Multi-scale in-situ non-destructive micro-XRF scanning analysis:

Implications for ore mineralogy, petrogenesis and micro-metallurgical assessments

Bruker Nano Analytics, Berlin, Germany

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Raič, S.
Molnar, F.
Michaux, S.

Botha, P.
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Sardisco, L.
Jones, S.

Lundström, M.

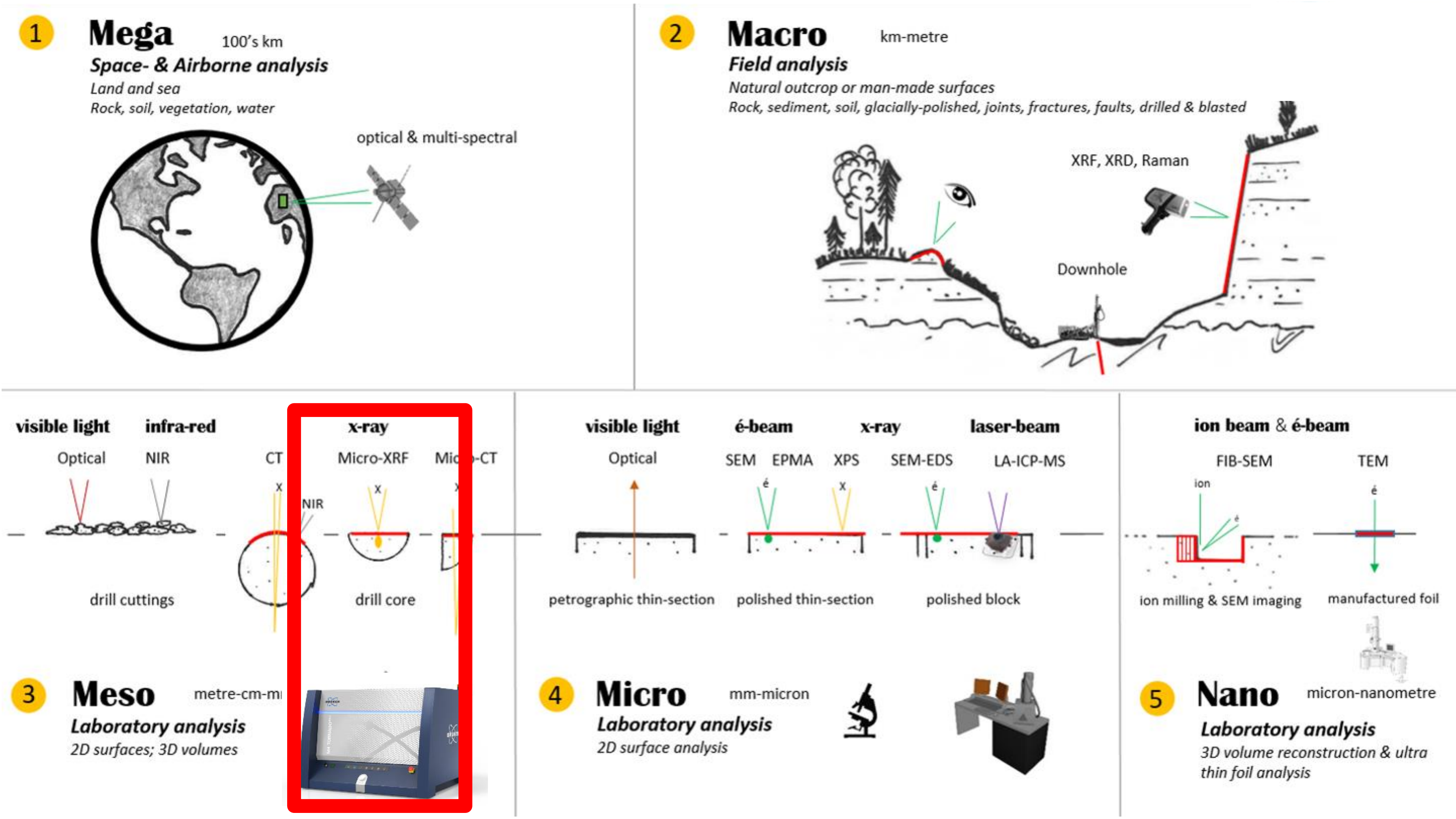
Multi-scale in-situ non-destructive micro-XRF scanning analysis:
Implications for ore mineralogy, petrogenesis and micro-metallurgical assessments

- The exploration process and metallurgical understanding occurs on scales that vary by numerous orders of magnitude.

- An important link in this chain is the transition from samples collected in the field to analysis in the laboratory.

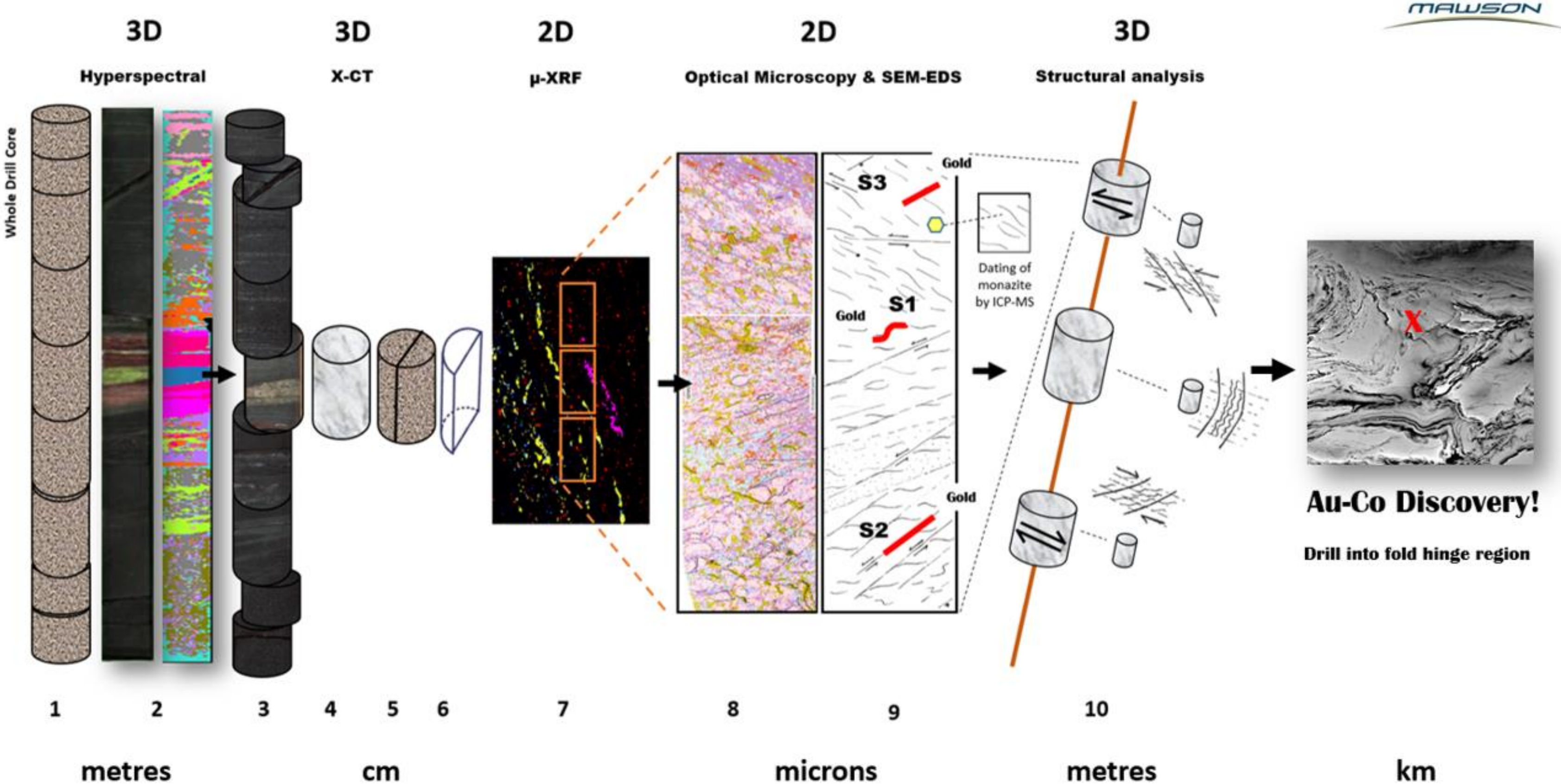
- Detailed characterisation of samples that employs geo-analytical techniques to achieve multi-scale, multi-modal, and multi-dimensional information (involving the integration of 2D, 3D and 4D imaging and analysis of rock samples).

Multi-scale Characterisation Workflow



Butcher AR (2020) Upscaling of 2D mineralogical information to 3D volumes for geoscience applications using a multi-scale, multi-modal and multi-dimensional approach. ***EMAS 2019****, Conference Proceedings Volume, Trondheim*, 19-23 May 2019.

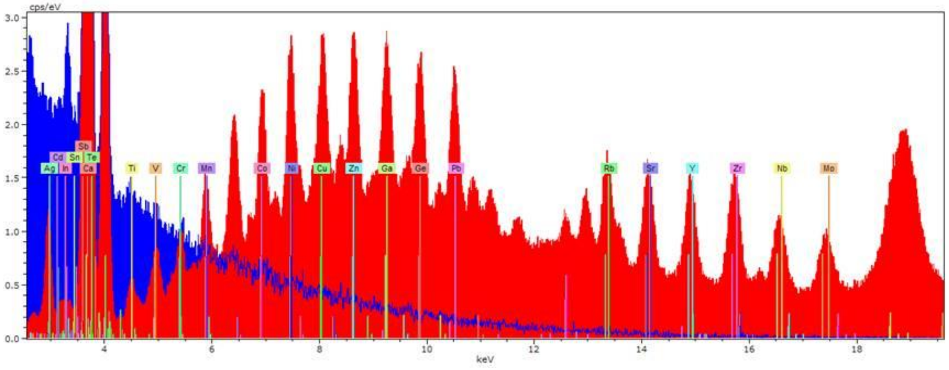
New Characterization Workflow



Butcher AR (2020) Upscaling of 2D mineralogical information to 3D volumes for geoscience applications using a multi-scale, multi-modal and multi-dimensional approach. ***EMAS 2019***, *Conference Proceedings Volume, Trondheim*, 19-23 May 2019.

Micro-XRF analysis:
Introduction

- **Little to no sample preparation**
- **Non-destructive**
- **Elemental information**
- **Small spot analysis**
- **Information from within the sample**
- **Meso-scale samples : Micro-scale information**
- **Quantification**
- **Mineralogy**

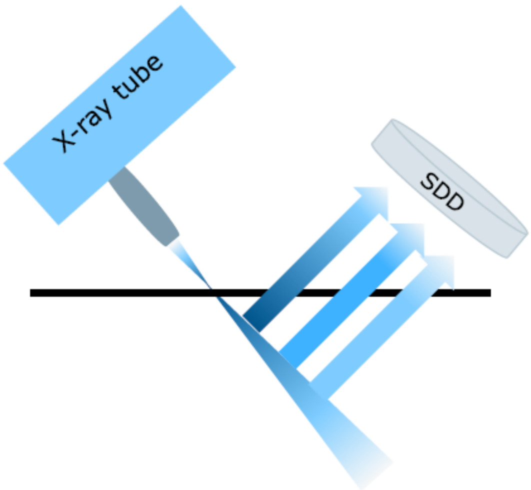


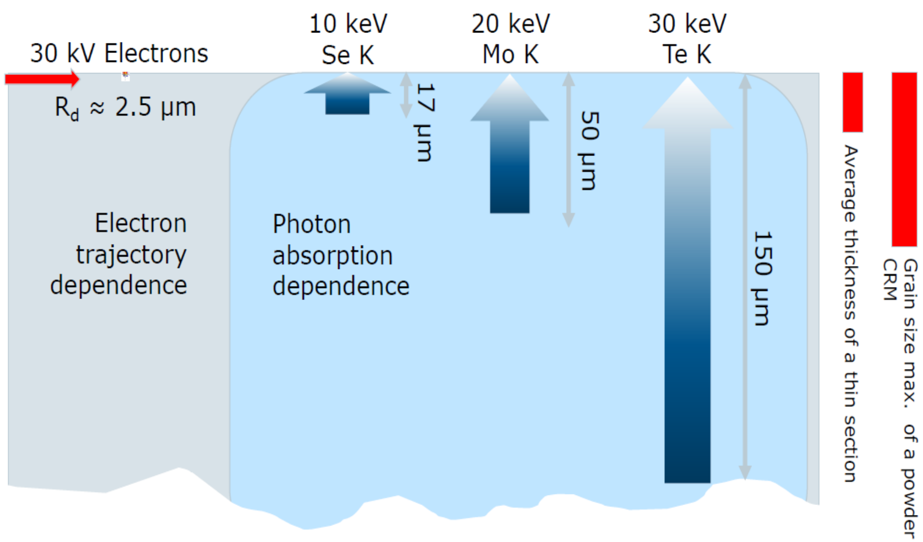
Spatial Resolution and Analyzed Volume:
Transmission and Attenuation

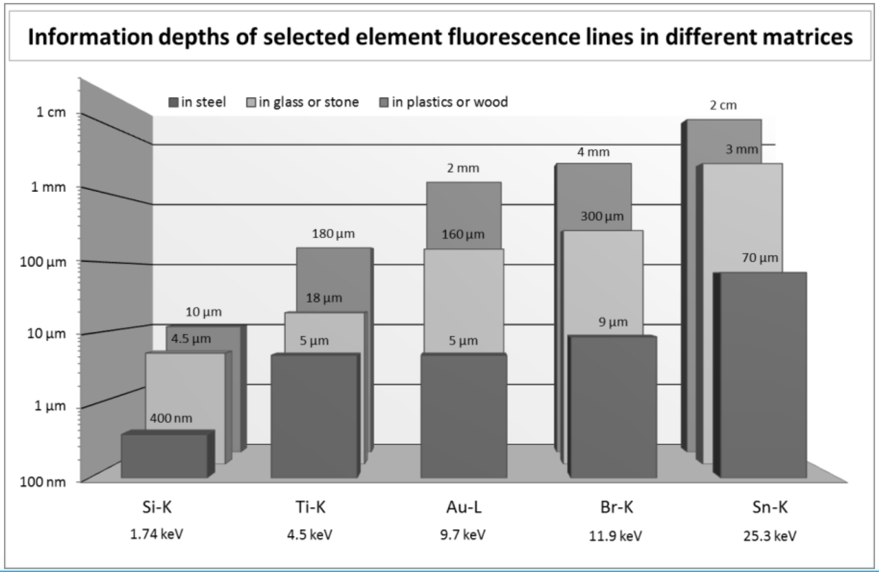
The transmission of X-rays is important for excitation of samples as well as for the fluorescence radiation.

Penetration depth: the depth that can still be excited

Information depth: the depth from which fluorescence X-rays can still reach the detector



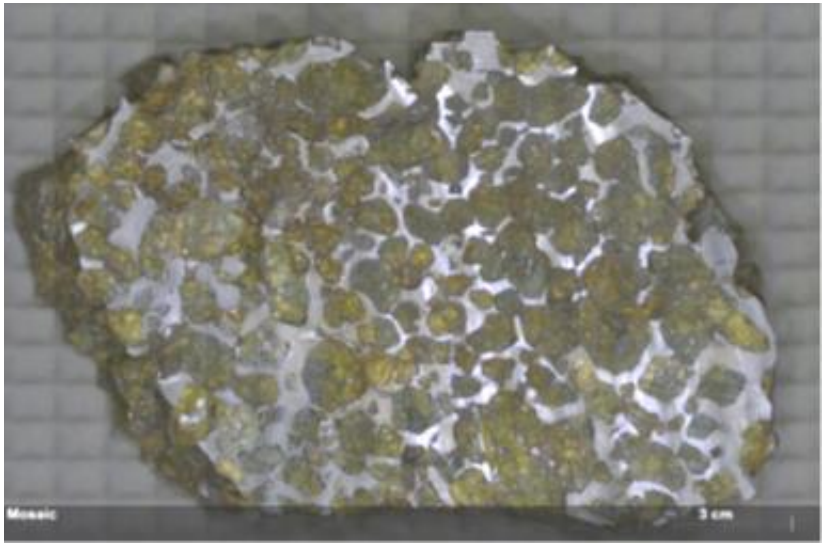


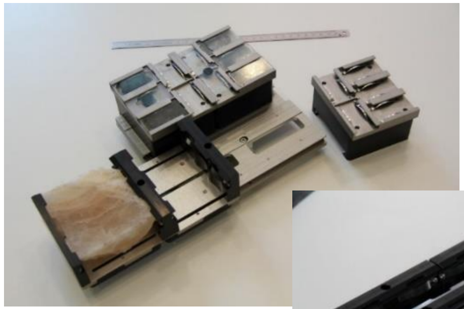


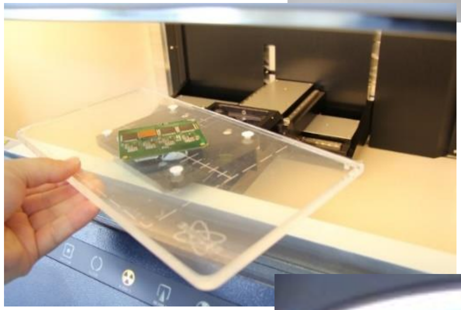
Micro-XRF: Comparison
Analytical Parameters and Conditions

Parameter	EDS: E-beam (SEM-EDS)	WDS: E-beam (SEM-WDS)	EDS: Micro-XRF
Analyzed Volume	Ø: few µm Information depth: µm; (depending primarily on electron energy)	Ø: few µm Information depth: µm; (depending primarily on electron energy)	Ø: 15-30 µm Information depth: µm to mm; (depending on analysed element and matrix)
Detectable Elements	Atomic number Z ≥ 4 (beryllium)	Atomic number Z ≥ 4 (beryllium)	Atomic number Z ≥ 6 (carbon)
Energy range	K- L –M – Lines (up to 20 keV)	70 eV – 3.6 keV (L- M- Lines)	K- L –M – Lines (up to 40 keV)
Concentration Range	Down to 1000 ppm	Down to 100 ppm	Down to 10 ppm
Quantification	Standard less and Standard based	Standard based	Standard less and standard based
Data collection	Simultaneously	Sequentially	Simultaneously
Sample Preparation	Sample needs to be electrically conductive (commonly carbon-coated), polishing required	Sample needs to be electrically conductive (commonly carbon-coated), polishing required	Electrical Conductivity not required, samples don´t need to be polished
Sample Stress	Heating due to absorbed electrons	Heating due to absorbed electrons	Minimal
Typical SEM beam current	Variable	Variable > 10 nA	N/A

Micro-XRF analysis:
Sample Types

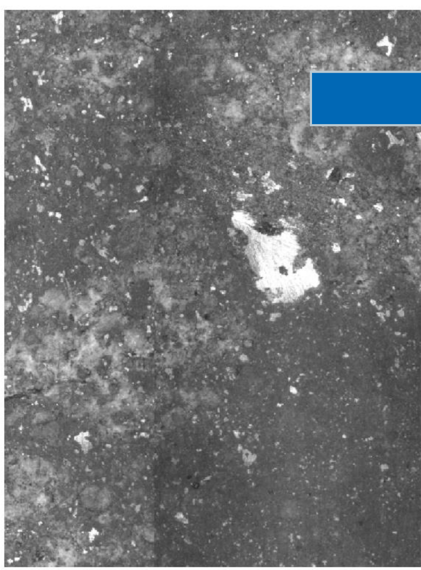




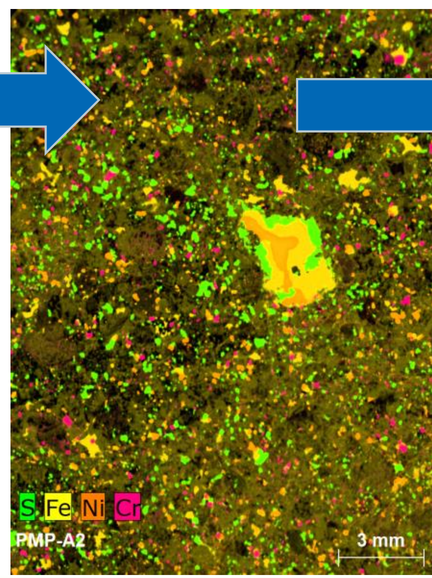


Micro-XRF analysis:
Hyperspectral Datasets

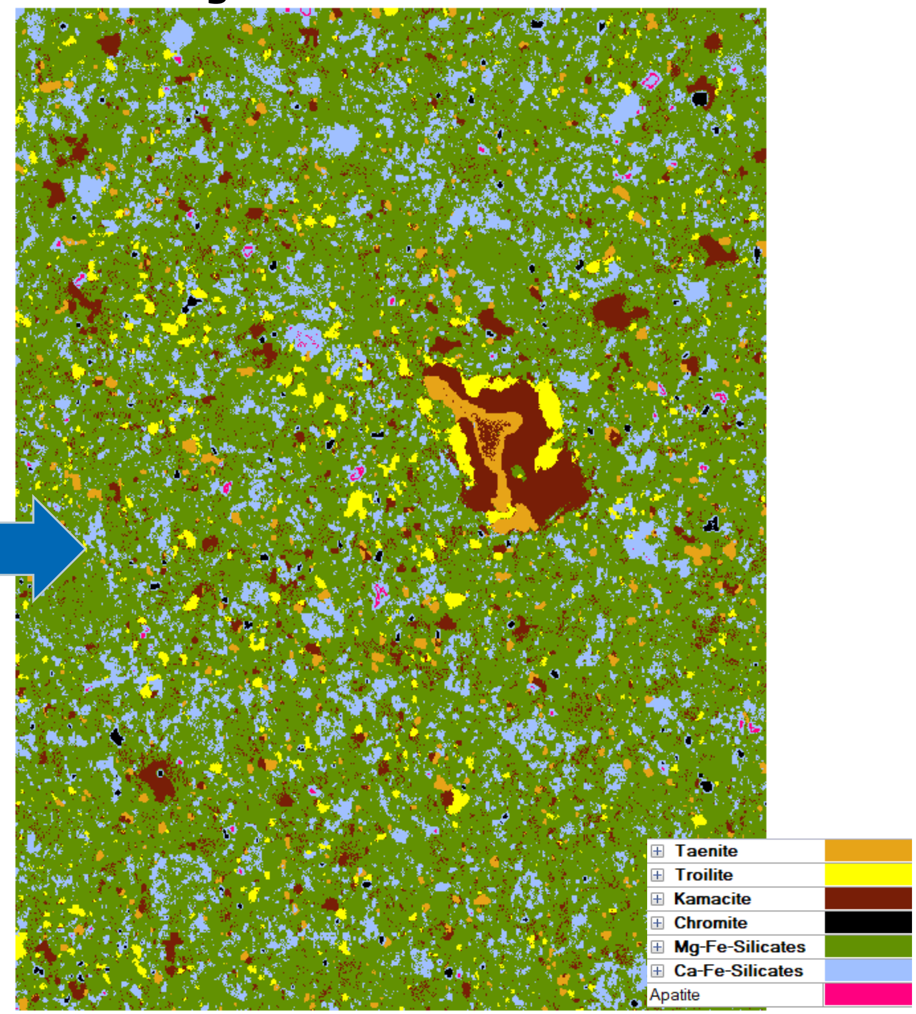
Optical



Elemental



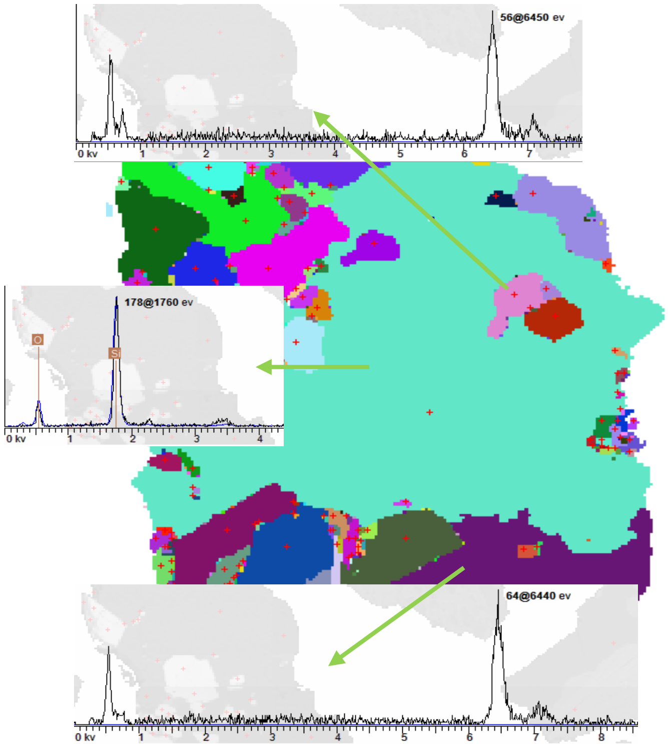
Mineralogical



AMICS: Automated Mineralogy

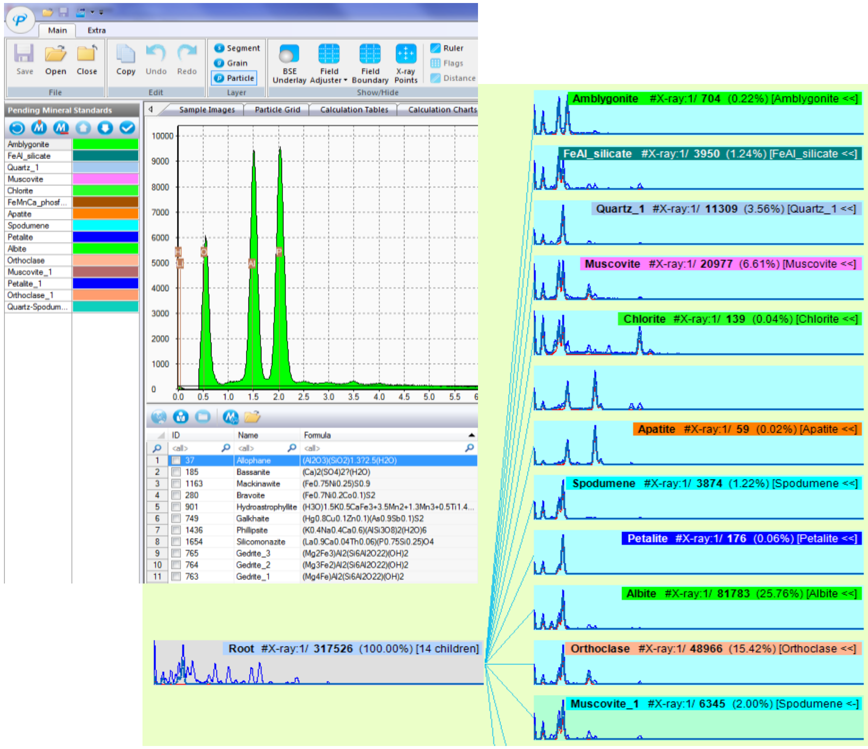
How does AMICS work?

X-ray Analysis



- Acquires spectrum for each segment

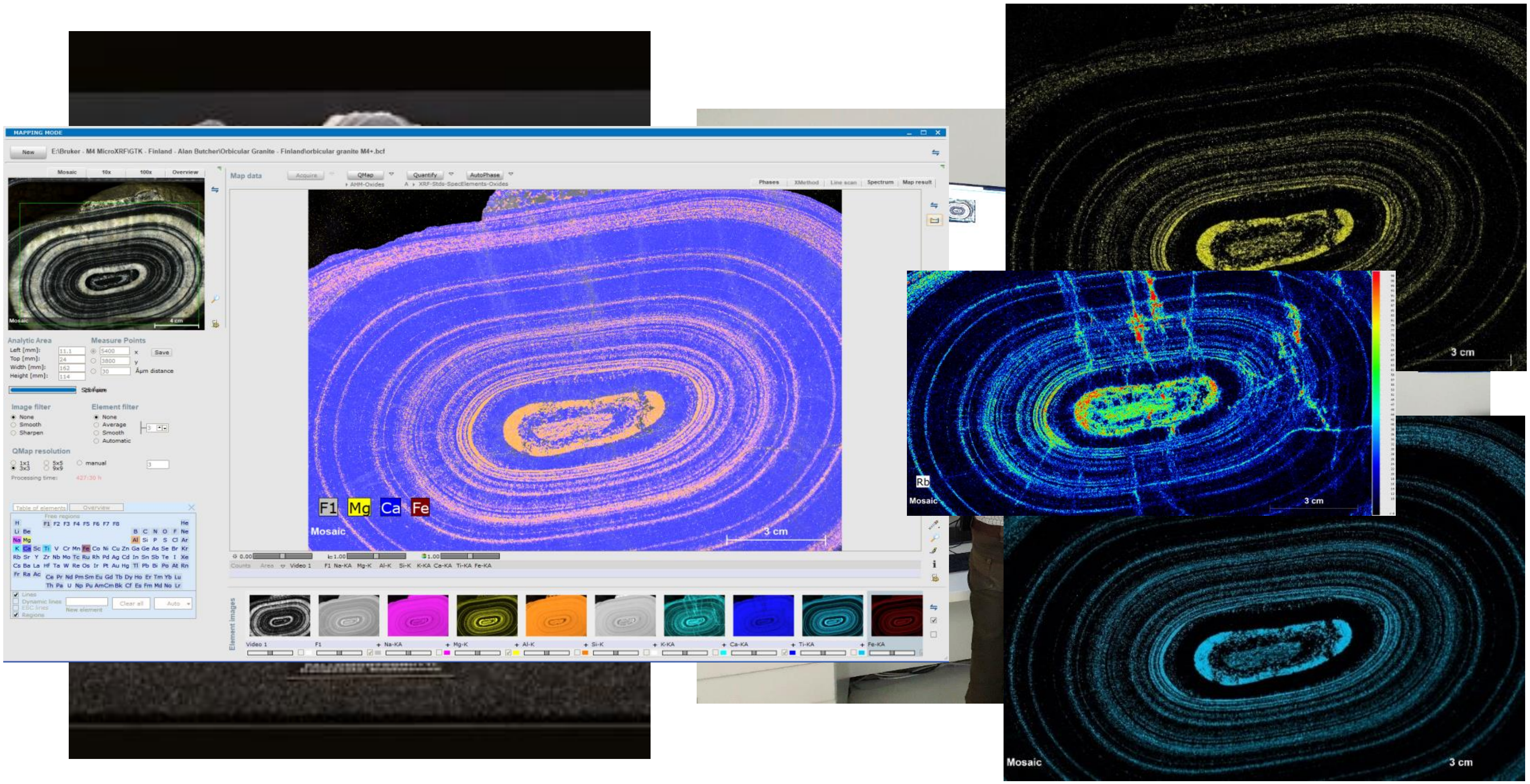
Classification



- Identifies minerals based on spectral information

Finland – Kuohenmaa

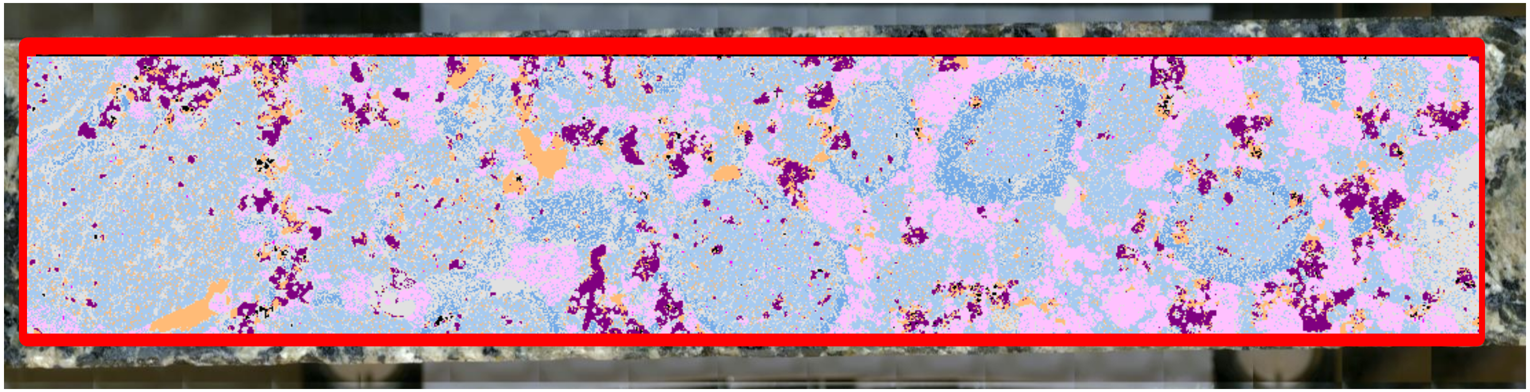
Orbicular Monzonite: Rock Sample 5 kg

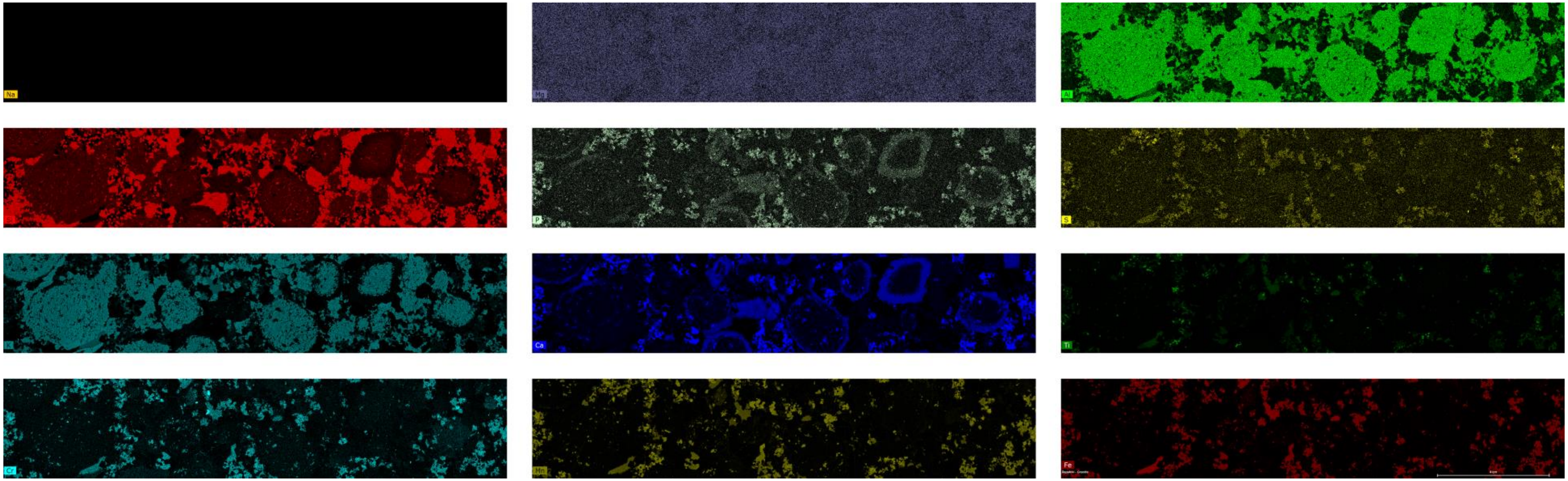


Kuohenmaa Quartz Monzonite Boulder

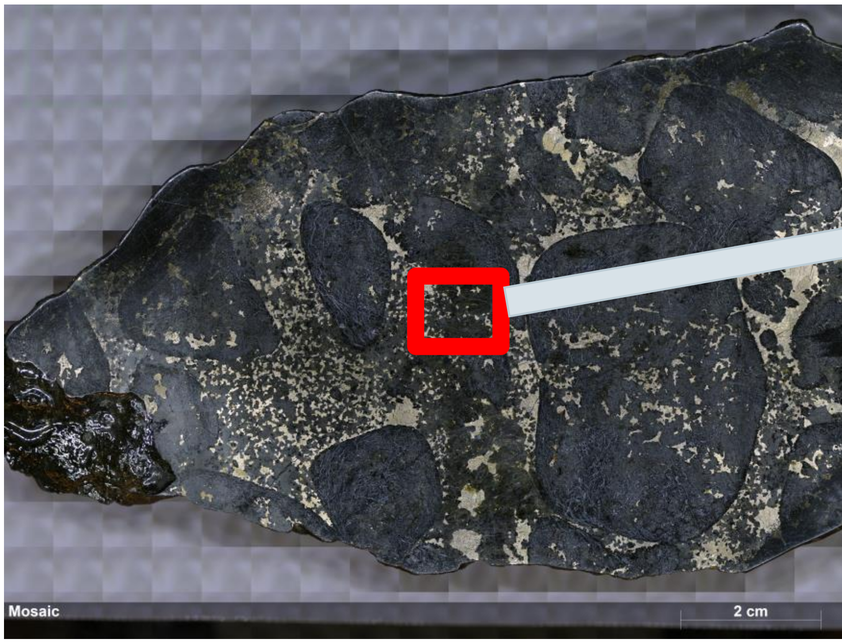
Sample courtesy of Aku Heinonen

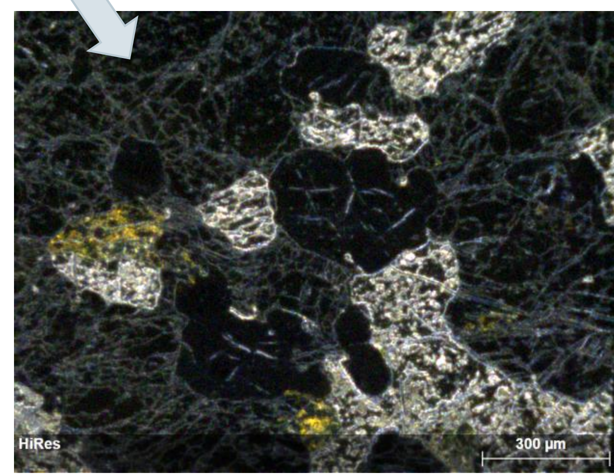
Finland – Rapakivi
Granite: Drill Core – 20 cm in Length





Finland – Kylmäkoski
Rock Sample: 3 kg and 20 cm



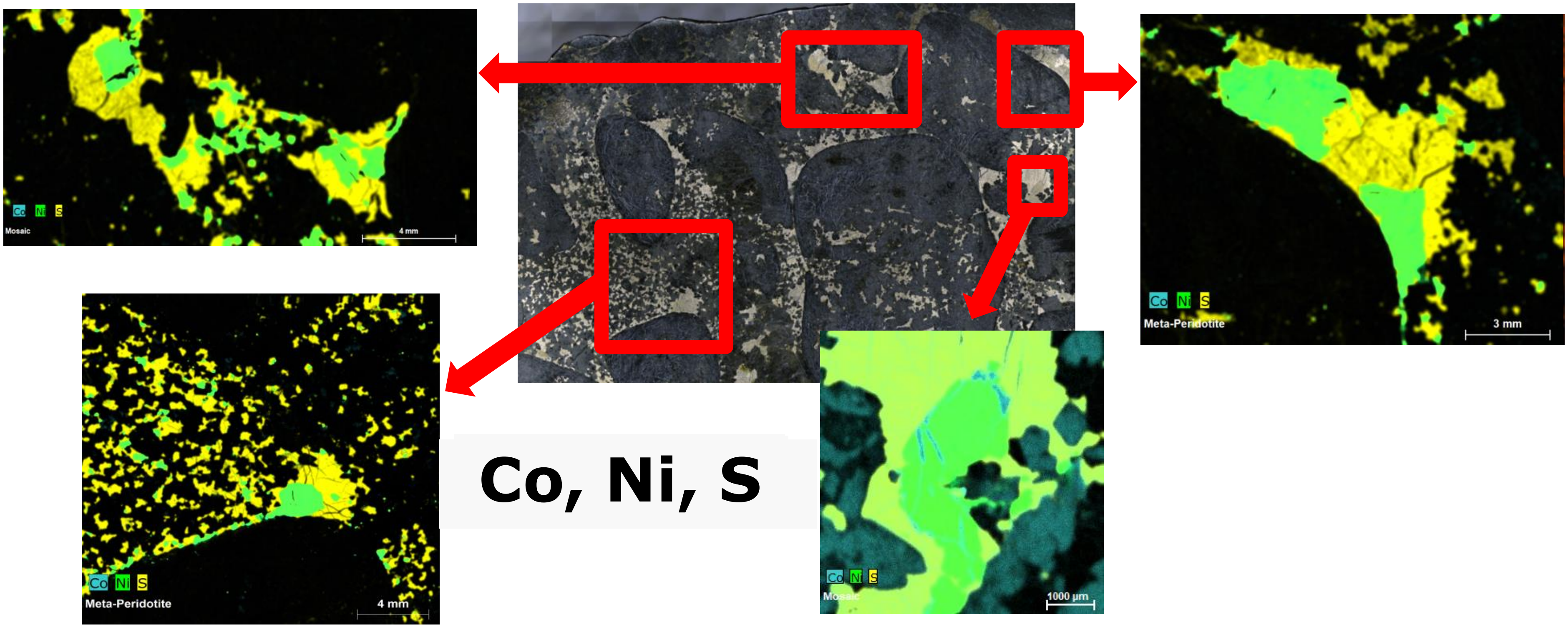


Orbicular Peridotite

Kylmäkoski nickel-copper deposit

Optical Images

Finland – Kylmäkoski
Elemental Mapping



Orbicular Peridotite

Kylmäkoski nickel-copper deposit

Elemental Images

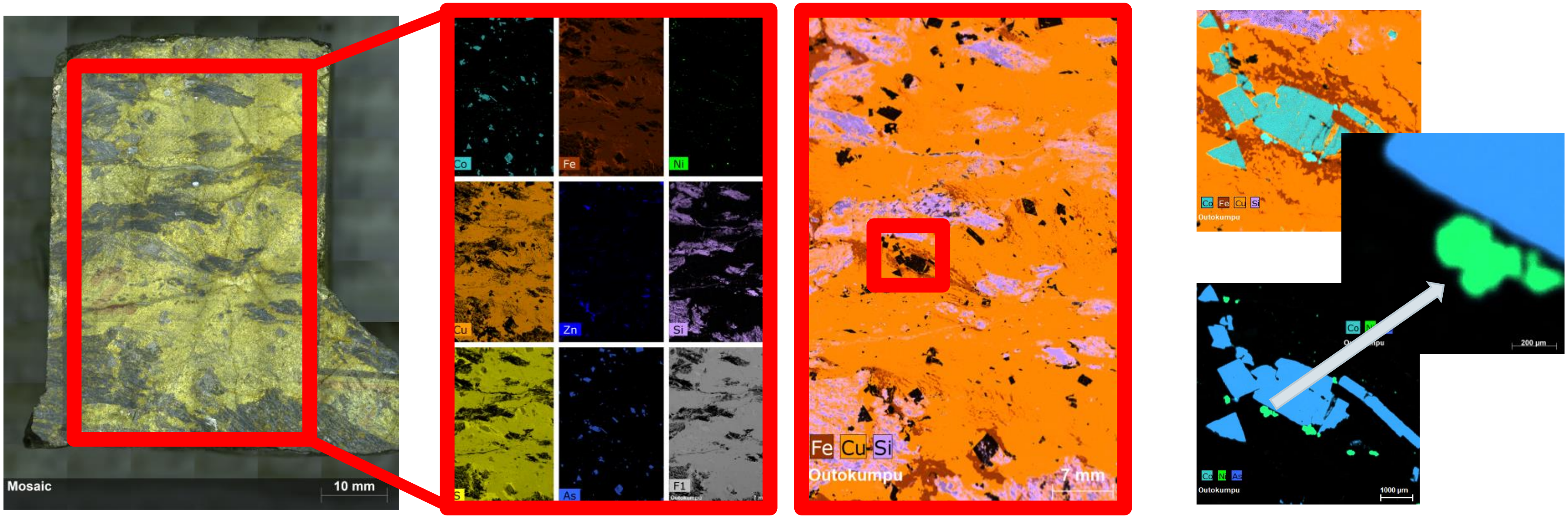
Finland - Outokumpu
Rock Sample: 5 micron mapping

Map Entire Sample

Interpretation

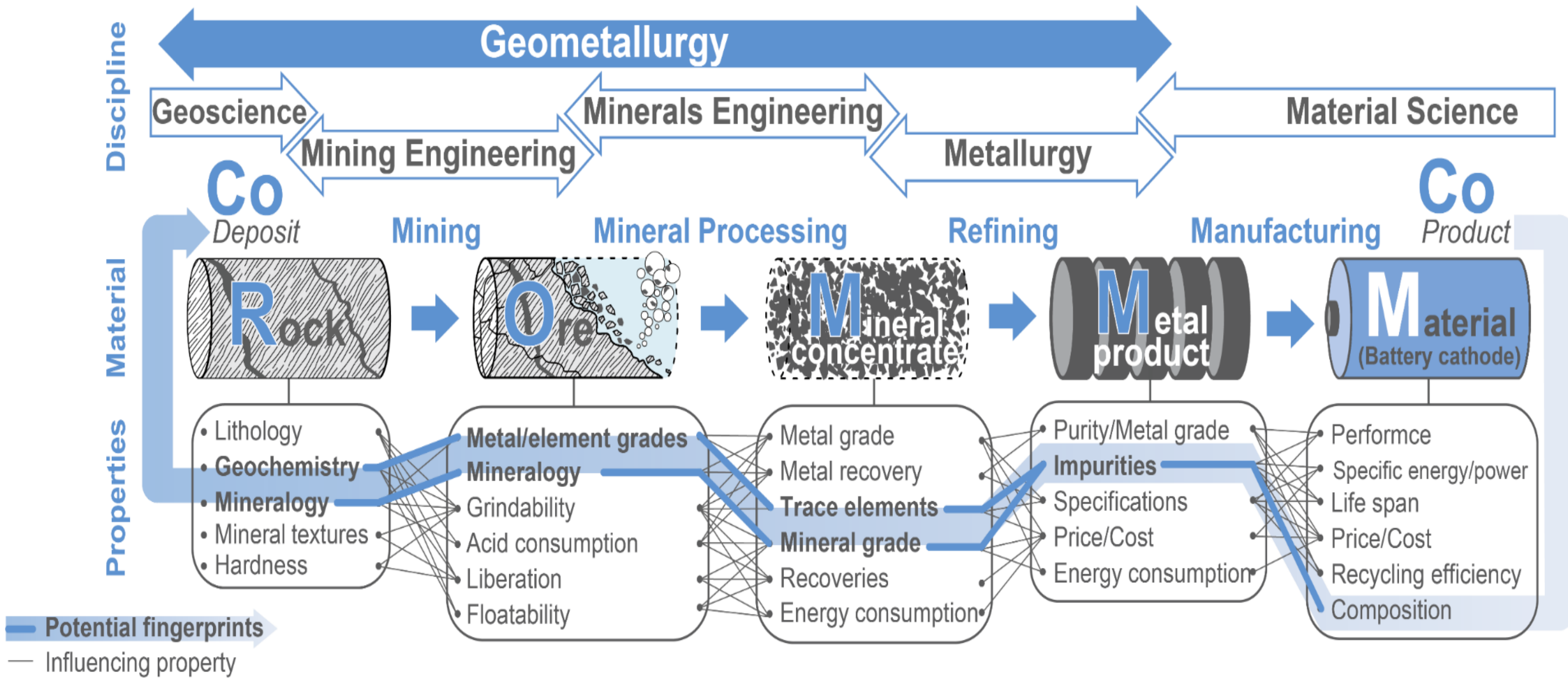
Mineralogy and
Element Distribution

Select Areas of
Interest for detailed
mapping



Geometallurgy:
Traceability Workflow

Integrated approach to improve value chain and responsible sourcing



Q. Dehaine, S. Michaux, J. Pokki, M. Kivinen and A.R. Butcher, *Battery minerals from Finland: Improving the supply chain for the EU battery industry using a geometallurgical approach*, European Geologist 49 (*In Press*).

Multi-scale in-situ non-destructive micro-XRF scanning analysis:
Implications for ore mineralogy, petrogenesis and micro-metallurgical assessments

- This includes characterization of:
  - Mineralogy and Distribution of Element(s) of Interest(s) and associated metals/elements,
  - Key mineralogical/geometallurgical ore properties,
  - Mineral associations
  - Micro-structural relationships

- The end result is an enhanced perspective on the commercial process mineralogy, incorporating details about liberation of both ore and gangue minerals at the early stages of any given project.

Finland – Raja Prospect, Rajapalot Project
Exploration Project: Au-Co (Mawson Oy)

Finland – Raja Prospect
Context and Objectives

Finland – Raja Prospect, Rajapalot Project
Exploration Project: Au-Co (Mawson Oy)

Context

The work forms part of the **BATCircle Project** currently underway at GTK, with Mawson Oy as a Partner, as well as other **geometallurgical research** at **GTK** in collaboration with Mawson Resources.

Objectives, Scientific and Analytical Question(s)

- Identify Co distribution
- Identify Co mineralogy
- Determine Co mineral associations
- Determine Co mineralogy size distribution

Finland – Raja Prospect
Context and Objectives

Minerals of economic interest at Rajapalot Project

Precious Metal Minerals

Gold

Native gold – electrum

Battery Minerals

Cobalt

Cobaltite: **CoAsS**

Linnaeite: **($Co^{+2}Co^{+3}{}_2S_4$)**

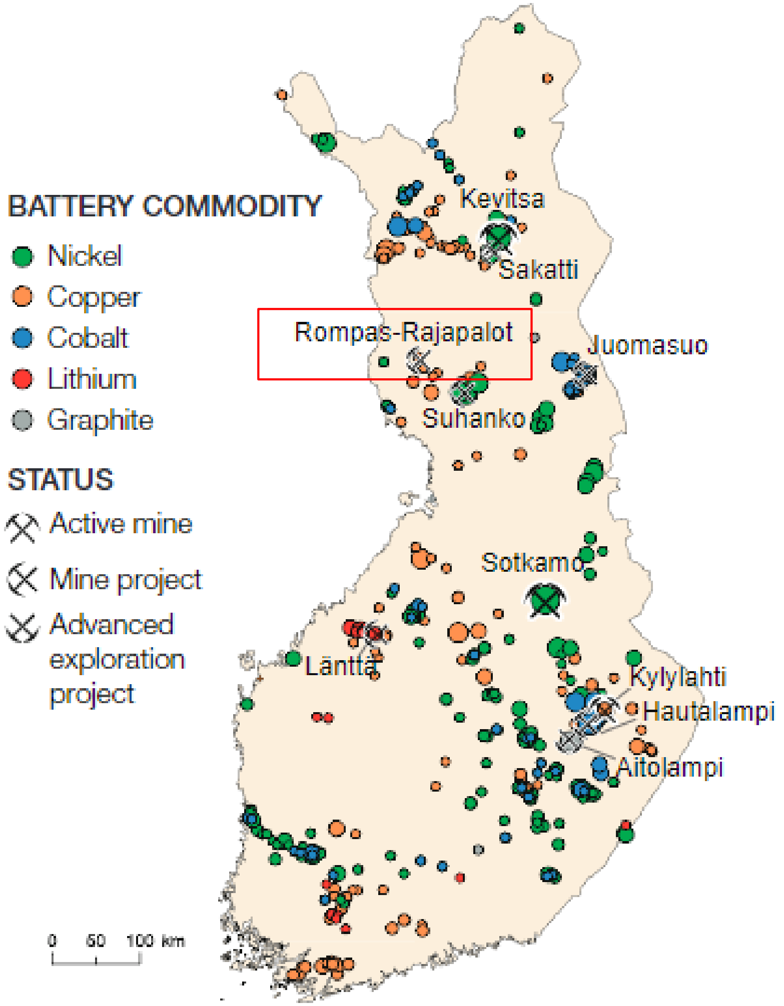
Cobaltian pentlandite: **($(Co, Ni, Fe)_9S_8$)**

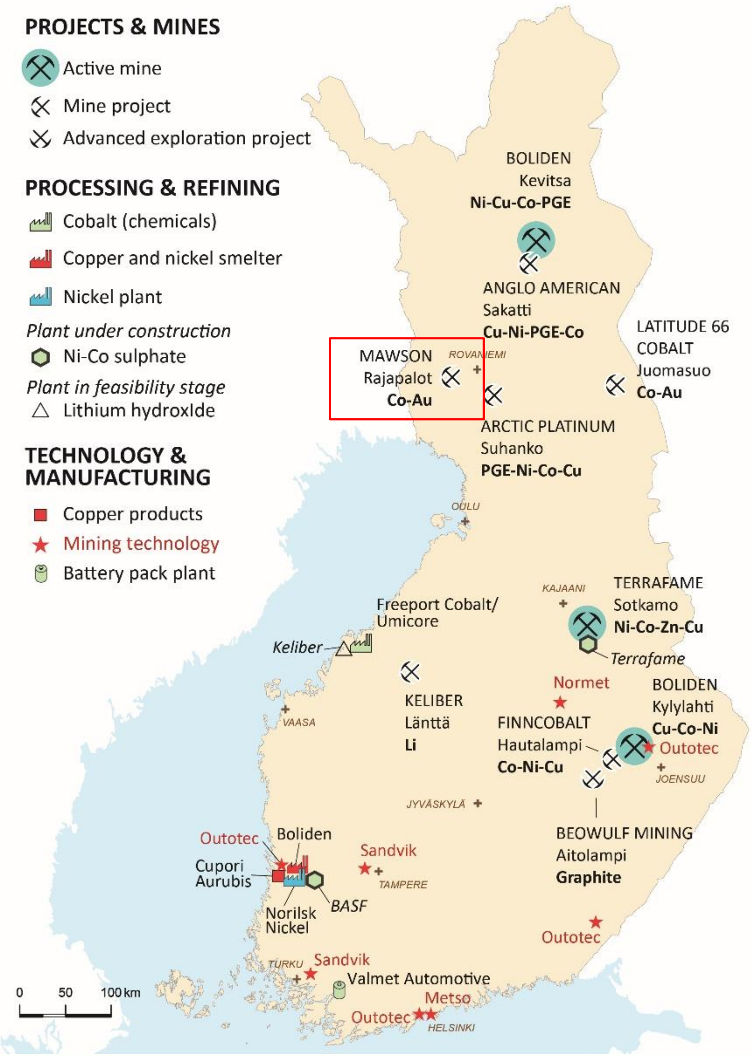
Co-Pyrite
Co-Pyrrhotite

Copper

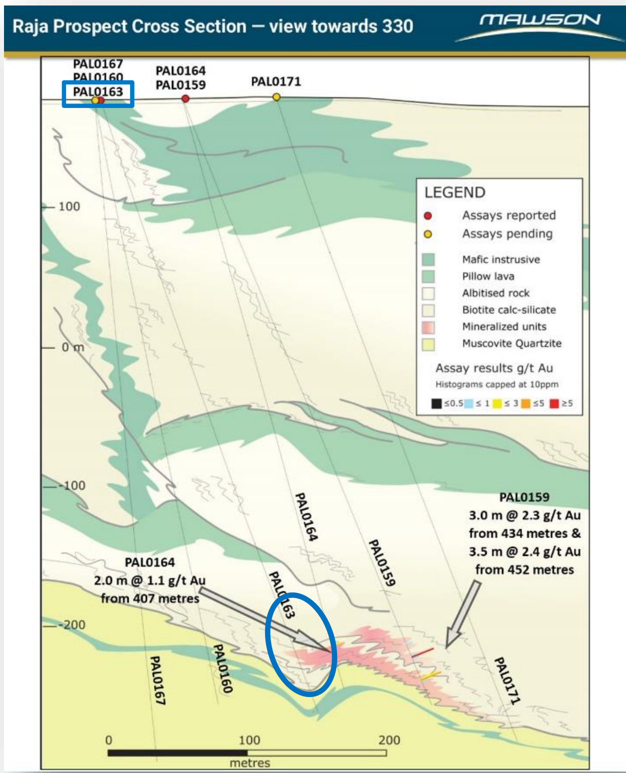
Chalcopyrite

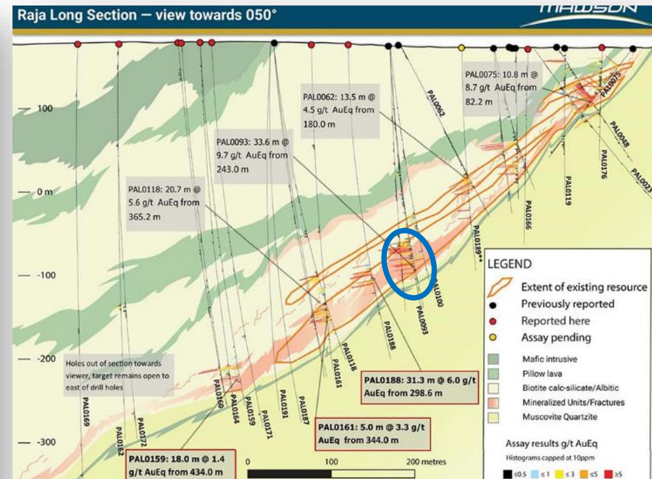
Finland – Rompas-Rajapalot Project
Sample Location and Geology

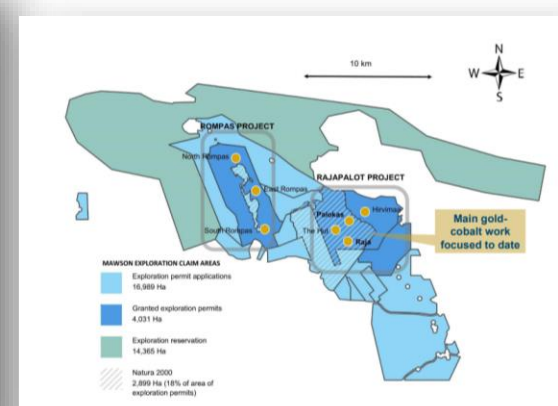




Finland – Rajapalot Project
Sample Location and Geology



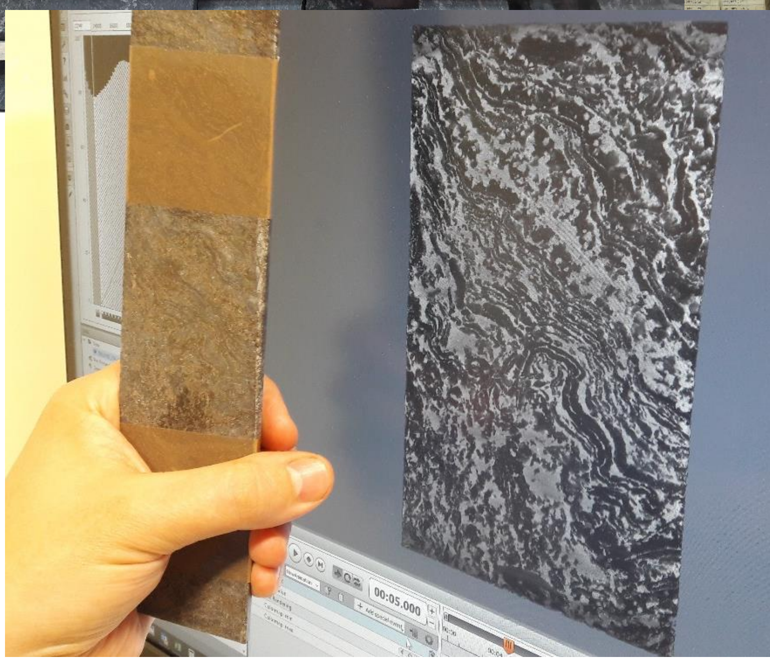


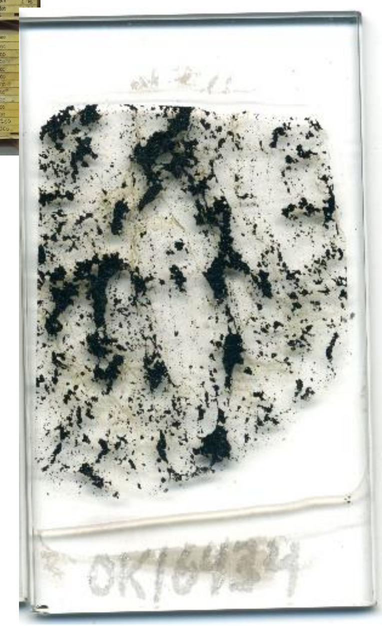


Location of **Raja Prospect**, within the **Rajapalot Project**, and position of drill hole **PAL0163** relative to gold-bearing units, with region of interest marked by a ring.

Source: *Mawson Resources*
http://mawsonresources.com/

Finland – Raja Prospect
Sample Description





Half cores
X-CT & **Micro-XRF**

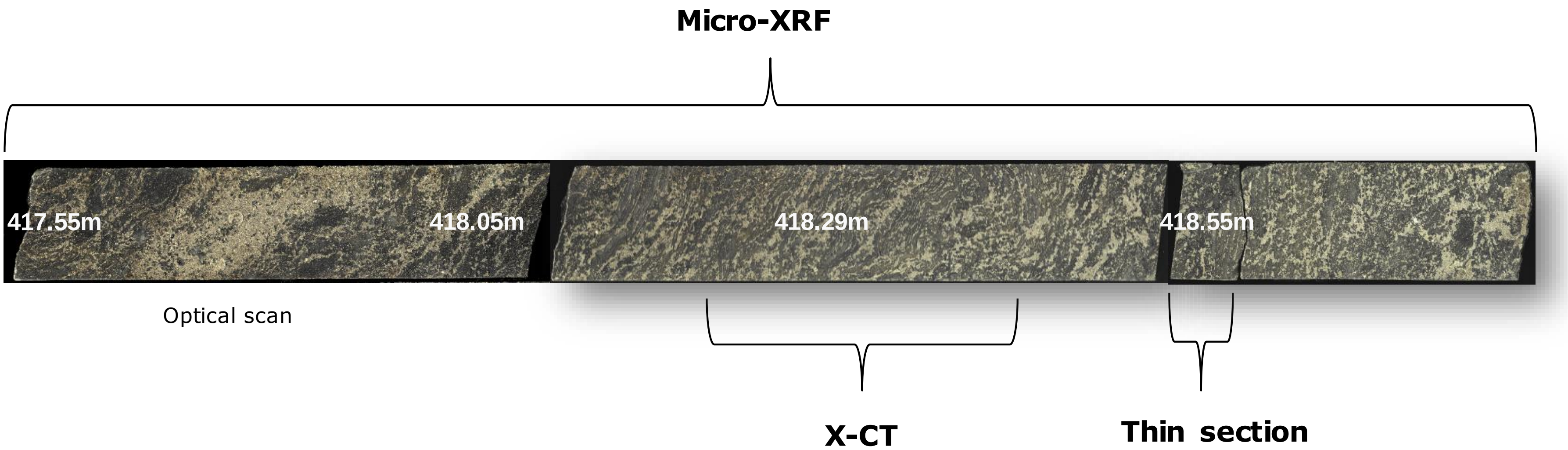
Thin sections
Automated Mineralogy & **Micro-XRF**
Off-cuts by QXRD

Finland – Raja Prospect

Drill Core: High cobalt intersection

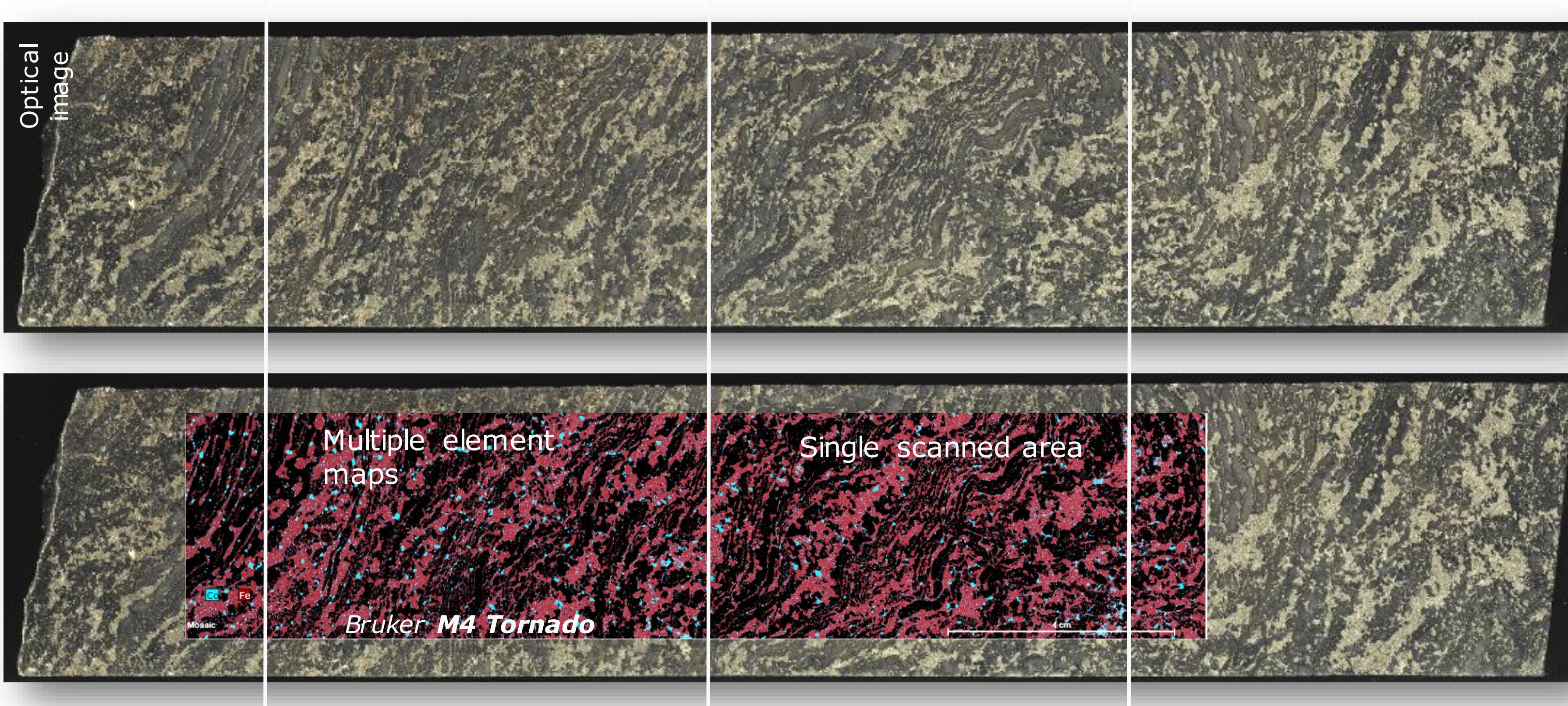
PAL0163

Total Assay: Co 9769.3 ppm



Finland – Raja Prospect
Multi-elemental map

PAL0163 - 418.29 m



Co Fe Other

Cobalt mineral Pyrrhotite Silicates

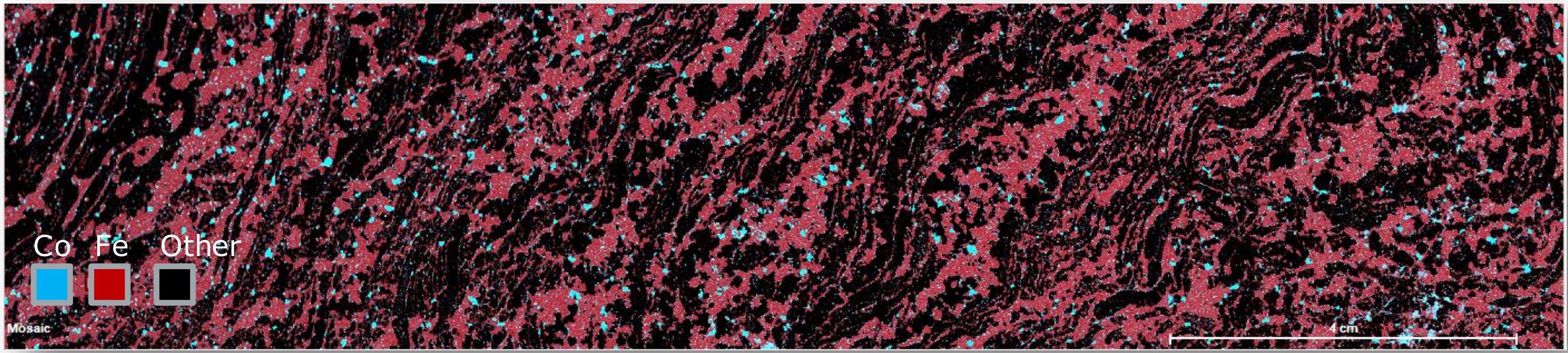
50 micron resolution

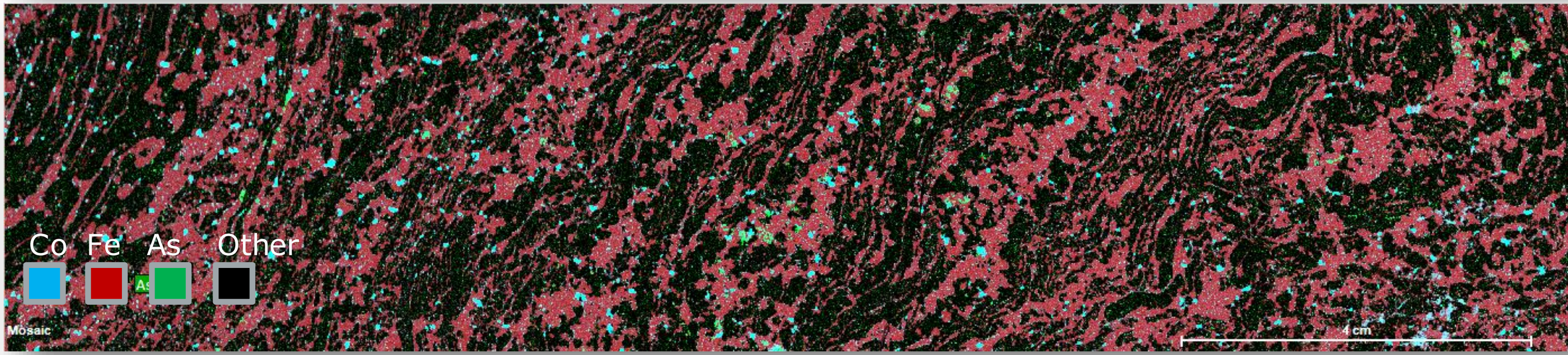
Linnaeite is associated with pyrrhotite

Finland – Raja Prospect
Multi-elemental map

PAL0163 - 418.29 m

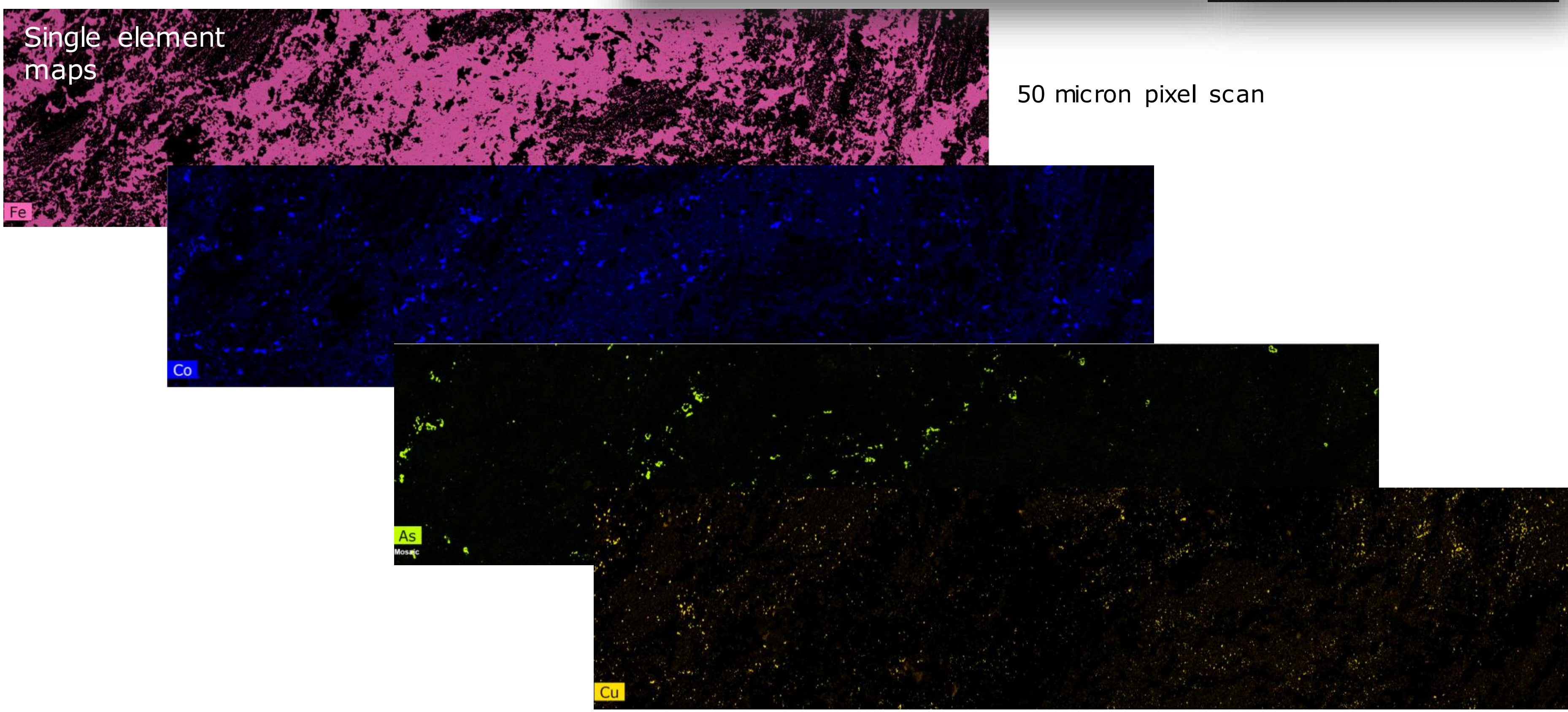
Linnaeite is associated with pyrrhotite; with patchy occurrences of cobaltite





Finland – Raja Prospect
Single-elemental map

PAL0163 - 418.29 m

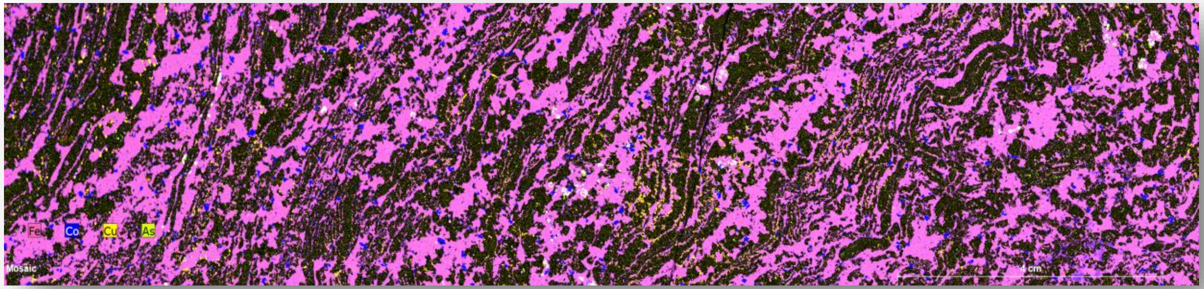


50 micron pixel scan

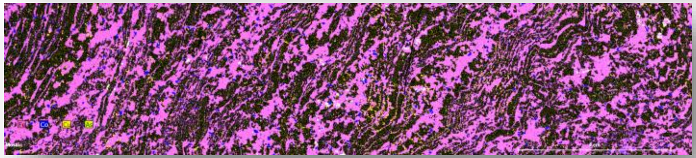
Micro-XRF
Analytical Conditions

Change in Resolution and Dwell Time impact Analytical Time:
Important to define what is the goal of analysis prior to Measurement

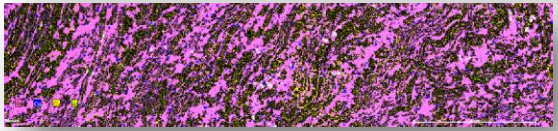
Variable Resolution



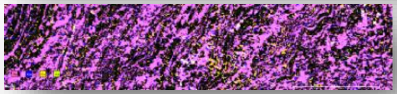
50 micron pixel scan
Less than 9 hours
total measurement time



100 micron pixel scan
Less than 3 hours
total measurement time



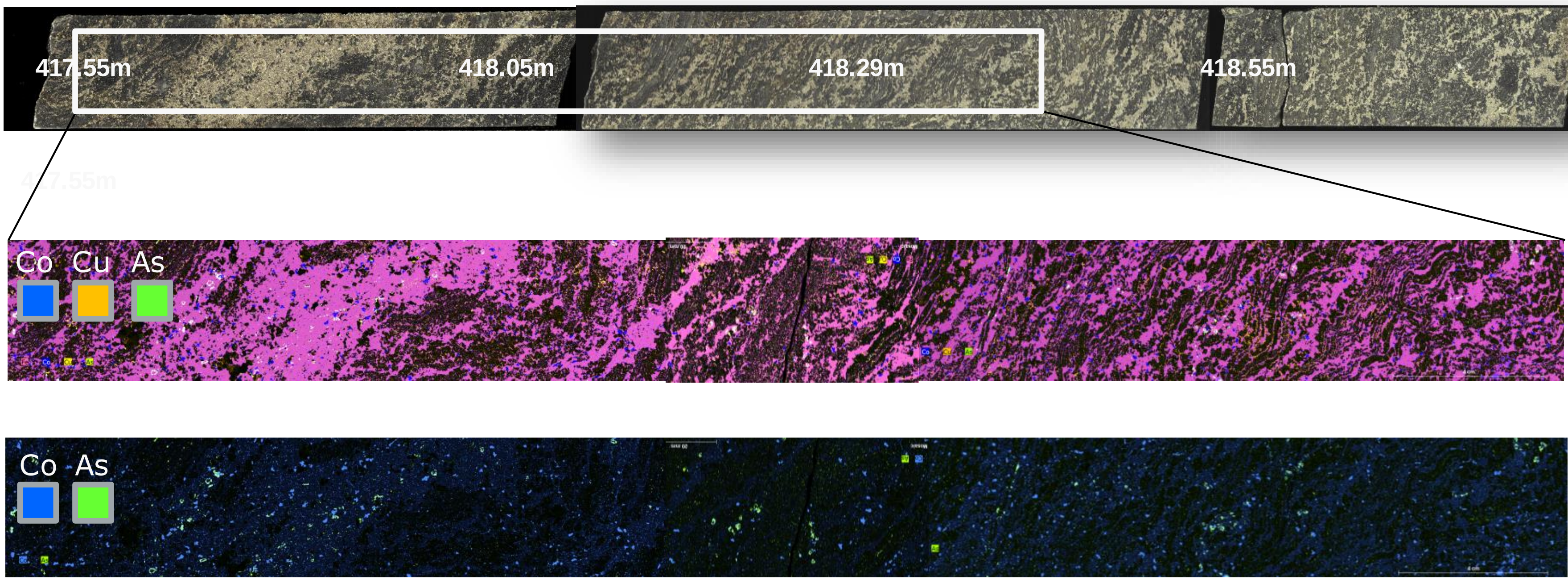
200 micron pixel scan
Less than 1 hour
total measurement time



500 micron pixel scan
Less than 1 hour
total measurement time

Finland – Raja Prospect
Multi-elemental map

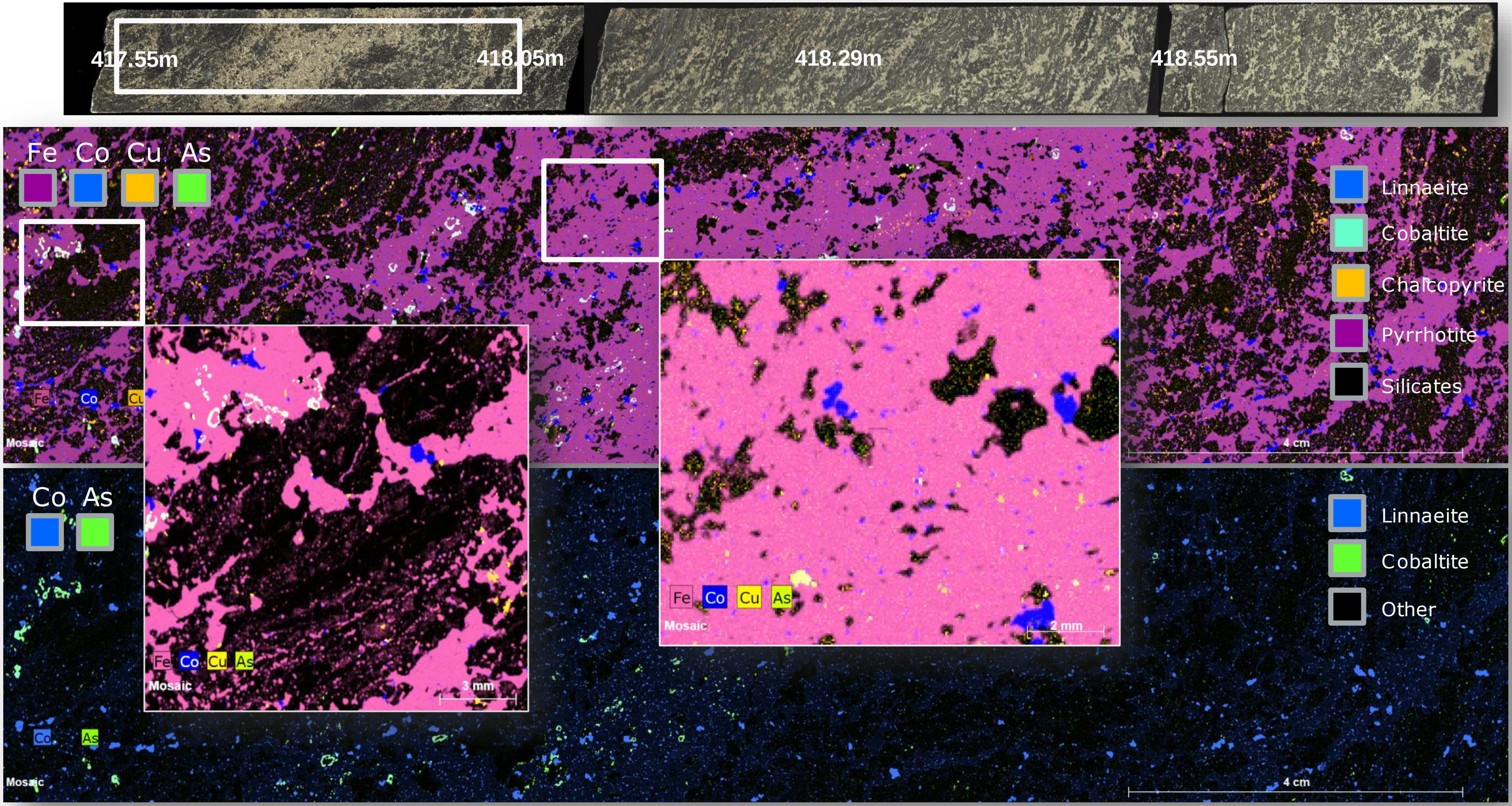
PAL0163 - 418.29 m



Finland – Raja Prospect

Large-area to small-area maps

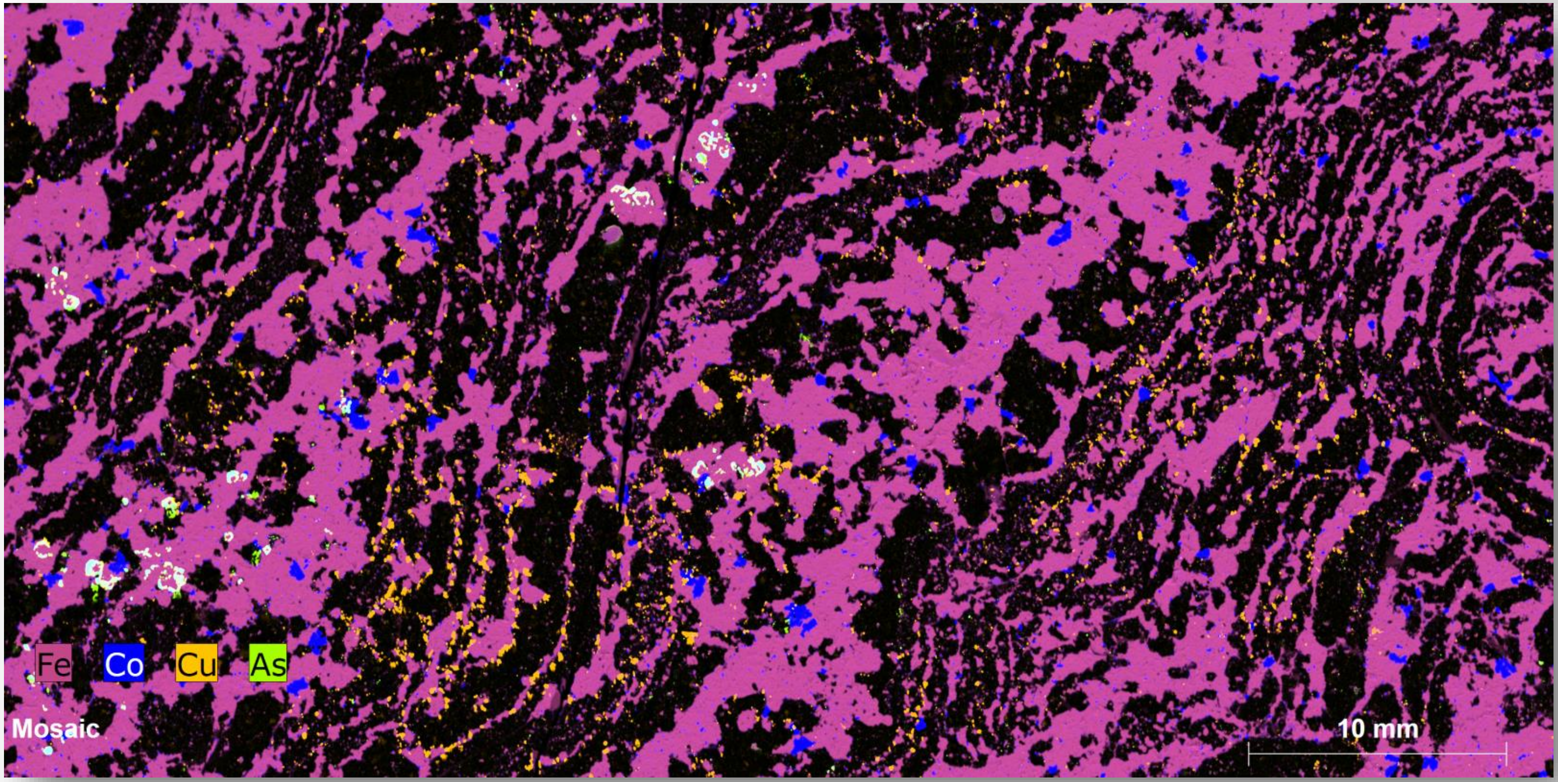
PAL0163 - 418.29 m



Finland – Raja Prospect
Small-area maps

PAL0163 - 418.29 m

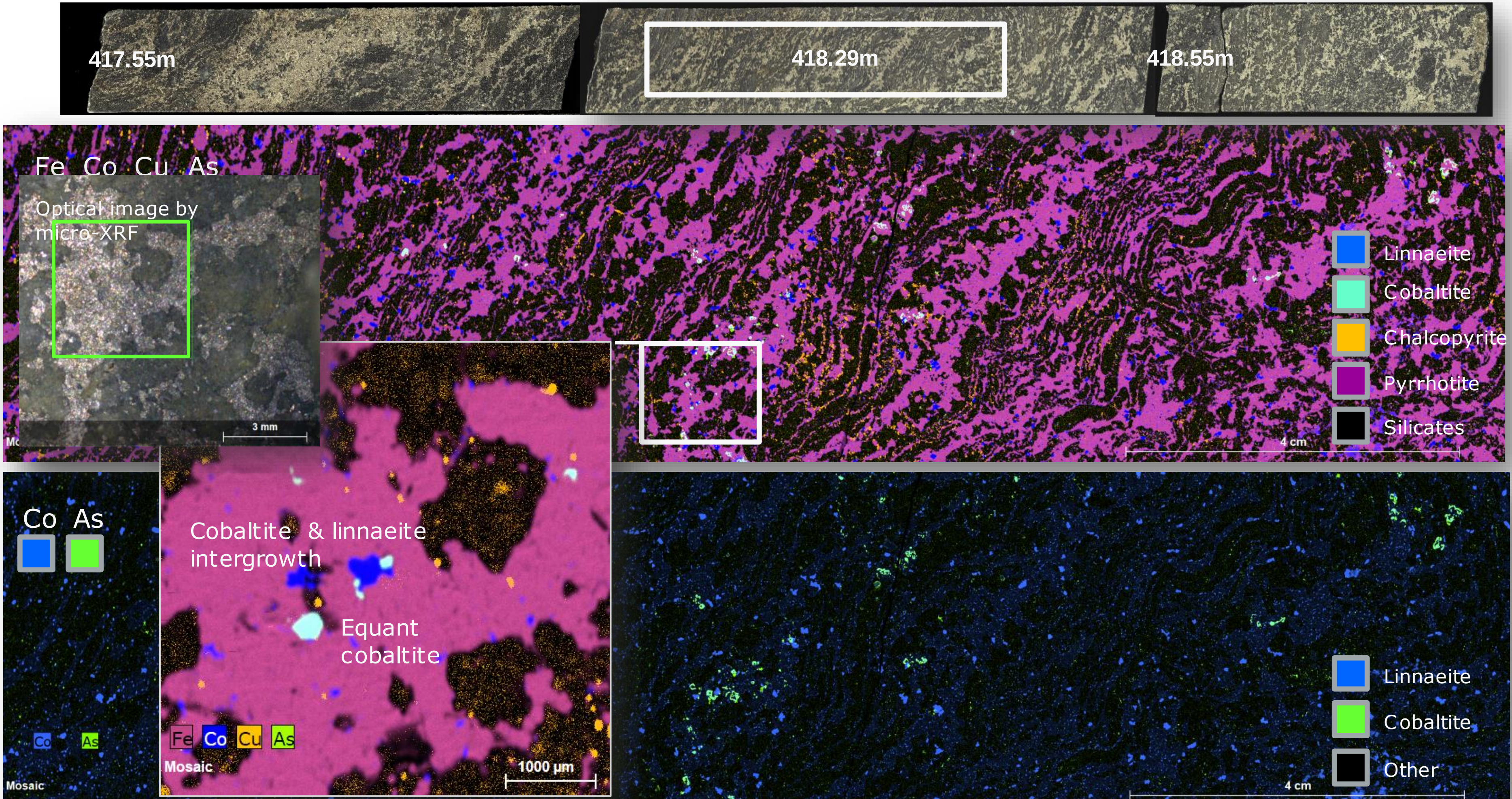
Cobaltite
Linnaeite
Pyrrhotite
Silicates
Chalcopyrite



20 micron pixel scan

Finland – Raja Prospect
Large-area to small-area maps

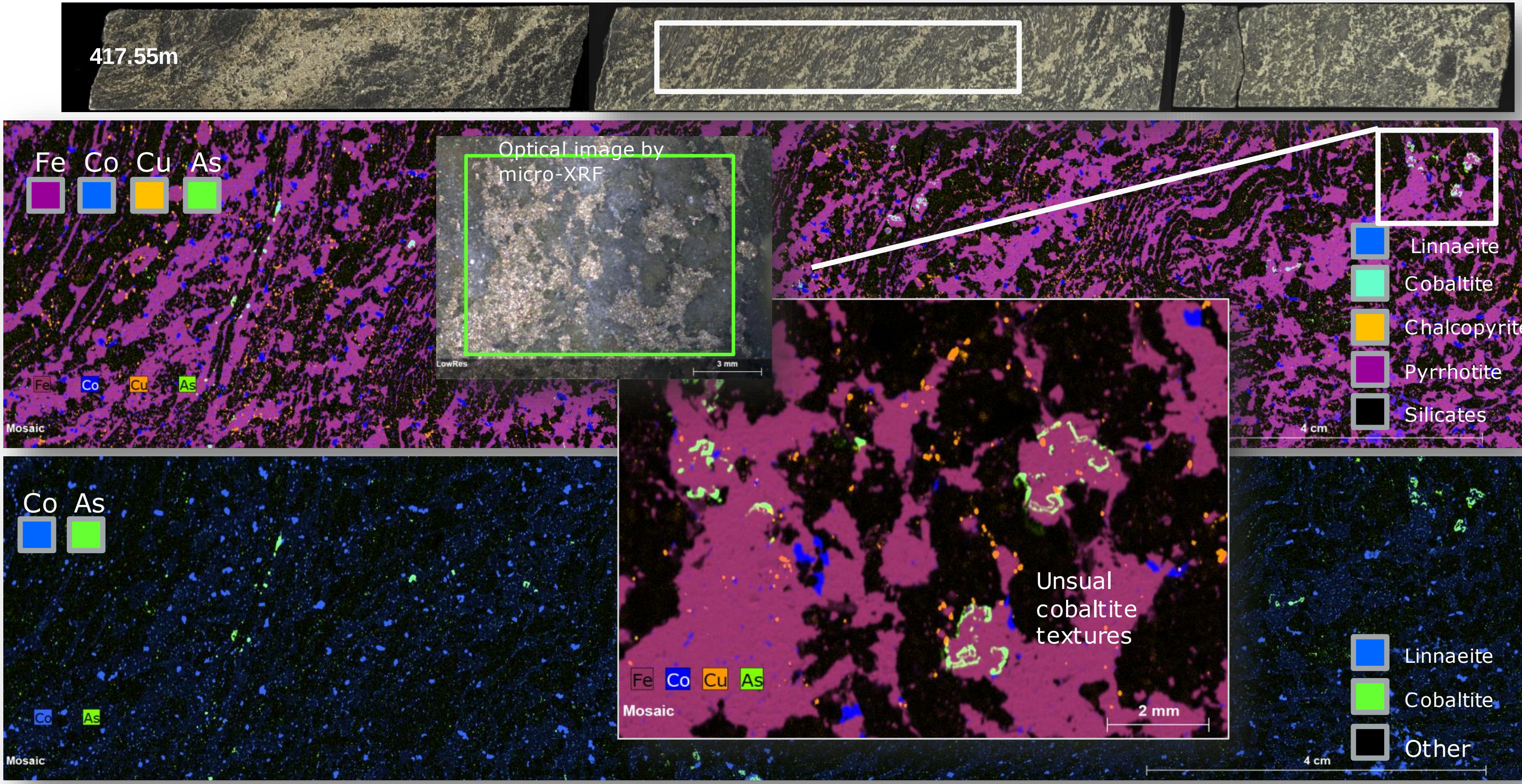
PAL0163 - 418.29 m



Finland – Raja Prospect

Large-area to small-area maps

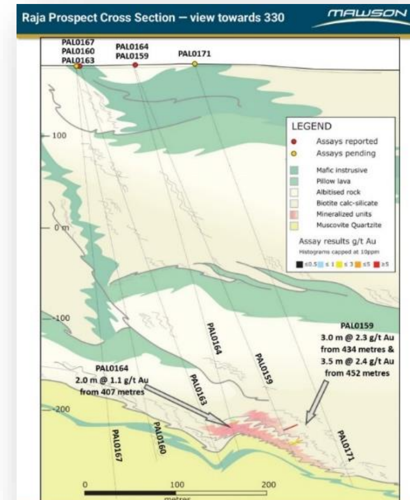
PAL0163 - 418.29 m

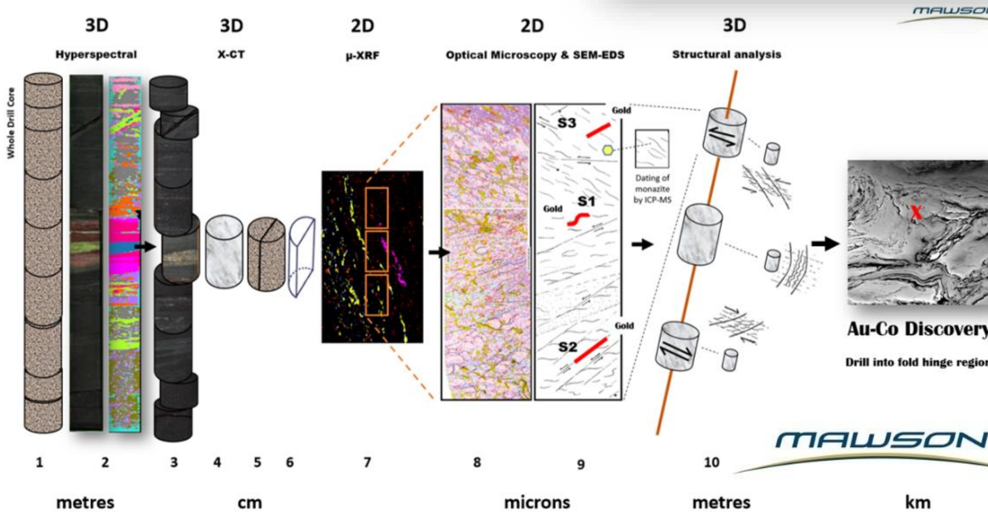


Finland – Raja Prospect

Mineralogy, Petrogenesis & Micro Geometallurgy

Multi-scale, multi-modal, multi-dimensional





MAWSON

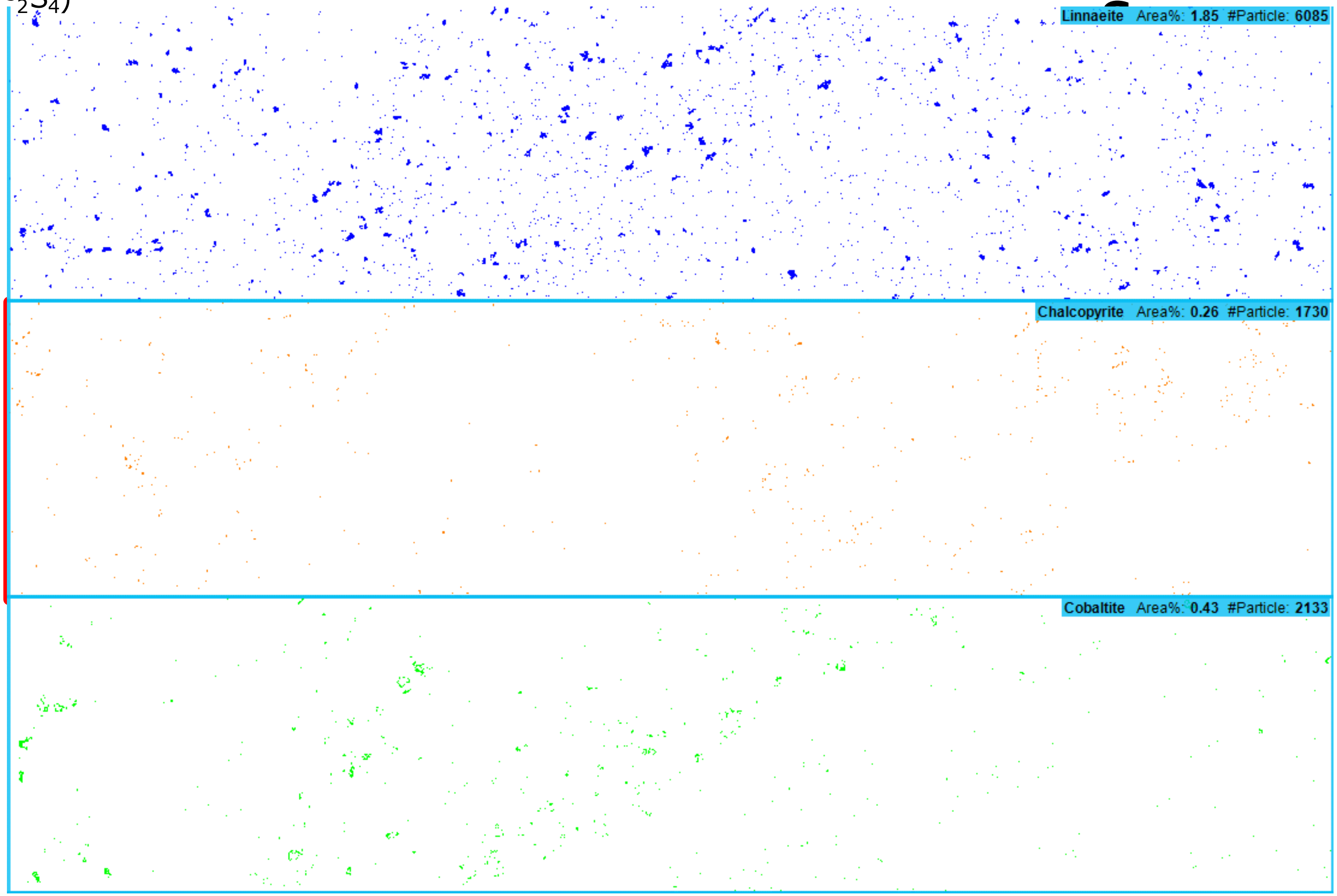
Finland – Raja Prospect, Rajapalot Project
Exploration Project: Au-Co (Mawson Oy)
AMICS: Mineralogy

Finland – Raja Prospect
AMICS: Mineralogy Map

Linnaeite ($Co^{+2}Co^{+3}_2S_4$)

Cobaltite (CoAsS)

Pyrrhotite	
Linnaeite	
Chalcopyrite	
Cobaltite	
Titanite	
Rutile	
Apatite	
Zircon	
Quartz	
Albite	
Phlogopite	
K-Feldspar	
Amphibole	
Chlorite	
Others	

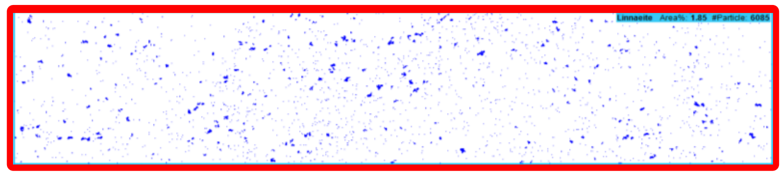


Finland – Raja Prospect

AMICS: Grain Size Distribution - Linnaeite (Co_3S_4)

Linnaeite ($Co^{+2}Co^{+3}_2S_4$)

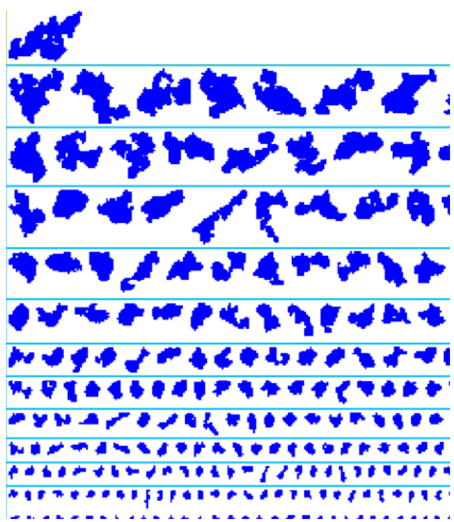
Cobaltite (CoAsS)

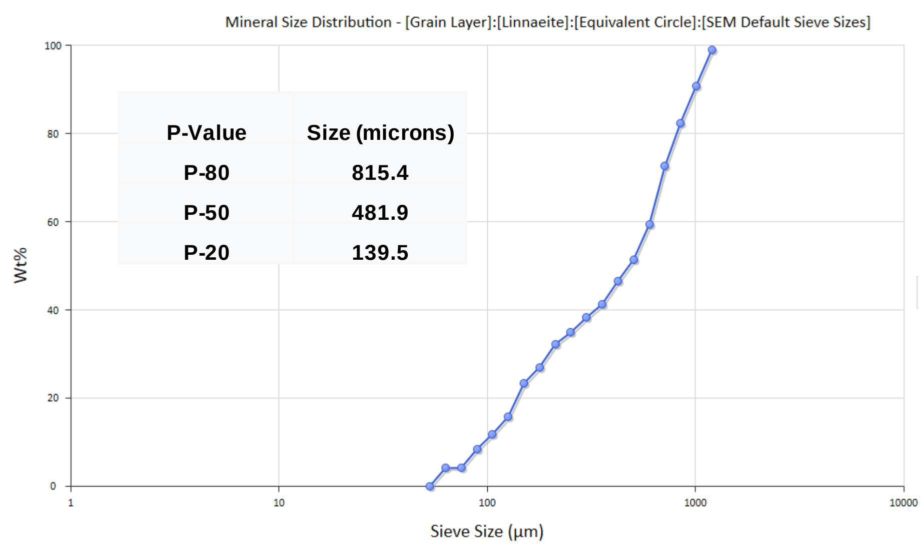


Scan

180 x 40 mm

Pyrrhotite	
Linnaeite	
Chalcopyrite	
Cobaltite	
Titanite	
Rutile	
Apatite	
Zircon	
Quartz	
Albite	
Phlogopite	
K-Feldspar	
Amphibole	
Chlorite	
Others	





Finland – Raja Prospect

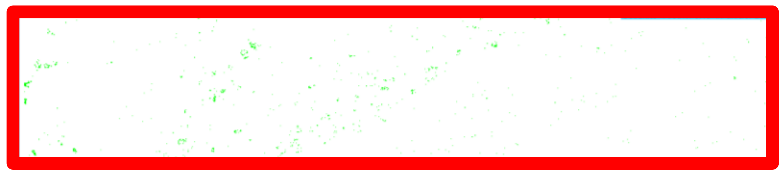
AMICS: Grain Size Distribution - Cobaltite (CoAsS)

Linnaeite ($Co^{+2}Co^{+3}{}_2S_4$)

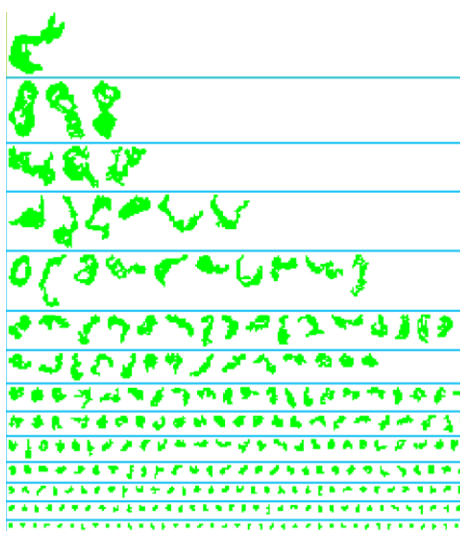
Cobaltite (CoAsS)

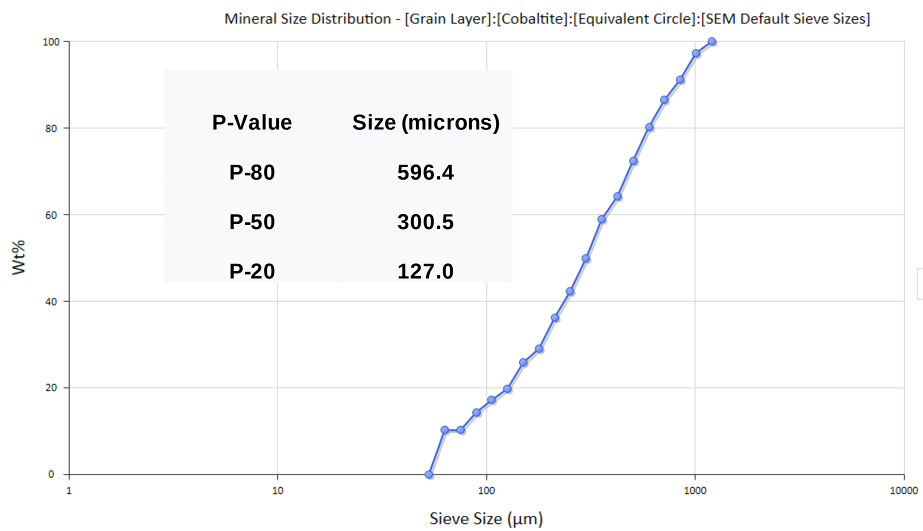
Scan

180 x 40 mm



Pyrrhotite	
Linnaeite	
Chalcopyrite	
Cobaltite	
Titanite	
Rutile	
Apatite	
Zircon	
Quartz	
Albite	
Phlogopite	
K-Feldspar	
Amphibole	
Chlorite	
Others	





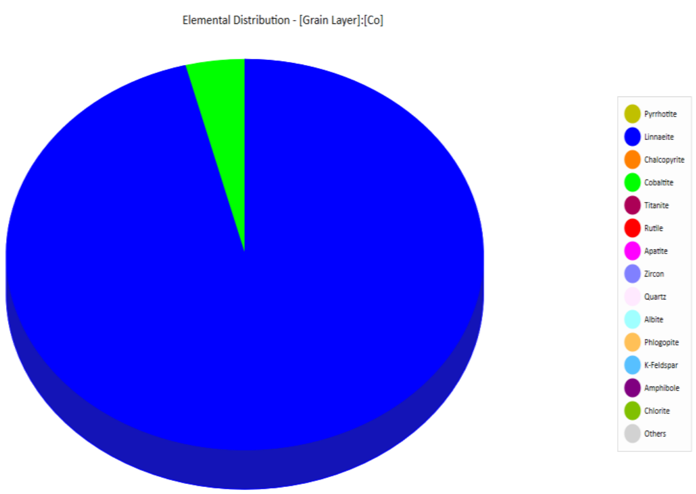
P-Value	Size (microns)
P-80	596.4
P-50	300.5
P-20	127.0

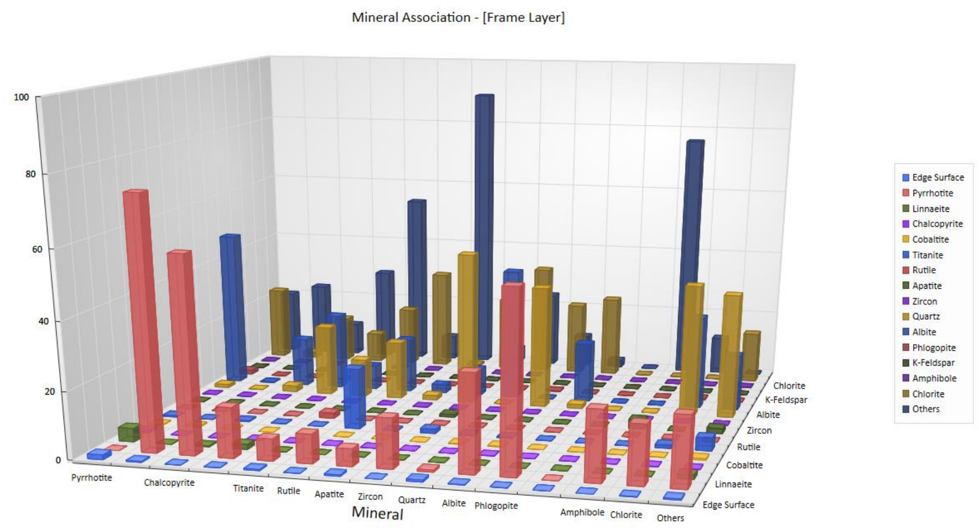
Micro XRF

AMICS: Element Distribution, Mineral Association

Elemental Distribution: How is the element of interest (EOI) distributed in each mineral? E.g. Co in Linnaeite vs Cobaltite.

Mineral Association: Identify how the minerals are associated with each-other, e.g. Cobaltite and Linnaeite and Pyrrhotite etc.

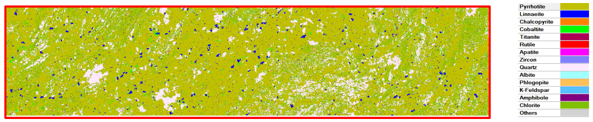




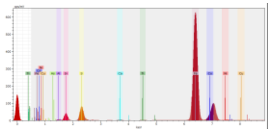
Finland – Raja Prospect

AMICS: Assay Calculations - Comparison

AMICS Back Calculated (Mineralogical)
Co concentration: 9340 ppm



AMICS Total Spectrum (Whole Rock)
Co concentration: 10400 ppm



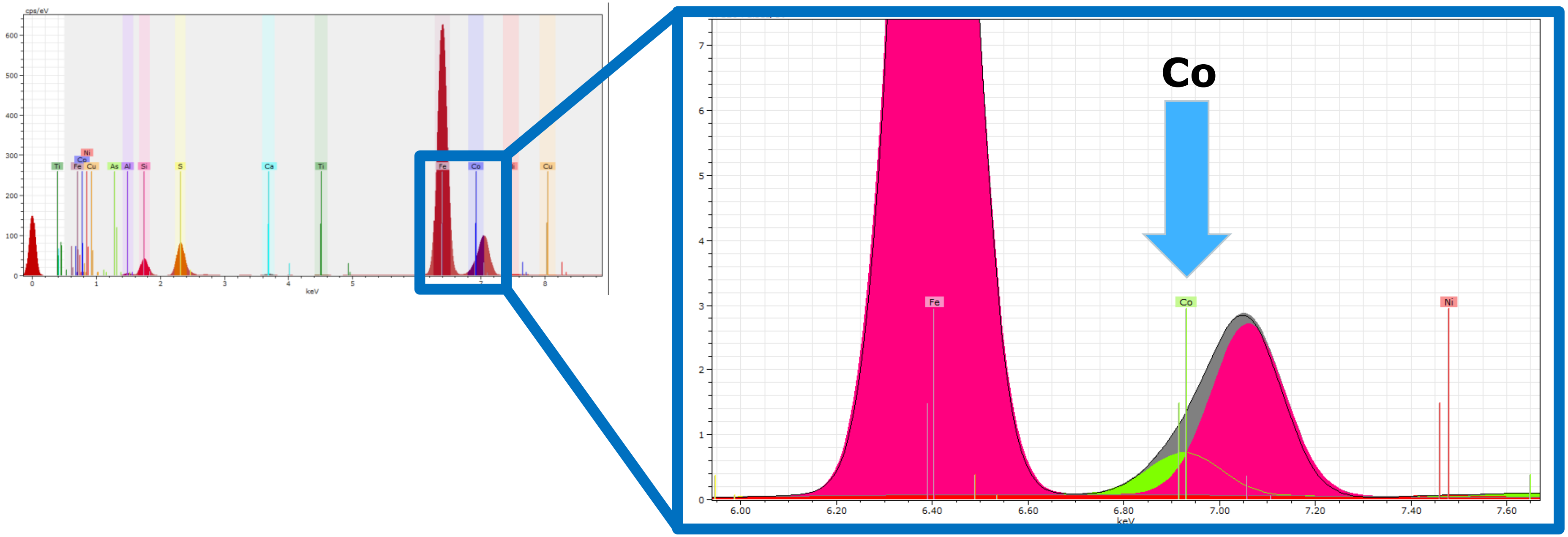
Cobalt content depths of 417.6 - 418.6 m
Chemical Assay Co concentration: 9769.3 ppm
(reported by Mawson Oy)

Micro XRF

AMICS: Assay Calculations

Total Spectrum (Whole Rock)

Co concentration: 1.04% (10400 ppm)



Deconvolution of Co (Cobalt) is important in the presence of Fe (Iron)

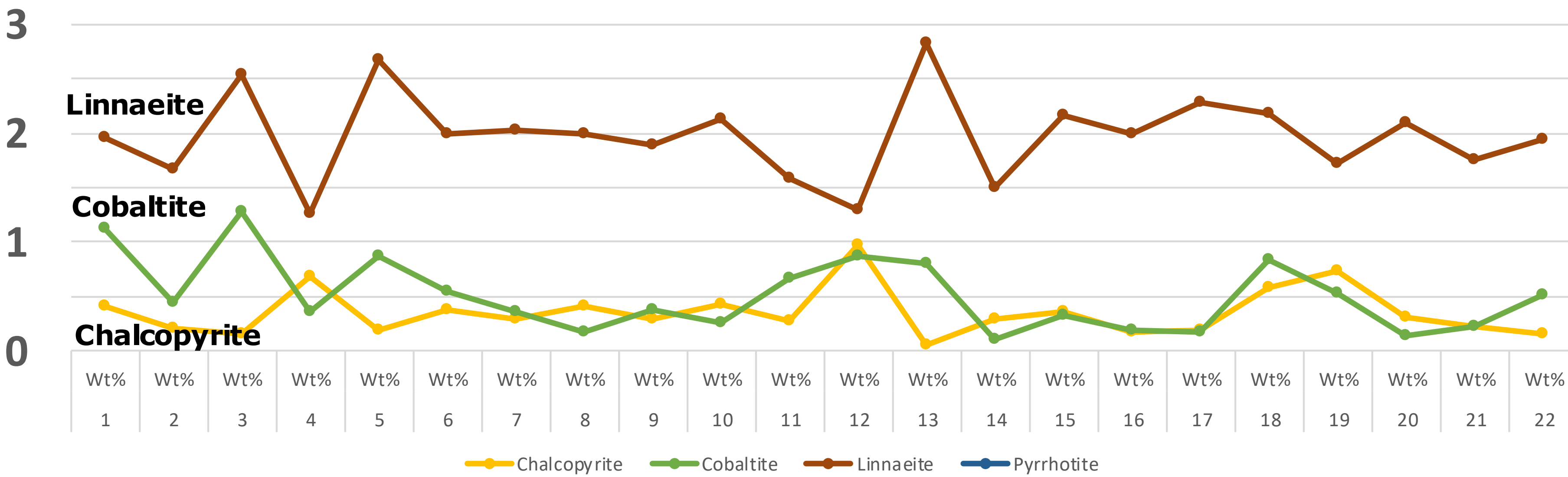
Micro XRF

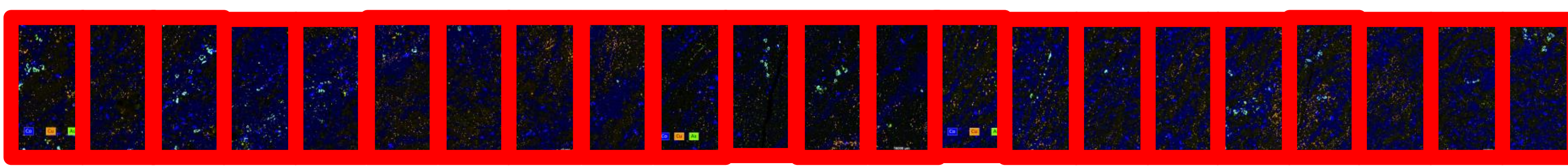
AMICS: Mineralogy Variation

Mineralogy Variation:

Thin Section Size - 30cm x 20cm

Linnaeite $(Co^{+2}Co^{+3}{}_2S_4)$
Cobaltite $(CoAsS)$





Large Mineralogical Variation on Thin Section Sized Specimens

Linnaeite Range: 1.4 % to 2.8 %

Factor of 2 Variation

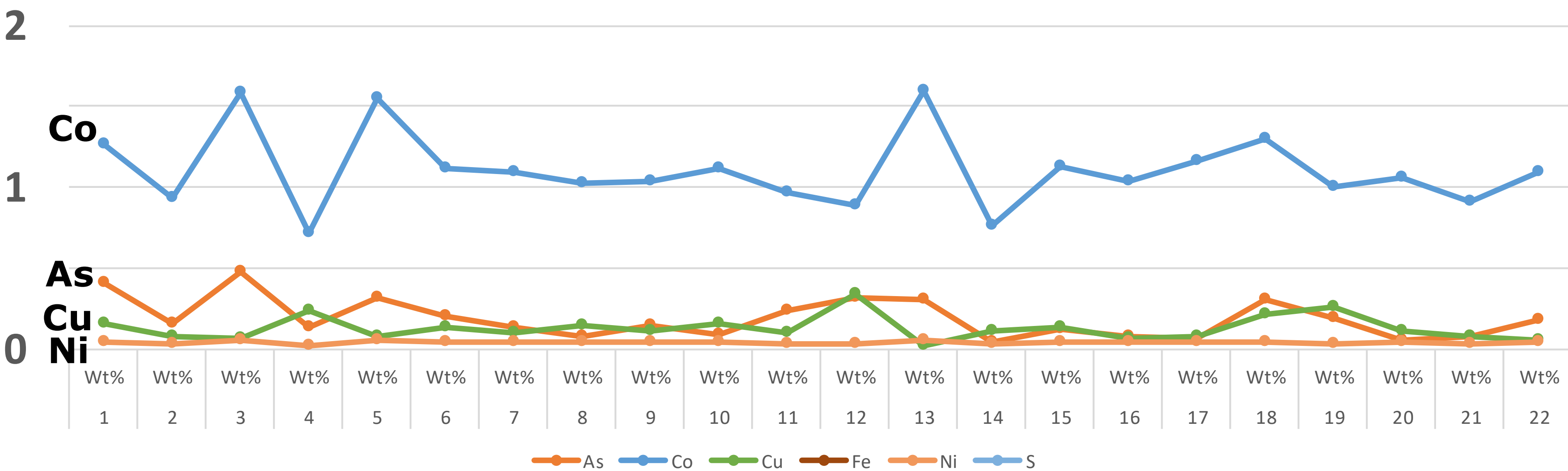
Micro XRF

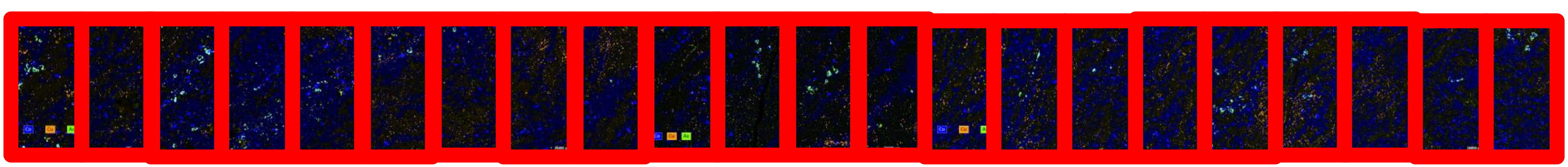
AMICS: Elemental Variation

Elemental Variation:

Thin Section Size - 30cm x 20cm

Linnaeite ($Co^{+2}Co^{+3}{}_2S_4$)
Cobaltite (CoAsS)





Large Elemental Variation on Thin Section Sized Specimens

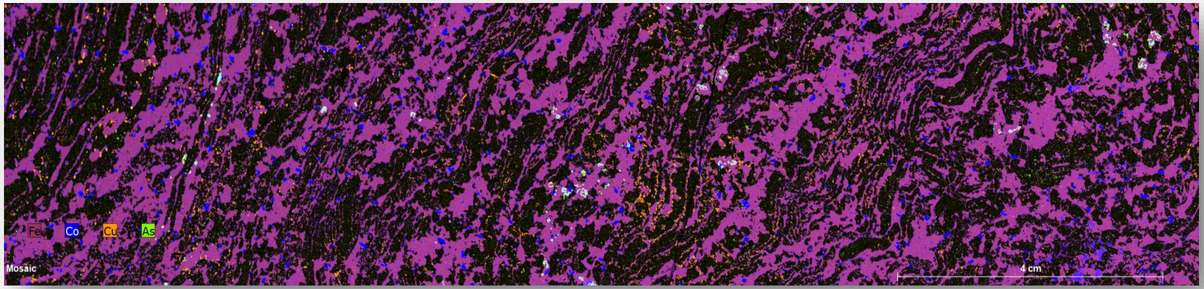
Co Concentration Range: 0.7 % to 1.5 %

Factor of 2 Variation

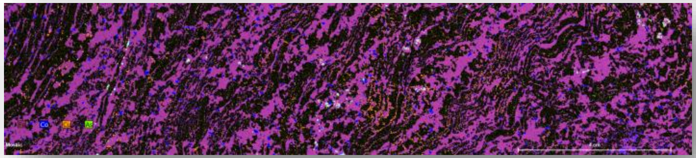
Micro XRF
Analytical Conditions

Change in Resolution and Dwell Time impact Analytical Time:
Important to define what is the goal of analysis prior to Measurement

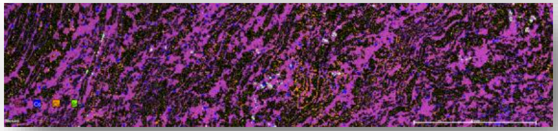
Variable Resolution



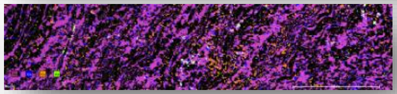
50 micron pixel scan
Less than 9 hours
total measurement time



100 micron pixel scan
Less than 3 hours
total measurement time



200 micron pixel scan
Less than 1 hour
total measurement time



500 micron pixel scan
Less than 1 hour
total measurement time

- Linnaeite
- Pyrrhotite
- Silicates
- Chalcopyrite

Micro XRF

AMICS: Potential Relevant Information

Modal Mineralogy: How much of each mineral is present.

Elemental Assay: How much of each element is present.

Elemental Distribution: How is the element of interest (EOI) distributed in each mineral? E.g. Co in Linnaeite vs Cobaltite.

Mineral Association: Identify how the minerals are associated with each-other, e.g. Cobaltite and Linnaeite and Pyrrhotite etc.

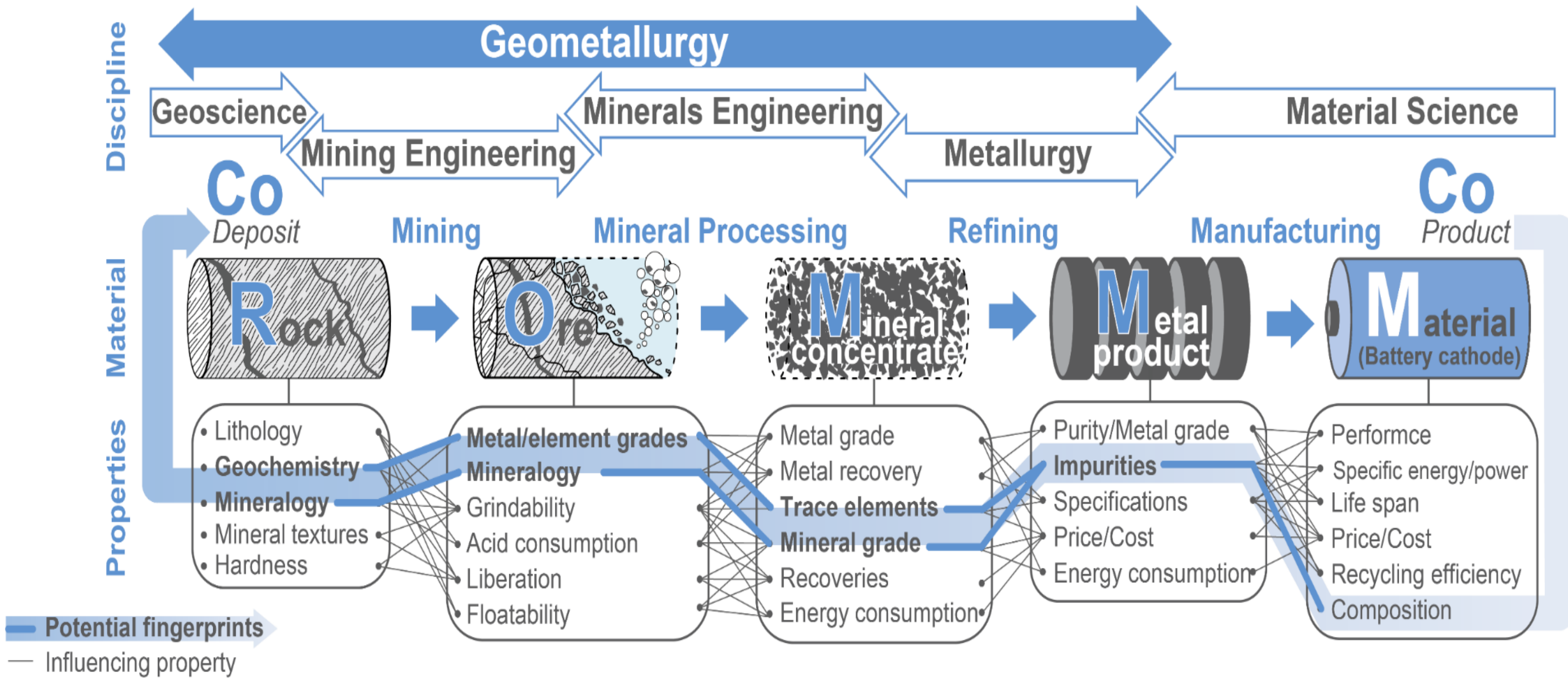
Grain Shape Factor: The shape of the grain, i.e. euhedral, elongated.

Mineral Density Distribution: Classify densities of minerals. Identify how the minerals are distributed among the densities.

Grade Recovery Curves: What % of minerals of interest (MOI) or element of interest (EOI) is recovered at what grade?

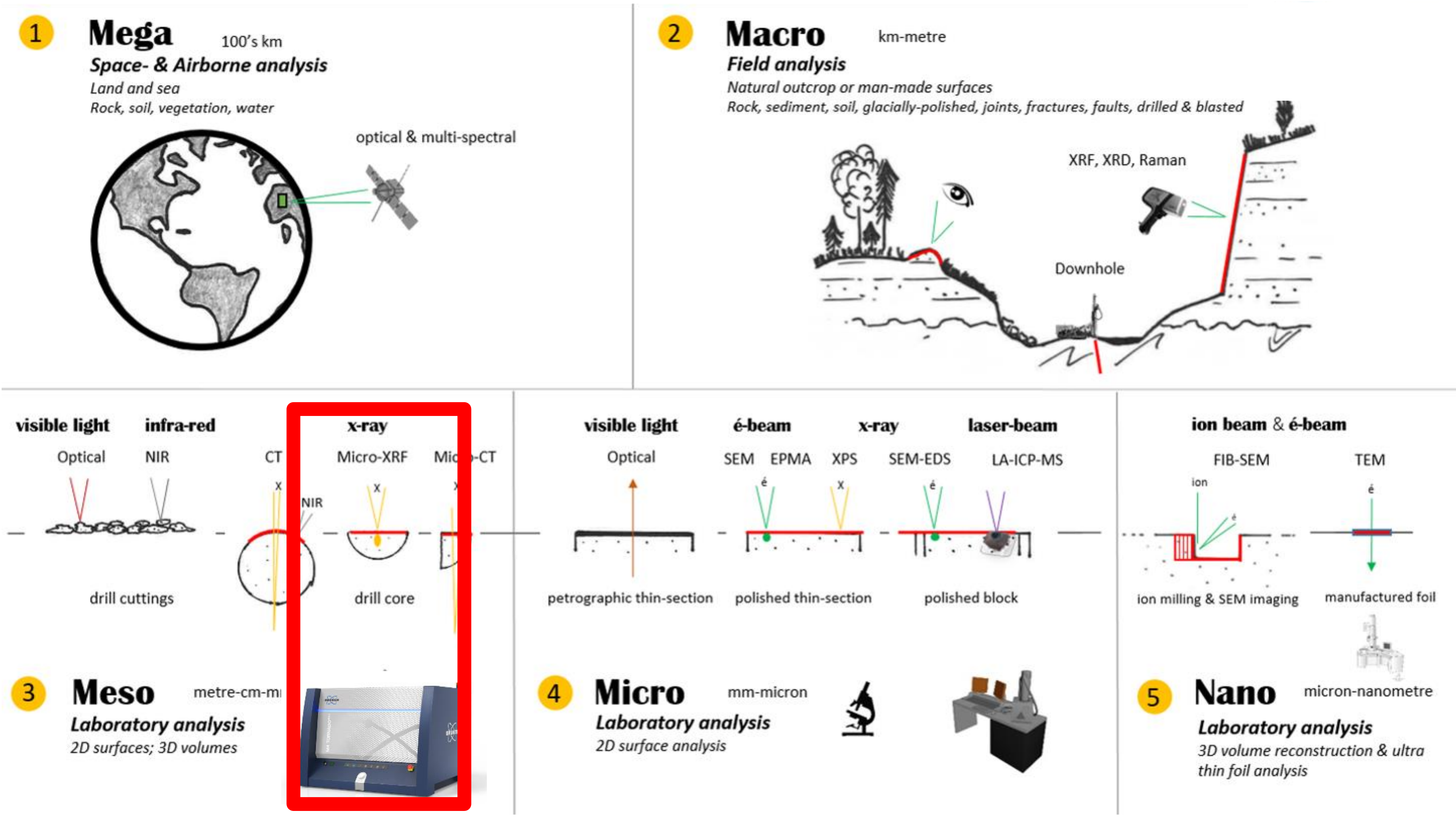
Geometallurgy:
Traceability Workflow

Integrated approach to improve value chain and responsible sourcing



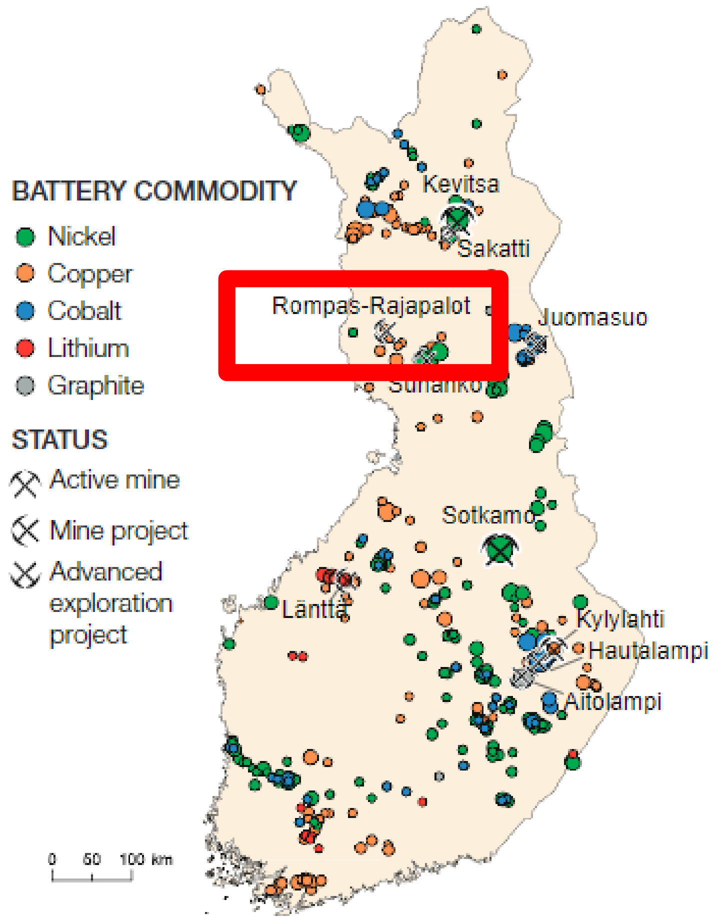
Q. Dehaine, S. Michaux, J. Pokki, M. Kivinen and A.R. Butcher, *Battery minerals from Finland: Improving the supply chain for the EU battery industry using a geometallurgical approach*, European Geologist 49 (*In Press*).

Multi-scale Characterisation Workflow



Butcher AR (2020) Upscaling of 2D mineralogical information to 3D volumes for geoscience applications using a multi-scale, multi-modal and multi-dimensional approach. ***EMAS 2019**, Conference Proceedings Volume, Trondheim*, 19-23 May 2019.

Nick Cook, President
Mawson Resources



Summary and Conclusions:
Micro-XRF

- Able to perform large area maps on a variety of samples:
  - Including Cut Rock Samples, Drill Core, Briquettes, Polished Sections

- Sample Preparation is Minimal for Micro-XRF
  - No carbon-coating
  - No polishing
  - Plane Parallel Surface only required

- Able to detect and resolve minor and trace elements

- Identification of high energy X-Ray lines

Summary and Conclusions
Mineralogy and Micro-Metallurgy

- Ability to measure cut half drill core without further sample preparation
- *Tens of centimetres-scale* sample analysed at *Tens of micron-scale* resolution
- Areas of interest at higher resolutions
- Elemental and Mineralogical information on same area
- Maps of elements and mineral distributions
- Determine textural and geometallurgical information using AMICS Automated Mineralogy Software

Extra InformationWebinars
Historic and Current

Webinars

www.bruker.com/events/webinars.html

Filter: EDS, WDS, EBSD, Micro-XRF on SEM

https://www.gtk.fi/

https://www.mawsonresources.com/

https://www.batcircle.fi/

More Information

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Questions and Answers

Are There Any Questions?

Please type in the questions you might have

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

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Innovation with Integrity