Processing of MA(or μ)-XRF Data with the M6 software

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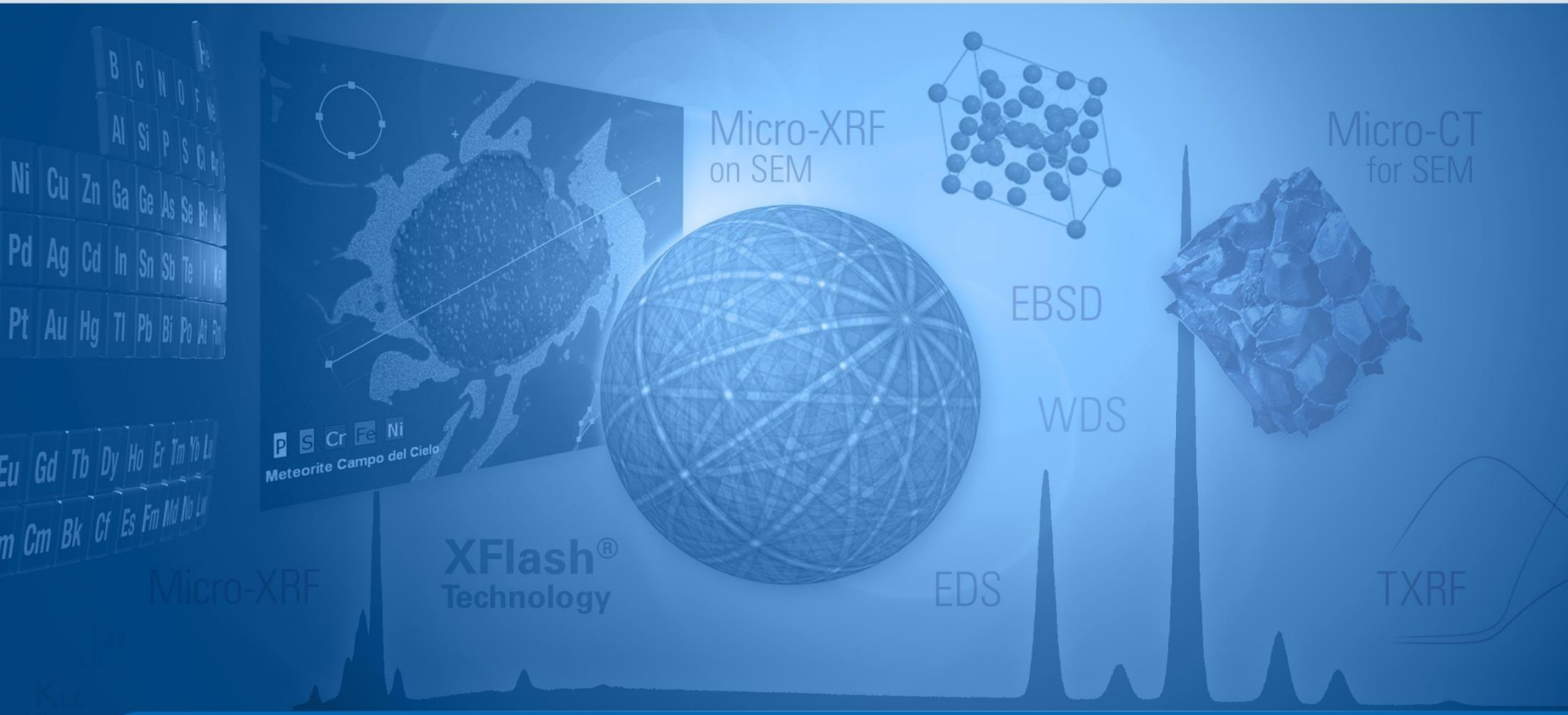
Bruker Nano Berlin



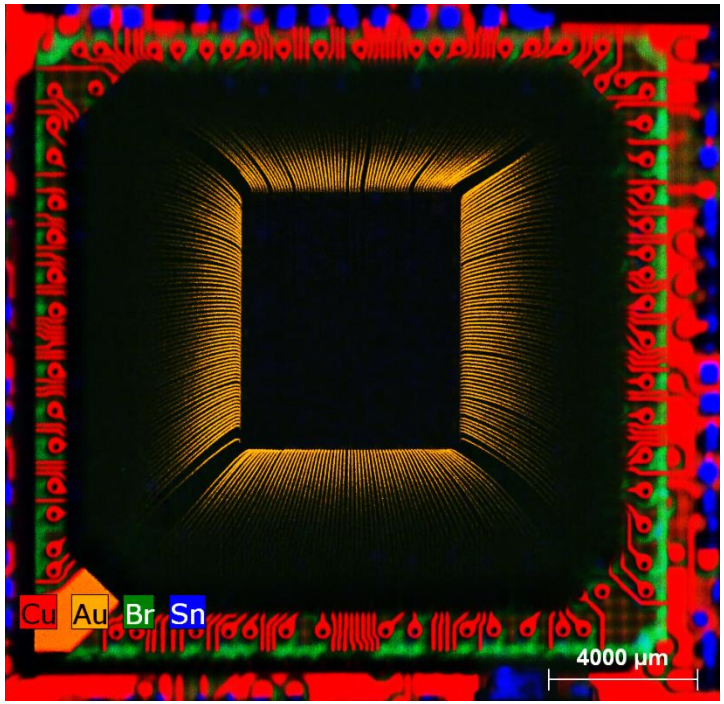
Outline

1. Introduction

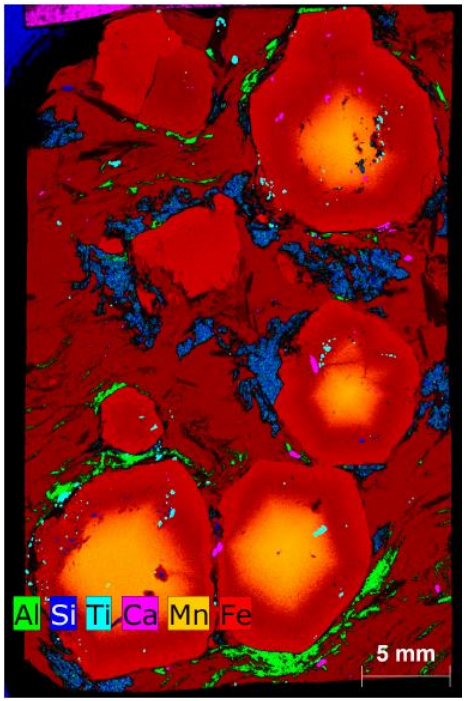
2. From the object to the data
   - The measurement parameters

3. From the data to the results: data mining tools advantages and disadvantages
   - ROI
   - Fast deconvolution
   - Convolution by forward calculation
   - Peak math, addition and subtraction of lines
   - and more….

Three main advantages of µ-XRF



Information from the depth of the sample



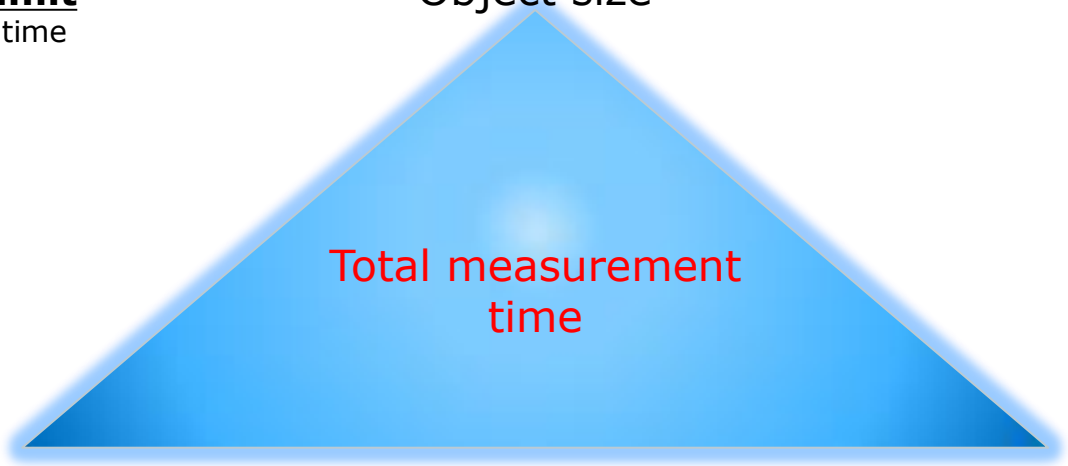
Trace element sensitive

No sample preparation

From the object to the data
The measurement parameters

'Soft' limit
Working time

Object size



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 → 300 to 800 µm
Drawings →100 to 300 µm

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

Dwell time

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

Dwell time reduction

Improving the signal
- larger detector → 60 mm²
- He flush

From the object to the data
The measurement parameters

Test painting

46 cm

36 cm

Variation of time

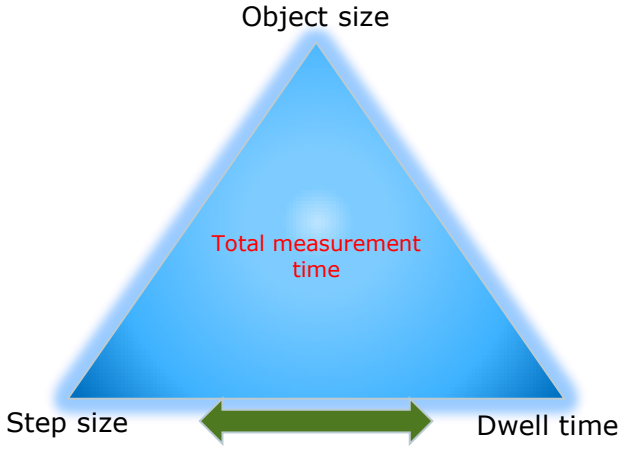
260 kpixel
800 µm / 3 ms

240 kpixel
800 µm / 700 ms

Variation of step size

4 Mpixel
200 µm / 8 ms

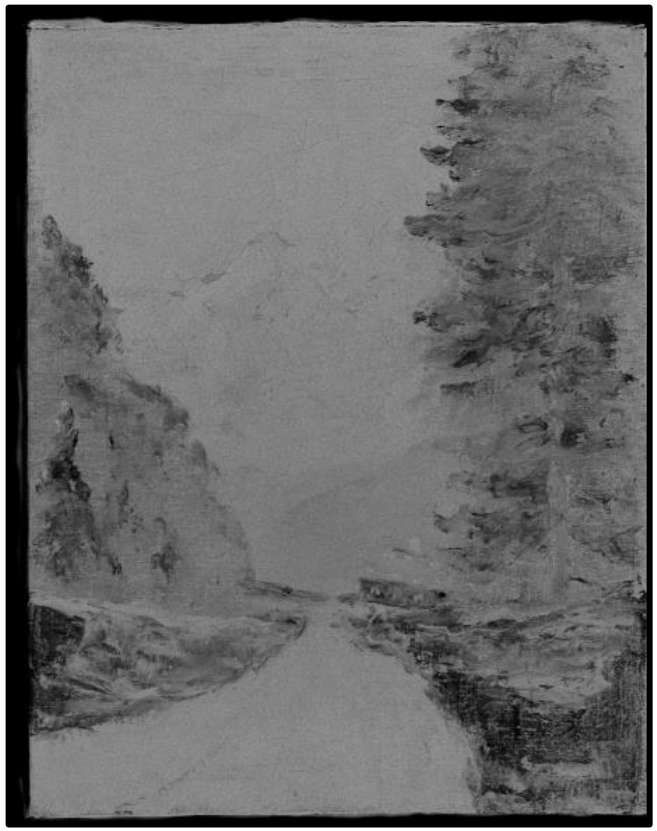
16 Mpixel
100 µm / 1 ms



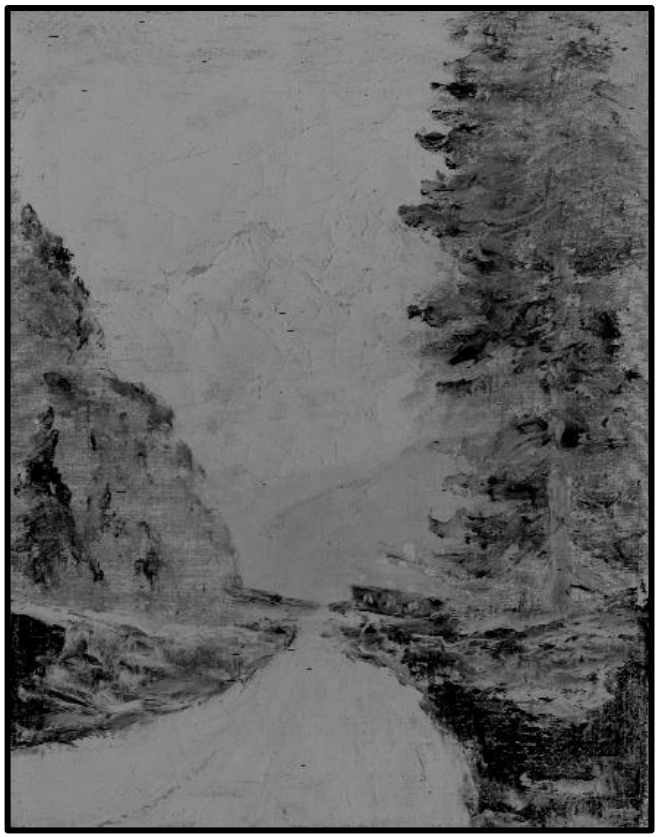
results

From the object to the data
The measurement parameters

800 µm / 8 ms



800 µm / 700 ms

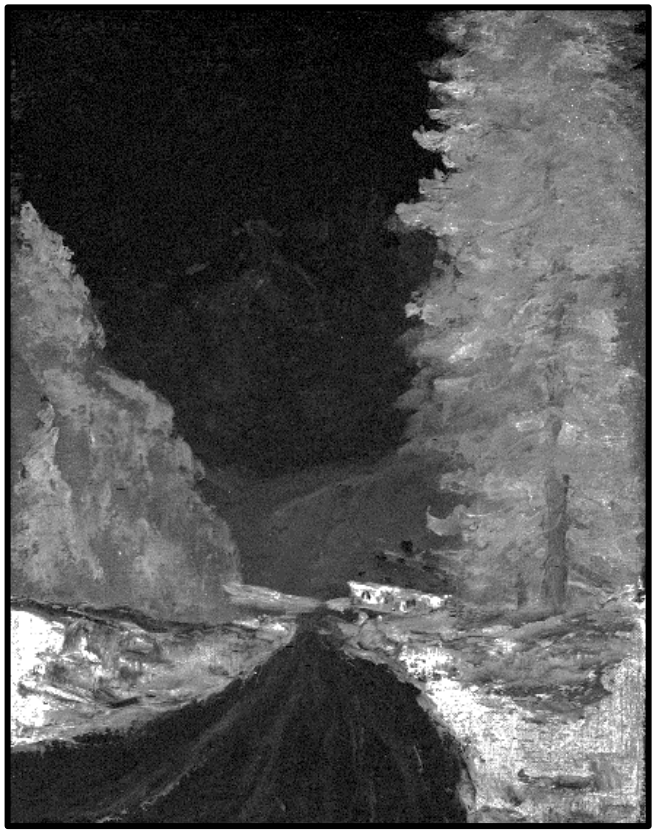


File	294 MByte		661 MByte
# of Spectra	260 kpixel		240 kpixel
Total time	1 h 35 min		48 h 32 min

From the object to the data
The measurement parameters

800 µm / 8 ms

800 µm / 700 ms



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From the object to the data
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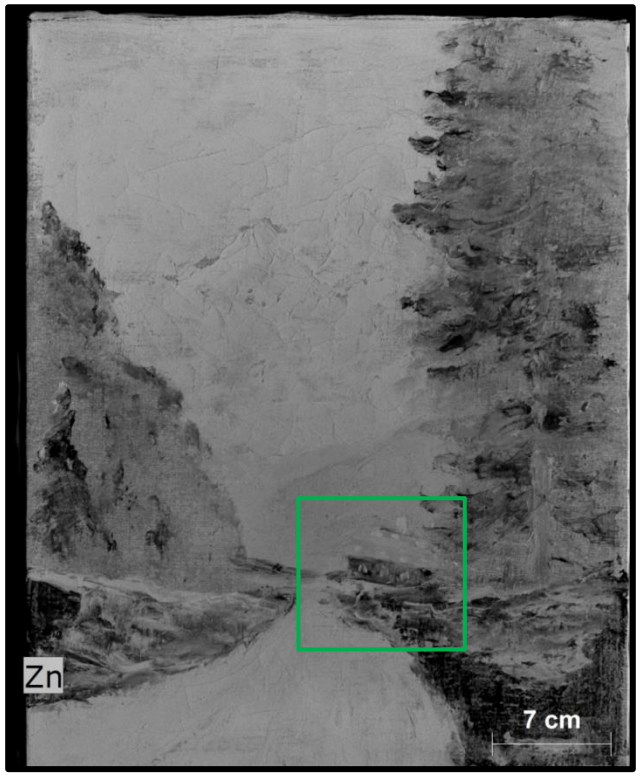
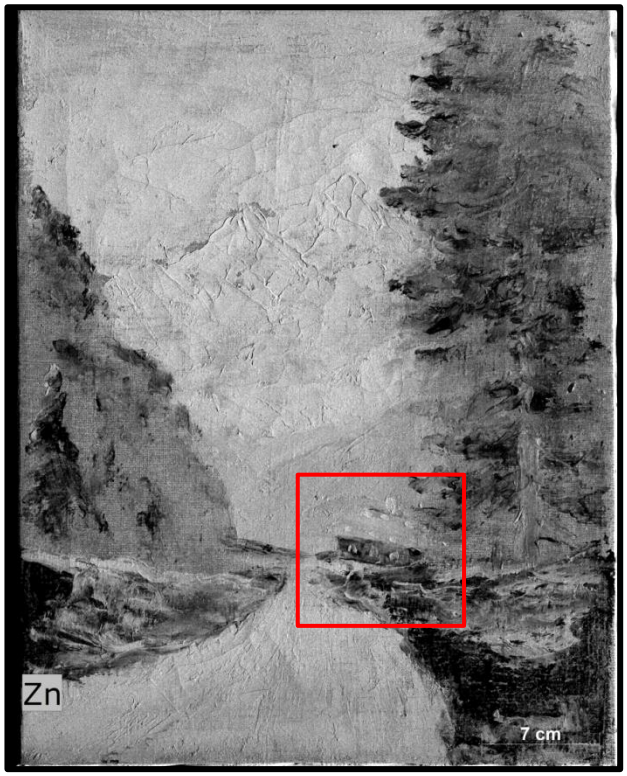
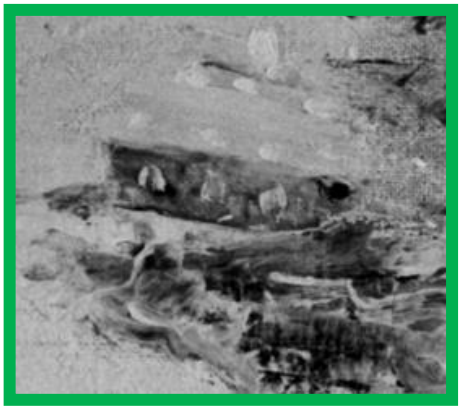


4 million



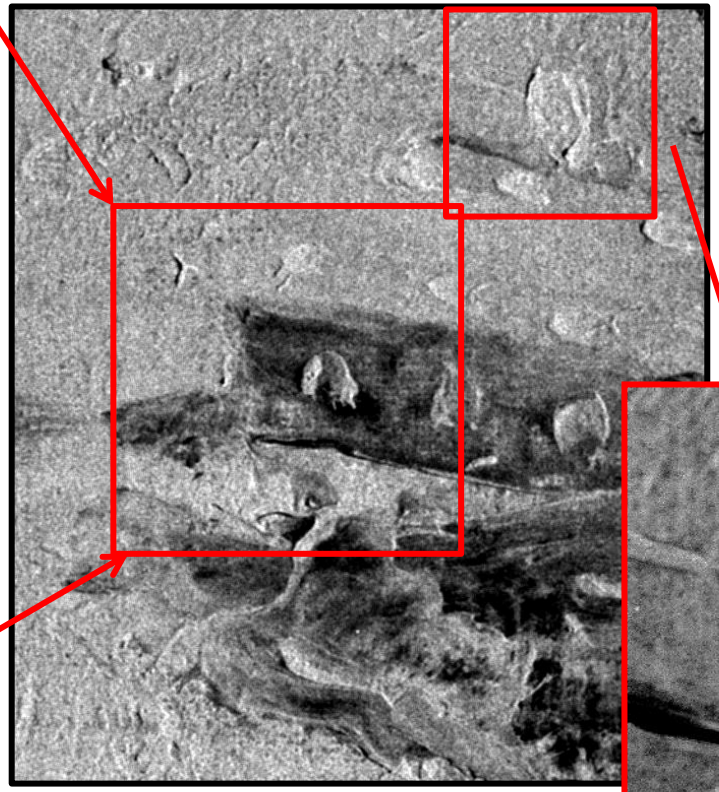
16 million



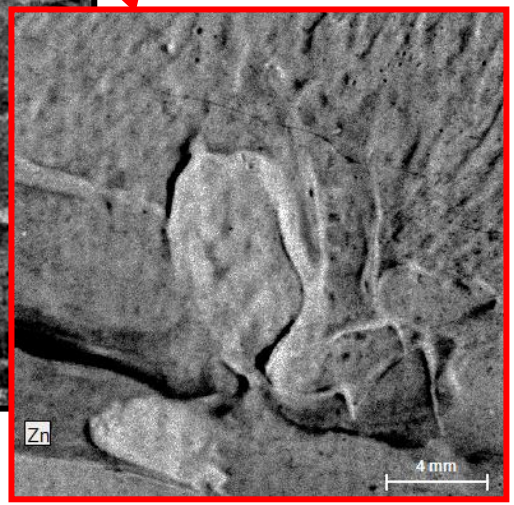
From the object to the data
The measurement parameters

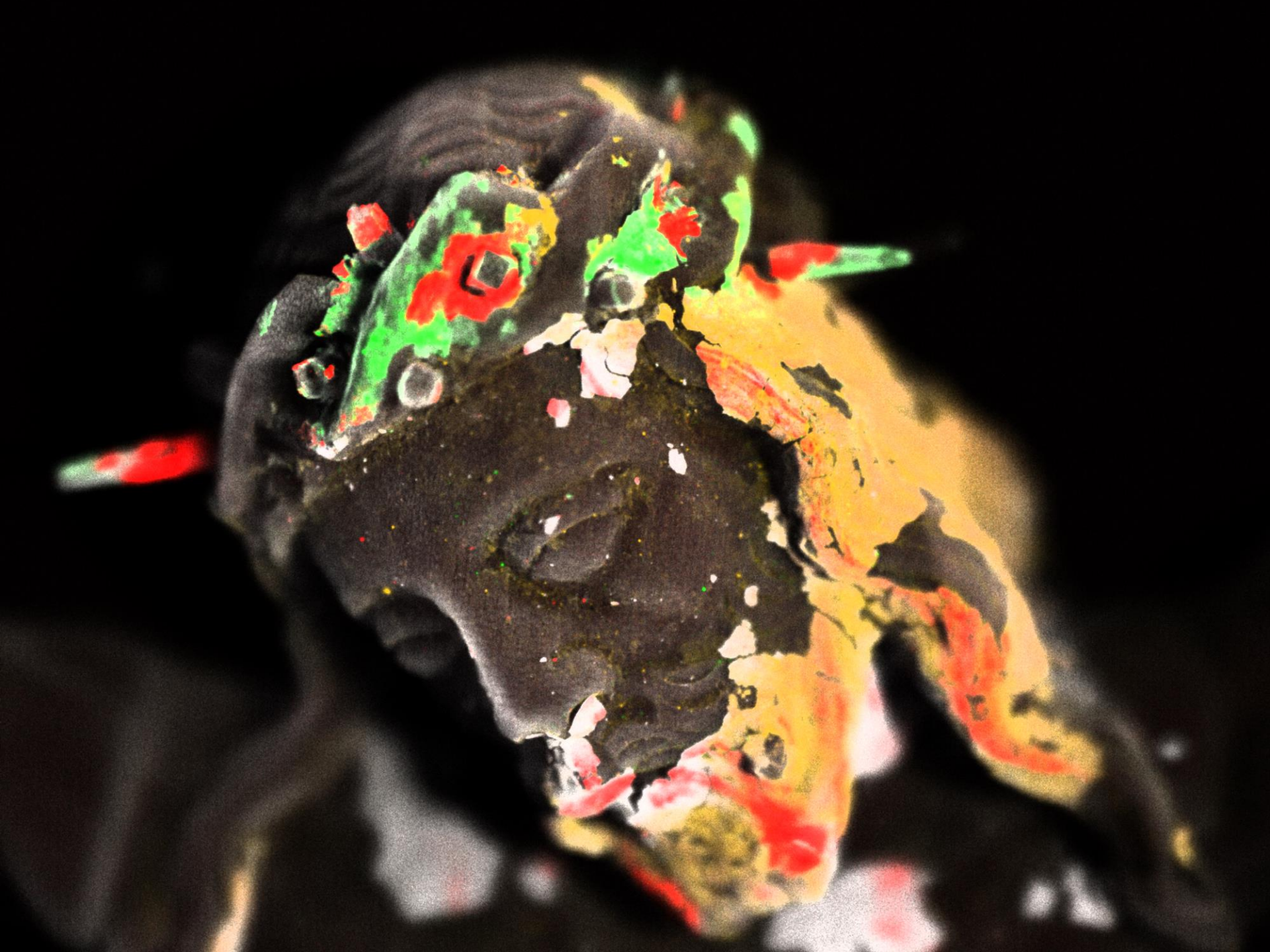
Detail measurement 1

Full scan 16 Mpixel

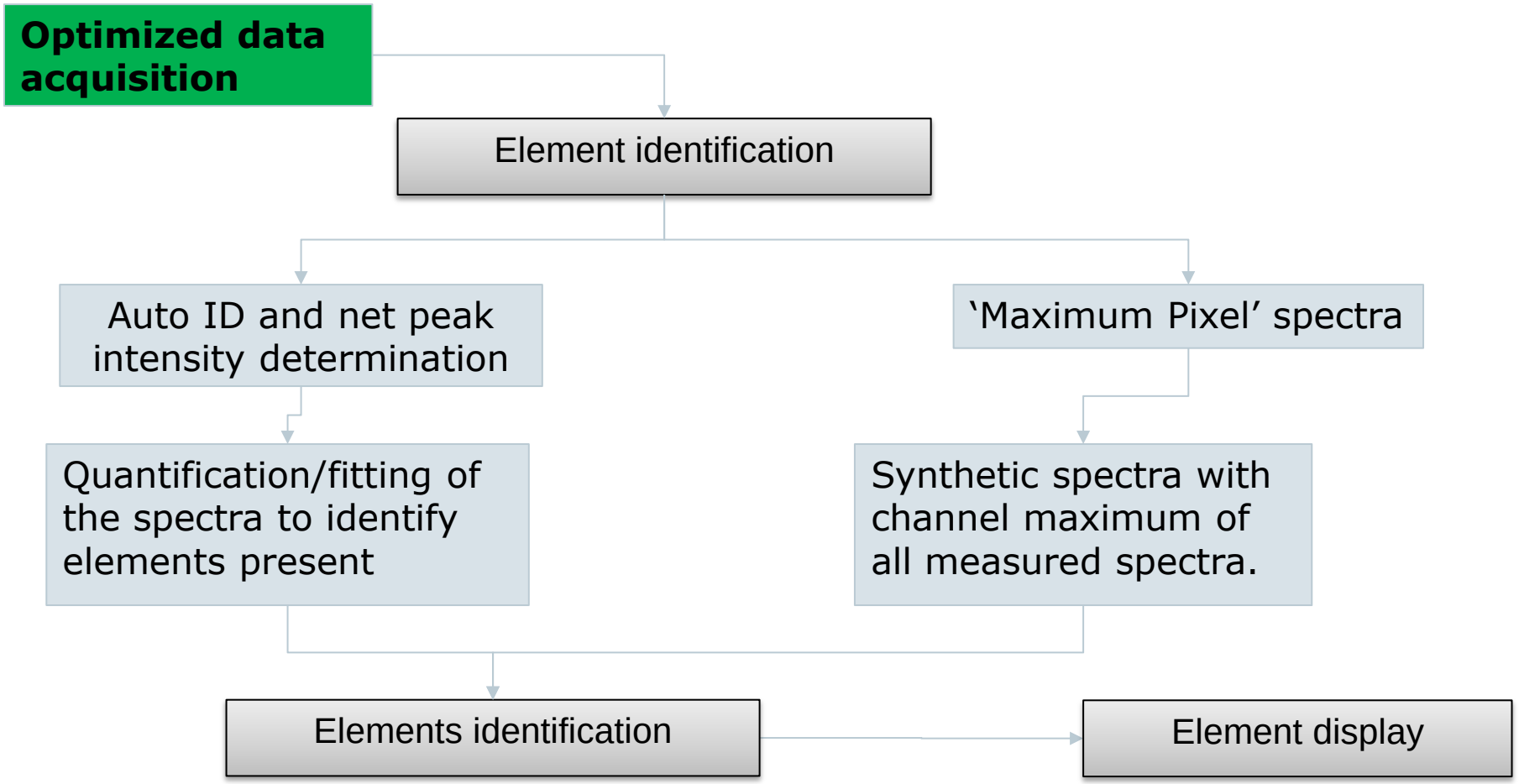


Detail measurement 2





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



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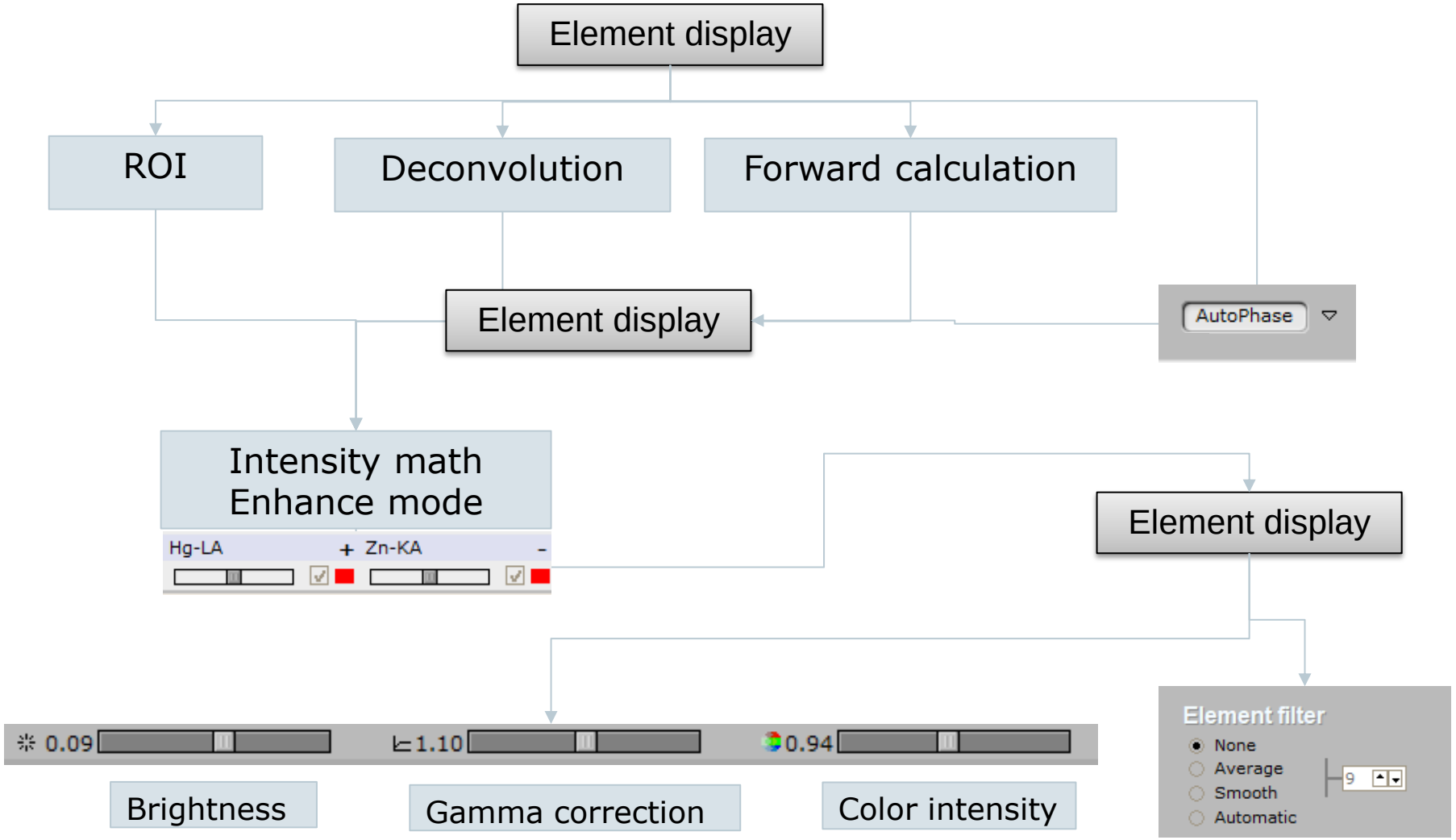
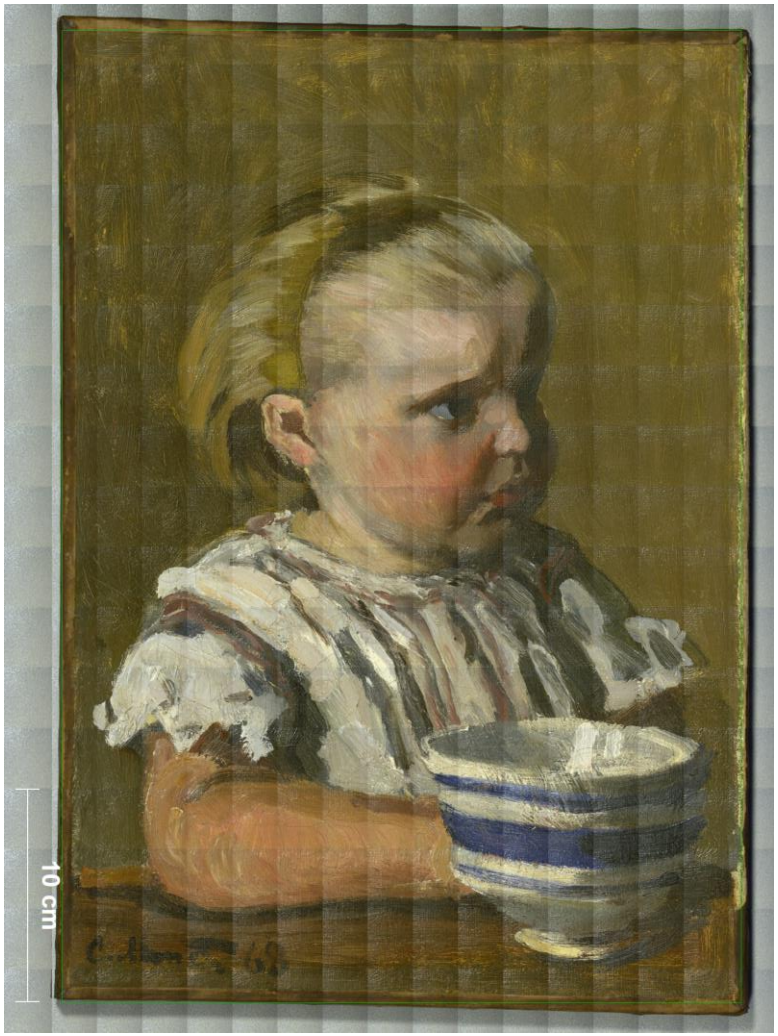


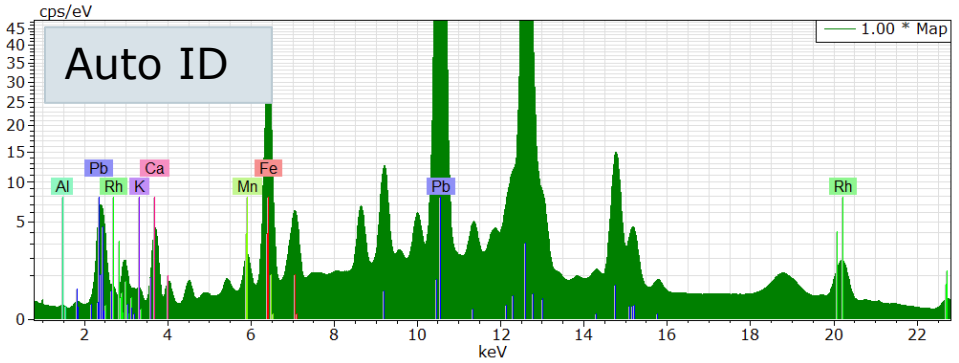
The Data:



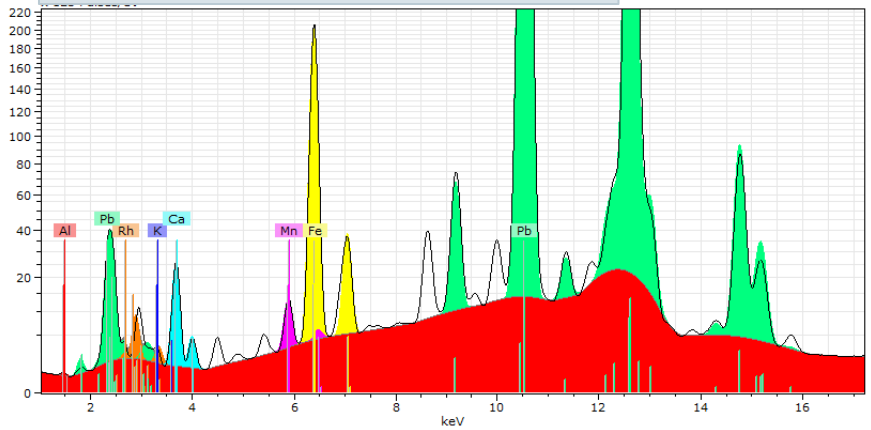
Mapping parameters		
Width:	916	pixel
	457.884	mm
Height:	640	pixel
	319.988	mm
Pixel Size:	500	µm
Total number of pixel:	586240	pixel
Acquisition parameters		
Frame count:	1	
Pixel time:	10	ms/pixel
Measure time:	1:14 h	
Overall time:	2:43 h	
Stage speed:	50.0	mm/s
Stage position (X,Y,Z):		
Tube parameter		
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

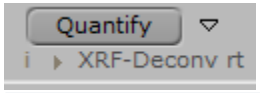
Data mining, Element identification:
Auto ID and Interactive quantification

1) The Auto ID does not identify all the elements. It based on a quantification of all possible elements.
2) Wrong identifications are possible!

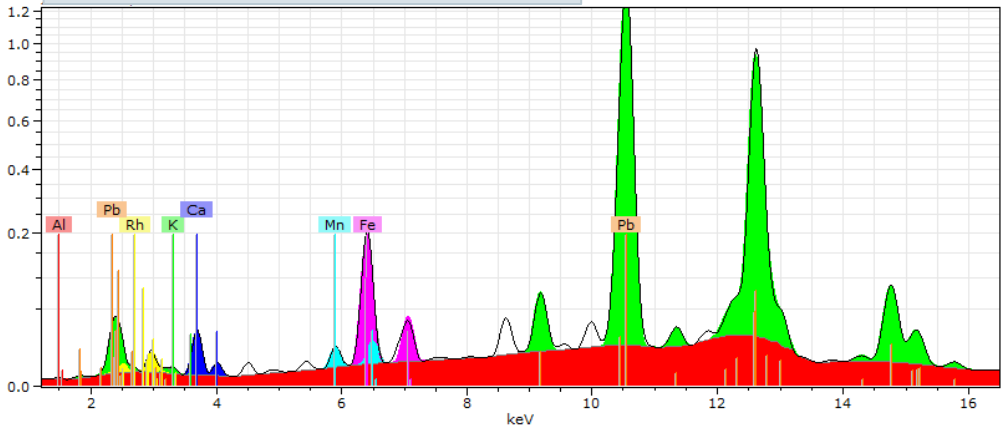


Fundamental parameter forward calculation





Bayes deconvolution

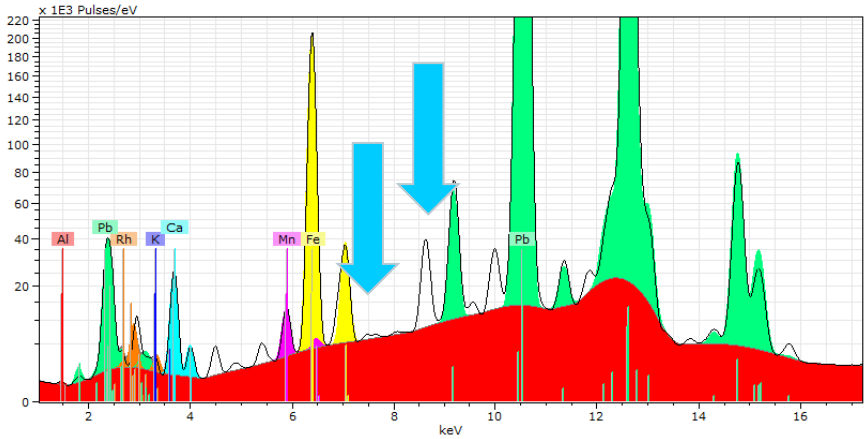


Data mining, Element identification:
Interactive quantification using FP model

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 think sample.
…which is rarely the case for a painting.

Fundamental parameter forward calculation

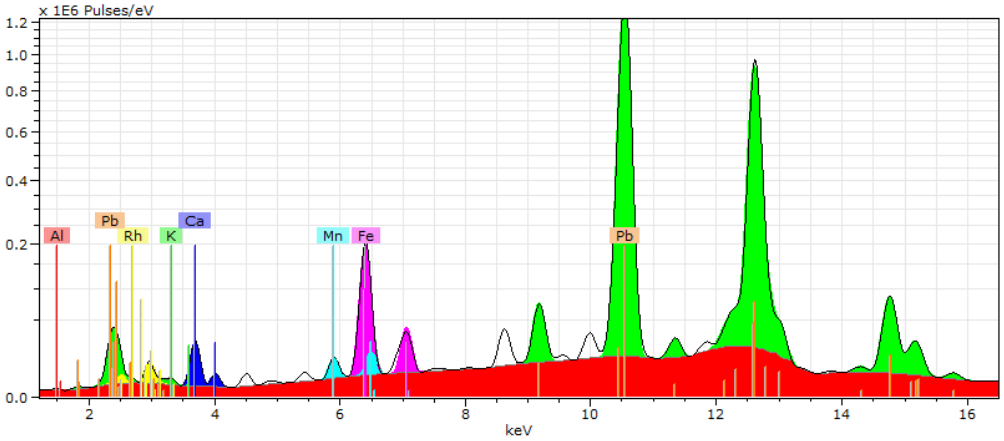


There is no forward calculated homogeneous, infinitely thick sample which produces a spectrum like the measured one. Therefore the fit cannot be perfect. However, for most of the samples the fit is surprisingly good. But problems might appear especially in the low energy range!

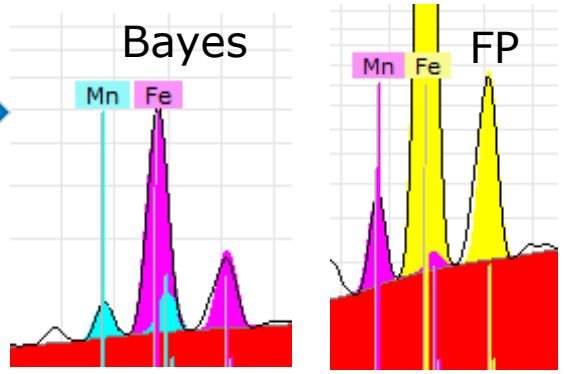
Data mining, Element identification:
Interactive deconvolution using Bayes

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

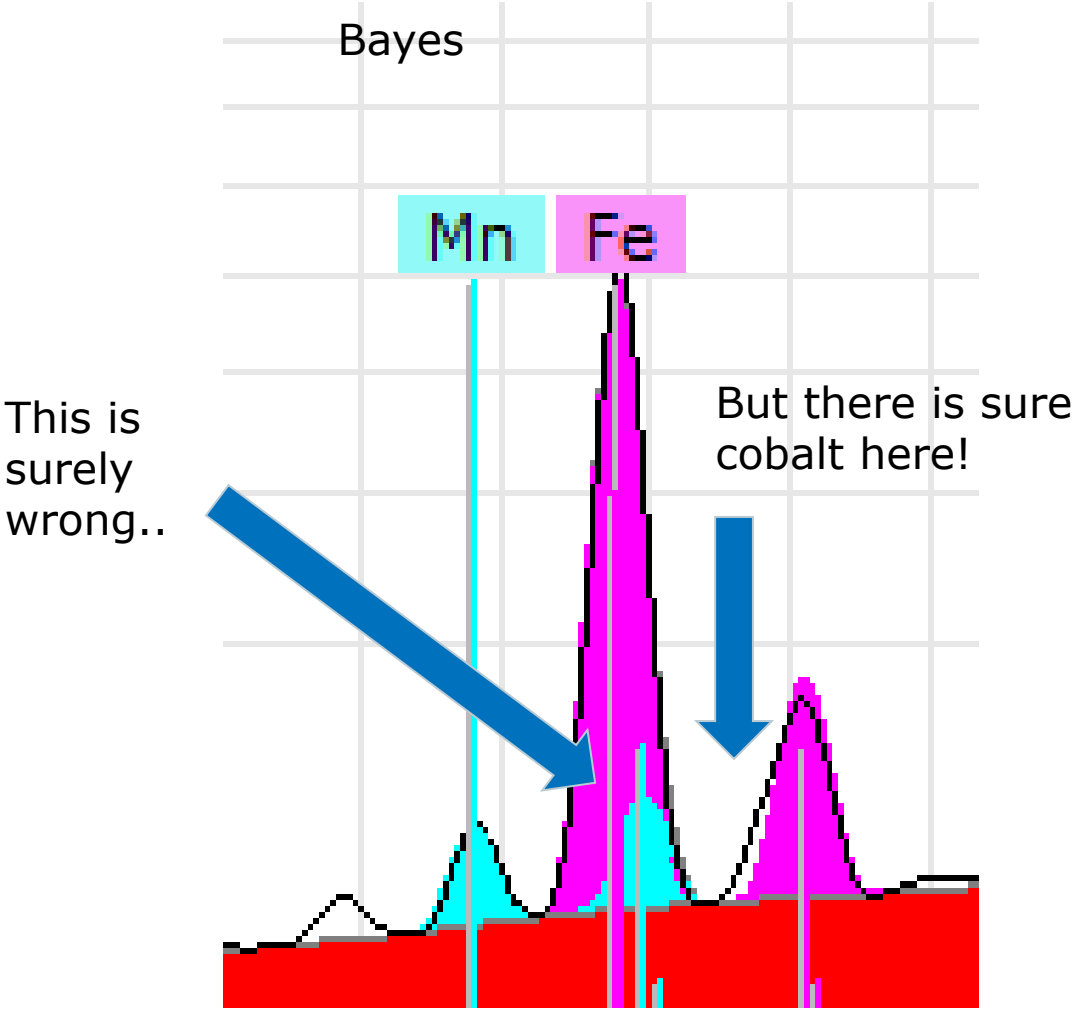


The Mn Kb line is overestimated in the Bayes deconvolution.



There is no correct solution, there are different tools, which have their pro and contra…

Data mining, Element identification:
Interactive deconvolution using Bayes



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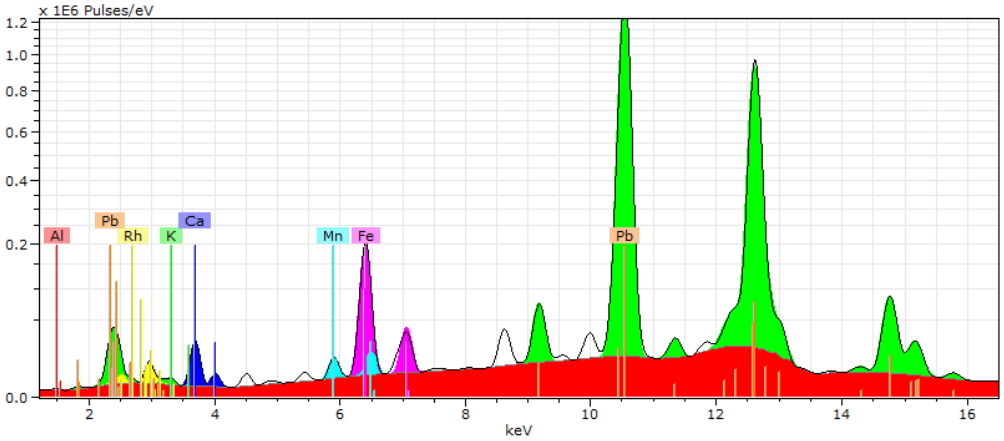
Bayes deconvolution



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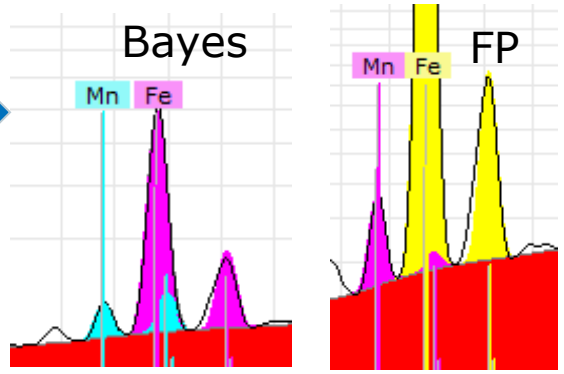

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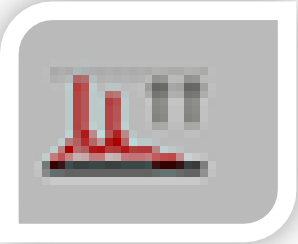
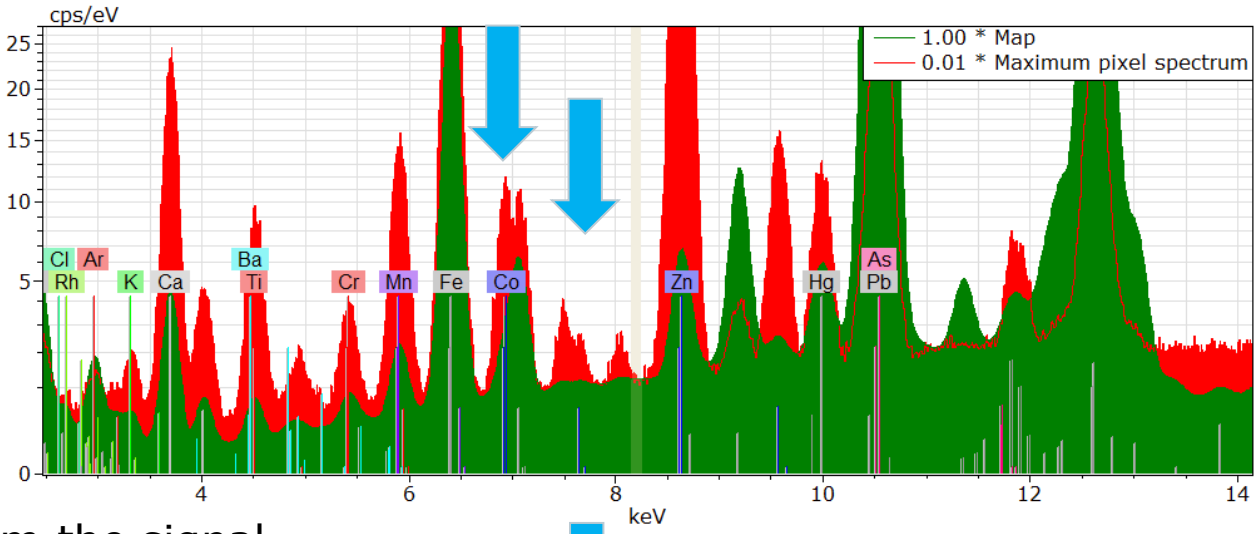
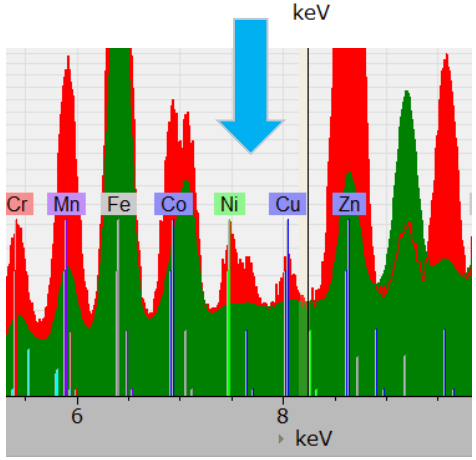
Data mining, Element identification:
Maximum Pixel spectra





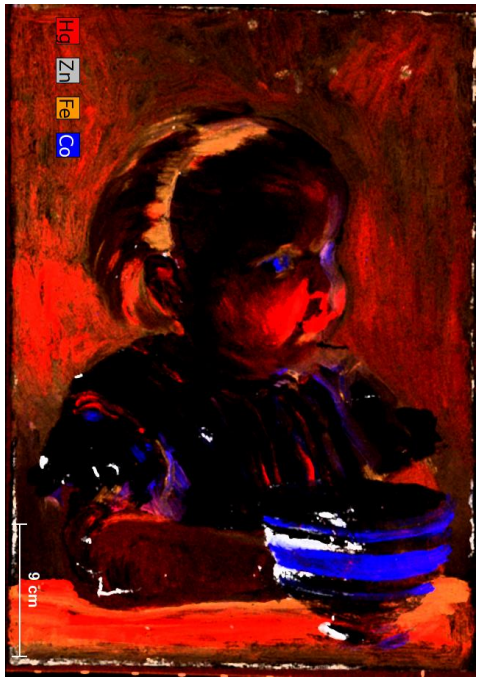
In the map spectrum the signal for Ni and Cu is "diluted" by the large number of spectra.
The Maximum pixel spectrum reflects the highest intensity per channel found in any pixel of the map.
Maximum pixel spectrum can be used to find hot spots in the data block.

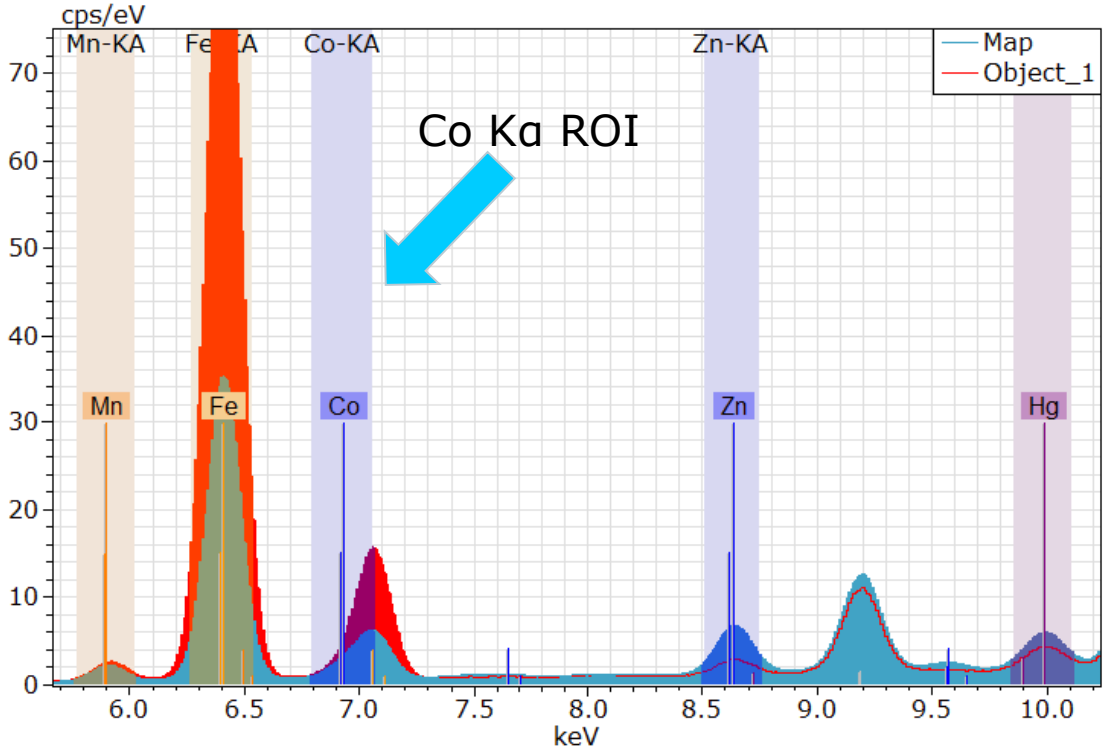


Easy identification of Ni and Cu presence somewhere in the sample.

An even the cobalt is no 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 Kβ from Fe.

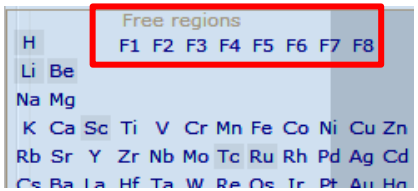
Extremely fast and robust!

Data mining, Element display:
Region of interest ROI

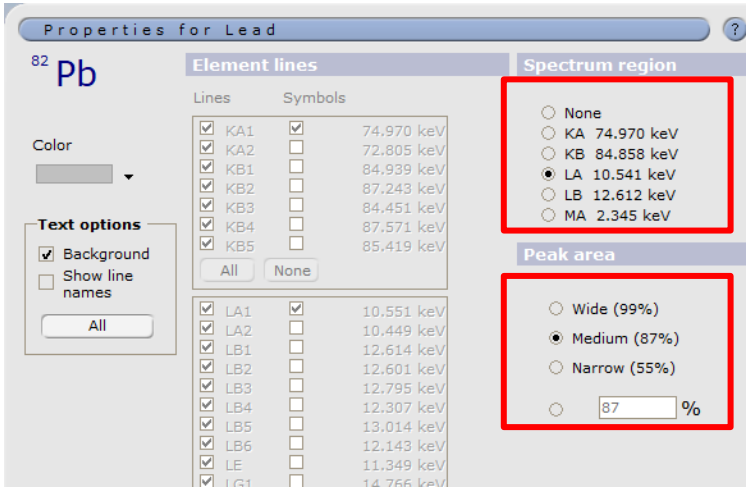
In the periodic table (right mouse on the element) it is possible to edit the lines that shall be used for the element display.

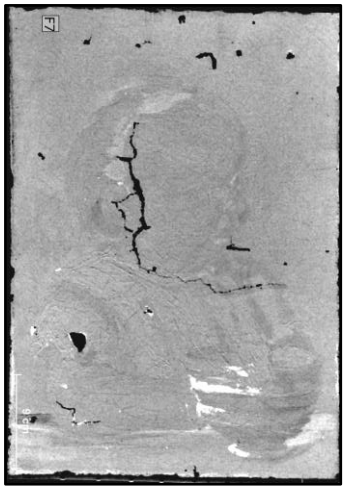
The Line as well as the width of the Region around the peak can be edited.

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.



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

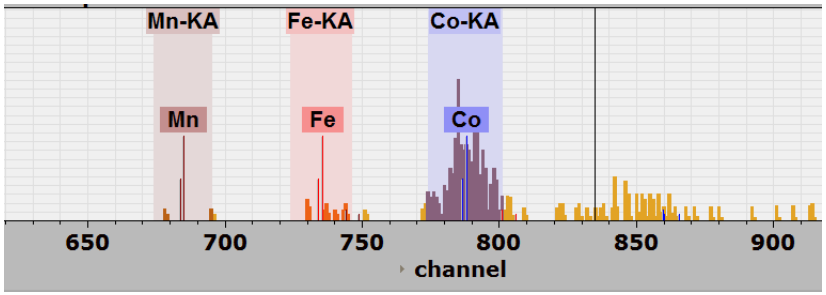




Total intensity

Compton

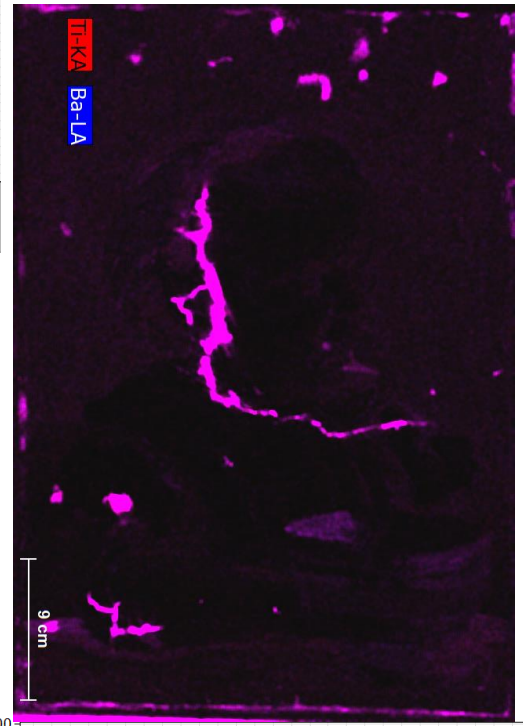
Data mining, Element display:
Fast deconvolution



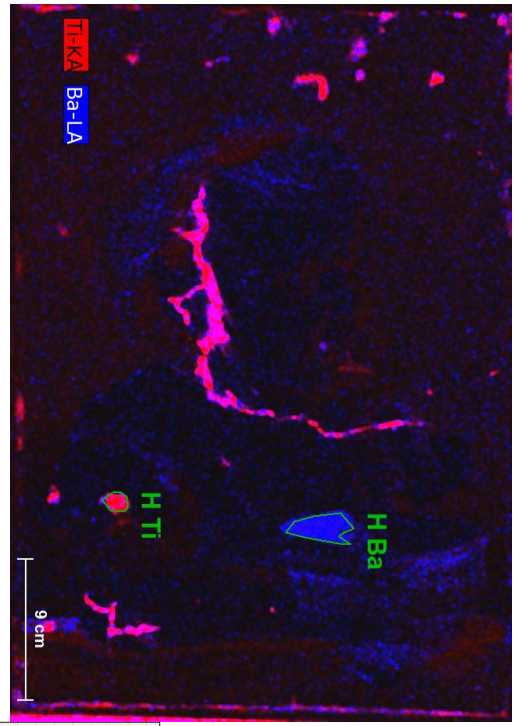
In the fast deconvolution (which is a fit) every count in every channel is weighted by the probability that a it belongs to one of the selected elements. As soon as this is calculated for each channel the complete data set is evaluated at high speed.

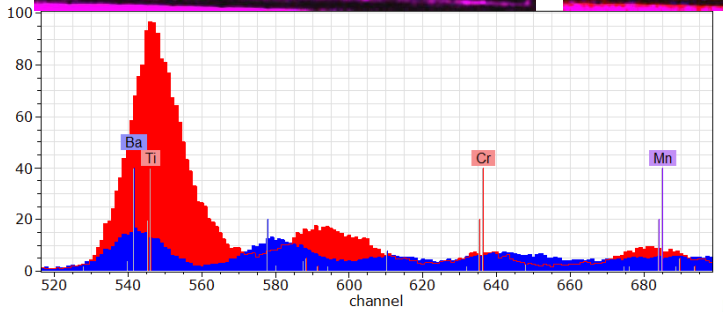
In this case 17 elements and half a million spectra were 'deconvoluted' in 30 seconds.

ROI

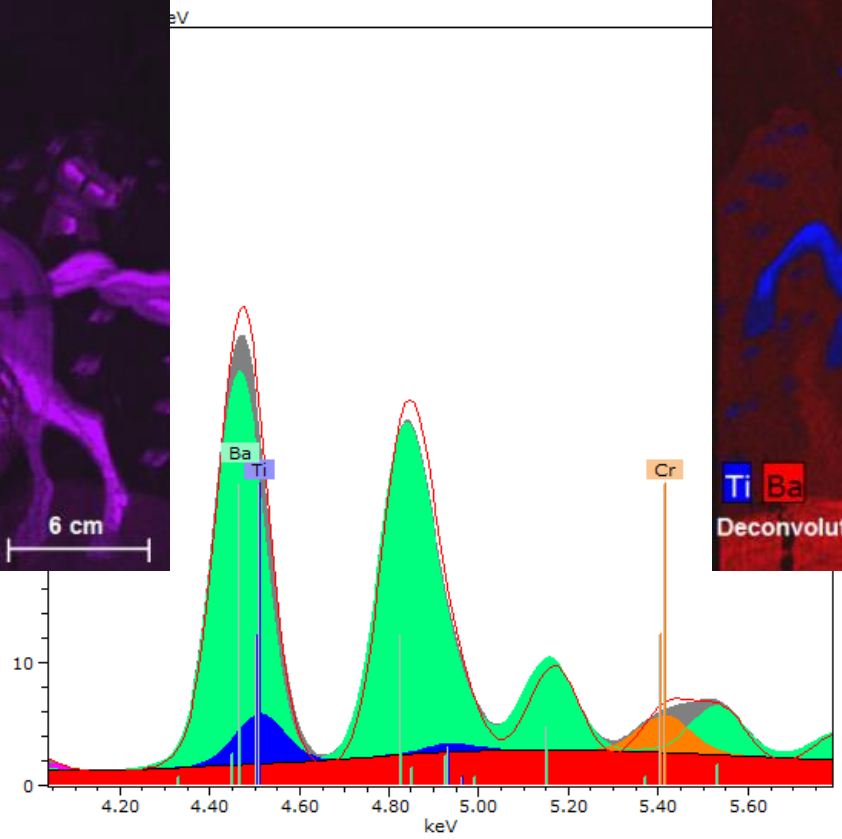


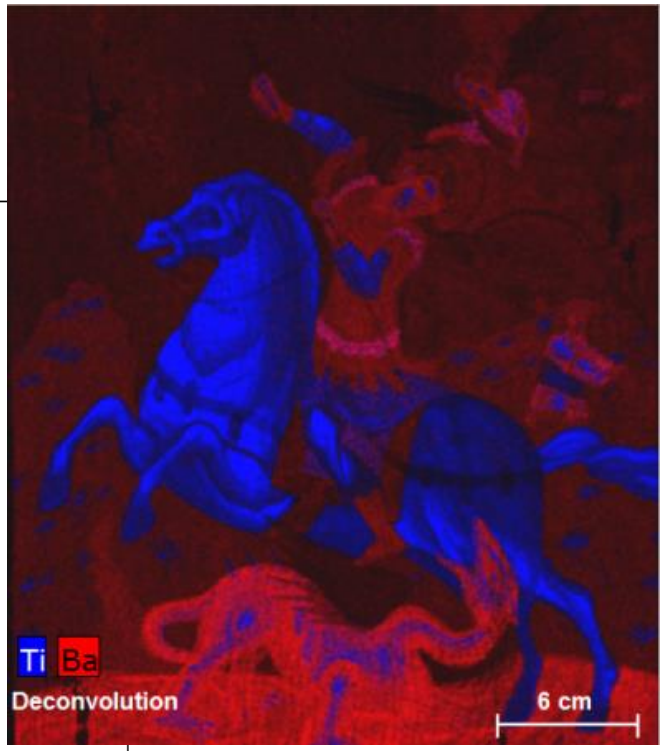
Fast deconvolution



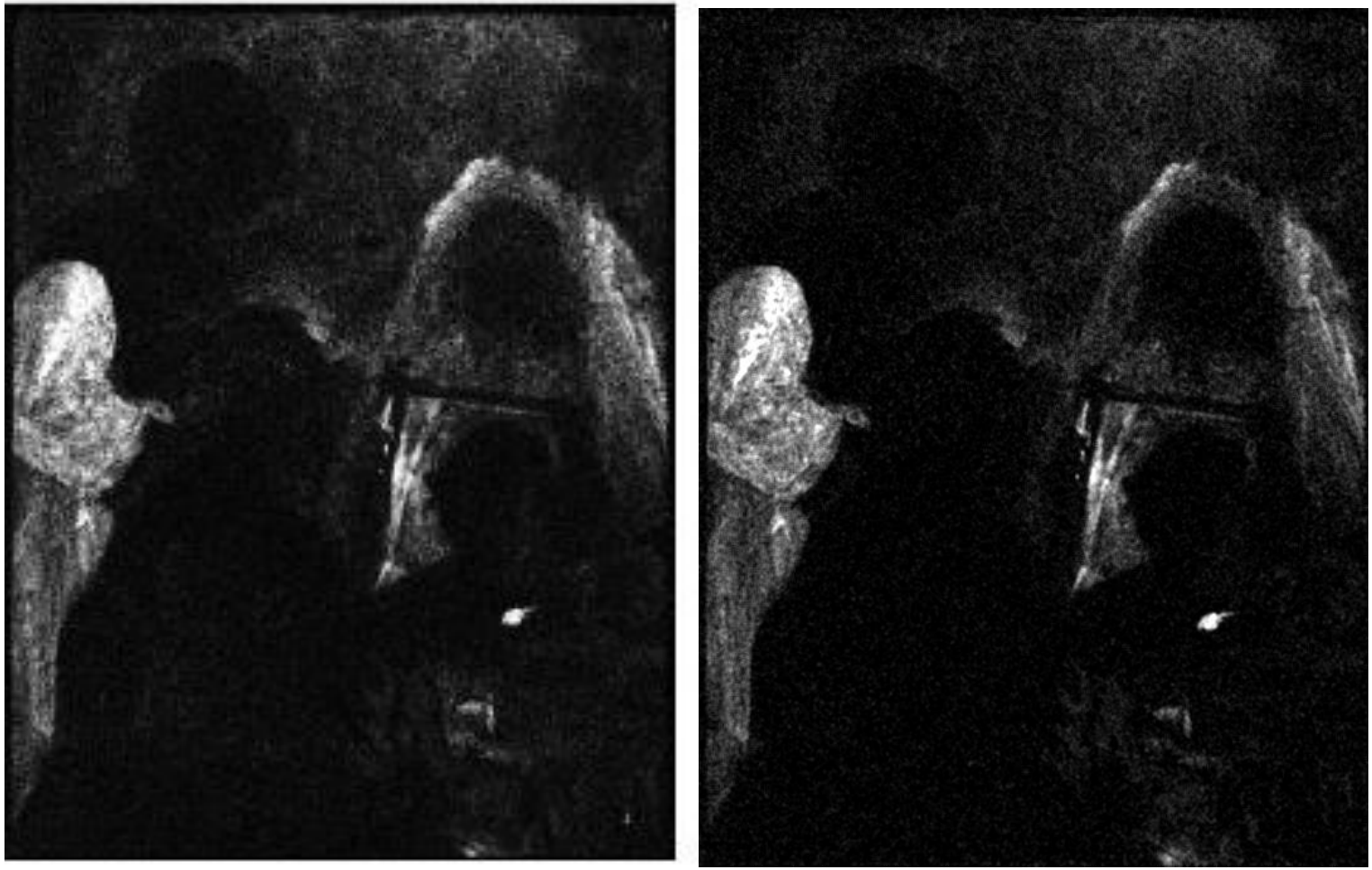


Data mining, Element display:
Fast deconvolution





Data mining, Element display:
Fast deconvolution



Data mining, Element display:
Fast deconvolution

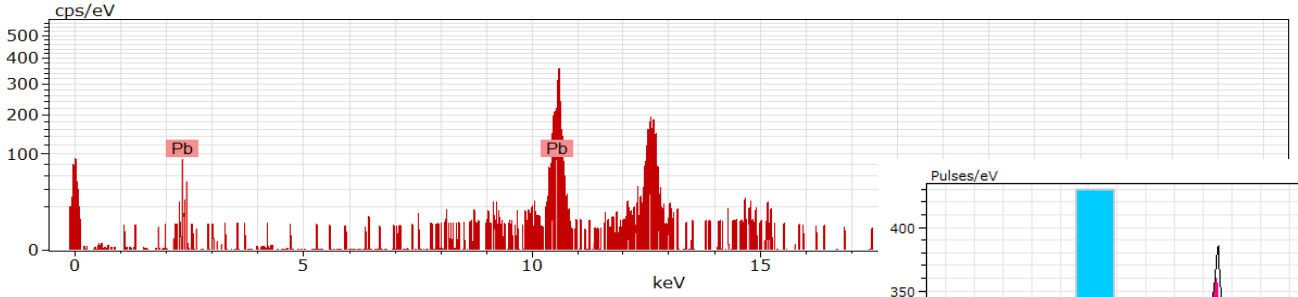
Processed by M. Alfeld

M6 Jetstream deconvolution

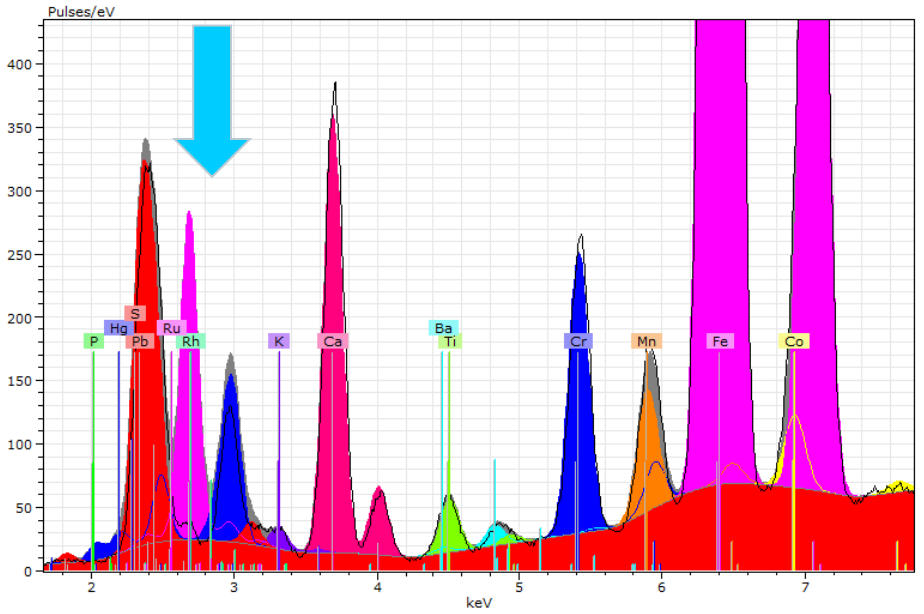
Data mining, Element display:
Forward calculation I

A 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.

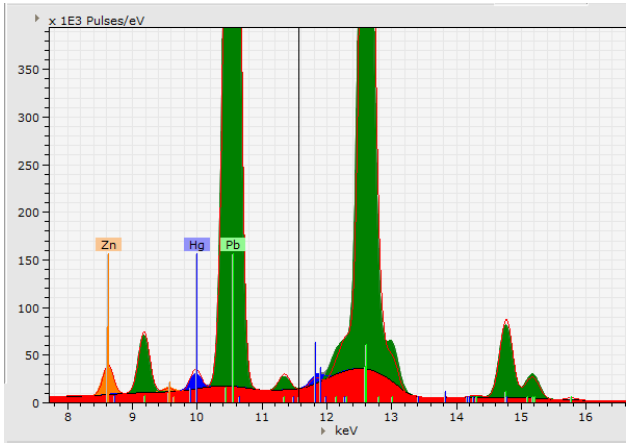


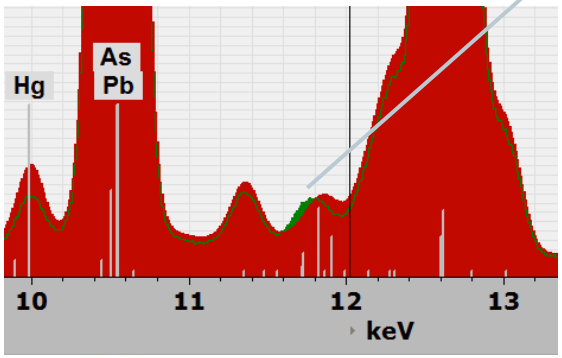
Best numerical solution might not be the best fit, as a non-ideal sample has no "correct" solution for the Sherman Equation



Data mining, Element display:
Forward calculation

Convolution

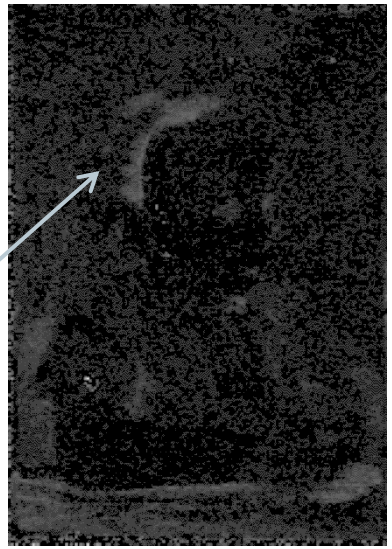




Multiple overlapping's Hg-Pb-As

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

As



Pb

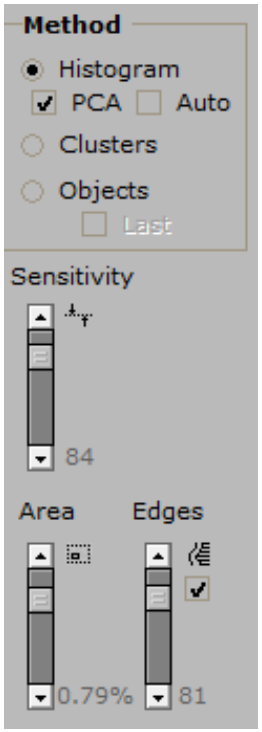
Hg

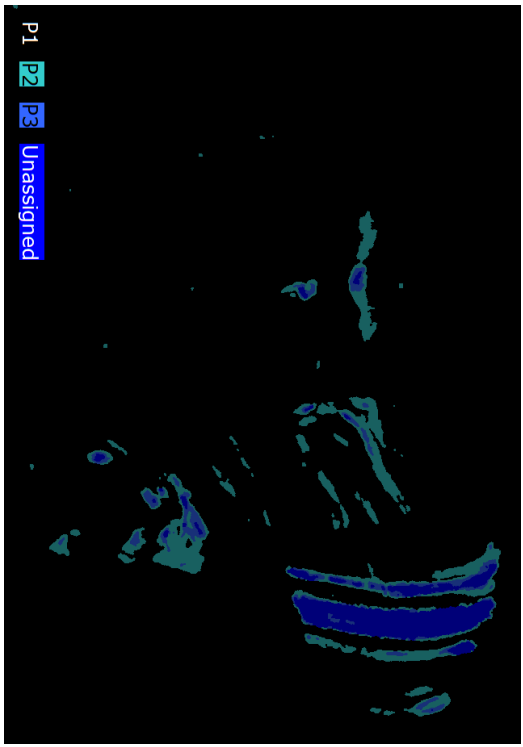
Data mining, Element display:
Phase analysis

Phase analysis is based on an algorithm that compares the intensity of a ROI in the spectra (element or selected free region) with all the other spectra in the data block and tries to find similarities. The Phase analysis can also be done by finding areas with similar spectra to a pre-selected object.

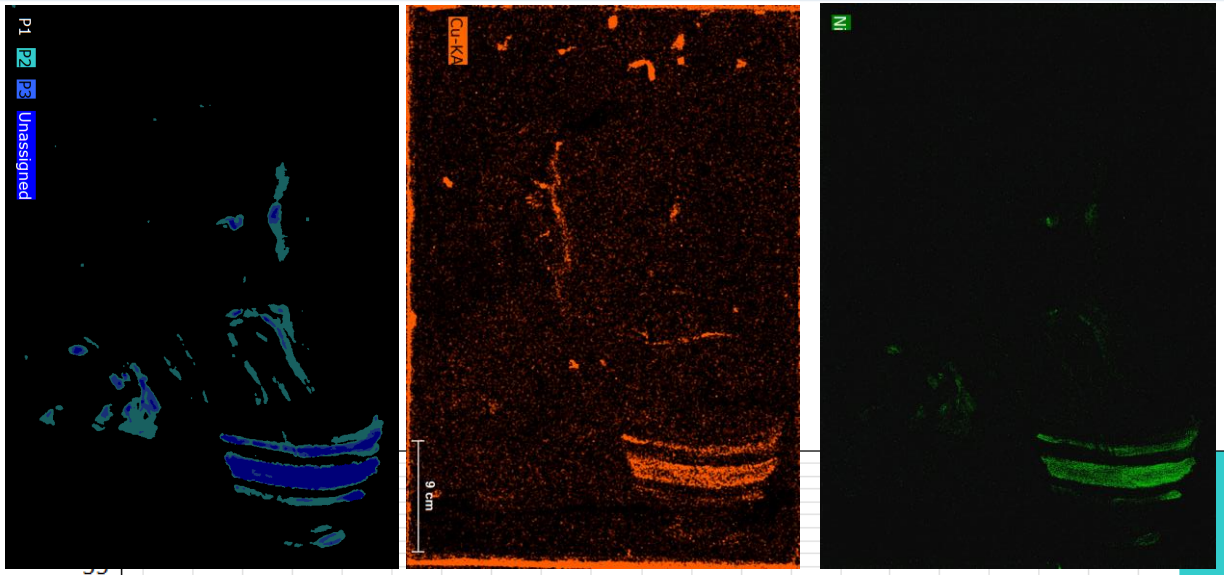
Recommended use e.g.: What trace element are associated to selected other elements e.g. Co.

Identified phases ‚P3' and ‚Unassigned' correlate to the Co image.

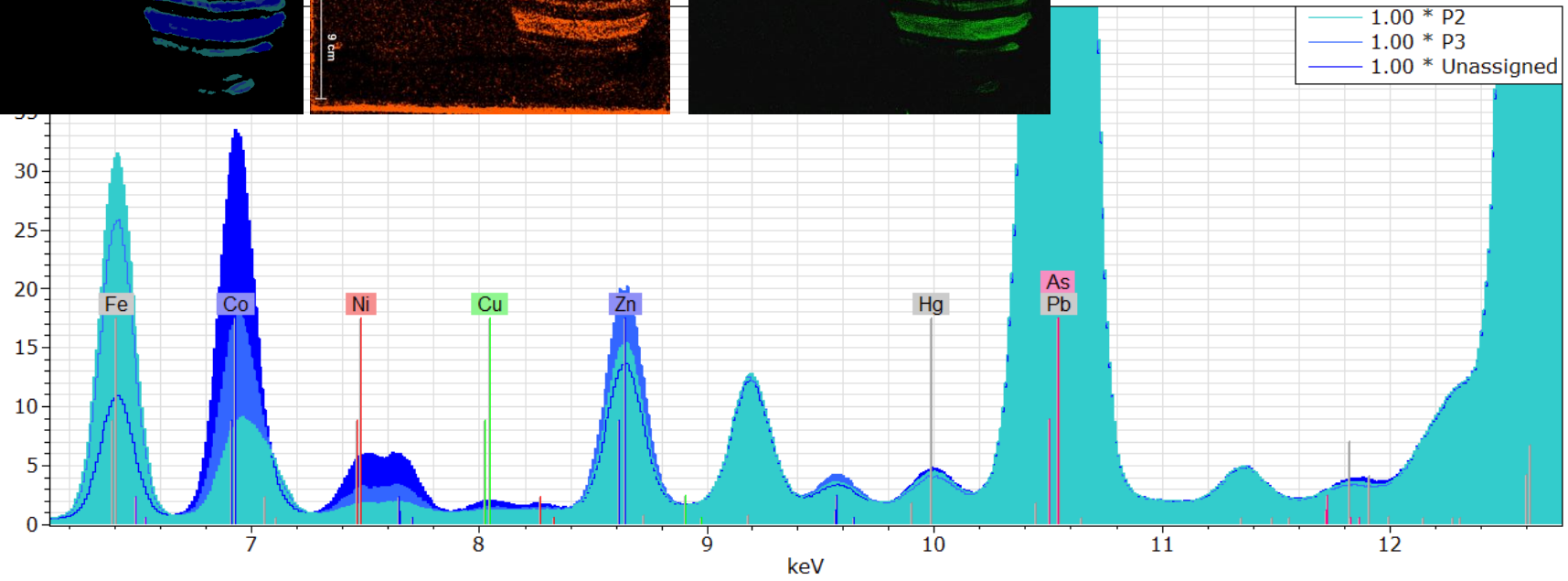




Data mining, Element display:
Phase analysis



The cobalt pigment, is associated to the presence of traces of Ni and Cu. Where the Ni comes only with the Co the Cu is also found on other locations of the painting.

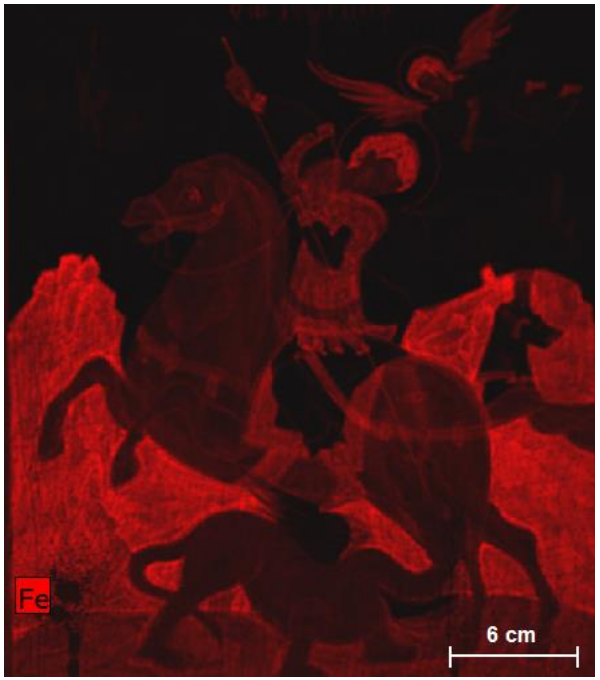


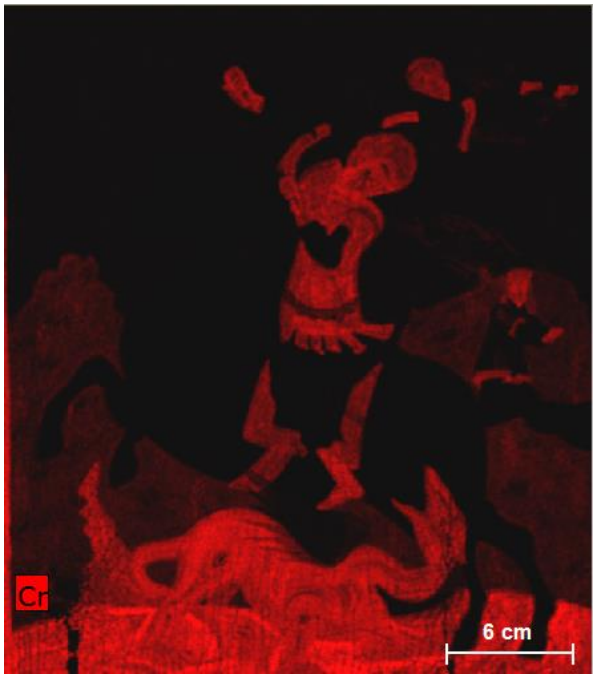
Data mining, Element display:
Enhance mode

Peak mathematics

Very fast, easy to implement, plenty of option

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





Data mining, Element display:
Enhance mode

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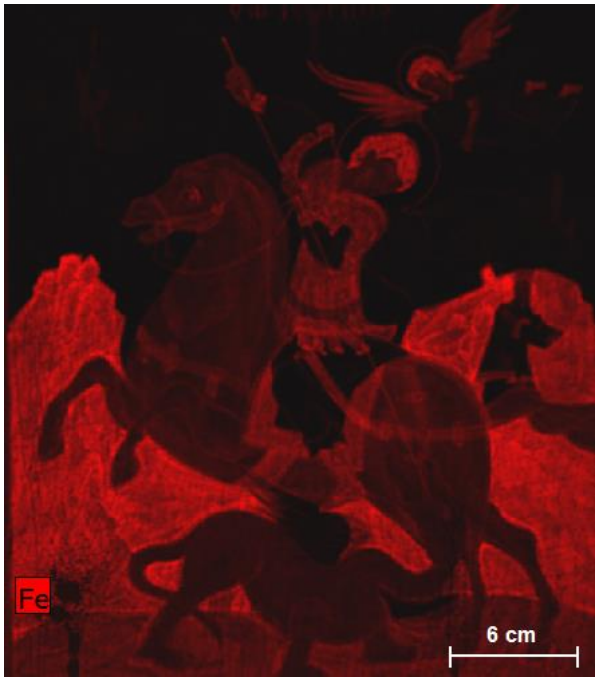

Data mining, Element display:
Enhance mode

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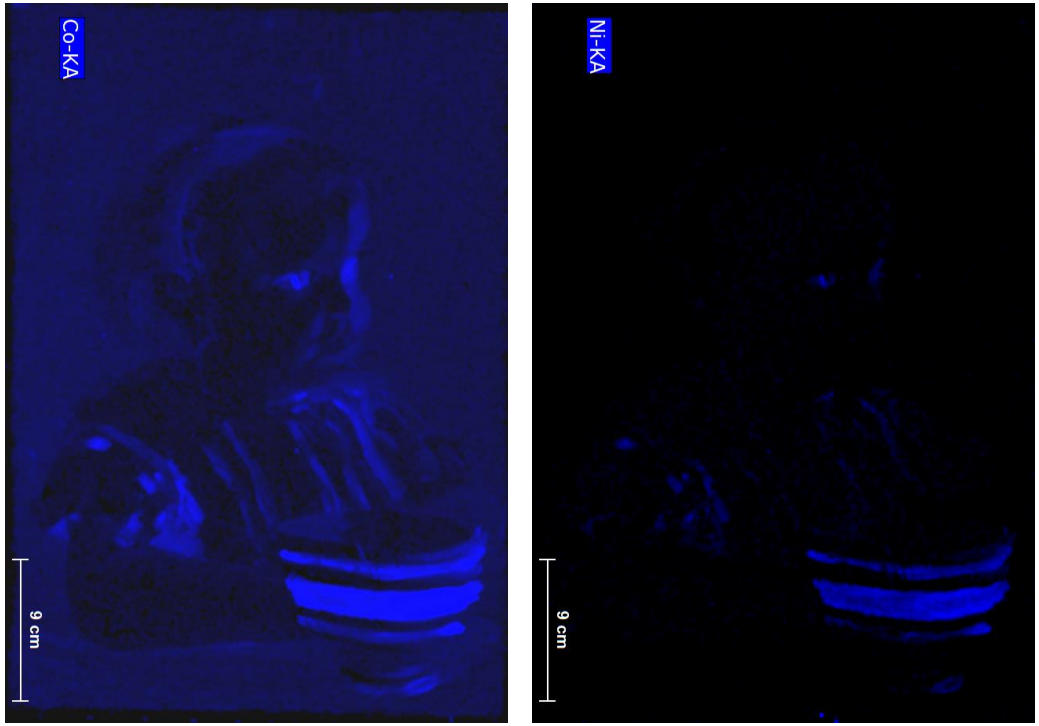

Data mining, Element display:
Enhance mode

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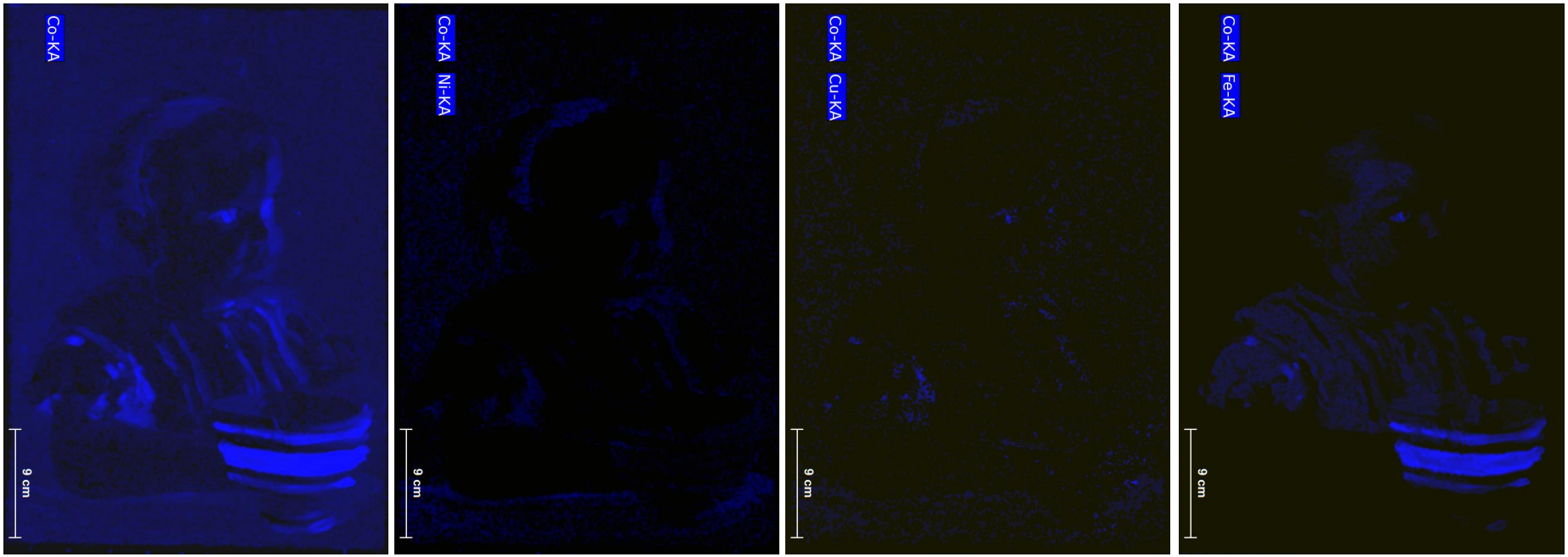


Co minus Ni

Good correlation

Co minus Cu

No corr. in the eyes

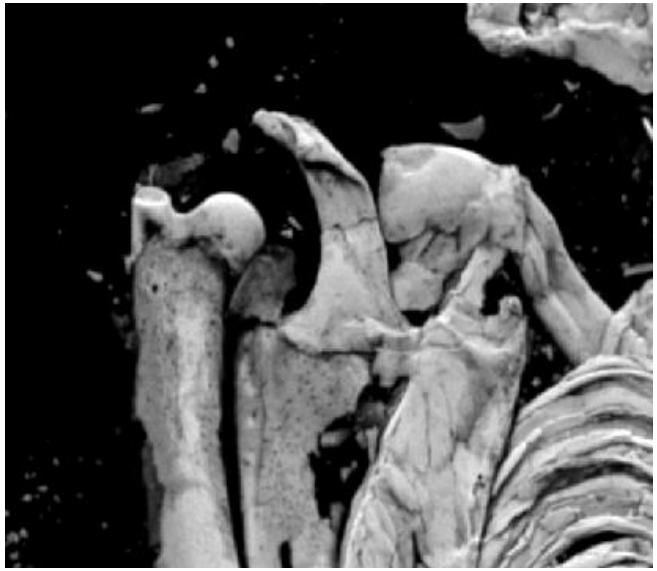
Co minus Fe

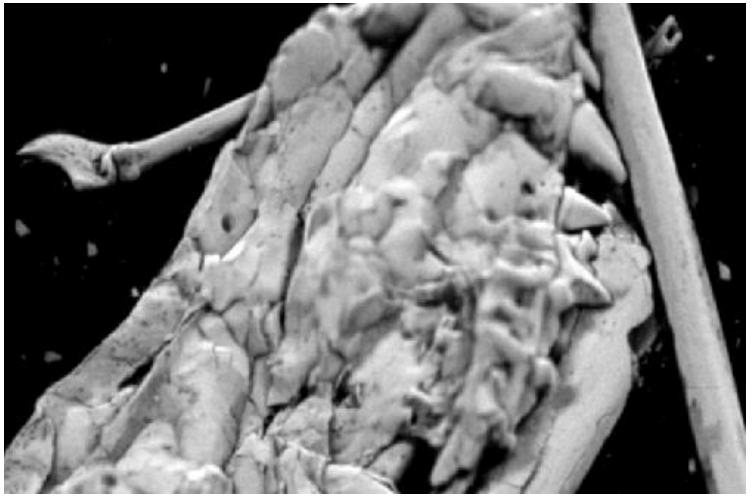
No correlation

Data mining, visualization:
Image parameters

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

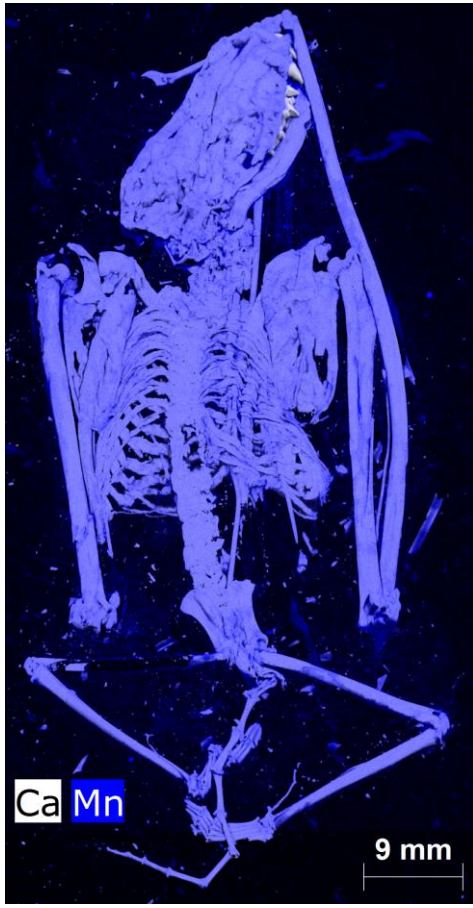
Data mining, visualization:
Image parameters, fossil bat with tissues





14 h scan with M4 Tornado
25 µm step size
5.6 million pixel
8 ms

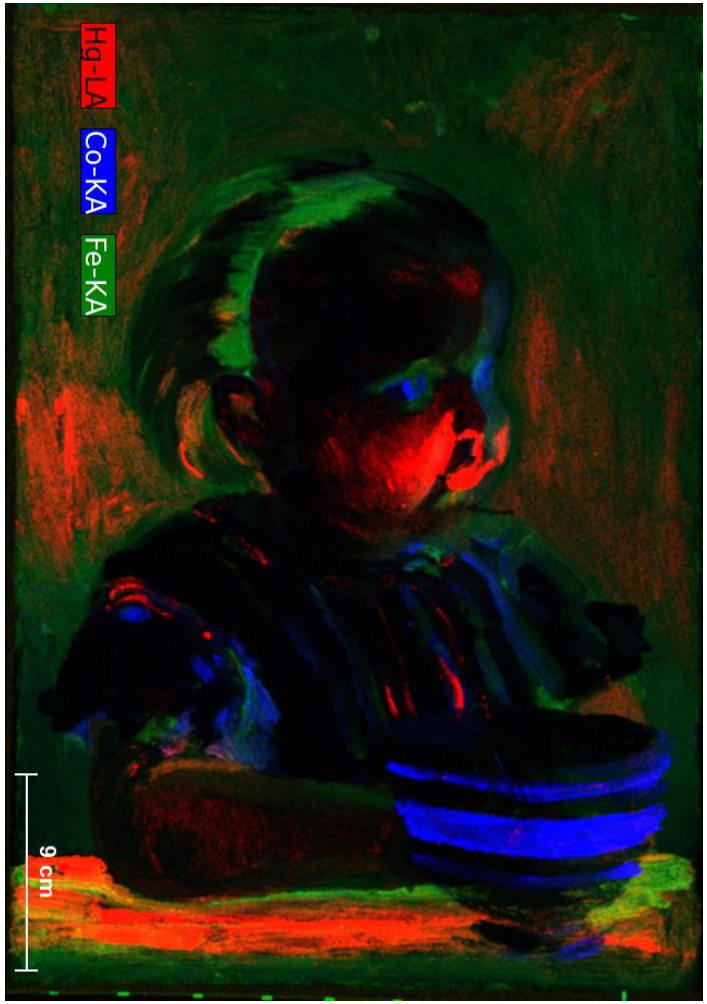
Data mining, visualization:
Image parameters, fossil bat with tissues

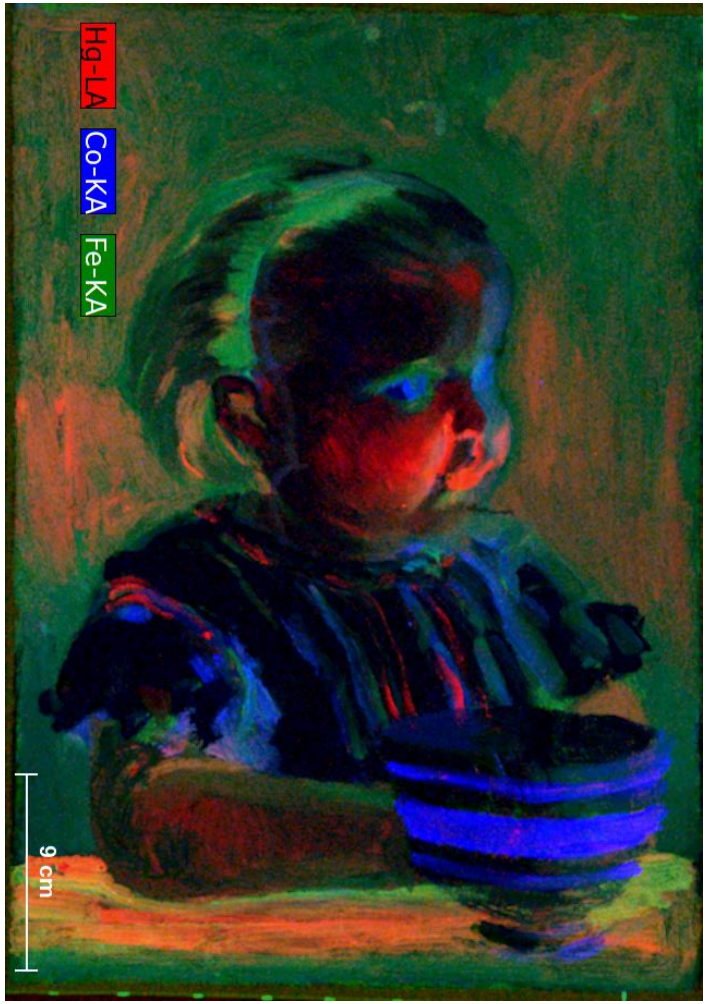


Increasing colour intensity

Combined to highlight Mn regions

Data mining, visualization:
Image parameters



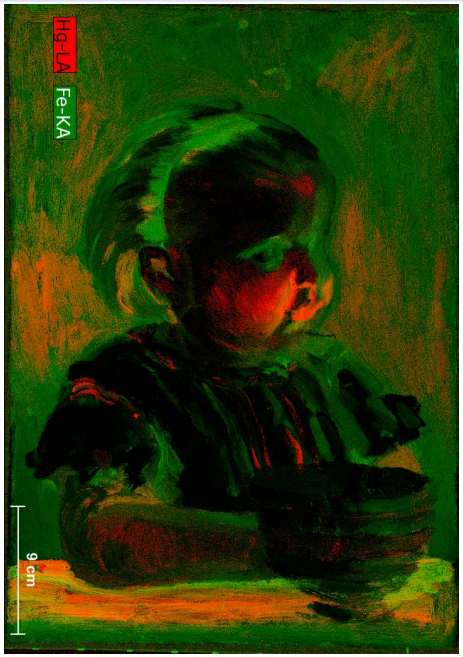


Gamma correction

Brightness

Color intensity

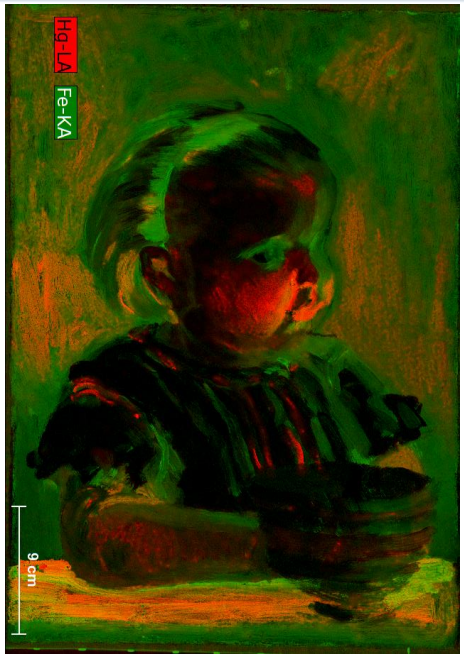
Data mining, visualization:
Element filter



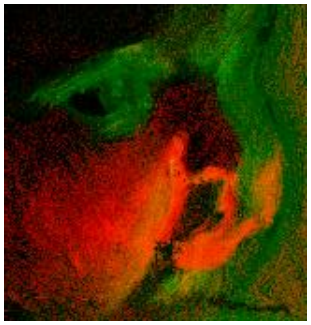
None

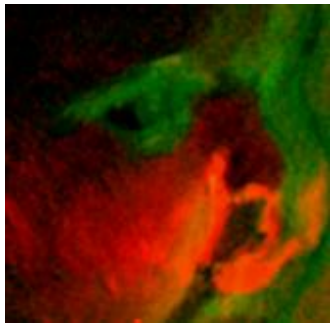
Average 3

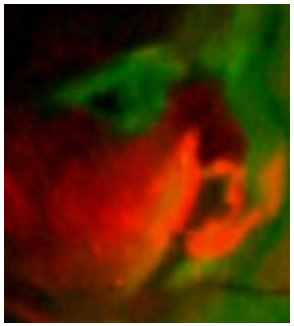
Smooth 3



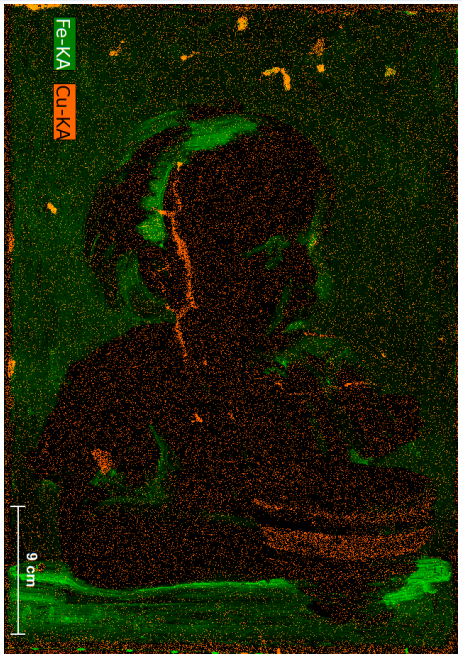
Automatic



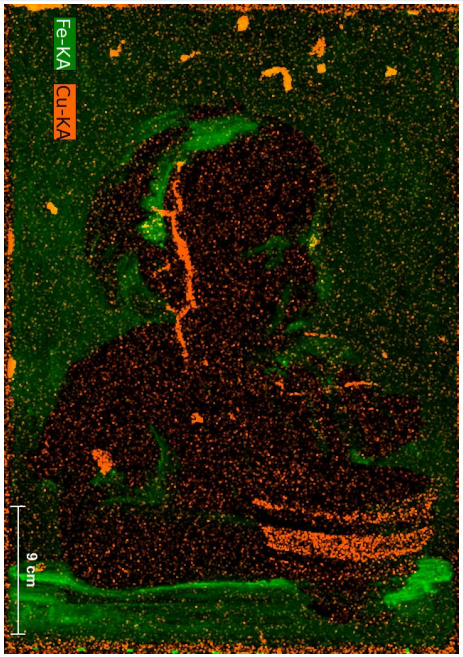




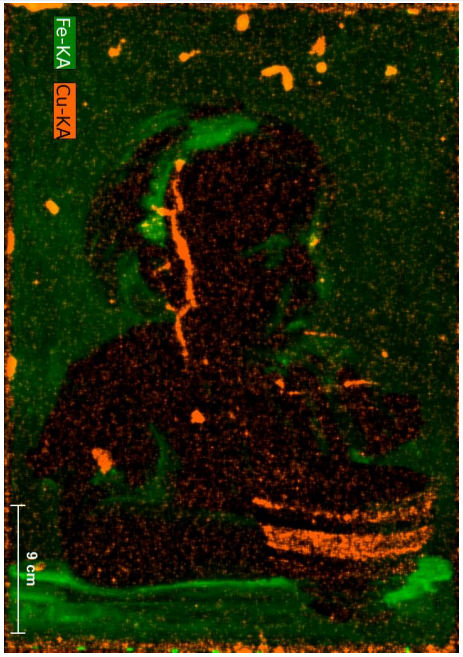
Data mining, visualization:
Image parameters



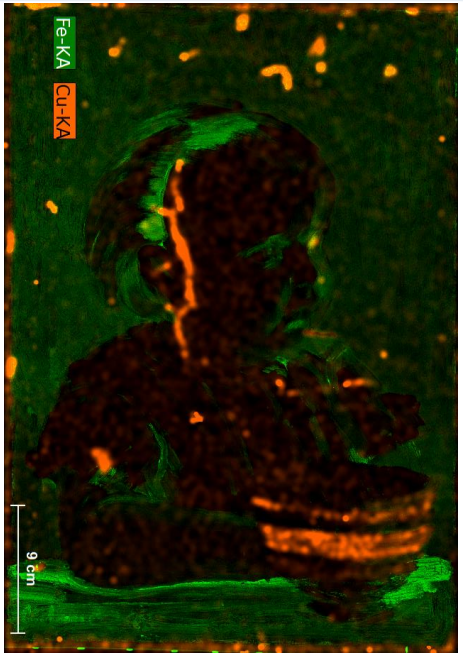
None



Average 3



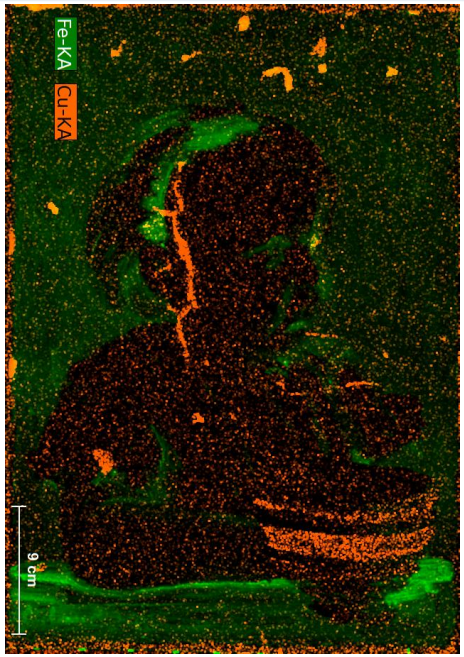
Smooth 3



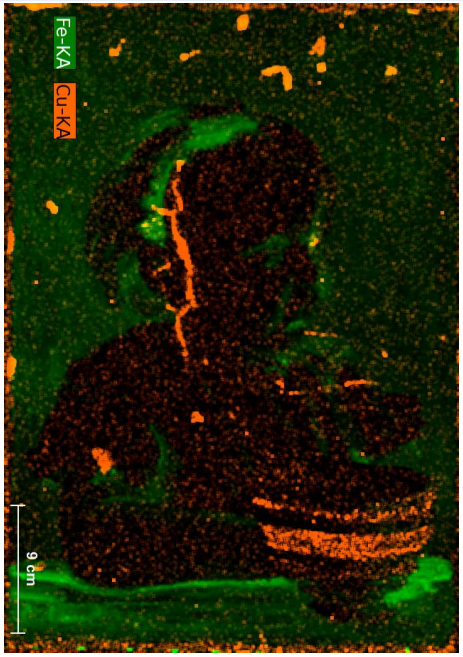
Automatic

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.

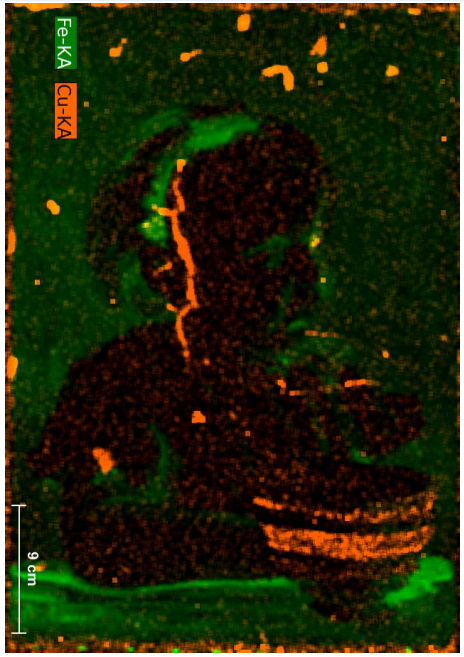
Data mining, visualization:
Image parameters



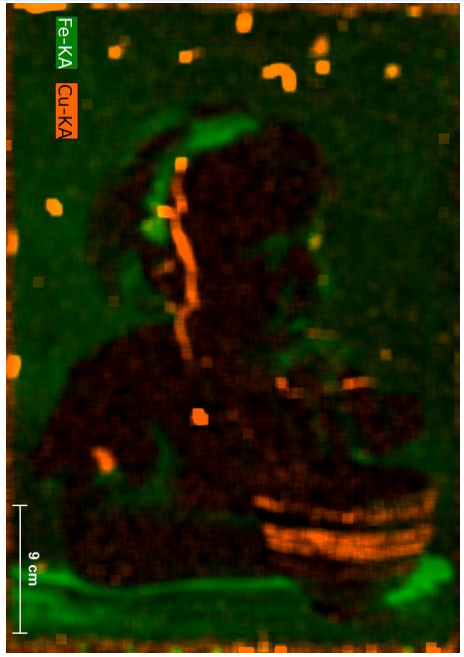
Average 3



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

Data mining:
Getting the data out…

Execute commands

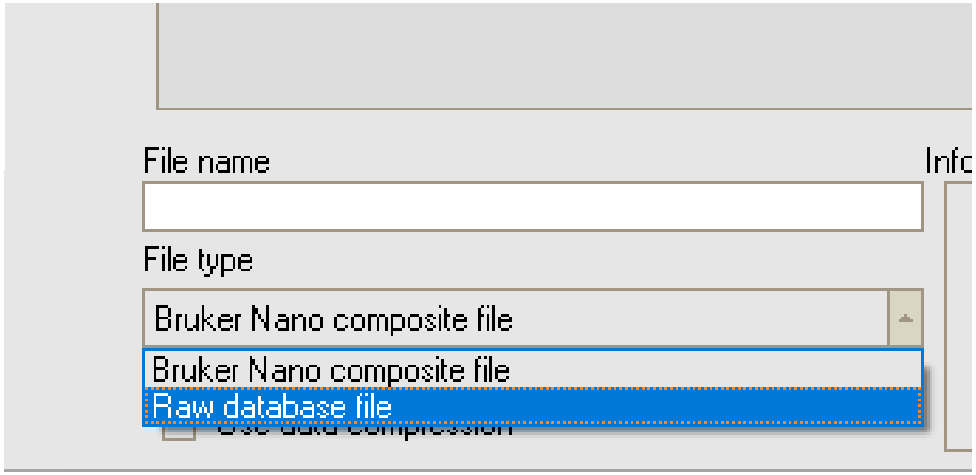
RCL commands | Script IDE

Script code: New | Open | Save | Save as | Compile | Debug | Run | Reset

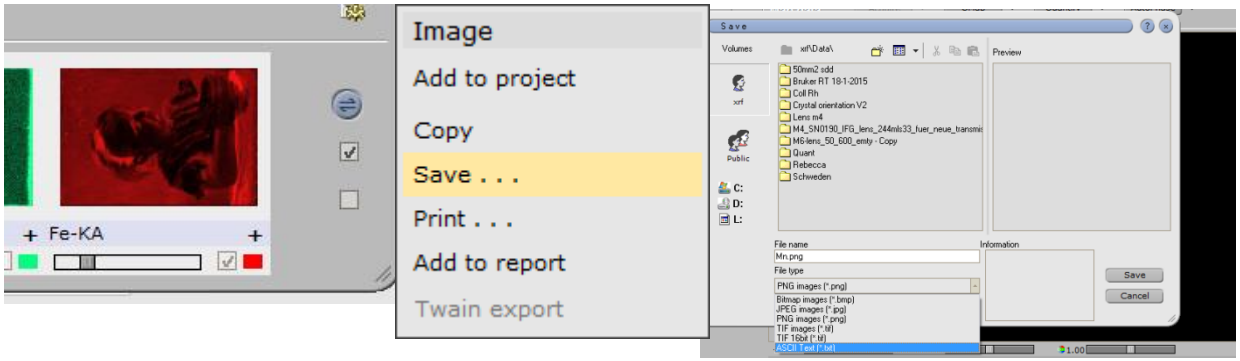
```
Program test;

var   MapFileName    : string;
      k,x,y          : integer;
      Width,
      Height,
      DetCount,
      ImgCount       : cardinal;
      Spectra        : TSpectrumArray;
      SpcFile        : TStringList;
      s              : string;
begin
  if GetOpenFileName('Load HyperMap file','Map files|*.bcf',MapFileName)=0 then
  begin
    SpcFile:=TStringList.Create;
    if HyMapLoadFromFile(MapFileName,Width,Height,DetCount,ImgCount)=0 then
    begin
      Writeln('Processing '+MapFileName);
      for y:=0 to Height-1 do
      begin
        Writeln('Read spectra line '+IntToStr(y)+' of '+IntToStr(Height-1));
        HyMapGetLineSpectra(0,Y,Width,Spectra);
        Writeln('Read spectra ready');
        if Length(Spectra)=Width then
        begin
          for X:=0 to Width-1 do
          begin
            //Writeln('Processing X '+IntToStr(x));
            // Alten Inhalt löschen
            SpcFile.Clear;
            // Datei-Inhalt erzeugen
            for k:=0 to Spectra[X].Header.ChannelCount-1 do
            begin
              s:=IntToStr(k);                                          // Kanalnummer
              s:=s+' '+FloatToStr(k*Spectra[X].Header.CalibrationLin+Spectra[X].Header.CalibrationAbs); // Energie
              s:=s+' '+FloatToStr(k*Spectra[X].Channels[k]);           // Kanalinhalt
              SpcFile.Add(s);
            end;
            SpcFile.SaveToFile('C:\M4 User\Xrf\Data\_Falk\forPTBpt3\Probe9_'+IntToStr(Y)+'_'+IntToStr(X)+'.txt');
          end;
        end;
      end;  // of Y
    end;
    SpcFile.Free;
  end;
end.
```

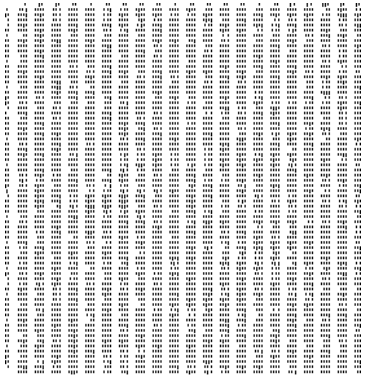
~ 100 kB per TXT file
Easily > 1 TB data sets for average sized maps

RAW is a very basic file format accessible with a wide variety of software tools.

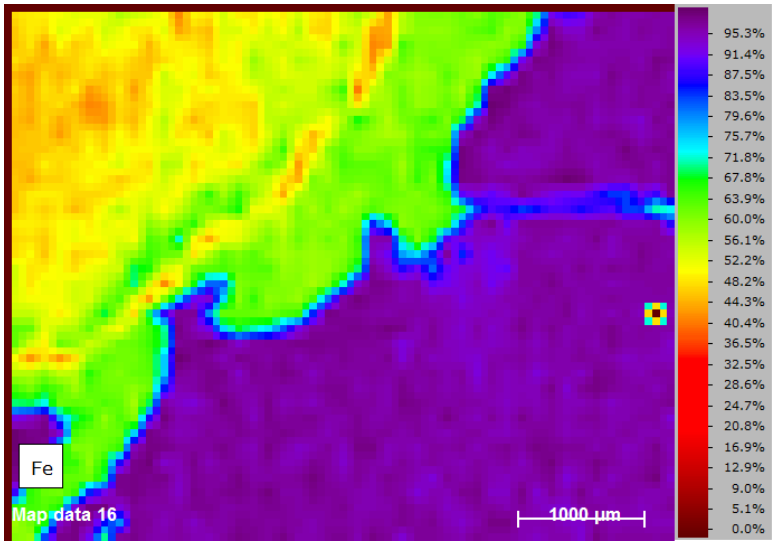


Data mining:
Getting the data out…

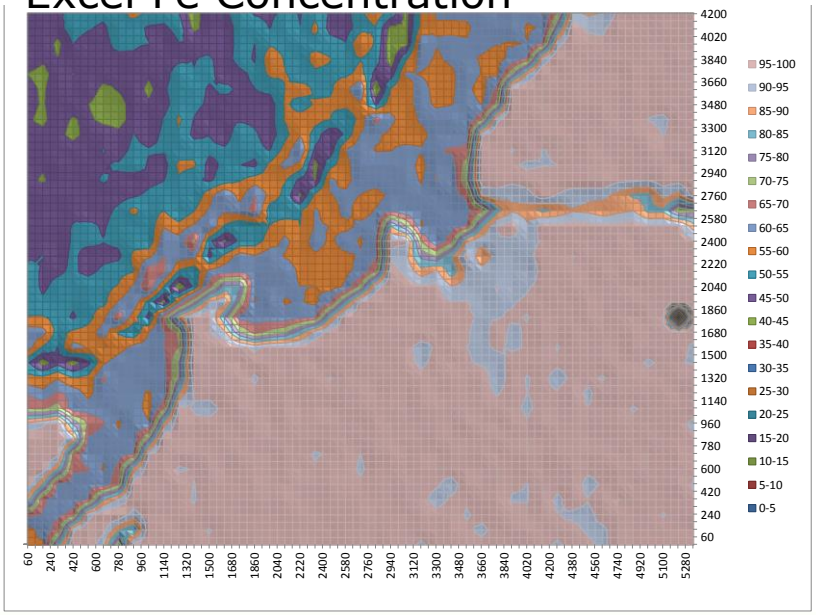




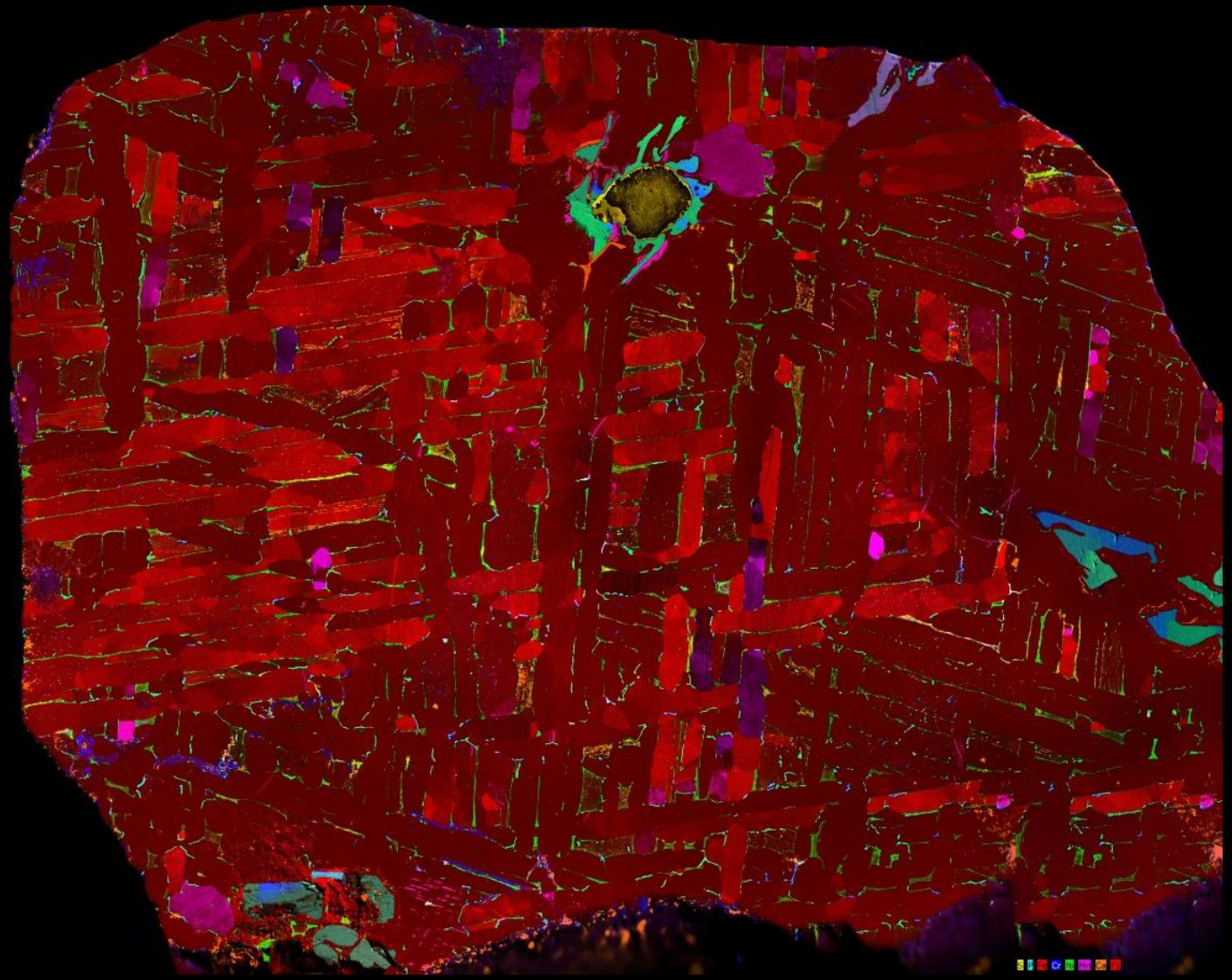
M6 Software



Excel Fe Concentration

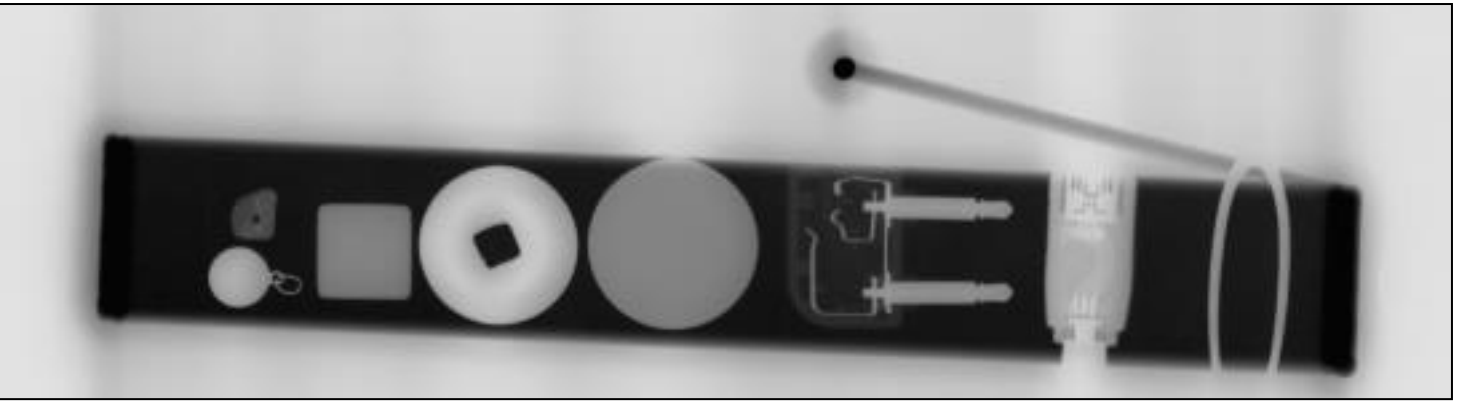


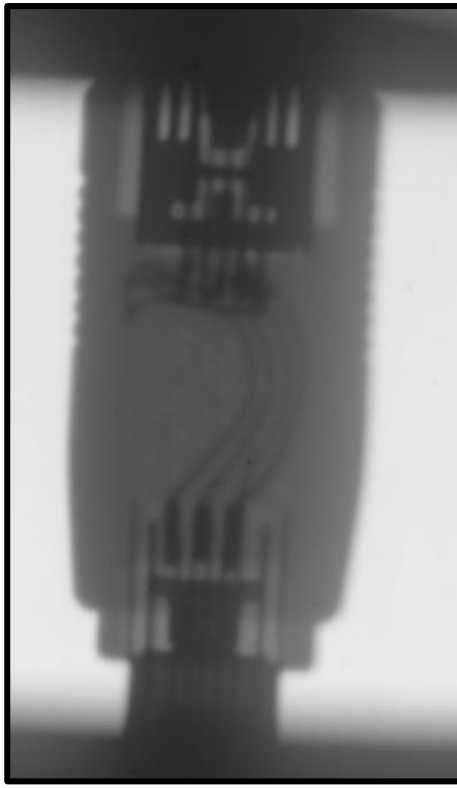
90 million Pixel
stitched from 40 single maps

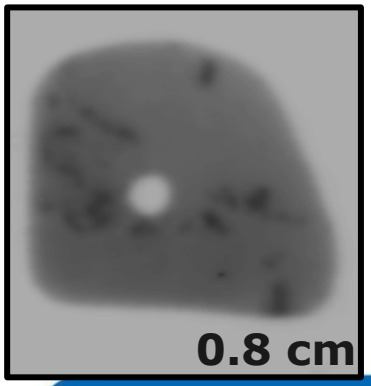


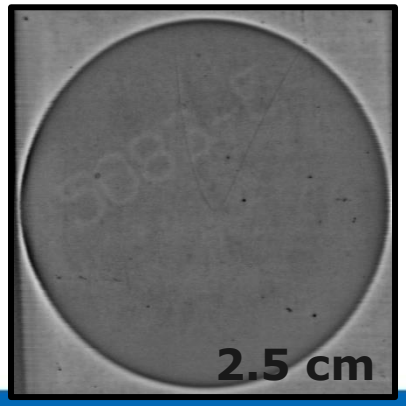
Transmission with the M6 Jetstream
First tests..

M6 scan over a selection of objects:
Gold medal, lapis lazuli fragment, glass, Cu alloy coin, Al alloy, airplane plug adapter connector, USB adaptor and gold earring.
Using X-ray plate from VMI 5100 MS-C
50 kV 600 µA 10 cm working distance 100 mm/s









Transmission with the M6 Jetstream Chimei Museum Taiwan, work in progress….

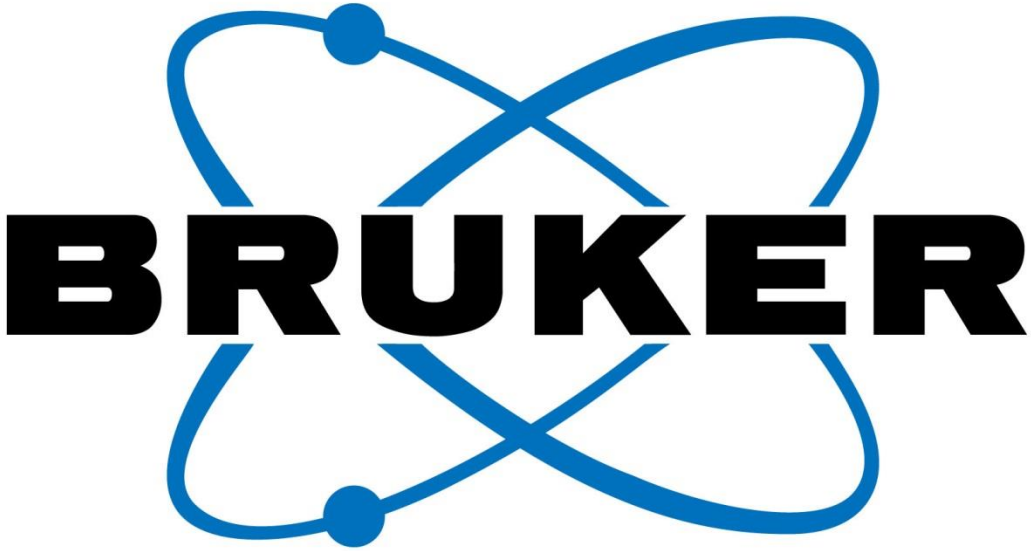
The Awakening Hour: an Interior with a Mother and A Child, Dutch School (19th Century)
Undated, Oil on panel

Fuji cassette-Type CC

AGFA CR MD4.0T general cassette

AGFA CR MD4.0T general cassette


Pb
Fe
Cu
Hg



Innovation with Integrity