

The M6 JETSTREAM: State of the Art



Bruker Nano Analytics, Berlin, Germany
Webinar, November 9, 2017

XFlash[®] Technology

Micro-XRF

Results

Series	Net	Primary energy		Tilt angle		Atom C. Error	
		[wt.%]	[wt.%]	[wt.%]	[wt.%]	[wt.%]	[wt.%]
Iron	K series 214751713	94,59	93,62	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	0,08	0,08	0,24	0,00		
Chromium	K series 99229	0,03	0,03	0,03	0,00		
Total	100,82	100,00	100,00				

M6 JETSTREAM Webinar

Outline



- Introduction

 - Presenters

 - The M6 Jetstream

 - Micro-XRF in art and conservation

- Producing the data

 - Measurement parameters and options

- Evaluating the data "DATA MINING"

 - ROI

 - fast deconvolution

 - convolution or forward calculation

 - ROI mathematics

 - image processing

- Live part

- Summary

M6 JETSTREAM Webinar

Presenters / Moderators



Falk Reinhardt

Application Scientist,
Bruker Nano Analytics, Berlin, Germany



Dr. Roald Tagle

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

M6 Jetstream micro-XRF spectrometer

Standard configuration



Instrument parameters

30 W micro-focus Rh tube with polycapillary lens
for excitation spot sizes from 100 to 500 μm
(for Mo-K α)

30 mm² silicon drift detector (SDD)
with energy resolution < 145 eV (for Mn-K α)
Option: 60 mm² detector

Sample stage with measurable area in one run
up to of **800 mm x 600 mm**

Sample stage speed up to **100 mm/s**, and 200 mm/s²

Optional **Helium flush** with flow controller



M6 Jetstream micro-XRF spectrometer

Instrument operation

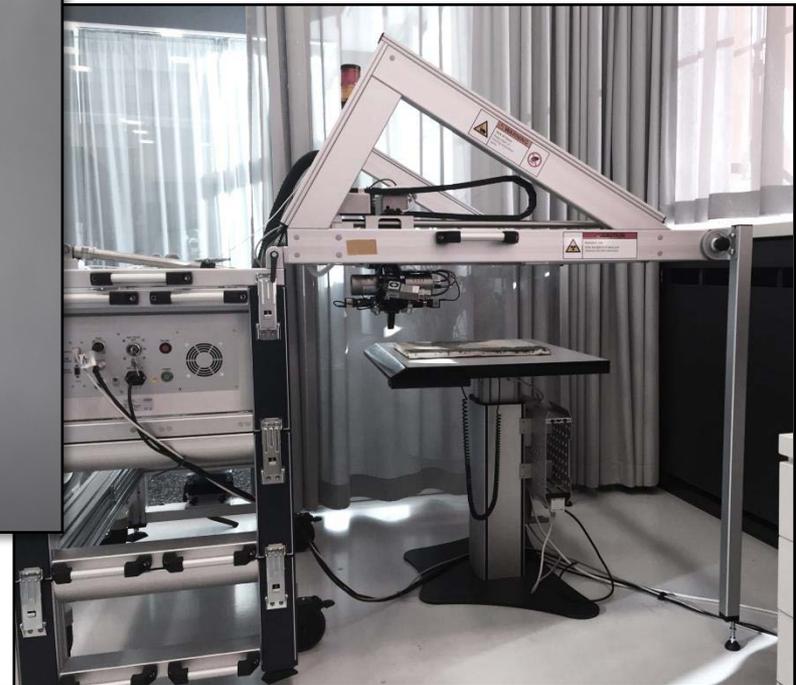


Vertical



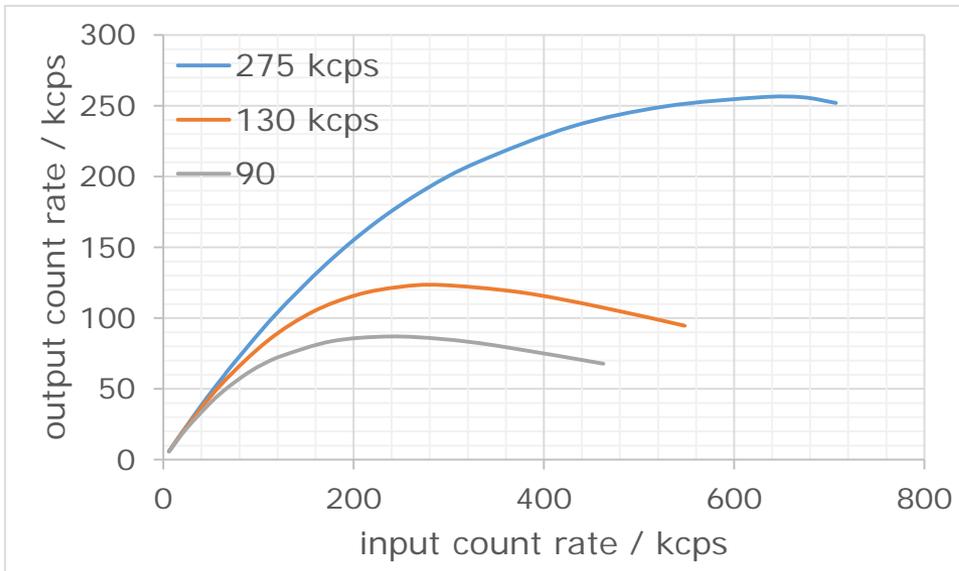
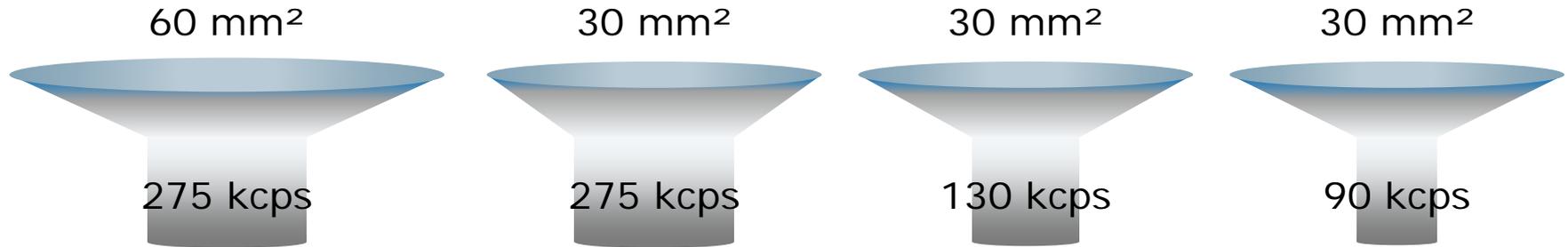
Vertical tilted

Horizontal



Improving the signal

Larger detectors and fast signal processing



Twice the detector size
(30 mm² to 60 mm²)



Output signal increased by ~ 60 %
(with the 275 kcps amplifier)



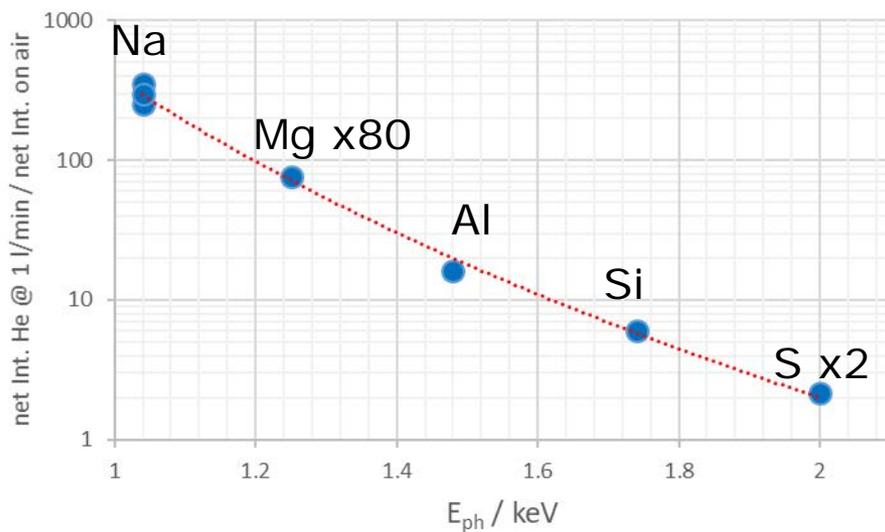
Significant reduction
of dead time
(and measurement time)

Improving the signal

He flush for light element detection

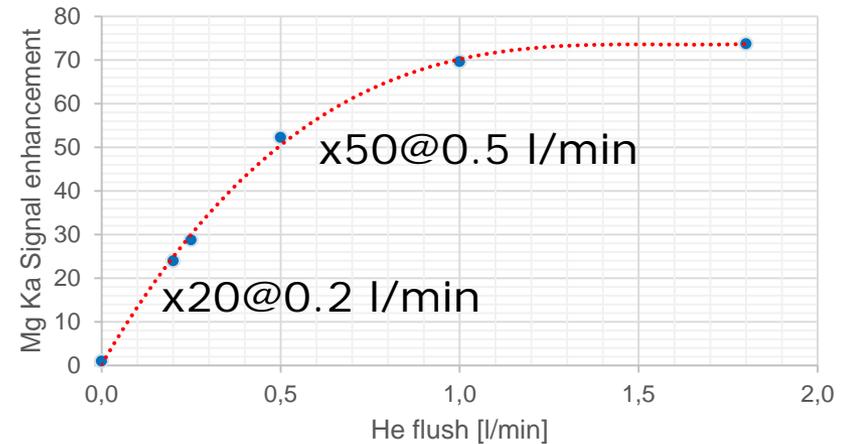


He flush adjustable, with integrated flow controller to operate between 0.2 l/min and 1.8 l/min



Values determined for a specific instrument, using pure element samples.

50 kV, 600 μ A, working distance set for 100 μ m spot, 30 mm² SDD, He flow rate: 1 l/min, in horizontal measurement orientation, Na-enhancement extrapolated



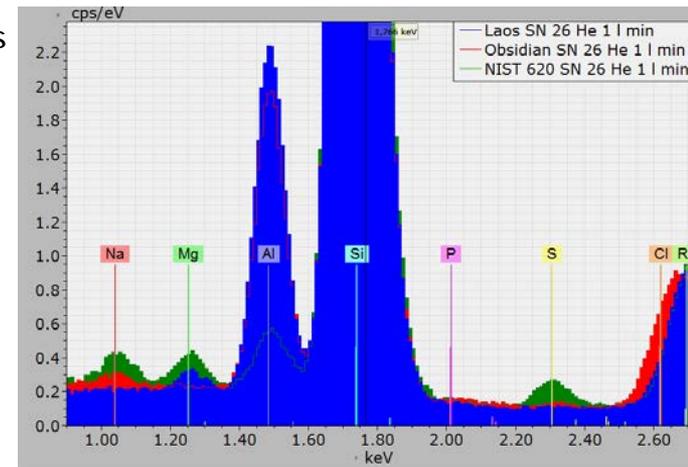
He consumption between 0.2 l/min and 0.5 l/min is a compromise between consumption and intensity gain

Na₂O in glass

NIST 620
14.39 wt.%

Obsidian
5.8 wt.%

Laos tektite
1.3 wt.%

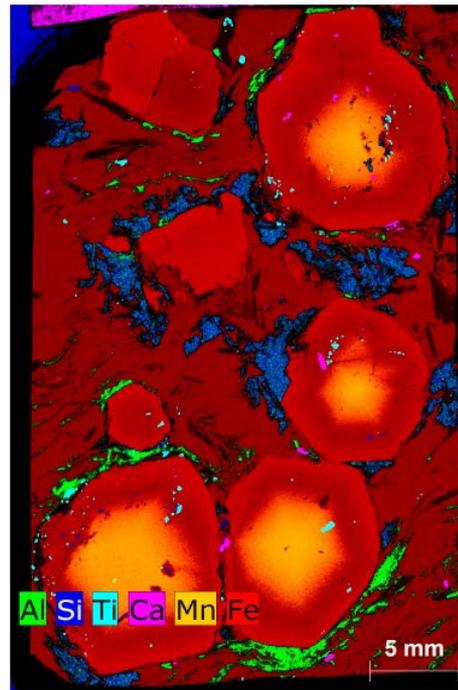


Micro-XRF features and benefits

At a glance



Information from the depth of the sample



Trace element sensitivity



No sample preparation

From the object to the data

The measurement parameters



'Soft' limit

Working time

Object size

Total measurement
time

Step size

Dwell time

Instrumental limit

File size:

Tested up to 33 Gbyte...

Scan dimension:

800 mm x 600 mm

Maximum speed:

100 mm/s

Maximum Acceleration:

200 mm/s²

Spot size:

Starting from 100 μm

Spectroscopic resolution:

<140 eV for 90 kcps

<145 eV for 130 kcps

<190 eV for 275 kcps

Step size

Should correspond to the size of the object that wants to be resolved

Painting \rightarrow 300 to 800 μm

Drawings \rightarrow 100 to 300 μm

Note: small step size allows to improve statistic by use of binning

Dwell time

Short (< 10 ms) \rightarrow main elements and or elements for which the instrument is sensitive



Dwell time
reduction

Improving the signal
- larger detector \rightarrow 60 mm²
- He flush

From the object to the data

The measurement parameters



Test painting



46 cm

36 cm

Variation of time

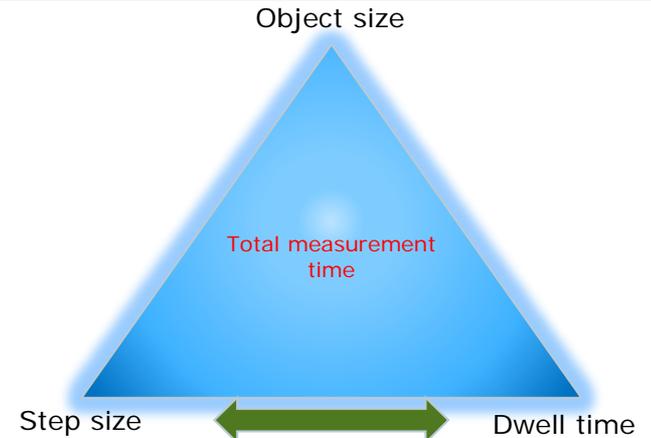
260 kPixel
800 μm / 3 ms

240 kPixel
800 μm / 700 ms

Variation of step size

4 MPixel
200 μm / 8 ms

16 MPixel
100 μm / 1 ms



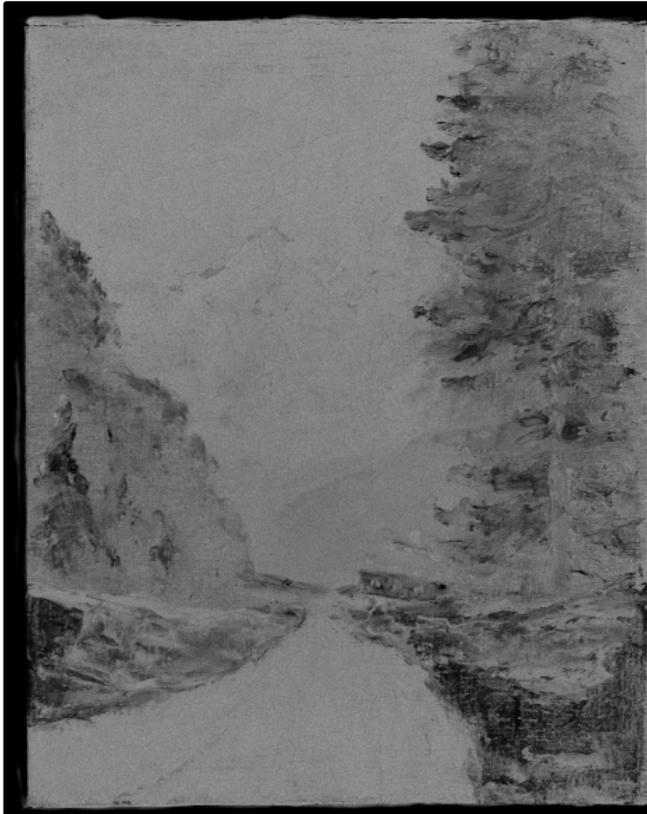
results

From the object to the data

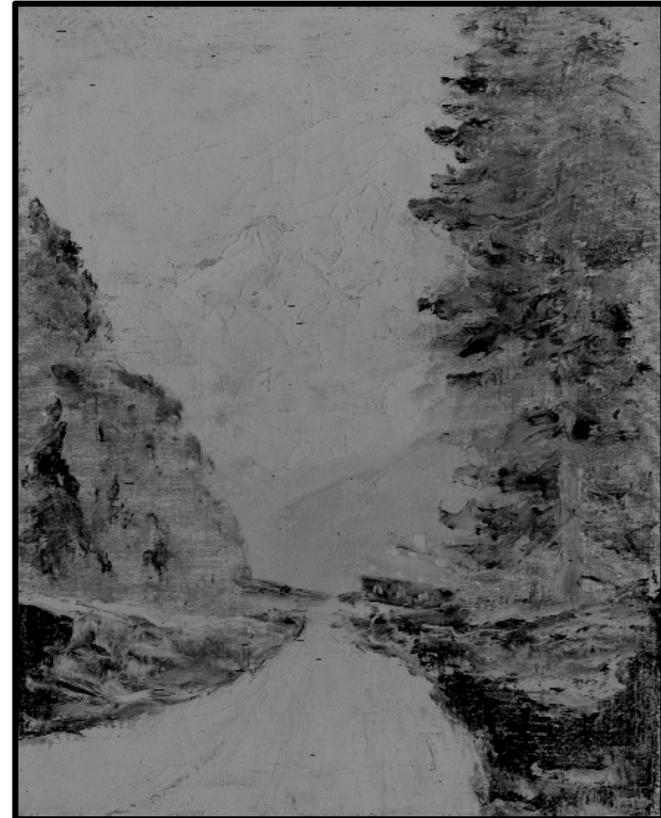
The measurement parameters



800 μm / 8 ms



800 μm / 700 ms



File	294 MByte		661 MByte
# of Spectra	260 kPixel		240 kPixel
Total time	1 h 35 min		48 h 32 min

From the object to the data

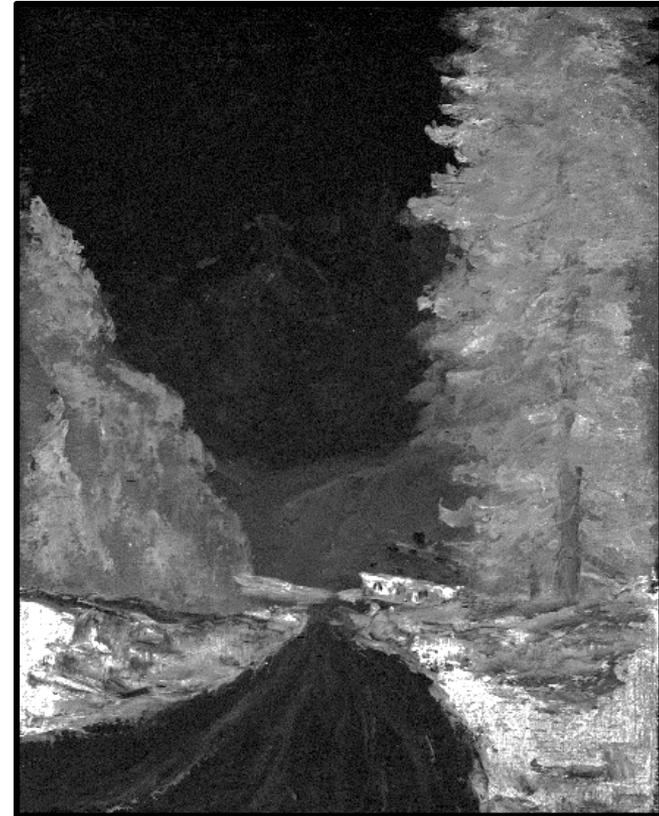
The measurement parameters



800 μm / 8 ms



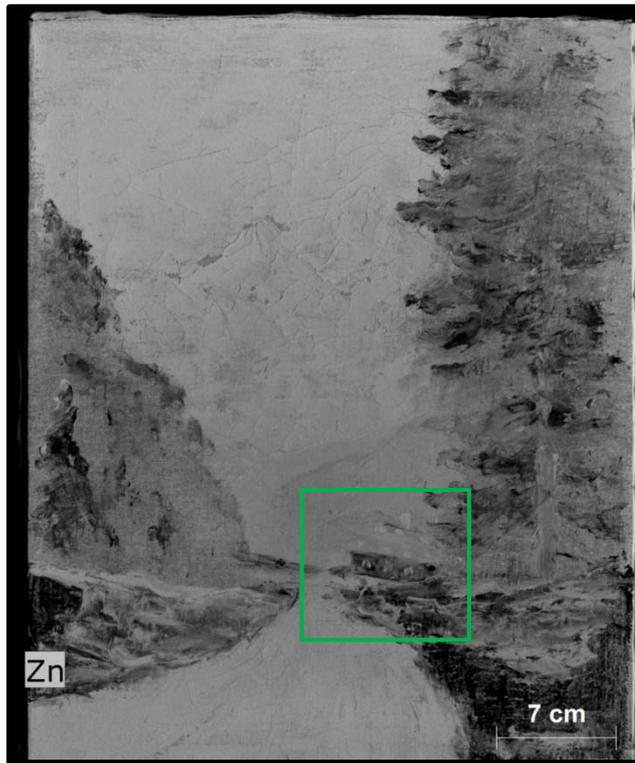
800 μm / 700 ms



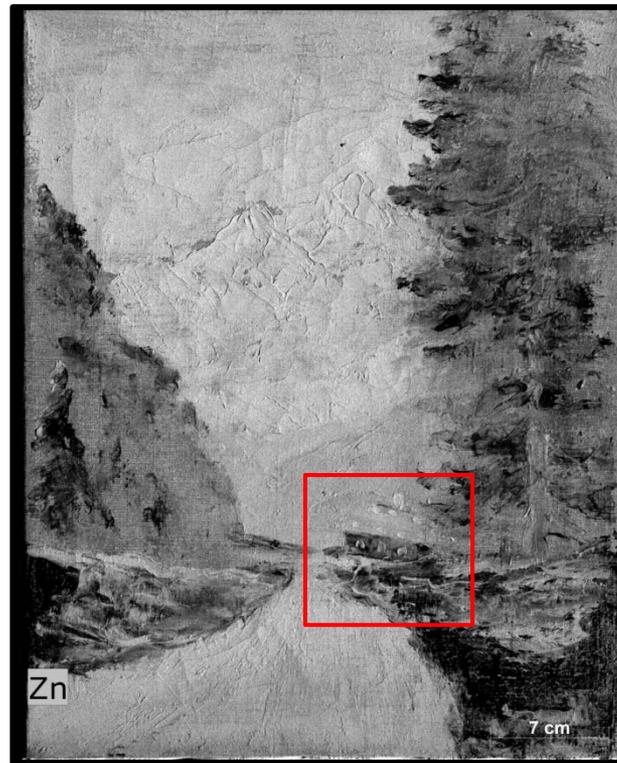
File	294 MByte		661 MByte
# of Spectra	260 kPixel		240 kPixel
Total time	1 h 35 min		48 h 32 min

From the object to the data

The measurement parameters



4 million



16 million



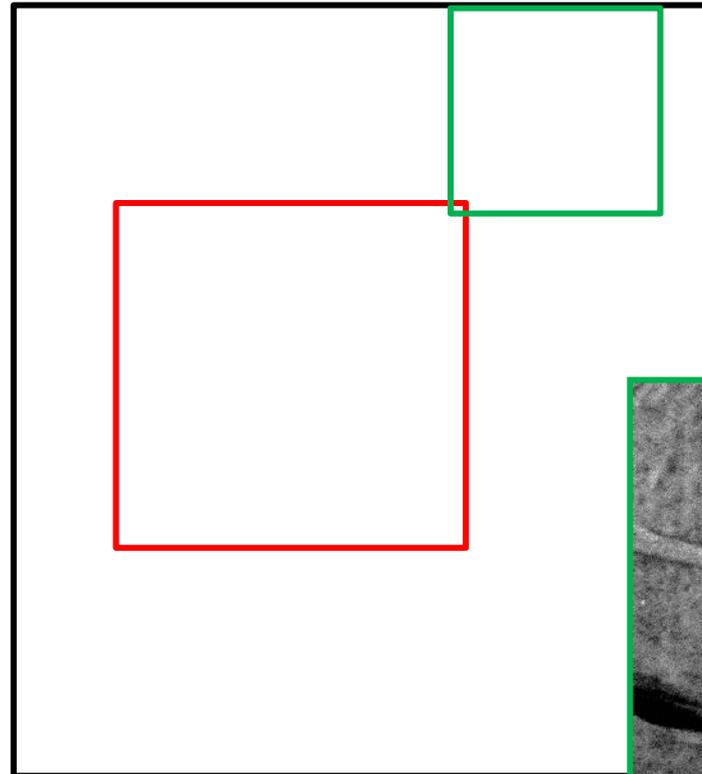
From the object to the data

The measurement parameters

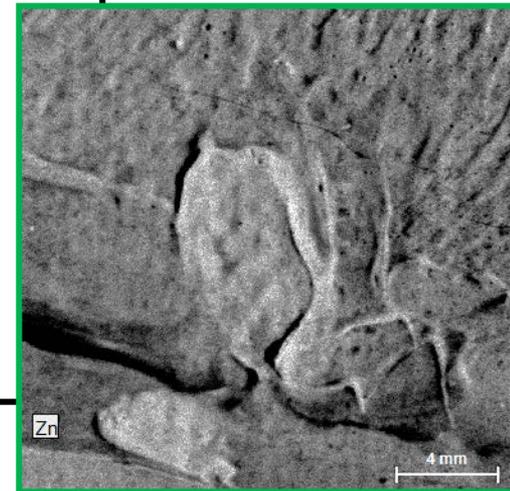


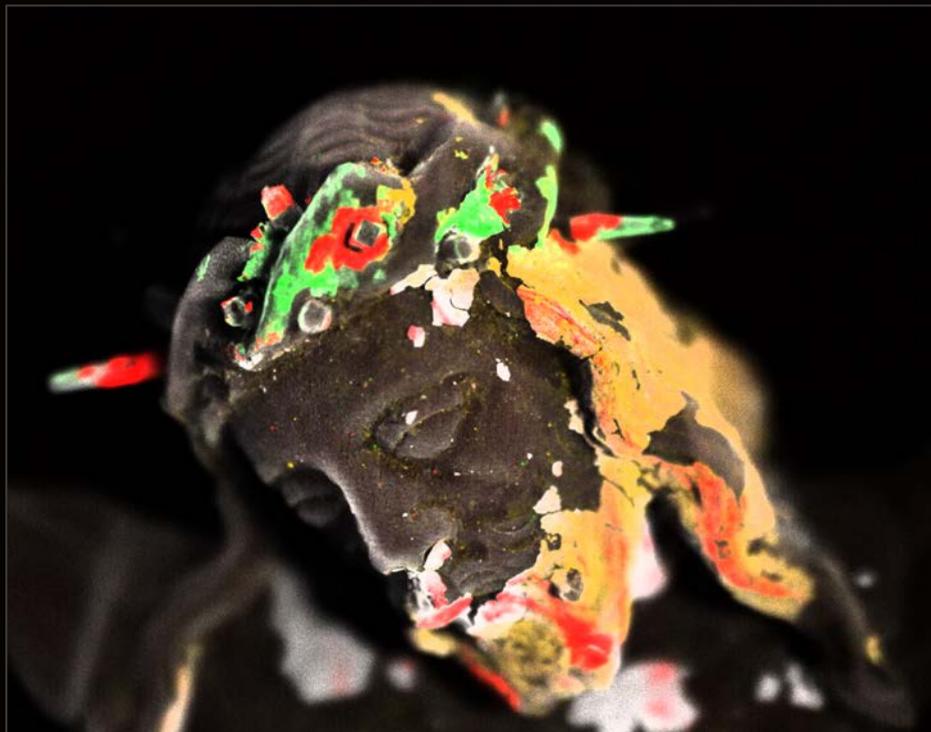
Detail measurement 1

Full scan 16 MPixel



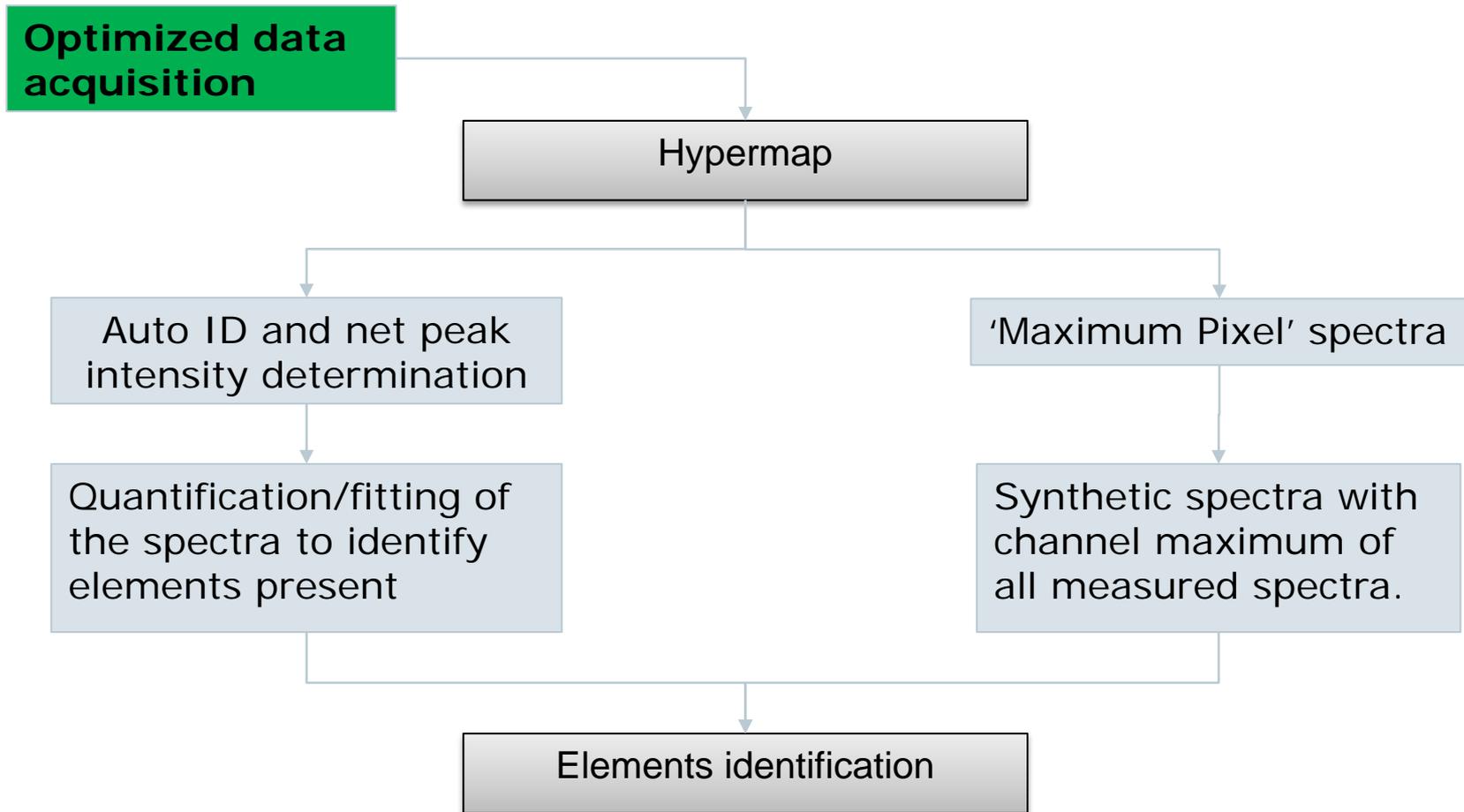
Detail measurement 2



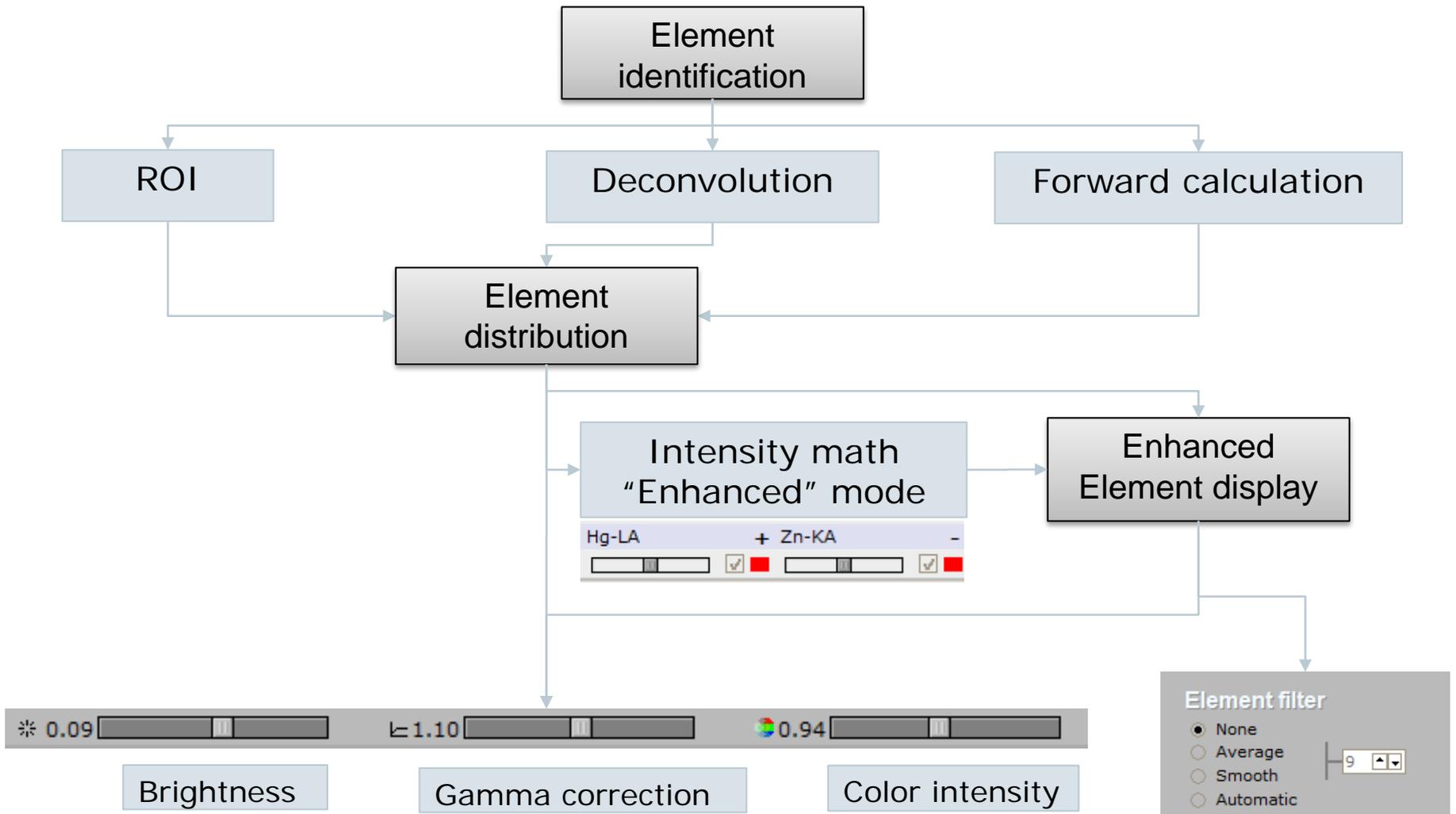


Pb Fe Cu Hg

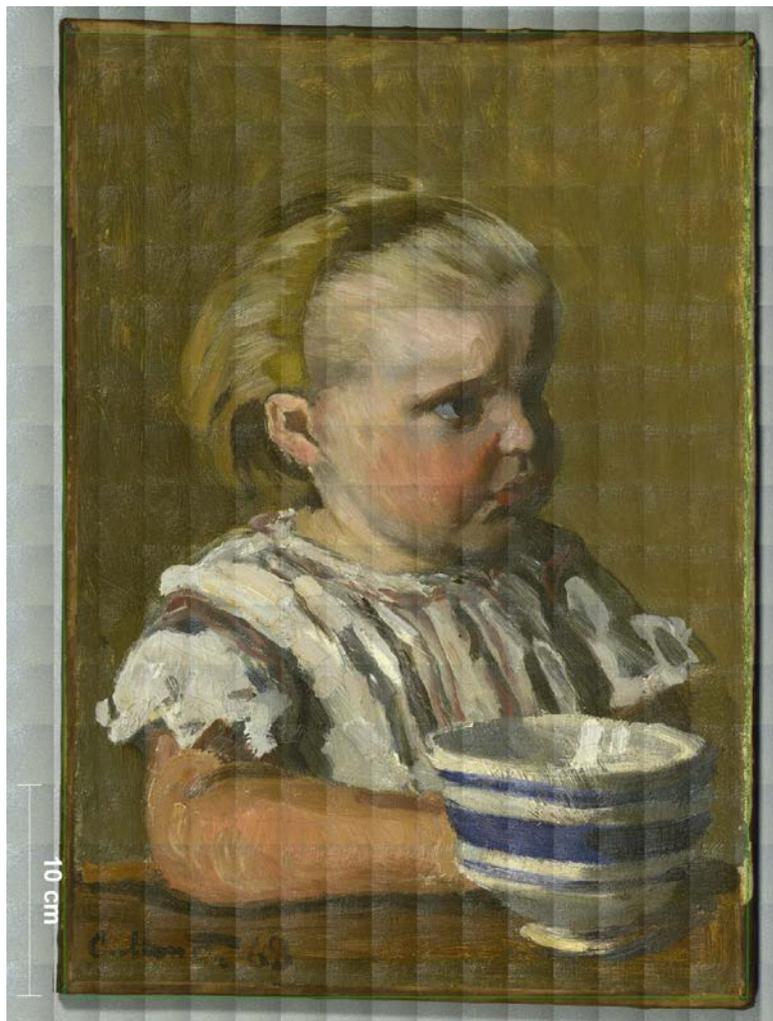
Now the data is there and the work starts: Data mining. What can be done?



Now the data is there and the work starts: Data mining. What can be done?



The Data:

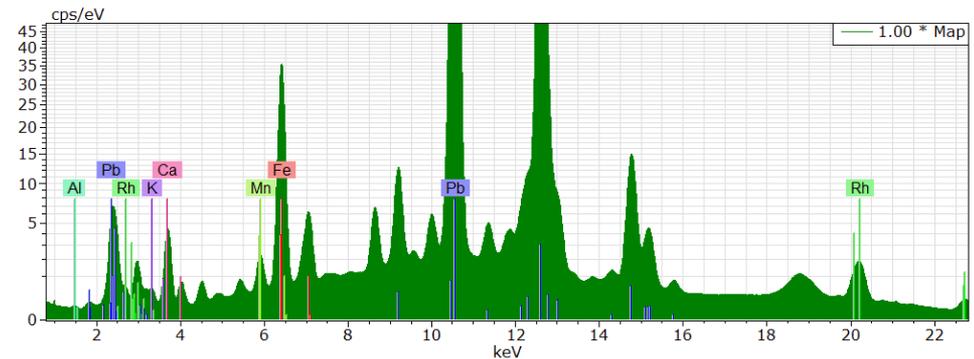


Mapping parameters		
Width:	916	pixel
	457.884	mm
Height:	640	pixel
	319.988	mm
Pixel Size:	500	μm
Total number of pixel:	586240	pixel
Acquisition parameters		
Frame count:	1	
Pixel time:	10	ms/pixel
Measure time:	1:14	h
Overall time:	2:43	h
Stage speed:	50.0	mm/s
Stage position (X,Y,Z):		
Tube parameter		
High voltage:	50	kV
Anode current:	600	μA
Filter:	Empty	
Optic:	Lens	
SpotSize:	25	
Chamber at:	Air 1055	mbar
Anode:	Rh	
Detector parameters		
Selected detectors:	1	
Max. pulse throughput:	275000	cps

Data mining, Element identification: Auto ID and Interactive quantification

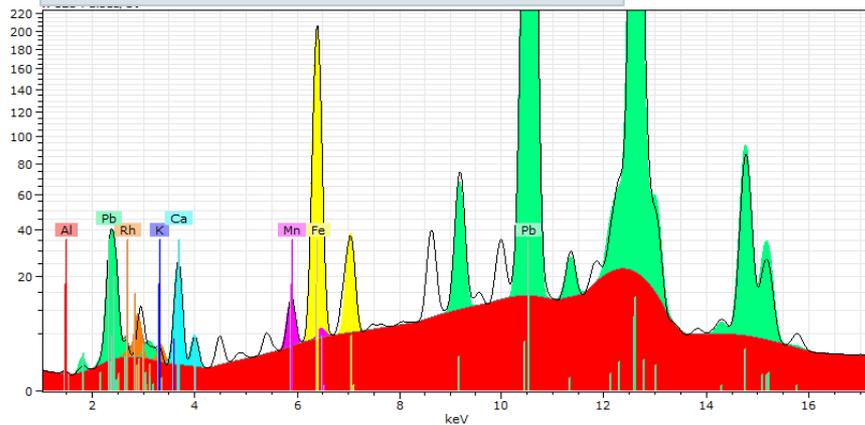


- 1) The Auto ID does not identify all the elements. It is based on a quantification of all "allowed" elements.
- 2) False identifications are possible!



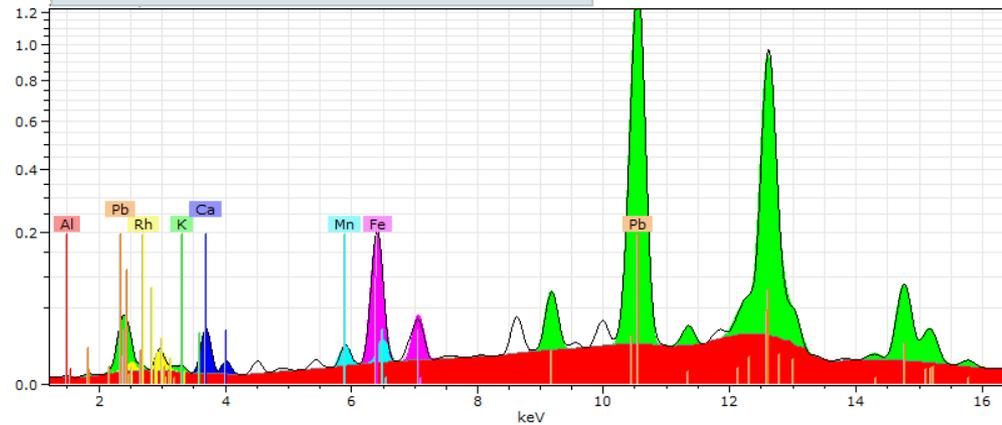
Quantify ▾ AutoPhase ▾
i ▶ Spectrum elements

Fundamental parameter
forward calculation



Quantify ▾
i ▶ XRF-Deconv rt

Bayes deconvolution



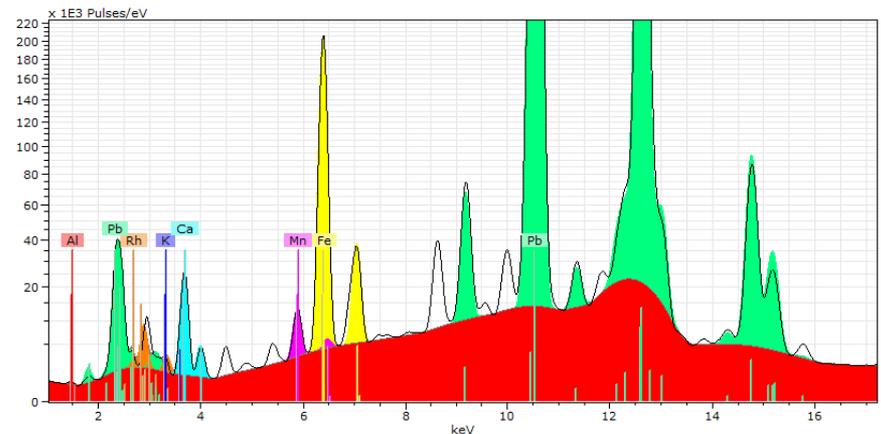
Data mining, Element identification: Interactive quantification using FP model



The M6's quantification iteratively varies the assumed sample composition and forward calculates the resulting spectra by repeatedly solving the Sherman Equation.

The prerequisite for a quantification is a homogenous, infinitely thick sample.
...which is rarely the case for a painting.

Fundamental parameter
forward calculation



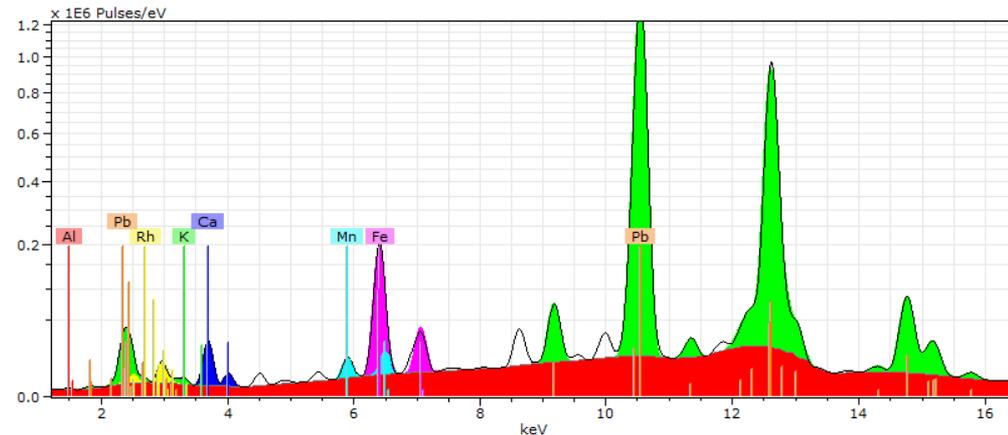
There is no forward calculated homogeneous, infinitely thick sample which produces a spectrum like the measured one. Therefore the fit cannot be perfect. However, for most of the samples the fit is surprisingly good. But problems might appear especially in the low energy range!

Data mining, Element identification: Interactive deconvolution using Bayes

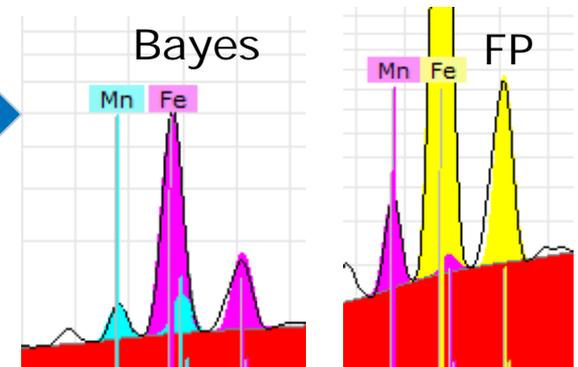


There is a possibility to determine the peak intensity by using a Bayes deconvolution. In this case a peak fit using Gaussian peaks is performed. However, since for example, the line ratios for the elements are not fixed, the deconvolution can run into some problems.

Bayes deconvolution

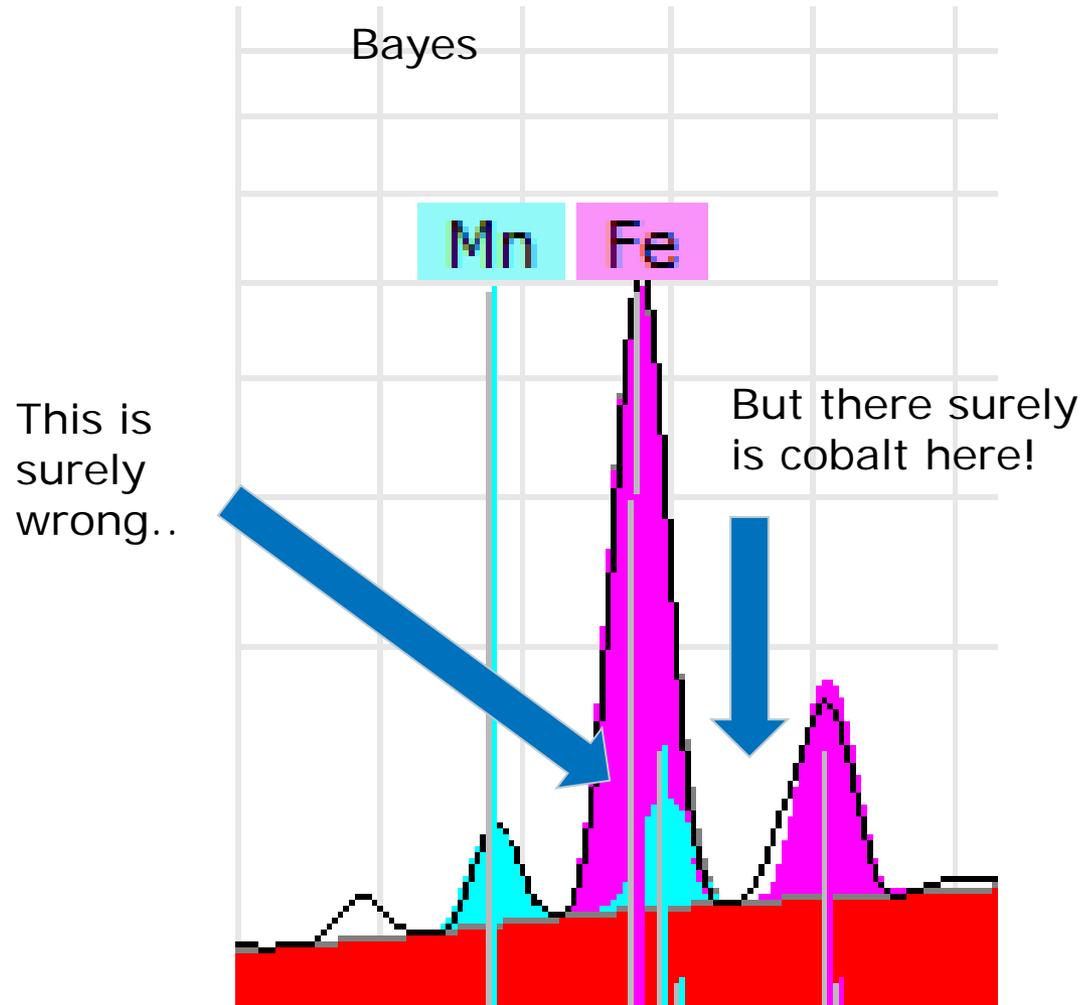


The Mn Kb line is overestimated in the Bayes deconvolution.



There is no correct solution, there are different tools, which have their pros and cons...

Data mining, Element identification: Interactive deconvolution using Bayes

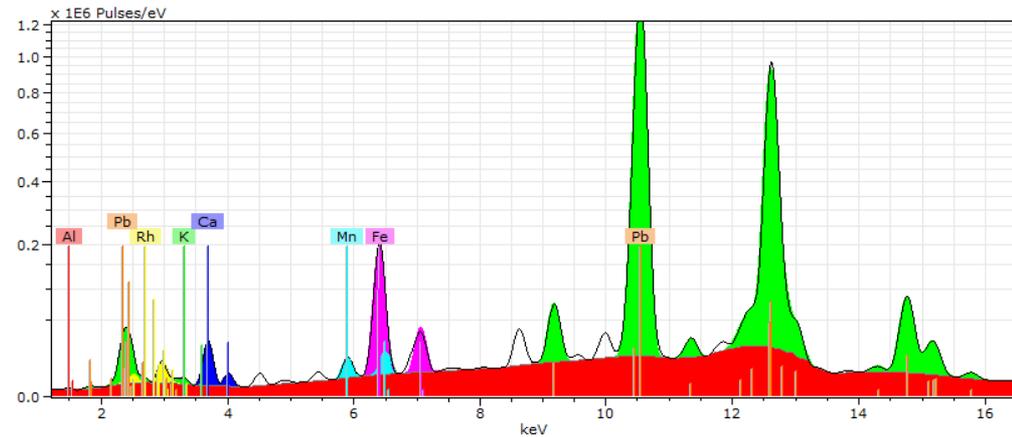


Data mining, Element identification: Interactive deconvolution using Bayes

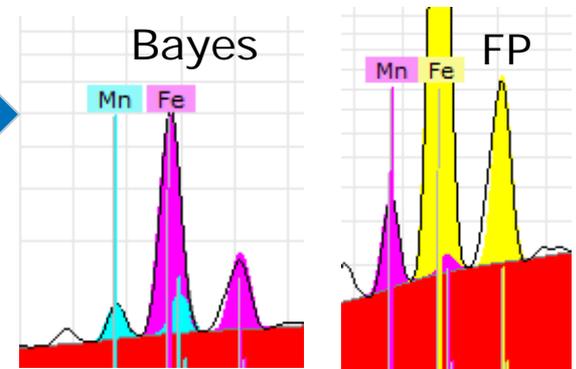


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Bayes deconvolution

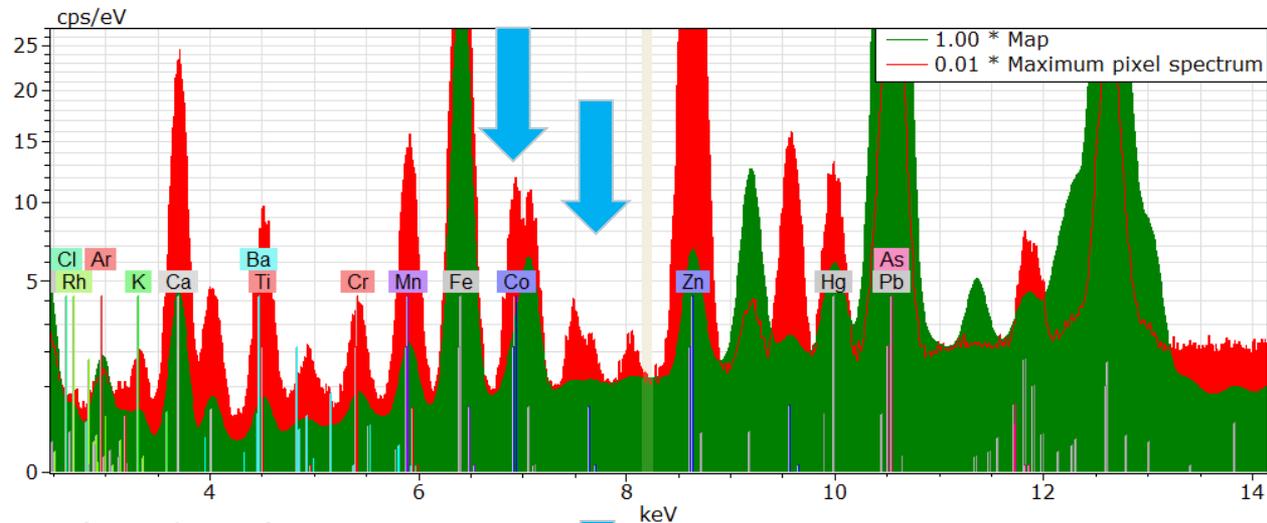
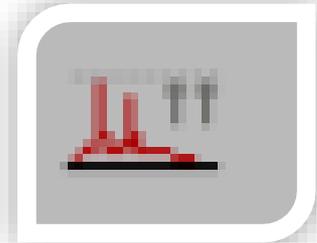


The Mn Kb line is overestimated in the Bayes deconvolution.

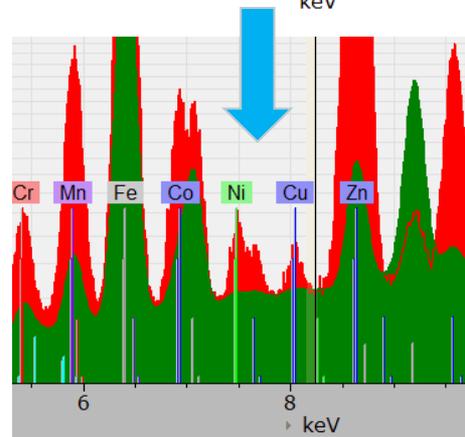


There is no correct solution, there are different tools, which have their pros and cons...

Data mining, Element identification: Maximum Pixel spectra



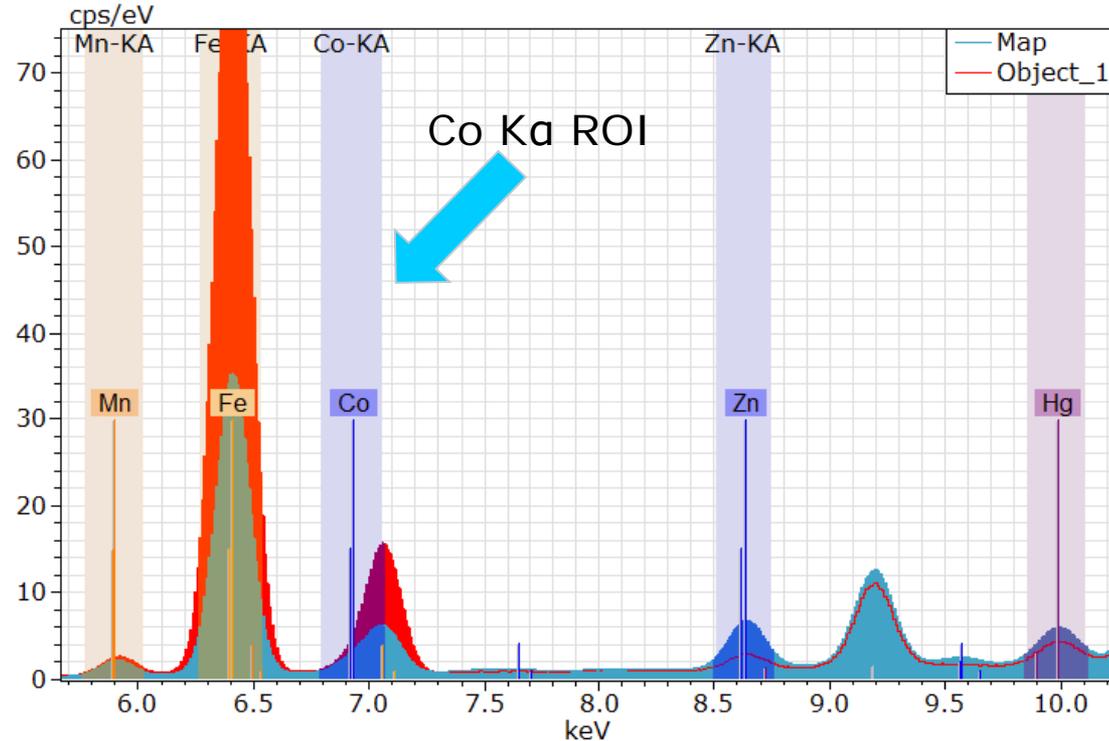
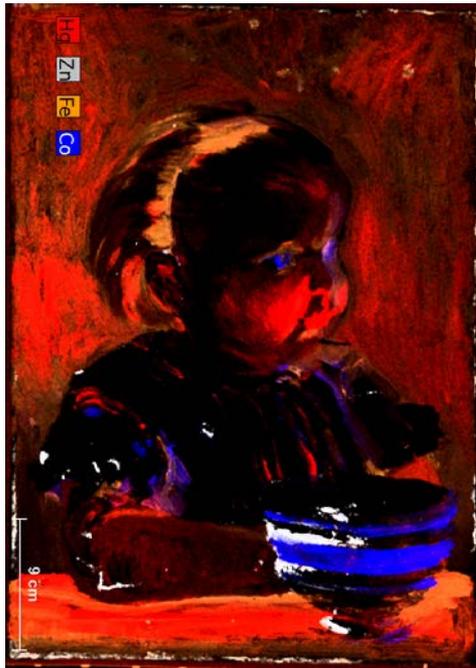
In the map spectrum the signal for Ni and Cu is "diluted" by the large number of spectra. The Maximum pixel spectrum reflects the highest intensity per channel found in any pixel of the map. Maximum pixel spectrum can be used to find hot spots in the data block.



Easy identification of Ni and Cu presence somewhere in the sample.

And even the cobalt is no longer in question!

Data mining, Element display: Region of interest ROI



The ROI element display does not correct for peak overlapping or background. Thus, f.e. the Co intensity contains also parts of the K β from Fe.

Extremely fast and robust!

Data mining, Element display: Region of interest ROI



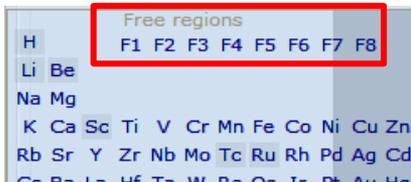
In the periodic table (right mouse on the element) it is possible to edit the lines that shall be used for the element display.

The Line as well as the width of the Region around the peak can be edited.

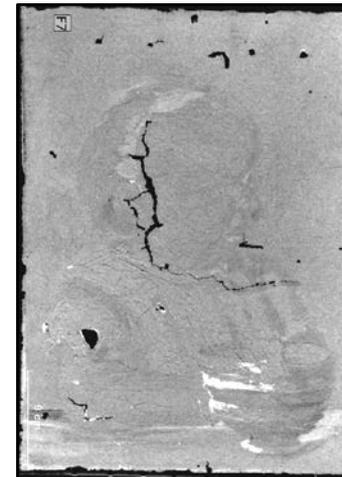
Note: only one line of an element can be displayed at a time. To display two lines of an element (Pb-M and -L) at the same time, a Free region can be used.

The screenshot shows the 'Properties for Lead' window. It includes a table of element lines with checkboxes for selection. The 'Spectrum region' section has radio buttons for 'None', 'KA 74.970 keV', 'KB 84.858 keV', 'LA 10.541 keV', 'LB 12.612 keV', and 'MA 2.345 keV'. The 'Peak area' section has radio buttons for 'Wide (99%)', 'Medium (87%)', and 'Narrow (55%)', with a text input field set to '87 %'.

Lines	Symbols	Energy (keV)
<input checked="" type="checkbox"/>	KA1	74.970 keV
<input checked="" type="checkbox"/>	KA2	72.805 keV
<input checked="" type="checkbox"/>	KB1	84.939 keV
<input checked="" type="checkbox"/>	KB2	87.243 keV
<input checked="" type="checkbox"/>	KB3	84.451 keV
<input checked="" type="checkbox"/>	KB4	87.571 keV
<input checked="" type="checkbox"/>	KB5	85.419 keV
<input checked="" type="checkbox"/>	LA1	10.551 keV
<input checked="" type="checkbox"/>	LA2	10.449 keV
<input checked="" type="checkbox"/>	LB1	12.614 keV
<input checked="" type="checkbox"/>	LB2	12.601 keV
<input checked="" type="checkbox"/>	LB3	12.795 keV
<input checked="" type="checkbox"/>	LB4	12.307 keV
<input checked="" type="checkbox"/>	LB5	13.014 keV
<input checked="" type="checkbox"/>	LB6	12.143 keV
<input checked="" type="checkbox"/>	LE	11.349 keV
<input checked="" type="checkbox"/>	LG1	14.755 keV



Free region can be use to display the intensity of any ROI in the spectrum, f.e. scattering background or total intensity.

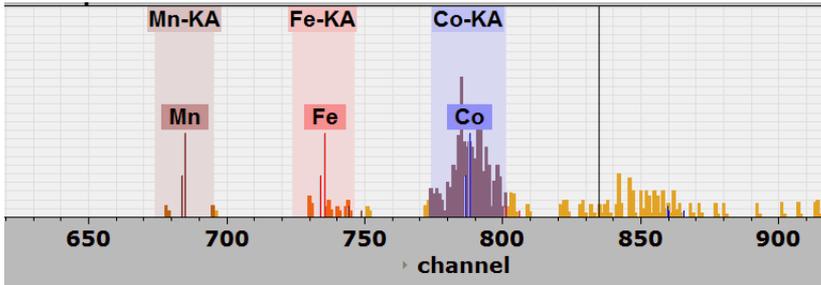


Total intensity



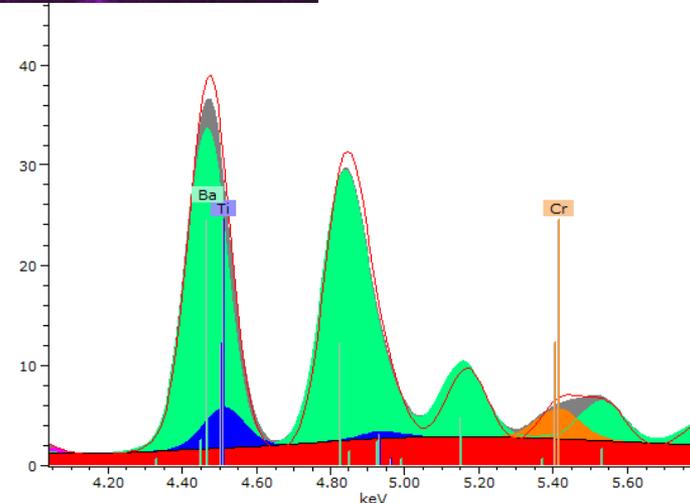
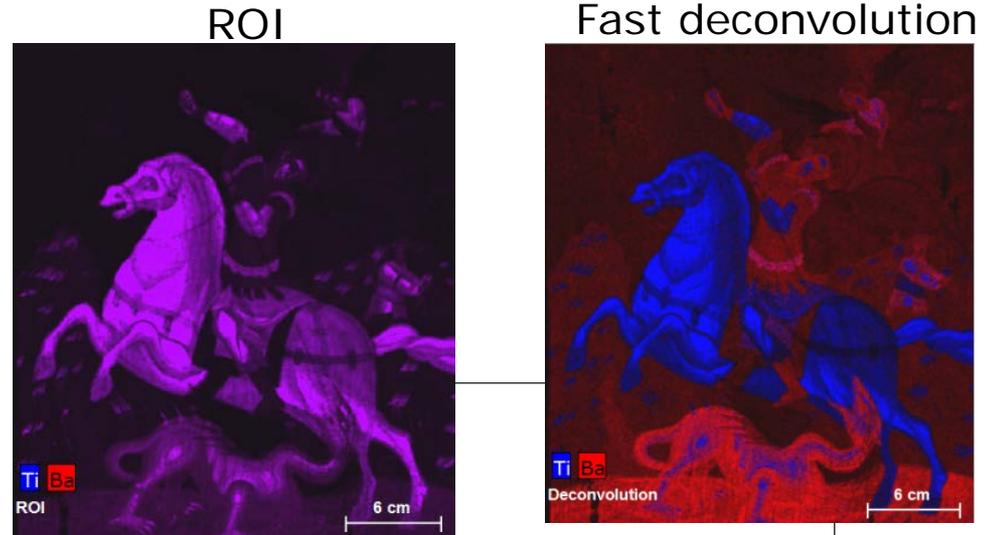
Compton

Data mining, Element display: Fast deconvolution



In the fast deconvolution (which is a fit) every count in every channel is weighted by the probability that it belongs to one of the selected elements. As soon as this is calculated for each channel the complete data set is evaluated at high speed.

In this case 17 elements and half a million spectra were 'deconvoluted' in 30 seconds.

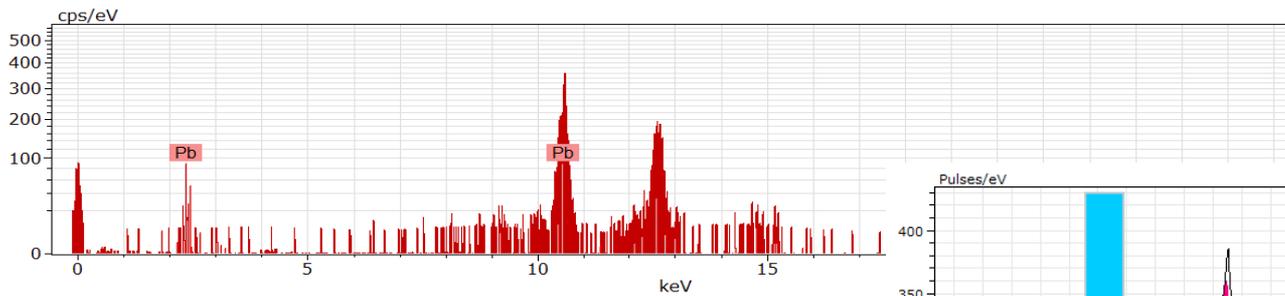


Data mining, Element display: Forward calculation I

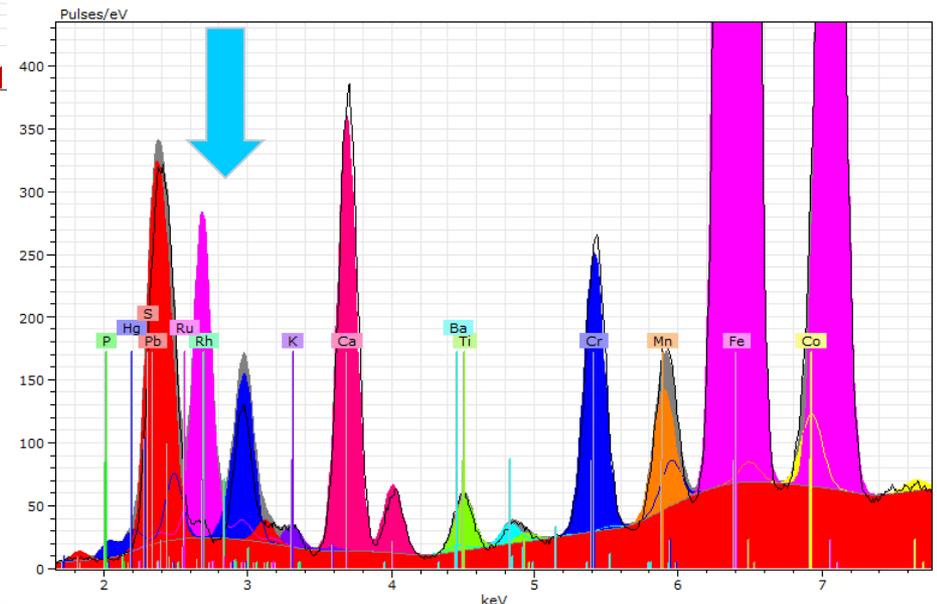


The quantification of a non-ideal sample is the main problem of this approach.

One spectrum acquired in short time might contain only a limited number of counts, e.g. 1500 counts in 4096 channels. Making an iterative spectrum fit and quantification unstable.



Best numerical solution might not be the best fit, as a non-ideal sample has no "correct" solution for the Sherman Equation



Data mining, Element display: Forward calculation



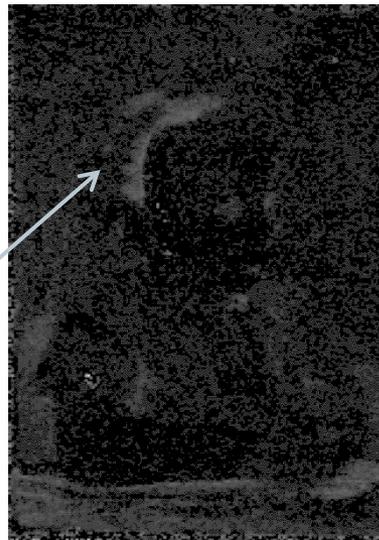
Convolution

Multiple overlapping's Hg-Pb-As

Very slow, the forward calculated spectrum assumes an infinitely and homogenous sample



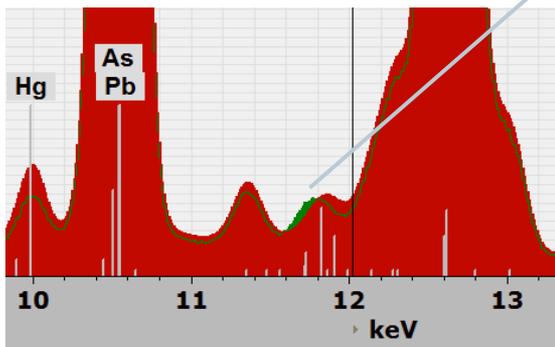
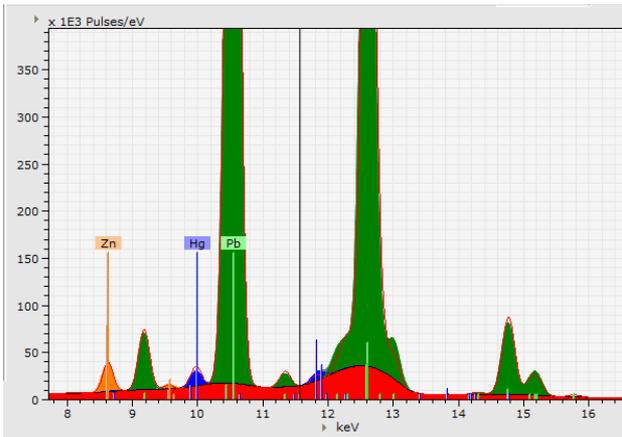
As



Pb



Hg



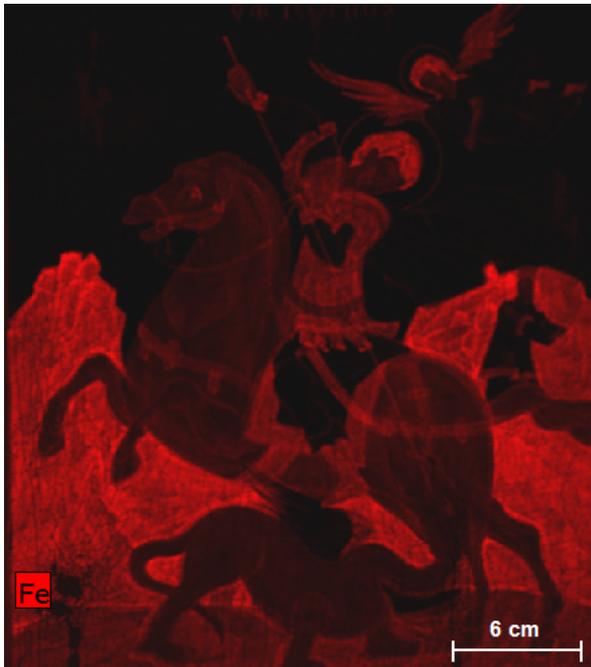
Data mining, Element display: Enhanced mode



Peak
mathematics

Very fast, easy to implement, plenty of options

A bit complex to understand at the beginning.
Requires experience (or talent)



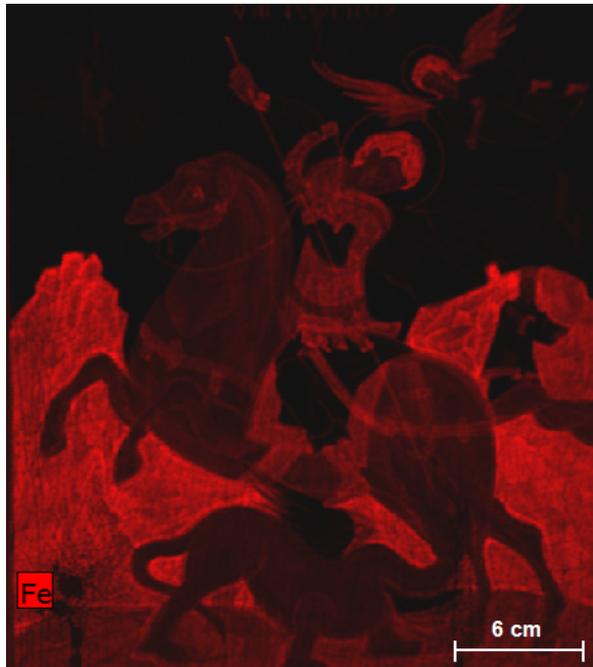
Data mining, Element display: Enhanced mode



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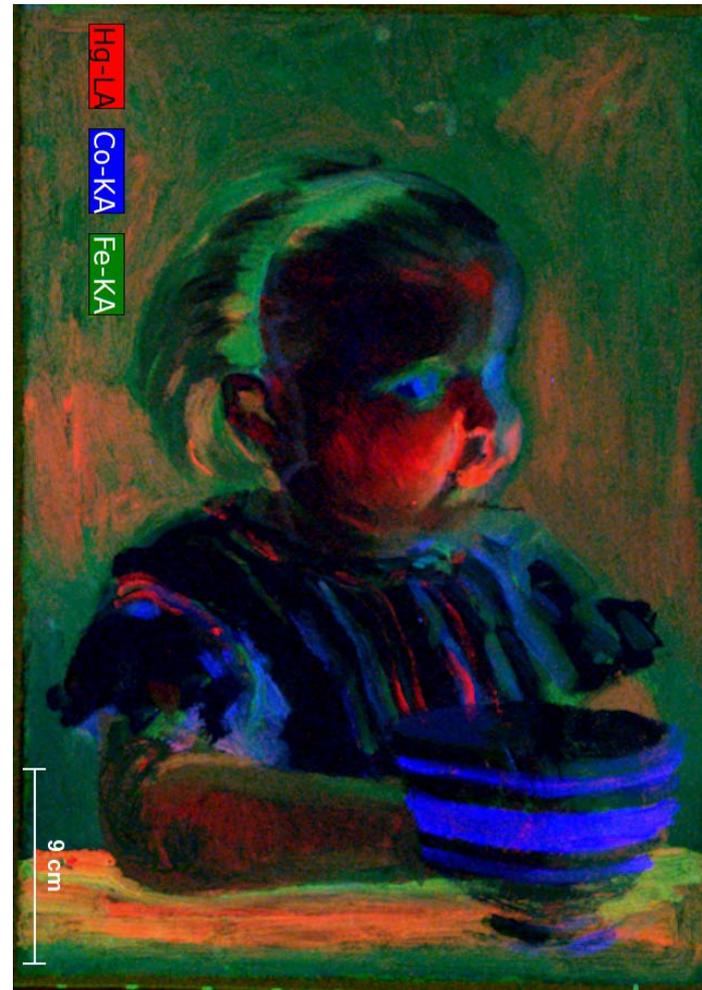
Data mining, visualization: Image parameters



By changing the slider position it is possible to highlight or oppress specific features



Data mining, visualization: Image parameters

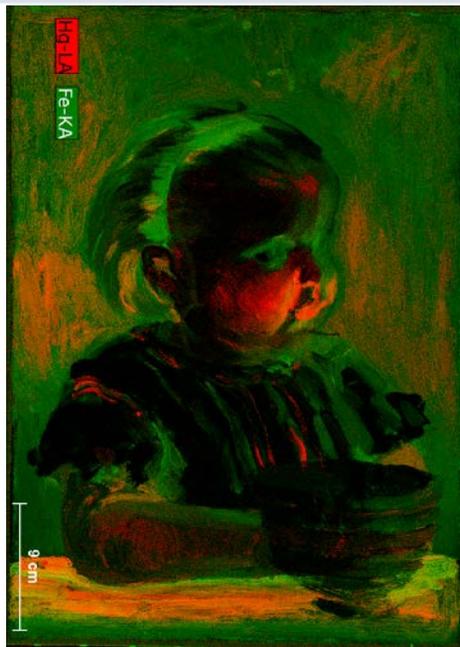


Gamma correction

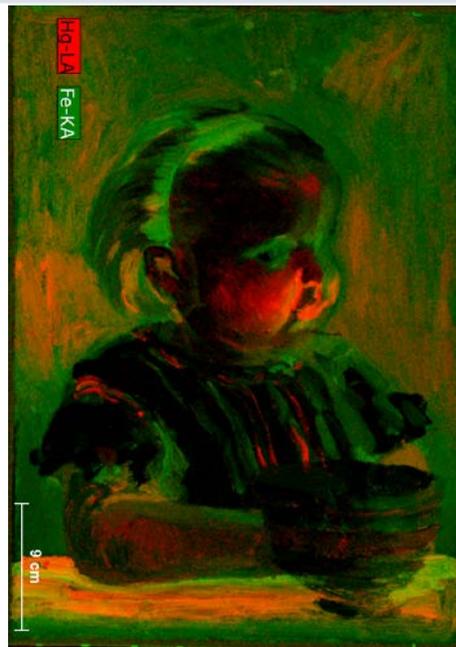
Brightness

Color intensity

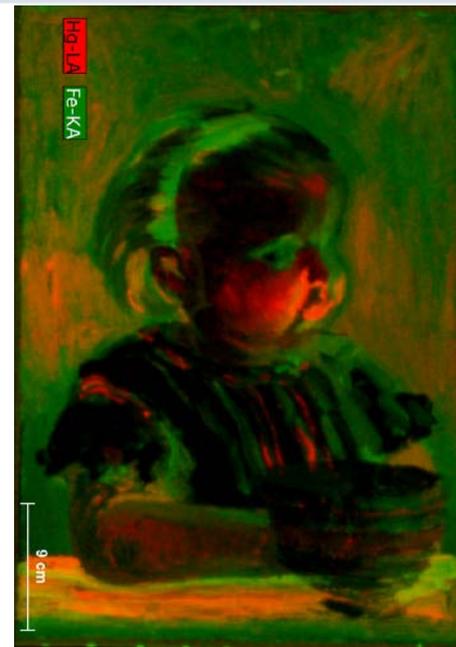
Data mining, visualization: Element filter



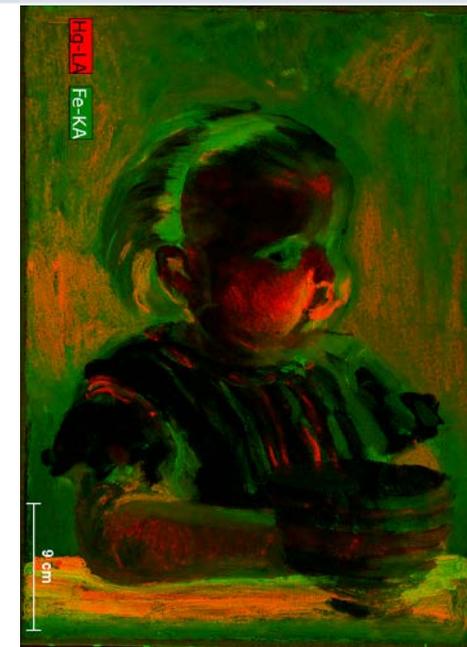
None



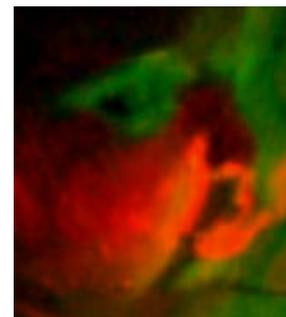
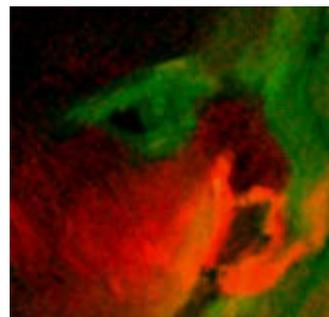
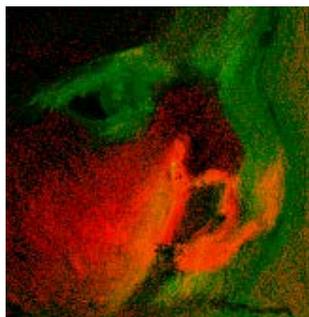
Average 3



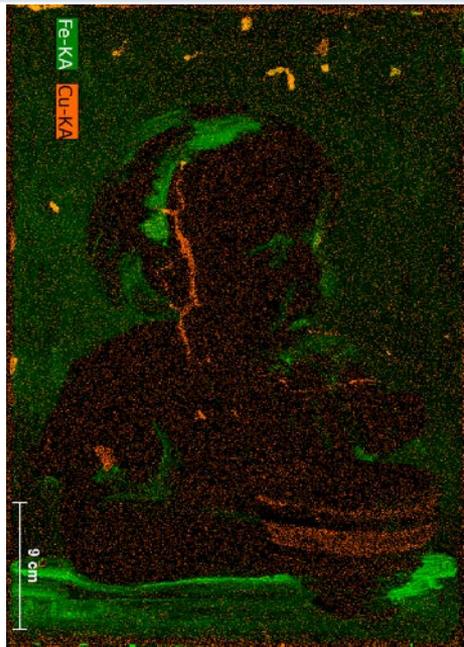
Smooth 3



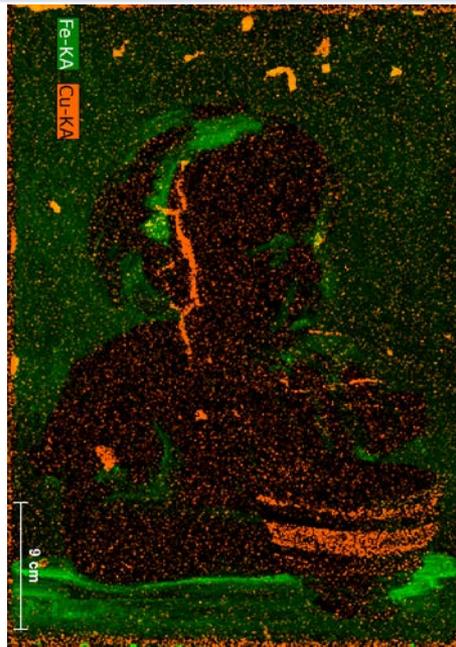
Automatic



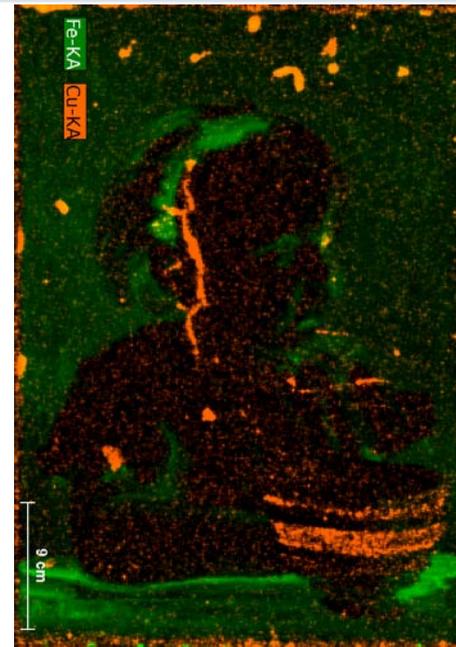
Data mining, visualization: Image parameters



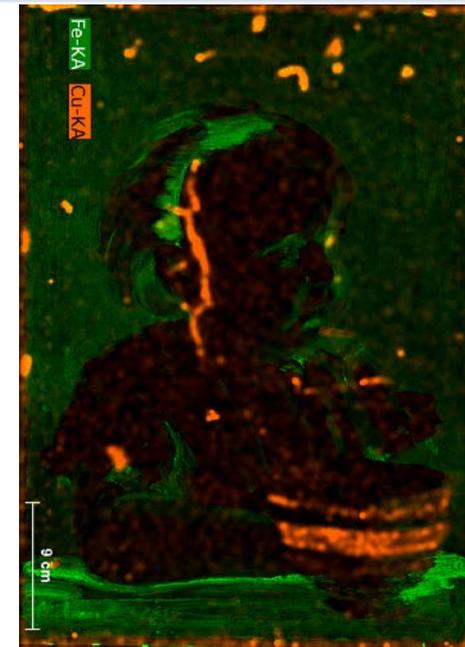
None



Average 3



Smooth 3

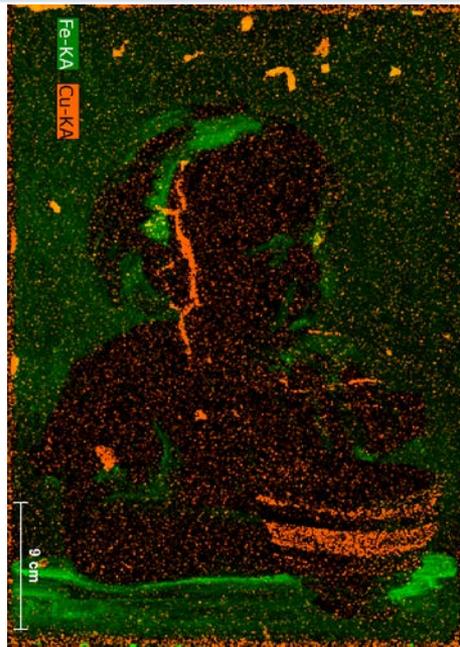


Automatic

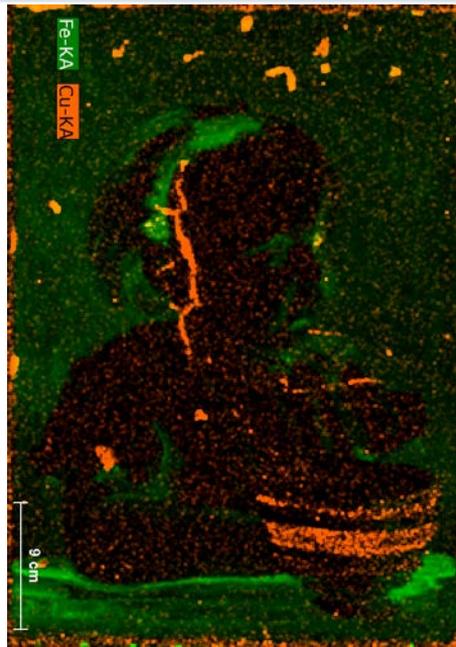
Automatic: every element displayed gets, after a statistical evaluation of the number of count in the data, an “optimal” binning or average number for the display.

... without telling, what was done for which element.

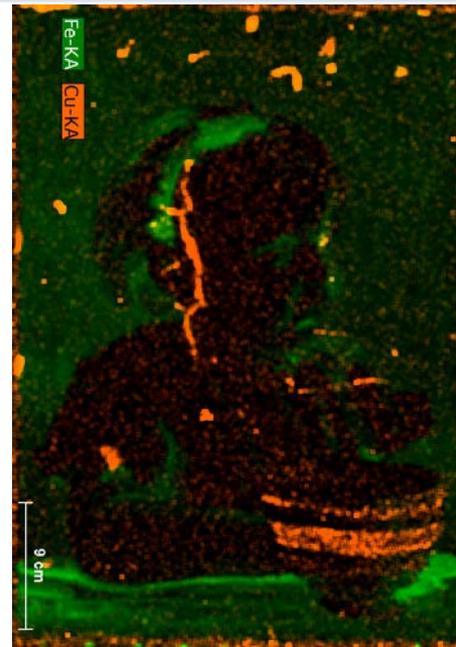
Data mining, visualization: Image parameters



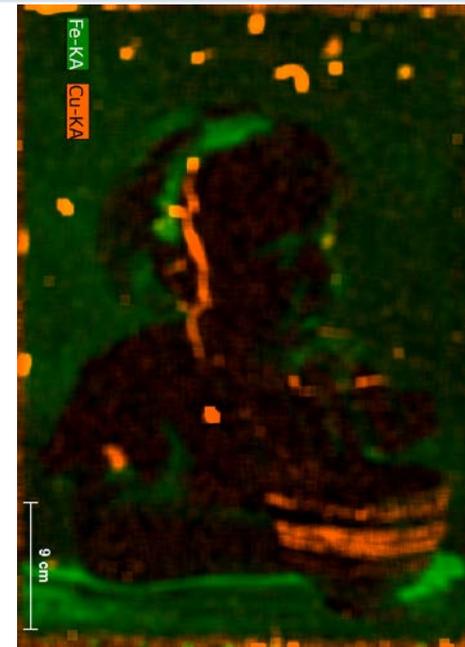
Average 3



Average 5



Average 7



Average 15

Data mining: Getting the data out...



The measured data is stored in a BCF file. This file contains all the information regarding the measurement.

The data can be extracted or converted in a format that can be read by other software.

Therefore 3 options are available:

- 1) Exporting all single spectra from the map using a script function
- 2) Saving the data block as a RAW file
- 3) Extracting the single element information in the 'element images' of the map window to a TXT file as a number matrix

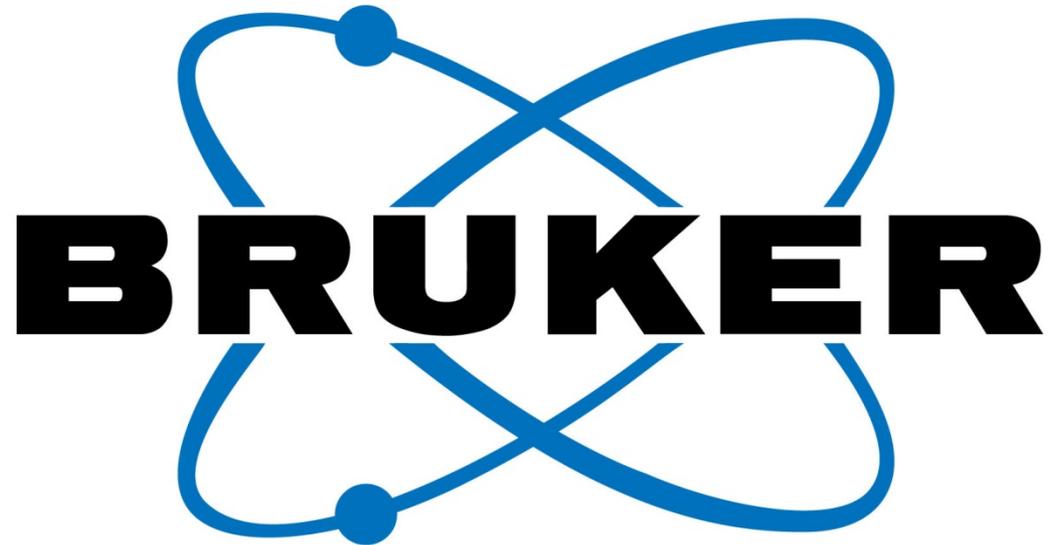


Questions, Thoughts or Comments?

If you have questions or want to contact us during the Webinar, please **type your questions**, thoughts, or comments in the **Q&A box** and **press Submit**.

We ask for your understanding if we do not have time to discuss all comments and questions within the session.

Any unanswered questions or comments will be answered and discussed by e-mail or in another Webex session.



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