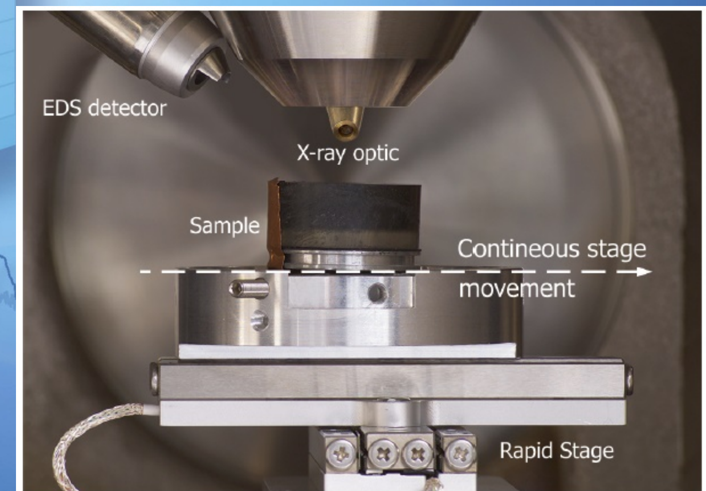
High Speed Mapping Using
Micro-XRF on SEM

Bruker Nano Analytics, Berlin, Germany
Webinar, November 6th, 2019

Micro-XRF on SEM



Presenters

Stephan Boehm

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

Andrew Menzies, PhD

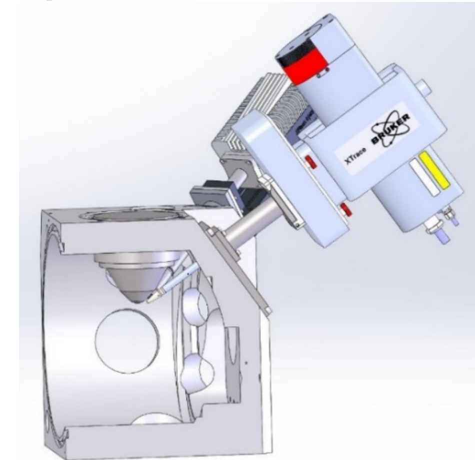
Sr Applications Geology and Mining,
Bruker Nano Analytics, Berlin, Germany

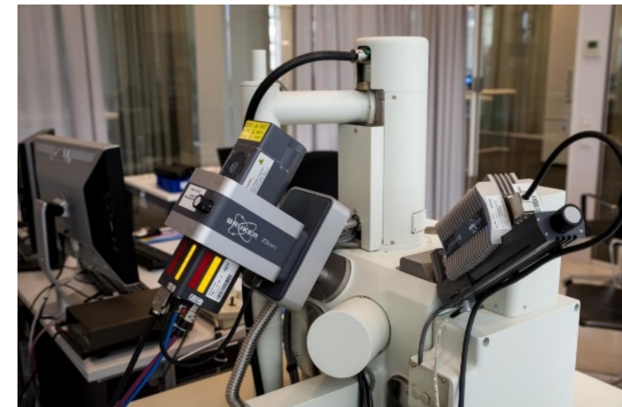
Overview

- Introduction / Presenters
- Introduction in SEM-XRF (XTrace)
- Differences between SEM-EDS / SEM-XRF - examples
- Rapid Stage Technical Description
- Example Applications and Benefits
- Summary and Conclusion

Introduction in SEM-XRF
At a glance

- Micro-XRF on an SEM with EDS analysis (SEM-XRF-EDS)
  - X-ray source will be adapted to an inclined SEM port
  - uses the same EDS detector (no extra detector required)
  - Same software package for EDS + XRF (ESPRIT 2.x)
- Non-destructive method for elemental analysis
  - Samples will be directly excited by photons
- Extended spectral range up to 40 keV
- Elemental range from Na to U
- Small spot analysis (35 µm X-ray spot)
- Low spectral background
- Information from within the sample
- Little or no sample preparation
- Quantification
  - standard less (based on fundamental parameters)
  - standard supported FP





SEM-XRF-EDS

ESPRIT 2.x Software & spectra comparison

SPECTRA

	cps/eV	Factor	Results [Mass-%(norm.)]	Sort: Value
EDS Acquisition 1.spx	0,95	0,136	E/X	O 30,79 Fe 21,36 Si 18,87 Al 15,24 Mg 8,96 Ca 4,10 Mn 0,40 Ti 0,21 Cr 0,09 Rh
XRF Acquisition 2.spx	0,04	1,000	E/X	O 30,79 Fe 21,36 Si 18,87 Al 15,24 Mg 8,96 Ca 4,10 Mn 0,40 Ti 0,21 Cr 0,09 Rh

0,14 * Acquisition 1.spx
1,00 * Acquisition 2.spx

cps/eV
Energy [keV]

Analytical Parameters and Conditions
SEM-XRF vs SEM-E-beam

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 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.

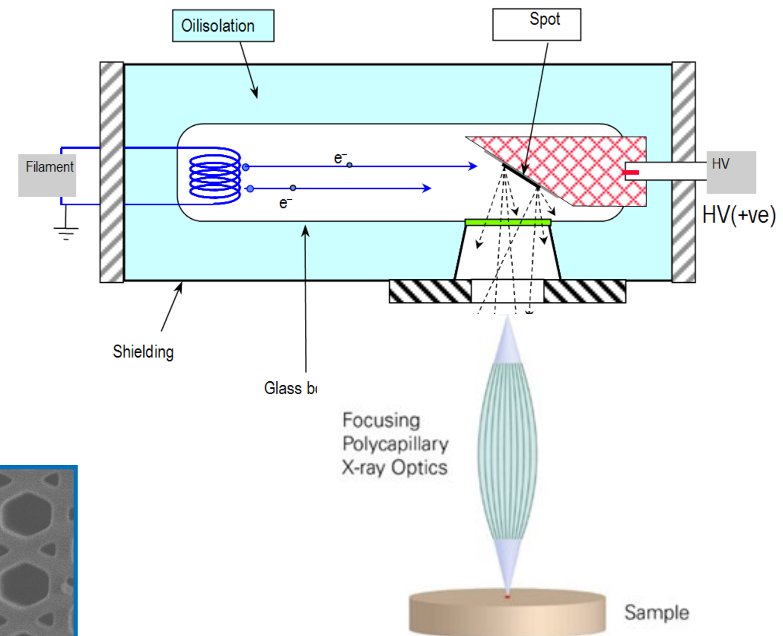
Introduction
SEM-XRF (XTrace)

Terminology

For clarity and simplicity, analysis and data using a **MicroXRF on SEM (XTrace)** will be referred to as **SEM-XRF** whilst traditional data gathered from an e-beam source (W or FEG) will be referred to as **SEM-EDS**

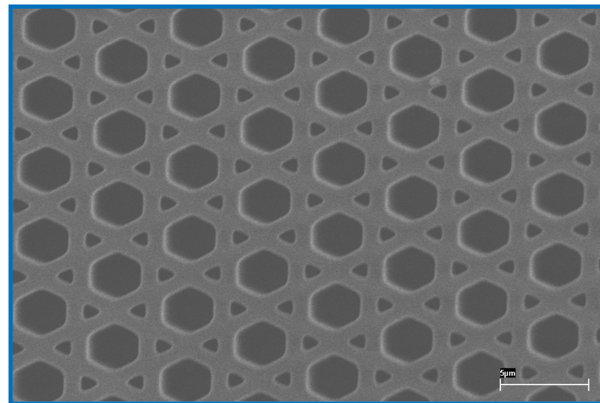
Bruker SEM-XRF
Hardware design

- Main components are an X-Ray tube with a focusing optic.
- Rhodium (Rh) is the commonly used tube material and does not overlap lines of typical analyte elements.
- Typically operates at 50 kV / 600 µA
- Tube radiation is captured by a polycapillary lens with large acceptance angle and focused onto the sample surface.



X-ray tube and optic schematics

XTrace source with polycapillary optic

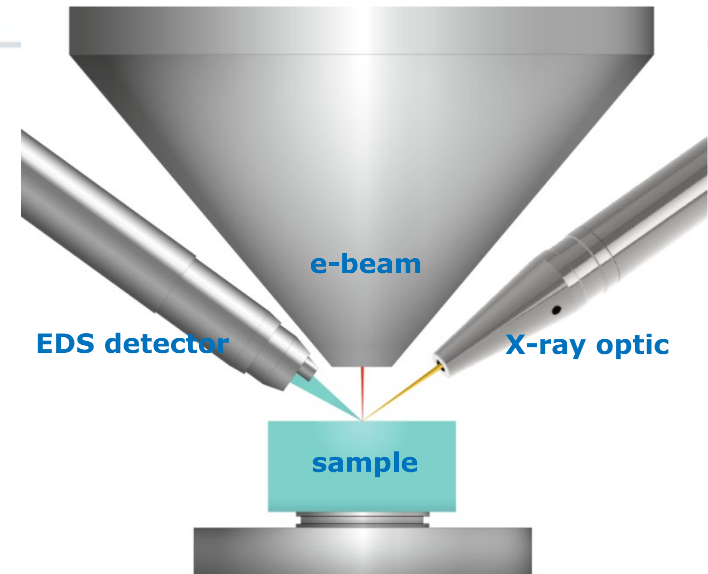


SEM image of a polycapillary structure. Inner diameter in the range of 2 µm

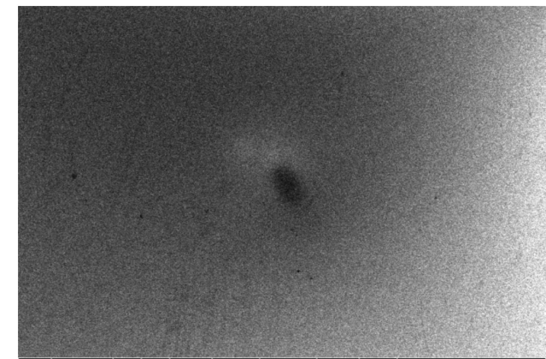
Bruker SEM-XRF
Installation of XTrace on a SEM

SEM – XRF setup & alignment

- X-ray source is (mechanically) adjustable and X-ray beam must be aligned to the position of the electron beam on the sample
- Re-alignment is only required when SEM WD was changed



- Beam alignment can be done by using the glass sample which comes with the system
- If electrical nonconductive materials (glass, paper) are excited with both electrons and X-rays, the position of the X-ray beam can be seen as a dark point
- Final adjustment has to be performed with special sample structures (structures on wafer sample)



X-ray spot on uncoated glass

Rapid Stage for SEM´s
Introduction

Bruker Nano Analytics, Berlin, Germany
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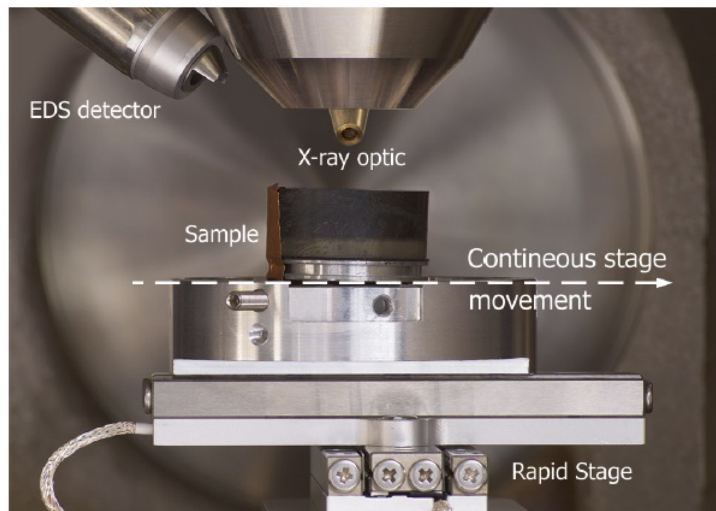
Element	At. No.	Line S.	EDS Mass [%] Norm.	XRF Mass [%] Norm.	Comb. Mass [%] Norm.	Certified Val. /M%
Oxygen	8	K-Series	45.71	45.58	46.03	46.82
Sodium	11	K-Series	10.54	10.32	10.61	10.68
Magnesium	12	K-Series	2.32	2.27	2.33	2.22
Aluminium	13	K-Series	1.34	0.89	0.89	0.95
Silicon	14	K-Series	33.94	34.99	34.18	33.70
Sulfur	16	K-Series	0.16	0.12	0.12	0.11
Potassium	19	K-Series	0.37	0.35	0.35	0.34
Calcium	20	K-Series	5.30	5.34	5.34	5.08
Titanium	22	K-Series	0.00	0.01	0.01	0.01
Iron	26	K-Series	0.28	0.03	0.03	0.03
Arsenic	33	K-Series	0.04	0.06	0.06	0.04
Strontium	38	L-Series	0.00	0.04	0.04	–

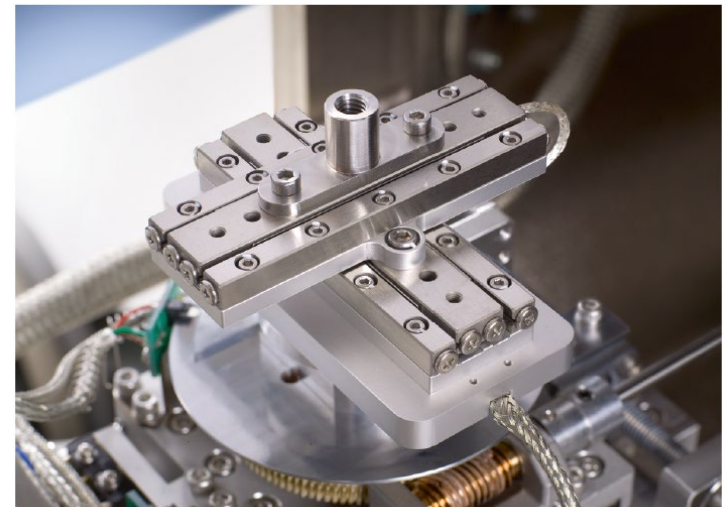
XRF: Oxygen quantification by stoichometry

Micro-XRF on SEM

Element Distribution Measurements
Rapid Stage

- Micro-XRF on SEM works with a fixed X-ray beam (X-ray beams cannot be raster as e-beams can).
- Consequently, elemental maps have to be acquired via stage movement.
- The **Rapid Stage** has been developed to enable high-speed mapping over large areas.
- It is mounted on top of an existing SEM stage, including stage adaption and sample holder.
- The Rapid Stage is controlled independently from the SEM stage and can operate up to a maximum travel speed of 4 mm/s.





Element Distribution Measurements
Rapid Stage

Rapid Stage Integration in ESPRIT

MAPPING

Sample | Standards | Microsc... | X-ray so... | Substage | Scan | EDS | Report | Project

Substage: Position X 0.0 µm; Position Y -0.8 µm; Velocity 5000.0 µm/s; Frame time 02:25 min; Pixel time 4.8 ms

Preview | Capture | Acquire | QMap

Loaded: C:\Users\messeds.ber\Desktop\SW test_Nov_2018\map_alignment_tool.bcf

Image extension

- [x] Activate

Width	10	X	25.7mm
Height	13	X	24.9mm

Map time

- ○ Manual
- ○ Measurement time [s] 100
- ◉ Cycles 1

Map area

◉ Full ○ Fixed ○ Variable

	[µm]	Points
Map width	12833.00	500
Map height	9571.00	375
Point distance	50.00	

SUBSTAGE MOVEMENT

	Current values	New values
Move speed [µm/s]	500	3000
Dwell time [ms]	25.7	4.3
Frame time	12:49min	02:08min

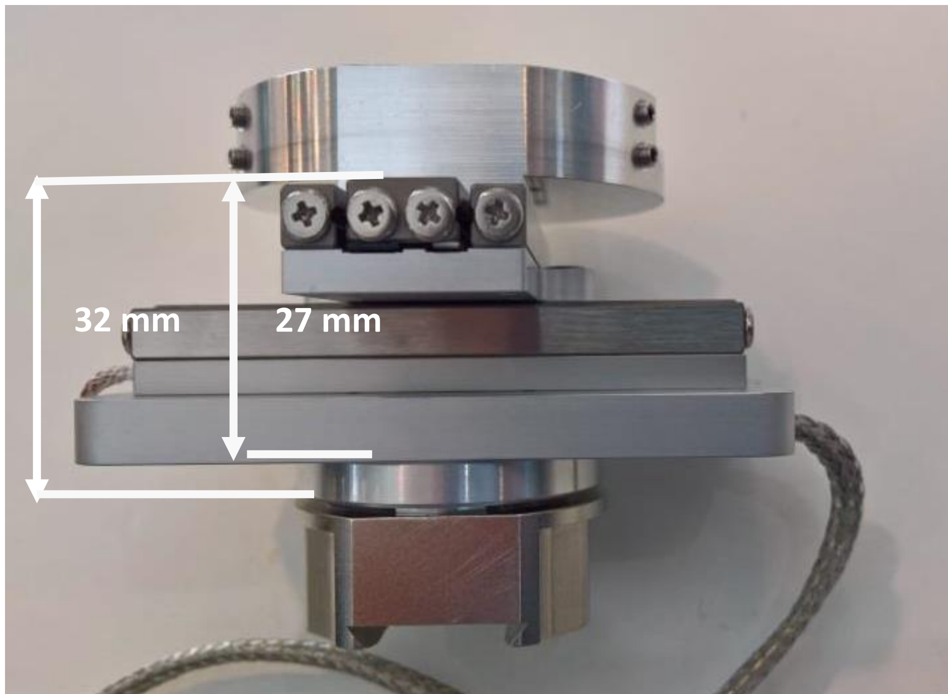
Reference | Apply

Rapid Stage Dimensions

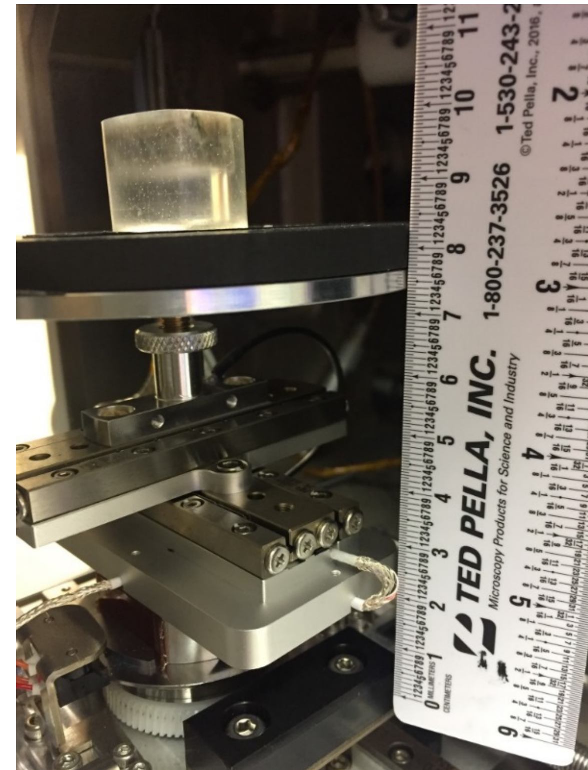
Stage itself (including x- and y linear positioners and basis plate): **27 mm**

Including footplate: **32 mm**

Without dovetail and sample holder → SEM depending

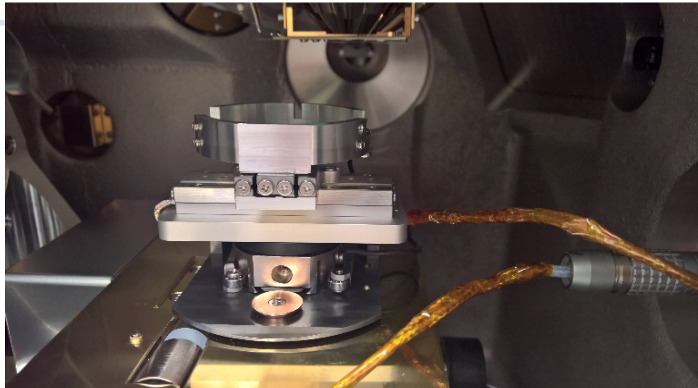


for Jeol IT 500 setup: **60 mm**

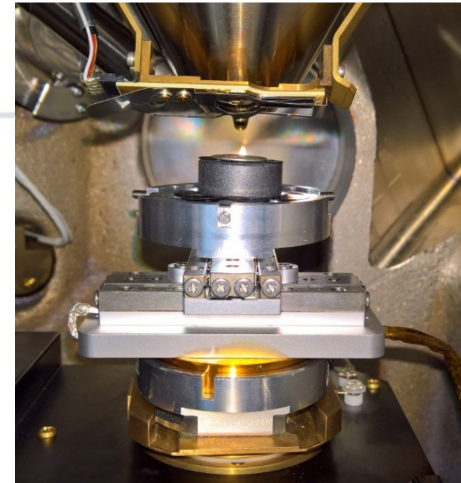


for Hitachi S3700N setup: **73 mm**

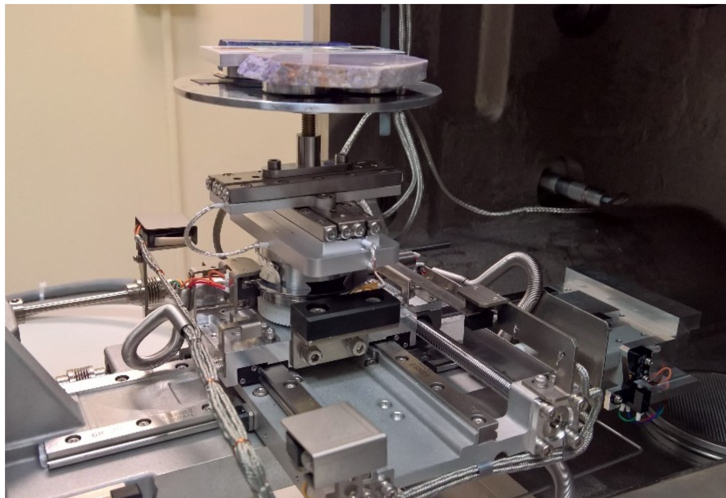
Rapid Stage Installations



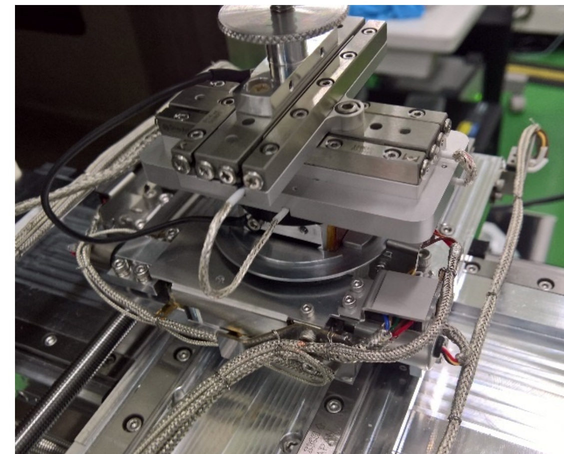
Jeol IT-500



Jeol JSM 6490



Hitachi S 3700N



Hitachi SU 3900

Rapid Stage performance
Variable Parameter – Pixel, Speed

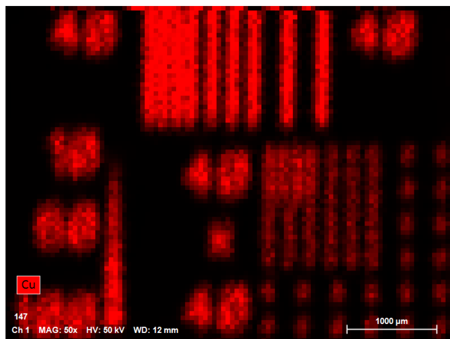
- Increased Stage Speed → Decreased Analytical time
- Increased Number of Pixel → Increased Analytical time

Image size (aspect ratio 4:3)	Dwell time per pixel	Scan Speed	Scanning time
200 px	20 ms	1.2 mm / sec	~ 11 min
200 px	8 ms	3 mm / sec	~ 4.5 min
400 px	40 ms	0.6 mm / sec	~ 82 min
400 px	4 ms	6 mm / sec	~ 8 min

Parameter that influence the spatial resolution

Cu on Si wafer sample (area: 2600 µm x 1950 µ)

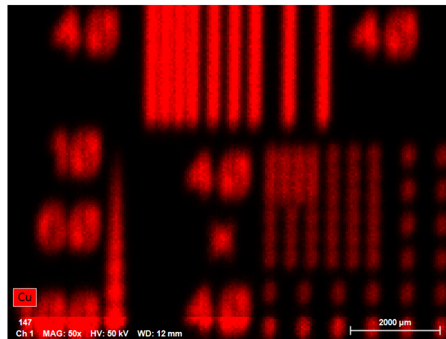
100 px – 10 ms/ pixel



Total map time: **75 sec**

- Image looks pixelated due to the low number of pixels and short integration time
- Step size: 26 µm
- Statistics not that good due to low dwell time

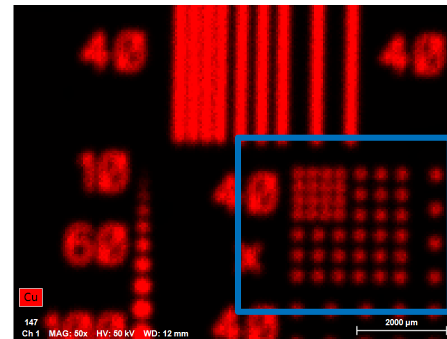
200 px – 40 ms/ pixel



Total map time: **20 min**

- Image looks better but the small points cannot be resolved due to the elliptically shape of the X-ray spot
- Step size: 13 µm
- Statistics improved due to longer dwell time

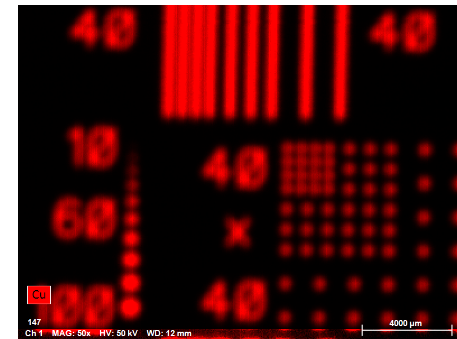
200 px – 40 ms/ px
32° sample tilt



Total map time: **20 min**

- Sample was tilted towards to the X-ray optics
- Small (20 µm) structures can be resolved much better
- * image distorted due to horizontal map while sample is tilted
- **Recommended conditions**

400 px – 400 ms/ px
35° sample tilt



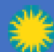
Total map time: **13 h**

- Oversampling yields only in minor improvements but results in long acquisition times
- Step size: 6.5 µm

Rapid Stage
Specification

Parameter	Description
Height	27 mm (without sample holder and SEM stage adaption)
Weight	300 g
Sample load	3 kg
Stage travel speed	4 mm /sec
Travel distance	50 mm
Vacuum resistance	10^{-7} mbar (higher vacuum resistance on request)
Resolution	< 1 nm

Examination of Heritage and Geological Materials Using Correlated Electron- and X-ray-Beam Microanalysis in the SEM



Smithsonian
Museum Conservation Institute

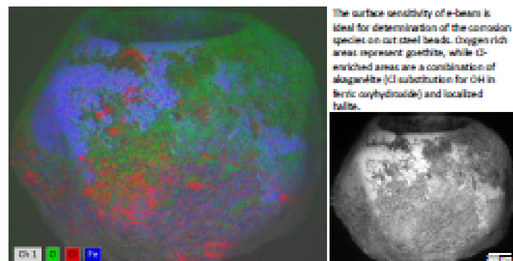
Edward P. Vicenzi & Thomas Lam

Background

A Bruker XTrace micro-XRF has been mounted onto an Hitachi S3700N SEM. A Rh source and one of two polycapillary optics aligned to the electron raster image were used to form a ~15, or 30 µm x-ray beam. This spatial resolution is defined as the lateral spot size at Cu K_α. A Bruker 6160 SDD was used to collect x-rays and XRF results were computed using the fundamental parameters approach to micro-XRF [1]. The information depth of XRF data scales with the energy of the peak and can be much greater (up to 10s-100s µm) for high energy x-rays compared to the x-ray emission depths produced by the electron beam in the SEM [2]. The XRF method therefore serves as a non-destructive complement to near surface electron beam microanalysis with increased sensitivity for trace elements. Here cut steel beads used in tanned hide garments from Native American Plains tribes dating to the late 19th to early 20th centuries has been examined for their composition and corrosion products [3]. Additionally, a jade from the Bursa Province of Turkey has been used to illustrate the virtues of correlated multi-beam scanning.

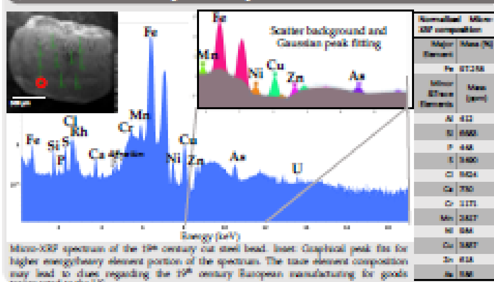
Electron Beam Imaging with Correlated µXRF Spectrometry

Electron Beam Raster Induced X-ray Imagery



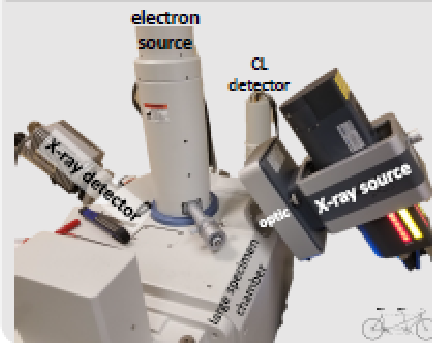
The surface sensitivity of e-beam is ideal for determination of the corrosion species on cut steel beads. Oxygen rich areas represent goethite, while Cl-enriched areas are a combination of akaganéite (Cl substitution for OH in ferric oxyhydroxide) and localized halite.

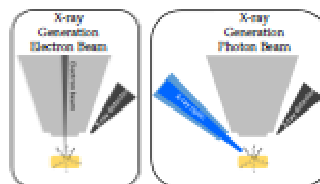
µXRF Point Analysis: Major, minor & trace elements



Micro-XRF spectrum of the 19th century cut steel bead. Inset: Graphical peak fits for higher energy/heavy element portion of the spectrum. The trace element composition may lead to clues regarding the 19th century European manufacturing for goods transported to the US.

The Analytical Dual Beam Microscope Tandem Analysis





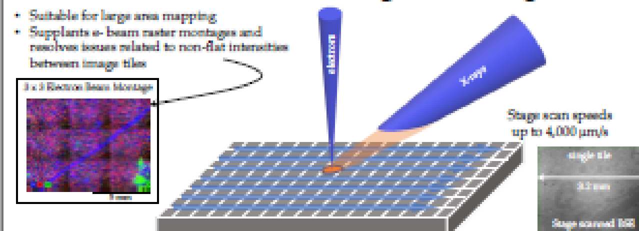
Instrumentation

Microscope	Hitachi S3700N
X-ray detector	Bruker XFlash 6160
X-ray source	Bruker XTrace
Rapid stage	Bruker Piezo Slip-Stick
X-ray optic	30 µm spot @ Cu K_α
CL detector	Gatan ChromaCL2

SEM Substage

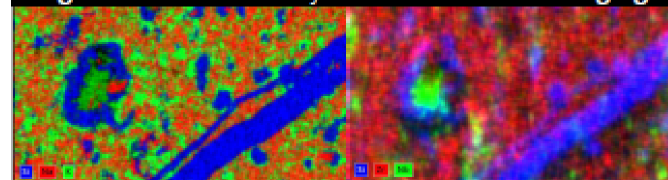
Electron -& Photon-Induced X-ray Imaging

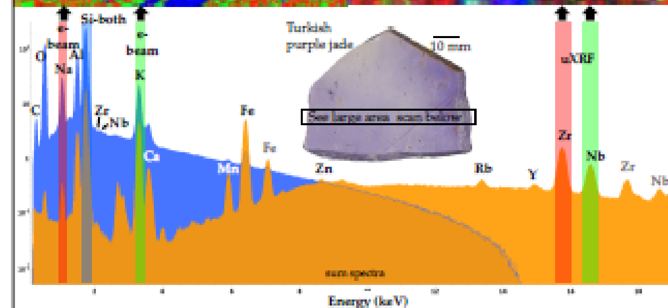
Fixed Beam(s) Stage Scanning



Spot E- Beam — Light element sensitivity

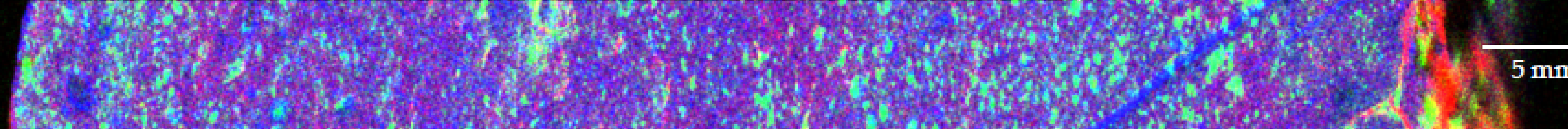
µXRF — Trace element imaging





Summary

- Electron beam analysis is a superior technique for the determination of light elements, near surface composition, and high quality quantification via standards-based quantification
- µXRF beam analysis yields superior trace element results for many metals (>10 of ppm) and probes more deeply into the specimen, particularly high energy lines
- Sequential fixed beam analysis offers a nondestructive method to extract new information about the history and origin of heritage and geologic materials from the analysis of correlated spot and image data

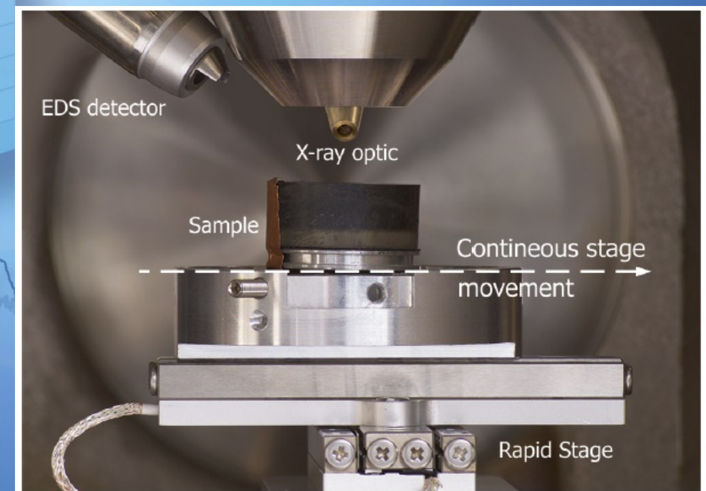


References: [1] M Haschke, "Laboratory Micro-X-Ray Fluorescence Spectroscopy" Springer (2014), 356 p. [2] M Haschke & S Boehm, in "Advances in Imaging and Electron Physics" ed. Hawkes (2017) p.1-68. [3] RD Rawlings, "The manufacture of cut steel studs and beads." Historical Metallurgy (1978) 12 (2):88-93.
[4] The authors gratefully acknowledge materials received from the reference collection of the Smithsonian Institution's National Museum of the American Indian and the Smithsonian Office of the Provost for support in the form of a Scholarly Studies award.

Examples

Bruker Nano Analytics, Berlin, Germany
Webinar, November 6th, 2019

Micro-XRF on SEM



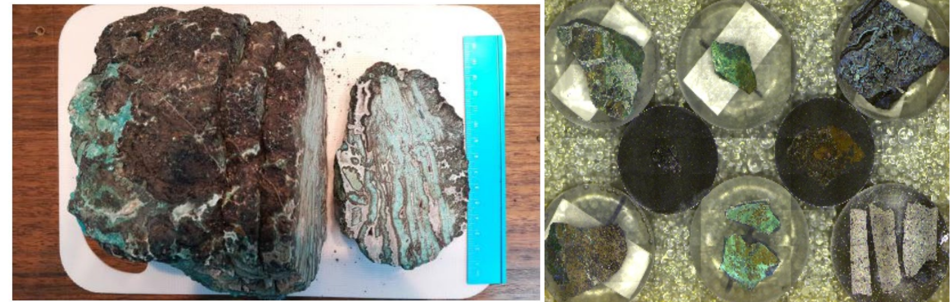
SEM-XRF (XTrace)
Rapid Stage: Applications

- **Large Area Maps Overview**
- **Applications**:
  - Geological
  - Environmental
  - Mining
  - Metallurgical
  - Archaeological
  - Industrial

Introduction
SEM-XRF (XTrace)

Analytical challenge

- Analysis of Large Areas:
  - Thin Sections / Probe Sections
  - Rock Slabs
  - Soil / Grain Samples



Why use Micro-XRF?

- Better sensitivity for element detection (Z>20)
- Detection of high energy X-ray lines

Terminology

For clarity and simplicity, analysis and data using a **MicroXRF on SEM (XTrace)** will be referred to as **SEM-XRF** whilst traditional data gathered from an e-beam source (W or FEG) will be referred to as **SEM-EDS**

Introduction
SEM-XRF (XTrace)

Specialised Rapid Stage for SEM-XRF (XTrace)

Maximum Analytical Area of Specialised Rapid Stage:

50 mm x 50 mm

Larger areas possible in combination with SEM-Stage

SEM-XRF (XTrace)

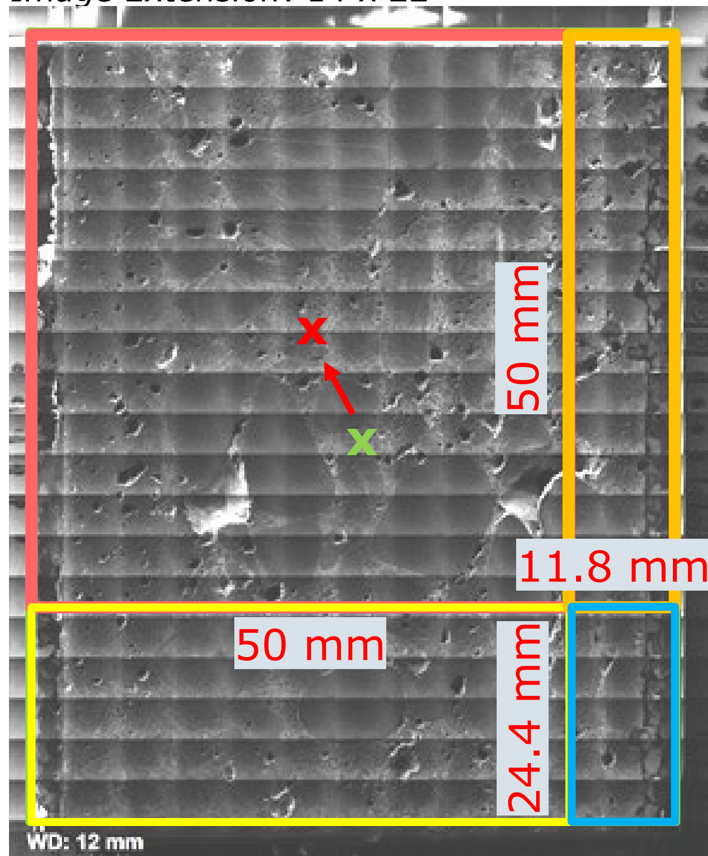
The source X-ray beam that interacts with the sample is in a fixed position. Therefore, the source X-ray beam cannot be controlled to raster as a standard SEM e-beam. Consequently, all mapping is via stage control (that is Stage Movement).

Large Area Maps
Rapid Stage + SEM Stage: SEM-XRF

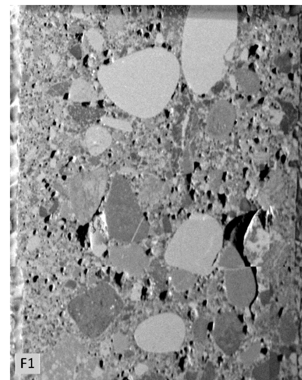
Large Samples: Concrete Block: 61.8 mm x 74.4 mm

Such samples require a combination of the Specialised high speed stage + SEM Stage. The sample is analysed in 4 maps which are mosaiced at the completion of the analysis.

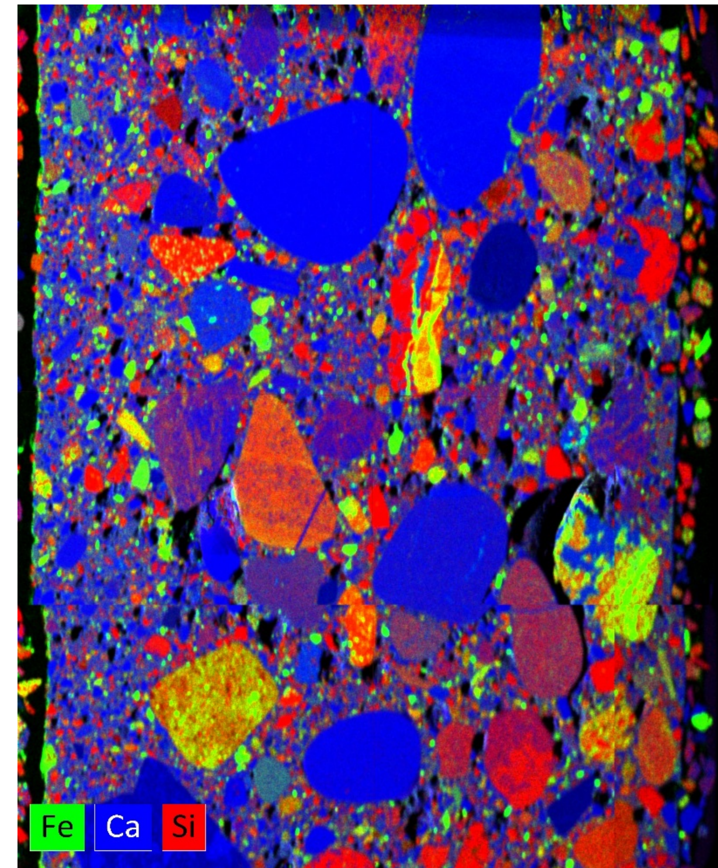
Image Extension: *14 x 22*



Photograph of the sample



X-Ray Intensity Map



Large Area Maps
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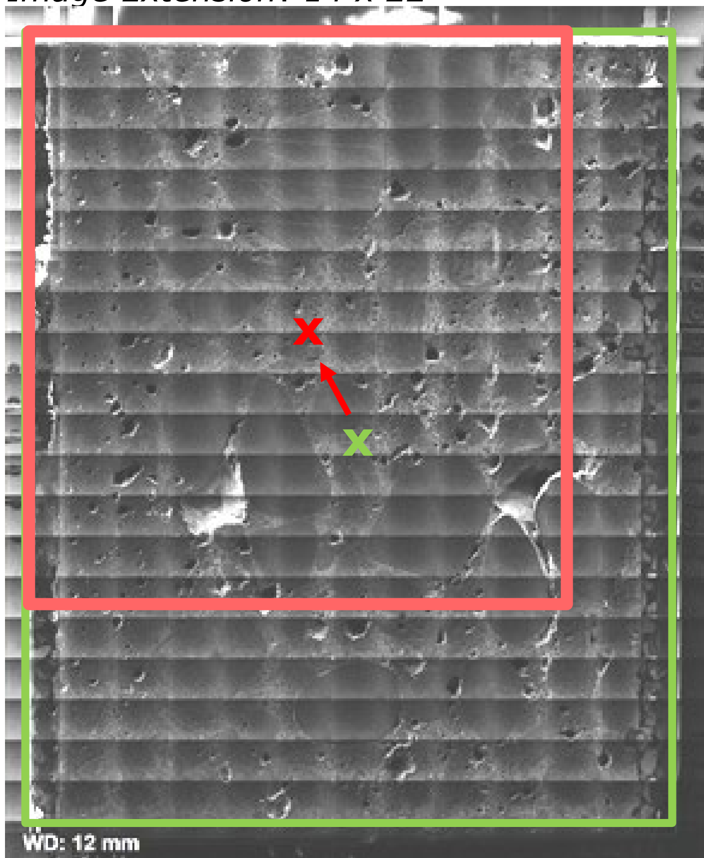
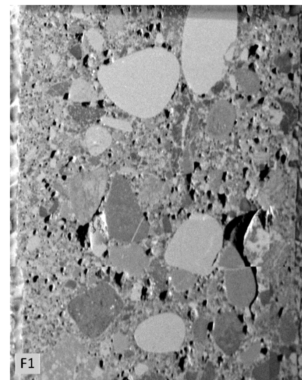
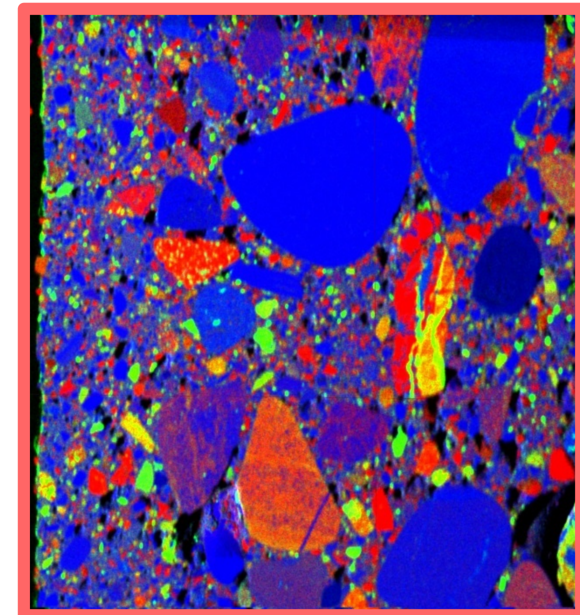


Photograph of the sample



X-Ray Intensity Map

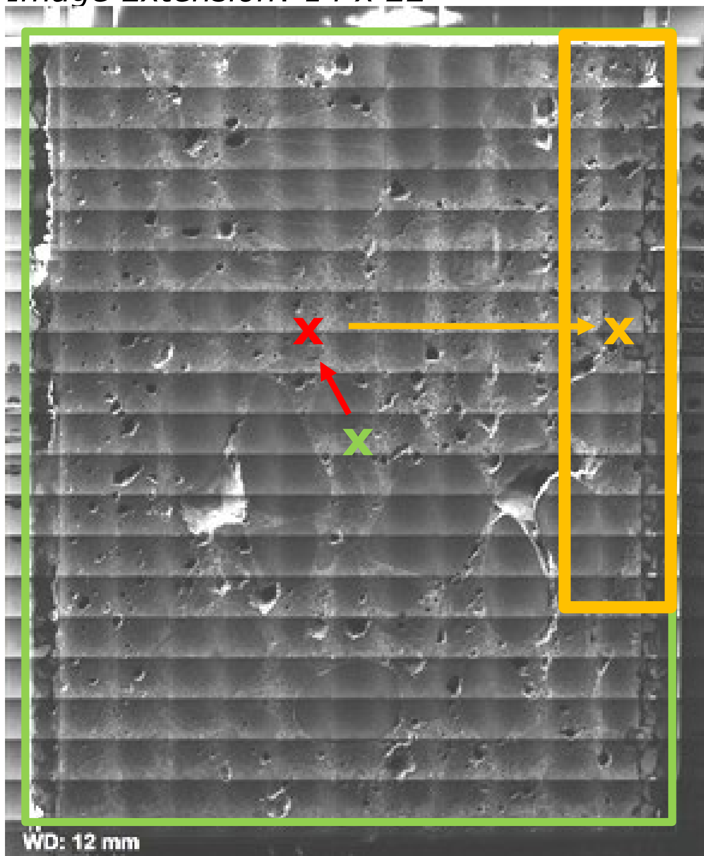


Large Area Maps
Rapid Stage + SEM Stage: SEM-XRF

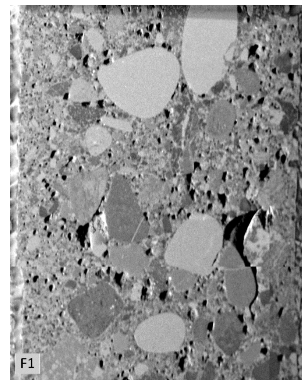
Large Samples: Concrete Block: 61.8 mm x 74.4 mm

Such samples require a combination of the Specialised high speed stage + SEM Stage. The sample is analysed in 4 maps which are mosaiced at the completion of the analysis.

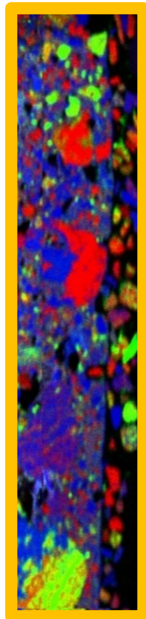
Image Extension: *14 x 22*



Photograph of the sample



X-Ray Intensity Map

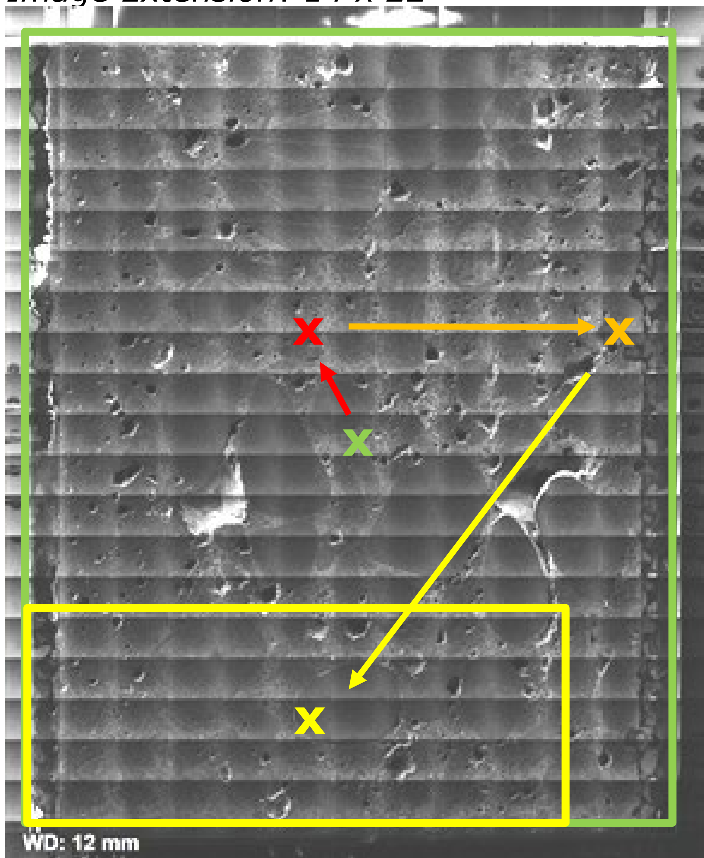


Large Area Maps
Rapid Stage + SEM Stage: SEM-XRF

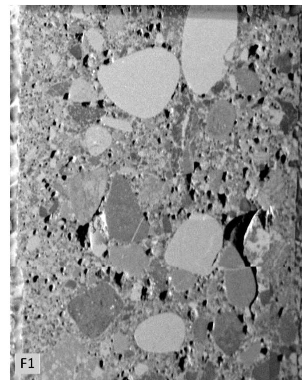
Large Samples: Concrete Block: 61.8 mm x 74.4 mm

Such samples require a combination of the Specialised high speed stage + SEM Stage. The sample is analysed in 4 maps which are mosaiced at the completion of the analysis.

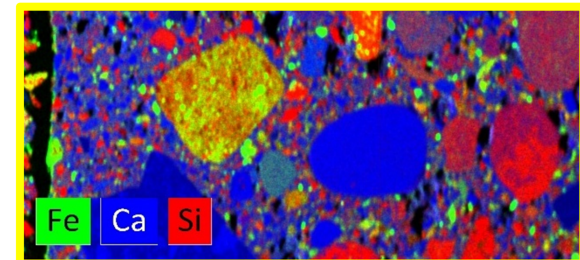
Image Extension: *14 x 22*



Photograph of the sample



X-Ray Intensity Map

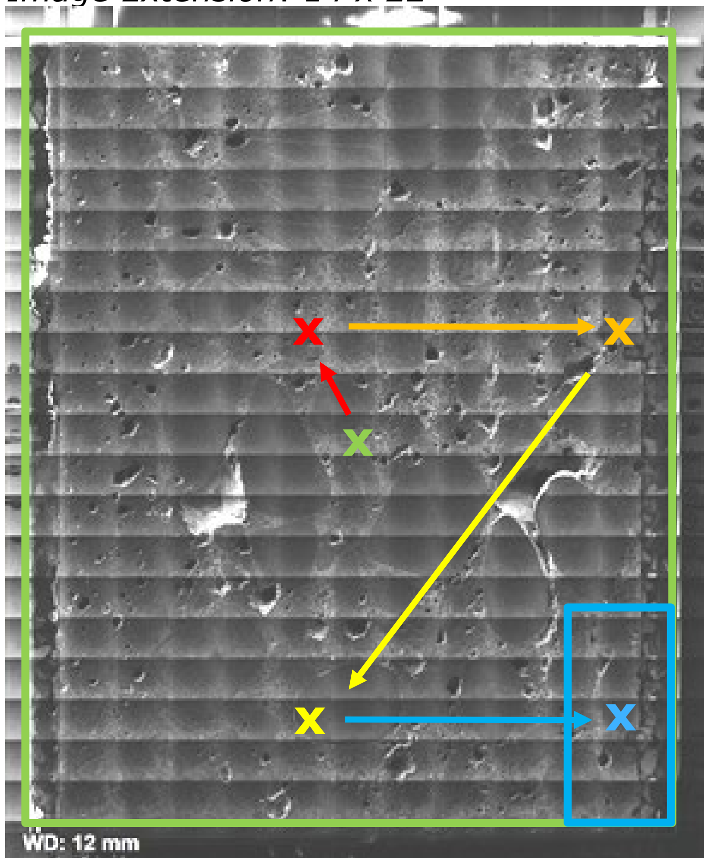


Large Area Maps
Rapid Stage + SEM Stage: SEM-XRF

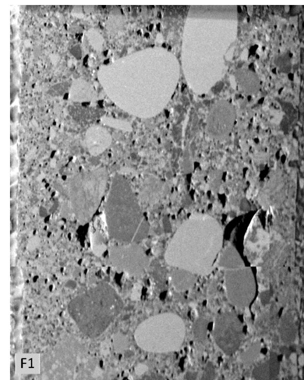
Large Samples: Concrete Block: 61.8 mm x 74.4 mm

Such samples require a combination of the Specialised high speed stage + SEM Stage. The sample is analysed in 4 maps which are mosaiced at the completion of the analysis.

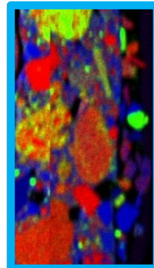
Image Extension: *14 x 22*



Photograph of the sample



X-Ray Intensity Map

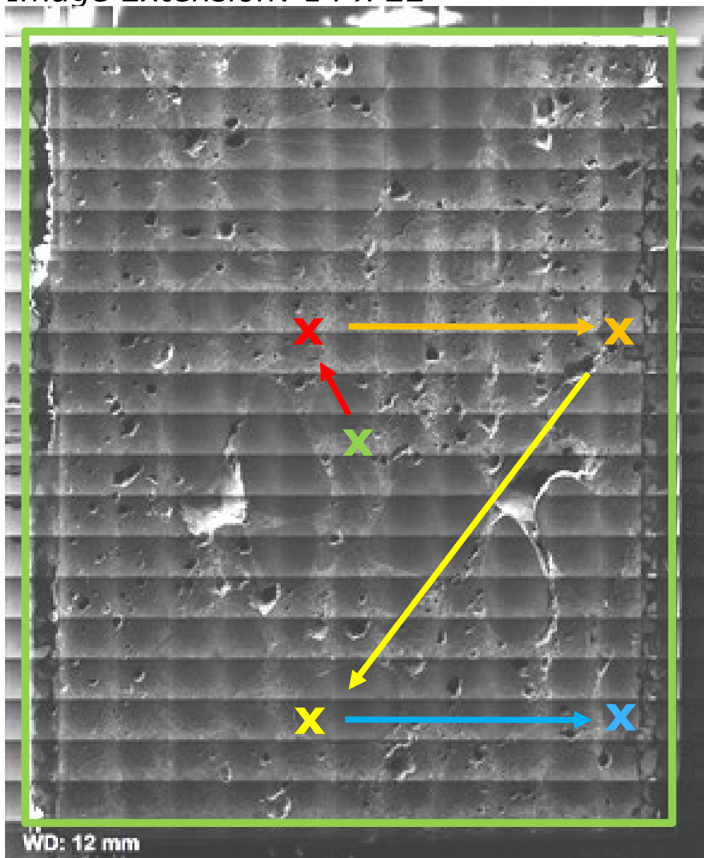


Large Area Maps
Rapid Stage + SEM Stage: SEM-XRF

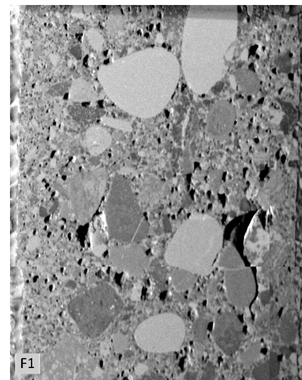
Large Samples: Concrete Block: 61.8 mm x 74.4 mm

Such samples require a combination of the Specialised high speed stage + SEM Stage. The sample is analysed in 4 maps which are mosaiced at the completion of the analysis.

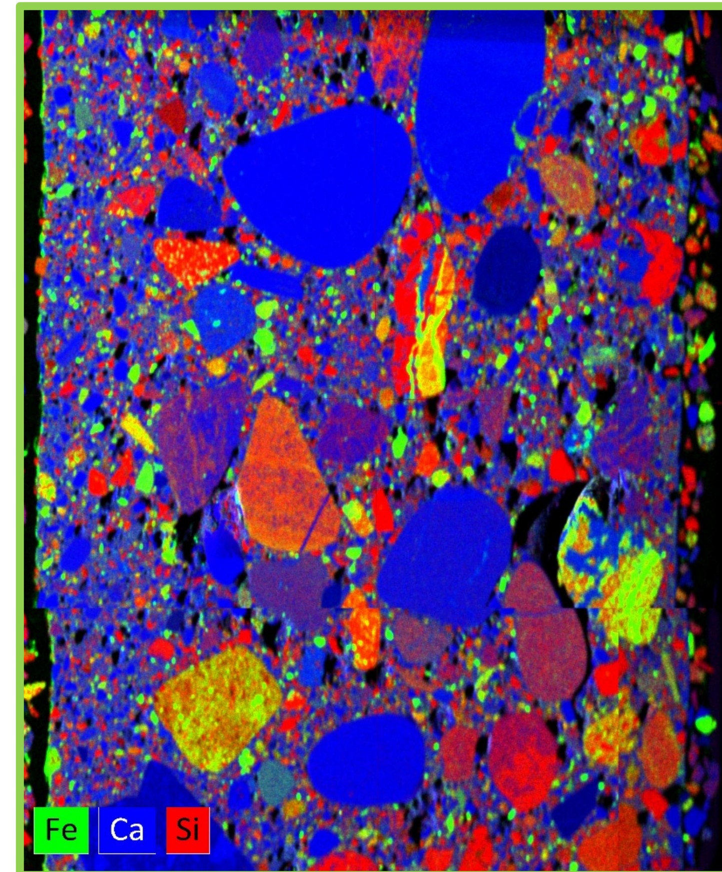
Image Extension: *14 x 22*



Photograph of the sample



X-Ray Intensity Map



Geological Applications:
Exotic-Cu Deposits

Bruker Nano Analytics, Berlin, Germany
Webinar, November 6th 2019

Geological Applications:
Exotic-Cu Deposits

320000 380000 440000 500000 560000 620000

7550000
7500000
7450000
7400000
7350000

Océano Pacífico

El Abra
Radomiro Tomic
Chuquicamata
Mina Sur
Calama
Sierra Gorda
El Tesoro
Mejillones
Carmen Alto
Baquedano
Antofagasta
Antofagasta

24
25
1
26
28
5

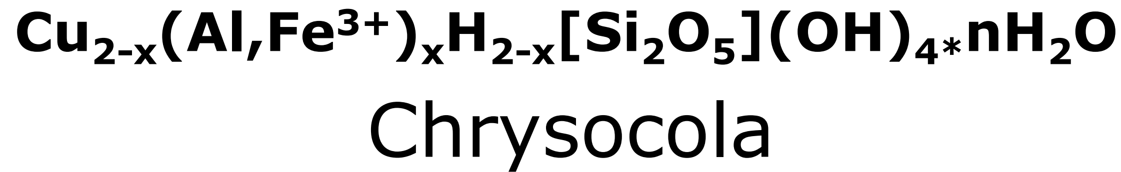
Image Landsat
© 2014 Mapcity

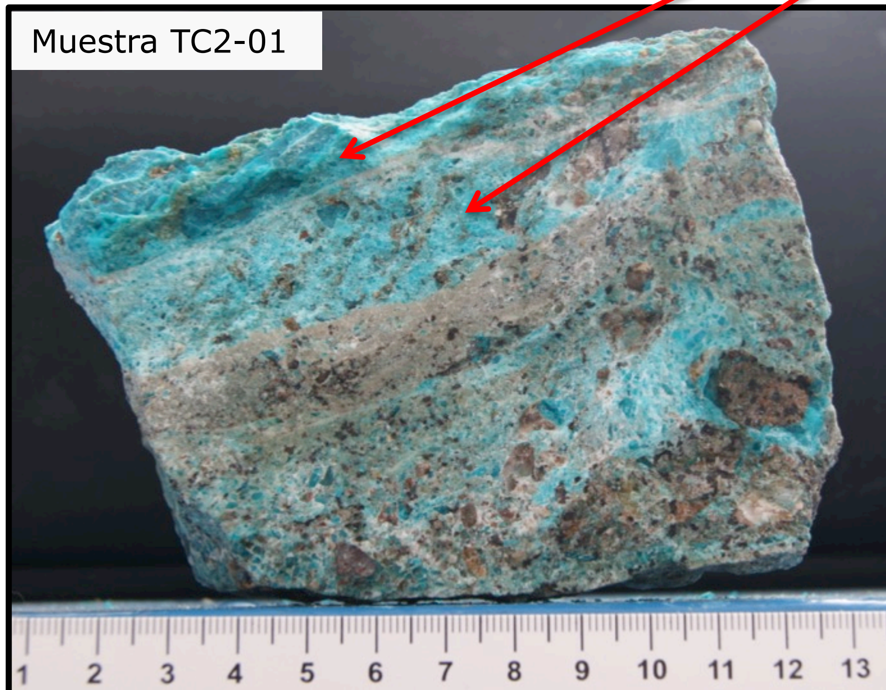
5

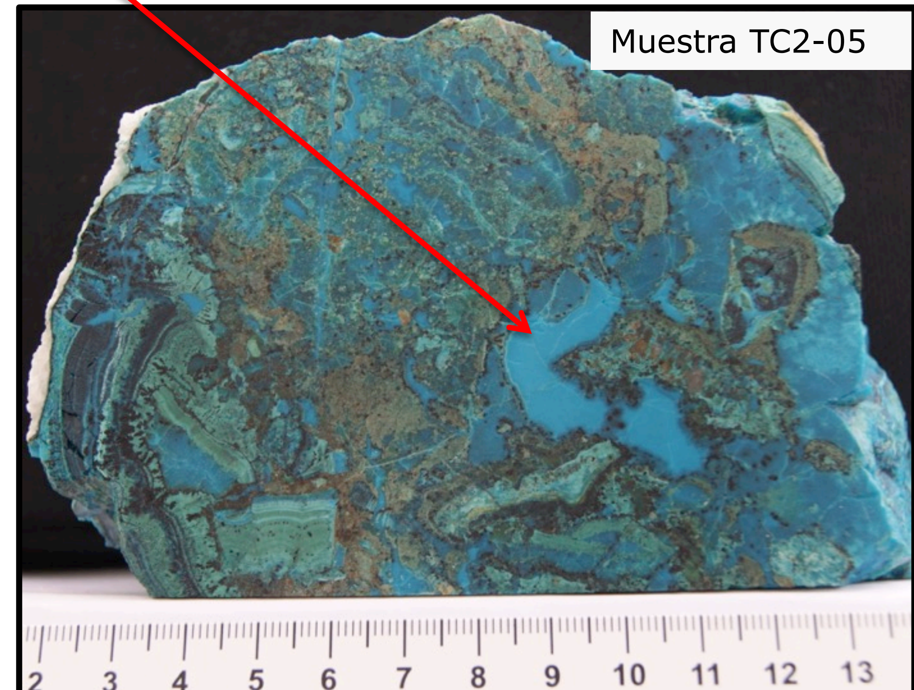
Geological Applications:
Exotic-Cu Deposits

- 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 – Rock Sample



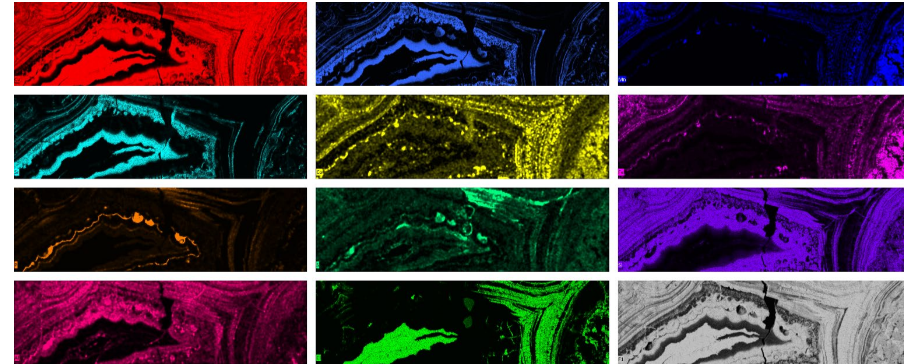




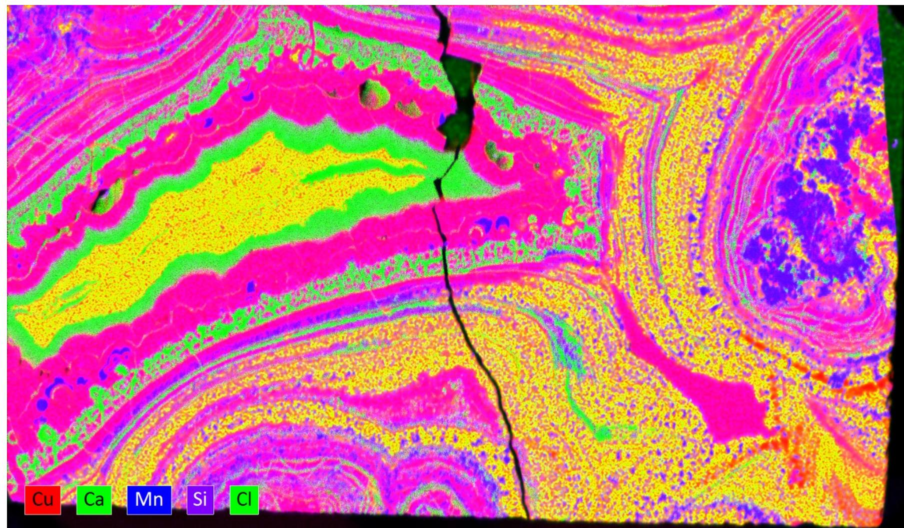
Geological Application:
Exotic Cu Deposits

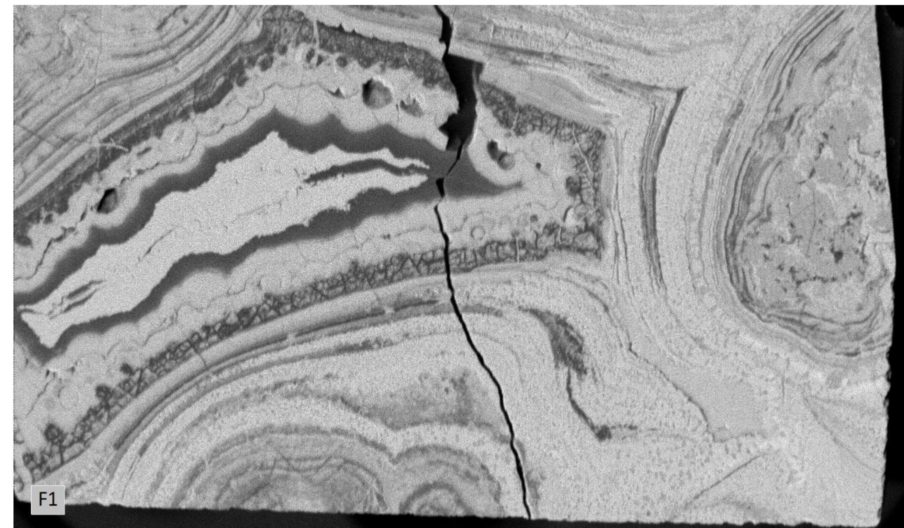
Large Area Map
Sample Size: Polished Section: 45 x 30 mm
Sample from El Tesoro, Chile.
Clearly Defined Elemental and Mineralogical Phases
Can identify the presence of trace elements, in this case, Cobalt (Co), Manganese (Mn), Strontium (Sr)

Analytical Parameters:
Tube Voltage: Rh at 50 kV
Anode Current: 600 µA
Pixel Spacing: 25 µm
Analytical Time: 101 mins



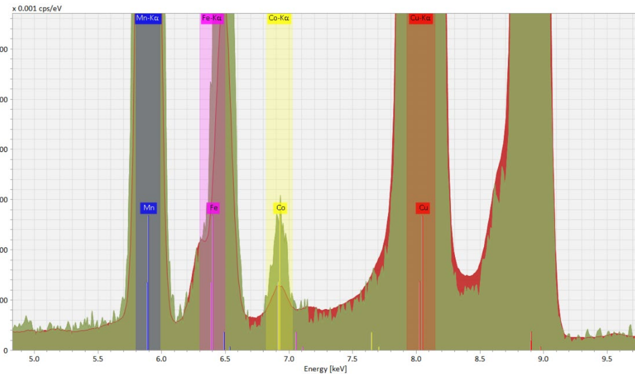
Top: Elemental Maps; Bottom Left: Mixed Elemental Map; Bottom, Right: X-Ray Intensity Map.



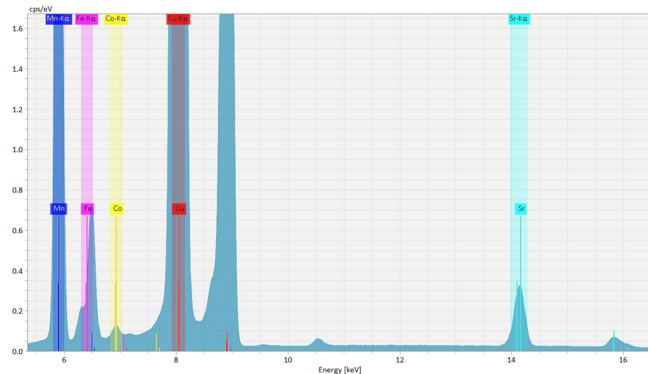


Exotic-Cu Deposits:
Elemental Mapping: Benefits of SEM-XRF

- Identify the presence of trace elements, e.g, Cobalt (Co) at 6.93 keV



- Identify high energy X-ray lines, e.g Strontium (Sr) at 14.14 keV



F1

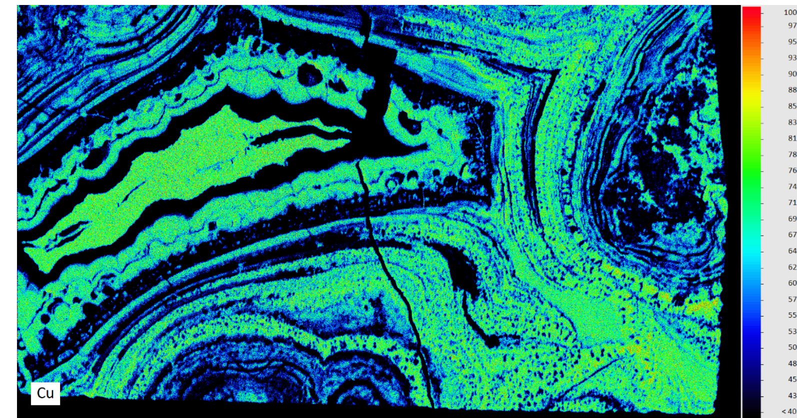
Sr

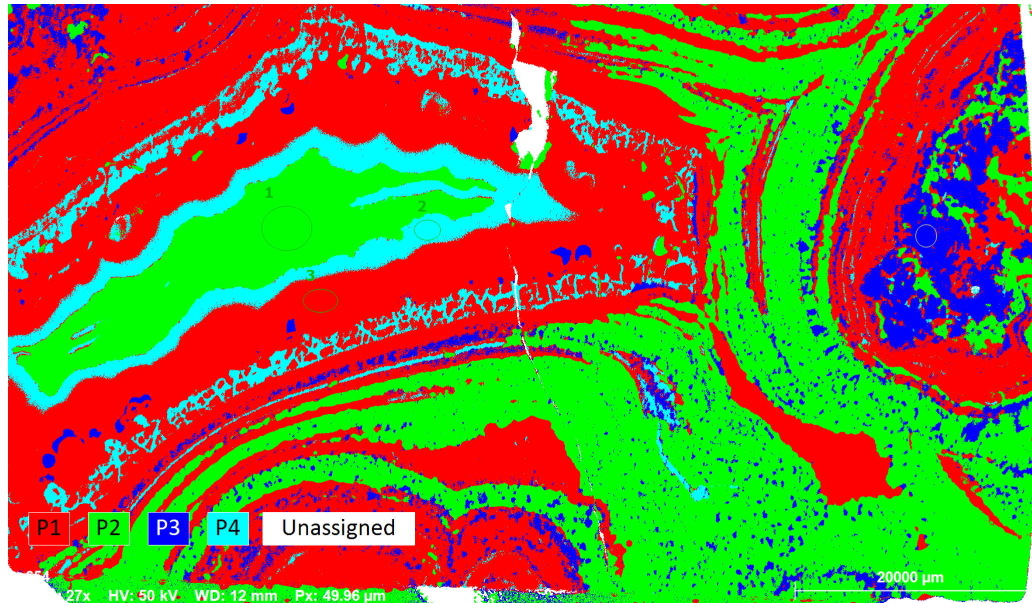
Co

Cu Ca

Exotic-Cu Deposits:
Mineral Phases

The results can be processed to determine various mineral phases and their area percentages and compositions within the sample, in this case, the various Cu phases.

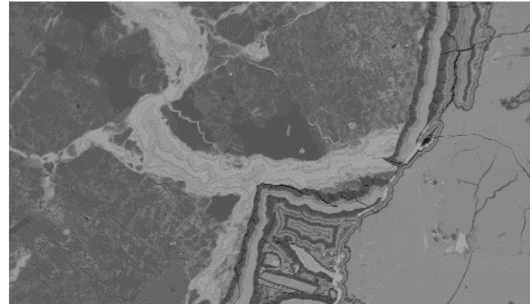




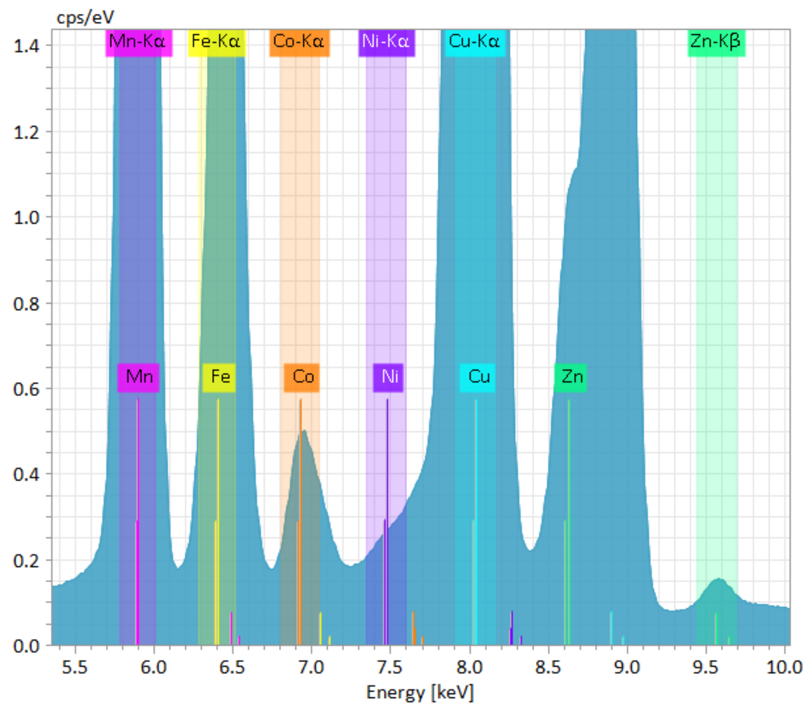
Counts/Pixel	Area
P1	45.6 %
P2	35.0 %
P3	7.9 %
P4	7.7 %
Unassigned	3.8 %

P1: Chrysocolla
P2: Atacamite
P3: Cu-Mn Wad
P4: Carbonate

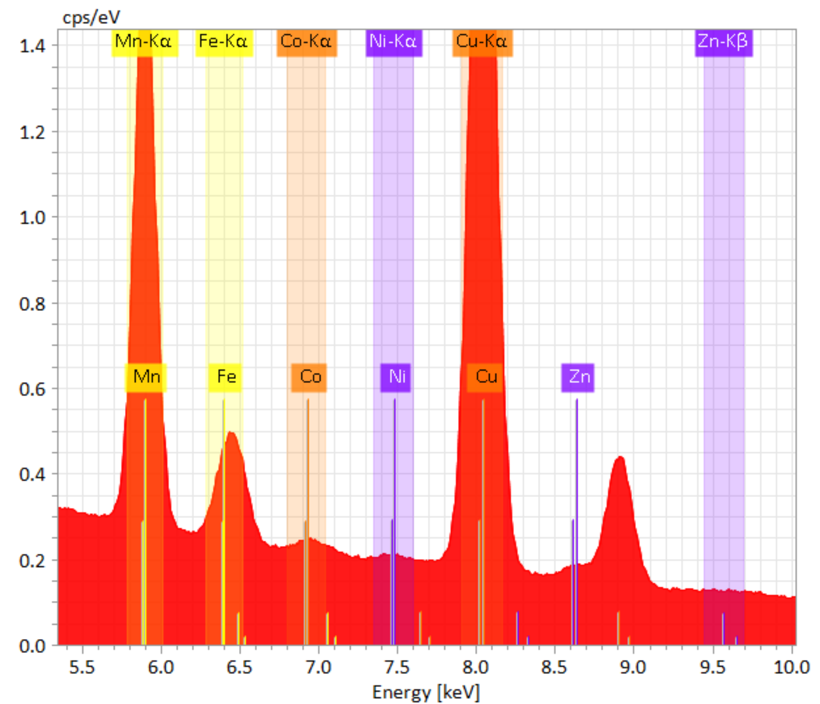
Exotic-Cu Deposits:
Elemental Mapping - Comparison



SEM-XRF (XTrace)



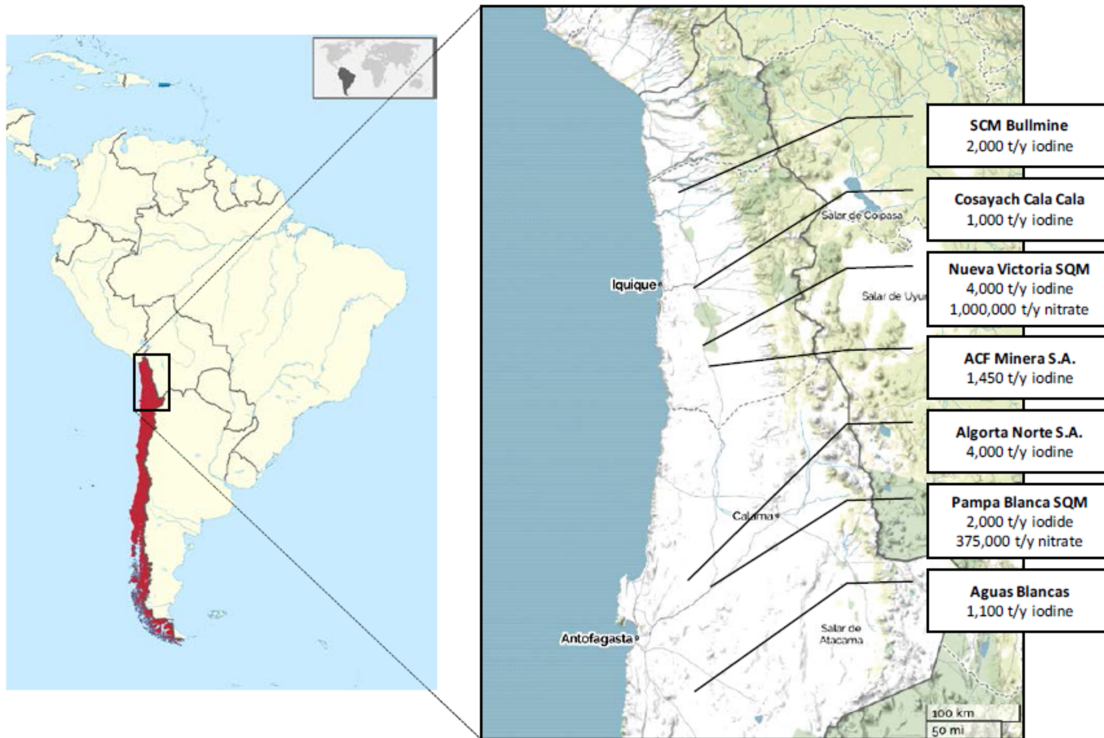
SEM-EDS (e-beam)



Geological Applications:
Nitrate Deposits – Rock Sample

Bruker Nano Analytics, Berlin, Germany
Webinar, November 6th 2019

Nitrate Deposits
Differentiation of Nitrates



Chilean operations of caliche exploitation in Northern Chile (Atacama Desert) and their annual production

Northern Chile:

Most people know this region for Cu production, which is also the largest Cu resource in the world.

In addition, because of its unique climate in the Atacama desert (hyper-arid), formed Nitrates and associated minerals:

- Iodates
- Chromates
- Sulphates

Largest Iodine resource in world, associated with nitrates.

SEM-XRF combined analysis

Differentiation of Nitrates in Caliche

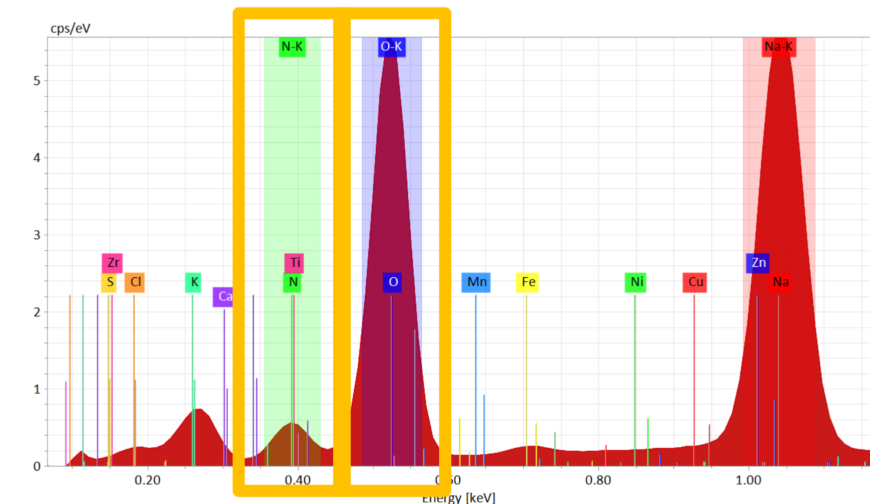
Detection of O and N:

Important for the identification and definition of minerals of interest

Important Minerals Groups:

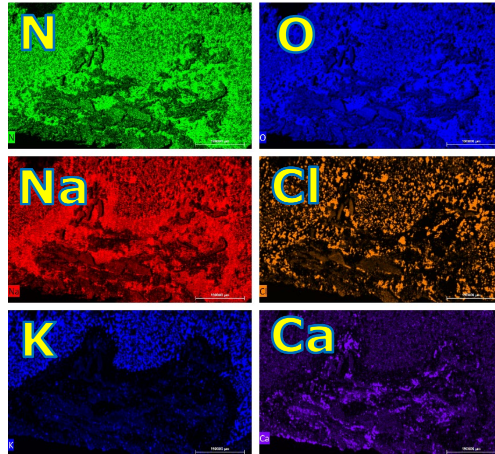
Salts, Nitrates, Iodates

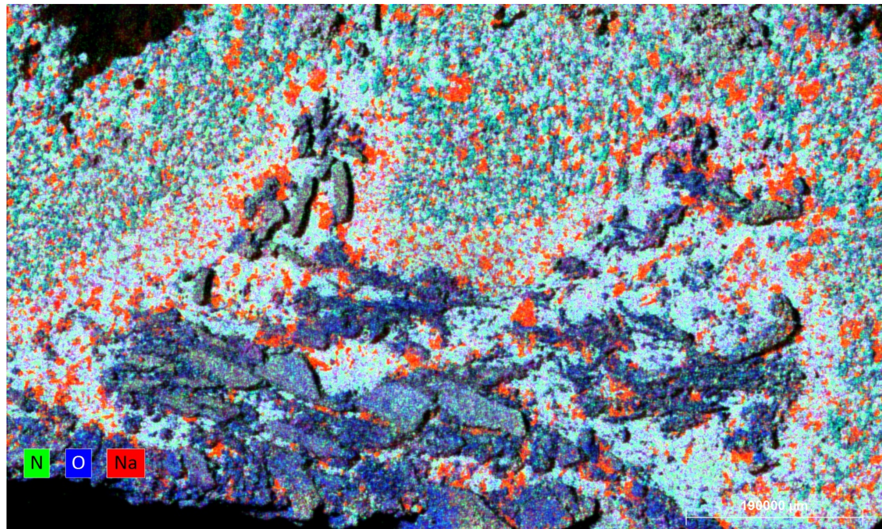
Nitratine	$NaNO_3$
Halite	$NaCl$
Niter	KNO_3
Langbeinite	$K_2Mg_2(SO4)_3$
Loeweite	$Na_{12}Mg_7(SO_4)_{13.15}H_2O$
Darapskite	$Na_3(NO_3)(SO_4).H_2O$
Ulexite	$NaCaB_5O_{9.8}H_2O$



SEM-XRF combined analysis
Differentiation of Nitrates in Caliche

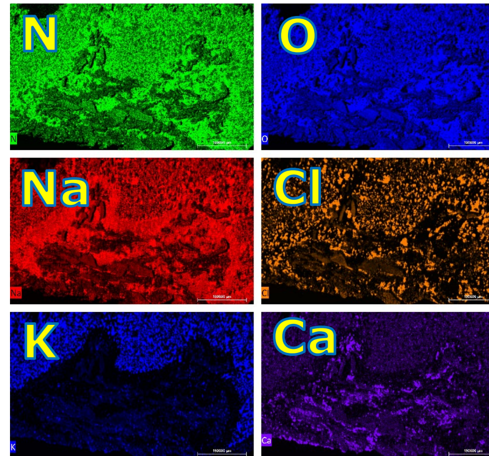
Association of N and Na: Nitratine ($NaNO_3$)

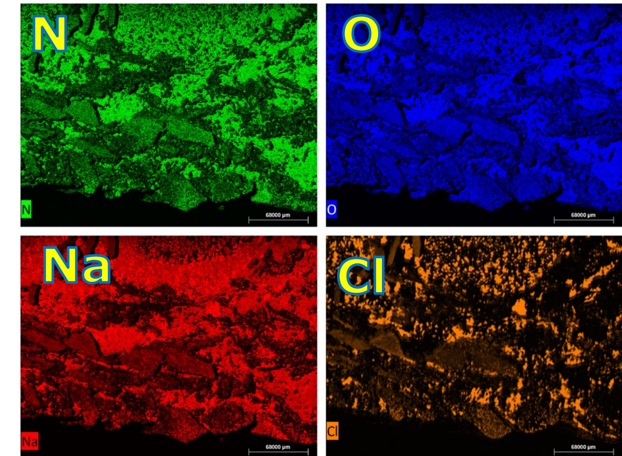


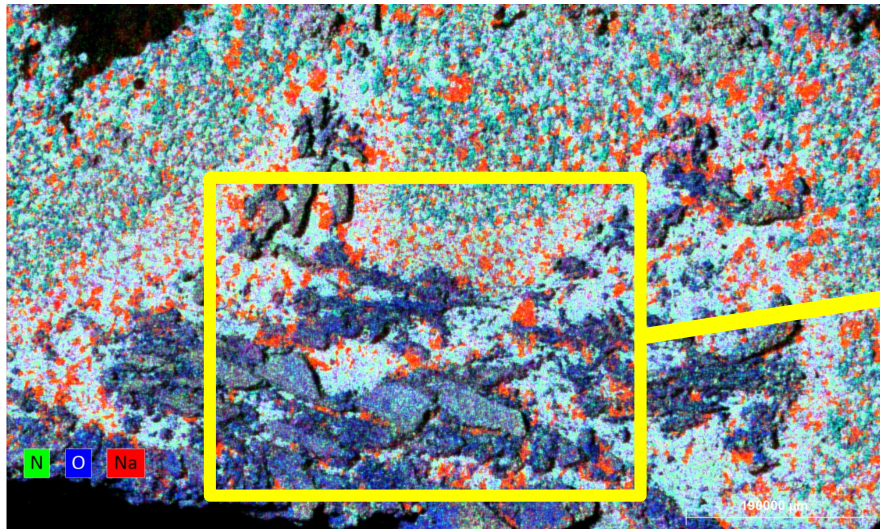


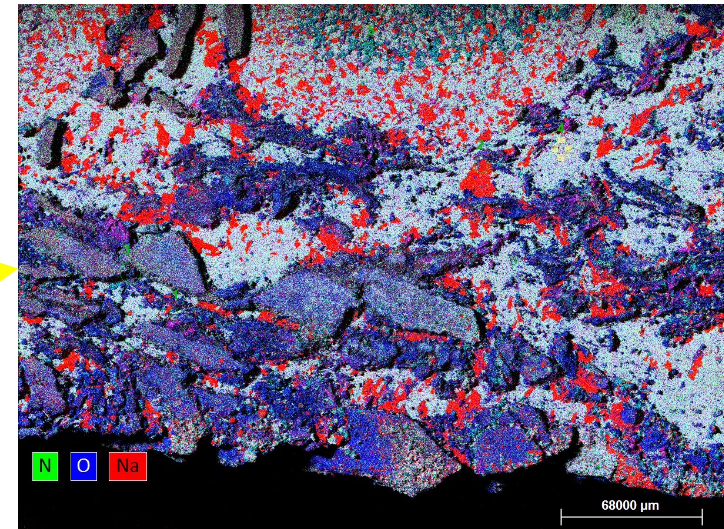
SEM-XRF combined analysis
Differentiation of Nitrates in Caliche

Association of N and Na: Nitratine ($NaNO_3$)









SEM-XRF combined analysis
Differentiation of Nitrates in Caliche

Detection of trace and high energy elements: Se, Br, Sr, Zr

Se 68000 µm

Br 68000 µm

Sr 68000 µm

Zr 68000 µm

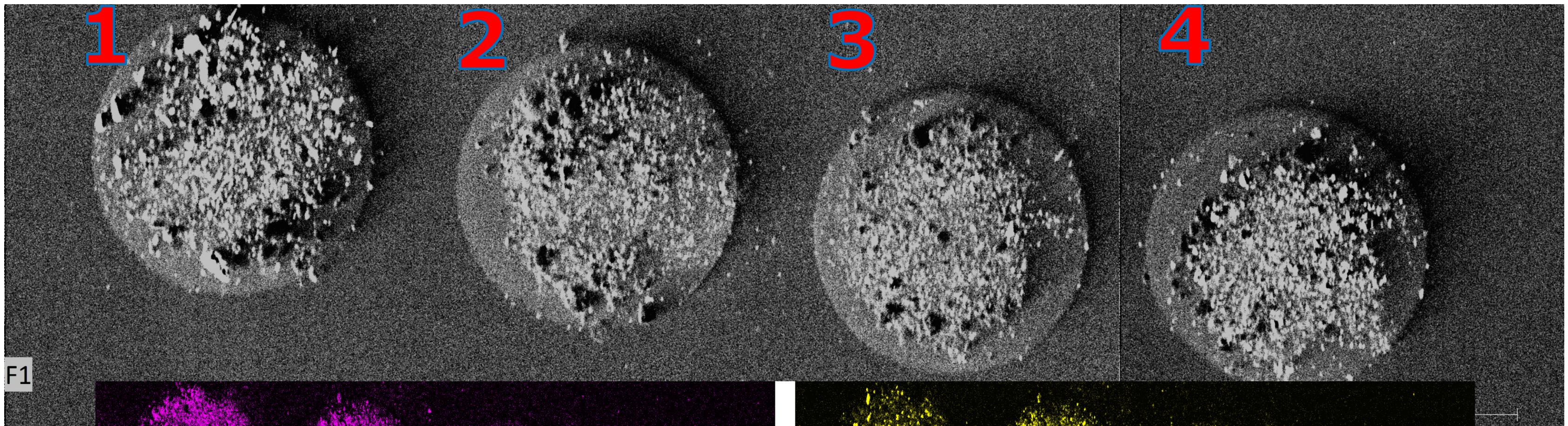
SEM-XRF combined analysis
Differentiation of Nitrates in Caliche

Detection of trace and high energy elements:
Se, Br, Sr, Zr

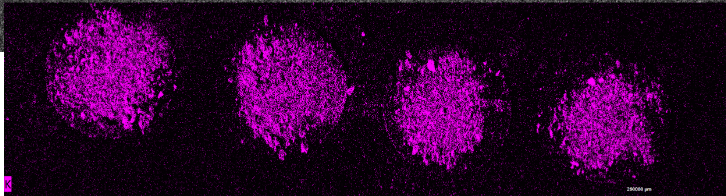
Environmental Applications:
Toxic Elements - Soil Sample and Rock Sample

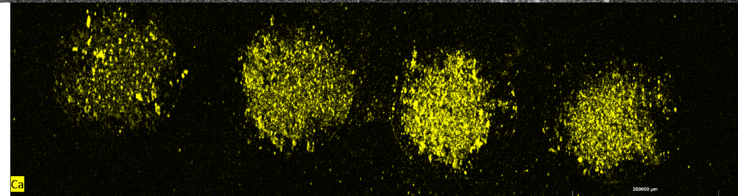
Bruker Nano Analytics, Berlin, Germany
Webinar, November 6th 2019

Analysis of Soil Samples:
Large Area Maps



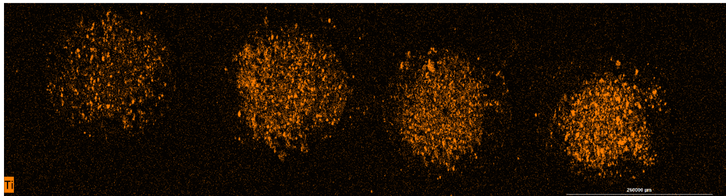
K

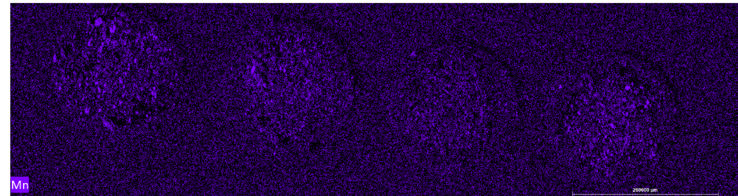




Ca

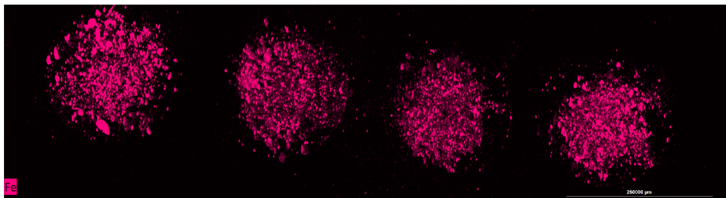
Ti

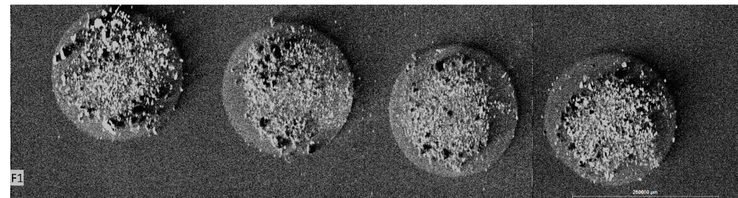




Mn

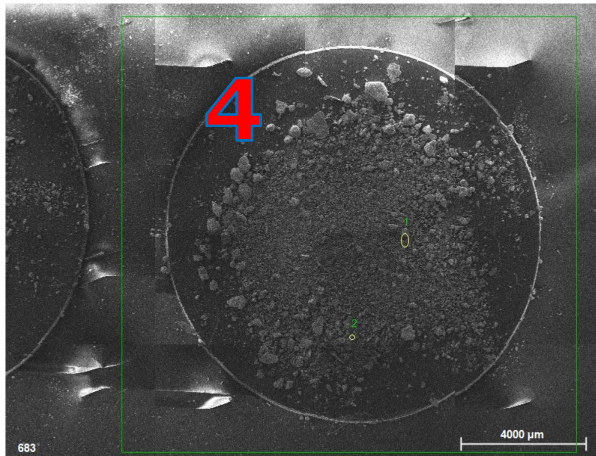
Fe

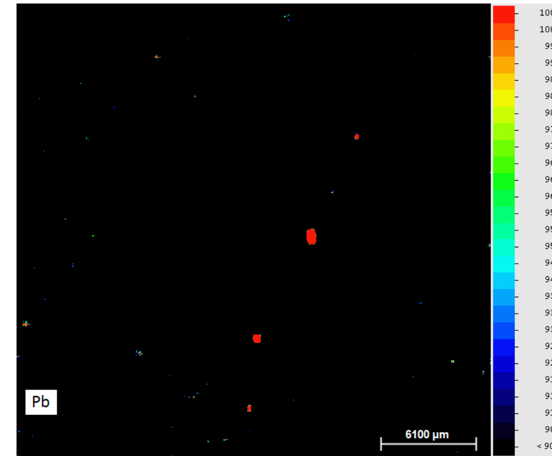




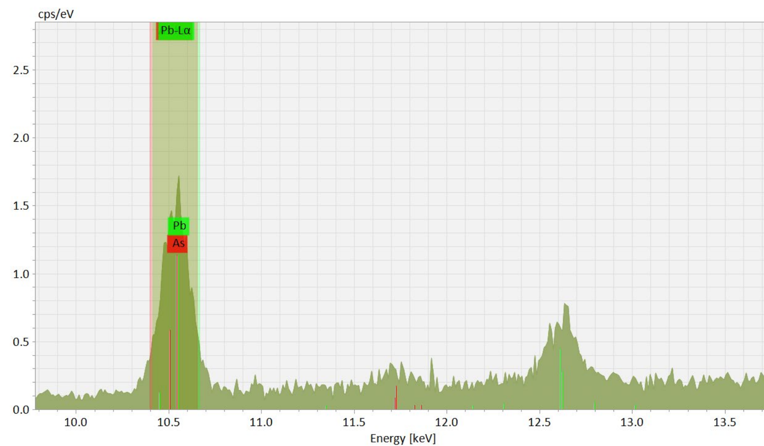
F1

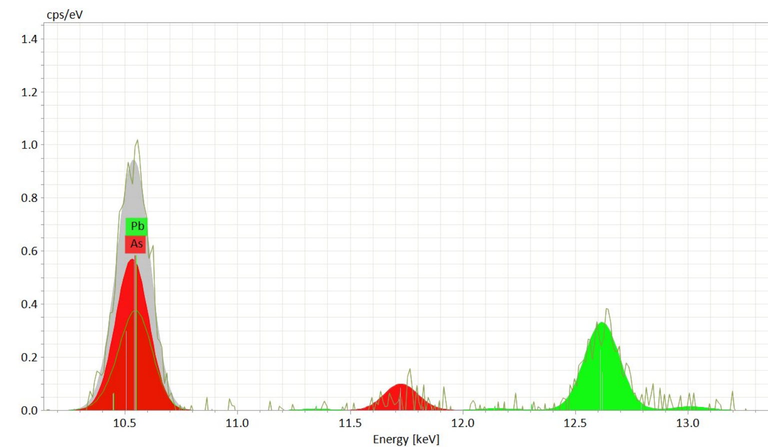
Analysis of Soil Samples:
Large Area Maps





Pb



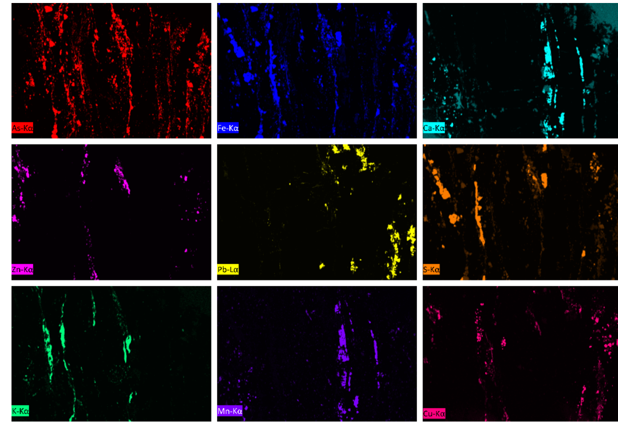


After Maximum Pixel Spectra determines presence of Trace elements.

Detailed investigation confirms presence of both Pb and As

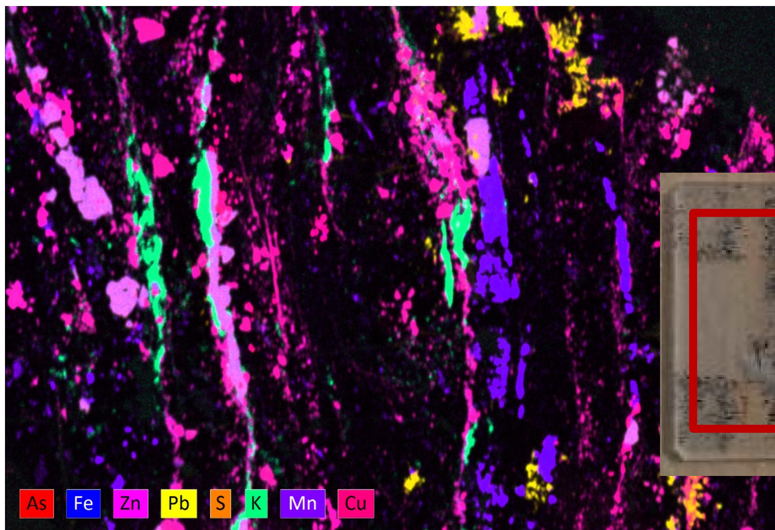
Analysis of Soil Bedrock:
Large Area Maps

Polished Sections:
Standard Size: 45 x 30 mm
Such samples can be completely analysed using the Specialised high speed stage only.
Example: *Soil Sample from Korea*



Analytical Parameters:
Tube Voltage: Rh at 50 kV
Anode Current: 600 uA
Pixel Spacing: 25 um
Analytical Time: 755 mins

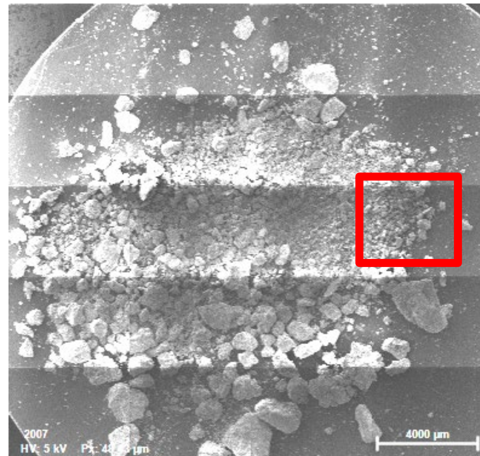
Top: Elemental Maps; Bottom Left: Mixed Elemental Map; Bottom, Right: X-Ray Intensity Map.

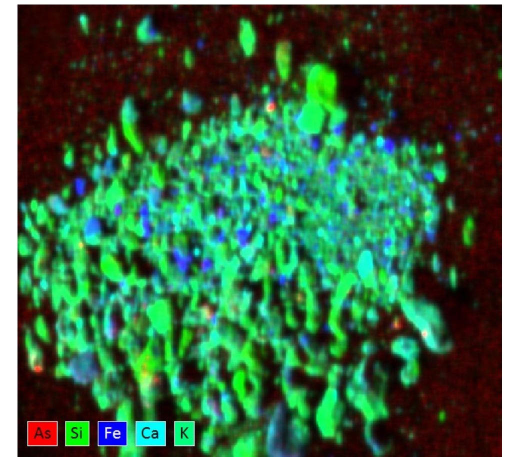


Analysis of Soil:
Large Area Maps

Large Samples
Loose Soil Sample: 40 mm circle
Such samples can be completely analysed using the Specialised high speed stage only.
Example: *Soil Sample from Korea*

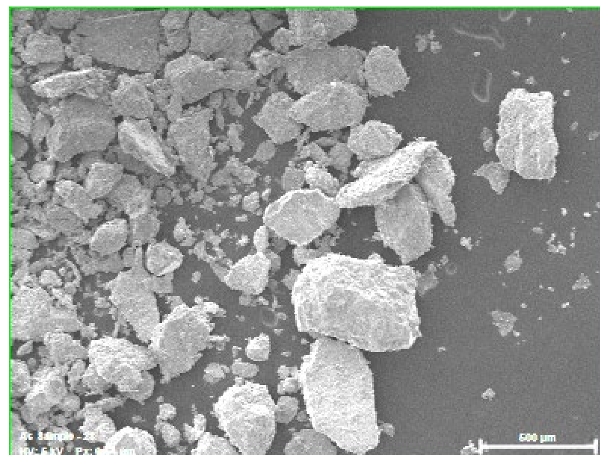
Analytical Parameters:
Tube Voltage: Rh at 50 kV
Anode Current: 600 uA
Pixel Spacing: 50 um
Analytical Time: 605 mins

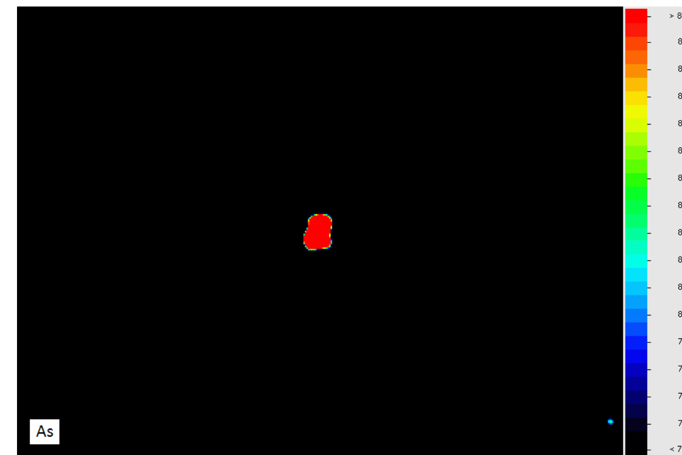




Top: Right: SEM Image and Left: Mixed Elemental Map
Bottom: Right: SEM Image and Left: Arsenic (As) Elemental Map

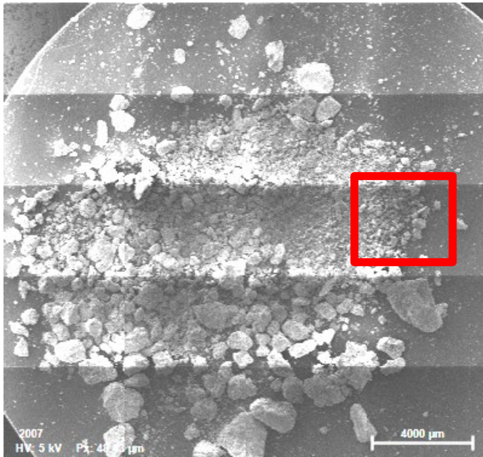
Analytical Parameters:
Tube Voltage: Rh at 50 kV
Anode Current: 600 uA
Pixel Spacing: 25 um
Analytical Time: 26 mins



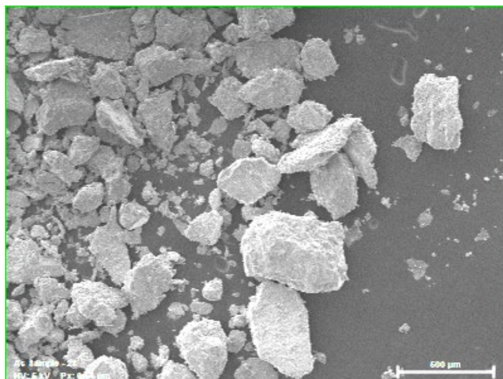


Analysis of Soil: Variable Parameters - Speed

Analytical Parameters:
Tube Voltage: Rh at 50 kV
Anode Current: 600 uA
Pixel Spacing: 20 um



Top: SEM Image
Bottom: SEM Image zoom
Right: Arsenic (As) Elemental Map

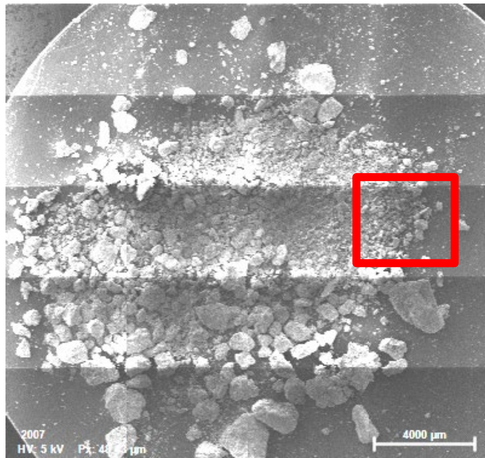


Increased Stage Speed:
(um per second)
500
1000
2000
3000
4000
5000

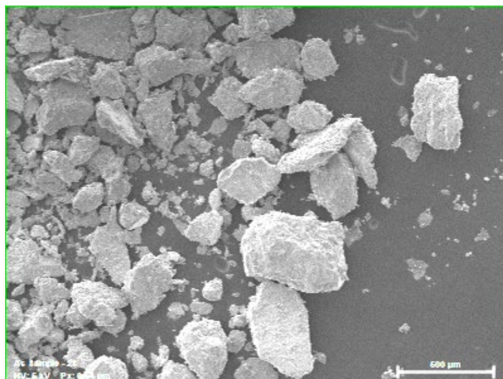
Decreased Analytical Time :
(minutes)
39
19
10
6
5
4

Analysis of Soil:
Variable Parameters - Pixel

Analytical Parameters:
Tube Voltage: Rh at 50 kV
Anode Current: 600 uA
Pixel Spacing: 20 um



Top: SEM Image
Bottom: SEM Image zoom
Right: Arsenic (As) Elemental Map



Increased Pixel Size
(micrometers)
Scan Speed
600
400
200
100

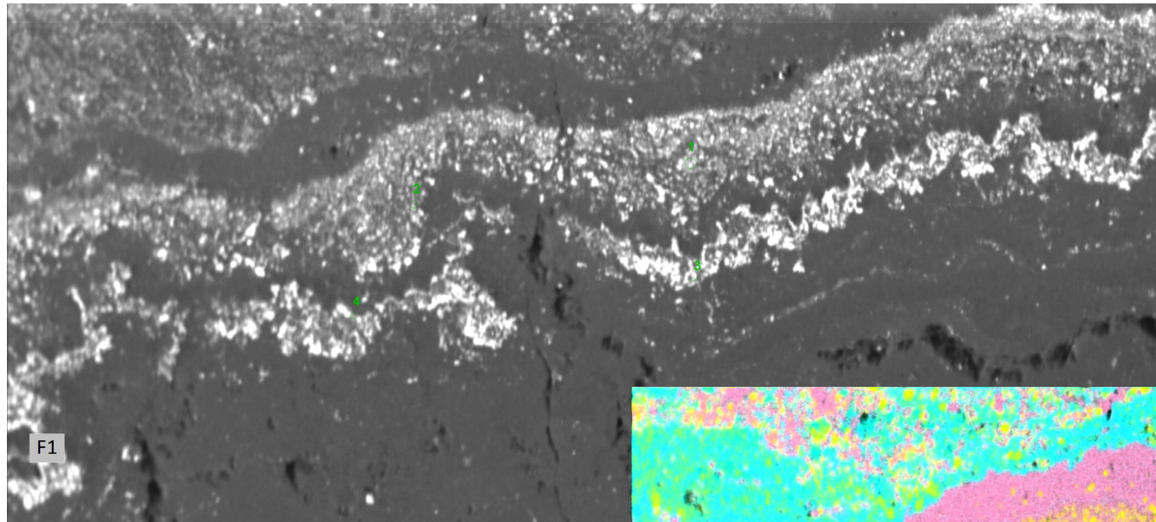
Decreased Analytical Time :
(minutes)
39
26
13
5

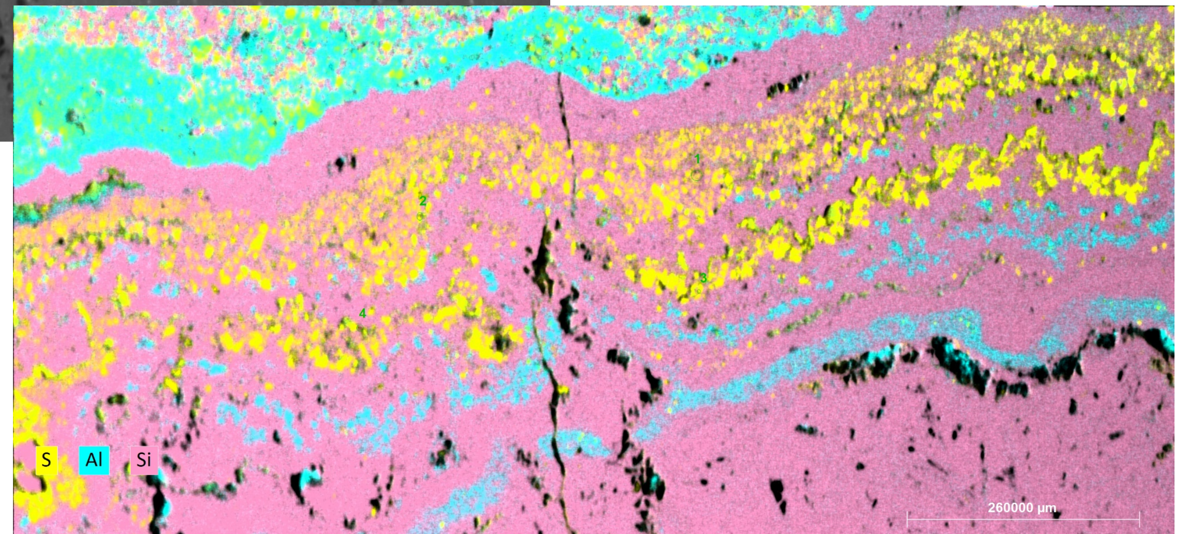
As
As
As
As

Mining and Exploration Applications: Epithermal Gold – Rock Sample

Bruker Nano Analytics, Berlin, Germany
Webinar, November 6th 2019

Mining and Exploration Applications: Epithermal Gold





Epithermal Gold-bearing rock sample from Karangahake, New Zealand

Mining and Exploration Applications: Epithermal Gold

S

K

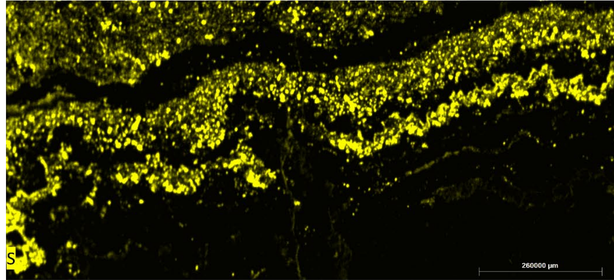
Al

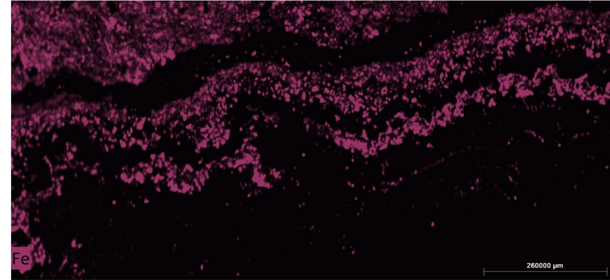
Si

Epithermal Gold-bearing rock sample from Karangahake, New Zealand

Mining and Exploration Applications: Epithermal Gold

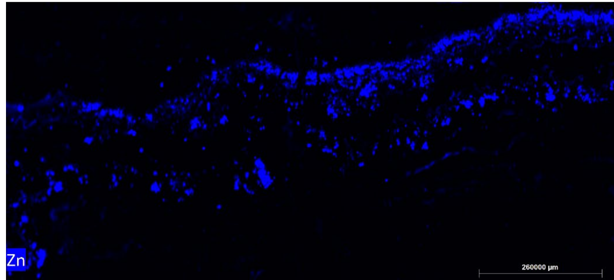
S

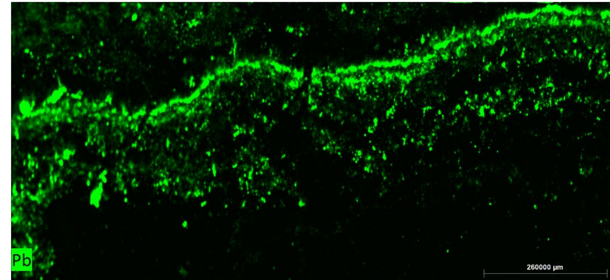




Fe

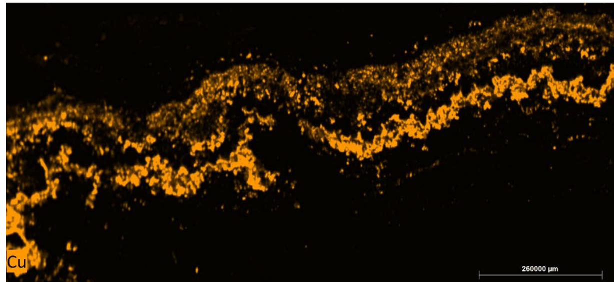
Zn

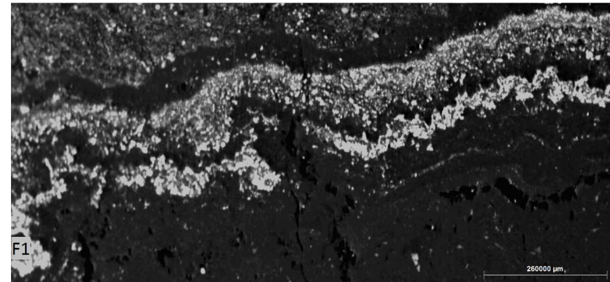




Pb

Cu





F1

Sulphide Mineralogy:

Banded Textures

Mining and Exploration Applications: Epithermal Gold

S

Fe

Zn

Pb

Cu

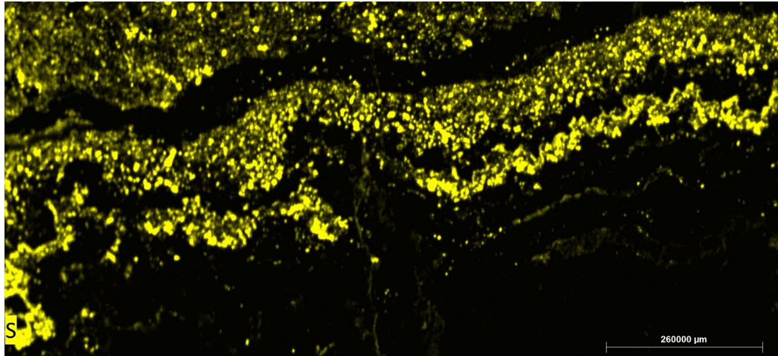
F1

Select various areas within the map to confirm composition and sulphide mineralogy.

The sulphide mineralogy is: pyrite, sphalerite, chalcopyrite, and galena.

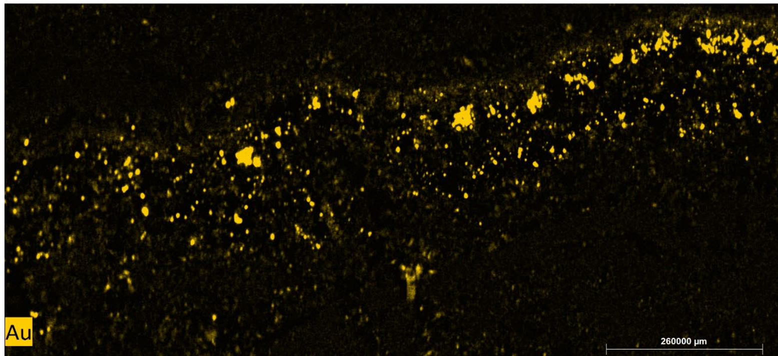
Mining and Exploration Applications: Epithermal Gold

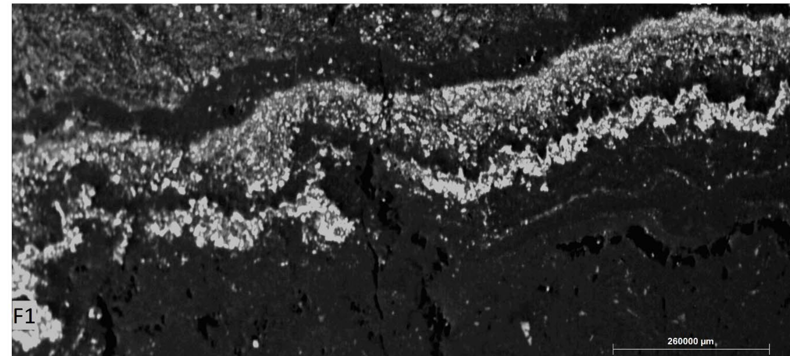
S



Ag

Au





F1

Search for Elements and Minerals of interest. Use "Maximum Pixel Spectrum" to identify elements not obvious in the "Total Map Spectrum".

Trace Mineral Phases and High Energy X-Ray Element Lines

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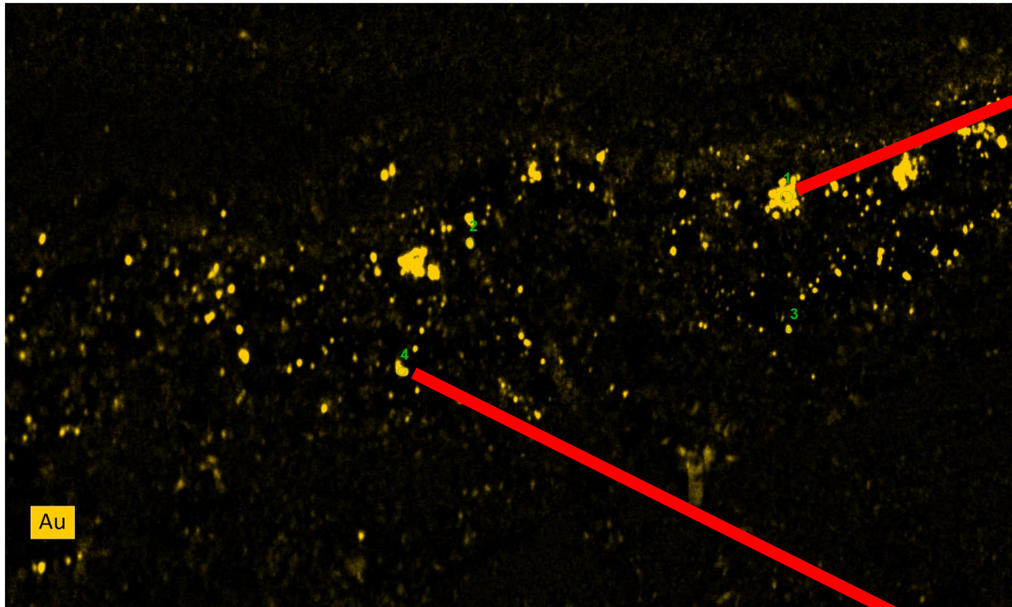
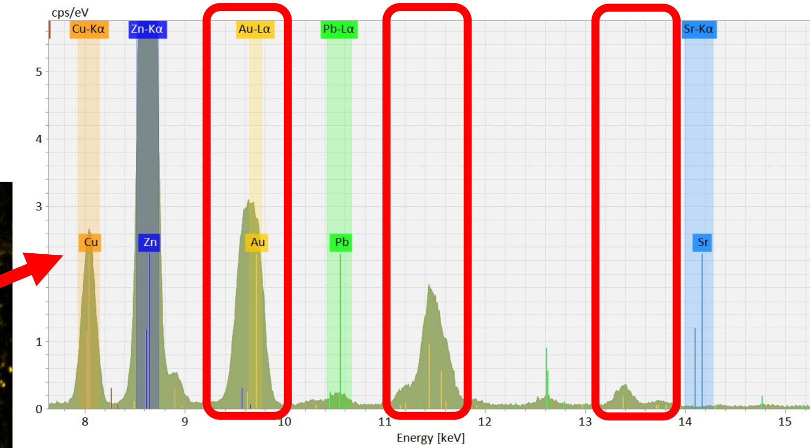
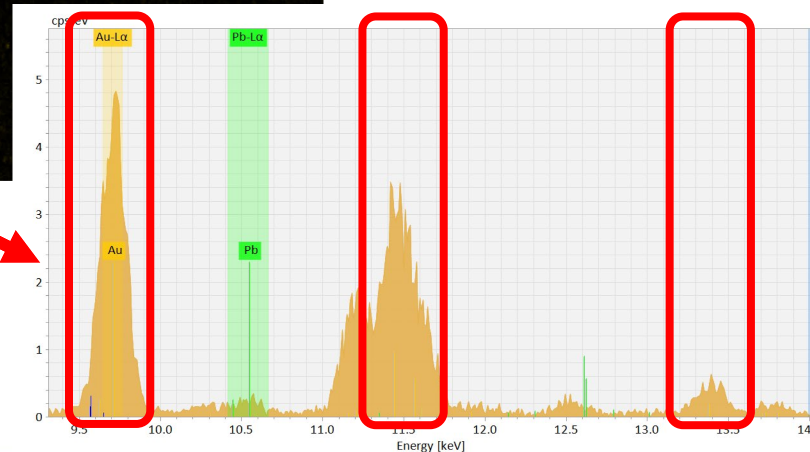






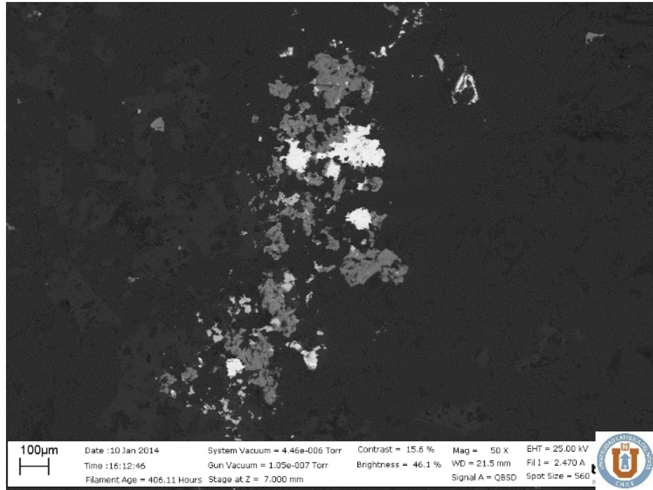
Presence of Au Mineralogy.

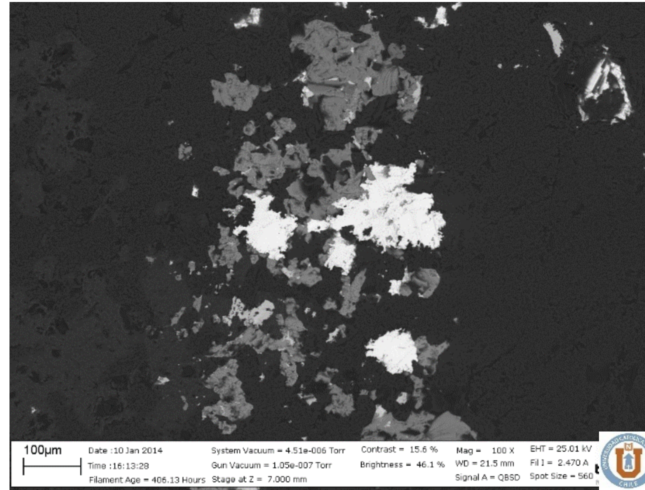
Often overlapping Peaks with Zn and Pb

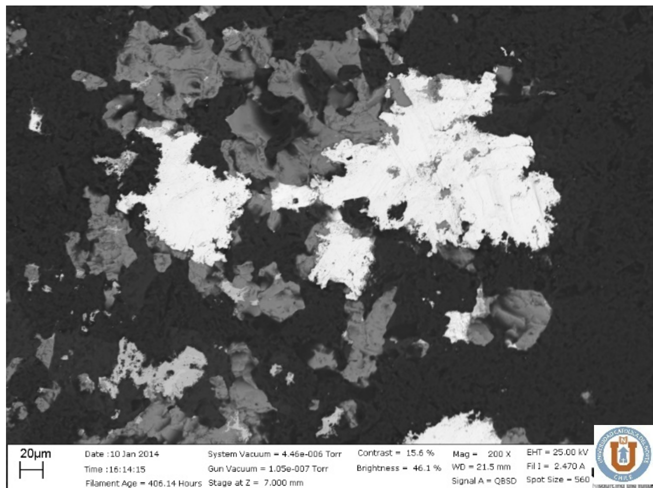
Confirmed by presence of all Au peaks:

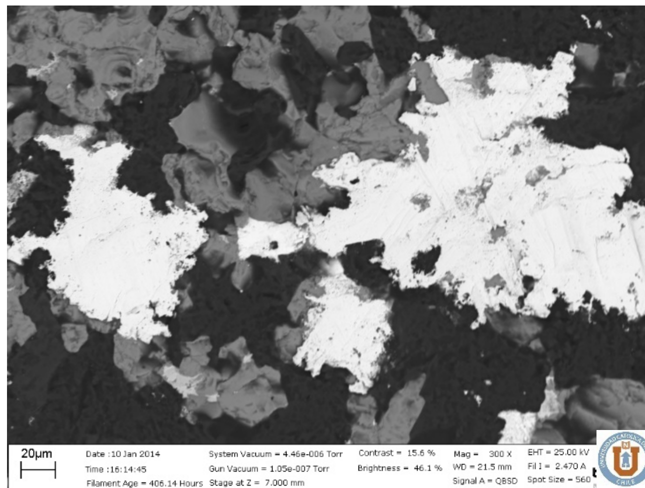
Au Lα (9.713 KeV) and Lβ (11.443 KeV)

Mining and Exploration Applications: Epithermal Gold









BSE Image

Magnification:

50X, 100X, 200X, 300X

Position:

Kar-SF1

Minerals of interest:

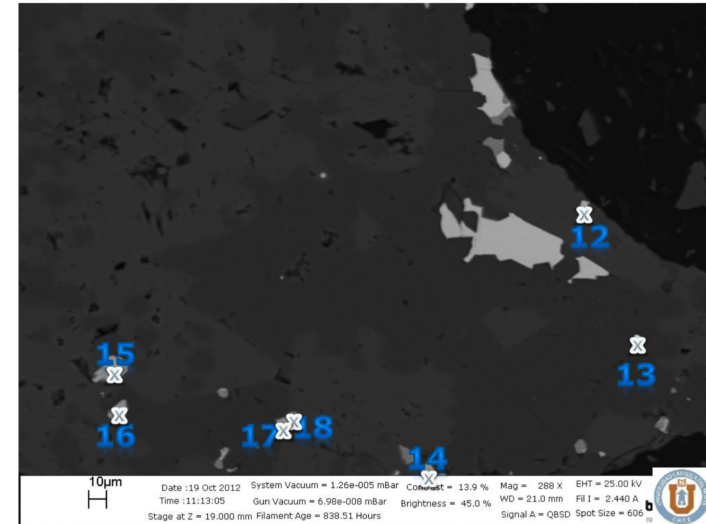
Electrum

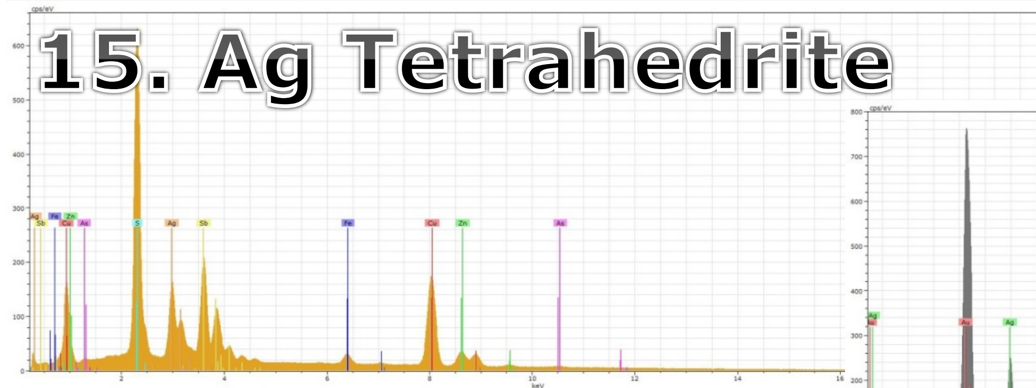
Acanthite

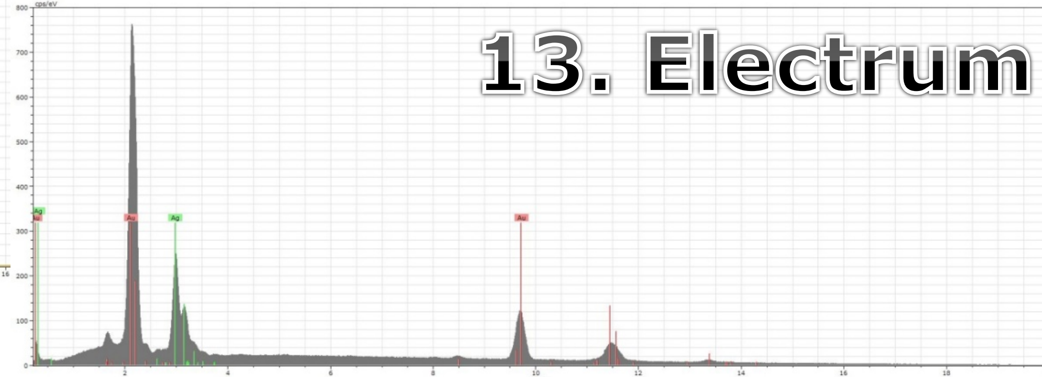
Tetrahedrite

Mining and Exploration Applications: Epithermal Gold

Mineral	Formula	Point(s)	Minor elements
Acanthite	Ag_2S	12	Sb, Se, Cu, As
Electrum	AuAg	13	
Tetrahedrite	$(Cu,Ag)_{10}(Zn,Fe)_2(As,Sb)_4S_{13}$	14, 15, 16, 18	Ag
Chalcopyrite	$CuFeS_2$	17	
Pyrite	FeS_2	4	







Metallurgical Applications:
Sphalerite – Mineral Samples

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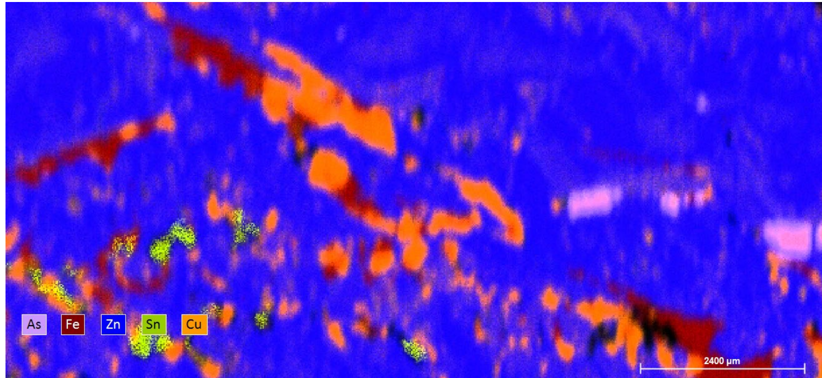
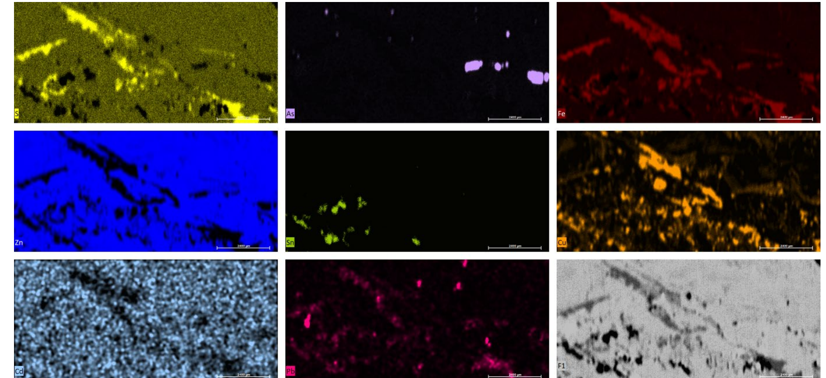
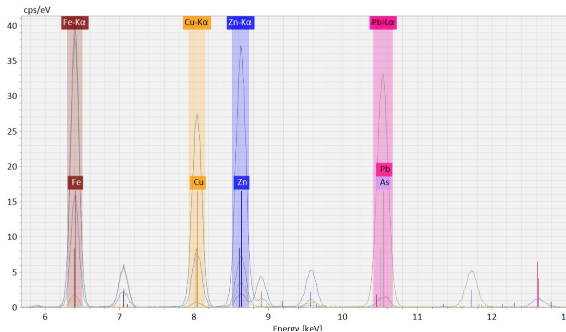
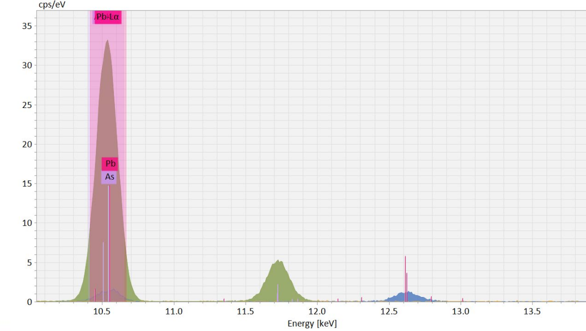
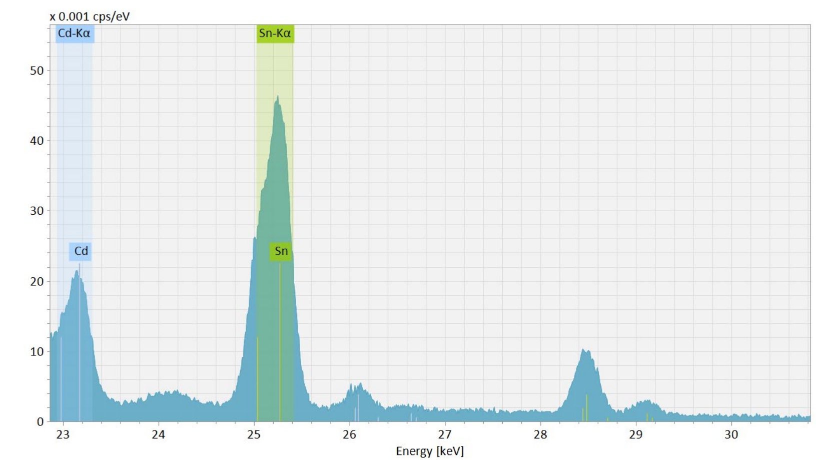
Metallurgical Applications: Sphalerite











Trace Mineral Phases

High Energy X-Ray Element Lines:

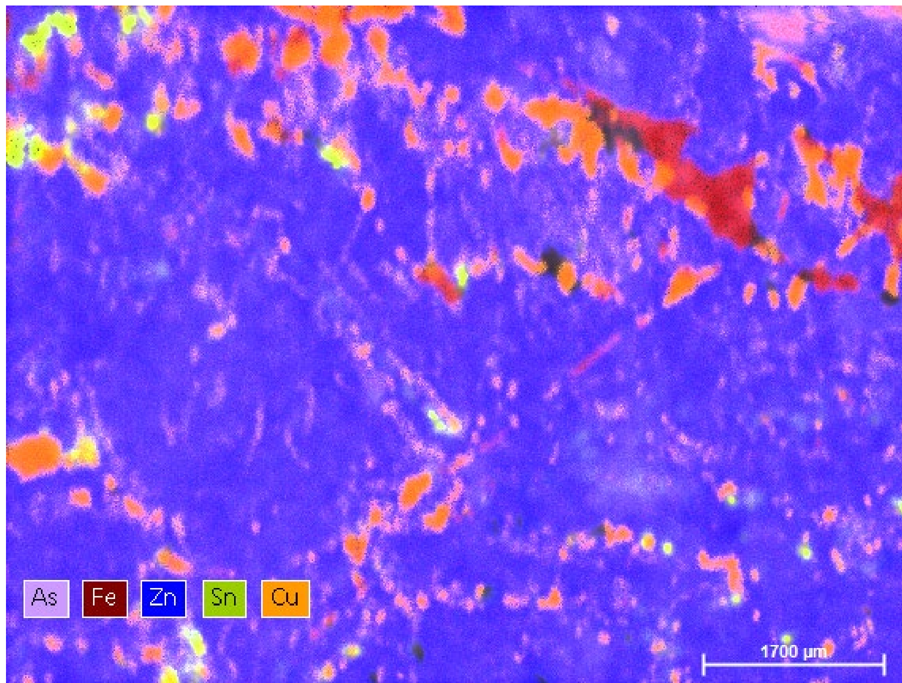
S, As, Fe, Zn, Sn, Cu, Cd, Pb

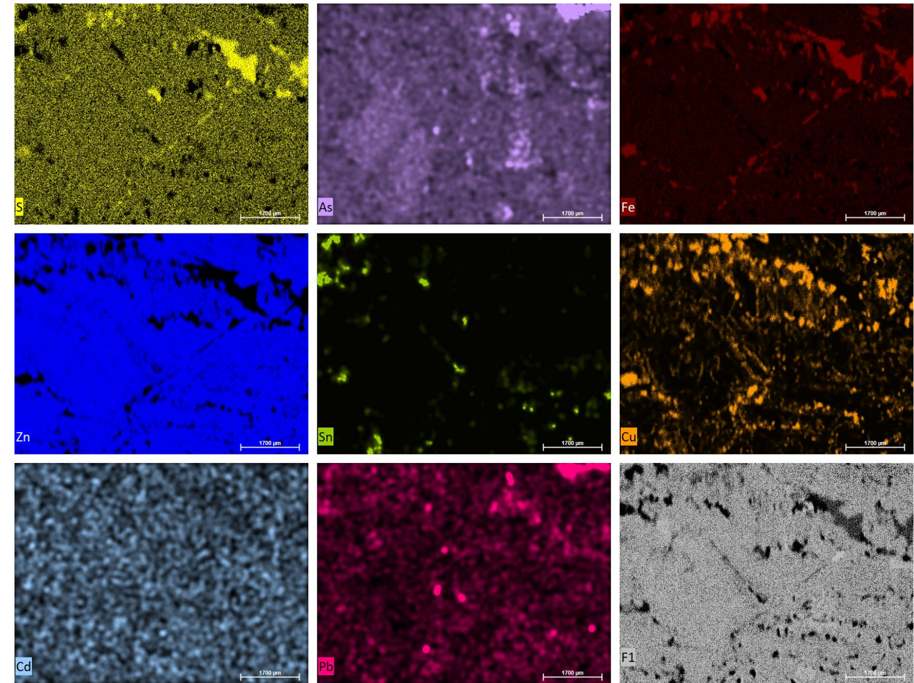
Metallurgical Applications:
Sphalerite

Trace Mineral Phases

High Energy X-Ray Element Lines:

S, As, Fe, Zn, Sn, Cu, Cd, Pb





Geological Applications:
Igneous Petrology - Mantle

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Geological Applications:
Igneous Petrology - Mantle

MAPPING

Loaded: C:\Data\Bruker - XTrace\Data\Applications - Geology\SouthAfrica-Mantle\AHM710_Speed0200_Pixel100.bcf

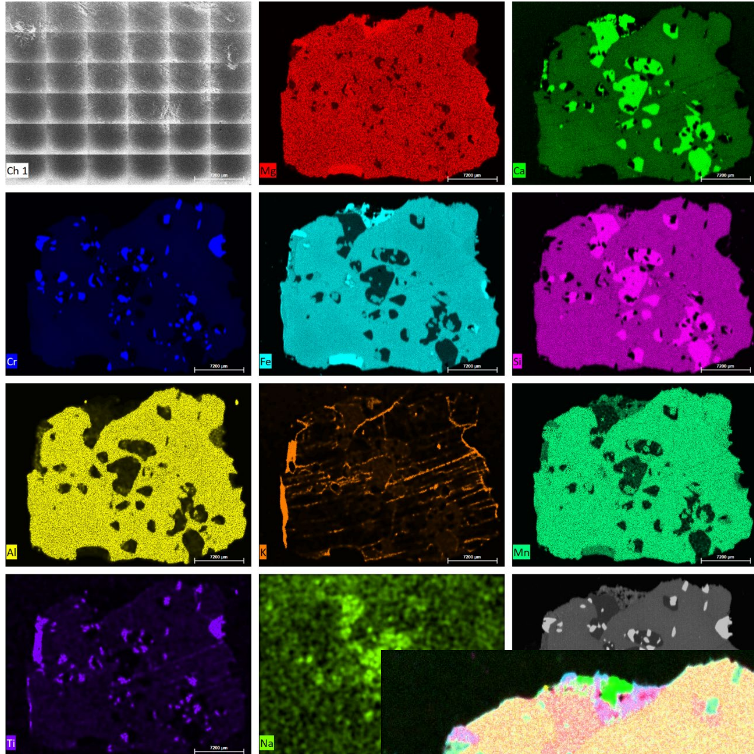
MAP INFORMATION

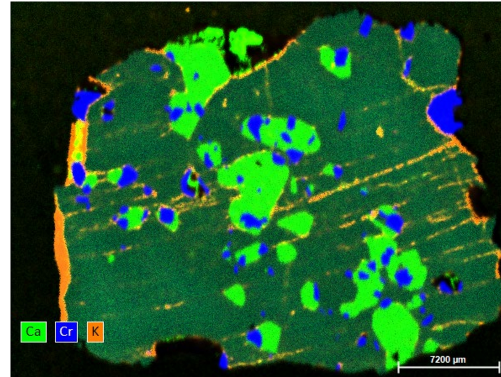
Mapping parameter		
Width:	600	pixel
	35893	µm
Height:	450	pixel
	27000	µm
Pixel size:	60	µm
Acquisition parameter		
Pixel time:	128	ms
Overall time:	577	min
Tube parameter		
High voltage:	50	kV
Anode current:	600	µA
Filter:	Empty	
Optic:	Lens	
Chamber at:	Vacuum	
Sample information		

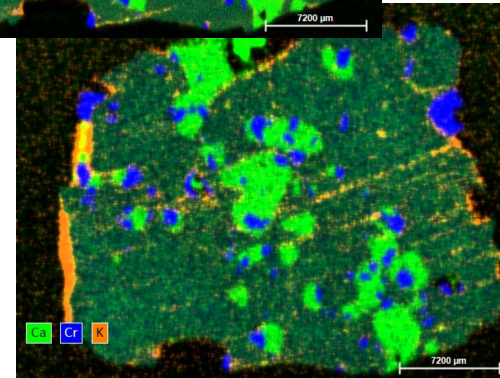
Ca Cr 7200 µm

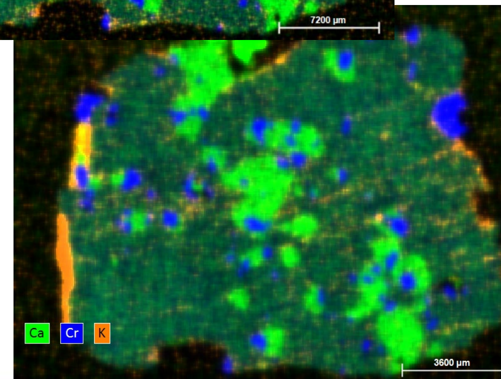
Ch 1 1.00 Mg-K 1.00 Ca-Kα 1.00 Cr-Kα 1.00 Fe-Kα 1.00 Si-K 1.00 Al-K 1.00 K-Kα 1.00 Mn-Kα 1.00 Ti-Kα 1.00 Na-Kα 1.00 F1 1.00

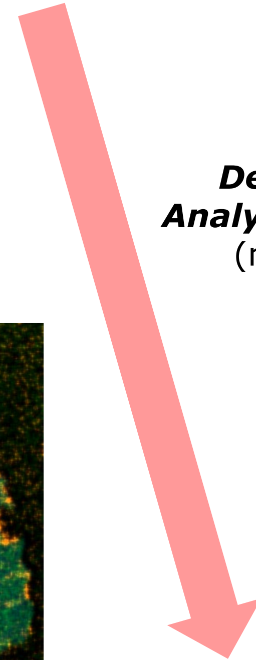
Geological Applications:
Igneous Petrology - Mantle







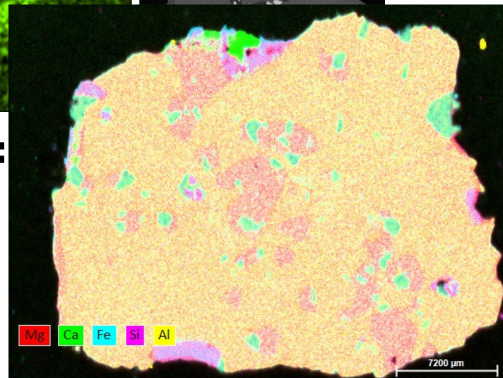




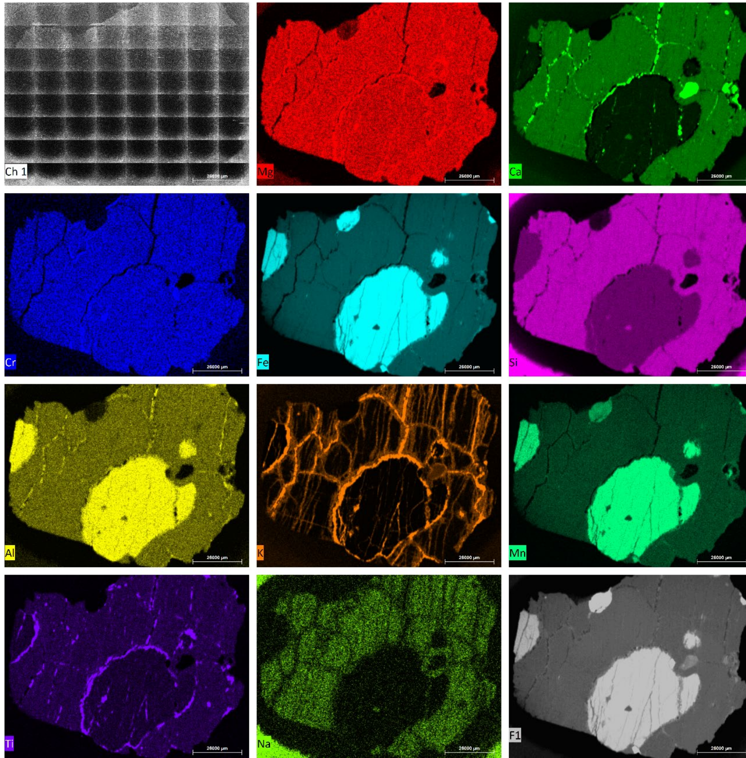
Decreased Analytical Time :
(minutes)
577
23
11

Mantle Peridotite:

Garnet
Chromite
Clinopyroxene
Olivine



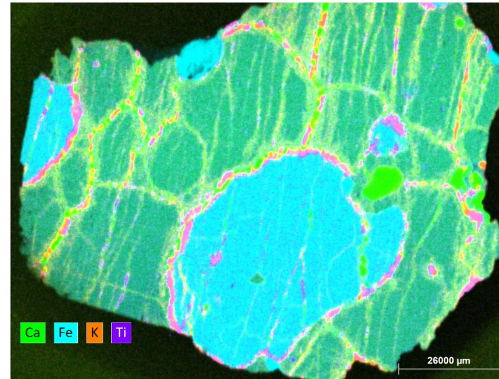
Geological Applications:
Igneous Petrology - Mantle

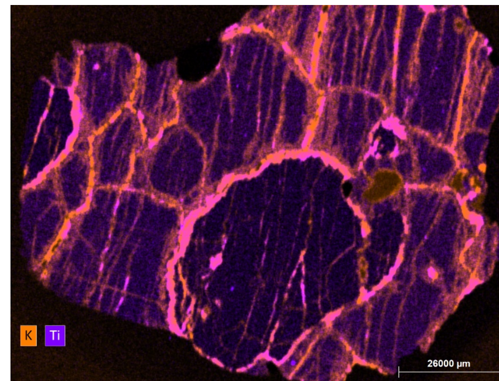


Mantle Eclogite:

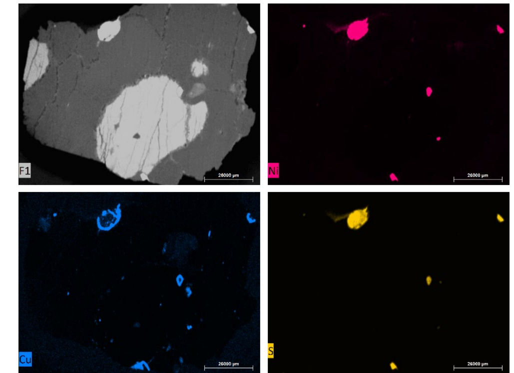
Clinopyroxene

Garnet





Metasomatic Interaction



Trace Phases

Industrial Applications:
Steel

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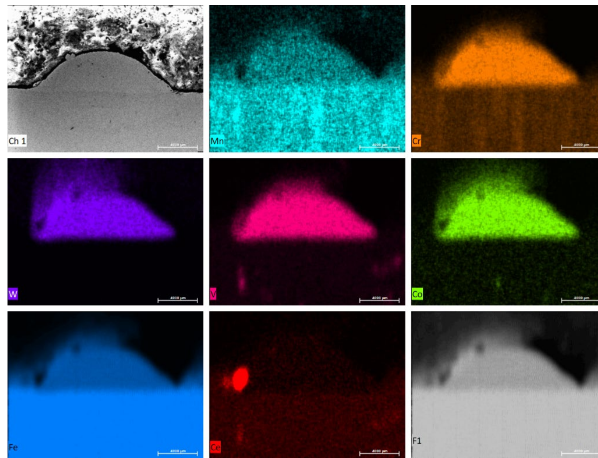
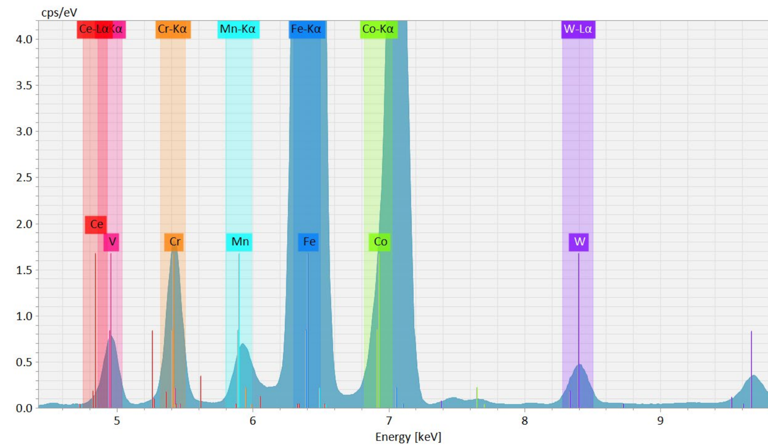
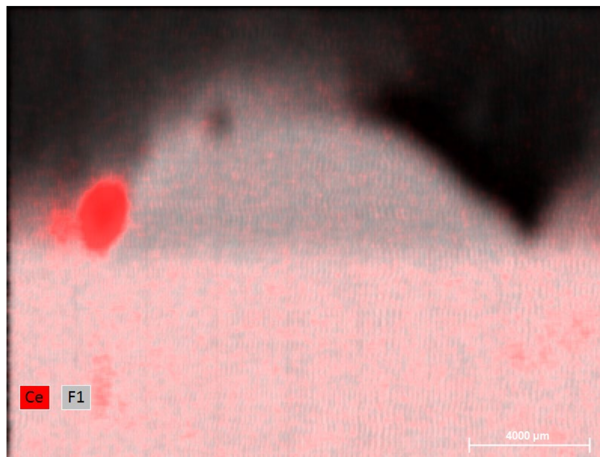
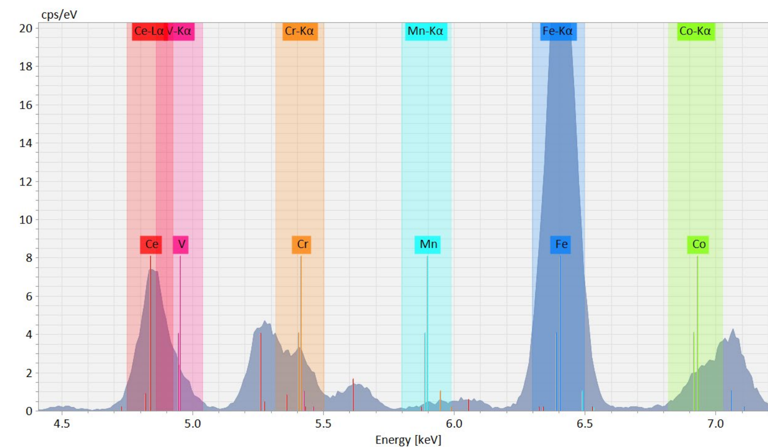
Industrial Applications:
Steel









Different Steel Types and impurities and reaction surfaces

Archaeological Applications:
Ancient Ceramic Fragments

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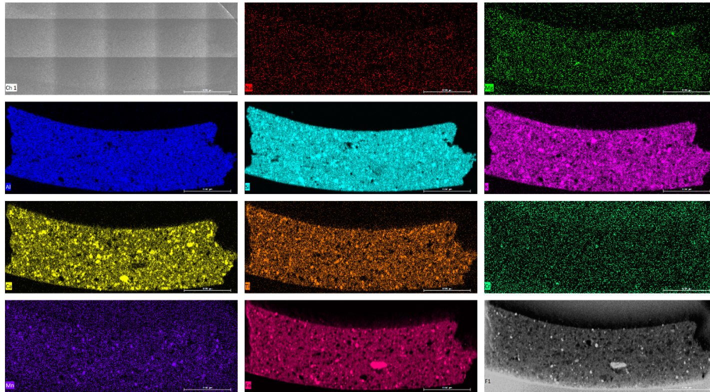
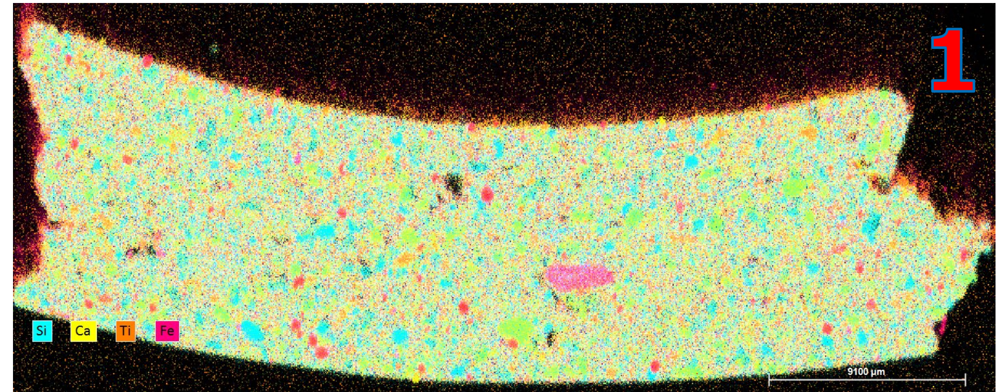
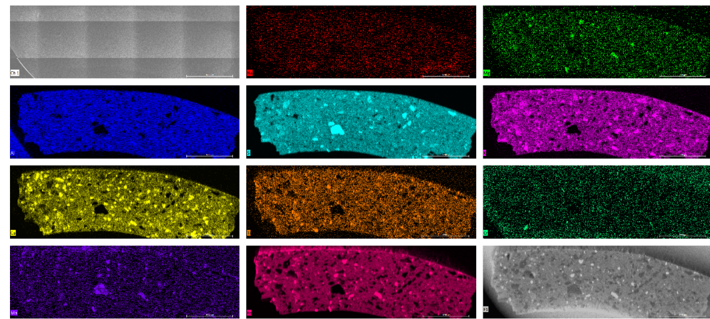
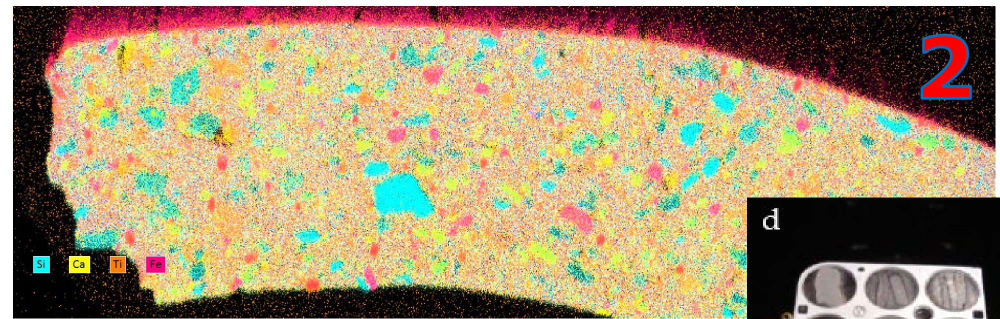
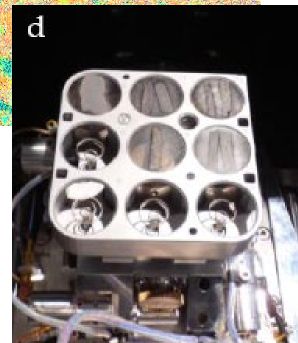
Analysis of Ceramics:
Large Area Maps











Top Row: SEM, Na, Mg; Second Row: Al, Si, K;

Third Row: Ca, Ti, Cr; Bottom Row: Mn, Fe, F1

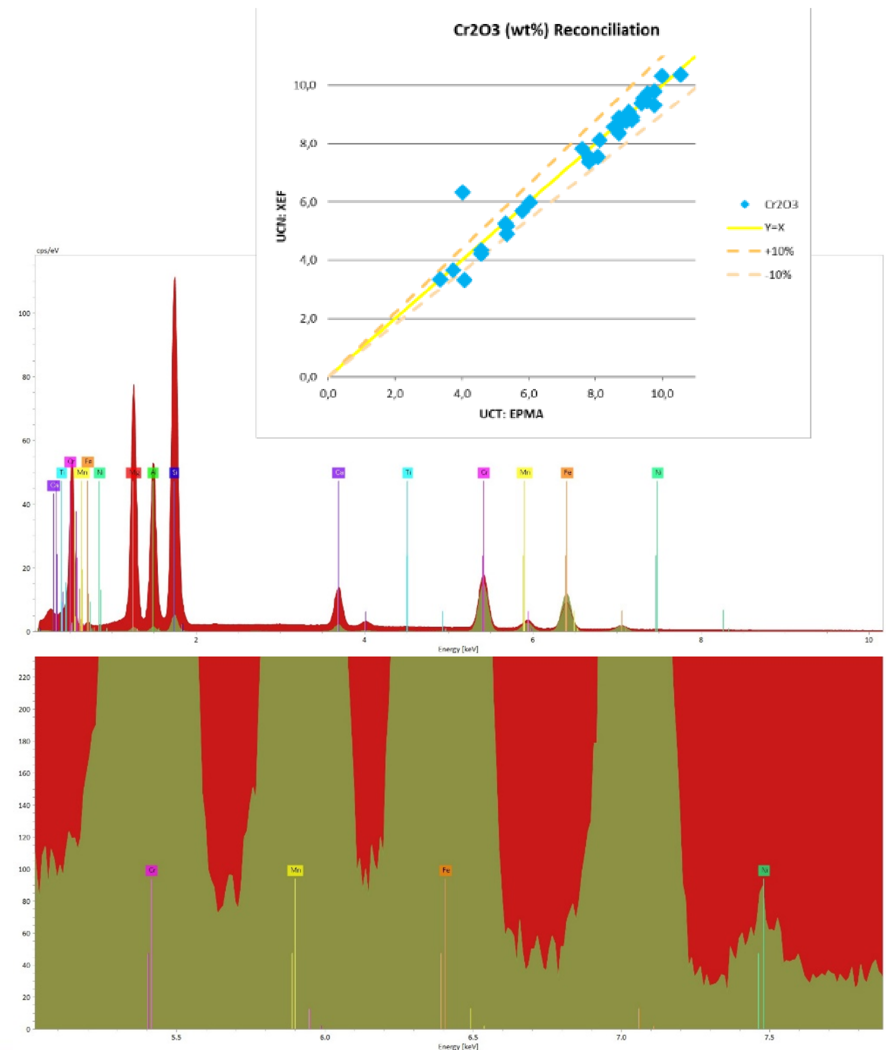
Ceramic Samples from Northern Chile

Identify different mineral phases – probable different source material

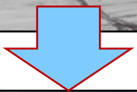
Quantification:
Point Analysis – Garnet

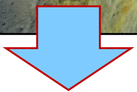
Major and trace elements

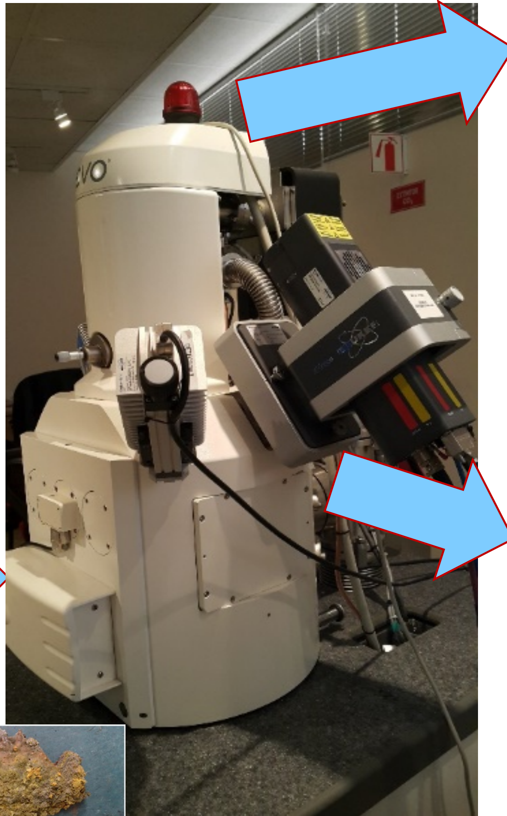
Element	Unit	90 sec	120 sec	180 sec
SiO2	(%)	39.04	39.17	39.20
TiO2	(%)	0.28	0.28	0.29
Al2O3	(%)	22.23	21.97	21.87
Cr2O3	(%)	0.11	0.11	0.11
FeO	(%)	21.16	21.05	21.02
MnO	(%)	0.49	0.48	0.48
MgO	(%)	12.29	12.57	12.63
CaO	(%)	4.35	4.31	4.33
Ni	(ppm)	26	18	28
Cu	(ppm)	3	5	4
Zn	(ppm)	173	143	150
Ga	(ppm)	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

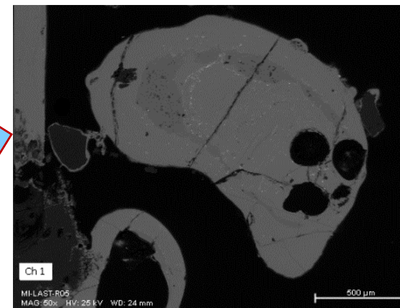


Workflow
SEM-XRF (XTrace)

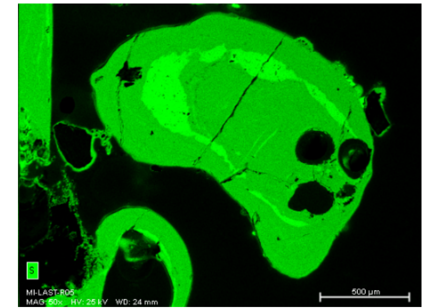




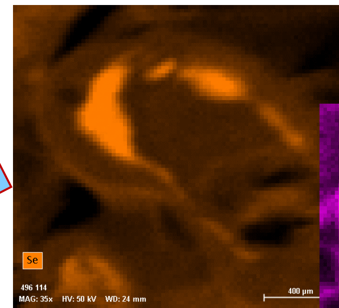


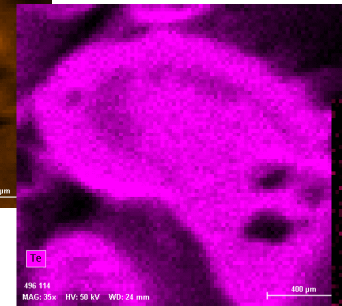


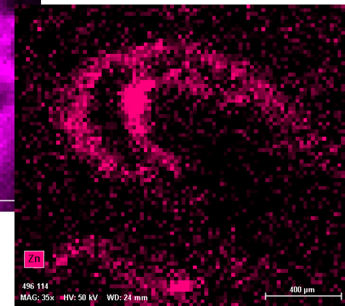
SEM-BSE Image



SEM-EDS Major Element Map: Sulphur (S)







SEM-XRF Trace Element Maps:
Selenium (Se), Tellurium (Te), and Zinc (Zn)

Summary and Conclusions:
SEM-XRF (XTrace)

- The analysis of samples in mircometer (μm) scale on a standard SEM.
- Able to perform large area maps on a variety of samples.
- Sample Preparation Minimal:
  - No carbon-coating
  - No polishing
  - Directly into the SEM
- Able to detect and resolve minor and trace elements at levels better than a (e-beam) SEM-EDS.
- Identification of high energy X-Ray lines that can not be identified by SEM-EDS.
- Can work in combination with SEM e-beam
  - Commonly Low-KV due to charging and sample interaction

Acknowledgements

John Gurney

Jeff Mauk

Marina Vargas

Monserrat Barraza

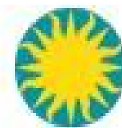
Manuel Inostroza

Felipe Aguilera

Sebastian Sola

Edward P.Vicenzi

Thomas Lam



Questions and Answers

BRUKER

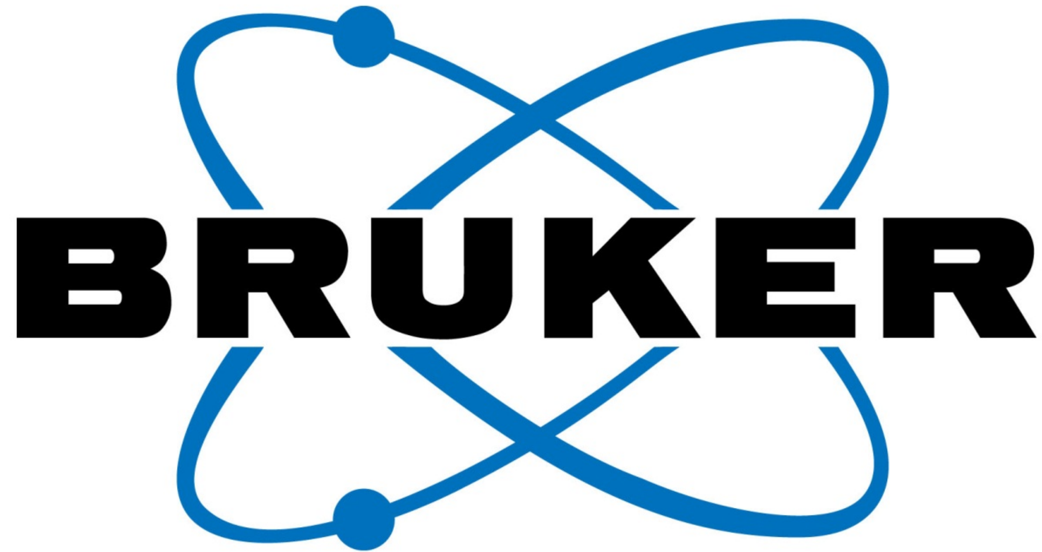
Are There Any Questions?

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

More Information

For more information, please contact us:

info.bna@bruker.com



Innovation with Integrity

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