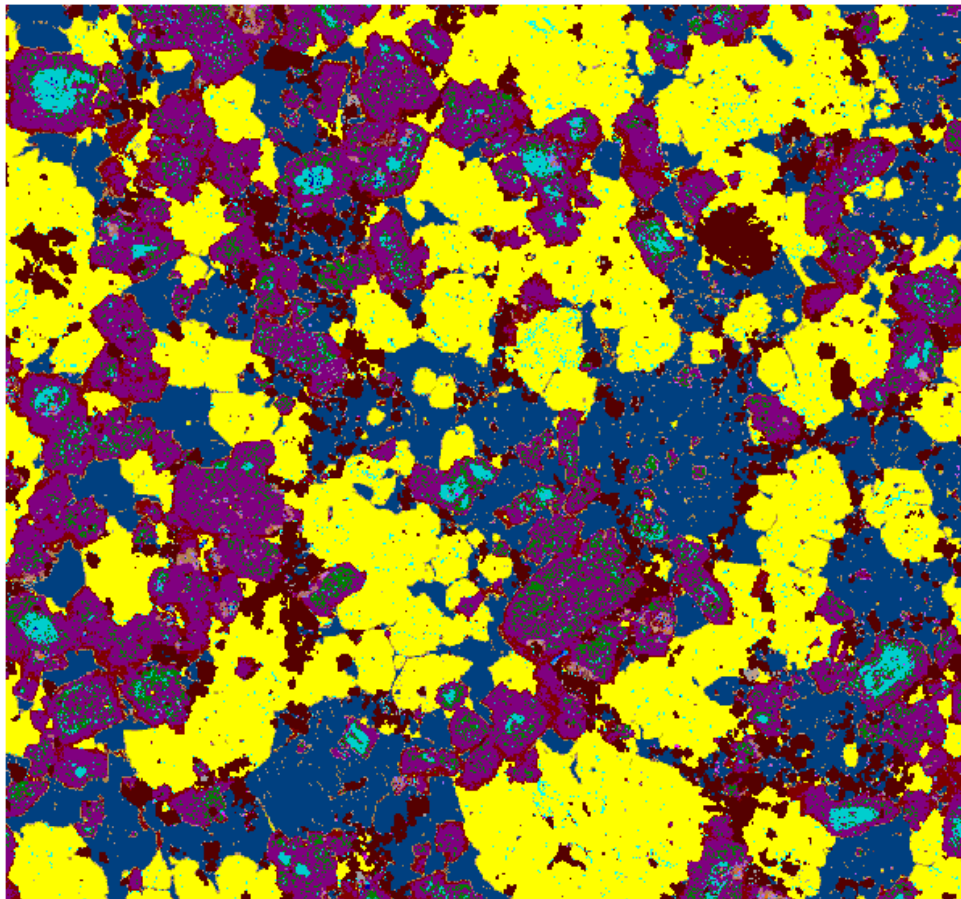


Binning Properties	
Binning Criteria	Automatic Clustering
Tolerance	80
Min Population	10
Seed Searching	80

- Albit\_0%Or100%Ab0%An
- Andesin\_5%Or55%Ab40%An
- Andesin\_5%Or60%Ab35%An
- Oligoclase\_5%Or75%Ab20%An
- Oligoclase\_5%Or80%Ab15%An
- Orthoclase\_100%Or0%Ab0%An
- Anorthite\_0%Or0%Ab100%An
- Bytownite\_5%Or15%Ab80%An
- Bytownite\_5%Or20%Ab75%An
- Labradorite\_5%Or35%Ab60%An
- Labradorite\_5%Or40%Ab55%An
- Anorthoclase\_15%Or65%Ab20%An
- Sanidin\_90%Or5%Ab5%An
- Sanidin\_85%Or10%Ab5%An
- Sanidin\_85%Or5%Ab10%An
- Sanidin\_80%Or15%Ab5%An
- Sanidin\_80%Or10%Ab10%An
- Sanidin\_80%Or5%Ab15%An
- Sanidin\_75%Or20%Ab5%An
- Sanidin\_75%Or15%Ab10%An
- Sanidin\_75%Or10%Ab15%An
- Sanidin\_70%Or25%Ab5%An
- Sanidin\_70%Or20%Ab10%An
- Sanidin\_70%Or15%Ab15%An
- Sanidin\_65%Or30%Ab5%An
- Sanidin\_65%Or25%Ab10%An
- Sanidin\_65%Or20%Ab15%An
- Anorthoclase\_15%Or70%Ab15%An
- Anorthoclase\_15%Or75%Ab10%An
- Anorthoclase\_15%Or80%Ab5%An
- Anorthoclase\_20%Or65%Ab15%An
- Anorthoclase\_20%Or70%Ab10%An
- Anorthoclase\_20%Or75%Ab5%An
- Anorthoclase\_30%Or65%Ab5%An

# Spectrum Tree

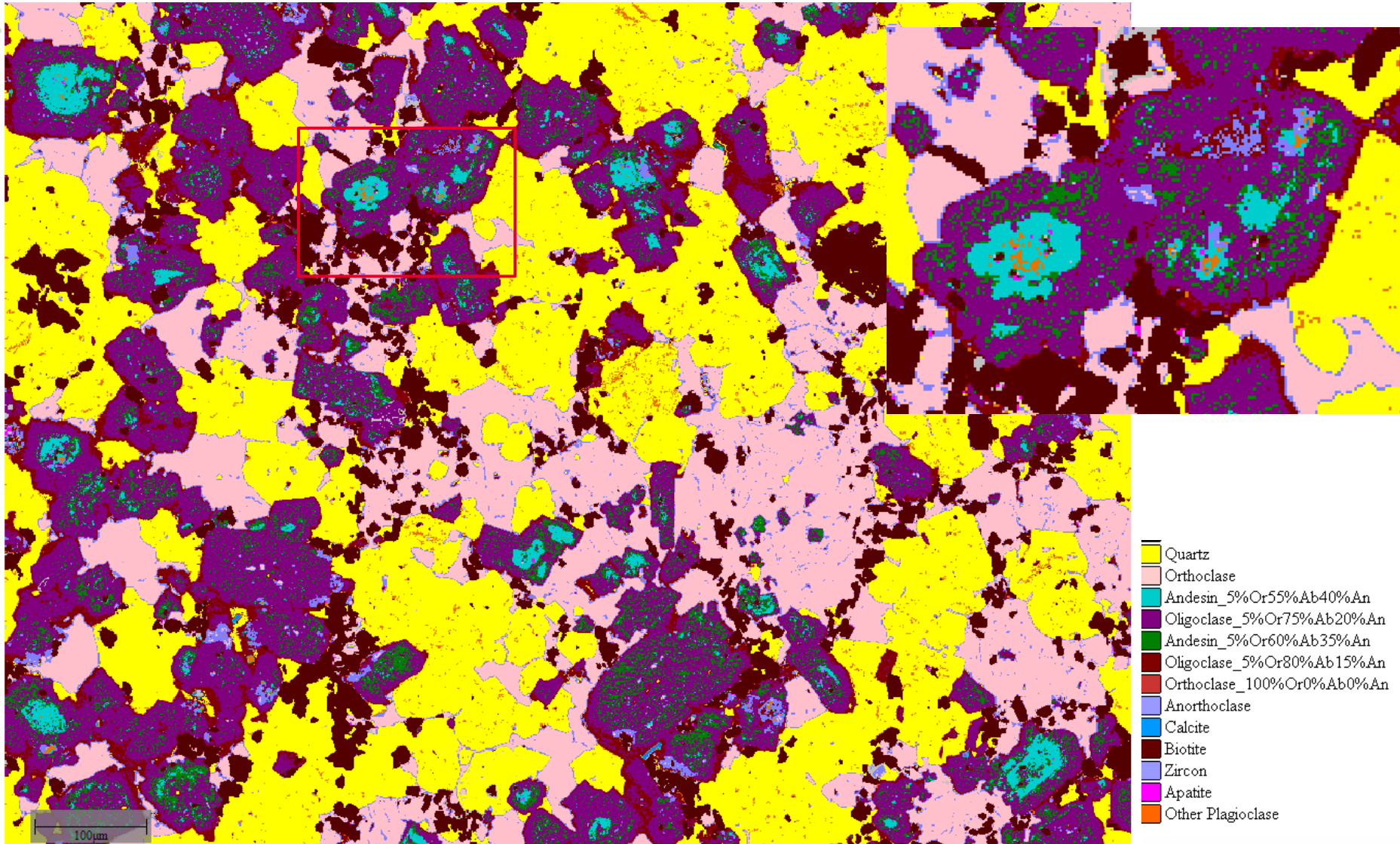
## Granite Zoned Plagioclase



200µm

- Biotite
- Quartz
- Orthoclase
- Calcite
- Zircon\_1
- Albit\_0%Or100%Ab0%An
- Andesin\_5%Or55%Ab40%An
- Andesin\_5%Or60%Ab35%An
- Oligoclase\_5%Or75%Ab20%An
- Oligoclase\_5%Or80%Ab15%An
- Orthoclase\_100%Or0%Ab0%An
- Anorthite\_0%Or0%Ab100%An
- Bytownite\_5%Or15%Ab80%An
- Bytownite\_5%Or20%Ab75%An
- Labradorite\_5%Or35%Ab60%An
- Labradorite\_5%Or40%Ab55%An
- Anorthoclase\_15%Or65%Ab20%An
- Sanidin\_90%Or5%Ab5%An
- Sanidin\_85%Or10%Ab5%An
- Sanidin\_85%Or5%Ab10%An
- Sanidin\_80%Or15%Ab5%An
- Sanidin\_80%Or10%Ab10%An
- Sanidin\_80%Or5%Ab15%An
- Sanidin\_75%Or20%Ab5%An
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- Sanidin\_75%Or10%Ab15%An
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- Sanidin\_65%Or30%Ab5%An
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- Anorthoclase\_15%Or75%Ab10%An
- Anorthoclase\_15%Or80%Ab5%An
- Anorthoclase\_20%Or65%Ab15%An
- Anorthoclase\_20%Or70%Ab10%An
- Anorthoclase\_20%Or75%Ab5%An
- Anorthoclase\_30%Or65%Ab5%An
- Quartz\_Kspar
- Apatite\_1
- Apatite\_K-spar
- Unknown
- Low\_Counts
- Un\_x\_rayed
- Shadows
- Pores

# 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



## Are There Any Questions?

Please type in the questions you might have  
in the Q&A box and press *Send*.

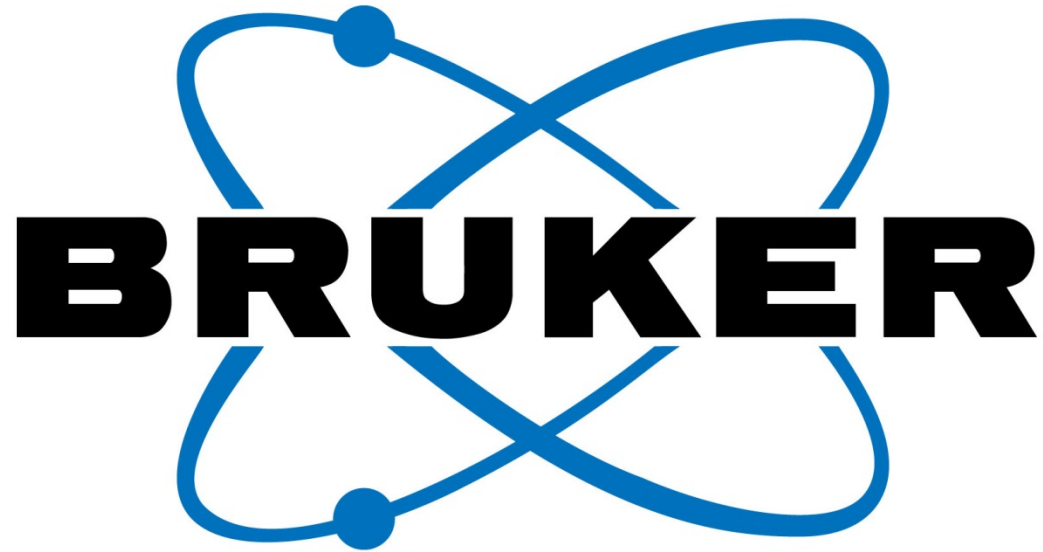
**For more information, please contact us:**

[samual.scheller@bruker.com](mailto:samual.scheller@bruker.com)

[gertrudia.gloy@bruker.com](mailto:gertrudia.gloy@bruker.com)

[info.bna@bruker.com](mailto: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