

# Advanced Data Mining in Micro-XRF



Bruker Nano Analytics, Berlin, Germany  
Webinar, September 4, 2018

Na	Mg		
K	Ca	Sc	Ti
Rb	Sr	Y	Zr
Cs	Ba	La	Hf
Fr	Ra	Ac	

V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn

Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr



Results		Primary energy	0.0 keV			
		Tilt angle	0.0°			
	Series	Net	[wt.%]	[wt.%]	Atom C. Error	
			[wt.%]	[wt.%]	[wt.%]	
Iron	K series	214751713	94.59	93.82	93.76	3.64
Nickel	K series	6274049	5.76	5.71	5.43	0.03
Copper	K series	7388	0.01	0.01	0.01	0.00
Zinc	K series	2017	0.00	0.00	0.00	0.00
Phosphorus	K series	89042	0.36	0.35	0.64	0.00
Sulfur	K series	37785	0.88	0.08	0.54	0.00
Chromium	K series	99229	0.03	0.03	0.03	0.00
	Total	100.82	100.00	100.00		

XFlash®  
Technology

Micro-XRF

## Are There Any Questions?

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

# M4 TORNADO Webinar

## Presenters



Falk Reinhardt

Application Scientist,  
Bruker Nano Analytics, Berlin, Germany



Dr. Roald Tagle

Sr. Application Scientist,  
Bruker Nano Analytics, Berlin, Germany

# M4 TORNADO Webinar

## Outline



- Introduction
  - Presenters
  - The M4 instrument
  - Micro-XRF
  - Position Tagged Spectroscopy
- Homogeneous samples? – Cu- and Fe-alloys
- Heterogeneous in 1 dimension – drill core
- Heterogeneous – concrete
- Live part – data extraction and evaluation
- Summary

# M4 TORNADO micro-XRF spectrometer

## Standard configuration



**30 W micro-focus Rh tube with polycapillary lens**  
for excitation spot sizes  $< 20 \mu\text{m}$  (for Mo-K $\alpha$ )  
Option: other target materials and second X-ray tube  
(collimated) for extended excitation conditions

**30 mm<sup>2</sup> silicon drift detector (SDD)**  
with energy resolution  $< 145 \text{ eV}$  (for Mn-K $\alpha$ )  
Option: second detector with second independent SPU  
for double pulse throughput  
Option: 60 mm<sup>2</sup> detector(s)

**Sealed sample chamber**  
with adjustable pressure between 1 mbar (for detecting light elements down to Na) and ambient pressure

Sample stage with measurable area of 190 mm x 160 mm

Maximum sample height 120 mm, maximum sample weight 5 kg

Sample stage speed up to 100 mm/s, minimum step size 4  $\mu\text{m}$

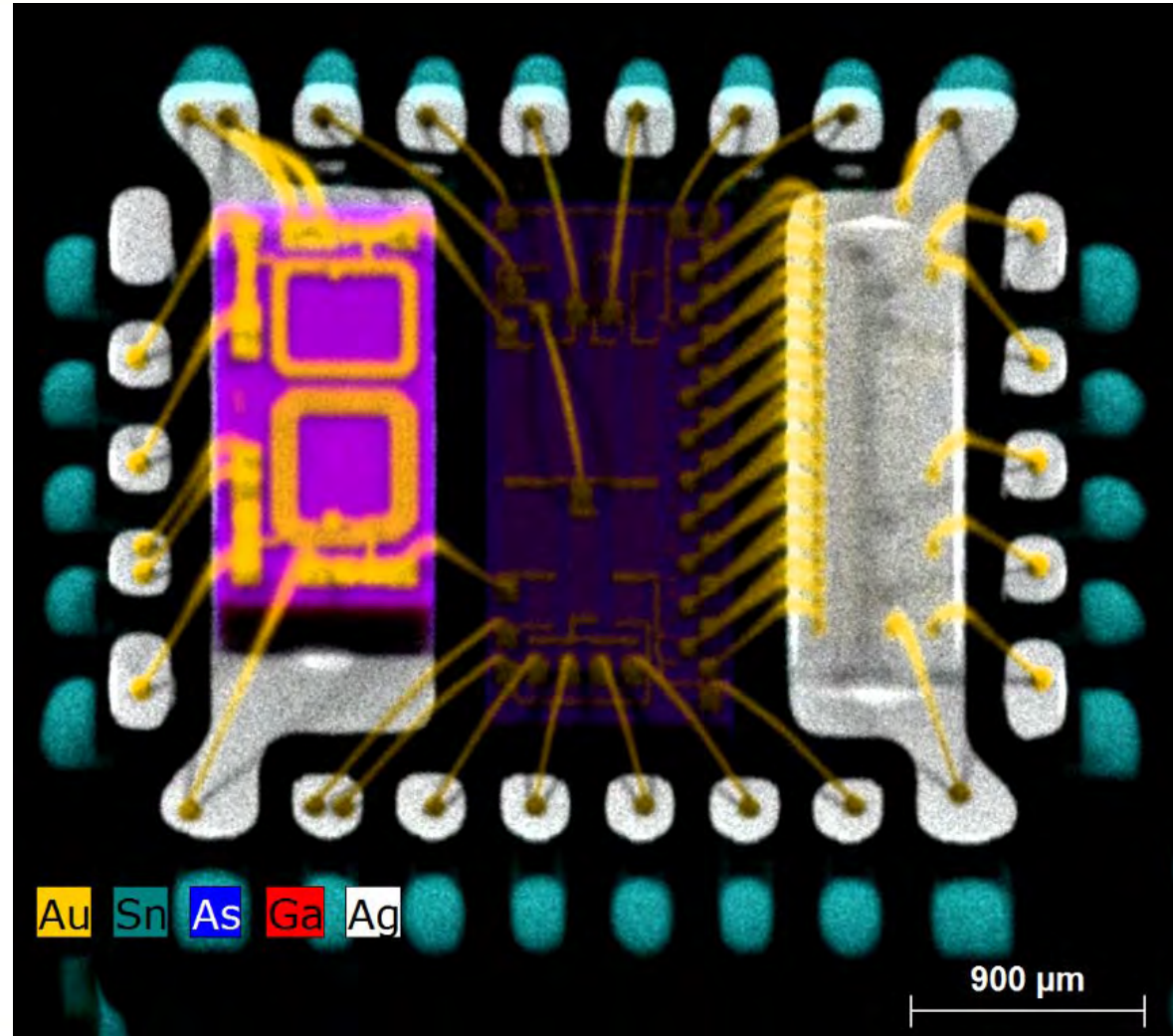
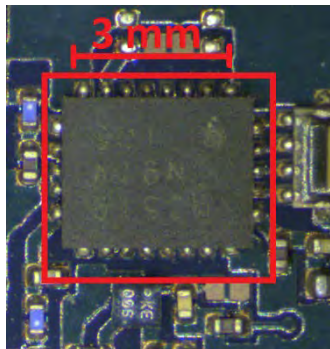


# Micro-XRF features and benefits

## At a glance



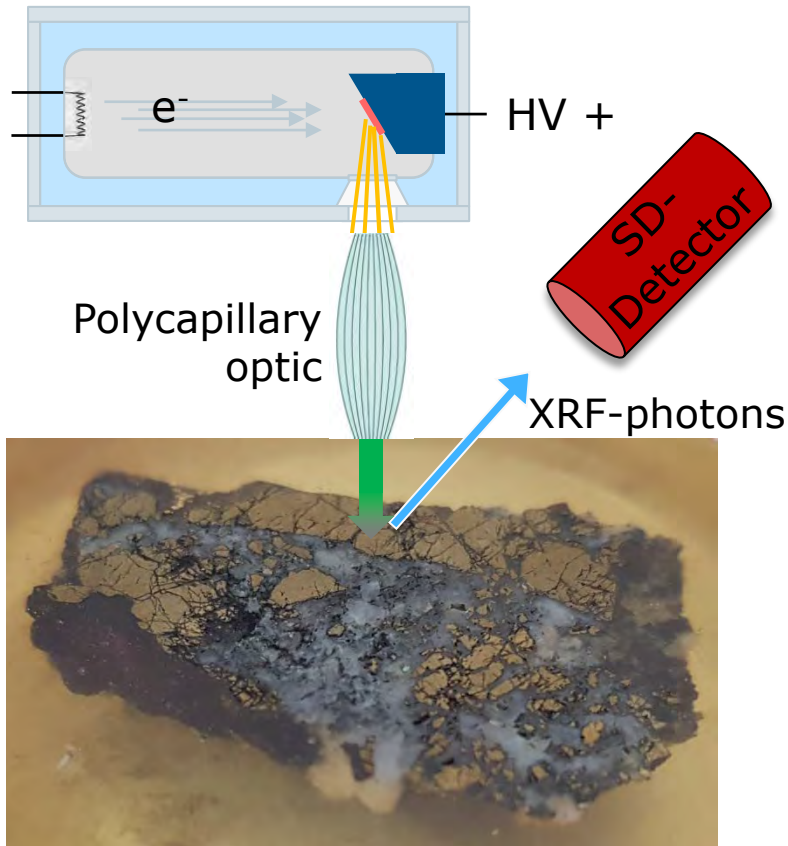
- Little to no sample preparation
- Non-destructive
- Elemental information
- Small spot analysis
- Information from within the sample
- Large-scale
- Quantification



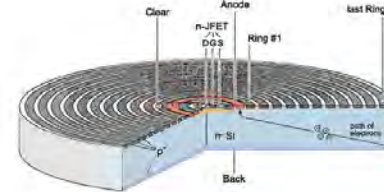
# X-Ray Fluorescence ...elemental analysis



## Excitation



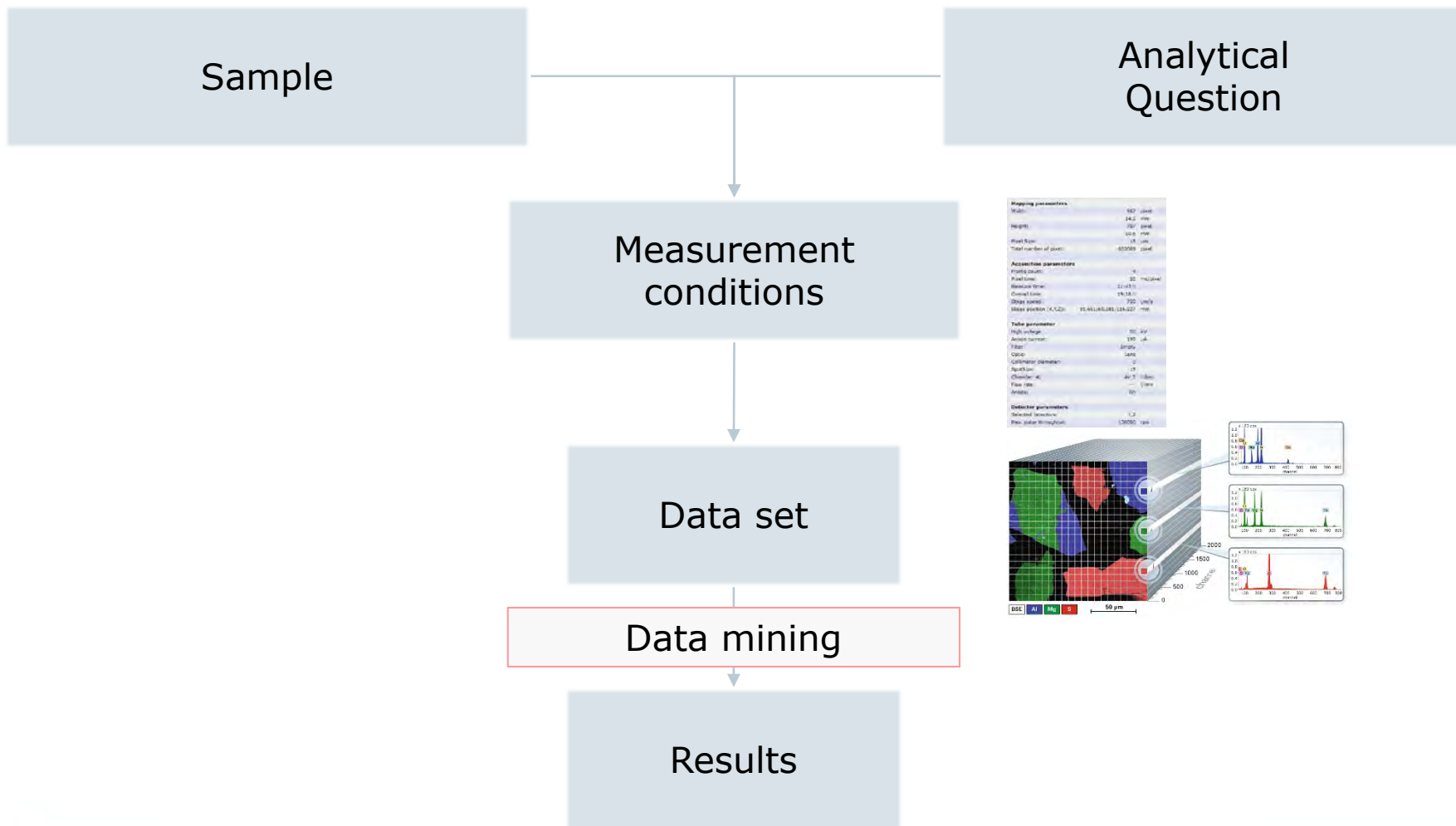
## XRF Detection



**Silicon Drift Detector**  
with XFlash® Technology

- X-rays can be guided onto a small spot
- Spatially resolved element-specific signal
- Intensity ratios of observed elemental lines can be used for quantification

# From question to results



Mapping parameters	
Width	162 pixel
Height	142 pixel
Pixel size	10.0 pixel
Total number of pixels	60368 pixel

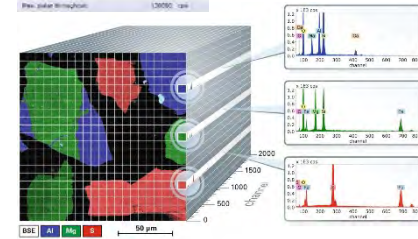
Acquisition parameters	
Points/scan	1
Pixel time	22.720/scan
Element time	11.077 s
Count rate	9128.0
Strip current	750 µm/s
Strip position (x, z)	11.611, 0.183, 128.622 mm

Tube parameter	
High voltage	10.0 kV
Anode current	110 µA
Filter	large
CRP	large
Scatterer diameter	1
Spot size	15
Detector ac	40.0°/scan
File name	11111111
Analysis	111

Detector parameters	
Detector resolution	1.2
Photo. detector	130500.128



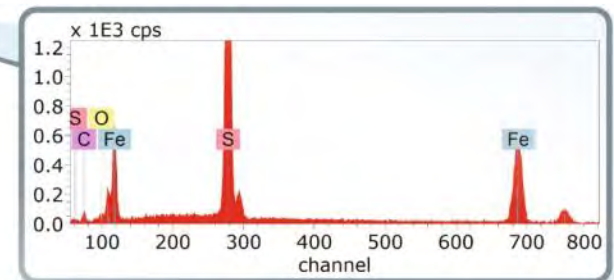
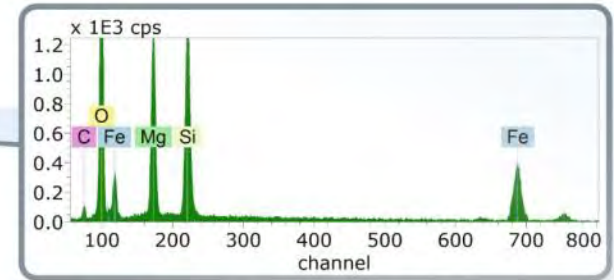
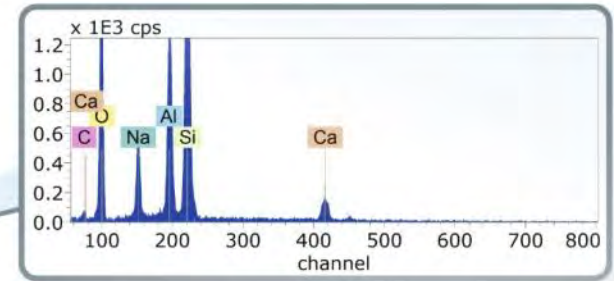
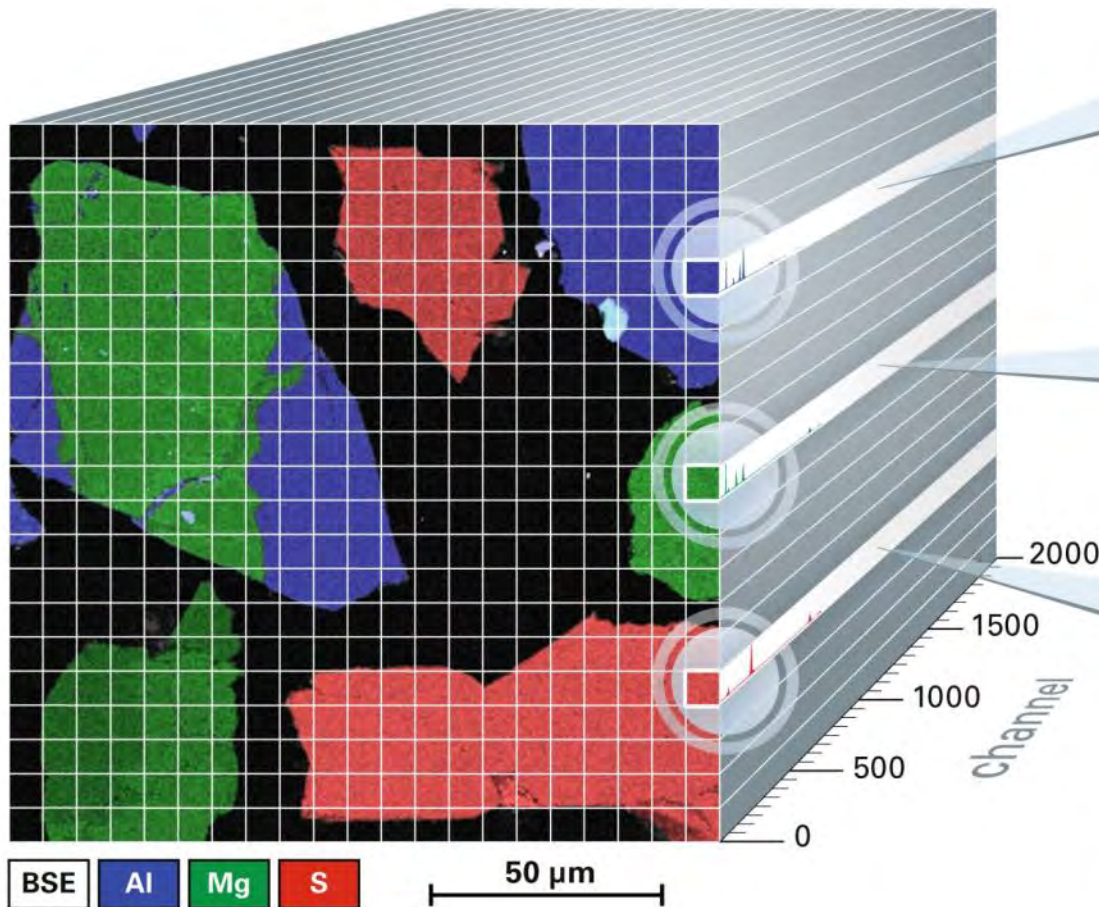


# PTS – Position Tagged Spectroscopy

all map data at hand



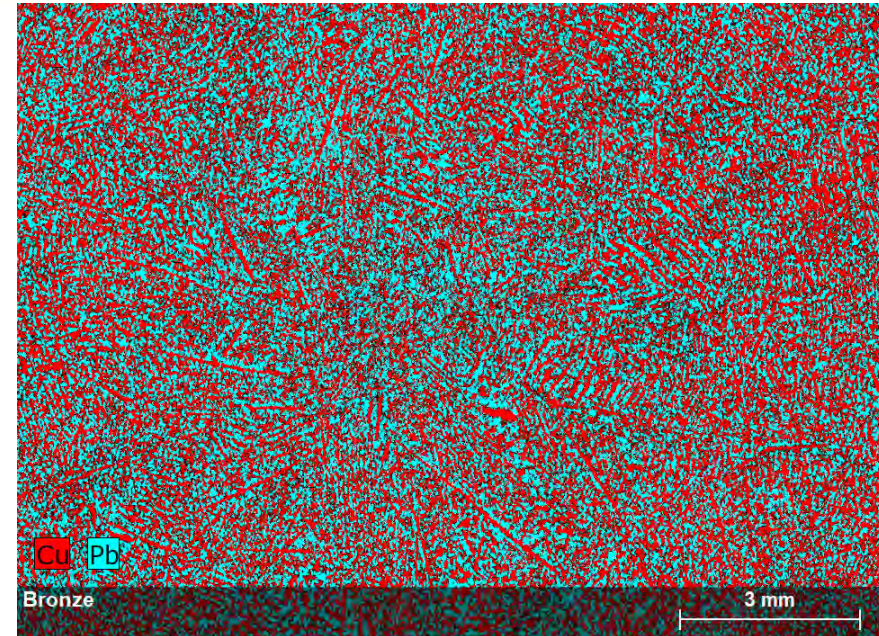
the „data cube “



# PTS – Position Tagged Spectroscopy all map data at hand



Analytical question:  
homogeneity of a bronze slab



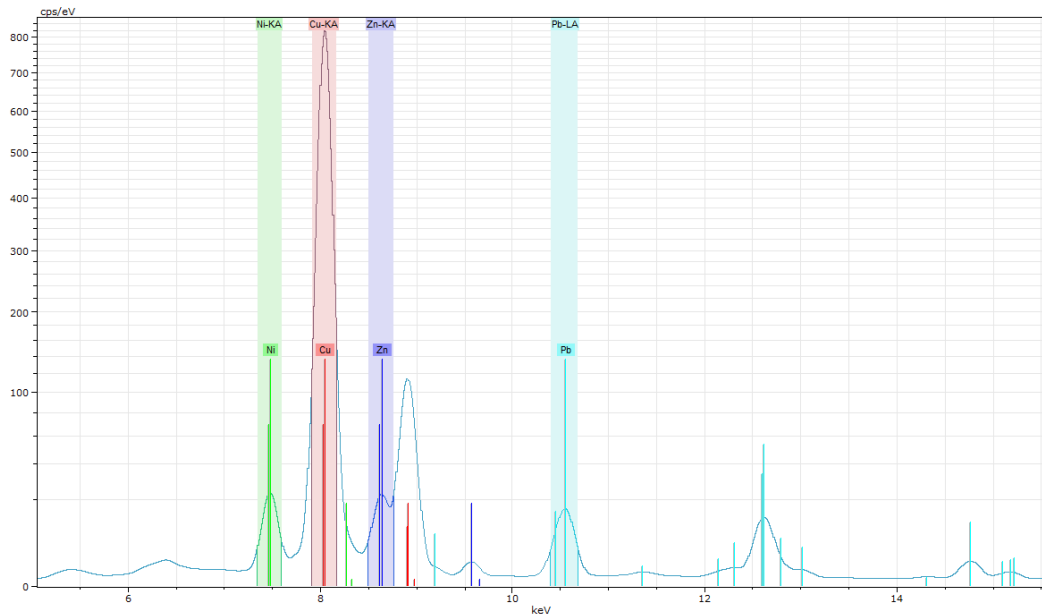
# PTS – Position Tagged Spectroscopy

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Analytical question:  
homogeneity of a bronze slab

Situation before measurement:  
All expected elements selected



# PTS – Position Tagged Spectroscopy

## all map data at hand

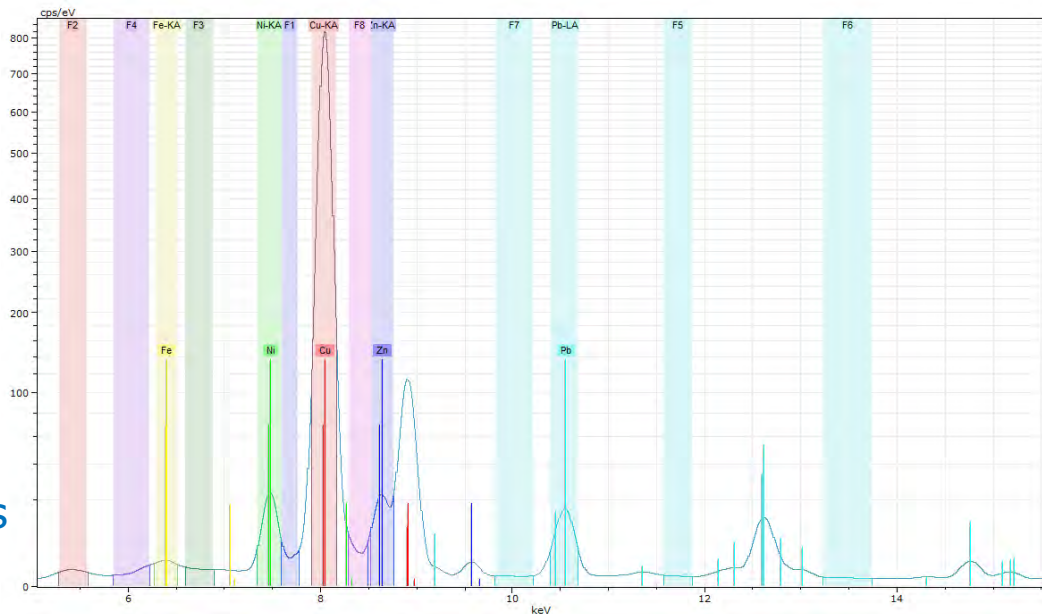


Analytical question:  
homogeneity of a bronze slab

Situation before measurement:  
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Post evaluation:

- Sum spectrum reveals additional peaks
- Identify unexpected elements
- Define 'free regions'



# PTS – Position Tagged Spectroscopy

## all map data at hand

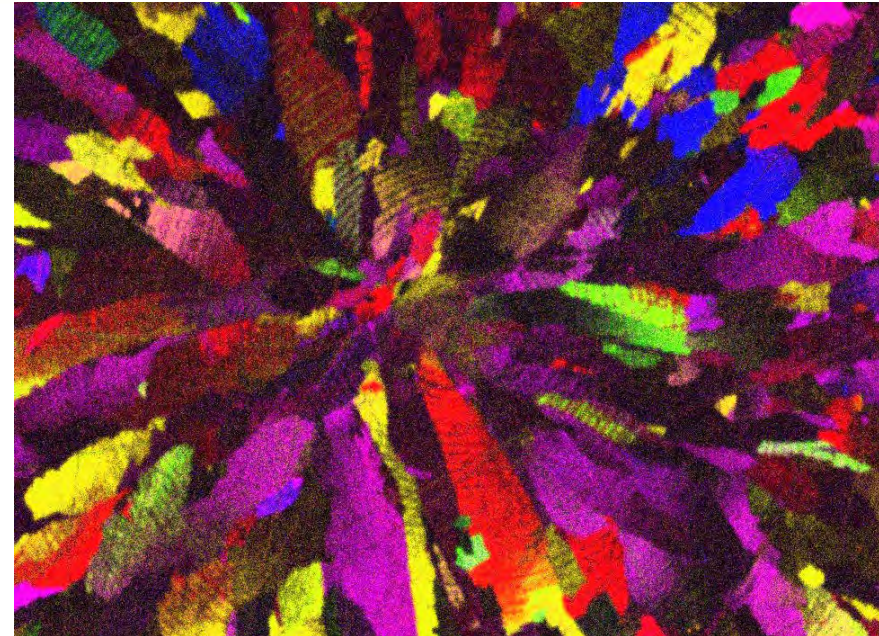


Analytical question:  
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Situation before measurement:  
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Post evaluation:

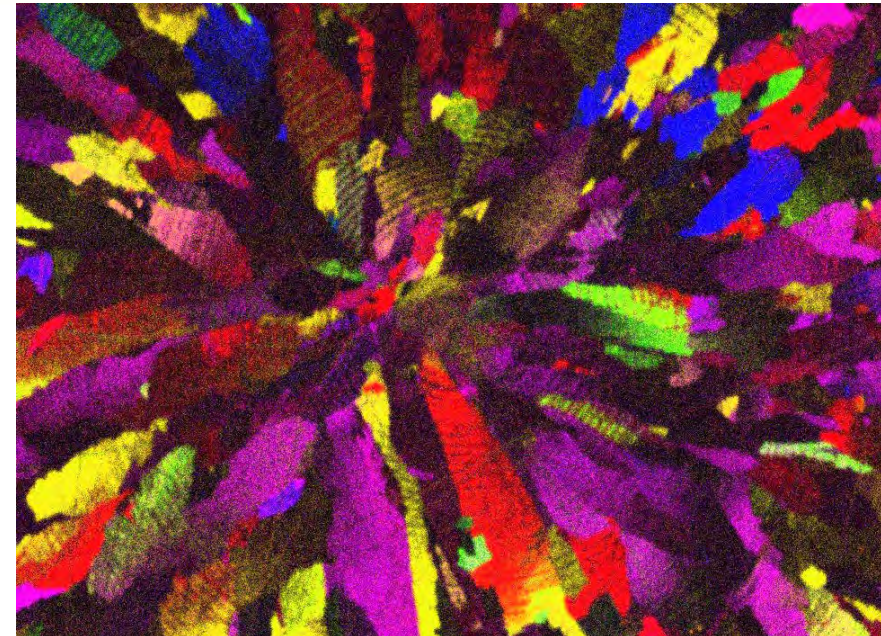
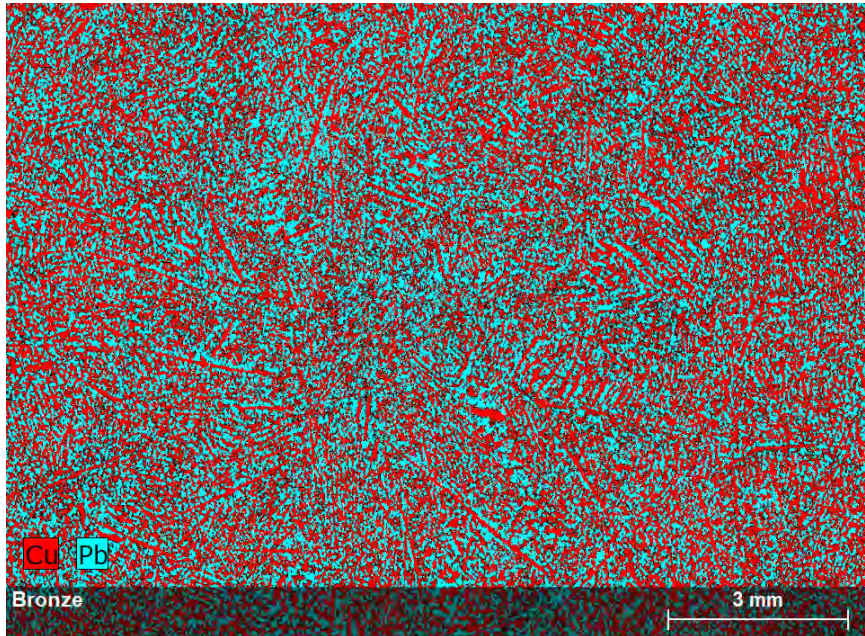
- Sum spectrum reveals additional peaks
- Identify unexpected elements
- Define 'free regions'



Here: the additional peaks are diffraction peaks, which reveal crystalline structure of the sample.

# PTS – Position Tagged Spectroscopy

all map data at hand



Here: the additional peaks are diffraction peaks, which reveal crystalline structure of the sample.

The data block contains all spectroscopic data and keeps it available for offline analysis.

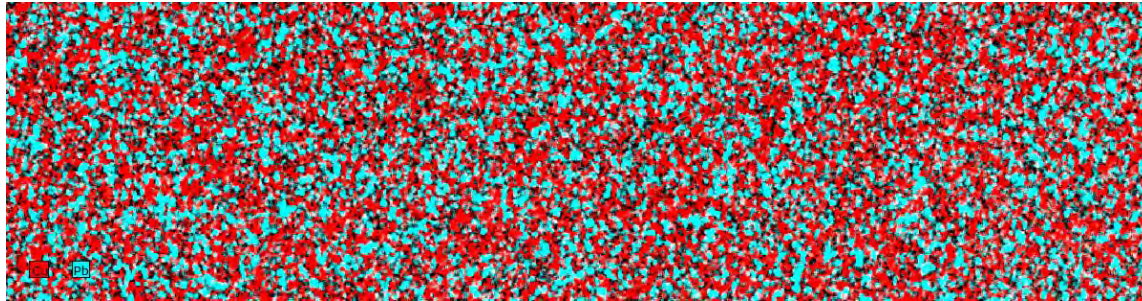
# M4 TORNADO Webinar Outline



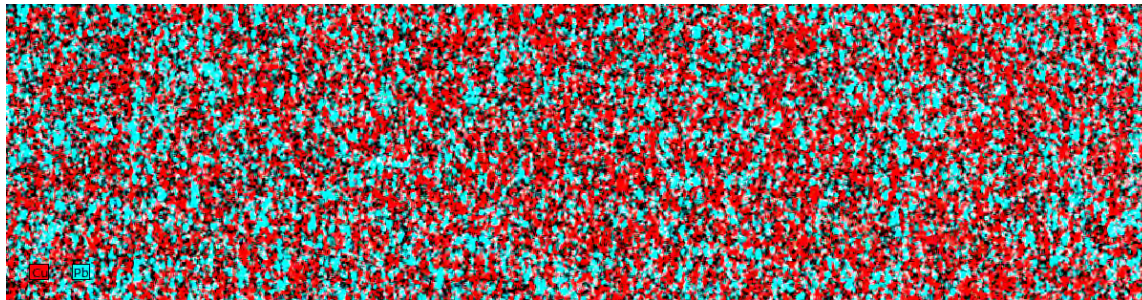
- Introduction
  - Presenters
  - The M4 instrument
  - Micro-XRF
  - Position Tagged Spectroscopy
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# Bronze

## Cu-Pb-distribution

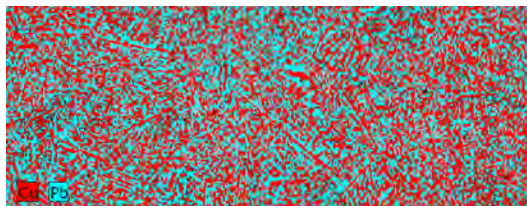


BAM  
ERM-EB 375  
Cu 58 %, Zn 39 % Pb 2.9 %

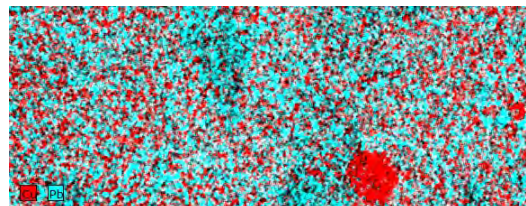


ARMI  
CDA 360 73B  
Cu 61.5 %, Zn 35.5 % Pb 1.9 %

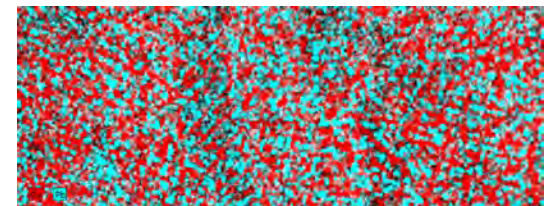
LGC Standards  
SUS RC36  
Cu 82.5 %, Zn 0.25 % Pb 9.6 %



ARMI  
CDA 932  
Cu 83.2 %, Zn 2.6 % Pb 6.8 %



ARMI  
CDA 397  
Cu 80.2 %, Zn 0.04 % Pb 9.2 %



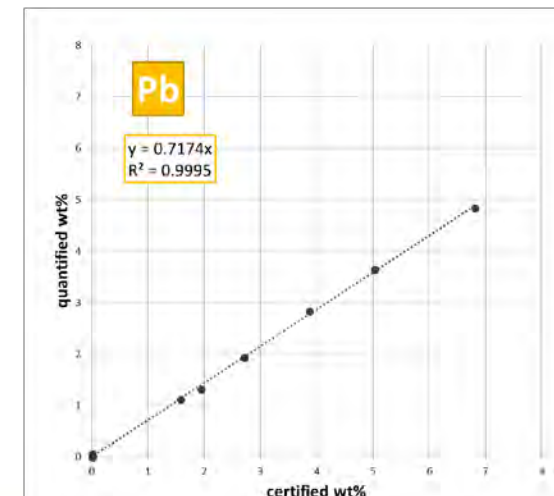
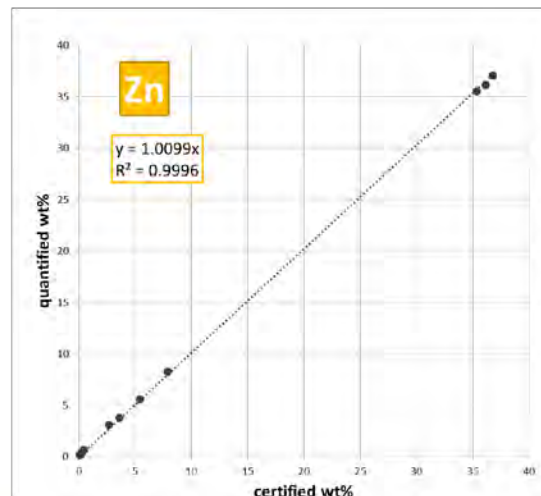
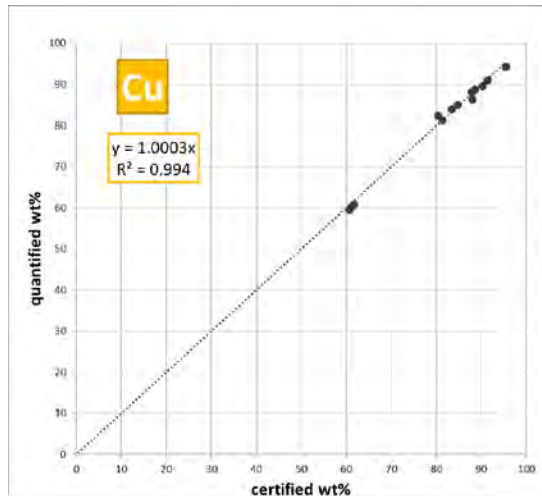


# Bronze

## FP-Quantification



Grade-IARM	Al	Co	Cr	Cu	Fe	Mn	Mo	Nb	Ni	P	Pb	Si	Sn	Ti	V	W	Zn	Zr
CDA314-72B	T	T	T	90.1	0.0	T	T	T	0.0	0.0	2.0	0.0	0.0	T	T	T	7.8	T
CDA360-73B	0.0010	0.0	T	61.5	0.2	0.0	T	T	0.1	0.0	2.7	0.0	0.2	T	T	T	35.3	T
CDA485-76B	0.0050	0.0	T	60.5	0.1	0.0	T	T	0.0	0.0	1.9	T	0.7	T	T	T	36.7	T
CDA510-77B	0.0010	T	T	95.2	0.0	0.0	T	T	0.0	0.1	0.0	0.0	4.7	T	T	T	0.0	T
CDA544-78B	0.0020	T	T	87.7	0.0	0.0	T	T	0.1	0.2	3.9	T	4.7	T	T	T	3.6	T
CDA623-79B	9.1900	0.0	0.0	88.4	2.1	0.2	T	T	0.1	0.0	0.0	0.0	0.0	T	T	T	0.0	T
CDA630-80B	10.1900	0.0	0.0	81.2	3.3	0.5	T	T	4.7	0.0	0.0	0.0	0.0	T	T	T	0.1	T
CDA642-81B	6.7000	T	0.0	91.2	0.0	0.0	T	T	0.0	0.0	0.0	1.8	0.0	T	T	T	0.2	T
CDA655-82B	0.0020	T	0.0	95.3	0.1	1.0	T	T	0.0	0.0	0.0	3.2	0.0	T	T	T	0.4	T
CDA706-84B	0.0020	0.0	0.0	87.9	1.3	0.6	T	T	10.0	0.0	0.0	0.0	0.0	T	T	T	0.1	T
CDA836-86C	0.0020	T	T	84.6	0.2	0.0	T	T	0.3	0.0	5.0	0.0	4.4	T	T	T	5.4	T
CDA857-87B	0.2000	0.0	0.0	60.9	0.3	0.0	T	T	T	0.0	1.6	0.0	0.8	T	T	T	36.1	T
CDA932-91C	0.0020	T	T	83.2	0.0	0.0	T	T	0.5	0.1	6.8	0.0	6.8	T	T	T	2.6	T
CDA937-BS937B-1	T	T	T	80.2	0.0	T	T	T	0.4	0.0	9.2	T	9.7	T	T	T	0.0	T



# Bronze

## FP-Quantification – Type calibration



**CONFIGURATION - SPECTRUM ELEMENTS**

**Elements**

Use spectrum elements  
 Use list elements  
 Search additional elements

Double click an element to open element editor Clear all

**Special properties of selected elements**

Compound	Fix %	Dec.	Diff.	Fact.
				1.00
Cu				0.92
Zn				0.91
Rh		<input checked="" type="checkbox"/>		1.00
Pb				1.37

**Global options**

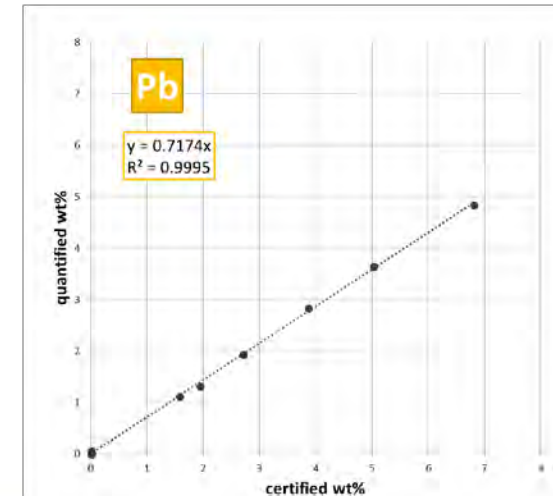
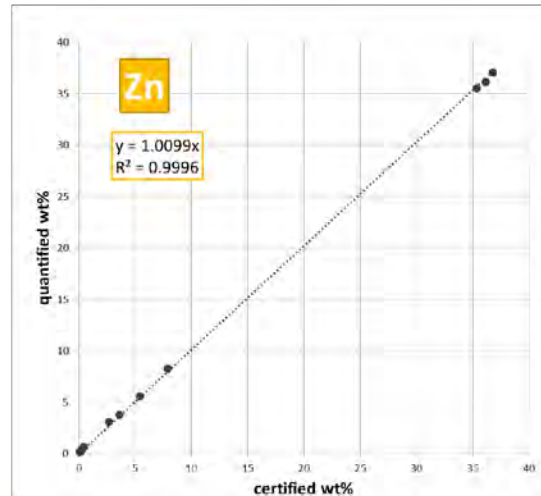
Background cycles:  Default  Manual 120

Minimum concentration: 0.00 %  NNLS

**Description**

Load... Save... OK Cancel

	Cu	Fe	Mn	Mo	Nb	Ni	P	Pb	Si	Sn	Ti	V	W	Zn	Zr
T	90.1	0.0		T	T	T	0.0	0.0	2.0	0.0	0.0	T	T	T	7.8
T	61.5	0.2	0.0	T	T	T	0.1	0.0	2.7	0.0	0.2	T	T	T	35.3
T	60.5	0.1	0.0	T	T	T	0.0	0.0	1.9	T	0.7	T	T	T	36.7
T	95.2	0.0	0.0	T	T	T	0.0	0.1	0.0	0.0	4.7	T	T	T	0.0
T	87.7	0.0	0.0	T	T	T	0.1	0.2	3.9	T	4.7	T	T	T	3.6
0.0	88.4	2.1	0.2	T	T	T	0.1	0.0	0.0	0.0	0.0	T	T	T	0.0
0.0	81.2	3.3	0.5	T	T	T	4.7	0.0	0.0	0.0	0.0	T	T	T	0.1
0.0	91.2	0.0	0.0	T	T	T	0.0	0.0	0.0	1.8	0.0	T	T	T	0.2
0.0	95.3	0.1	1.0	T	T	T	0.0	0.0	0.0	3.2	0.0	T	T	T	0.4
0.0	87.9	1.3	0.6	T	T	T	10.0	0.0	0.0	0.0	0.0	T	T	T	0.1
T	84.6	0.2	0.0	T	T	T	0.3	0.0	5.0	0.0	4.4	T	T	T	5.4
0.0	60.9	0.3	0.0	T	T	T	T	0.0	1.6	0.0	0.8	T	T	T	36.1
T	83.2	0.0	0.0	T	T	T	0.5	0.1	6.8	0.0	6.8	T	T	T	2.6
T	80.2	0.0		T	T	T	0.4	0.0	9.2		9.7	T	T	T	0.0



# Bronze

## FP-Quantification – Type calibration



**CONFIGURATION - SPECTRUM ELEMENTS**

**Elements**

Use spectrum elements  
 Use list elements  
 Search additional elements

Double click an element to open element editor Clear all

**Special properties of selected elements**

Compound	Fix %	Dec.	Diff.	Fact.
				1.00
Cu				0.92
Zn				0.91
Rh		<input checked="" type="checkbox"/>		1.00
Pb				1.37

**Global options**

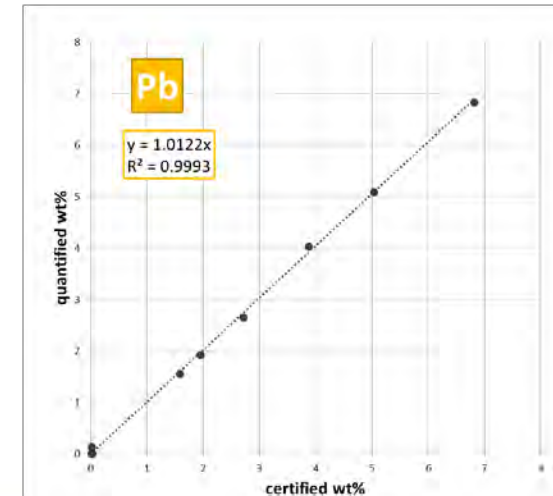
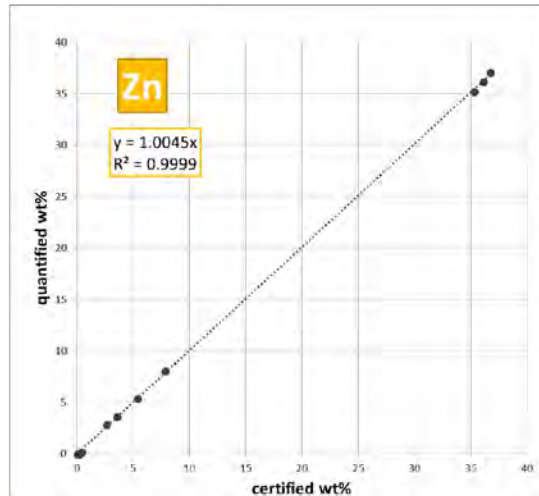
Background cycles:  Default  Manual 120

Minimum concentration: 0.00 %  NNLS

**Description**

Load... Save... OK Cancel

	Cu	Fe	Mn	Mo	Nb	Ni	P	Pb	Si	Sn	Ti	V	W	Zn	Zr
T	90.1	0.0		T	T	T	0.0	0.0	2.0	0.0	0.0	T	T	T	7.8
T	61.5	0.2	0.0	T	T	T	0.1	0.0	2.7	0.0	0.2	T	T	T	35.3
T	60.5	0.1	0.0	T	T	T	0.0	0.0	1.9	T	0.7	T	T	T	36.7
T	95.2	0.0	0.0	T	T	T	0.0	0.1	0.0	0.0	4.7	T	T	T	0.0
T	87.7	0.0	0.0	T	T	T	0.1	0.2	3.9	T	4.7	T	T	T	3.6
0.0	88.4	2.1	0.2	T	T	T	0.1	0.0	0.0	0.0	0.0	T	T	T	0.0
0.0	81.2	3.3	0.5	T	T	T	4.7	0.0	0.0	0.0	0.0	T	T	T	0.1
0.0	91.2	0.0	0.0	T	T	T	0.0	0.0	0.0	1.8	0.0	T	T	T	0.2
0.0	95.3	0.1	1.0	T	T	T	0.0	0.0	0.0	3.2	0.0	T	T	T	0.4
0.0	87.9	1.3	0.6	T	T	T	10.0	0.0	0.0	0.0	0.0	T	T	T	0.1
0.0	84.6	0.2	0.0	T	T	T	0.3	0.0	5.0	0.0	4.4	T	T	T	5.4
0.0	60.9	0.3	0.0	T	T	T	0.0	0.0	1.6	0.0	0.8	T	T	T	36.1
T	83.2	0.0	0.0	T	T	T	0.5	0.1	6.8	0.0	6.8	T	T	T	2.6
T	80.2	0.0		T	T	T	0.4	0.0	9.2		9.7	T	T	T	0.0



# Bronze

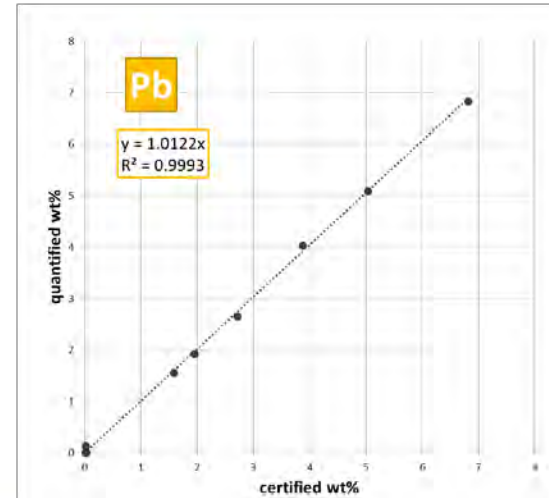
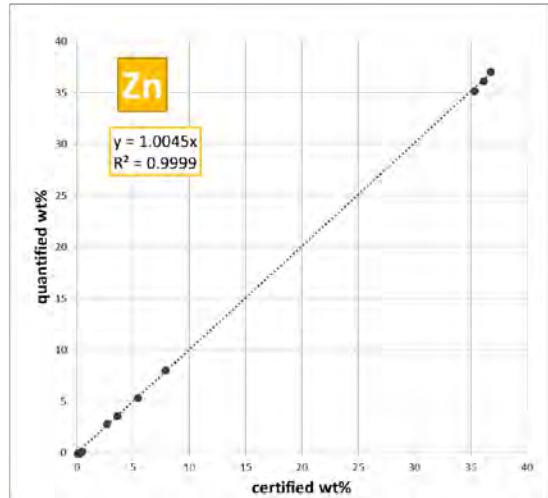
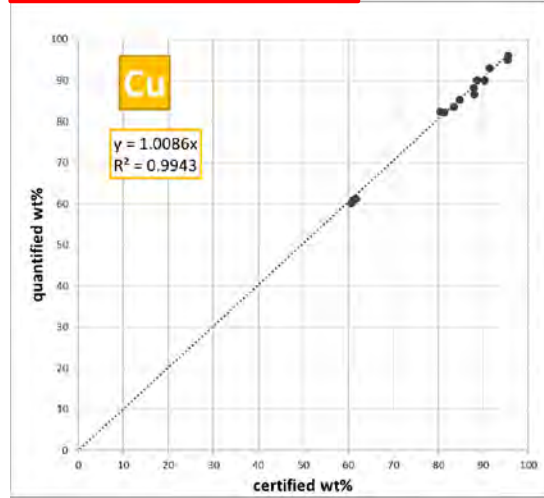
## FP-Quantification – Type calibration



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CDA314-72B	T	T	T	90.1	0.0	T	T	T	0.0	0.0	2.0	0.0	0.0	T	T	T	7.8	T
CDA360-73B	0.0010	0.0	T	61.5	0.2	0.0	T	T	0.1	0.0	2.7	0.0	0.2	T	T	T	35.3	T
CDA485-76B	0.0050	0.0	T	60.5	0.1	0.0	T	T	0.0	0.0	1.9	T	0.7	T	T	T	36.7	T
CDA510-77B	0.0010	T	T	95.2	0.0	0.0	T	T	0.0	0.1	0.0	0.0	4.7	T	T	T	0.0	T
CDA544-78B	0.0020	T	T	87.7	0.0	0.0	T	T	0.1	0.2	3.9	T	4.7	T	T	T	3.6	T
CDA623-79B	9.1900	0.0	0.0	88.4	2.1	0.2	T	T	0.1	0.0	0.0	0.0	0.0	T	T	T	0.0	T
CDA630-80B	10.1900	0.0	0.0	81.2	3.3	0.5	T	T	4.7	0.0	0.0	0.0	0.0	T	T	T	0.1	T
CDA642-81B	6.7000	T	0.0	91.2	0.0	0.0	T	T	0.0	0.0	0.0	1.8	0.0	T	T	T	0.2	T
CDA655-82B	0.0020	T	0.0	95.3	0.1	1.0	T	T	0.0	0.0	0.0	3.2	0.0	T	T	T	0.4	T
CDA706-84B	0.0020	0.0	0.0	87.9	1.3	0.6	T	T	10.0	0.0	0.0	0.0	0.0	T	T	T	0.1	T
CDA836-86C	0.0020	T	T	84.6	0.2	0.0	T	T	0.3	0.0	5.0	0.0	4.4	T	T	T	5.4	T
CDA857-87B	0.2000	0.0	0.0	60.9	0.3	0.0	T	T	T	0.0	1.6	0.0	0.8	T	T	T	36.1	T
CDA932-91C	0.0020	T	T	83.2	0.0	0.0	T	T	0.5	0.1	6.8	0.0	6.8	T	T	T	2.6	T
CDA937-BS937B-1	T	T	T	80.2	0.0	T	T	T	0.4	0.0	9.2	T	9.7	T	T	T	0.0	T

Compound	Fix %	Dec. Diff.	Fact.
Cu			1.00
Zn			0.92
Rh		✓	1.00
Pb			1.37

- Compound
- Stoichiom. elements
- Fix concentration
- Deconvolution only
- Excluded element
- Difference element



# Fe-alloy

## Overview and Measurement Conditions



### Mapping parameters

Width:	3139 pixel
	78.48 mm
Height:	1473 pixel
	36.825 mm
Pixel Size:	25 $\mu\text{m}$
Total number of pixel:	4623747 pixel

### Acquisition parameters

Frame count:	1
Pixel time:	3 ms/pixel
Measure time:	1:56 h
Overall time:	6:20 h
Stage speed:	8.3 mm/s
Stage position (X,Y,Z):	

### Tube parameter

High voltage:	50 kV
Anode current:	198 $\mu\text{A}$
Filter:	Empty
Optic:	Lens
SpotSize:	25
Chamber at:	Air 20 mbar
Anode:	Rh

### Detector parameters

Selected detectors:	
Max. pulse throughput:	130000 cps

Mapped area: 78.5 mm x 36.8 mm

Pixel size: 25  $\mu\text{m}$

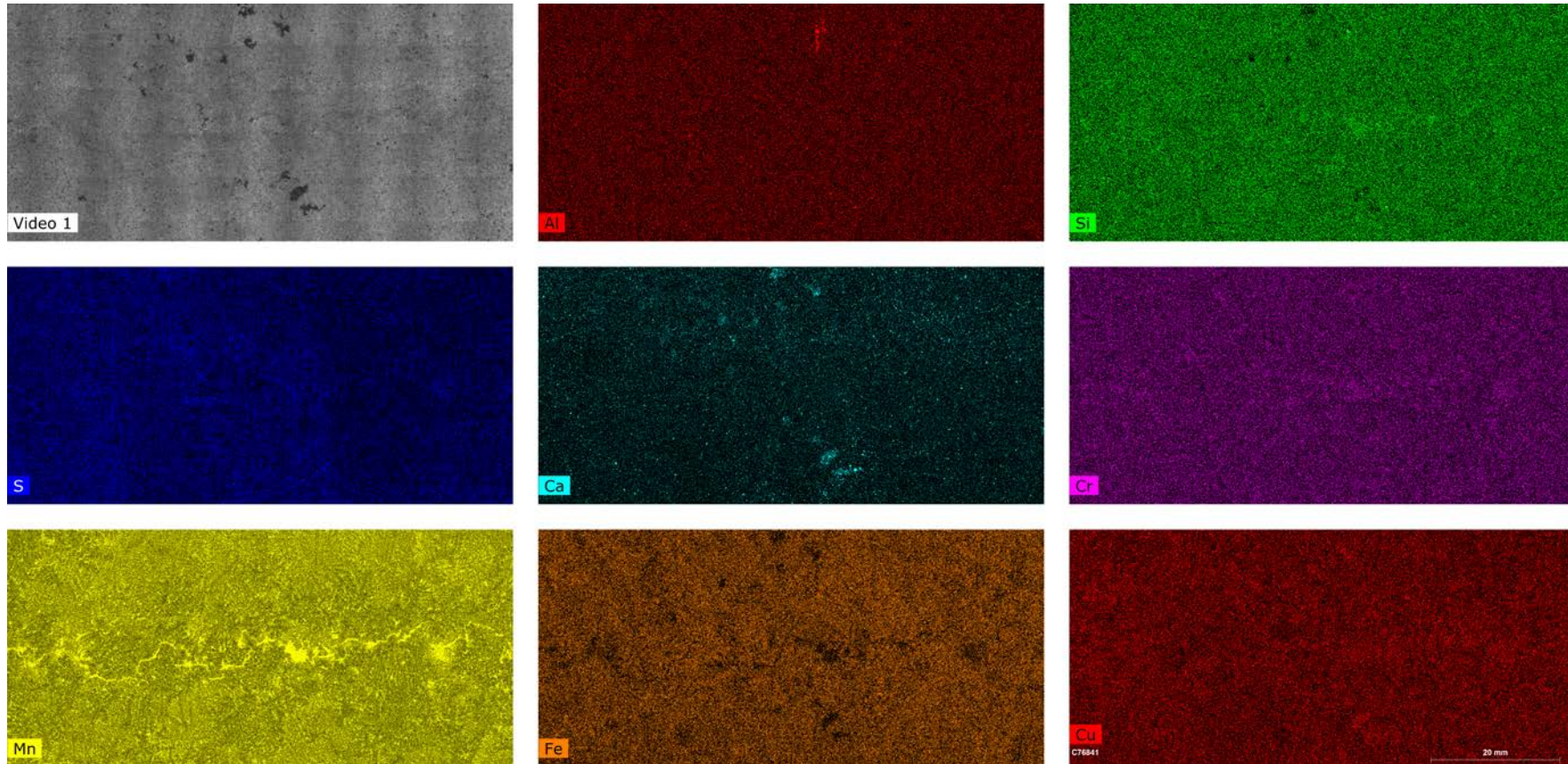
Dwell time: 3 ms/pixel

Overall: 4.6 Mpixel, 6:20 h.

# Fe-alloy Element Distributions



Net intensity distributions for the identified elements.

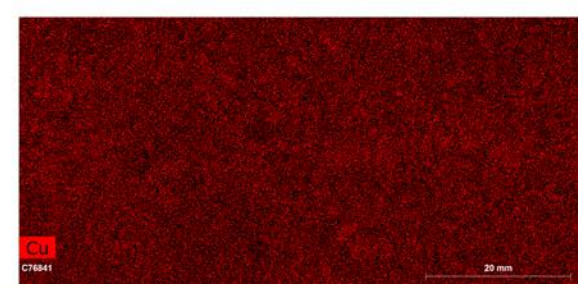
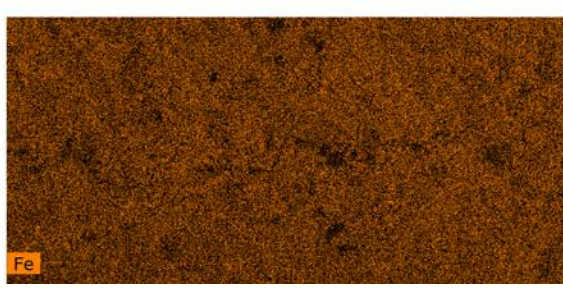
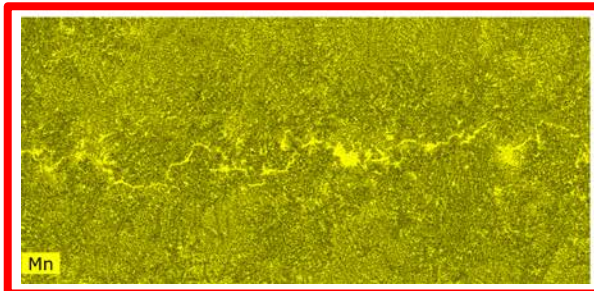
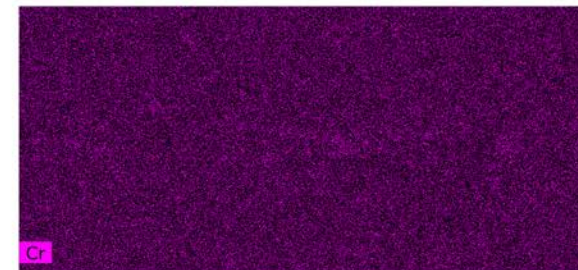
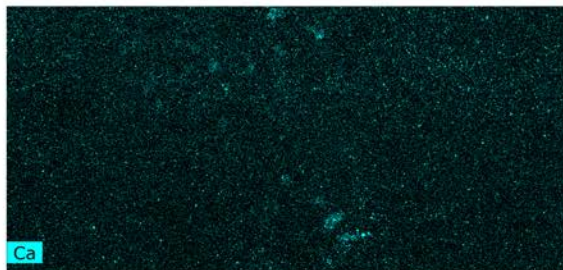
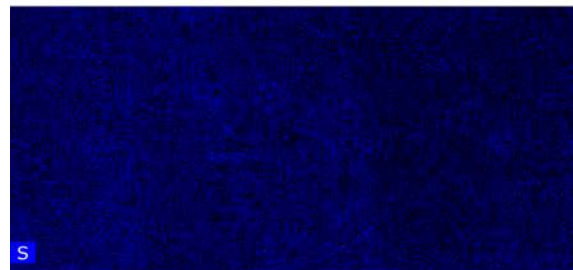
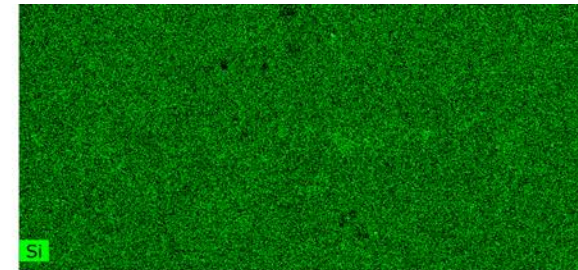
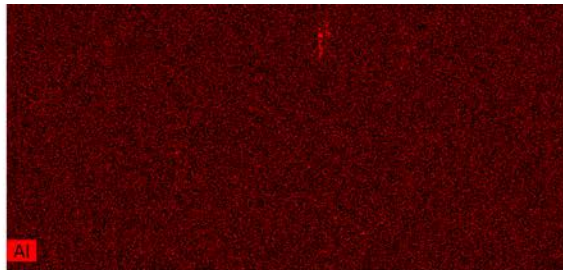
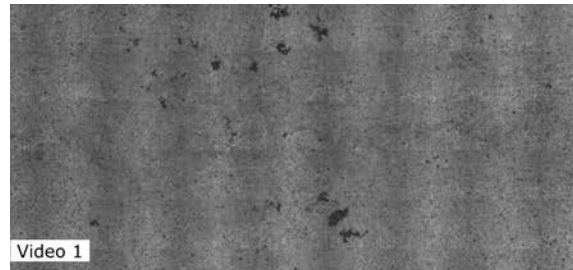


# Fe-alloy Element Distributions



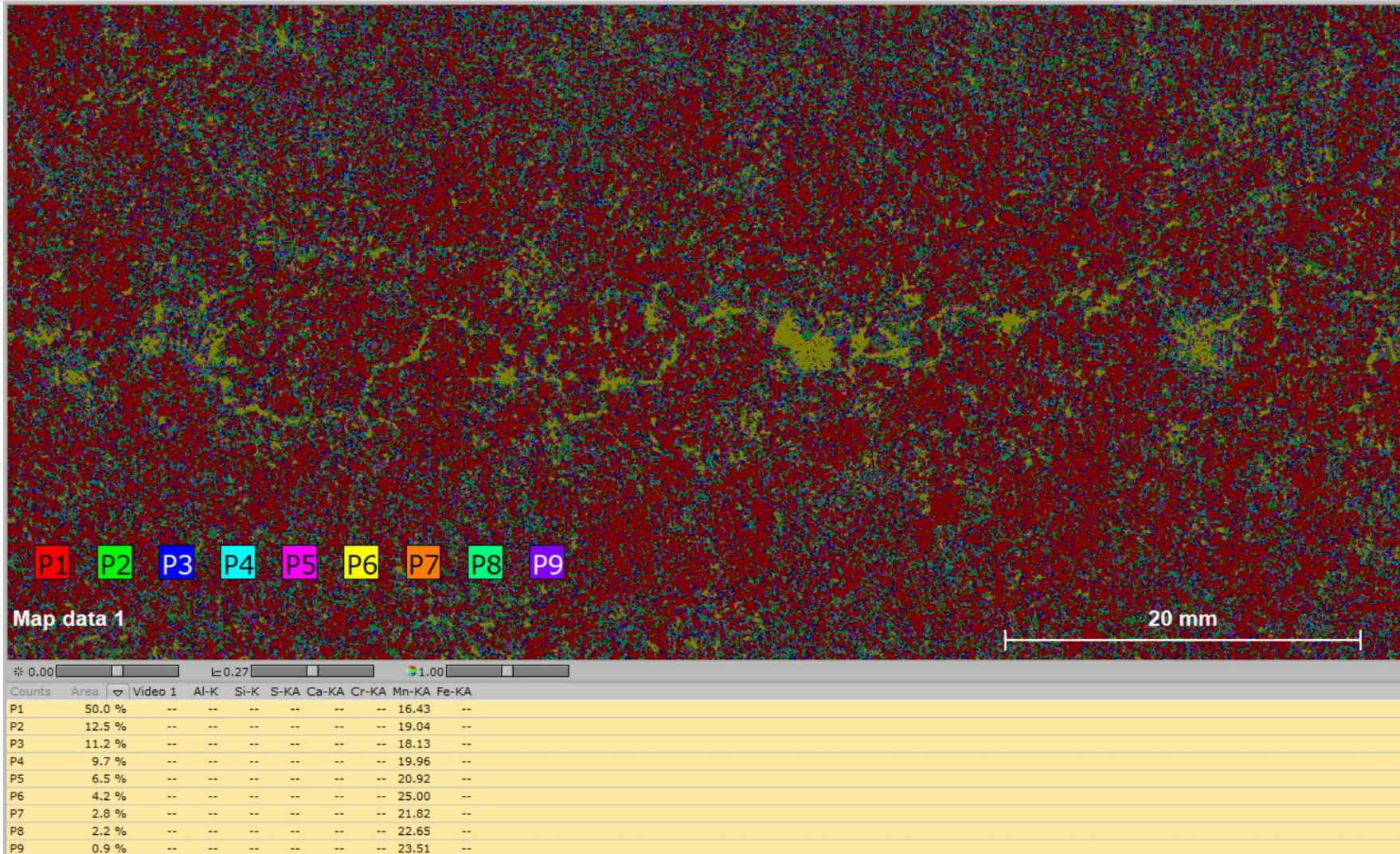
Net intensity distributions for the identified elements.

Mn shows an interesting 'horizon'.



# Fe-alloy

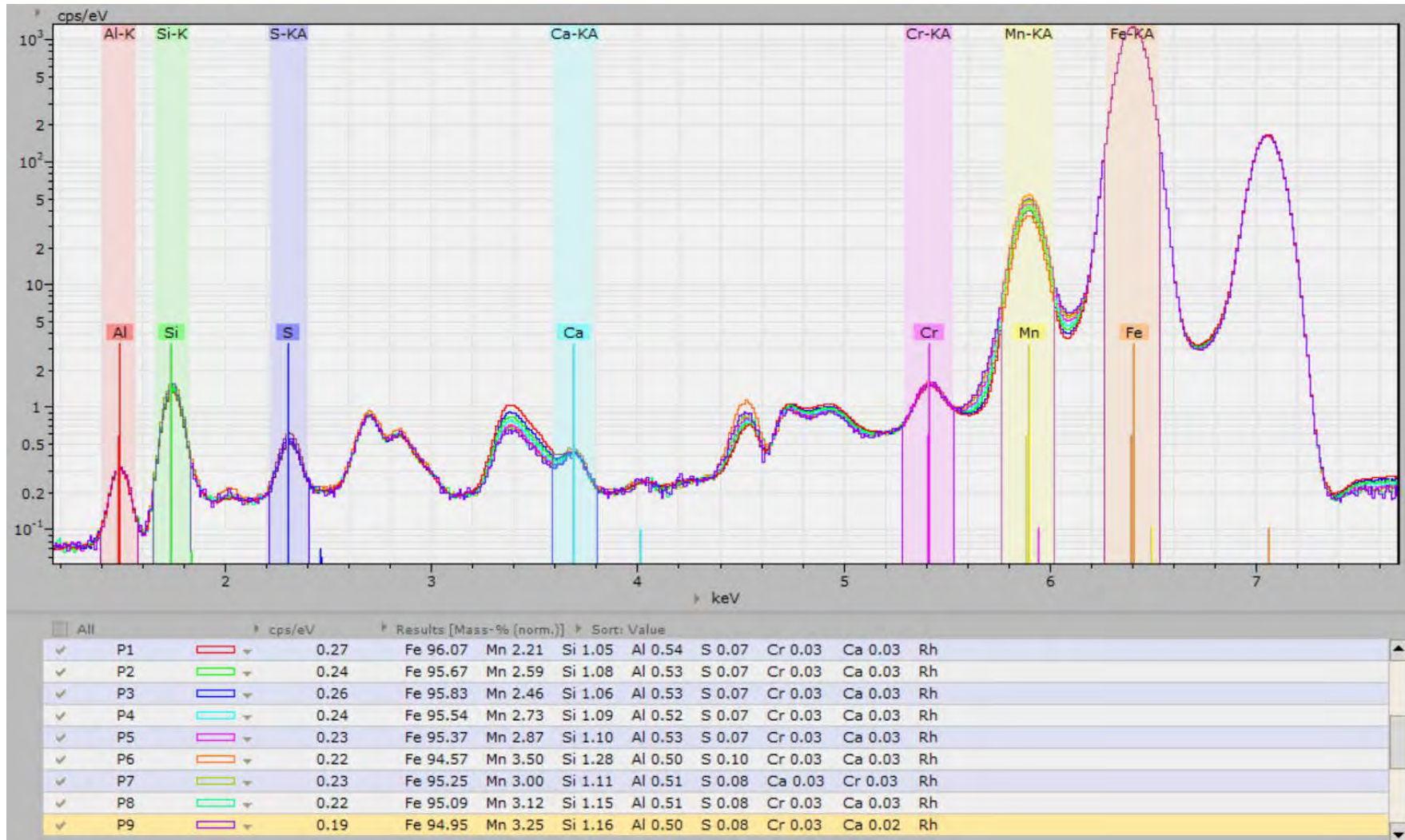
## Mn distribution – phase analysis





# Fe-alloy

## Mn distribution – phase spectra



# M4 TORNADO Webinar Outline

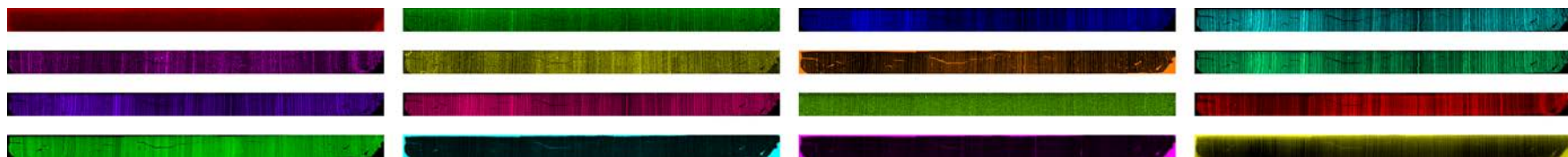


- Introduction
  - Presenters
  - The M4 instrument
  - Micro-XRF
  - Position Tagged Spectroscopy
- Homogeneous samples? – Cu- and Fe-alloys
- Heterogeneous in 1 dimension – drill core
- Heterogeneous – concrete
- Live part – data extraction and evaluation
- Summary

# Drill core Lake sediment

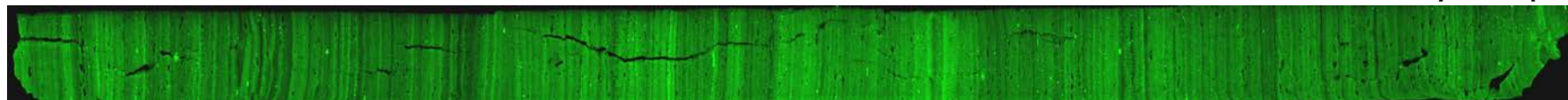


Na Mg Al Si P S Cl  
K Ca Sc Ti V Cr Mn Fe Co Ni Cu Zn Ga Ge As Se Br



Fe

Intensity map



Si



K

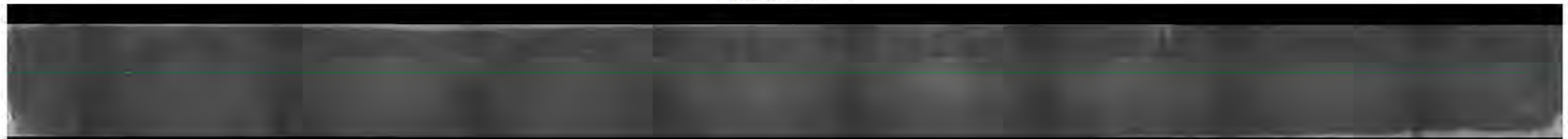
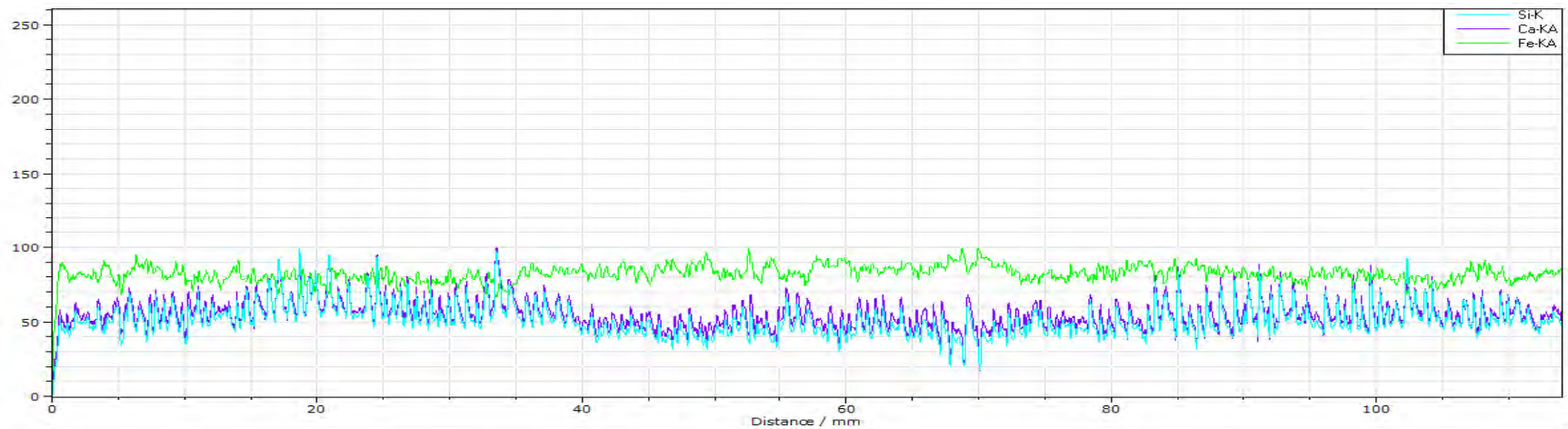


Ca



# Drill core

## Line object

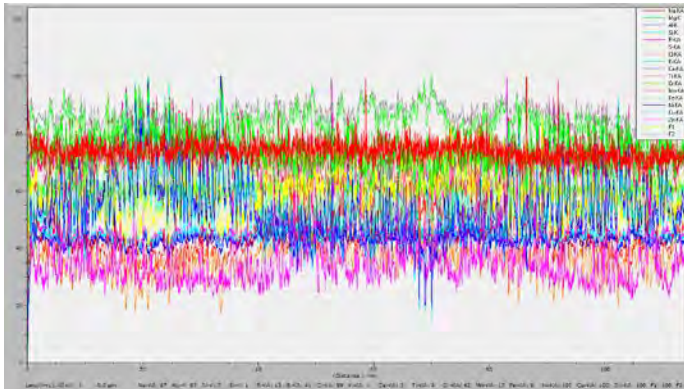


A line can be drawn over the map and the line can be widened to improve statistics. Selected elements can then be plotted directly in the software. For additional plot options the data can be extracted to make it accessible for other plot software.

# Drill core Exporting data



The line extracted from the map can be quantified to obtain the net intensity in the line work space



Quantification can be done using the Oxide method

Configuration - Oxides

Elements

Use spectrum elements  
 Use list elements  
 Search additional elements

Double click an element to open element editor Clear all

Special properties of selected elements

Compound	Fix %	Dec.	DJF	Fact.
C	CO <sub>2</sub>			1.00
Na	Na <sub>2</sub> O			1.00
Mg	MgO			1.00
Al	Al <sub>2</sub> O <sub>3</sub>			1.00
Si	SiO <sub>2</sub>			1.00
P	P <sub>2</sub> O <sub>5</sub>			1.00
S	SO <sub>3</sub>			1.00
K	K <sub>2</sub> O			1.00
Ca	CaO			1.00
Ti	TiO <sub>2</sub>			1.00
Cr	Cr <sub>2</sub> O <sub>3</sub>			1.00
Mn	MnO			1.00
Fe	Fe <sub>2</sub> O <sub>3</sub>			1.00
Ni	NiO			1.00
Zn	ZnO			1.00
As	As <sub>2</sub> O <sub>3</sub>			1.00

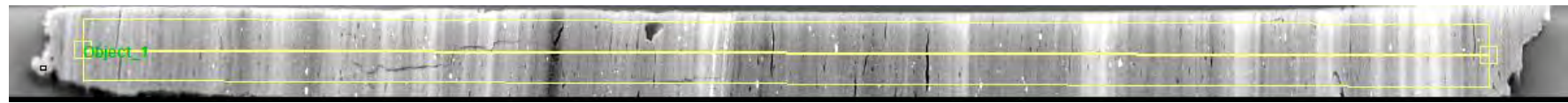
Legend

- Fixed list
- Compound
- Stoichiometric elements
- Fix concentration
- Deconvolution only
- Excluded element
- Difference element

Quantification results (wt.-%, net intensities) can be exported to excel

Spectrum	O	Na	Mg	Al	Si	P	S	Cl	K	Ca	Ti	Cr
Point 9	0.00	0.00	0.00	16.00	194.00	0.00	0.00	27393.00	0.00	262.00	75.00	16.00
Point 1	0.00	13.00	0.00	20.00	154.00	0.00	0.00	26134.00	21.00	336.00	156.00	0.00
Point 2	0.00	0.00	4.00	35.00	413.00	0.00	0.00	24117.00	385.00	727.00	573.00	1.00
Point 3	0.00	3.00	0.00	119.00	1129.00	0.00	0.00	26148.00	528.00	1129.00	999.00	5.00
Point 4	0.00	0.00	67.00	236.00	2207.00	0.00	0.00	24473.00	1080.00	1545.00	1470.00	99.00
Point 5	0.00	27.00	0.00	425.00	4499.00	0.00	0.00	24251.00	1916.00	2668.00	2436.00	0.00
Point 6	0.00	21.00	18.00	824.00	7769.00	0.00	54.00	22713.00	3366.00	4200.00	3793.00	133.00
Point 7	0.00	0.00	2.00	6153.00	9953.00	38.00	0.00	19702.00	4292.00	4820.00	3863.00	273.00
Point 8	0.00	17.00	24.00	1129.00	11794.00	123.00	0.00	20188.00	4993.00	8208.00	4788.00	130.00
Point 9	0.00	8.00	89.00	1296.00	12191.00	77.00	59.00	20363.00	4843.00	5473.00	4933.00	297.00
Point 10	0.00	0.00	12.00	1291.00	12999.00	69.00	32.00	19203.00	5294.00	5923.00	5130.00	136.00
Point 12	0.00	0.00	0.00	1676.00	14743.00	72.00	73.00	18159.00	6474.00	6934.00	6177.00	176.00
Point 13	0.00	10.00	13.00	2057.00	16630.00	153.00	78.00	17042.00	7228.00	7692.00	6911.00	159.00
Point 14	0.00	0.00	33.00	2063.00	17169.00	258.00	122.00	15844.00	7478.00	8219.00	7093.00	328.00
Point 16	0.00	0.00	38.00	2079.00	18657.00	172.00	181.00	13090.00	8541.00	9277.00	8273.00	458.00
Point 19	0.00	44.00	63.00	2241.00	20642.00	126.00	194.00	13991.00	9232.00	9232.00	8406.00	500.00
Point 17	0.00	19.00	116.00	2396.00	22154.00	236.00	331.00	12286.00	9718.00	10073.00	8766.00	949.00
Point 18	0.00	4.00	80.00	2516.00	28989.00	171.00	295.00	10793.00	11296.00	11731.00	10413.00	462.00
Point 19	0.00	13.00	196.00	3248.00	32391.00	124.00	249.00	9479.00	11191.00	12973.00	11191.00	708.00
Point 20	0.00	0.00	150.00	3582.00	32133.00	203.00	285.00	8978.00	13810.00	13320.00	11675.00	943.00
Point 21	0.00	0.00	103.00	3467.00	30919.00	284.00	298.00	8406.00	13252.00	12755.00	11334.00	458.00
Point 22	0.00	59.00	153.00	3303.00	29116.00	380.00	292.00	8379.00	12677.00	12021.00	10440.00	454.00
Point 23	0.00	14.00	155.00	3239.00	29005.00	447.00	316.00	9034.00	12410.00	11716.00	10633.00	477.00
Point 24	0.00	0.00	154.00	3056.00	26094.00	307.00	323.00	9177.00	11404.00	10970.00	9953.00	374.00
Point 25	0.00	10.00	137.00	3114.00	27791.00	368.00	225.00	9880.00	11961.00	11604.00	10714.00	512.00

# Drill core Plot vs. Image



- Line scan data
- Add to project
- Save...
- Print...
- Add to report
- Graphic
- Copy
- Save...
- Twain export

**CONFIRM**

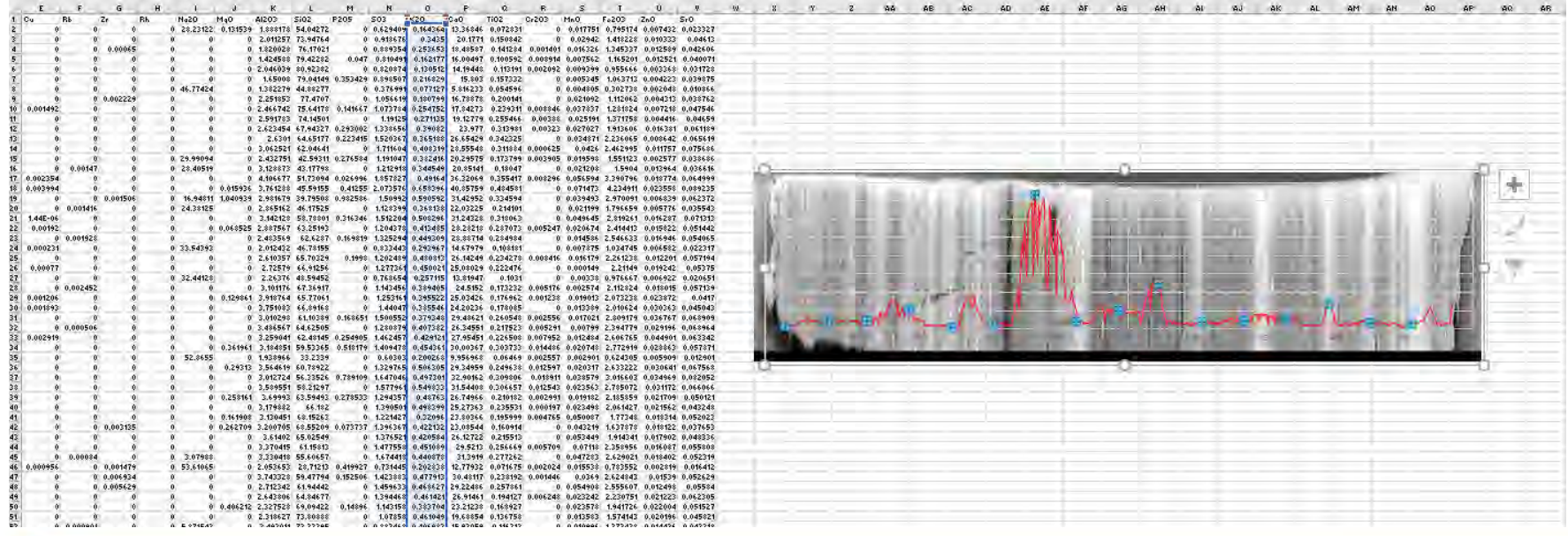
? Do you want to save point spectra too?

Yes  No

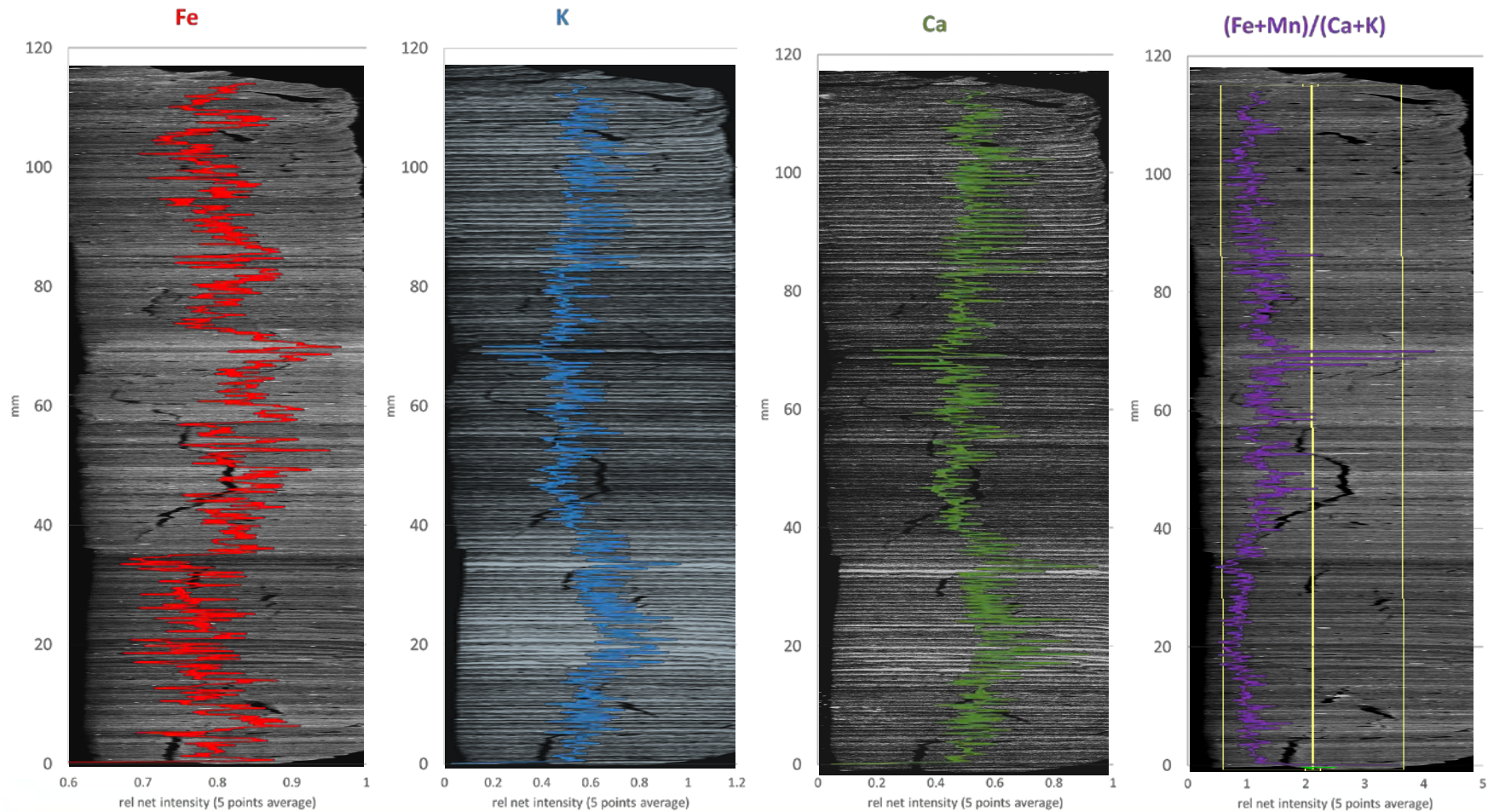
Quantify  
Oxides

Results Profiles Spectrum

- Add to project
- Copy
- Save...
- Print...
- Add to report
- Twain export



# Drill core Plot vs. Image



# M4 TORNADO Webinar

## Outline



- Introduction
  - Presenters
  - The M4 instrument
  - Micro-XRF
  - Position Tagged Spectroscopy
- Homogeneous samples? – Cu- and Fe-alloys
- Heterogeneous in 1 dimension – drill core
- Heterogeneous – concrete
- Live part – data extraction and evaluation
- Summary



# Concrete

## A heterogeneous sample



- 4 h scan on a region from left to right (thin green frame)
- gradient in Cl distribution (orange) clearly visible

Map information

**Mapping parameter**

Width:	2820 pixel
	56,4 mm
Height:	540 pixel
	10,8 mm
Pixel Size:	20 $\mu\text{m}$
Total number of pixel:	1522800 pixel

**Acquisition parameter**

Frame count:	1
Pixel time:	10 ms/pixel
Measure time:	4:13 h
Overall time:	4:48 h

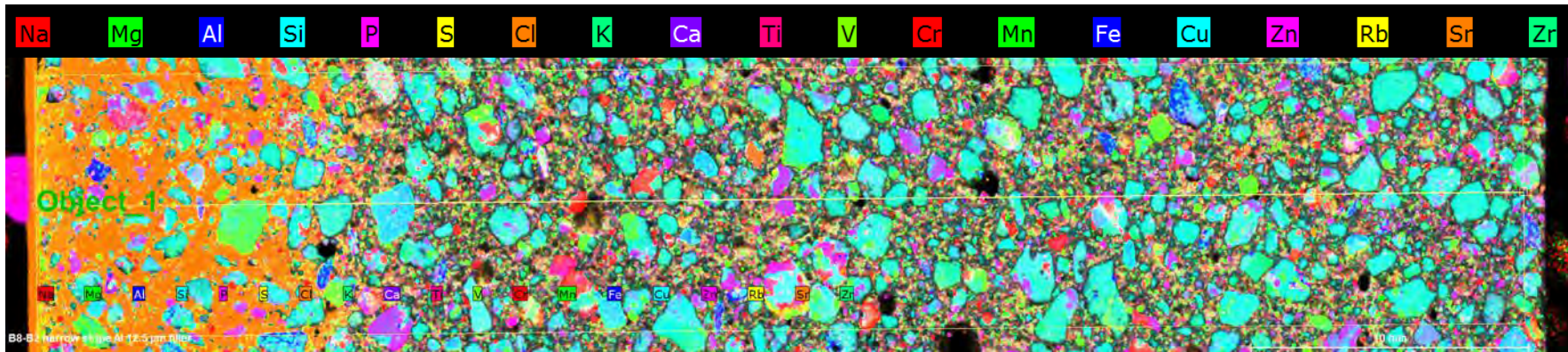
**Tube parameter**

High voltage:	50 kV
Anode current:	600 $\mu\text{A}$
Filter:	Empty
Optic:	Lens
Chamber at:	Vacuum 20,1 mbar
Anode:	Rh

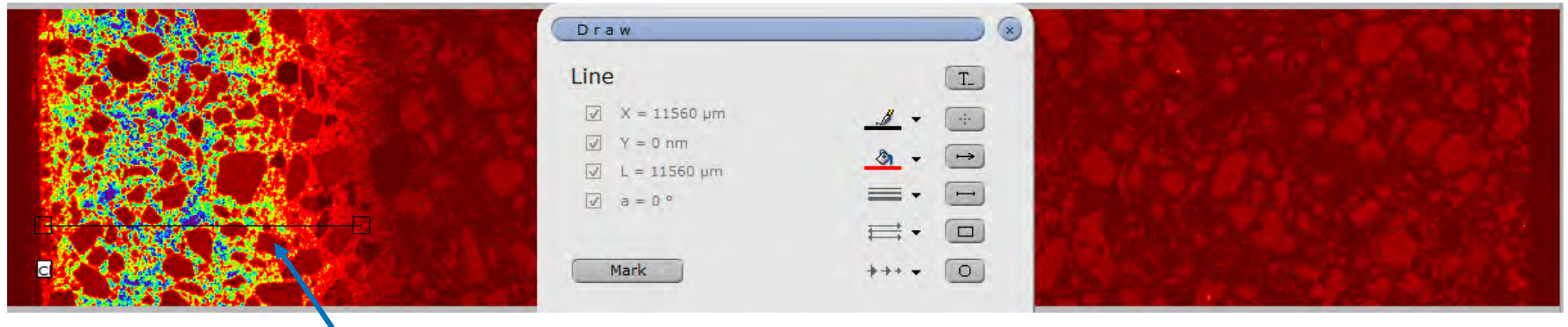
**Detector parameter**

Selected detectors:	1,2
---------------------	-----

Close



# Concrete Cl gradient in cement



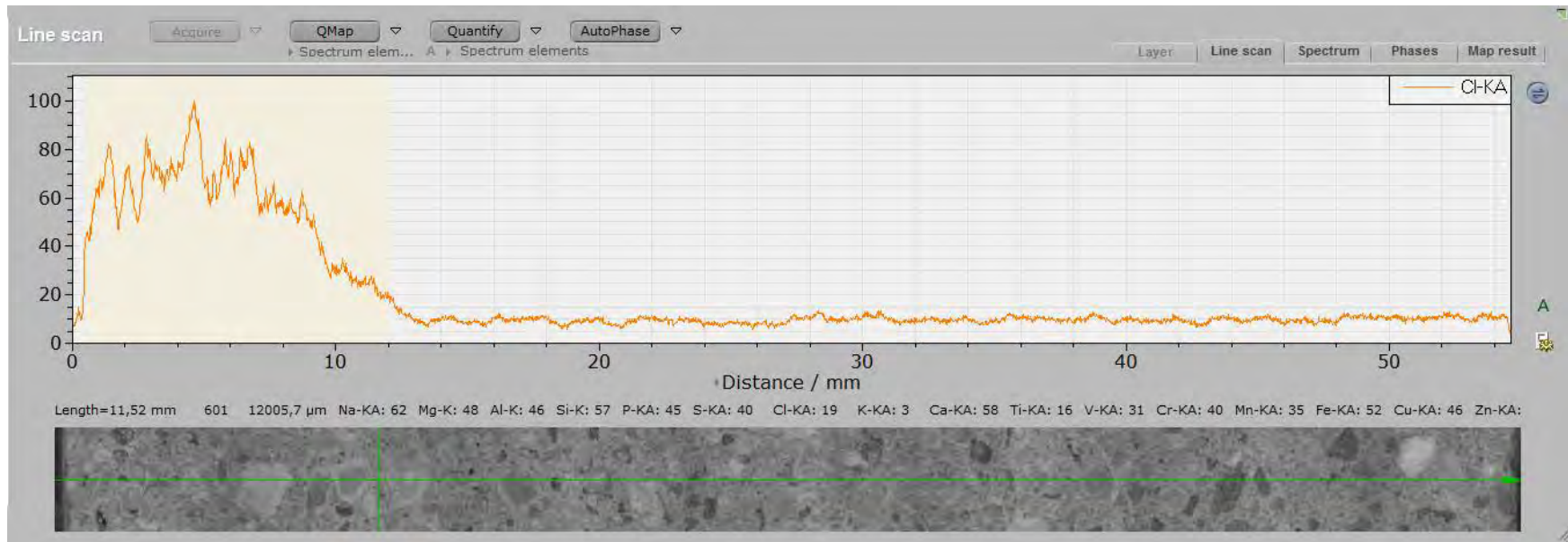
False color representation of the chlorine concentration

Obvious gradient

With the drawing tool a **line** has been drawn which is shown to be 11.5 mm long which gives an indication of the depth of Cl diffusion

# Concrete

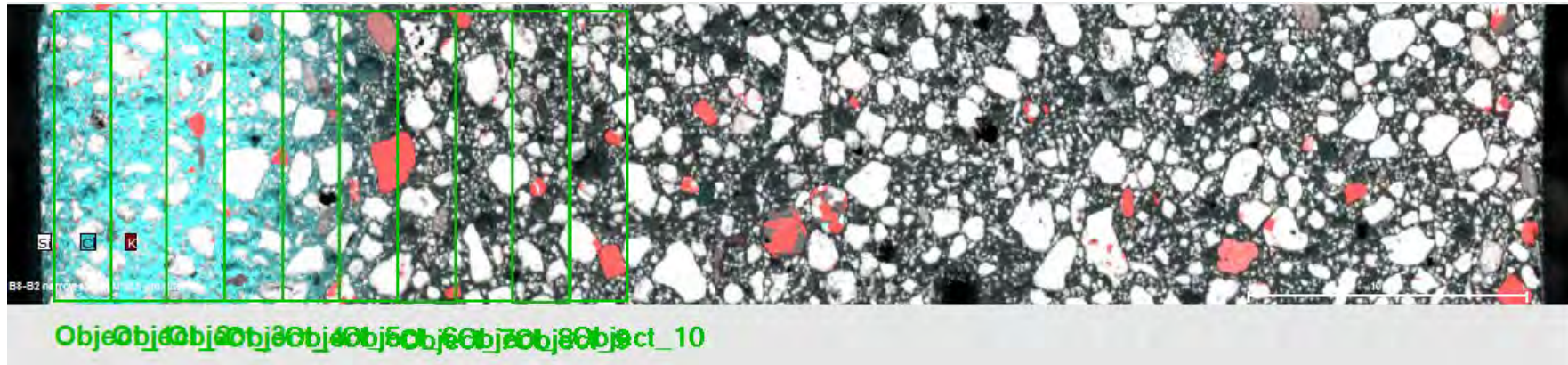
## Cl depth from line scan



A line object can be drawn into the the Hypermap data. Therewith a line profile along the sample can be generated.  
The Cl signal is increased in an area of which again is found to be  $\sim 11.5$  cm in length

# Concrete

... a more quantitative approach: objects



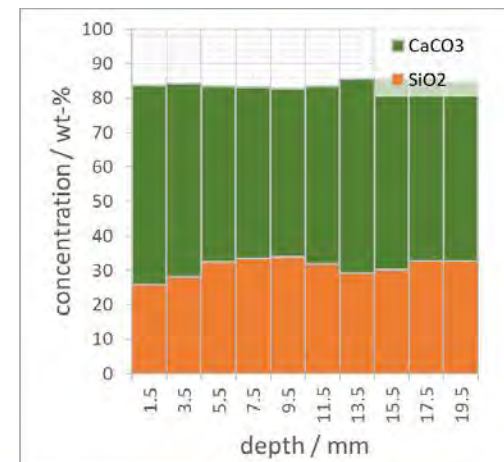
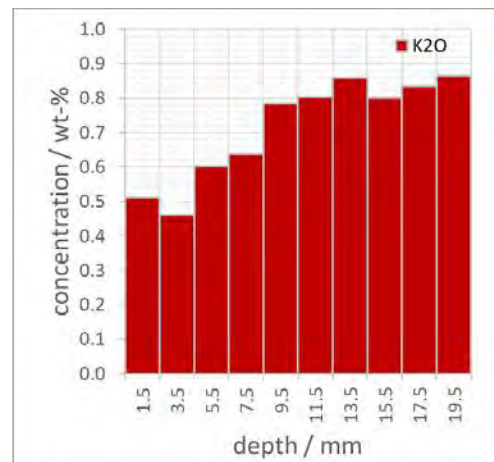
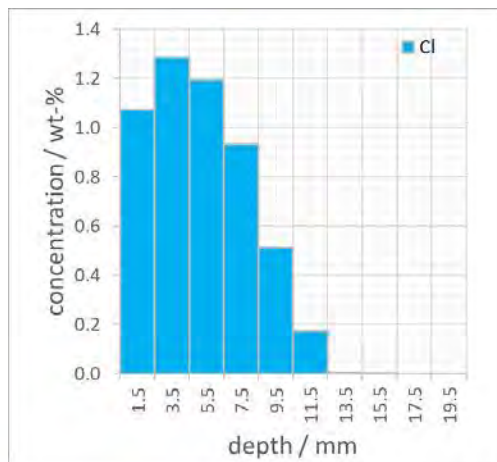
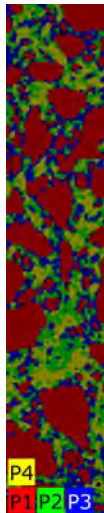
Draw objects into data cube and get their dimensions using the "draw tools"



# Concrete phase analysis and concentrations



- Load each object
- Select only a signature element of the cement (here P)
- Press auto phase
- Select the cement phase
- Create sum spectrum
- Move the sum spectrum to the project (or save to disk)
- Repeat for all objects
- Import all phase sum spectra into point mode
- Quantify with appropriate method
- Export results table to Excel → plot



# M4 TORNADO Webinar Outline



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  - Micro-XRF
  - Position Tagged Spectroscopy
- Homogeneous samples? – Cu- and Fe-alloys
- Heterogeneous in 1 dimension – drill core
- Heterogeneous – concrete
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# Some data mining Live



# Summary

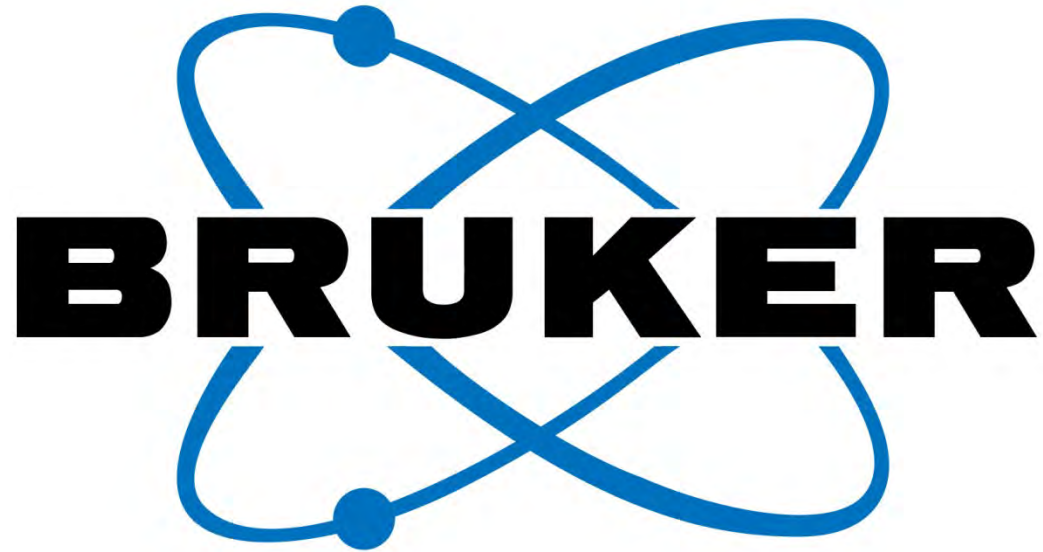


- Micro-XRF helps you understand (the inhomogeneity of) your sample
- The position tagged spectroscopy (HyperMap) allows to access all spectral information at a later time
- Any spectrum in the software can be quantified (but often shouldn't)
- To get a results, at first clarify the actual analytical question
- Find the correct measurement conditions
- Multiple approaches to useful results are possible for inhomogeneous samples
  - Use net peak areas as their significance is less affected by matrix effects
  - Use phase analysis to identify „homogeneous“ parts of a sample
  - Use data export for advanced results arithmetics
  - For quantification a type calibration usually corrects for sample effects
- Find an adequate results visualization (ideally supported by numbers)



## Are There Any Questions?

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



Innovation with Integrity