Advancements in Microanalysis with micro-XRF on SEM

Bruker Nano Analytics, Berlin, Germany
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Presenters

Stephan Boehm

Product Manager Micro-XRF/SEM
Bruker Nano Analytics, Berlin, Germany

Andrew Menzies, PhD

Applications Specialist,
Universidad Católica del Norte (Chile), Department of Geological Sciences

Overview

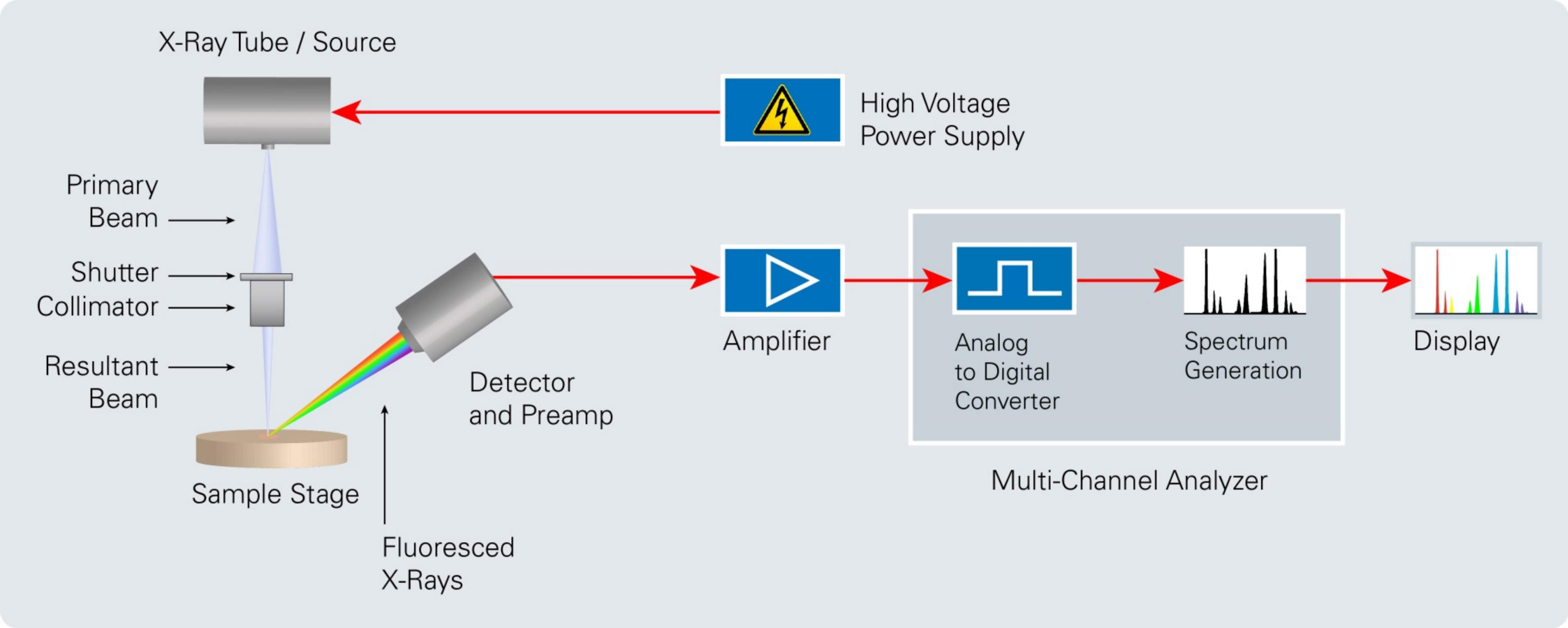
- Introduction / Presenters
- Design & principle of micro-XRF/SEM
- Differences between EDS / XRF - examples
- Applications:
  - Mantle Minerals – Diamond Exploration
  - Volcanic Fumeroles
  - Exotic Cu Deposits
  - Epithermal Quartz
  - Mineral Zonation

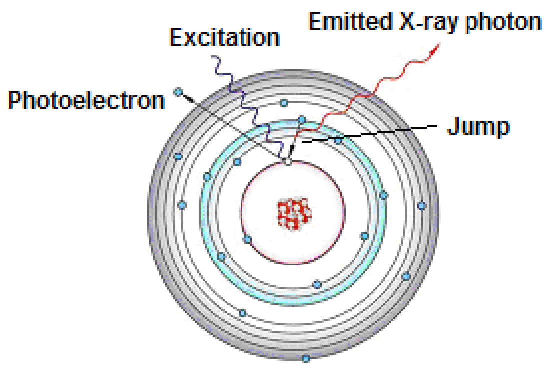
Introduction to Micro-XRF/SEM

At a glance

- Non-destructive method for elemental analysis
- Small spot analysis
- Low spectral background
- Information from within the sample
- Little or no sample prep
- Quantification
  - standard less (based on fundamental parameters)
  - standard supported FP

Introduction to Micro-XRF
Basic layout



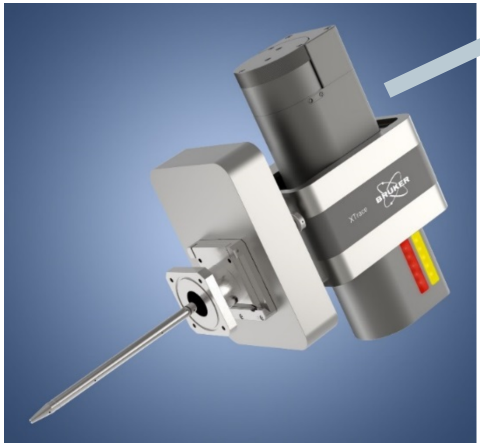


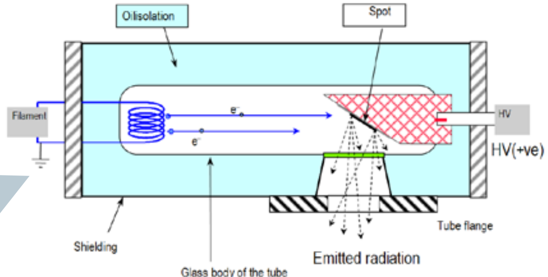
- Excitation of an atom by high energetic radiation
- Generation of a vacancy
- Fill up this vacancy by outer electrons
- Emission of a characteristic X-ray photon
- Fluorescence yield depends on atomic number

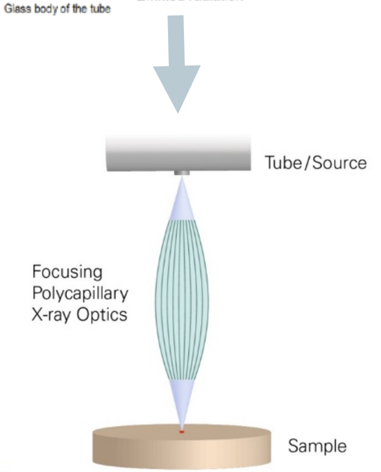
  (low for low atomic number and vice versa)

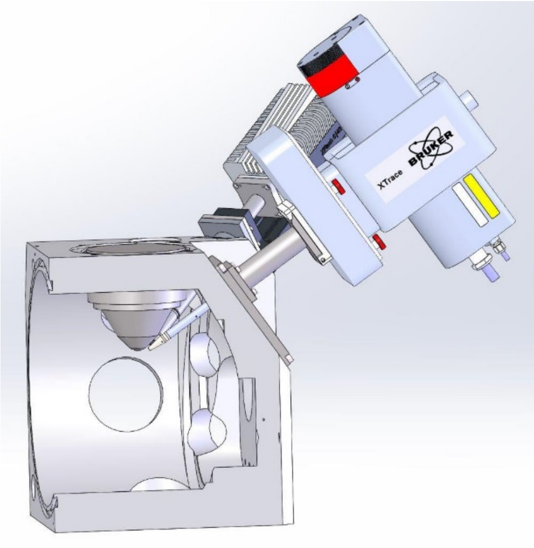
Bruker micro-XRF source for SEM´s
Hardware design

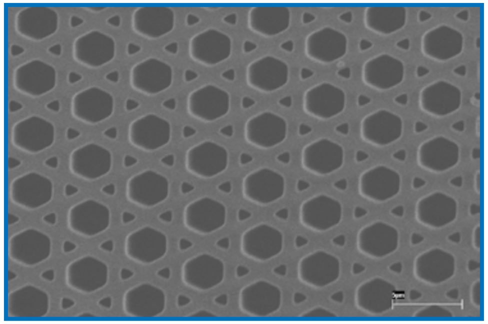
- Main components are a micro-focus X-Ray tube with a focusing optic.
- These components will be adapted to an inclined SEM port
- Other components are a High voltage generator for the X-ray tube and electronic control (separate electronic box)







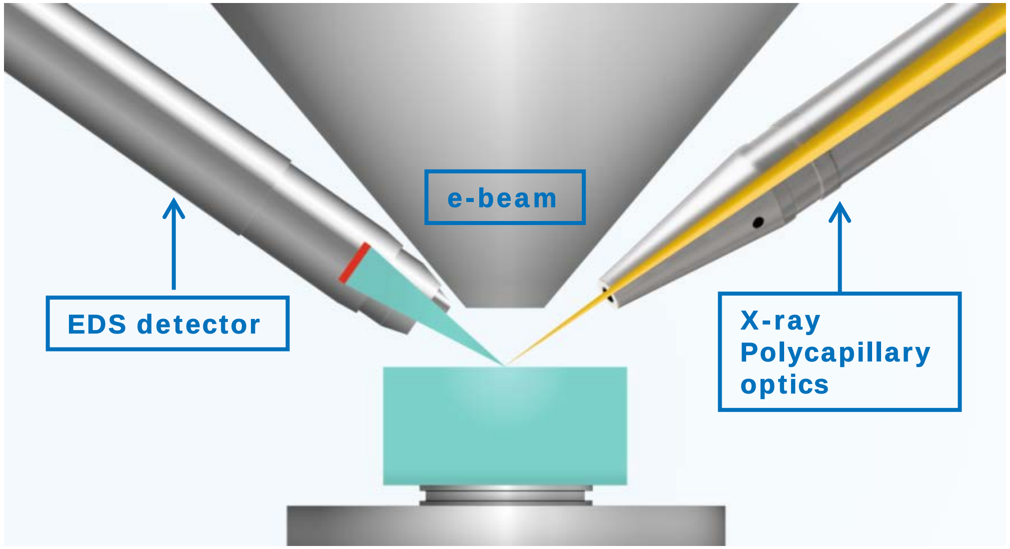


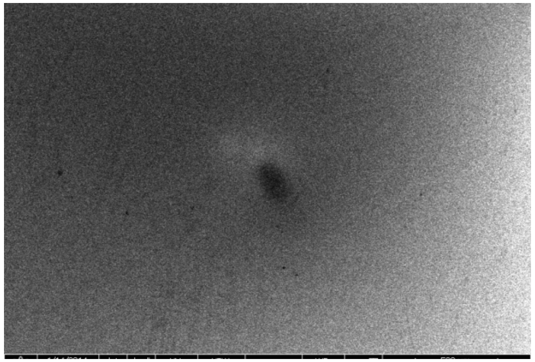


SEM image of a polycapillary structure. Inner diameter in the range of 2 µm

Bruker micro-XRF source for SEM´s
Working regime

- Since the measurement position is given by the e-beam the X-ray source is adjustable in order to align the X-ray spot to the e-beam focus for the given WD
- XTrace is aligned to a given SEM WD → sample focusing will be done by SEM stage
- Focal distance of X-ray optics is ~ 16 mm → no interference with BSD



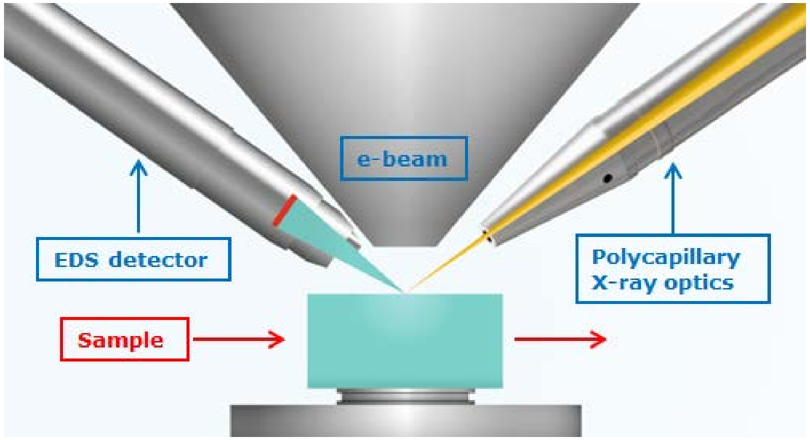


Uncoated glass makes the X-ray spot visible

Differences between SEM/EDS - XRF

XRF Elemental Mapping

- **Working principle:**
  - Polycapillary X-ray optics is fixed, changing the measurement position will be done by moving the stage



Principle of XRF/ SEM stage map

- **Differences to e-beam map**
  - Spatial resolution for e-beam is better due to the smaller spot
  - E-beam elemental map is faster because beam deflection is very fast compared to stage movement
  - The information depth for X-ray excitation is larger, i.e. deeper sample regions contribute to the measured signal
  - For not flat samples the measured distribution are influenced by the direction of excitation and detection. This means shadowing effects are possible due to the inclined angle for X-ray excitation or detection

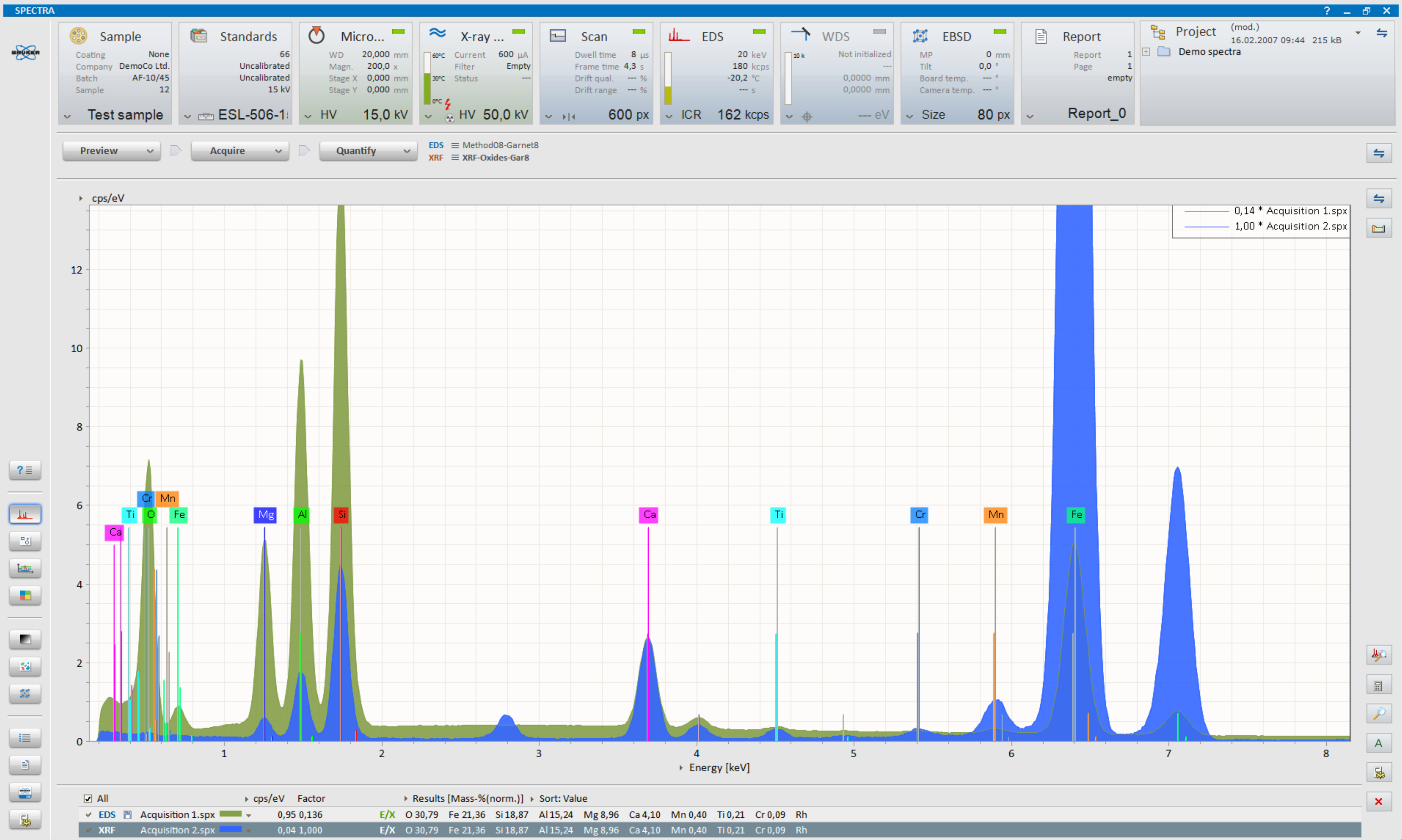
Bruker micro-XRF source for SEM´s
Software

- What kind of data we can obtain? Same data as for e-beam excitation:
  - Multipoint measurements
  - Line scan
  - Elemental mapping

- Software package for XRF spectra evaluation and quantification is completely integrated in Bruker ESPRIT 2.x EDS software which means beside SEM control und EDS analysis:
  - the control of the tube parameters,
  - measurement time,
  - XRF element distribution measurements (mapping, line-scan)
  - spectrum accumulation and their complete evaluation will be performed by one software package which makes the handling easy to use

Bruker micro-XRF source for SEM´s

ESPRIT 2.x Software



XTrace Installation´s

Important parameters and analytical conditions

micro-XRF and e-beam systems during automated mineralogical analysis

micro-XRF	Parameter	e-beam (SEM)
Ø: 15-30 µm Information depth: µm to mm; (depending on analysed element and matrix)	**Analyzed Volume**	Ø: few micrometers Information depth: µm; (depending primarily on electron energy)
Atomic number Z ≥ 11 (sodium)	**Detectable Elements**	Atomic number Z ≥ 4 (beryllium)
20 µg/g to 100%; (depending on analysed element)	**Concentration Range**	1000 µg/g to 100%;
Generated by scattered tube radiation on the sample into the detector (second order effect)	**Spectral Background**	Generated by continuous bremsstrahlung (first order effect)
Electrical Conductivity not required	**Sample Preparation**	Sample needs to be electrically conductive (commonly carbon-coated)
Minimal	**Sample Stress**	Heating due to absorbed electrons
Standard based or standardless	**Quantification**	Standard based or standardless
Spectral fitting (hierarchical)	**Mineral Classification**	Elemental concentration ranges (hierarchical)

Data presented in the table are principally based on the Bruker M4 Tornado[AMICS] micro-XRF and the QEMSCAN (model E430) Zeiss EVO SEM. Data updated from Haschke and Böhm (2017). Values presented in the table represent typical values and normal ranges of analysis, but are not limited to these values, and under specific circumstances values outside of those present may be preferable.

Important parameters and analytical conditions

micro-XRF and e-beam systems during automated mineralogical analysis

micro-XRF		Parameter	e-beam (SEM)	
Normal Range	Typical Value	Analytical Values[3]	Typical Value	Normal Range
20 - 50	50	High Voltage Excitation (kV)	25	10 - 25
100 – 600 µA	200 µA	X-ray Excitation or Beam Current (µA / nA)	5 nA	
20 - 200	50	Pixel Spacing (µm)	5	1 - 50
10 - 500	50	Dwell Time (ms)		
		X-Ray Count	1000	1000 - 10000
90 - 275	130	Spectroscopic Amplifier (kcps)	275	
1 - 2	1	Number of Spectrometers	4	2 - 4

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Geological Applications:
XTrace Installation - UCN

Geological Aplications

- **Applications**:
  - Mantle Minerals – Diamond Exploration
  - Volcanic Fumeroles
  - Exotic Cu Deposits
  - Epithermal Quartz
  - Mineral Zonation
- **Differences between EDS / XRF – examples**
- **Point Analyses, Line Scans, and Elemental Mapping**

Geological Applications:
Ultramafic Plutonic Rocks: Definitions

Terminology

Peridotites are distinguished from pyroxemites with > 40% olivine

Peridotites are subdivided into:

- **dunite, harzburgite, lherzolite and wehrlite**

Pyroxenites are subdivided into :

- **orthopyroxenite**, **websterite**, y **clinopyroxenite**

Streckeisen (1973, 1976)

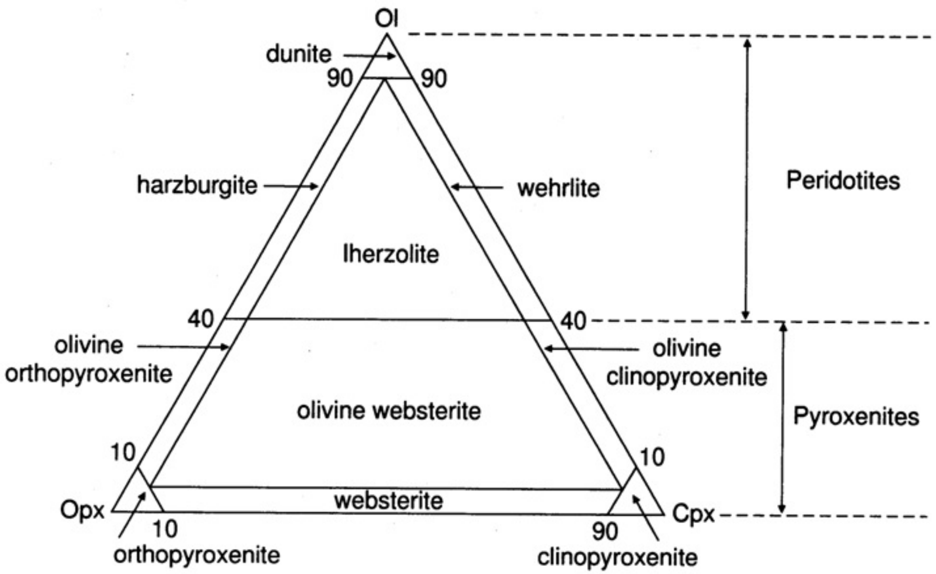


Figure 8. *Classification of peridotites and pyroxenites as recommended by the International Union of Geological Sciences. Modal proportions are in volume percent. Reproduced from Dawson (1980).*

Geological Applications:
Mantle Minerals

Mantle Minerals		
Garnet	Peridotite Pyropo	Red Purple
Orthopyroxene	Peridotite Enstatite	Green
Spinel	Peridotite Chromite	Black
Olivine	Peridotite	Green
Ilmenite	Peridotite	Black
Garnet	Eclogite Pyropo-Almandine	Orange
Clinopyroxene	Peridotite Cr-Diopside	Green

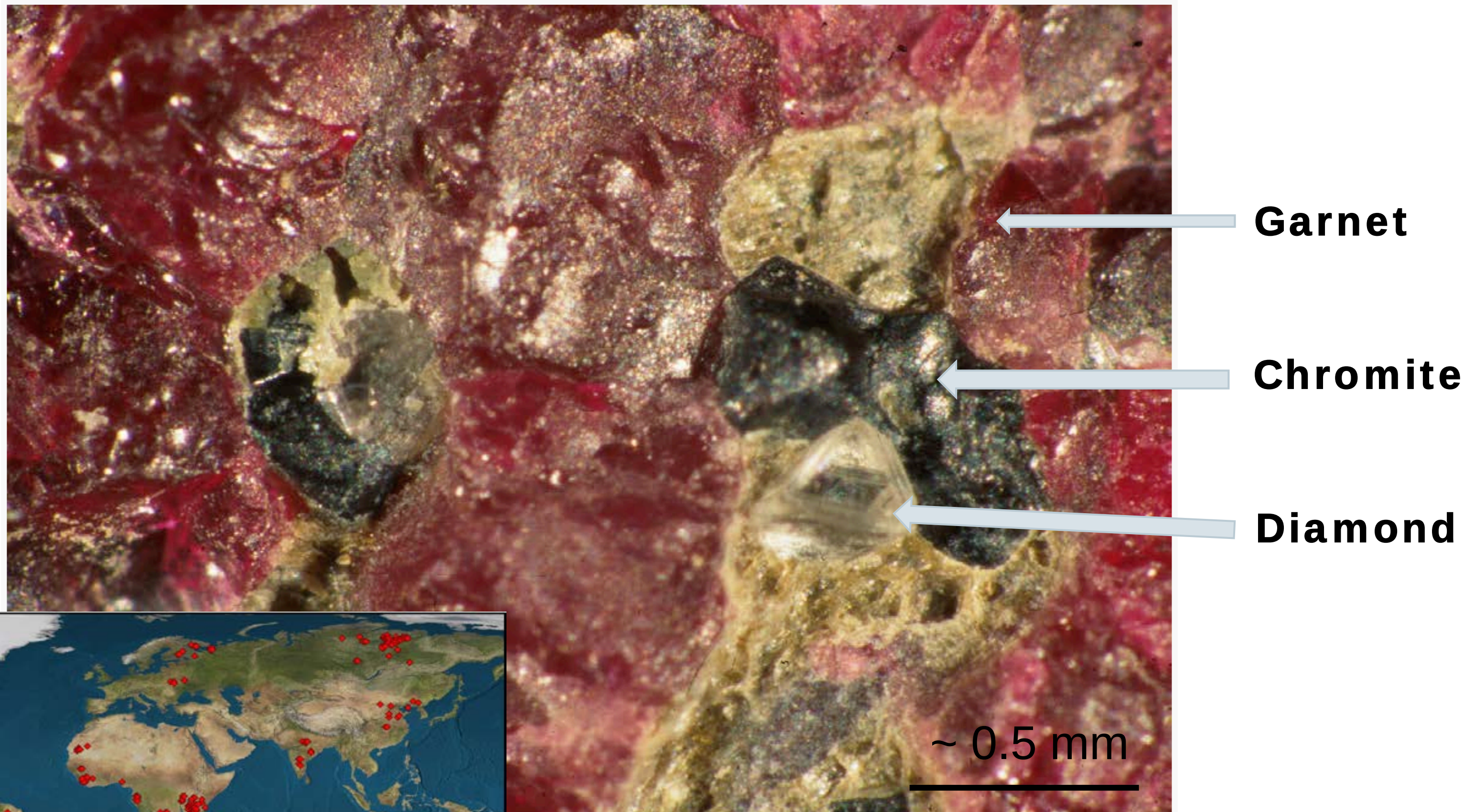
Kimberlite Indicator Minerals (KIM´s)

Geological Applications:
Mantle Minerals - Composition

Garnet	• $(Ca,Mg,Fe)_3(Al,Cr,Fe^{3+})_2(SiO4)_3$ • Minor: Mn y Ti, y Trace: Ni
Olivine	• $(Mg,Fe)_2SiO_4$ • Minor: Ca, Mn, y Trace: Ni
Clinopyroxene	• $(Ca,Na)(Mg,Fe,Al)(Si,Al)_2O_6$ • Minor: Cr
Orthopyroxene	• $(Mg,Fe)SiO_3$ • Minor: Al
Spinel	• $(Mg,Fe)(Al,Cr,Fe^{3+})_2O_4$ • Minor: Ti

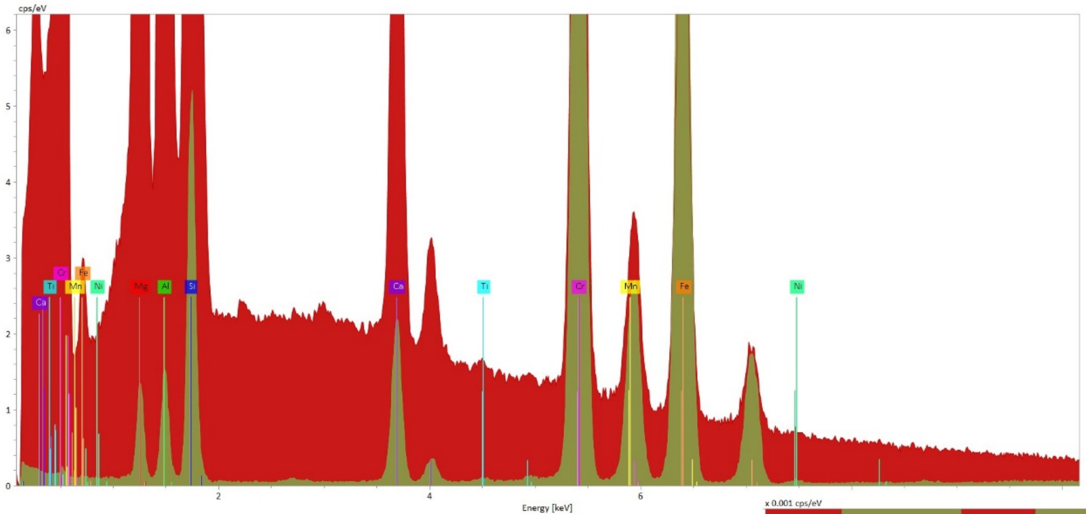
Geological Applications:

Mantle Minerals - Diamonds



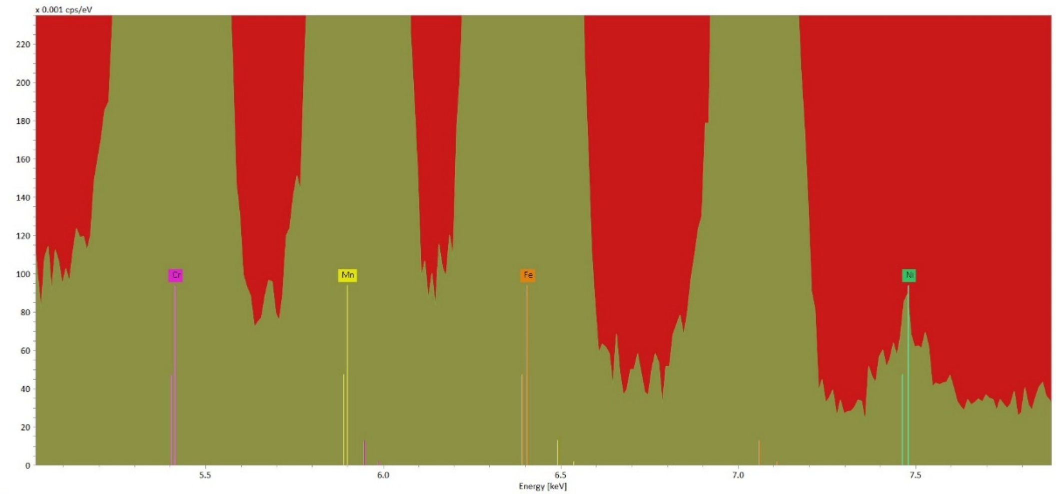
Geological Applications:
Point Analysis – Mantle Minerals

Comparison of SEM-EDS and XRF-EDS spectra for the same point.



Sample AHM-D2

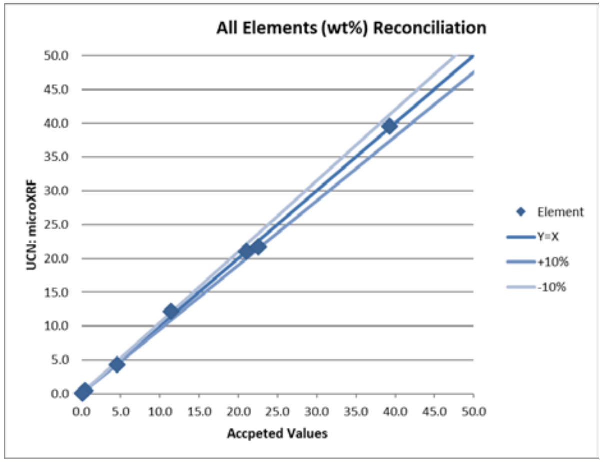
Peridotitic garnet



Geological Applications:
Point Analysis – Mantle Minerals

Comparison XRF-EDS spectra for the same point: analytical precision

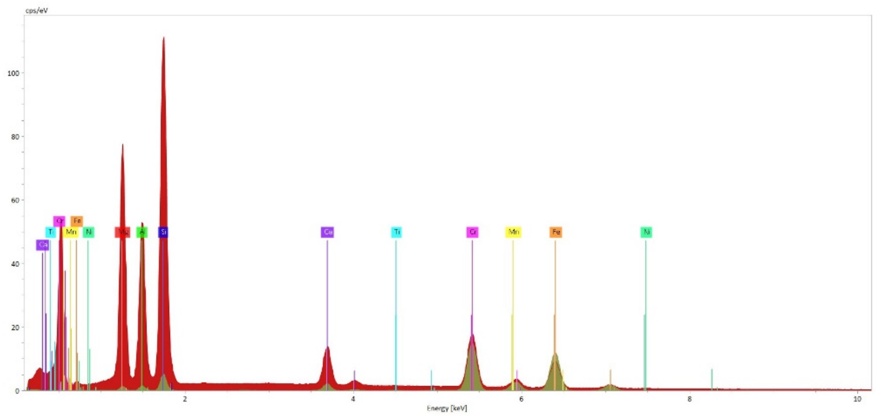
Element	Unit	EPMA Values	XRF-EDS Values	Std Dev	Maximum	Minimum	Range
SiO2	(%)	39.28	39.22	0.18	39.48	38.89	0.58
TiO2	(%)	0.28	0.30	0.02	0.33	0.26	0.07
Al2O3	(%)	22.51	21.68	0.31	22.21	21.23	0.98
Cr2O3	(%)	0.12	0.11	0.01	0.11	0.09	0.02
FeO	(%)	21.00	21.41	0.20	21.85	21.18	0.67
MnO	(%)	0.47	0.47	0.01	0.50	0.46	0.04
MgO	(%)	11.44	12.22	0.32	12.62	11.57	1.05
CaO	(%)	4.57	4.36	0.05	4.45	4.29	0.16

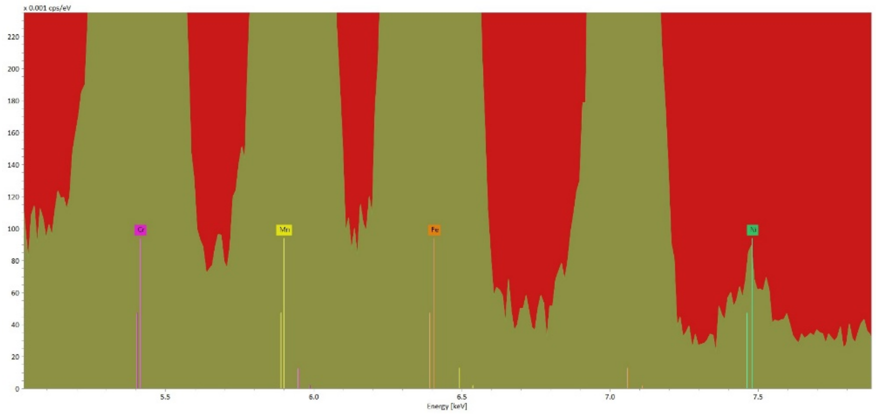


Geological Applications:
Point Analysis – Mantle Minerals

Major and trace elements

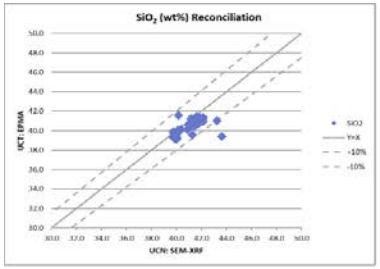
Element	Unit	90 sec	120 sec	180 sec
SiO2	(%)	39.04	39.17	39.20
TiO2	(%)	0.28	0.28	0.29
Al2O3	(%)	22.23	21.97	21.87
Cr2O3	(%)	0.11	0.11	0.11
FeO	(%)	21.16	21.05	21.02
MnO	(%)	0.49	0.48	0.48
MgO	(%)	12.29	12.57	12.63
CaO	(%)	4.35	4.31	4.33
Ni	(ppm)	26	18	28
Cu	(ppm)	3	5	4
Zn	(ppm)	173	143	150
Ga	(ppm)	7	0	28
Ge	(ppm)	17	22	17
As	(ppm)	28	28	28
Rb	(ppm)	41	69	59
Sr	(ppm)	28	0	28
Y	(ppm)	2	28	3
Zr	(ppm)	157	157	171
Nb	(ppm)	1	28	0

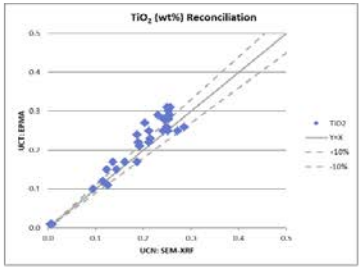


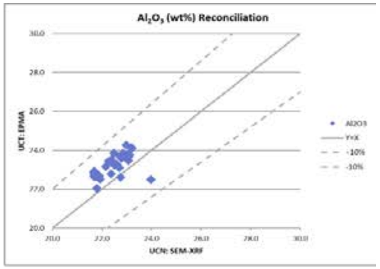


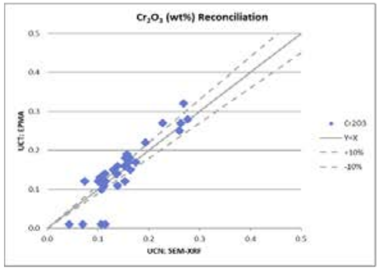
Geological Applications:
Point Analysis – Mantle Minerals

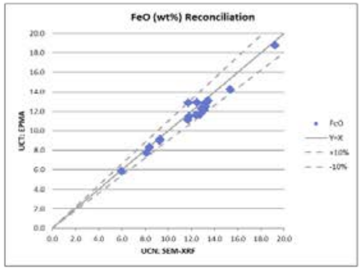
Comparison of analytical results on a per element basis between EPMA and SEM-XRF (Xtrace) for 33 eclogitic garnets from Newlands kimberlite in South Africa.

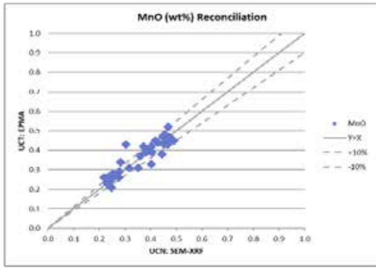


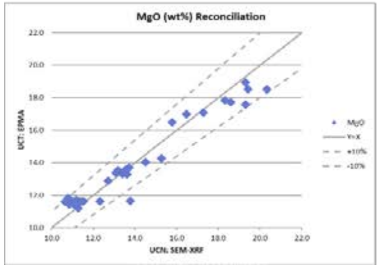


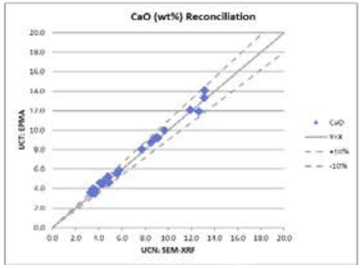


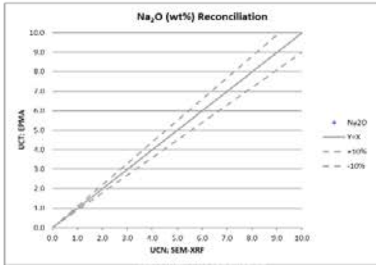






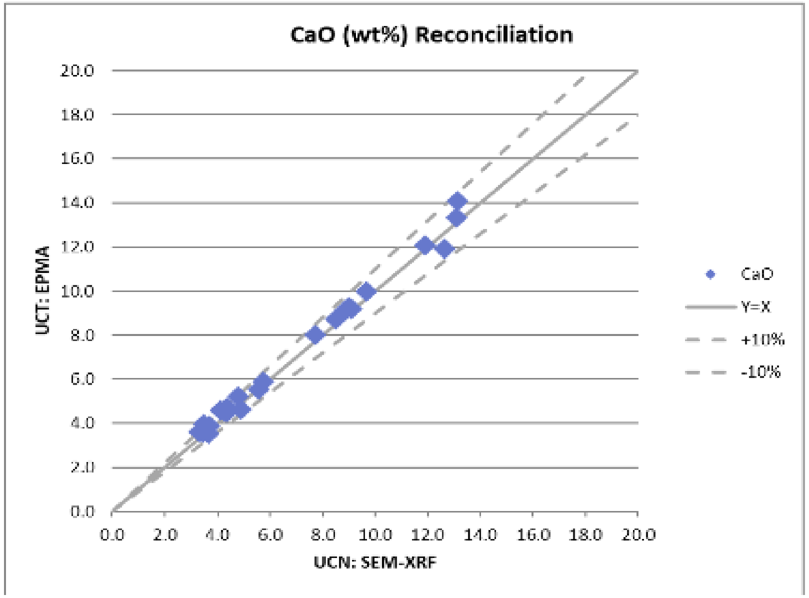


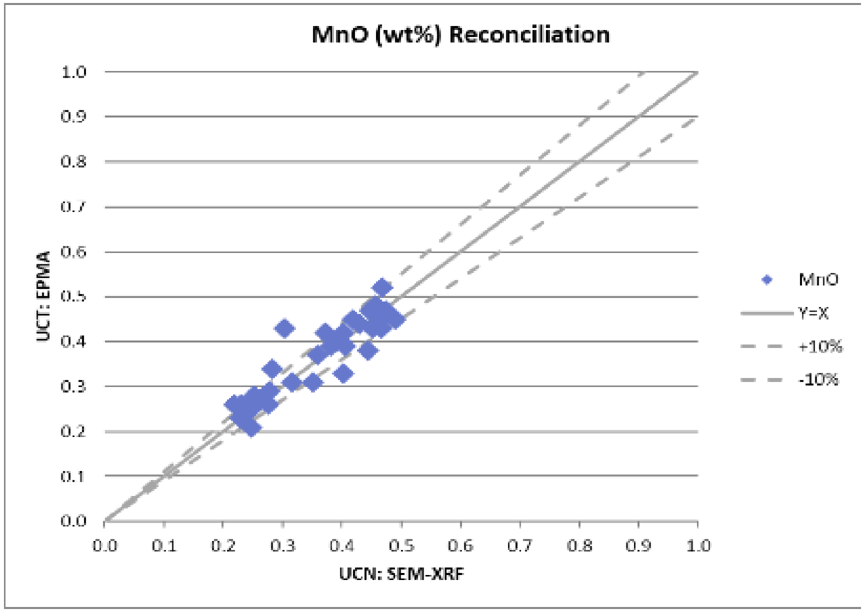




Geological Applications:
Point Analysis – Mantle Minerals

Comparison of analytical results on a per element basis between EPMA and SEM-XRF (Xtrace) for 33 eclogitic garnets from Newlands kimberlite in South Africa.

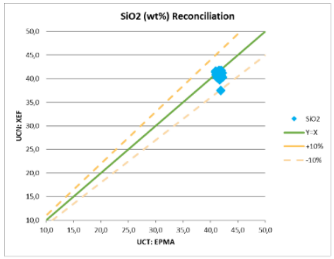


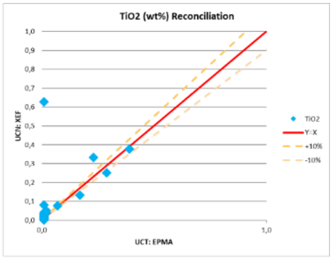


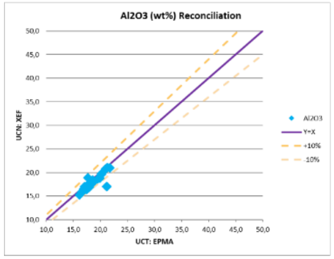
Geological Applications:

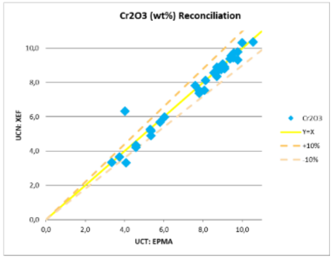
Point Analysis – Mantle Minerals

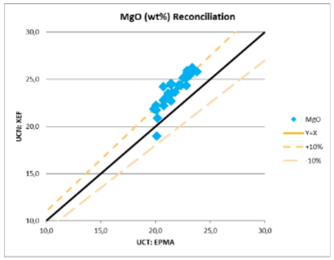
Comparison of analytical results on a per element basis between EPMA and SEM-XRF (Xtrace) for 34 peridotitic garnets from Newlands kimberlite in South Africa.

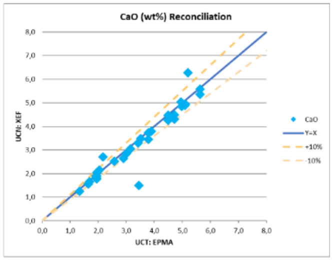


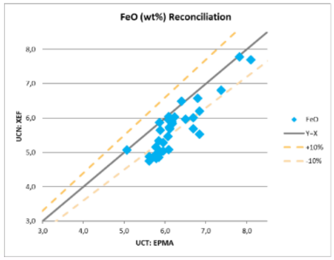


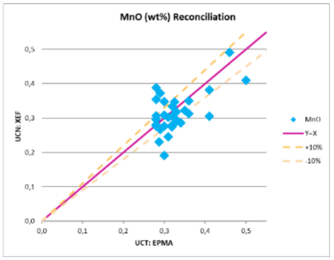


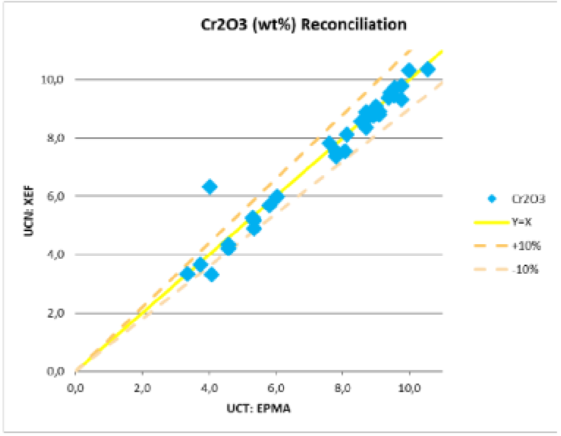


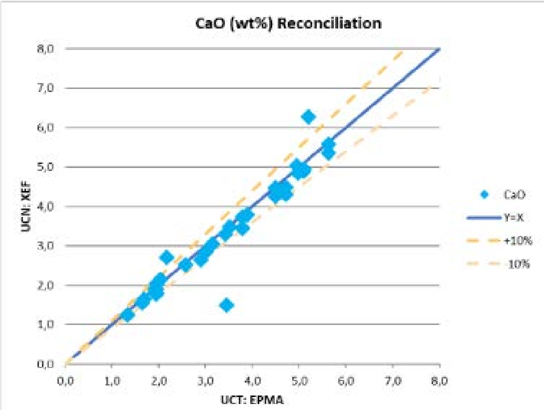




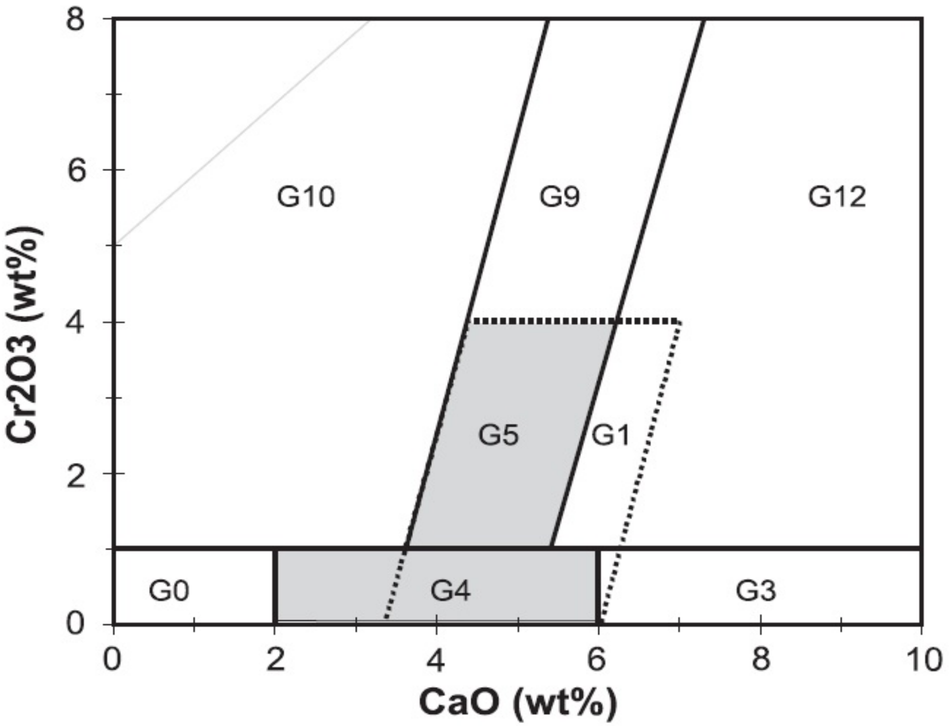


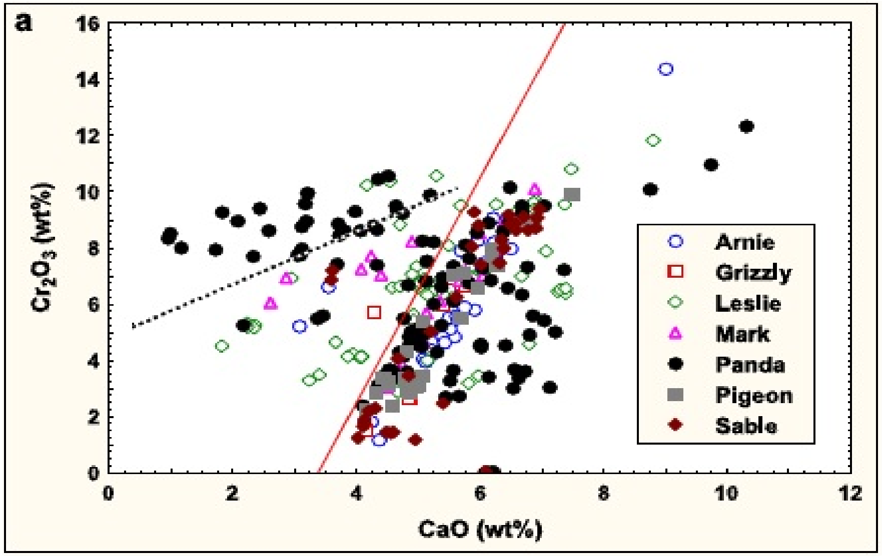






Geological Applications:
Garnet: Cr_2O_3 - CaO





Reference: Grütter et al., Lithos 77 (2004) 841–857.

Solid line (Gurney,1984) separates G10 (sub-calcic) and G9 (calcic) fields.

Dashed line (Grütter and Sweeney, 2000) represents the graphite–diamond constraint (GDC).

Geological Applications:
Point Analysis – Mantle Minerals

(i) EMPA

(ii) SEM-EDS

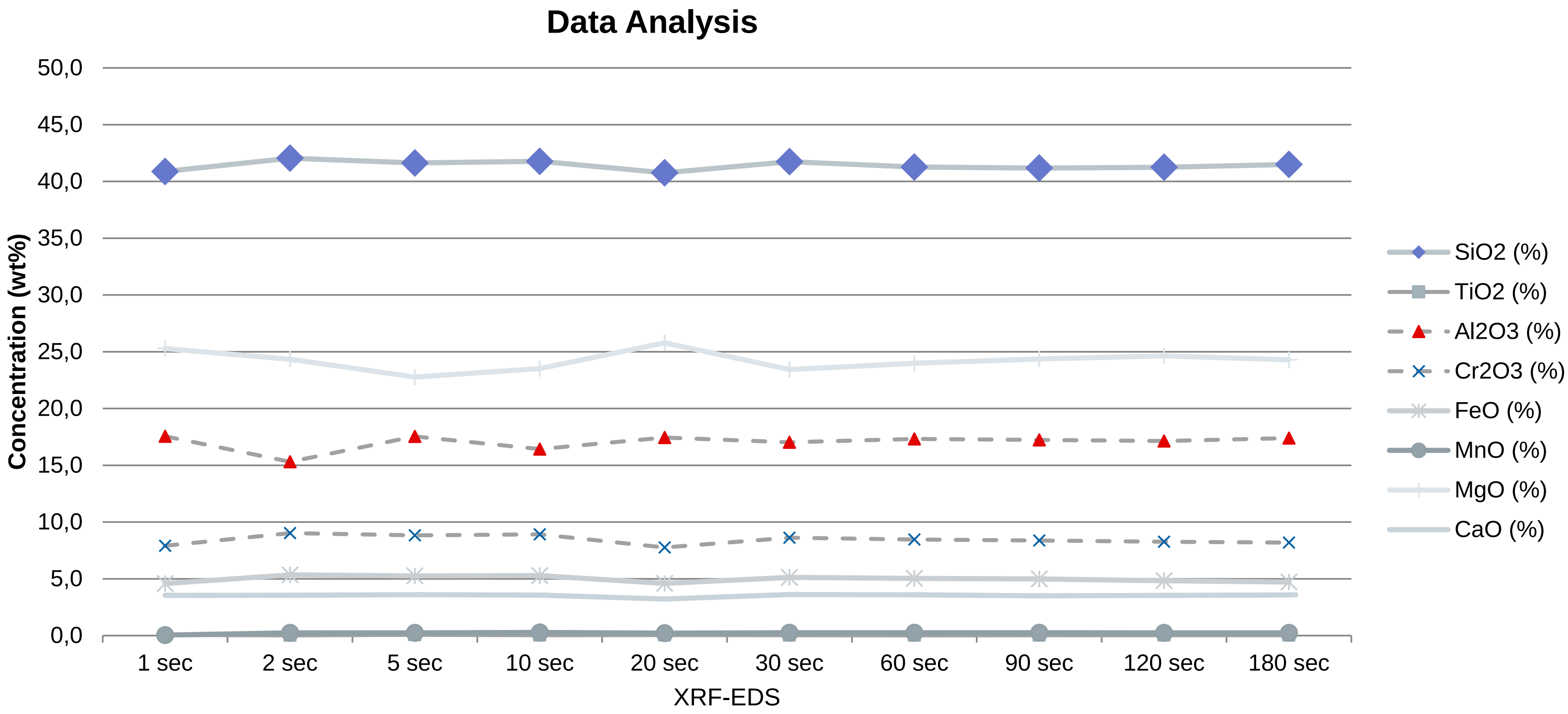
(iii) XTrace

	Garnet	Garnet	Garnet	Garnet	Garnet	Garnet	Garnet	Garnet	Garnet	Garnet
	AHM K1	AHM K2	AHM K3	AHM K4	AHM K5	AHM K6	AHM K7	AHM K8	AHM K9	AHM K10
	1	2	3	4	5	6	7	8	9	10
UCT (EMPA)										
SiO_2	39.28	40.06	39.43	39.42	39.55	39.91	39.14	40.16	39.80	39.35
TiO_2	0.28	0.31	0.28	0.30	0.26	0.28	0.29	0.25	0.25	0.29
Al_2O_3	22.51	22.92	22.05	22.49	22.61	22.77	22.66	23.16	22.89	22.70
Cr_2O_3	0.12	0.10	0.01	0.12	0.15	0.11	0.01	0.01	0.13	0.14
FeO	21.00	20.57	20.73	20.85	21.06	20.91	21.10	18.78	20.42	20.97
MnO	0.47	0.43	0.45	0.43	0.44	0.47	0.46	0.42	0.47	0.52
MgO	11.44	11.74	11.19	11.65	11.60	11.62	11.58	12.87	11.61	11.80
CaO	4.57	4.68	4.61	4.56	4.50	4.51	4.69	5.21	4.57	4.62
Na_2O	0.08	0.08	0.11	0.09	0.11	0.10	0.09	0.12	0.09	0.10
UCN (SEM-EDS)										
SiO_2	39.74	39.74	39.77	41.33	39.91	39.76	39.93	40.22	39.80	39.82
TiO_2	0.26	0.27	0.25	0.23	0.27	0.26	0.26	0.26	0.27	0.26
Al_2O_3	22.57	22.50	22.59	23.57	22.57	22.58	22.55	22.96	22.60	22.67
Cr_2O_3	0.10	0.11	0.10	0.07	0.12	0.10	0.08	0.03	0.11	0.09
FeO	20.94	20.99	20.90	18.14	20.83	20.92	20.89	18.50	20.88	20.78
MnO	0.47	0.47	0.46	0.40	0.47	0.48	0.47	0.42	0.47	0.47
MgO	11.35	11.36	11.35	12.14	11.45	11.38	11.28	12.61	11.34	11.36
CaO	4.56	4.56	4.58	4.11	4.38	4.51	4.54	5.00	4.53	4.55
Na_2O										
UCN (XTrace)										
SiO_2	40.02	40.14	39.90	43.63	41.27	40.10	40.02	40.39	40.13	39.69
TiO_2	0.25	0.26	0.25	0.25	0.25	0.24	0.26	0.24	0.25	0.23
Al_2O_3	21.92	21.69	21.79	23.98	22.76	21.69	21.74	22.16	21.67	21.89
Cr_2O_3	0.10	0.11	0.11	0.07	0.13	0.11	0.11	0.04	0.10	0.11
FeO	21.87	22.06	21.86	13.58	20.14	22.05	22.07	19.23	21.92	21.93
MnO	0.45	0.47	0.47	0.30	0.43	0.47	0.47	0.40	0.47	0.47
MgO	11.06	10.86	11.28	13.77	10.66	11.01	10.97	12.72	11.14	10.78
CaO	4.33	4.42	4.35	4.41	4.37	4.32	4.37	4.81	4.31	4.90
Na_2O										

Geological Applications:
Point Analysis – Mantle Minerals

Comparison XRF-EDS spectra for the same point: analytical time

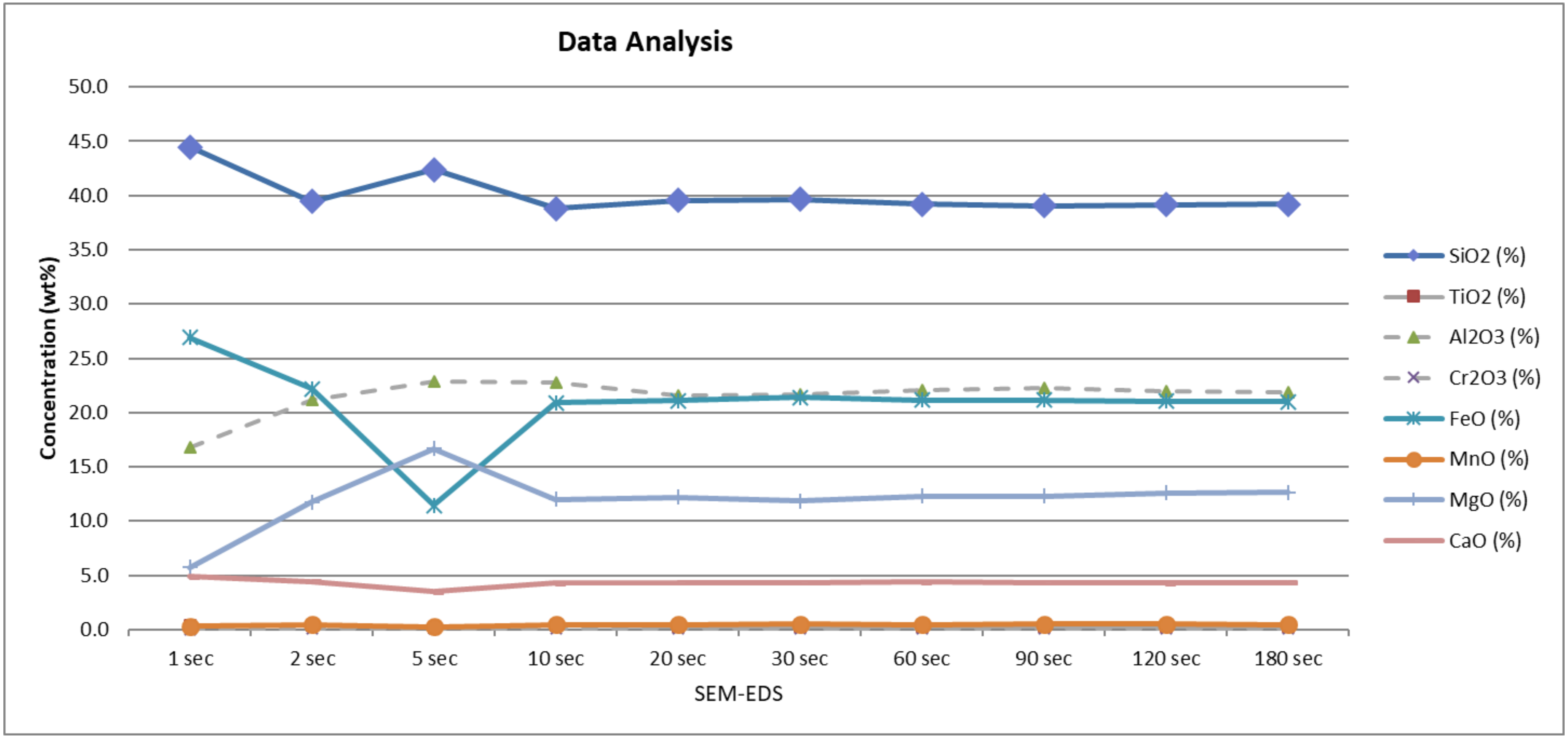
Peridotitic Garnet



Geological Applications:
Point Analysis – Mantle Minerals

Comparison XRF-EDS spectra for the same point: analytical time

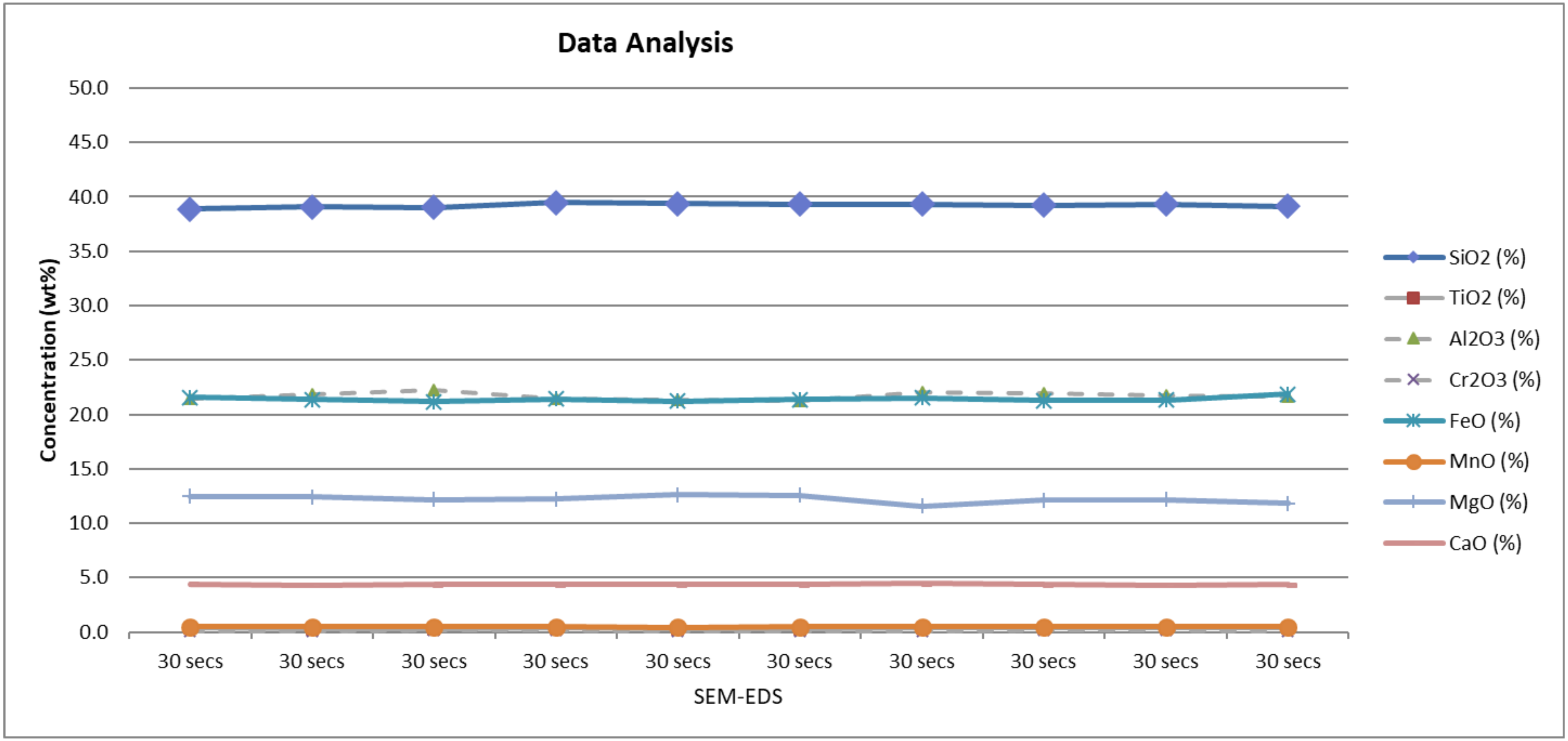
Eclogitic Garnet



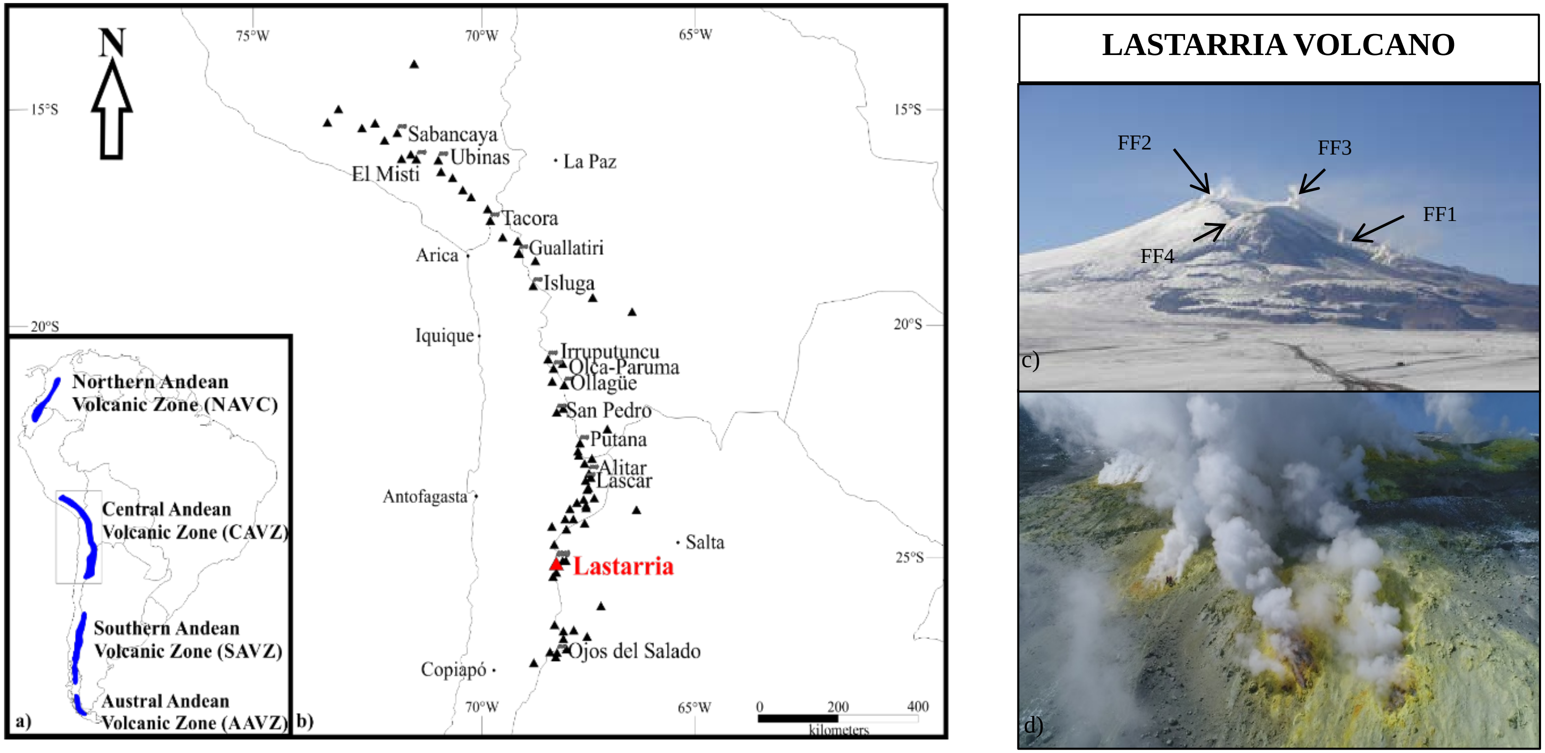
Geological Applications:
Point Analysis – Mantle Minerals

Comparison XRF-EDS spectra for the same point: analytical precision

Eclogitic Garnet

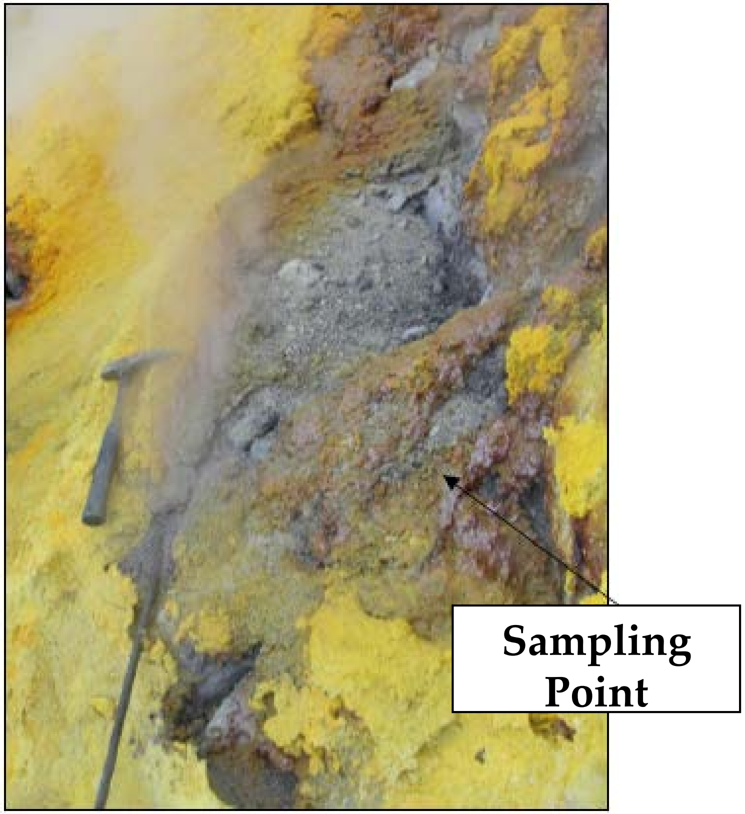


Geological Applications:
Volcanic Fumeroles



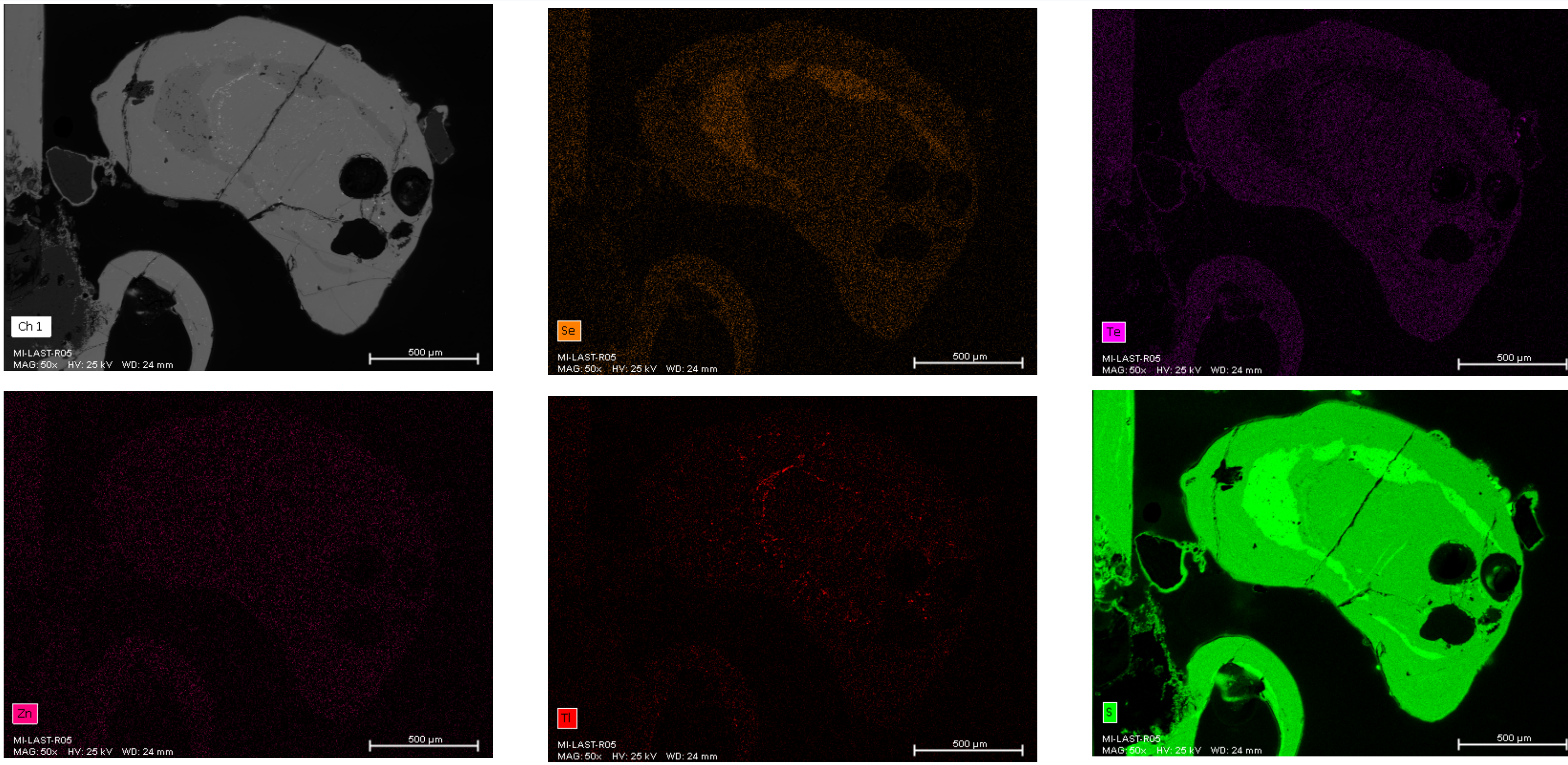
Location map of Lastarria volcano and fumarolic fields: a) South America and their respective volcanic zones; b) the Central Andean Volcanic Zone; c) Lastarria volcano and their four fumarolic fields (FF); d) Zoom to fumarolic field 1.

Geological Applications:
Volcanic Fumeroles



Images of sample MI-LAST-R05 and its sample point from Lastarria volcano.

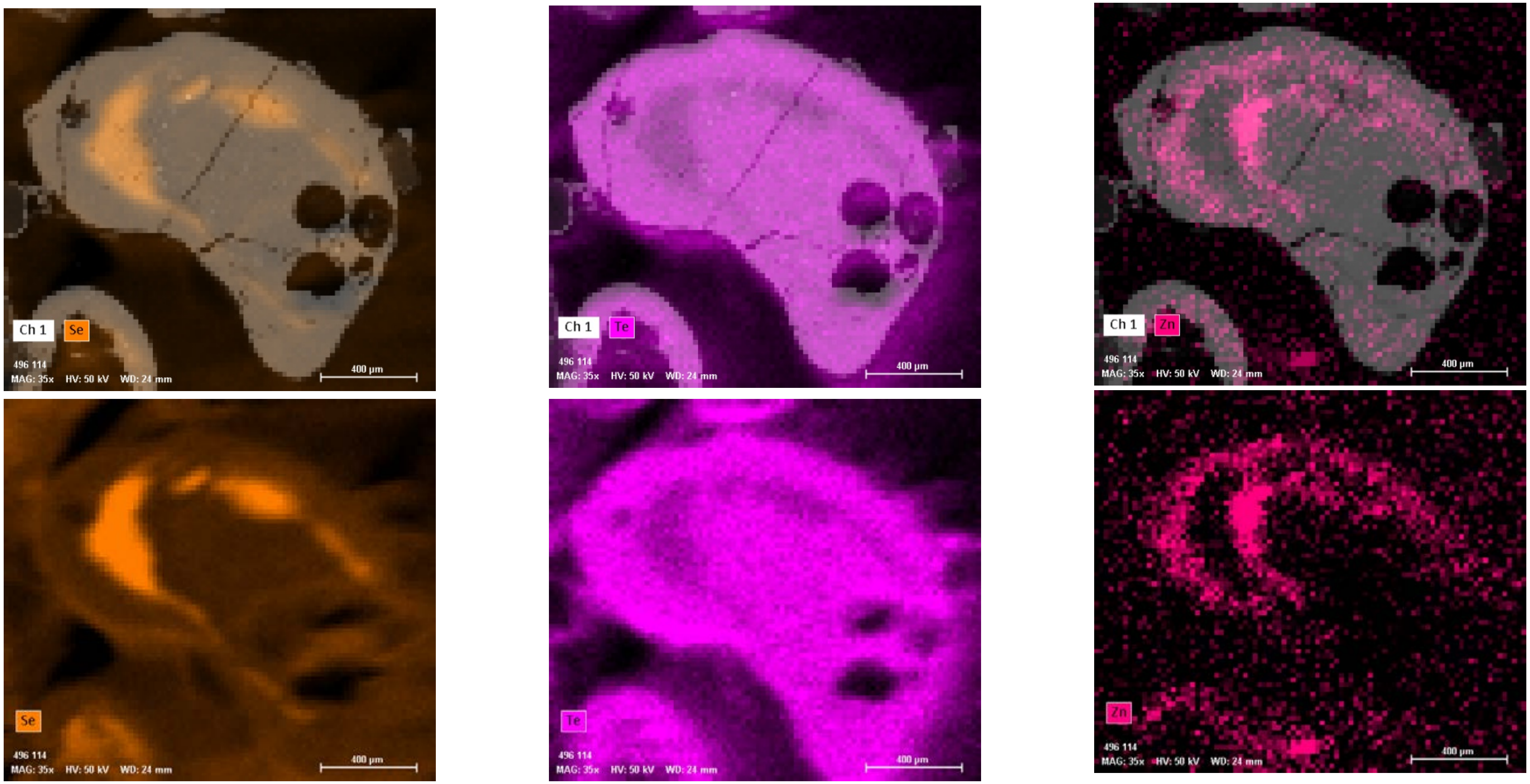
Geological Applications:
Volcanic Fumeroles



SEM-EDS (x-beam) elemental maps and BSE image of sample MI-LAST-R05. Pixel size 6 um.

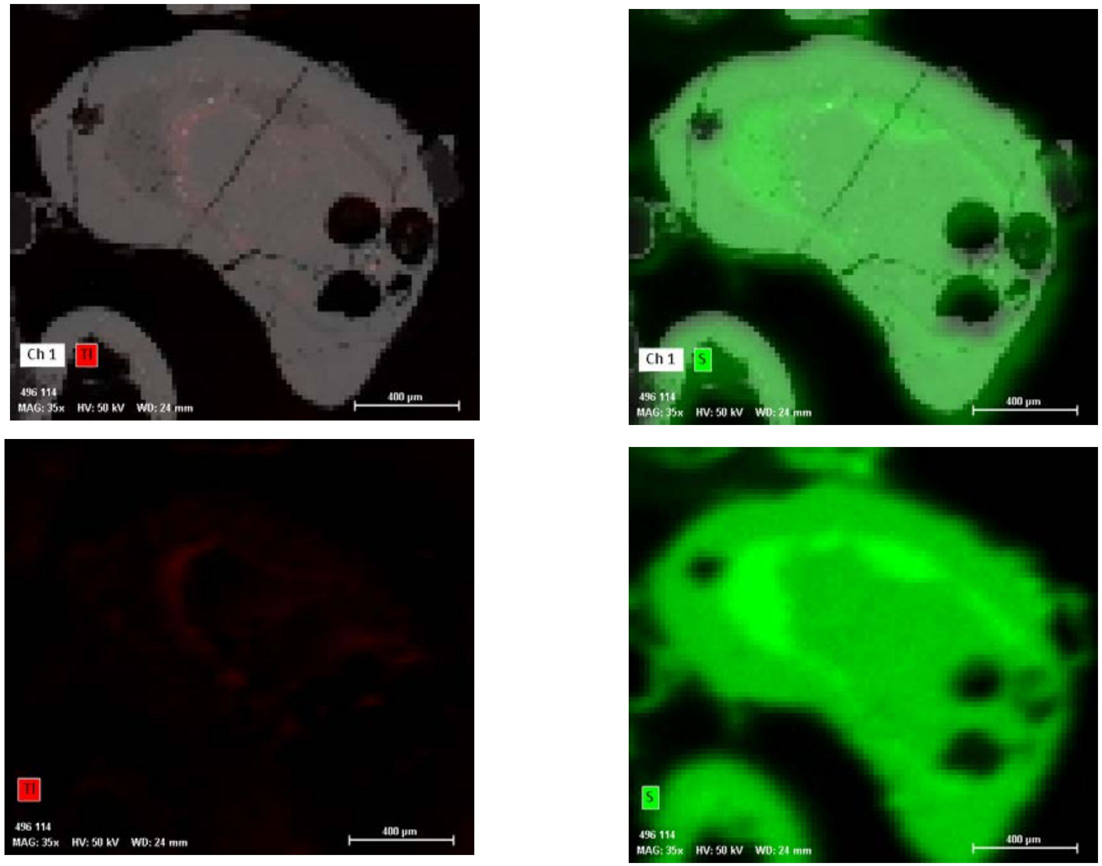
Top Row: BSE Image, Se, and Te. Bottom Row: Zn, Tl and S distributions.

Geological Applications:
Volcanic Fumeroles



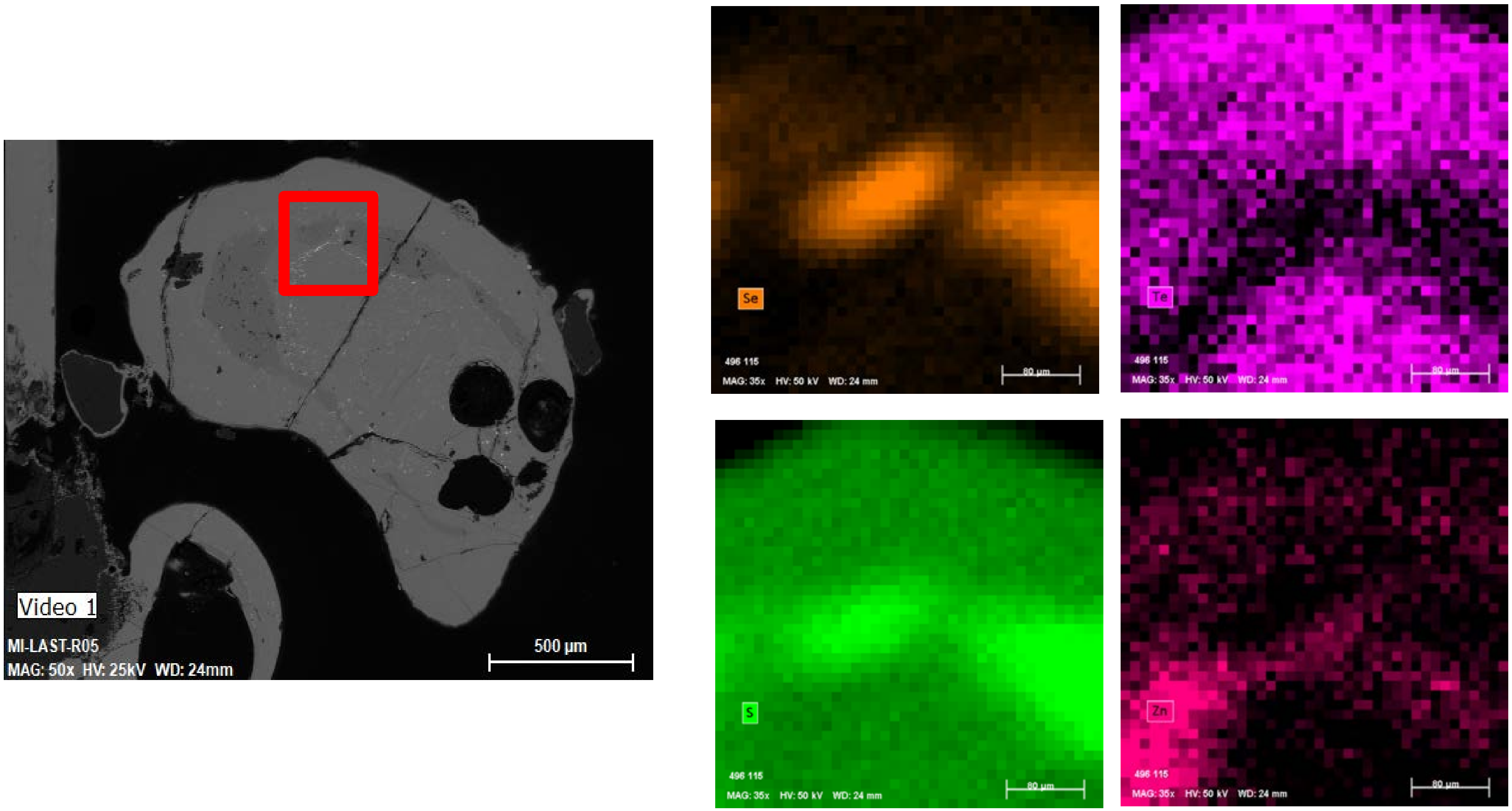
XRF-EDS elemental maps and BSE image of sample MI-LAST-R05.
Pixel size 20 um. Dwell time 5s.
Top Row: BSE Image overlain with Se, Te and Zn distributions.
Bottom Row: Se, Te and Zn distributions. .

Geological Applications:
Volcanic Fumeroles



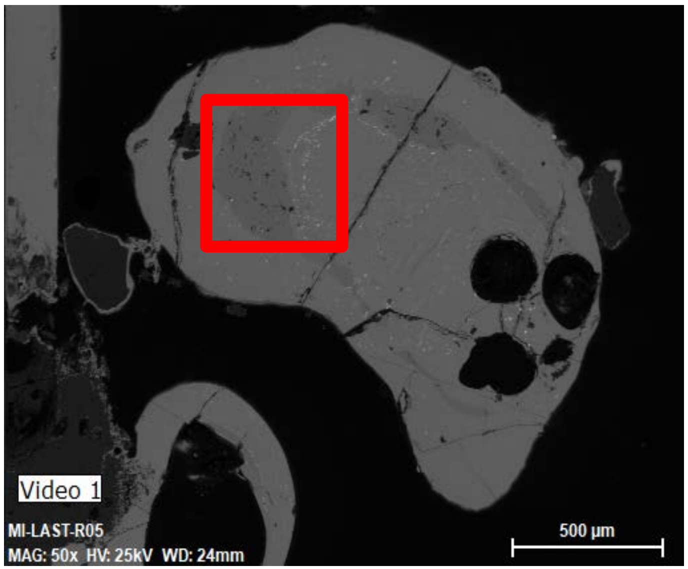
XRF-EDS elemental maps and BSE image of sample MI-LAST-R05.
Pixel size 20 um. Dwell time 5s.
Top Row: BSE Image overlain with Tl and S distributions.
Bottom Row: Tl and S distributions.

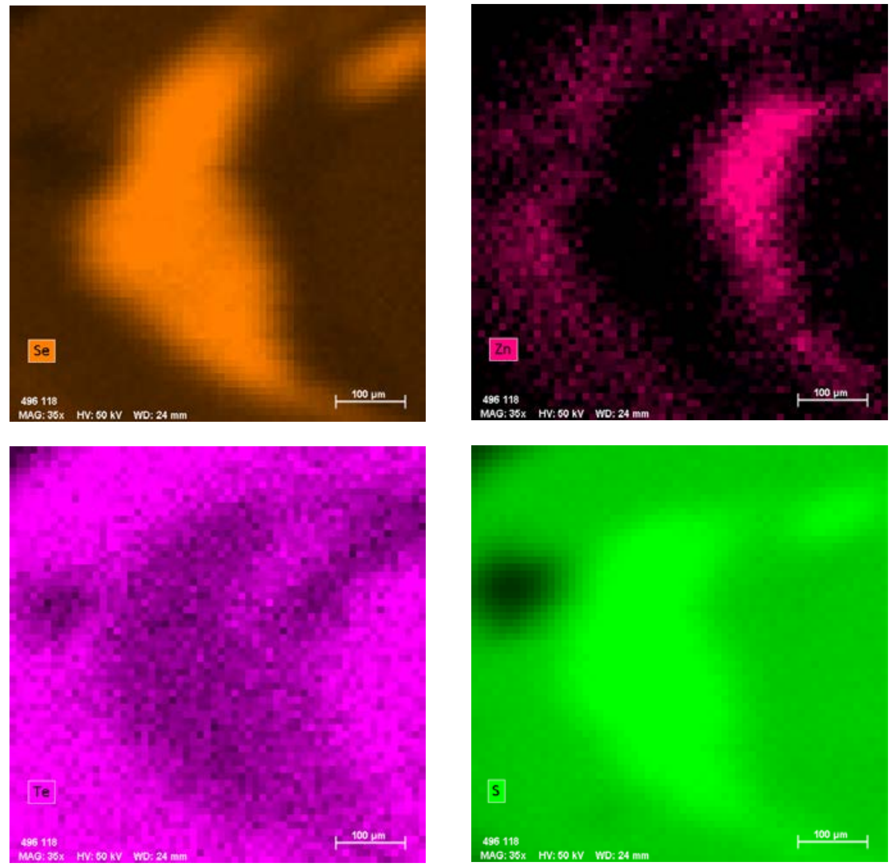
Geological Applications:
Volcanic Fumeroles



BSE Image and XRF-EDS elemental maps for Se, Te, Zn and S of indicated area from sample MI-LAST-R05.
Pixel size 10 um. Dwell time 1s.

Geological Applications:
Volcanic Fumeroles

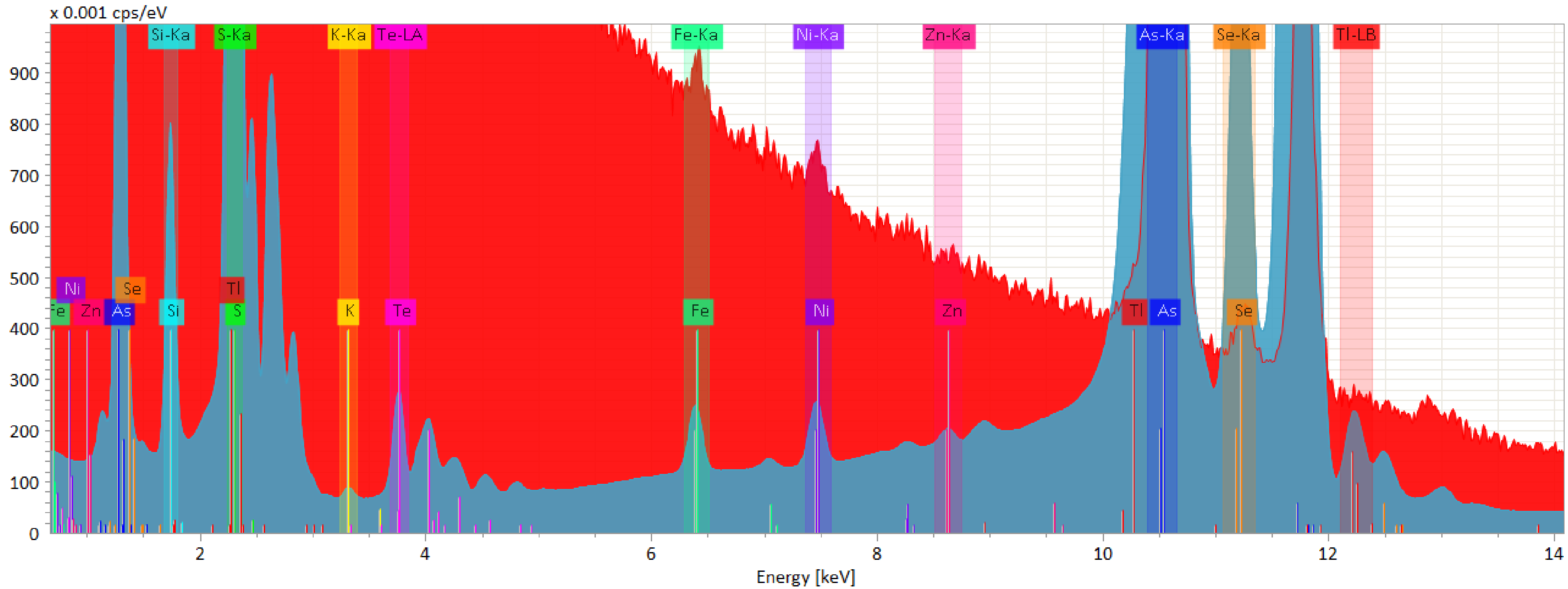




BSE Image and XRF-EDS elemental maps for Se, Te, Zn and S of indicated area from sample MI-LAST-R05.
Pixel size 10 um. Dwell time 5s.

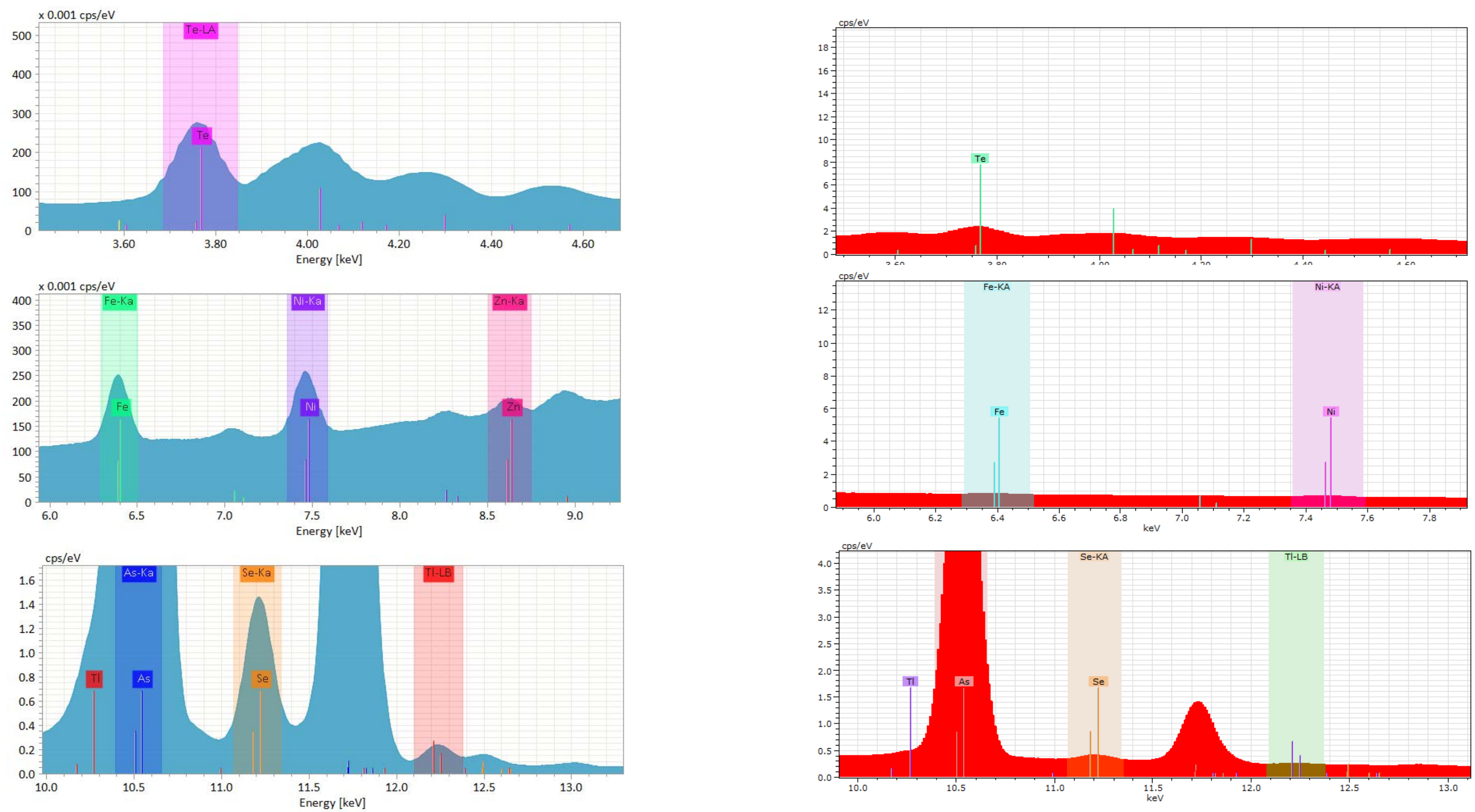
Geological Applications:
Volcanic Fumeroles

Comparison between XRF-EDS (XTrace) and SEM-EDS (e-beam) total map spectra, which highlights the capabilitites of the XRF-EDS to detect trace concentrations of elements not visible in the SEM-EDS spectrum.
Note the distinct peaks observable in the X-Ray beam image (blue) at various energies that are not present in the e-beam spectrum (red), and the significantly lower noise in the x-beam spectra.



Geological Applications:
Volcanic Fumeroles

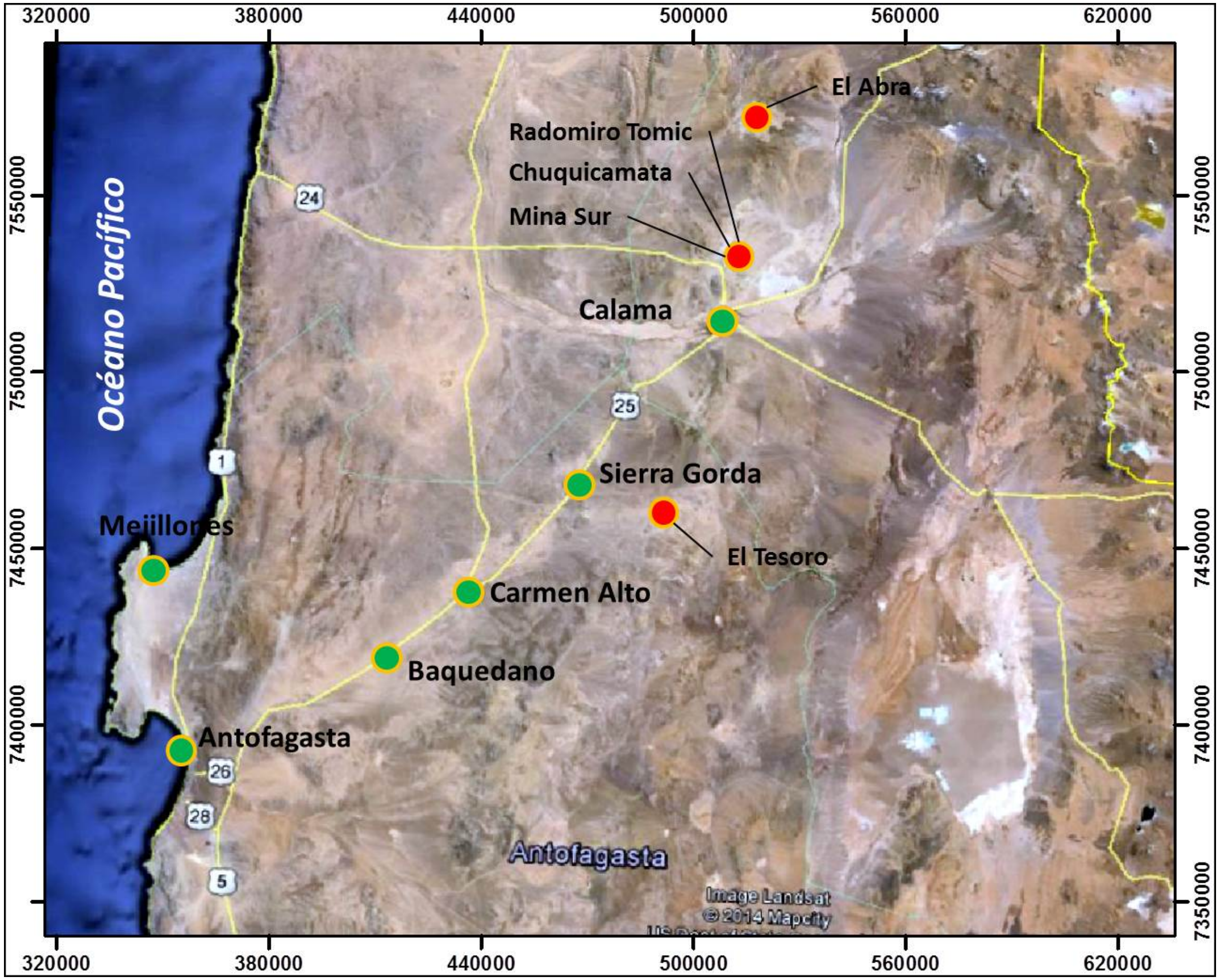
Comparison between XRF-EDS (XTrace) and SEM-EDS (e-beam)
Left column: X-Ray beam spectrum (blue); Right column: E-beam spectrum (red).



Geological Applications:
Exotic-Cu Deposits

- Exotic-Cu deposits often form in the vicinity of the parental porphyry system due to the lateral migration of Cu-bearing fluids.
- Mineralisation in this type of deposit comprises different species of copper minerals and mineraloids broadly defined as green-copper (*cobre-verde*) and black-copper (*cobre-negro*) ores.
- The analysis and subsequent definition of Cu-bearing minerals from exotic-Cu deposits is extremely complex due to the fine scaled textures and compositional variation.
- This is particularly true for so-called “black-copper” minerals. both Cu-wad and Cu-pitch, specifically related to the Mn concentrations, as well as numerous minor and trace elements such as: Mg, Al, Fe, Si, P, Ca, P, Cl, Co and S.

Geological Applications:
Exotic-Cu Deposits

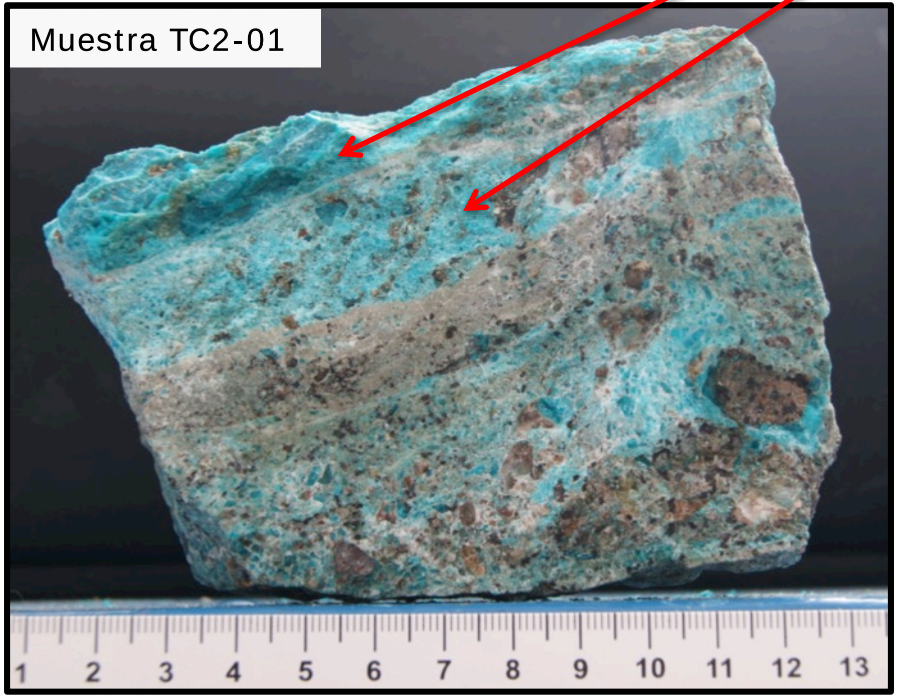


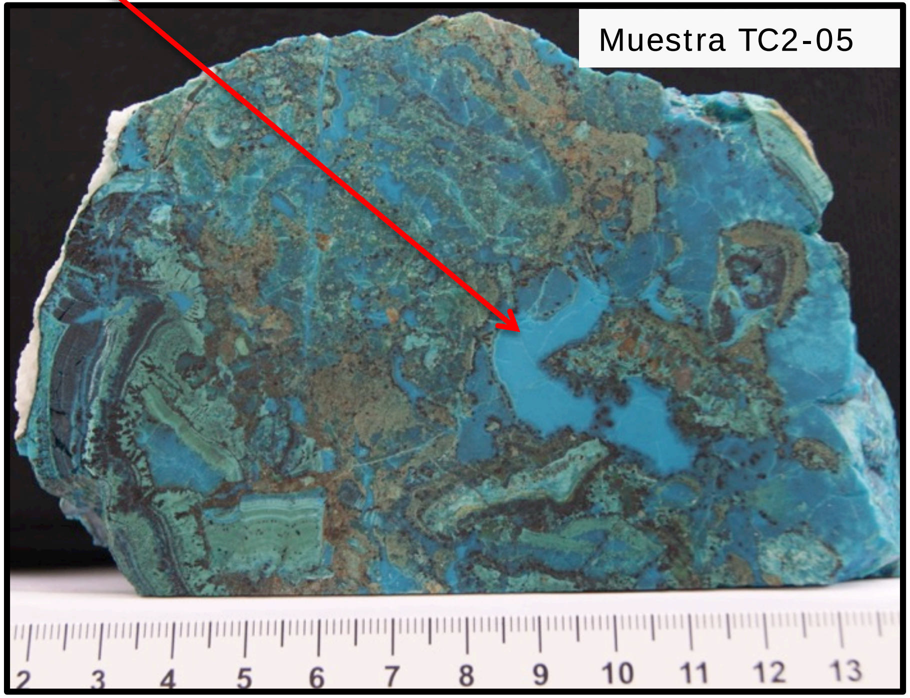
5

Geological Applications:
Exotic-Cu Deposits

$$Cu_{2-x}(Al,Fe^{3+})_xH_{2-x}[Si_2O_5](OH)_4 * nH_2O$$

Chrysocola

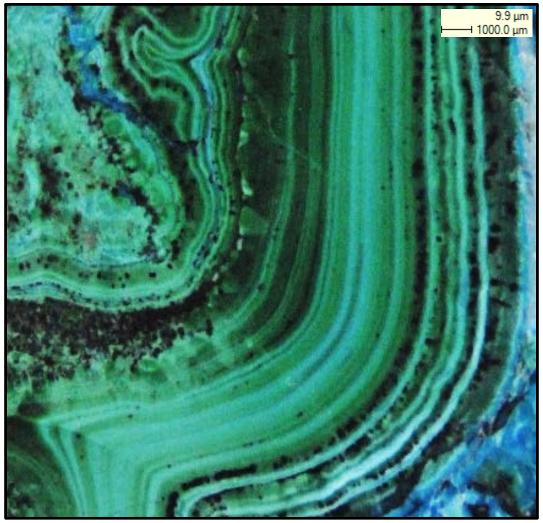




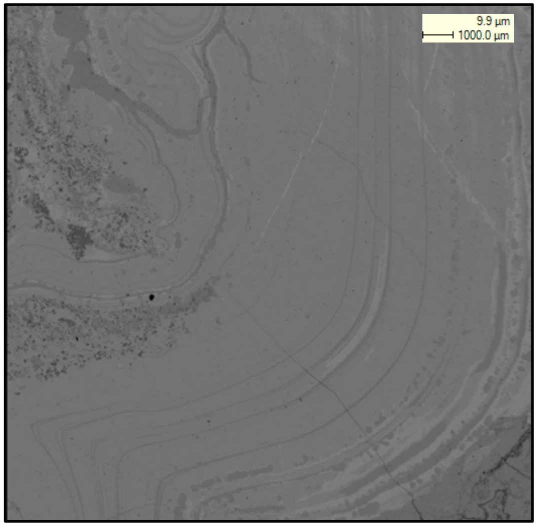
Geological Applications:
Exotic-Cu Deposits

Sample **AHM – ET – 003 – A**

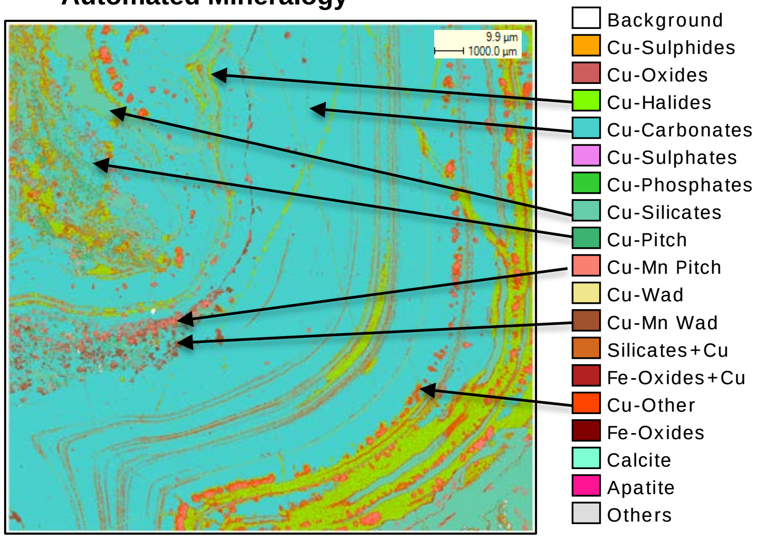
Optical Image

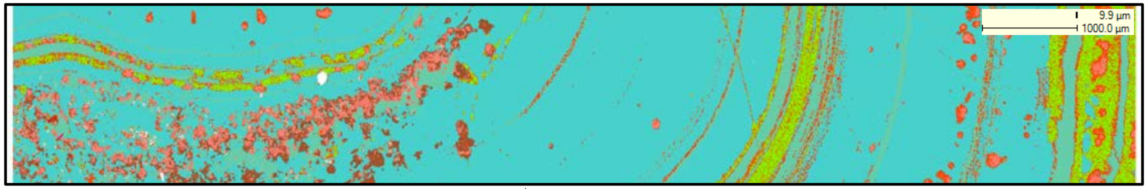


BSE



Automated Mineralogy



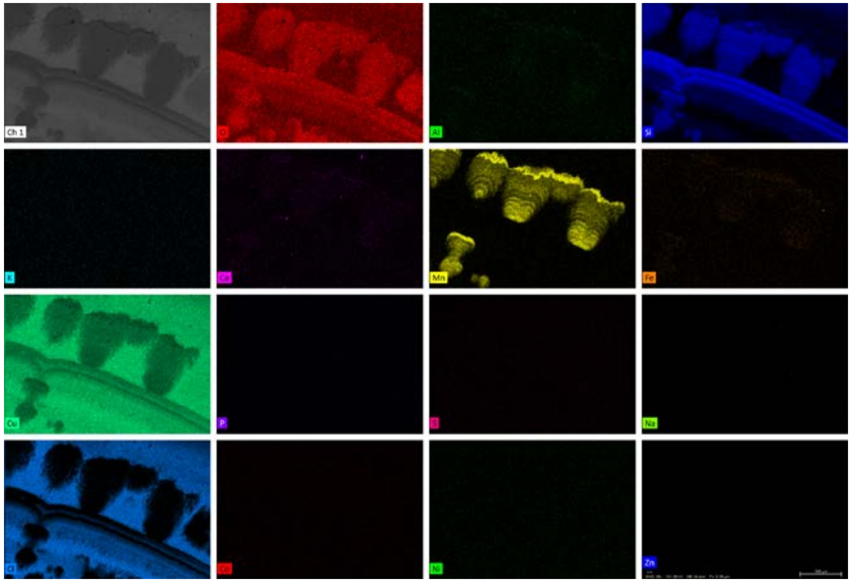


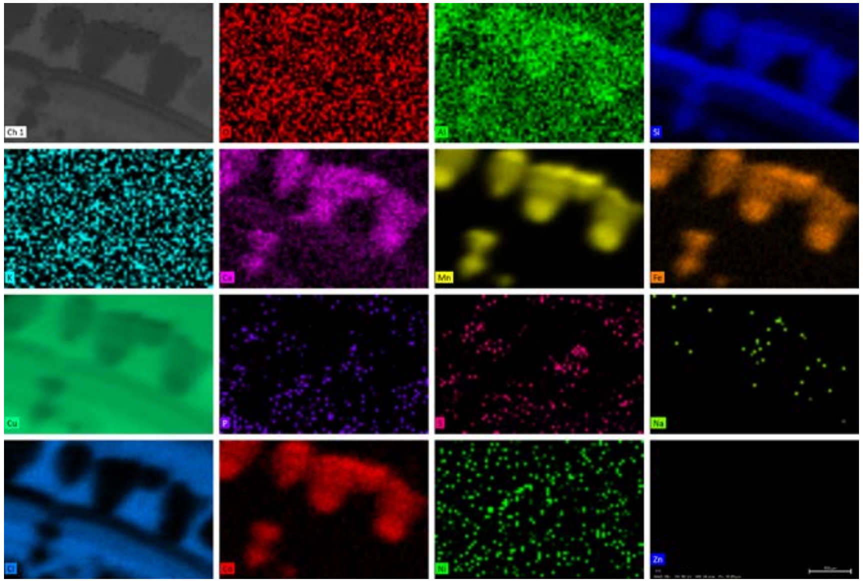
Geological Applications:
Exotic-Cu Deposits

Sample from El Tesoro, Chile.

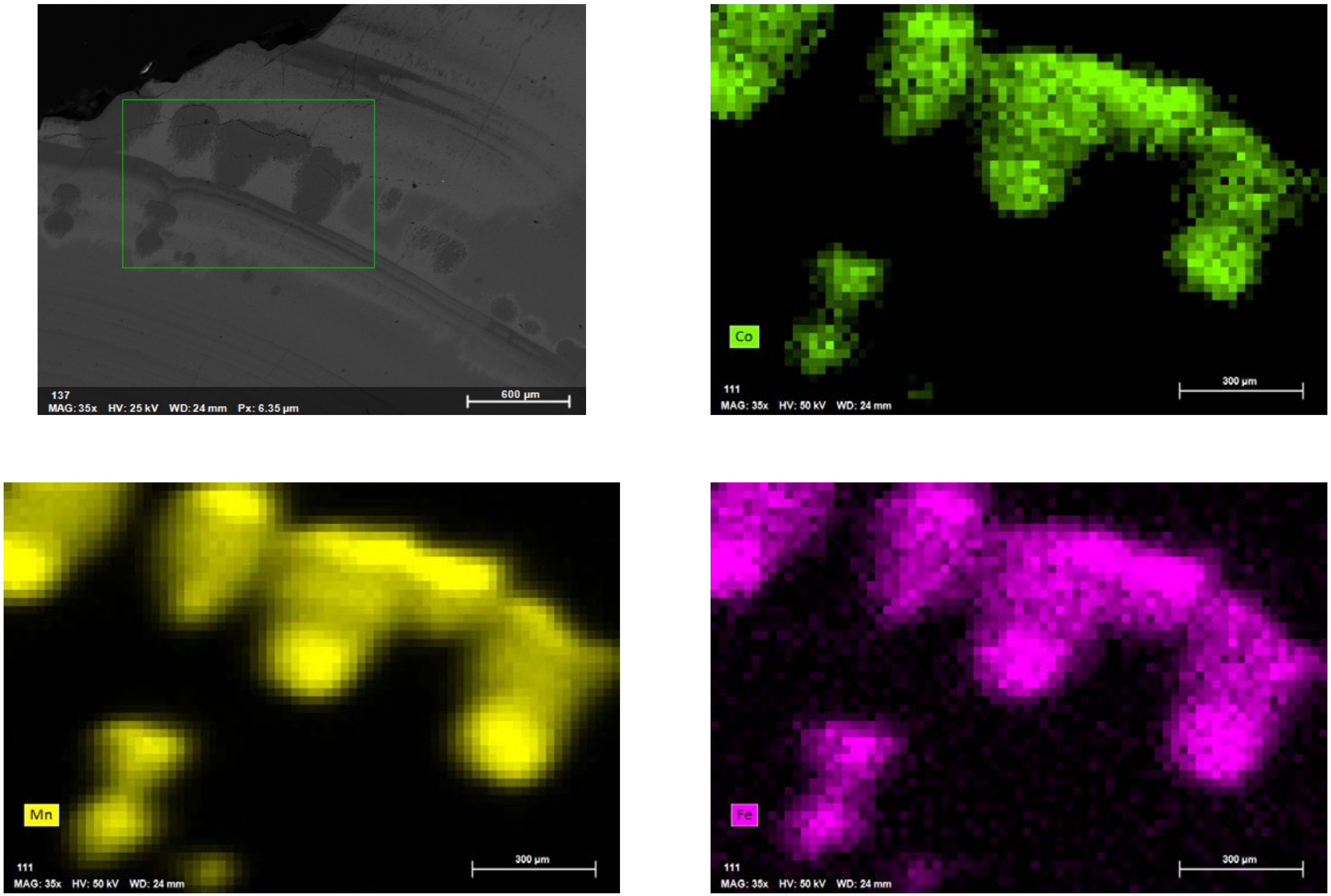
Elemental Maps Comparison between SEM-EDS (left) and XRF-EDS (right).

XRF-EDS: Increased signal intensity for trace elements: Al, Ca, Fe, P, Co
SEM-EDS: Better resolution





Geological Applications:
Exotic-Cu Deposits



Geological Applications:
Exotic-Cu Deposits

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Elemental Maps Comparison between SEM-EDS (left) and XRF-EDS (right).

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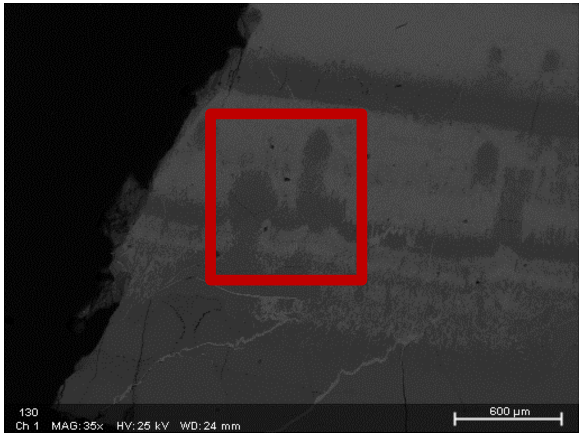
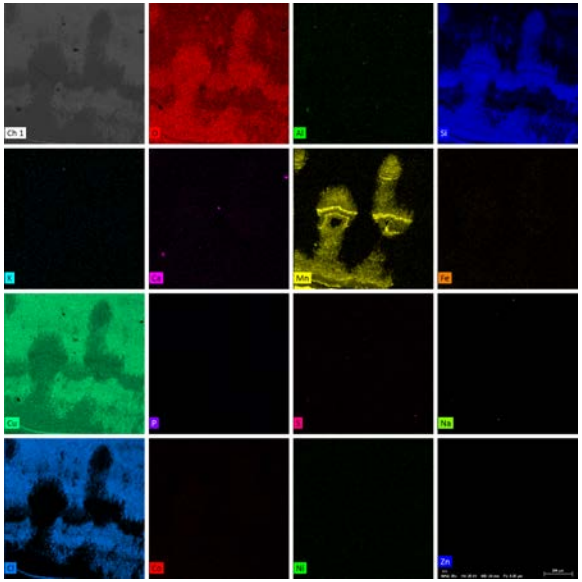
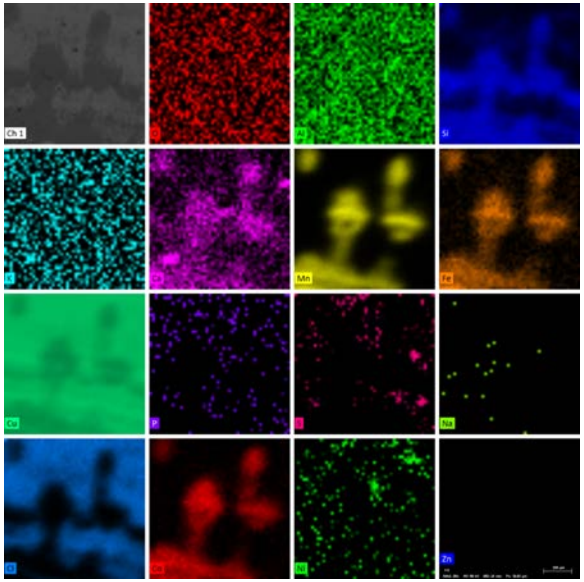
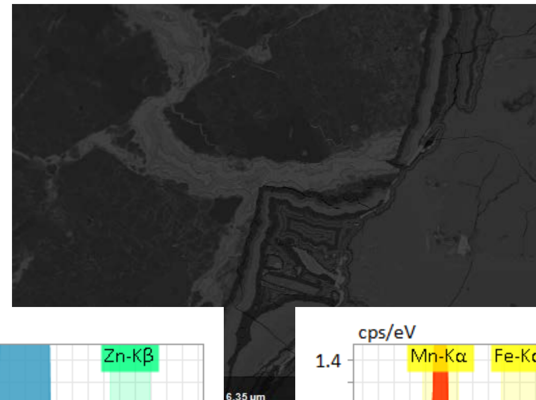


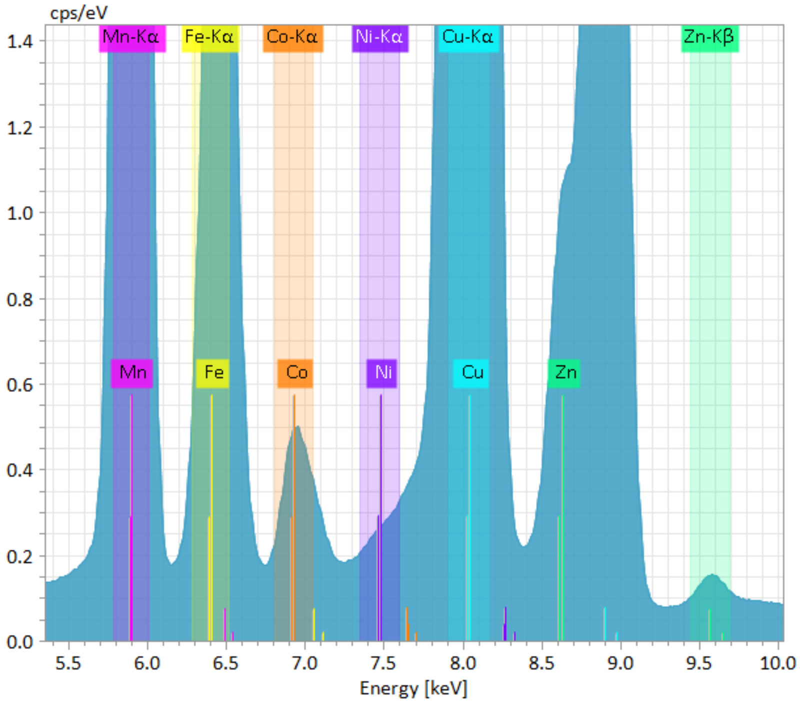




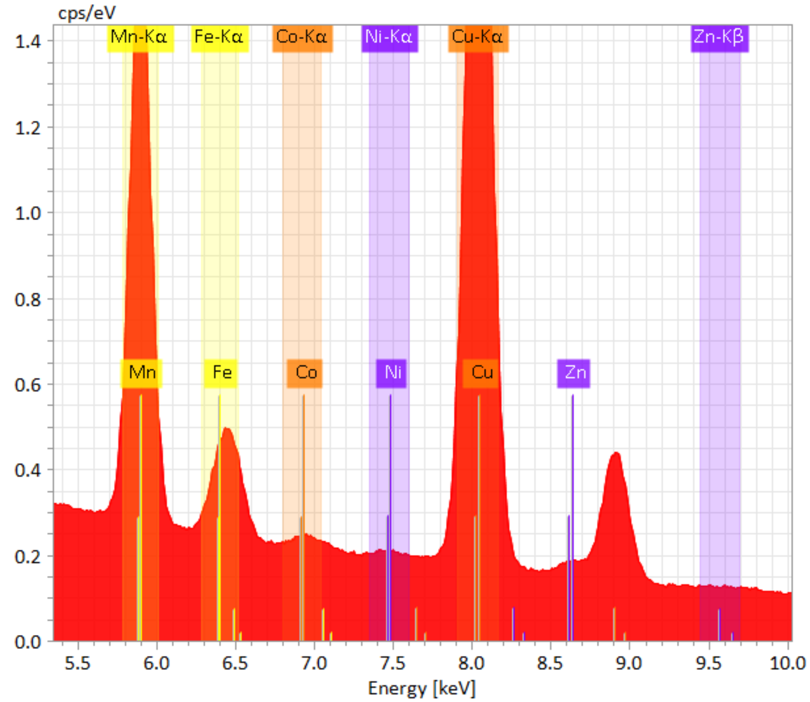
Geological Applications:
Exotic-Cu Deposits



XRF-EDS (XTrace)



SEM-EDS (e-beam)



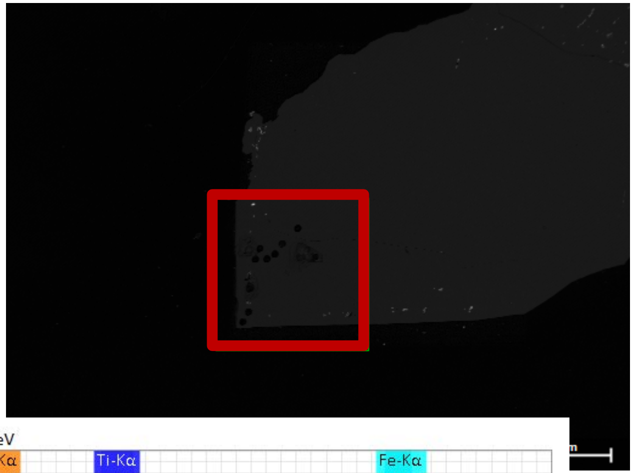
Geological Applications:
Ti-in-Quartz

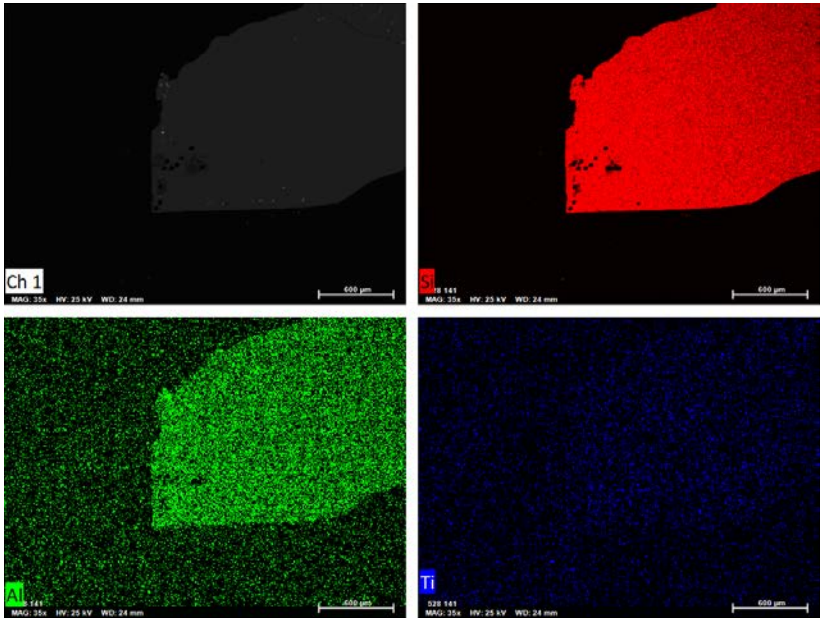
Sample: Epithermal Quartz

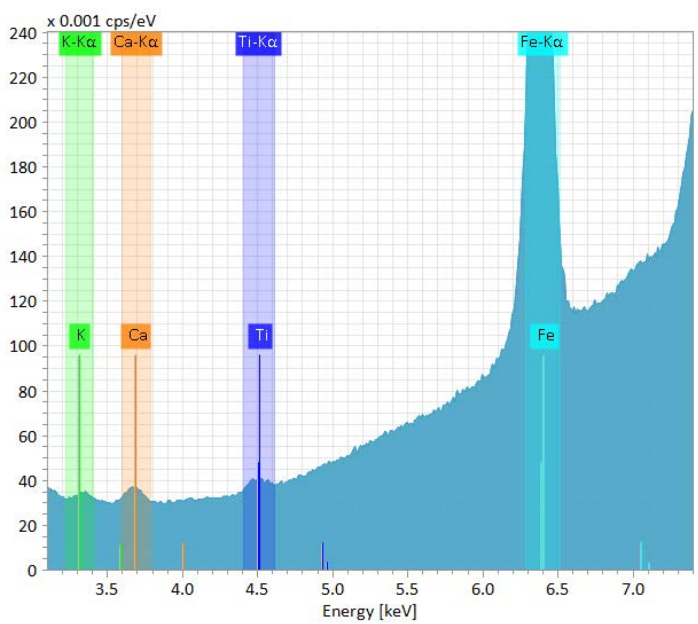
Ti concentration ranges from 0 – 120 ppm

Quartz grain border: low Ti (<20 ppm)

Consistent with LA-ICPMS results







Geological Applications:
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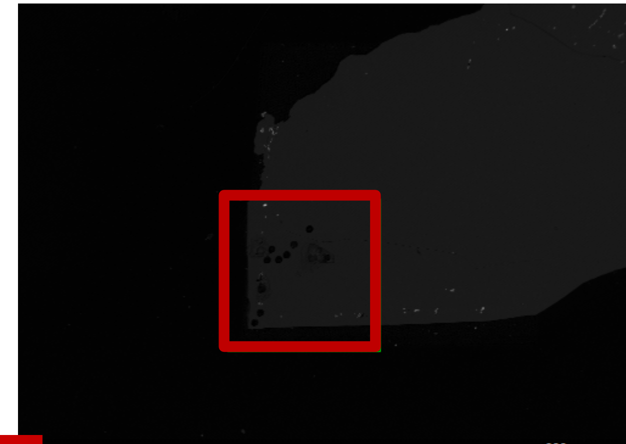
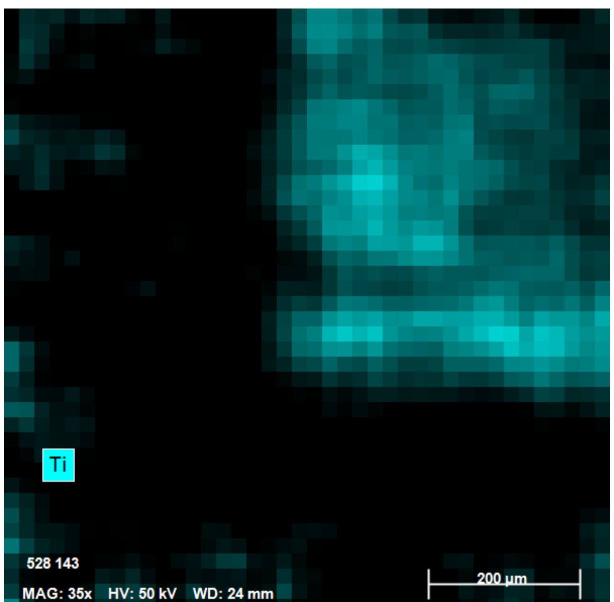
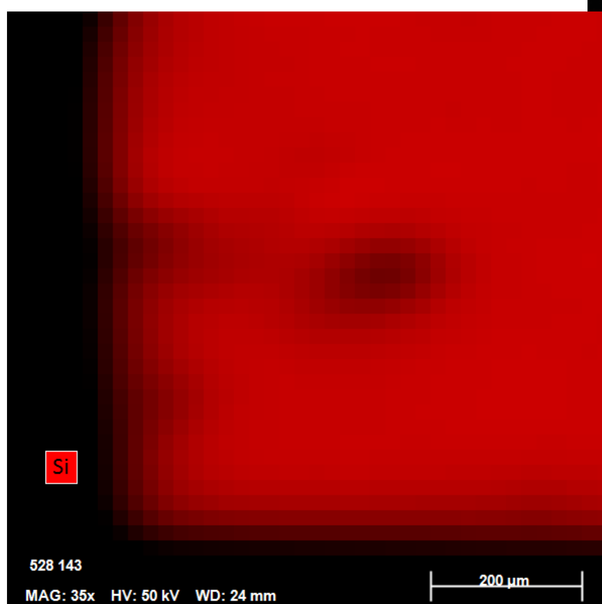
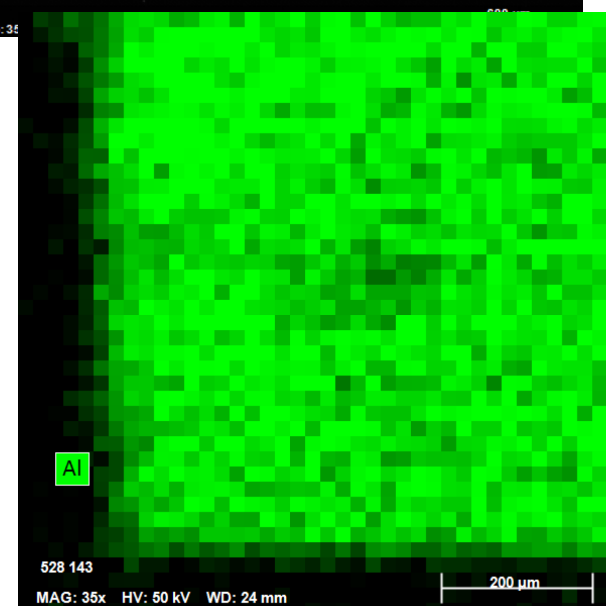


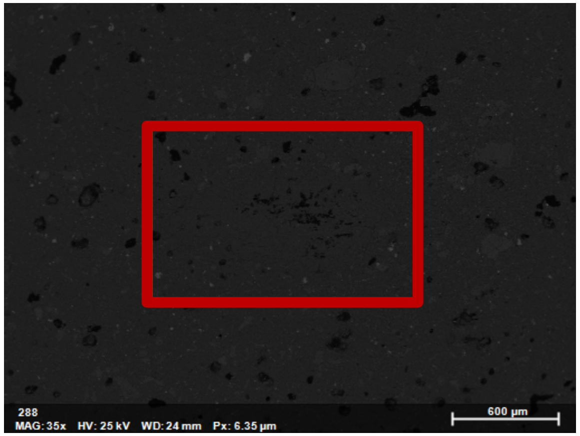


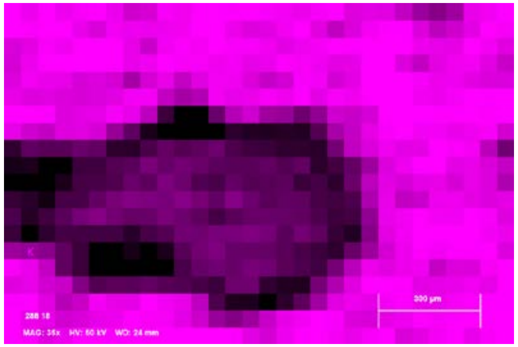


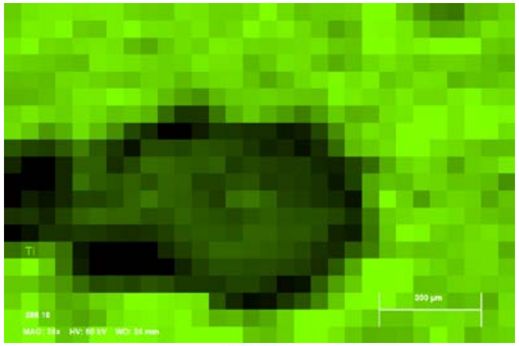


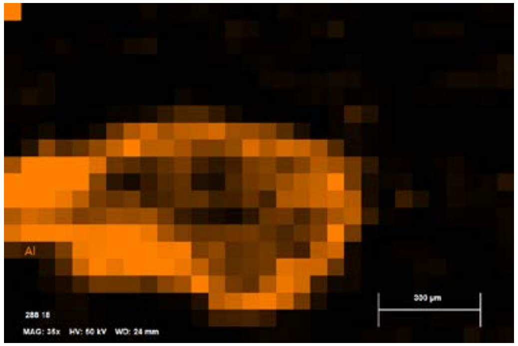
Geological Applications:
Mineral Zonation

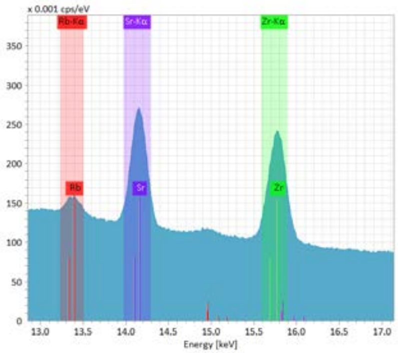
Sample:
Central Volcanic Zone, Chile.
Plagioclase Mineral
Elemental zonation

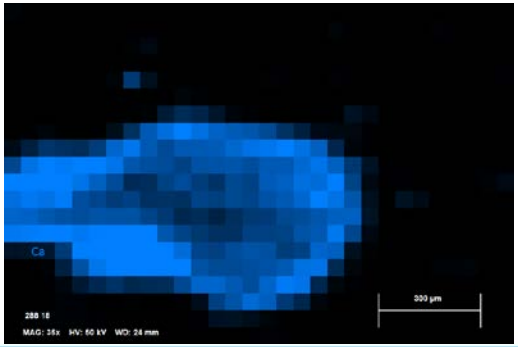


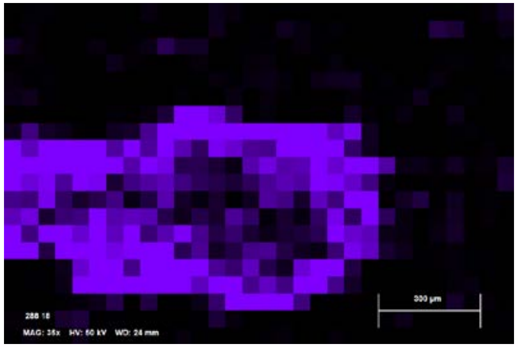








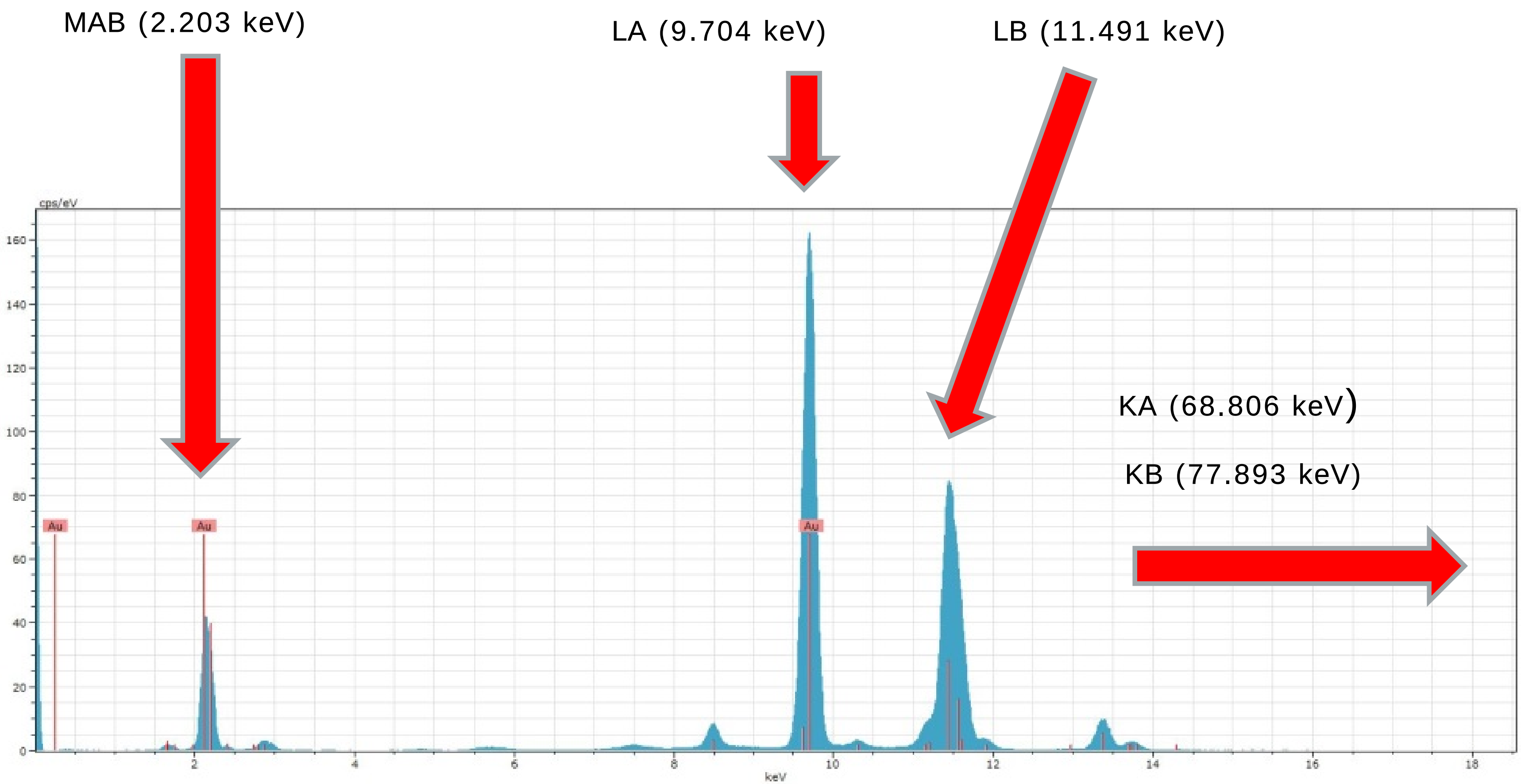




First Row: K and Ti
Second row: Al
Third Row: Ca and Sr

Interaction Volume: Interpretation

Gold: L-lines vs M-lines



Interaction Volume: Interpretation

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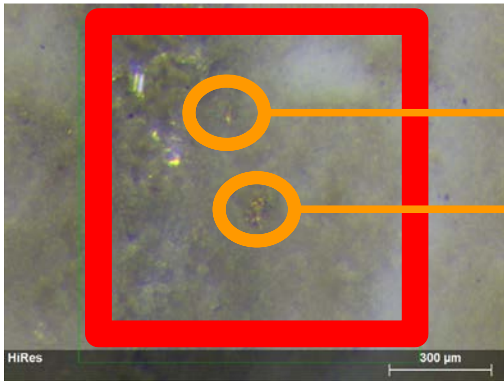
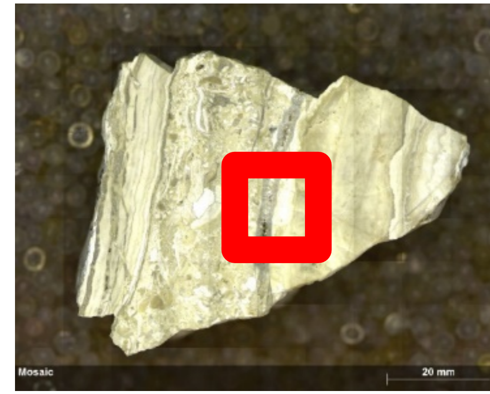
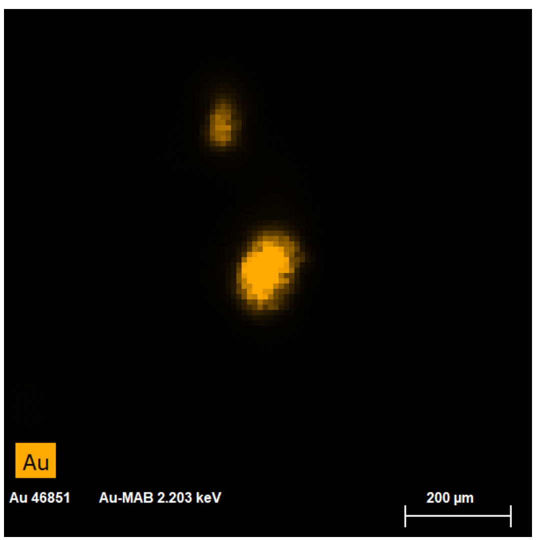


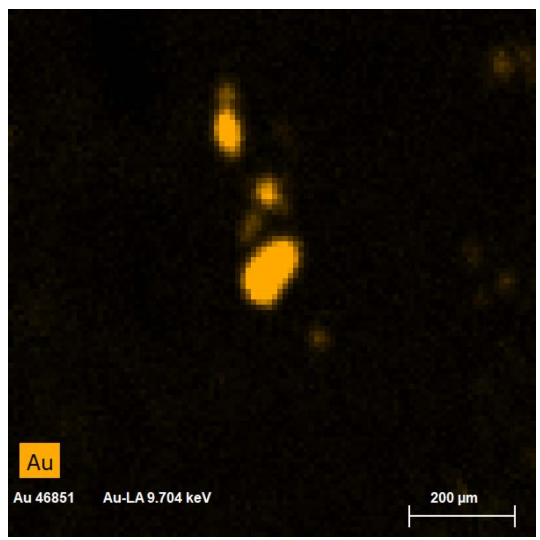
Small Gold Grain on Surface (<10 um)

Large Gold Grain on Surface (50 um)

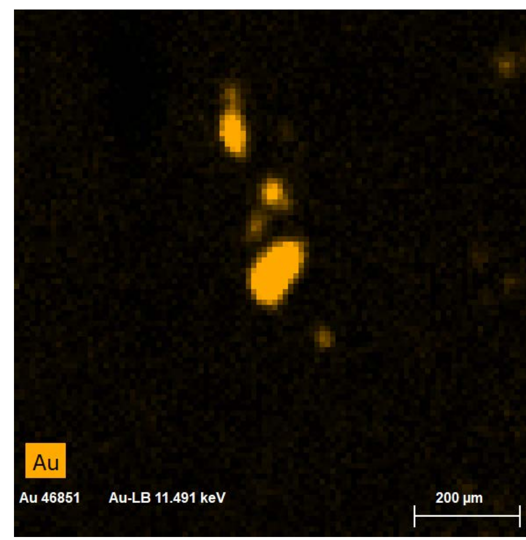




MAB (2.203 keV)



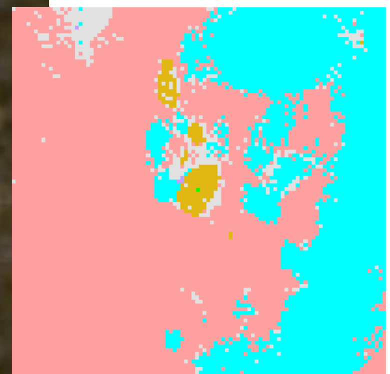
LA (9.704 keV)

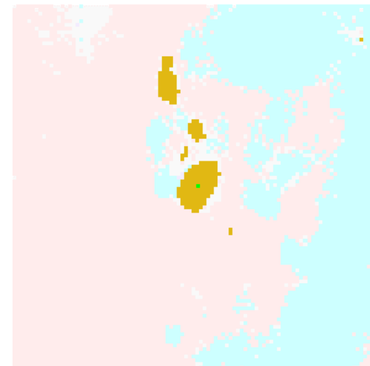


LB (11.491 keV)

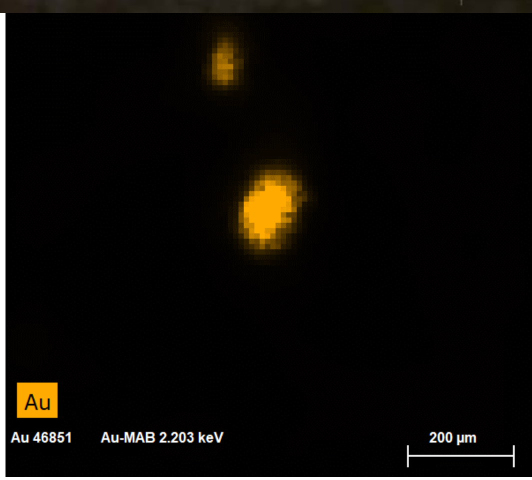
Interaction Volume: Interpretation

Gold: L-lines vs M-lines

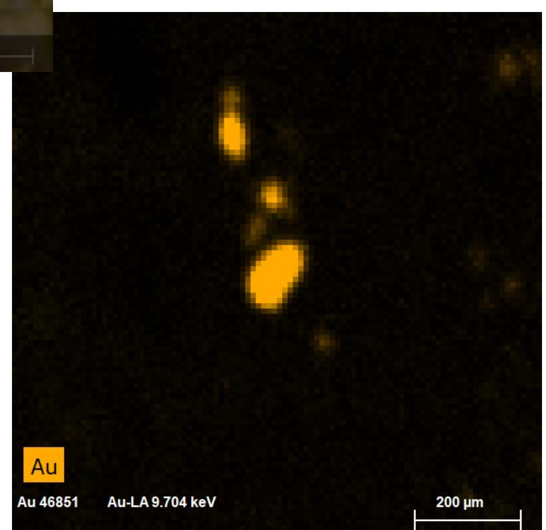




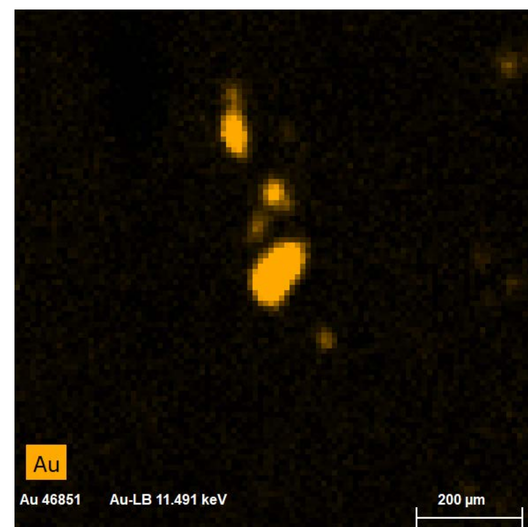
LA (9.704 keV)
LB (11.491 keV)
KA (68.806 keV)
KB (77.893 keV)



MAB (2.203 keV)



LA (9.704 keV)



LB (11.491 keV)

Elemental Mapping

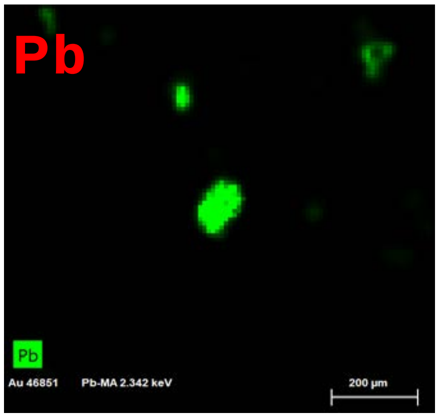
Overlapping K, L and M- Lines

Elemental Energy lines (keV) for Au, Zn and Pb

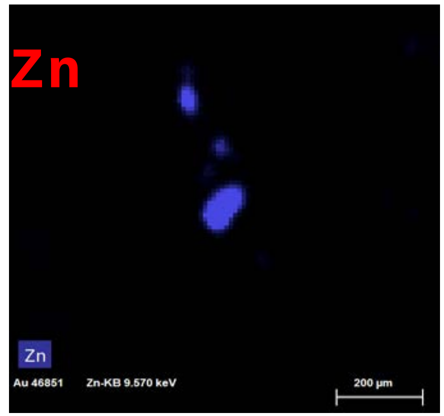
Symbol	Kα	Kβ	Lα	Lβ	Mα	Mβ
Au	68,806	77,982	9,713	11,443	2,123	2,203
Zn	8,637	9,570	1,012	1,035		
Pb	74,970	84,939	10,551	12,614	2,342	2,444

Elemental Mapping
Overlapping K, L and M- Lines

MA (2.342 keV)

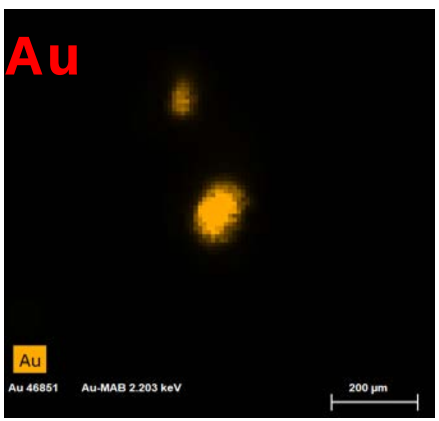


KB (9.570 keV)

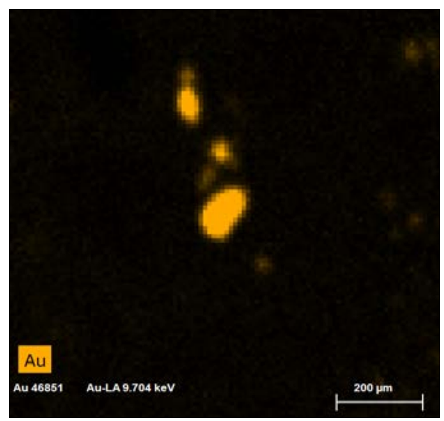


Large Gold Grain on Surface (50 um)

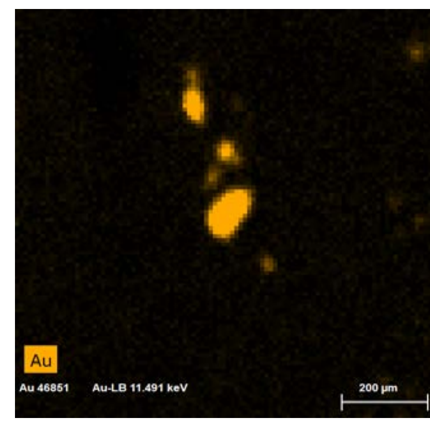
Small Gold Grain on Surface (<10 um)



MAB (2.203 keV)



LA (9.704 keV)



LB (11.491 keV)

Elemental Mapping:
Pixel Spacing vs Dwell Time

Analytical time

pixel dwell time (ms)

5 10 20 50

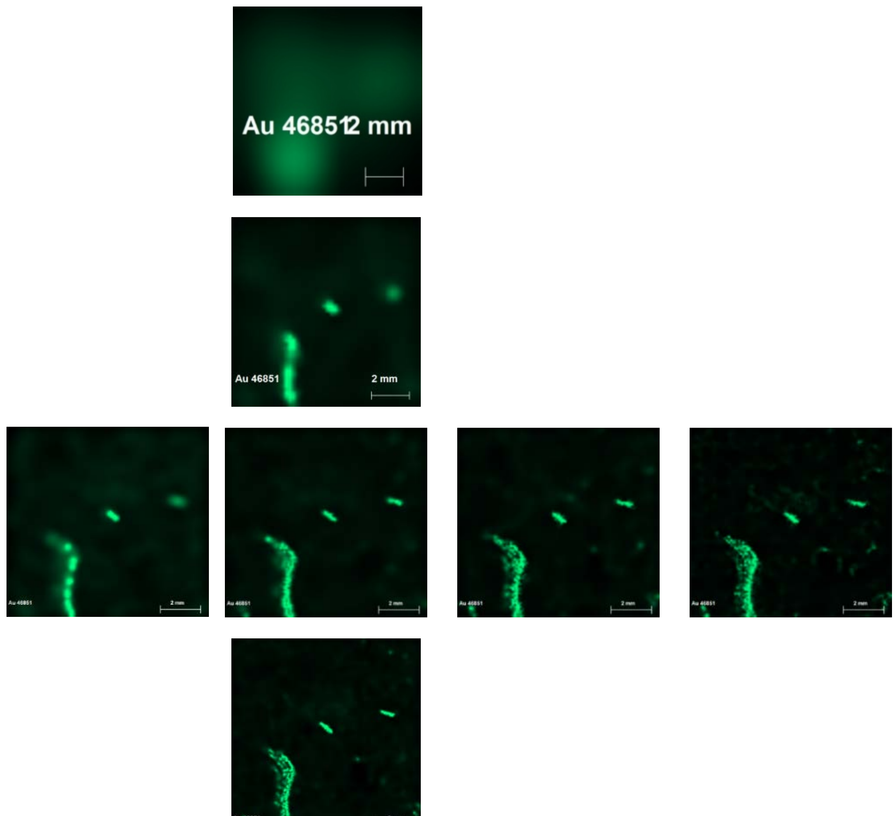
pixel spacing (um)

500

200

100

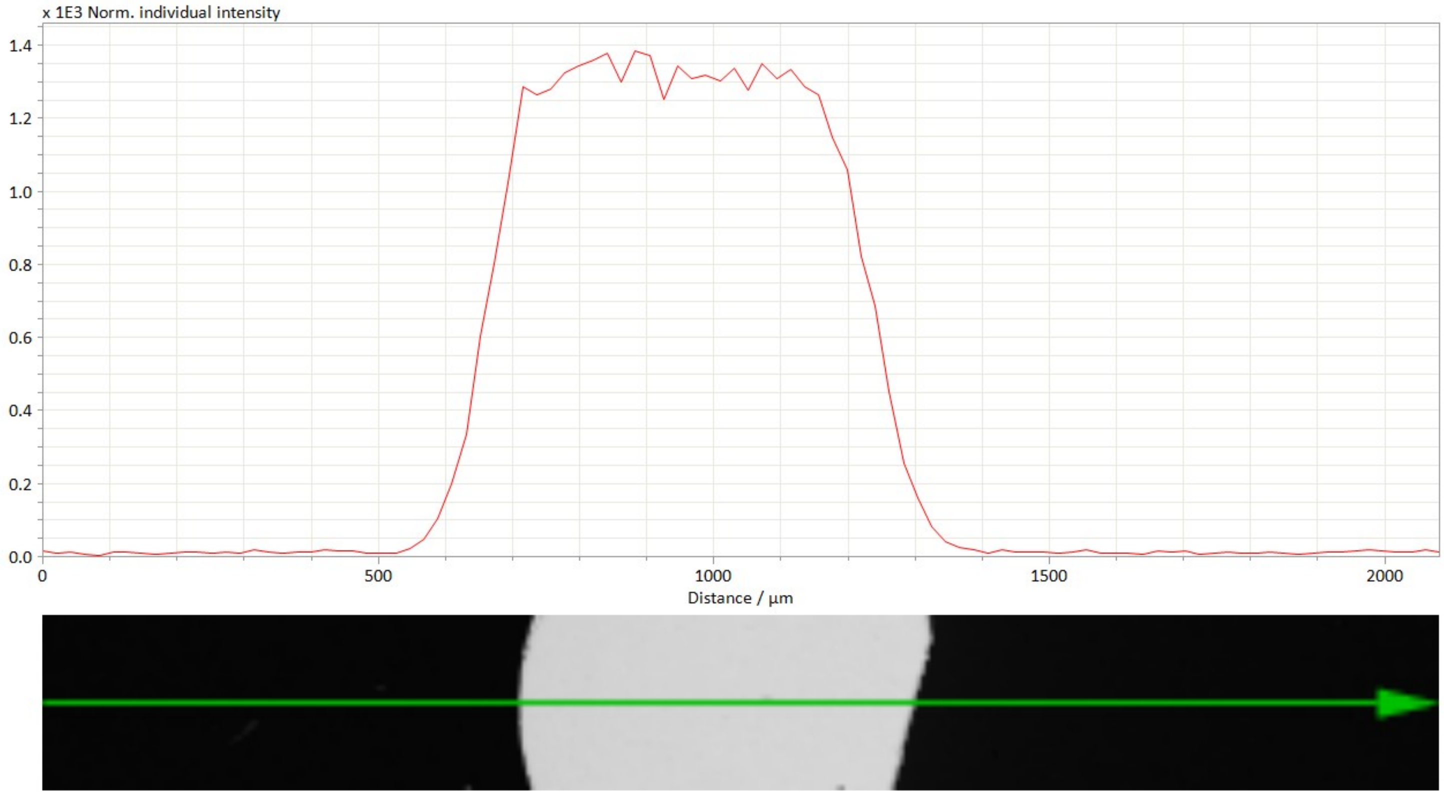
50



Analytical Area
10 x 10 mm

Xray Tube Position Calibration

- Questions:



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Patricia Muñoz

Are There Any Questions?

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

More Information

For more information, please contact us:

stephan.boehm@bruker.com

info.bna@bruker.com

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