BRUKER NANO ANALYTICS' CULTURAL HERITAGE WEBINAR SERIES 2023

Quantification by means of XRF – Illustrated on Cultural Heritage samples

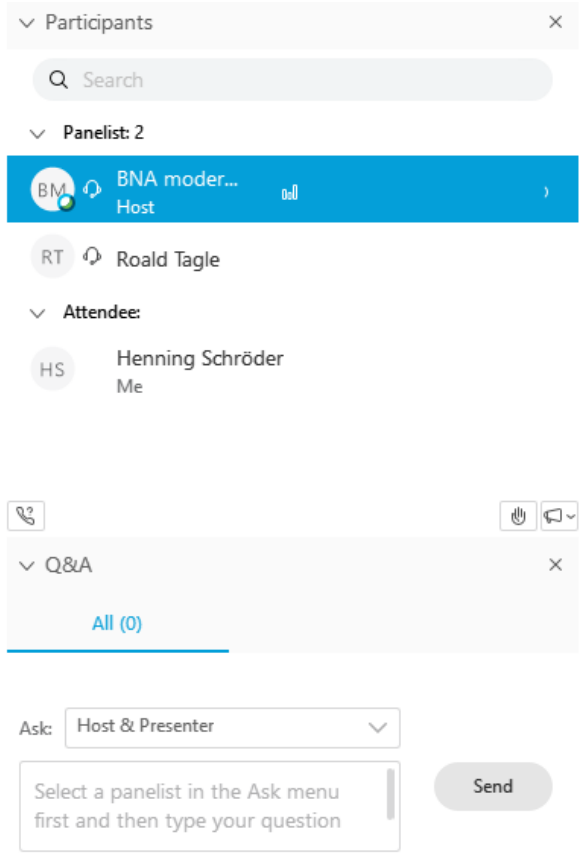
Micro-XRF Applications Team
Bruker Nano Analytics

Art & Conservation Webinar Series
Quantification by means of XRF in Cultural Heritage Studies

If you have questions during this webinar,

please **type your questions**, thoughts, or comments in the

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



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

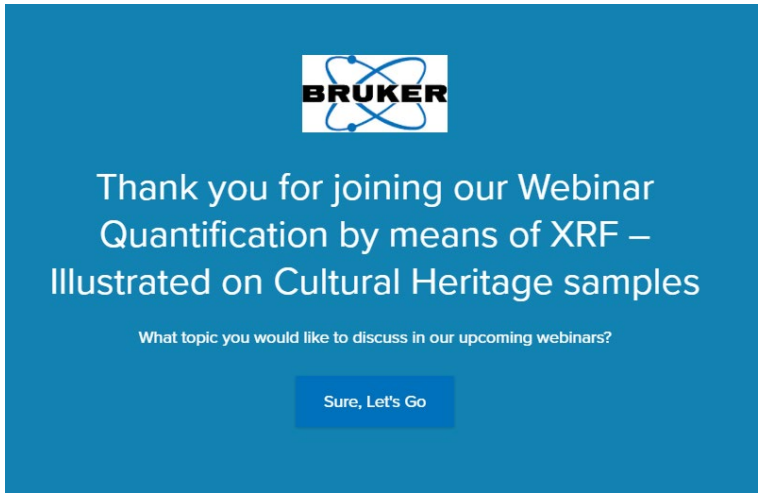
Any unanswered questions or comments will be answered and discussed by e-mail or in another WebEx session.

Art & Conservation Webinar Series
Quantification by means of XRF in Cultural Heritage Studies

There are many topics involving XRF in Cultural Heritage!

We are interested to know which topic(s) you would like to learn more about in upcoming webinars?

- For live sessions: At the end of this webinar, you will be automatically redirected to a short survey via the Bruker Webex side.
- For on-demand: You can find a link in the video description or follow the QR code on the right.



The Speakers

Dr. Roald Tagle

- Head of XMP Application,
  Bruker Nano Analytics, Berlin, Germany

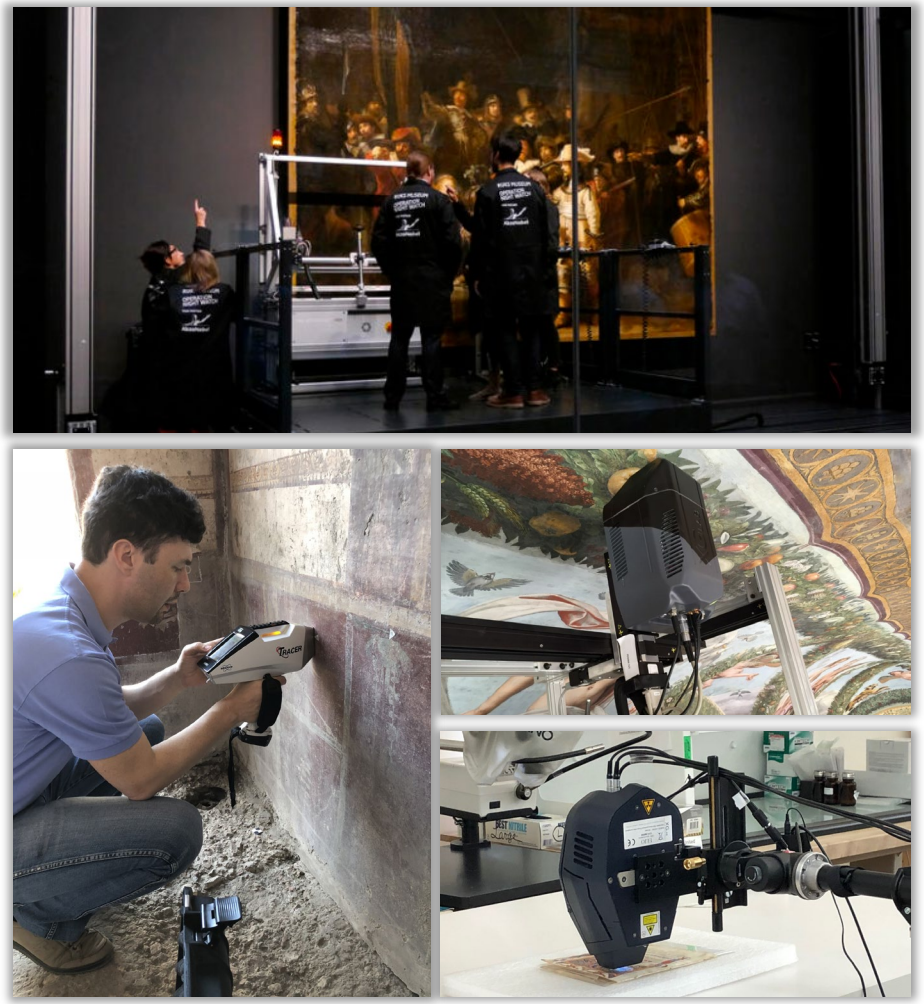
Mareike Gerken M.A.

- Application Scientist XMP specialized in Cultural Heritage
  Bruker Nano Analytics, Berlin, Germany

X-ray fluorescence analysis
A key technology for Cultural Heritage

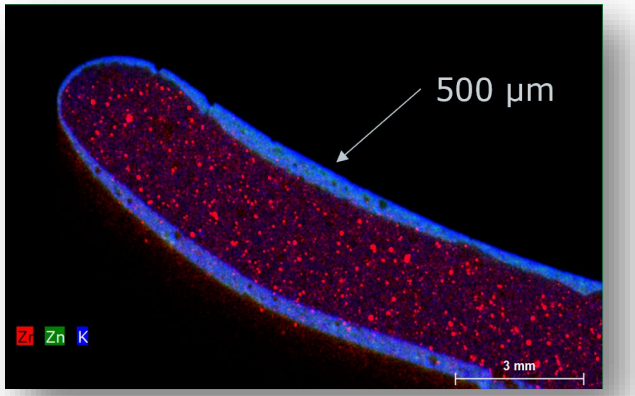
Today, XRF analysis has proven to be one of the fundamental and substantial analytical techniques for material analysis in Heritage Science due to the following reasons:

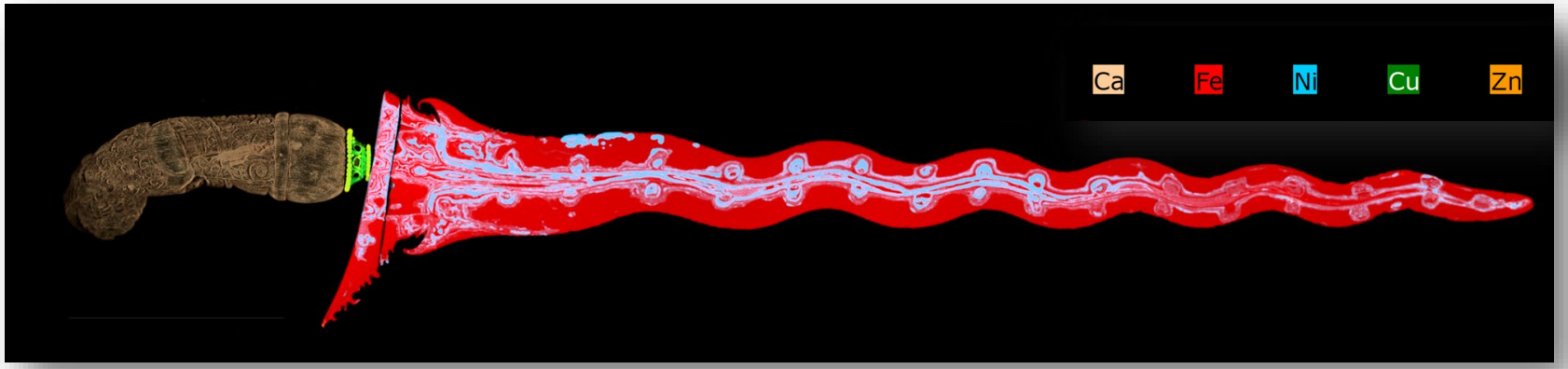
- Multi-elemental sensitivity.
- Non-invasive nature.
- Fast and reliable results.
- Usually no sample preparation required, potentially needed, depending on the sample and the analytical question.
- Applicable to a wide range of materials or objects, as well as research objectives.
- Applicable as qualitative, semi-quantitative and quantitative analysis tool.

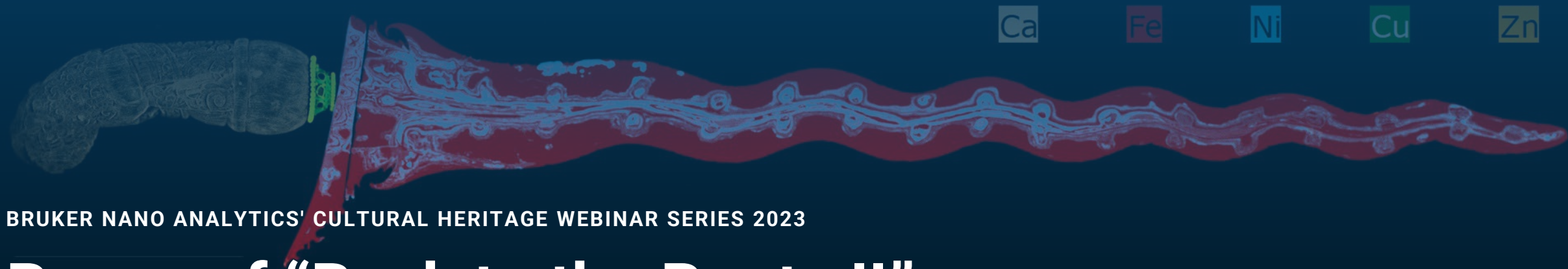


Overview

- This webinar focuses on XRF **quantification of Cultural Heritage related materials.**
- The next session, on December 7th 2023, will focus on the study and quantification potentials of **non-ideal samples in Heritage Science.**
- Further next year, we will continue discussing the analysis of **three-dimensional objects,** aiming on qualitative and quantitative analysis, its potentials and limitations.







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Recap of “Back to the Roots II” Discussing quantitative analysis

Recap: What are Qualitative, semi-quantitative and quantitative analysis principles

Compositional Analysis

Qualitative Analysis
- What is in the sample?

→ Element identification

Quantitative Analysis
- How much of it is in the sample?

Semi-quantitative
- What is/are the main elements
- Which elements are correlated or anti-correlated?
- What are the relative abundancies?

→ Results in (relative) intensities

Quantitative
- What is the mass deposition or amount of the elements in the sample?

→ Results in %, ppm, mg/cm², or similar

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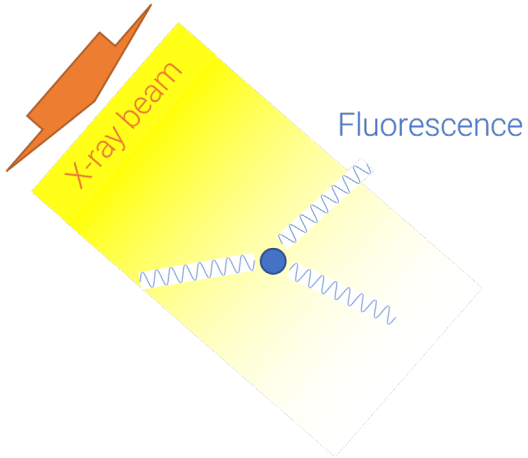
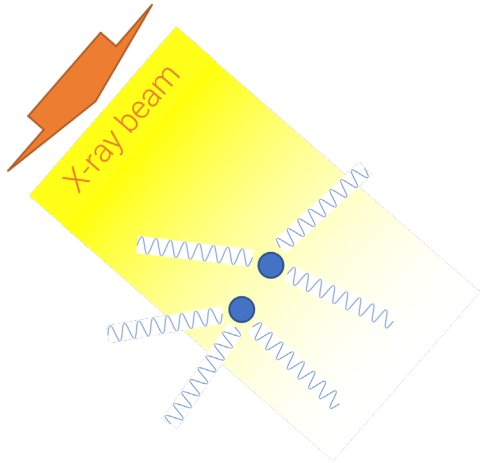
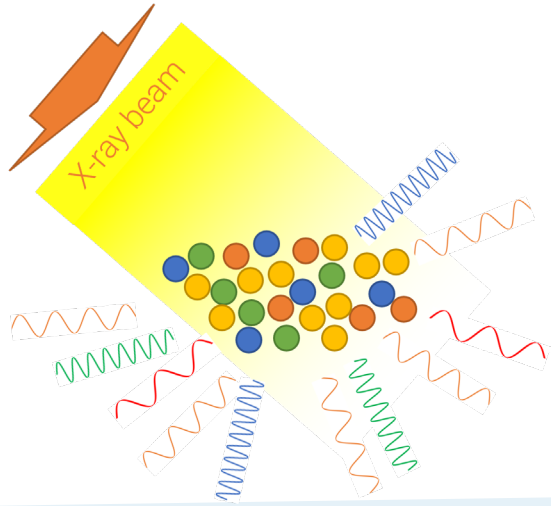
Recap: Quantitative X-ray fluorescence analysis Counting atoms

- XRF is widely known for quantitative analysis. Why? Because it works so straightforwardly.
- An atom in an X-ray beam will produce element specific fluorescence radiation.
- Two atoms of the same type will produce twice as much fluorescence radiation.
- Many different atoms in the X-ray beam will produce many photons of the characteristic fluorescence radiation.
  - Detecting the radiation with wavelength- or energy-dispersive detectors enables qualitative analysis.
  - Counting the number of incoming photons allows to **count the atoms** → quantitative analysis.

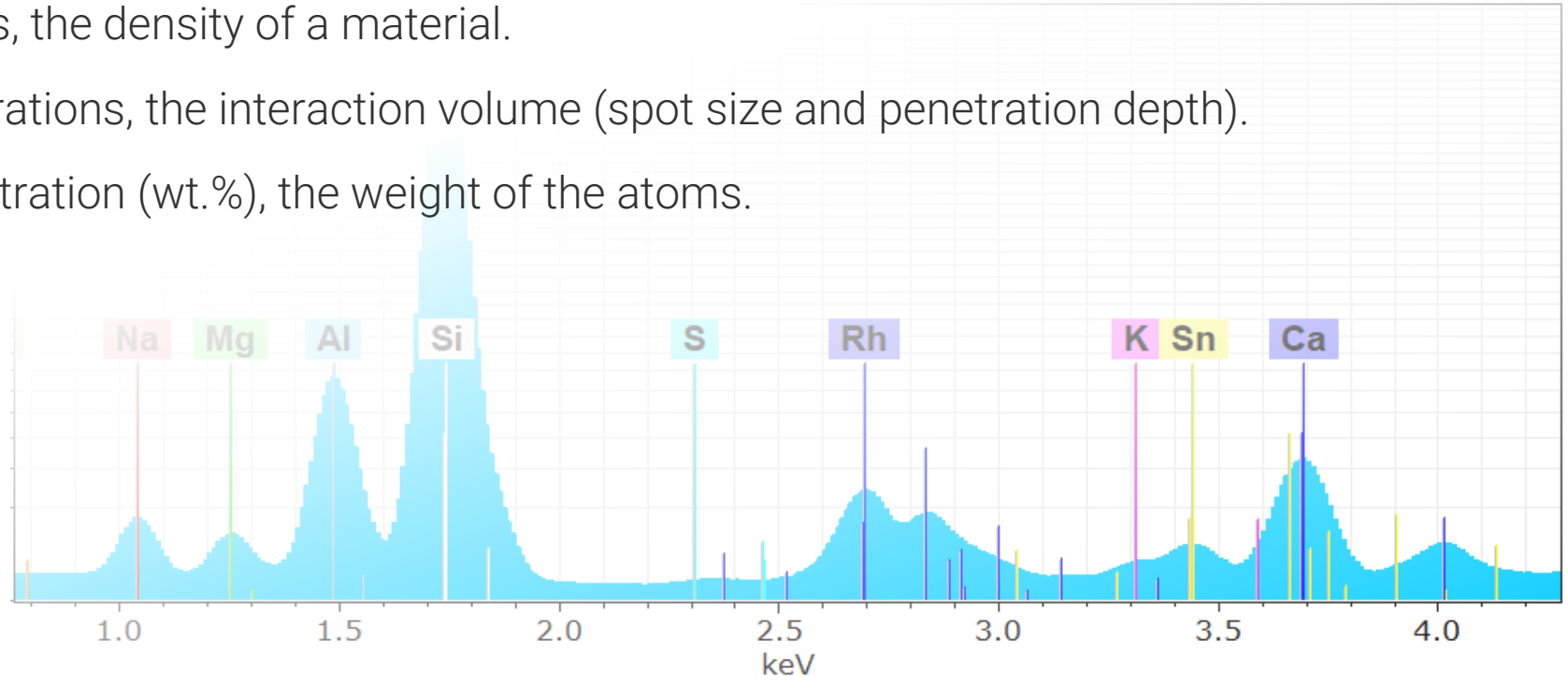






Recap: Quantitative X-ray fluorescence analysis From atoms to “wt.%”

- With additional information, it is possible to convert this number of “atoms in an X-ray beam” to more useful units:
  - To get the mass coverage, the size of the irradiated area.
  - To get a layer thickness, the density of a material.
  - To get atomic concentrations, the interaction volume (spot size and penetration depth).
  - To get to mass concentration (wt.%), the weight of the atoms.



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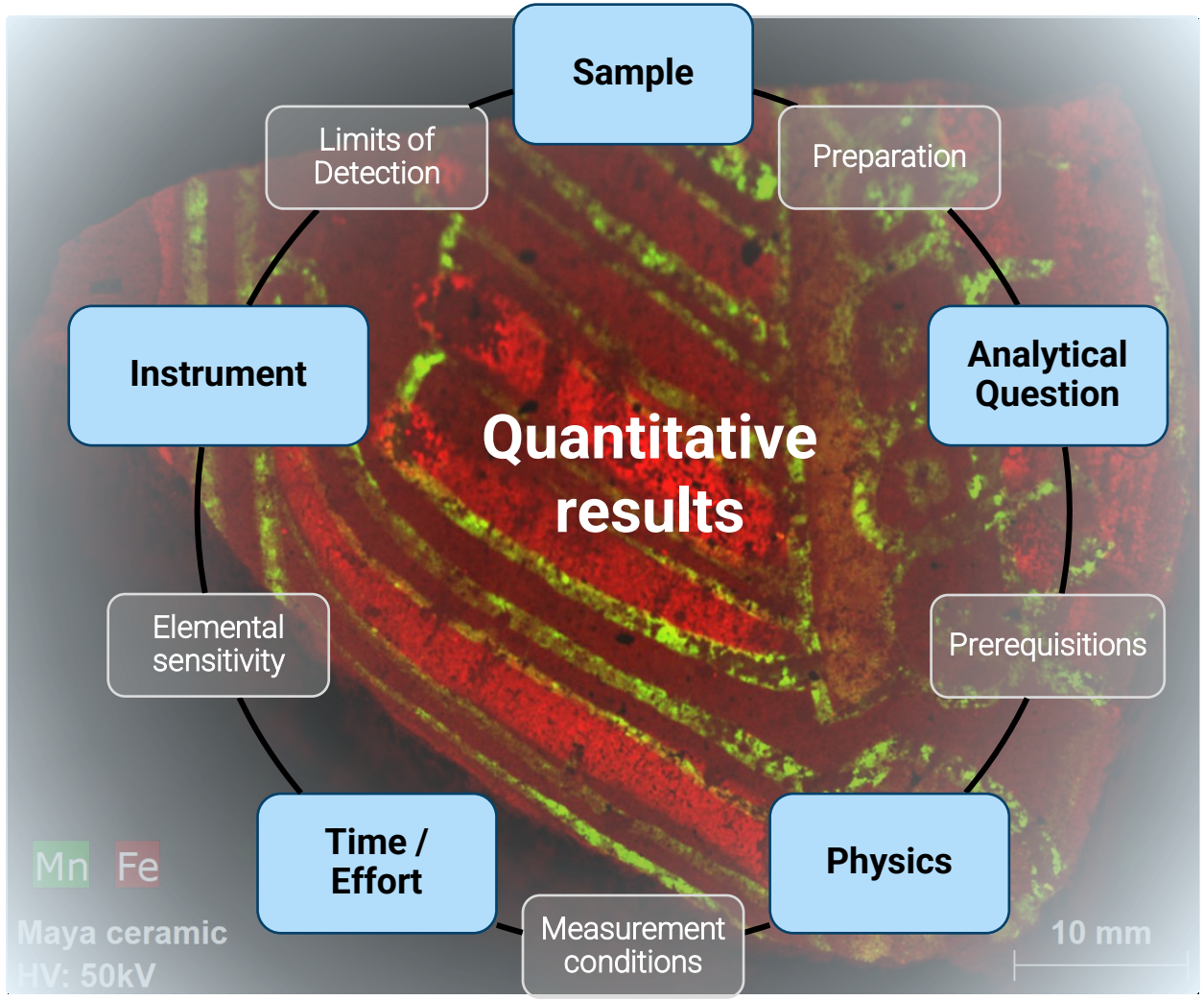
Physics unveils possibilities, technology harnesses potentials, while the synergy between the sample and the analytical query forges the path

Quantitative X-ray fluorescence analysis
A complex matter…

Quantitative results emerge as a convolution of:

- the sample
- the analytical question
- the applied effort
- the available time
- the analysts' talent
- the capabilities of the available instrumentation



Quantitative X-ray fluorescence analysis
What type of samples can be quantified?

- For quantification purposes, the sample needs to have a defined composition within the analytical volume.
- Yet, the **analytical approach** as well as the **sample preparation** applied can be adapted to suit the requirements for quantification.
  - ➢ Any sample can be quantified, the best approach and the potential output depends on the research question, the sample itself and the effort planned to spend!
    - ➢ *With the correct sample preparation, even a painting can be quantified!*

➢ As you can imagine, quantification of Cultural Heritage is very special, because of the need for it to be non-invasive!

Credits:
Des McKenzie
Australia

Quantitative X-ray fluorescence analysis
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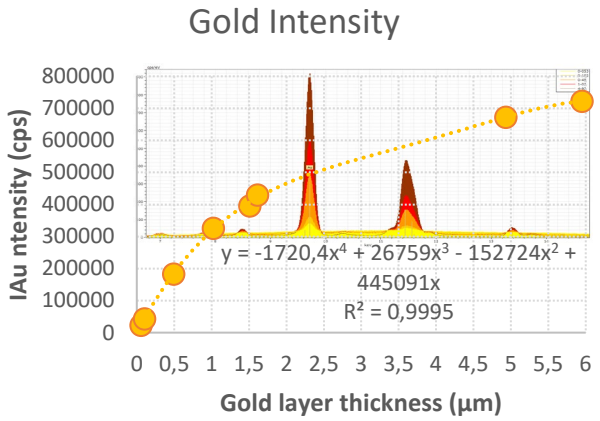
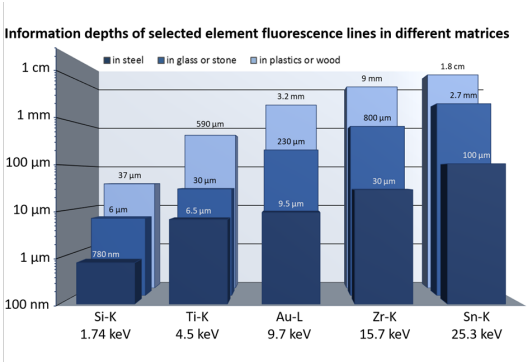
Quantitative X-ray fluorescence analysis
Understanding your sample… from the XRF point of view

Each material has its own intrinsic structure and composition that impacts not only the analysis itself but also its outcomes. Likewise, each instrument has its own specifications that equal its performance.
Understanding both is the key to getting significant and analytically relevant results!

Sample Determinants

- Information depth
- Homogeneity





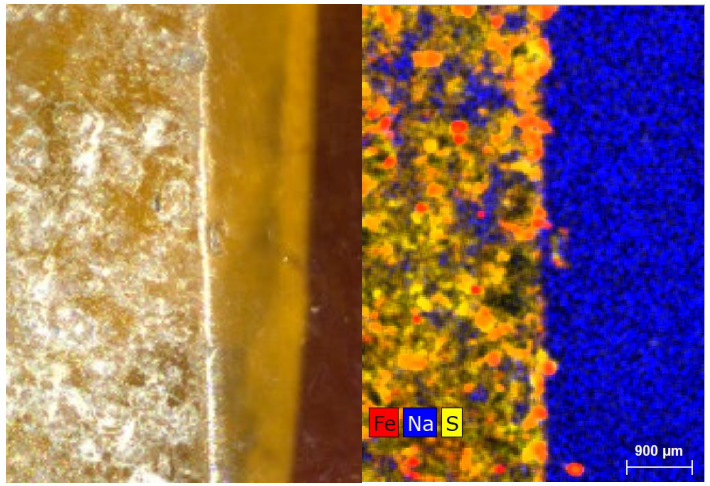
The Copper CHARM set: 10.1111/arcm.12117

Quantitative X-ray fluorescence analysis
Information depth

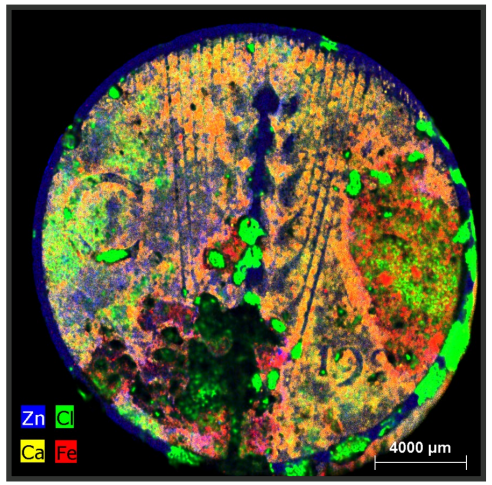
The surface of the material might “show” a composition not representative for the sample itself.
For example, a layer of pigment, corrosion or contamination during use or ground deposition can strongly affect the analytical outcome.

This becomes especially problematic when studying light elements that do not have a high information depth.

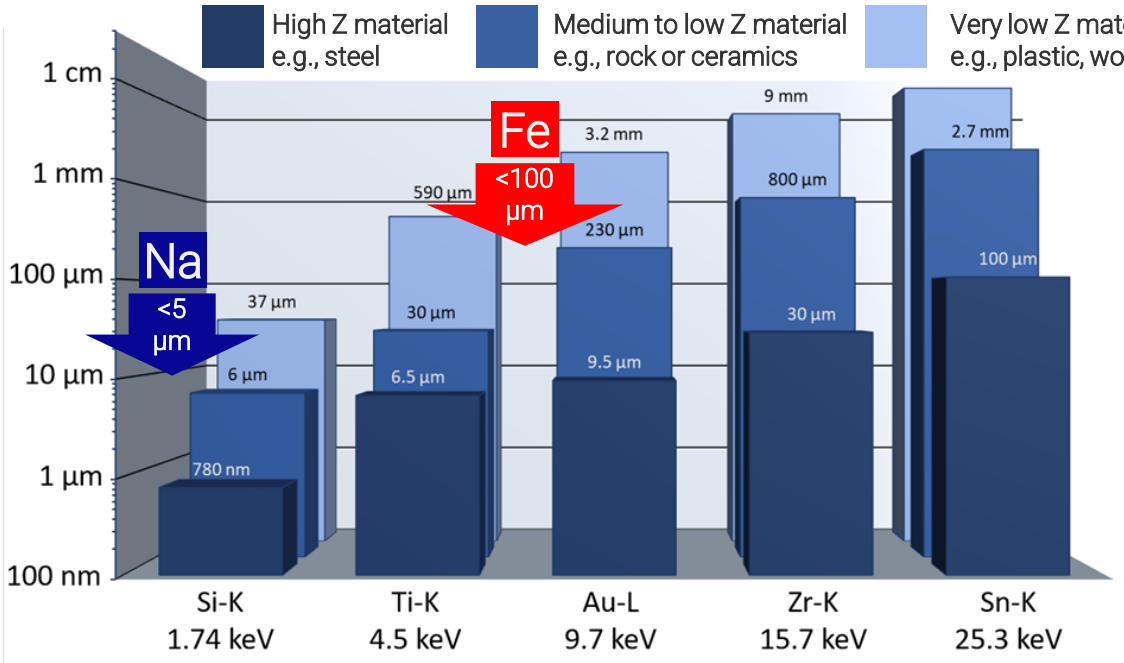
Corroded Glass



Coin with Patina

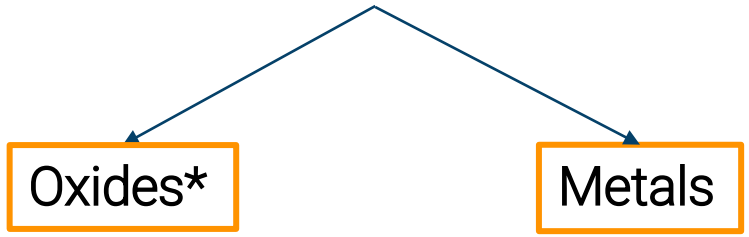


Information depths of selected element fluorescence lines in different matrix.
Depths from which 90 % of the generated intensity can escape the sample



Quantitative X-ray fluorescence analysis
Sorting materials

- Today, we would like to cover two types of material groups:



- Both groups require their specific quantification approach, strongly related to the sample itself.

Painted ceramic

Bronze statue

* Note: “Oxides” are a simplification! Lead glass is an oxide, but it needs to be treated like a metal as it likewise is a high-density high-Z material. On the other hand, aluminum or magnesium alloys are metals but low-density low-Z materials and hence, more similar to what we here define as oxides!

Cultural Heritage materials
Oxides (Low Z materials)

What types of materials do we consider as „Oxides“?

- Ceramics
- Glass (excluding lead glass)
- Porcelain
- Stone

Which research questions can be answered using XRF?

- Provenance (Origin and dating)
- Composition
- Technology and production
- State of preservation and previous treatments



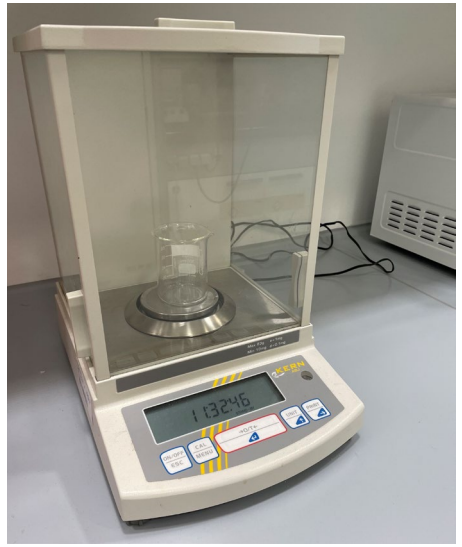

Quantification of Oxides …With standards or without?

There are two distinct approaches to quantitative XRF:

- **Empirical**: Element concentrations can be deduced by comparison of the sample spectrum with spectra of sufficiently similar standard samples with known compositions.

- **FP-based**: All physical effects in XRF are reasonably well-understood nowadays and their probabilities are tabulated. Thus, a quantification based on these fundamental parameters and a lot of math (FP quantification) can be performed.

https://en.wikipedia.org/wiki/Weighing_scale



We will discuss the pros and cons and highlight the advantages of a hybrid approach using

Standard-supported FP quantification

Quantification of Oxides …With standards or standard free (FP)?

Ideally, for a Quantification the samples are:

- Homogeneous
- Infinitely thick
- All matrix-relevant elements in the sample are either detectable and their amount known or they are in a fixed ratio to a detectable element.

?

If this IS the case, standard-less or reference material-free (FP methods) are well suitable with possibilities to increase performance by using reference materials

If this is NOT the case, reference samples are recommended. Otherwise, the complexity of the task increases significantly together with the uncertainty of the results

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Let’s get more practical

Quantification of Oxides
A “secret” often not told for light matrix samples

Yes…matrix effects influence the quantification;
BUT this is just one of the many factors…

How strong is the influence in reality?

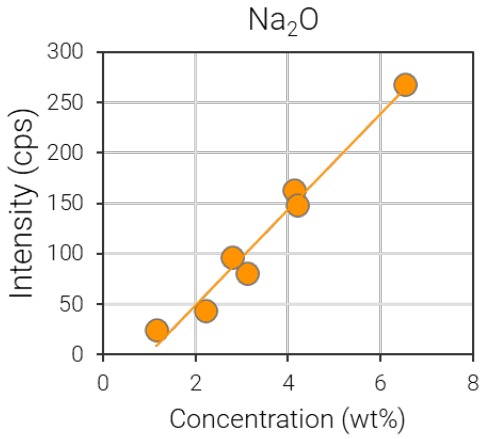
We know it is proportional to the atomic number of the element as well as to its relative abundance in a sample

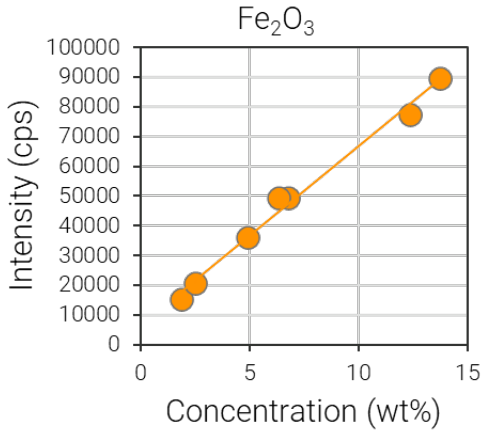
An average silica glass and a ceramic:

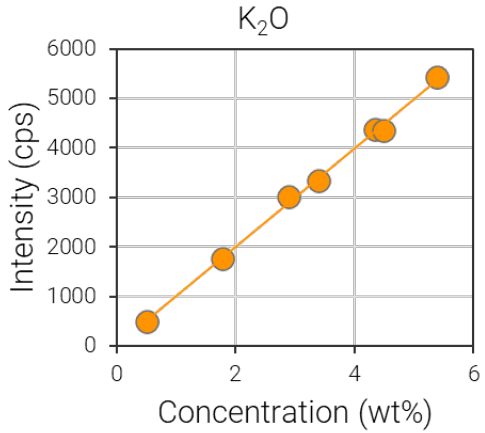
	Glass	Ceramic
Oxygen	~ 47%	~ 40 %
Silica	~ 34 %	~ 30 %
Na, Mg, Al, S, K, Ca	~ 20 %	~ 25 %
Fe	< 1%	< 5 %
Rb, Sr, Y, Zr	<< 1%	<< 1%

> 90 % light and very light elements

A simple linear relation!







The selected analyzed references samples show a nice linear correlation for major elements despite the relatively high Fe content.

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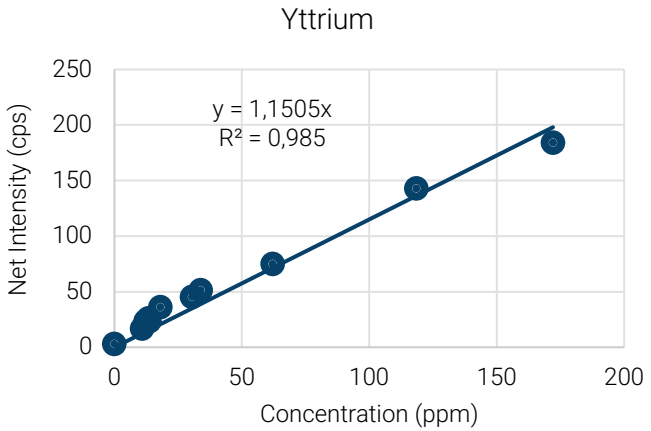
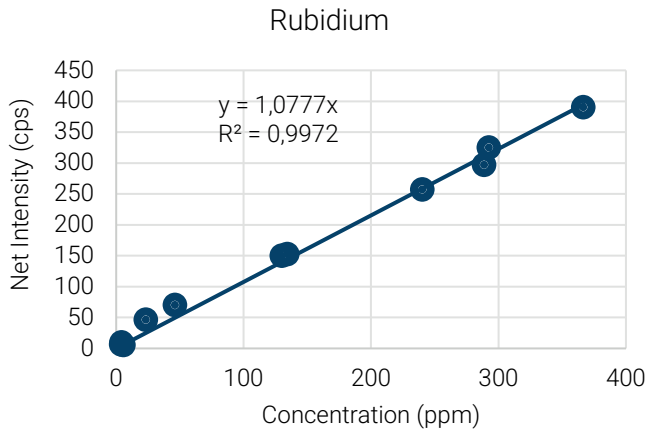


For trace elements in XRF a simple linear correlation can be expected as the matrix effects are negligible.



Quantification of Oxides
The “real life” sample quantification

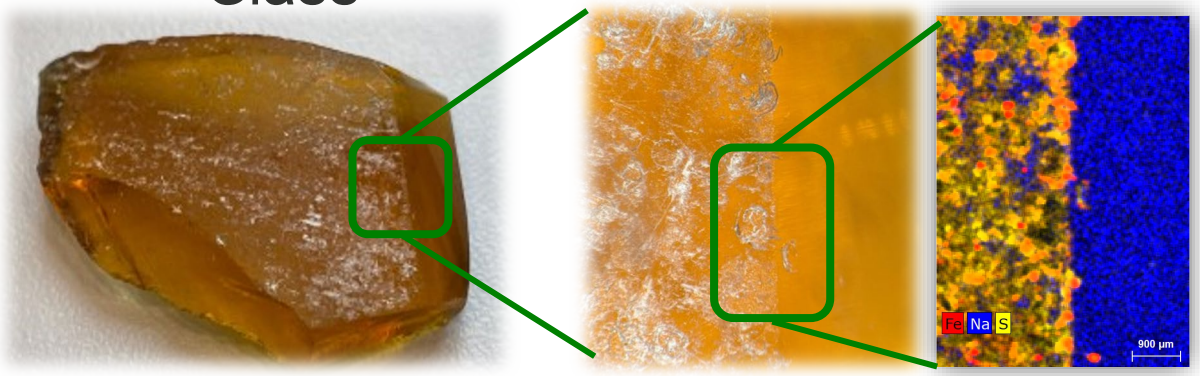
Quantification of light matrix samples by XRF should not be a problem at all…

But it is… and one of the main reasons is the sample!

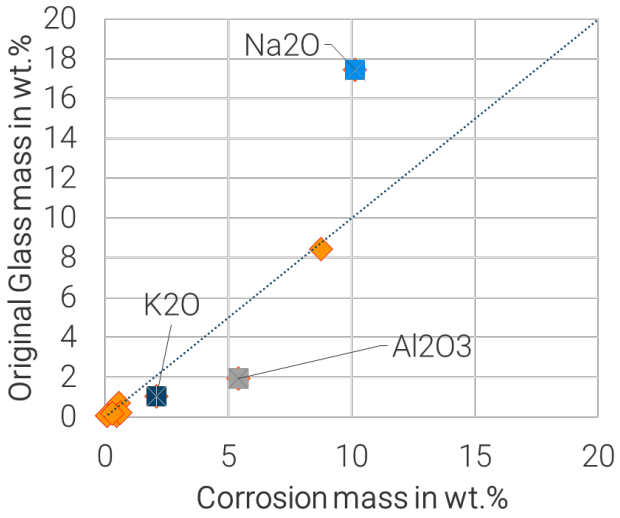



Quantification of Oxides
The “real life” sample quantification

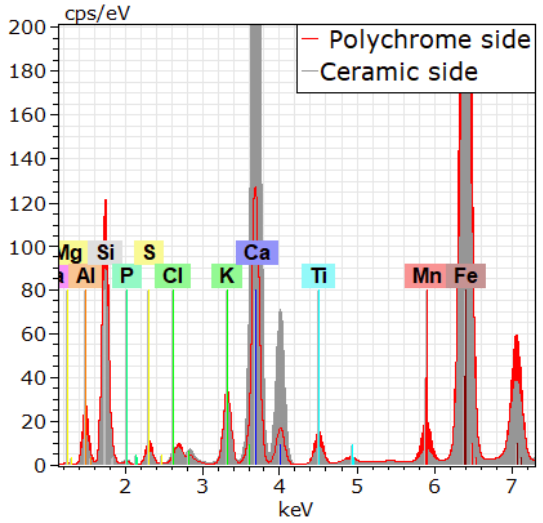
Glass

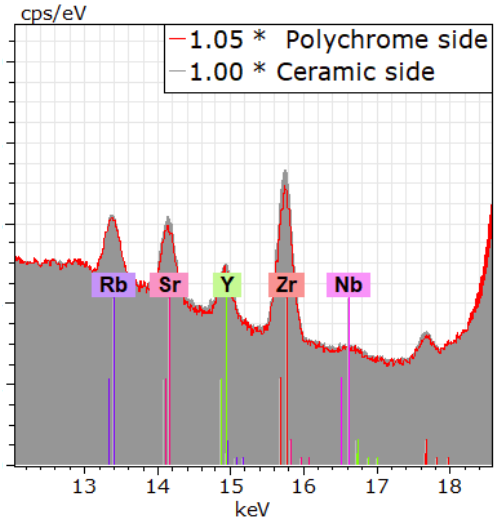


Compound	Corrosion surface	Polished surface
Na_2O	10.12	17.44
MgO	0.57	0.7
Al_2O_3	5.4	1.93
SiO_2	71.52	69.88
SO_3	0.46	0.06
K_2O	2.09	1.05
CaO	8.76	8.43
TiO_2	0.09	0.06
MnO	0.09	0.07
Fe_2O_3	0.28	0.17

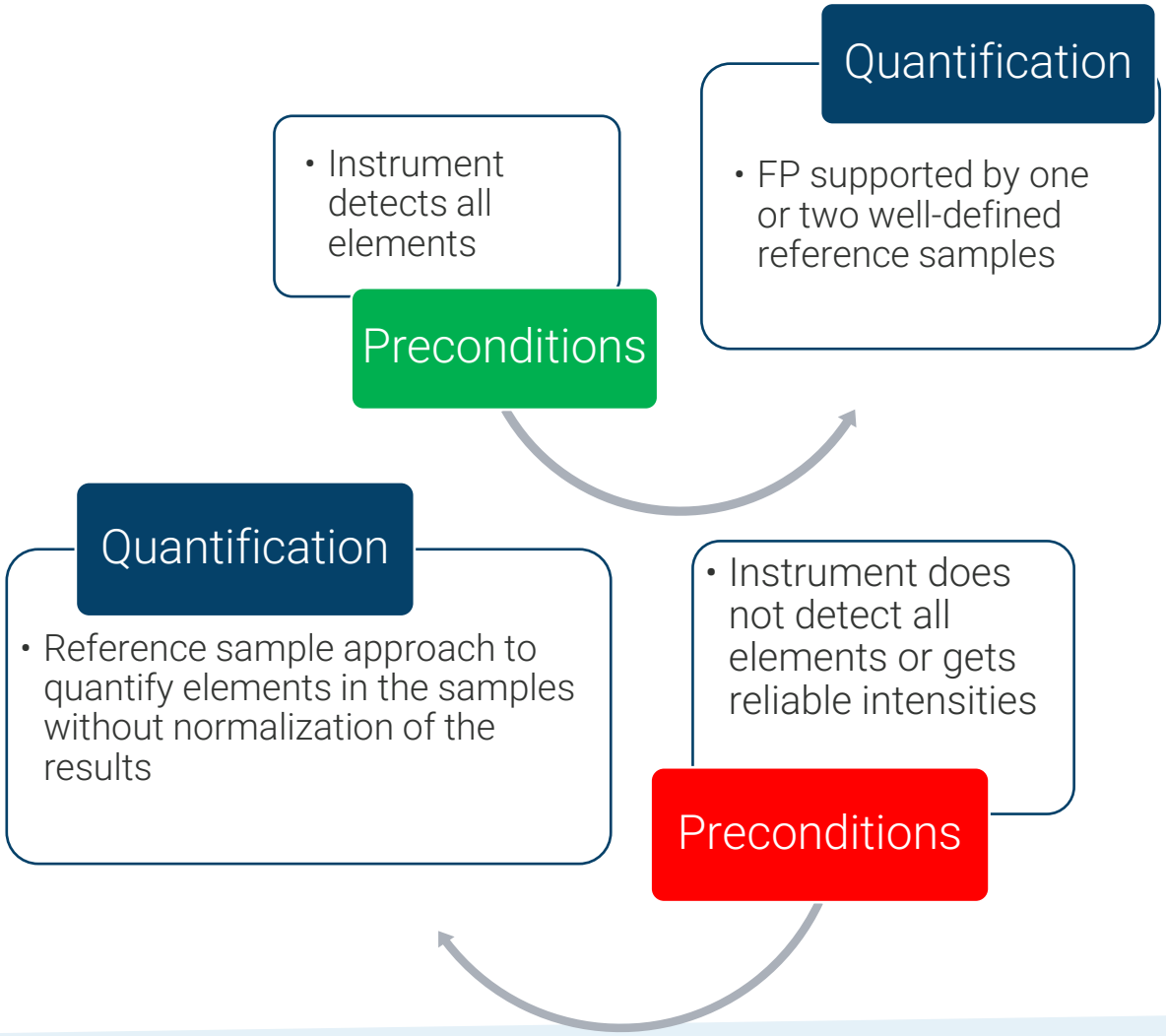


Ceramic





Quantification of Oxides
The “real life” sample quantification



Recommendations for avoiding typical pitfalls:

If samples are not homogeneous within the analytical volume, e.g., glass is corroded, ceramic is painted or contaminated by environmental deposition

- quantification of elements by using low-energy lines is not recommended, as the numbers might be meaningless
- quantification should focus on non- or less-disturbed elements such as Pb, U, Th, Rb, Sr, Y or Zr
- If low-Z elements are needed, a sample preparation is required

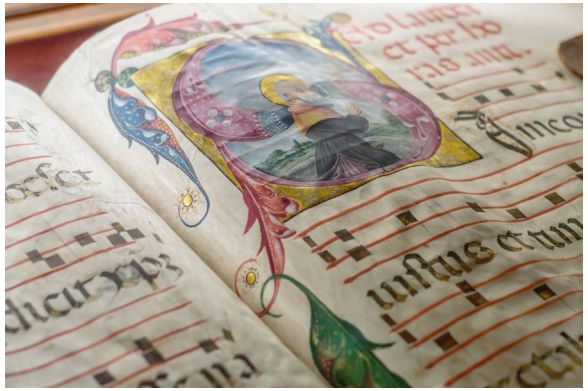
Cultural Heritage materials
Metals

What types of metals can be analyzed?

- Alloys
- Gildings
- Corrosion

Which research questions can be answered using XRF?

- Provenance (origin and dating)
- Composition
- Technology and production
- State of preservation and previous treatments

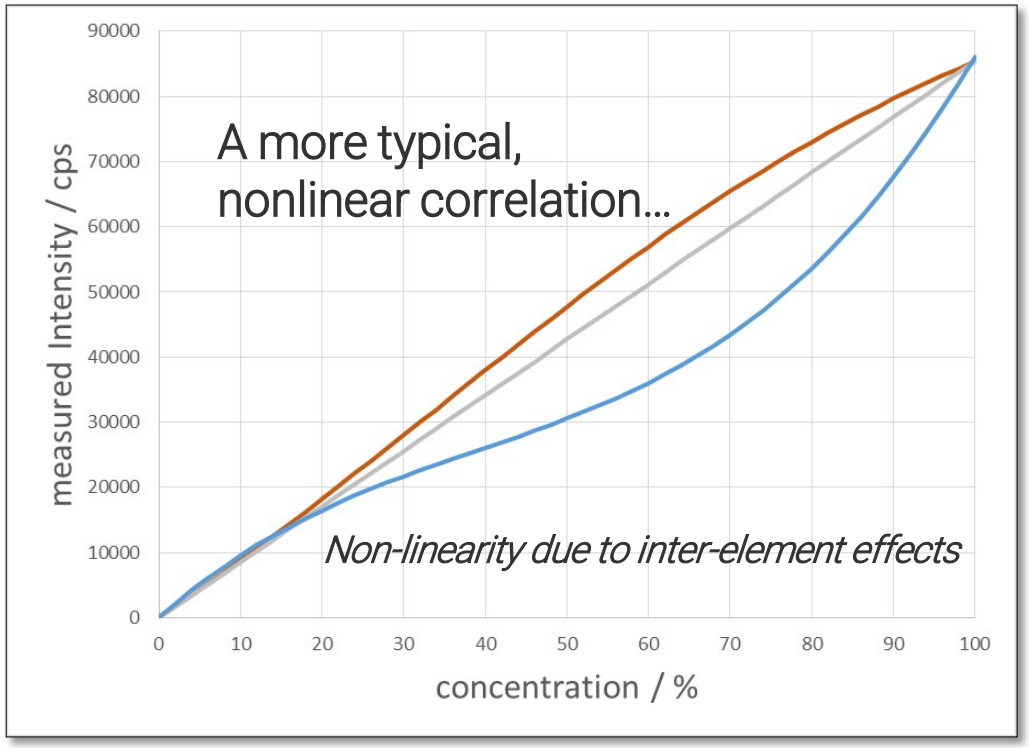



Quantification of Metals (high-density / high-Z materials) Considerations from the quantification point of view

Differences to light matrix materials

- Simple linear relation between intensity and concentration is disturbed due to inter-element effects
  - Absorption
  - Excitation
- Full validity of calibration based on reference samples is restricted to the reference material range
- Inhomogeneity due to large-scale metal segregation, e.g. Cupper alloys

… A complex relationship

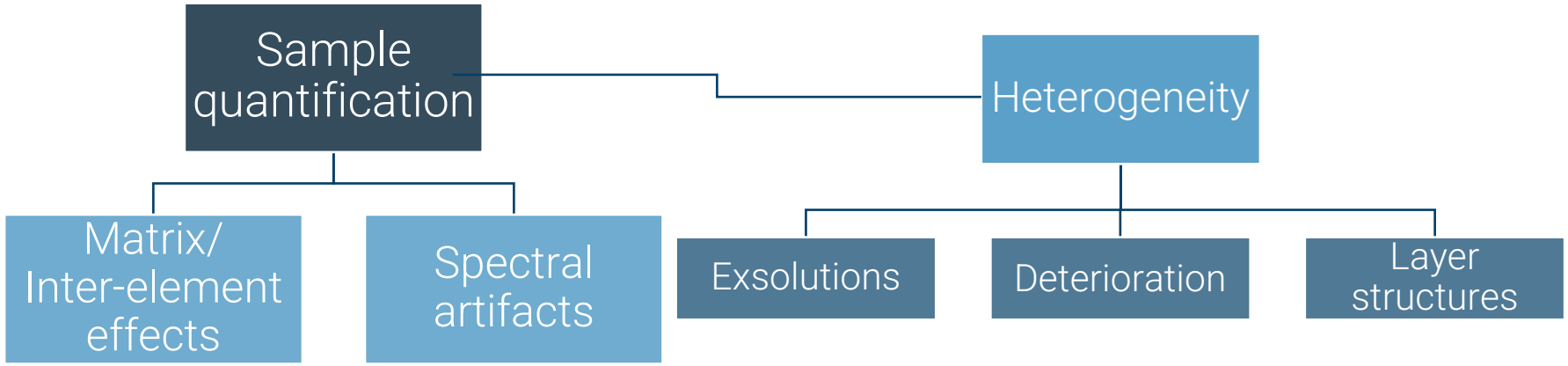


Quantification of Metals (high-density / high-Z materials) The “real life” sample quantification

Some preconditions for “good” quantification are easier to achieve with metals:

- Infinitely thick
- Matrix-relevant elements in the sample are all detectable
- The use of FP can correct for matrix effects and the use of reference materials improve results

But pitfalls can come from the sample again:



Quantification of Metals (high-density / high-Z materials) …With standards or standard-free (FP)?

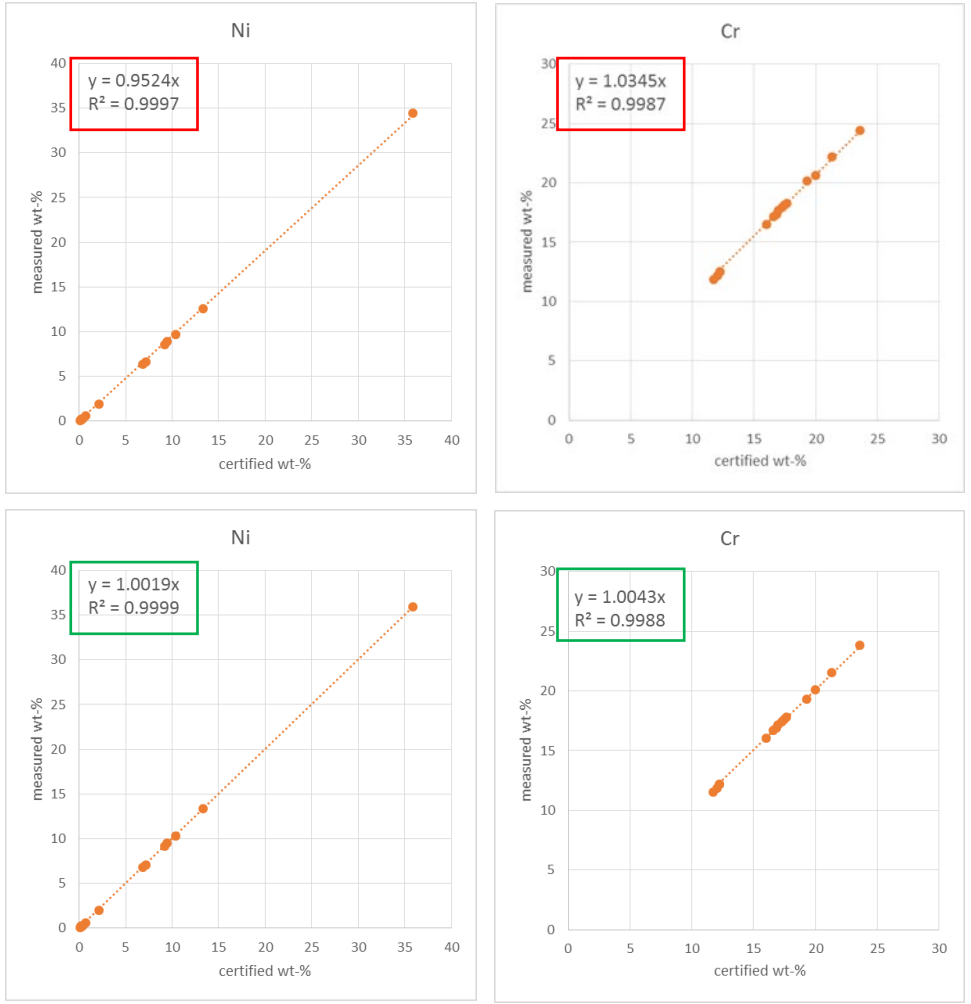
Fundamental Parameter (FP) has multiple advantages when studying high-density materials!

- Physics are well-described.
- FP algorithm includes inter-element effects, such as absorption or secondary excitation!

Due to the well-described physics, the quantified values fall on a straight linet

The lack of trueness is related to undescribed effects such as tertiary excitation, or the sample itself.

Such a linear correlation can easily be corrected using reference materials.

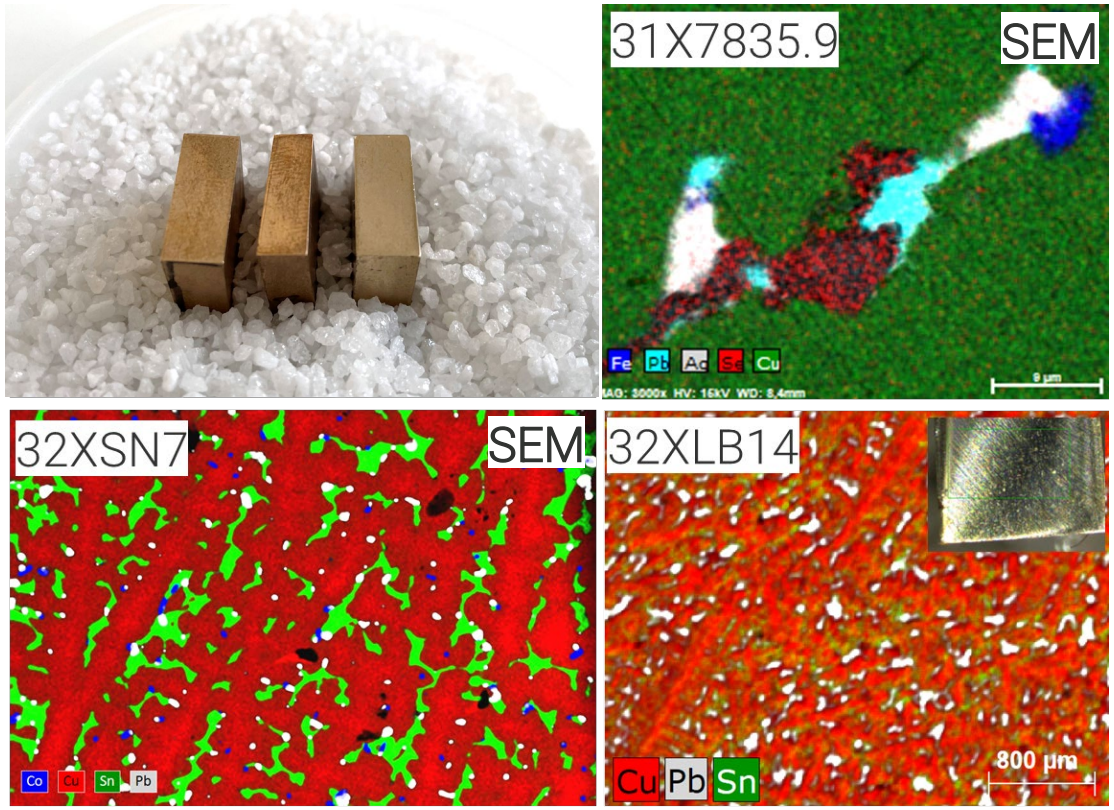


Stainless steel

Quantification of Metals (high-density / high-Z materials) Heterogeneity

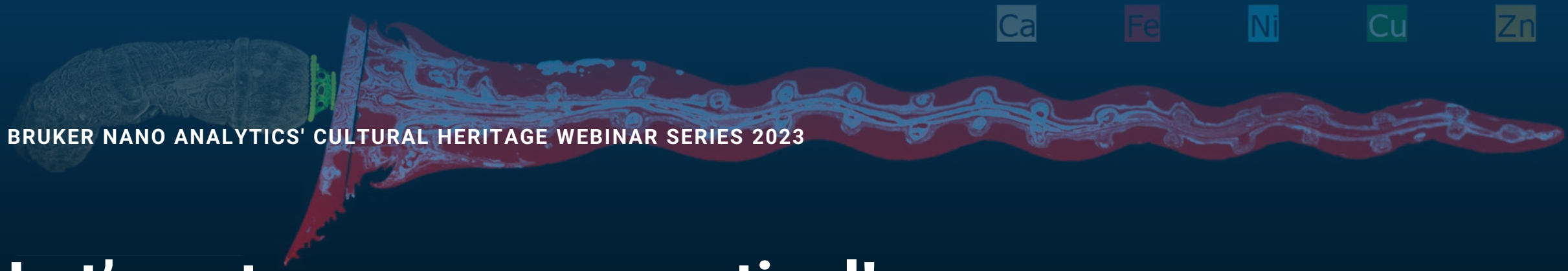
The Copper CHARM set: Historic bronze alloys

- Trace metals in historic alloys are likely to segregate from the matrix metal or migrate over time.
- Considering the resulting heterogeneity, the analytical approach needs to be adapted to avoid false results.
- As visible in the lower-right corner measurements on the same sample may result in widely varying Pb values
- The small spot size of the M4 TORNADO of 20 µm “allows” to resolve the exsolutions.
- This effect is not visible using instruments with larger spot sizes (≥ 1 mm) such as the TRACER 5g or ELIO.



Ni	Cu	Zn	As	Rh	Ag	Cd	Sn	Sb	Au	Pb	Bi
0.25	87.58	0.29	0.00	0.00	0.12	0.00	7.02	0.06	0.00	4.32	0.17
0.23	84.75	0.33	0.05	0.00	0.11	0.01	6.20	0.03	0.00	7.84	0.31
0.18	54.82	0.15	0.10	0.00	0.15	0.01	5.70	0.02	0.00	37.37	1.39

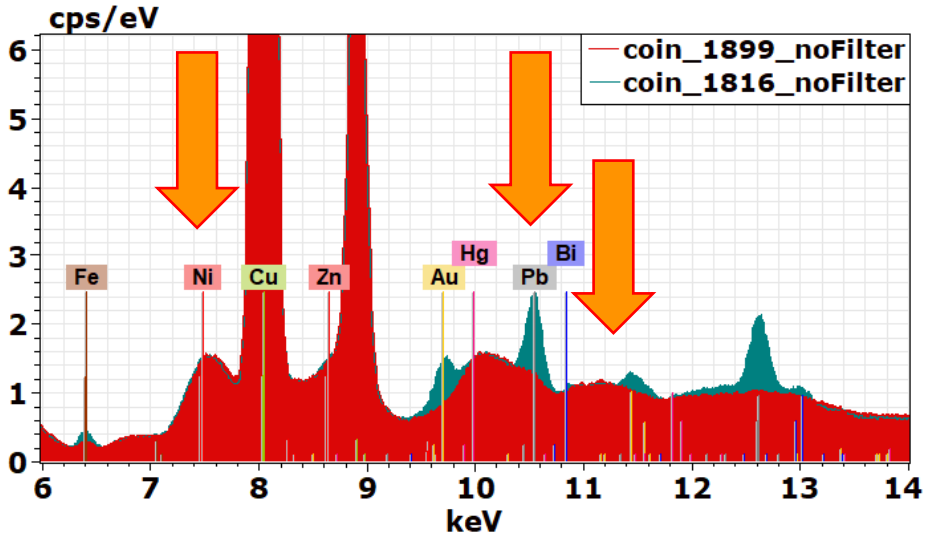
The Copper CHARM set: 10.1111/arcm.12117



BRUKER NANO ANALYTICS' CULTURAL HERITAGE WEBINAR SERIES 2023

Let’s get even more practical! Money throughout the centuries

Quantification of Metals
Getting rid of spectral artifacts



Diffractions peaks

Unclear presence of Ni, Zn, Hg or Bi in the sample.

Depending on its origin, historic silver **contains trace elements, such as gold**. Yet, in the 1880s, Berhard Moebius patented an electrochemical procedure in Europe and USA to separate gold and silver.

This marks the beginning of gold-free silver coins and can be used as a *terminus ante quem*.

But how can we properly identify traces that are hidden below diffraction peaks?

https://de.wikipedia.org/wiki/Bernhard_Moebius

Quantification of Metals
Getting rid of spectral artifacts

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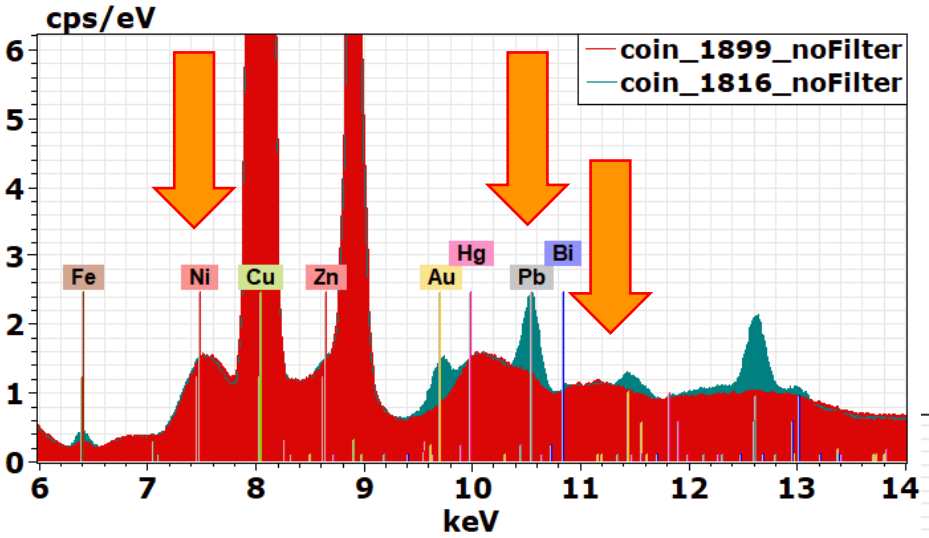

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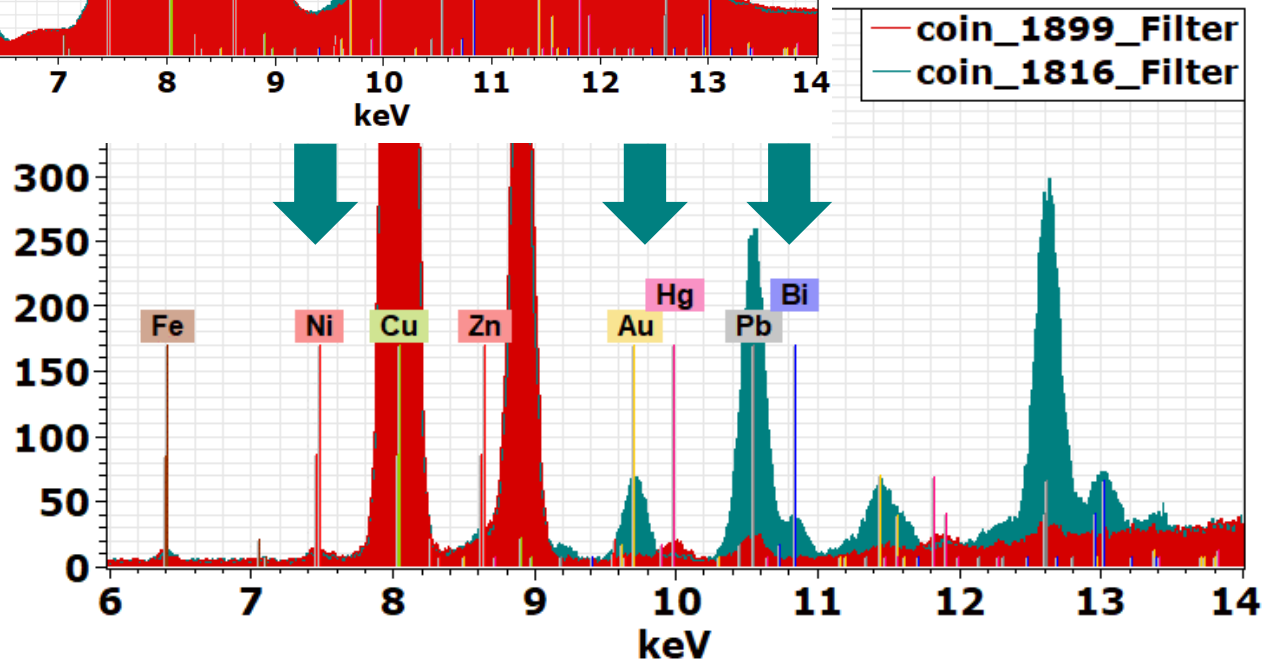
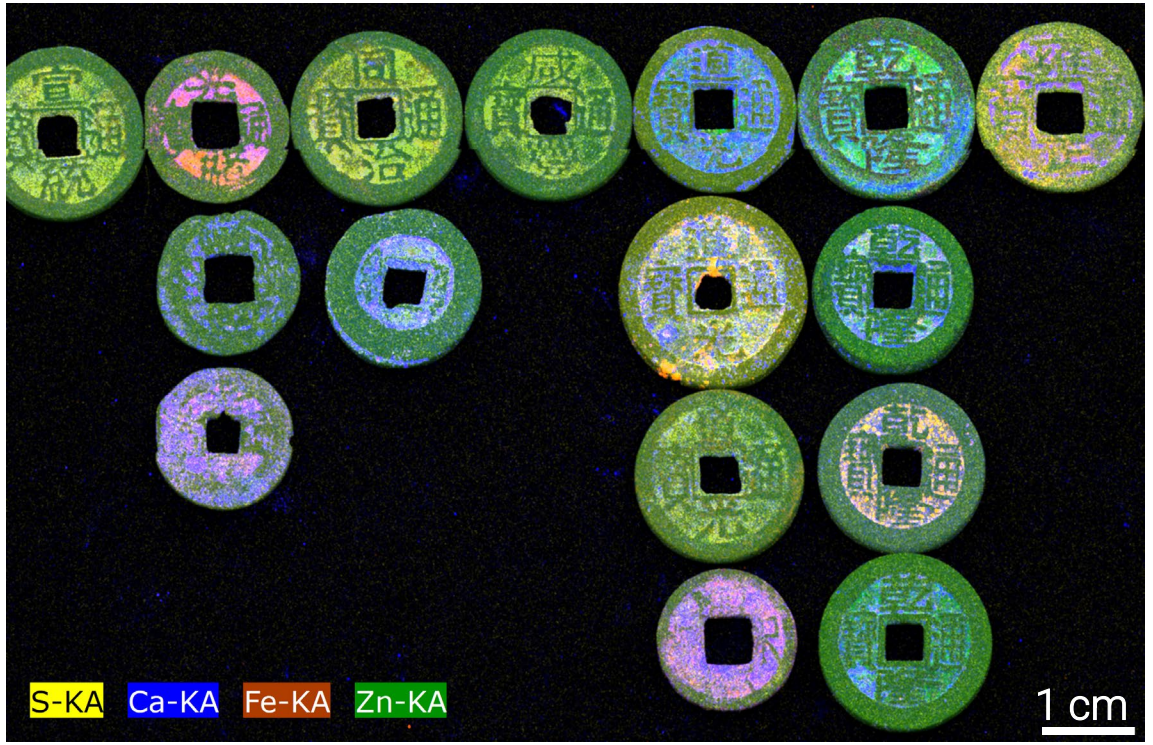


Quantification of Metals
Impact of surface effects and deterioration of the sample

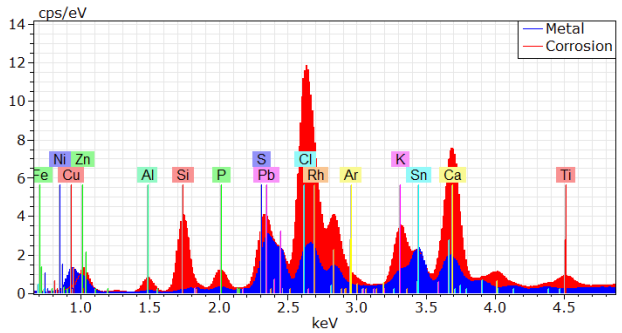
A set of Chinese coins dated 1667-1911.



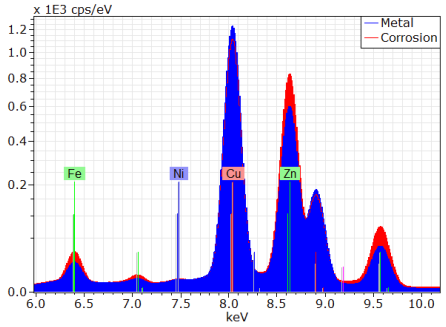
Micro-XRF mapping using an M4 TORNADO reveals the heterogeneity of the samples' surfaces due to corrosion and patina layers.

Quantification of Metals
Impact of surface effects and deterioration of the sample

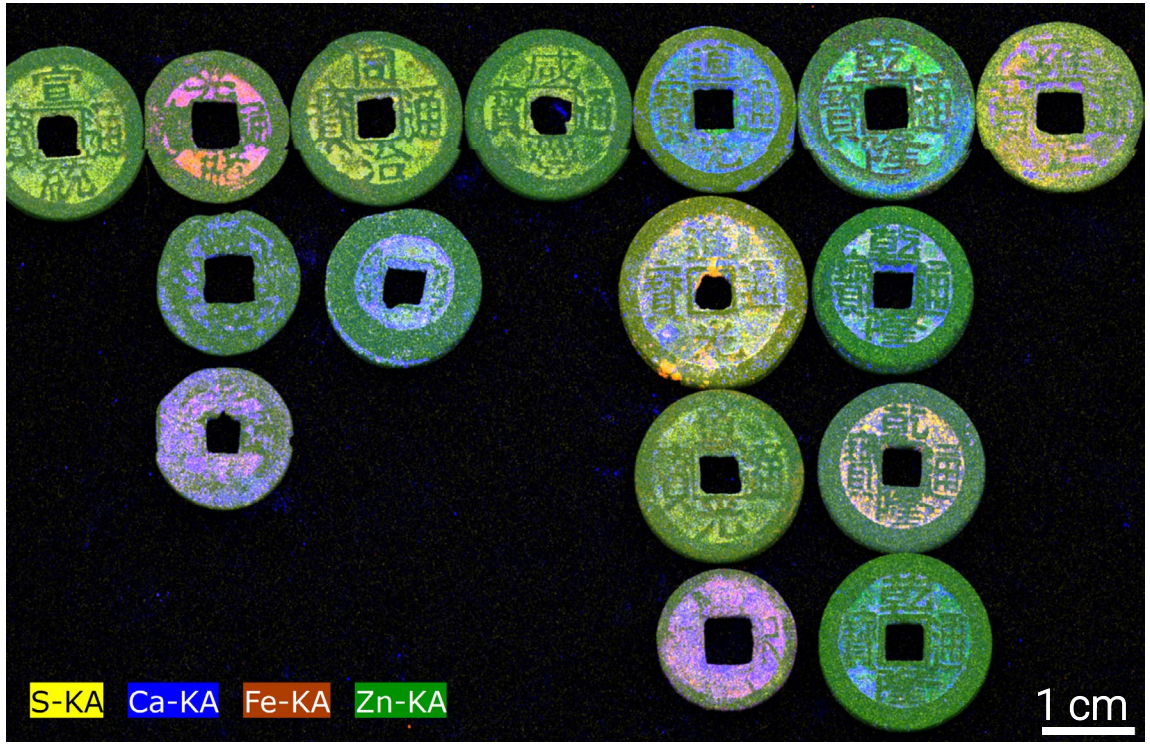
- Depending on the composition, metal surfaces can corrode or enrich in patina.
- This affects the XRF results especially in the lower energy range due to:
  - attenuation or
  - deposition of material.



Marker elements of corrosion and patina visible in the lower energy range.



Enrichment of Zn from the coin metal in the corrosion layer.



Micro-XRF mapping using an M4 TORNADO reveals the heterogeneity of the samples' surfaces due to corrosion and patina layers.

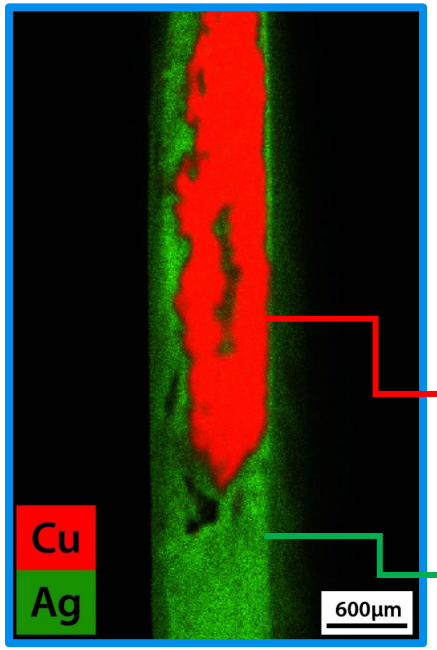
Quantification of Metals
Impact of surface effects and deterioration of the sample

Era	Date	Coin no.	Area of measurement	Mn	Fe	Cu	Zn	As	Se	Ag	Sn	Sb	Pb	Bi
Kong Xi	1667-1722	17	corroded area	0.05	1.06	61.61	32.34	0.03	0.00	0.01	0.68	1.88	2.00	0.00
			polished area	0.04	0.65	62.38	32.40	0.02	0.00	0.01	0.72	1.94	1.75	0.00
Yong zheng	1722-1735	16	corroded area	0.07	1.09	61.65	32.37	0.12	0.00	0.02	0.60	1.14	1.84	0.00
			polished area	0.08	1.04	62.23	32.37	0.11	0.00	0.04	0.69	1.30	1.94	0.00
Qian long	1735-1796	15	corroded area	0.00	0.46	57.02	35.41	0.62	0.00	0.00	0.25	0.04	5.87	0.00
			polished area	0.00	0.44	57.62	35.56	0.58	0.00	0.00	0.31	0.05	5.29	0.00
		14	corroded area	0.00	0.76	58.20	33.03	0.18	0.00	0.01	1.55	0.24	5.77	0.02
			polished area	0.00	0.77	58.86	33.30	0.16	0.00	0.00	1.70	0.27	4.73	0.01
		13	corroded area	0.00	0.39	56.89	35.66	0.18	0.00	0.00	0.67	0.03	5.90	0.00
			polished area	0.00	0.37	58.13	35.79	0.14	0.00	0.00	0.79	0.04	4.47	0.00
		12	corroded area	0.00	0.72	58.35	32.36	0.35	0.01	0.00	0.98	0.02	6.20	0.16
			polished area	0.00	0.57	48.87	43.31	0.25	0.00	0.00	0.70	0.01	5.79	0.13
Dao guang	1820-1850	11	corroded area	0.01	1.92	64.09	24.61	0.84	0.00	0.01	0.85	0.78	5.61	0.06
			polished area	0.00	0.95	61.51	30.23	0.54	0.00	0.01	0.69	0.68	5.23	0.04
		10	corroded area	0.04	1.16	61.15	35.05	0.01	0.00	0.00	0.44	0.17	1.86	0.00
			polished area	0.04	1.10	60.93	34.84	0.00	0.00	0.00	0.56	0.28	2.16	0.00
		9	corroded area	0.00	3.05	54.77	35.25	0.59	0.00	0.01	0.00	0.73	4.50	0.01
			polished area	0.00	1.25	57.35	32.40	0.35	0.00	0.00	0.01	0.72	7.30	0.01
		8	corroded area	0.00	1.00	60.41	33.56	0.62	0.00	0.00	0.00	0.71	3.18	0.03
			polished area	0.00	0.94	61.17	33.73	0.55	0.00	0.00	0.00	0.73	2.31	0.03
Xian feng	1850-1861	7	corroded area	0.03	0.61	60.17	36.80	0.00	0.00	0.01	0.41	0.67	1.14	0.01
			polished area	0.03	0.55	60.27	36.29	0.01	0.00	0.01	0.45	0.67	1.63	0.03
Tong zhi	1861-1875	6	corroded area	0.00	0.49	52.08	33.11	3.13	0.00	0.03	0.00	3.12	7.26	0.23
			polished area	0.00	0.57	54.78	33.28	3.98	0.00	0.04	0.00	3.48	3.32	0.15
		5	corroded area	0.02	0.68	58.70	37.13	0.00	0.00	0.01	0.54	1.00	1.38	0.03
			polished area	0.03	0.82	60.15	35.71	0.00	0.00	0.01	0.59	0.99	1.63	0.02
Guang xu	1875-1908	4	corroded area	0.01	1.32	59.47	29.80	0.72	0.00	0.02	0.88	0.50	5.55	0.05
			polished area	0.00	0.98	60.29	31.52	0.53	0.00	0.00	0.76	0.43	5.17	0.03
		3	corroded area	0.00	0.78	58.79	32.89	0.32	0.00	0.00	0.88	0.10	5.38	0.02
			polished area	0.00	0.71	58.36	33.17	0.35	0.00	0.01	1.24	0.15	5.72	0.03
		2	corroded area	0.01	1.00	60.54	32.02	0.46	0.00	0.00	0.70	0.44	4.06	0.02
			polished area	0.00	0.88	63.91	28.38	0.44	0.00	0.01	0.80	0.50	4.67	0.03
Xuan tong	1908-1911	1	corroded area	0.03	0.77	60.88	35.21	0.00	0.00	0.00	0.42	0.97	1.38	0.02
			polished area	0.03	0.84	59.38	36.22	0.00	0.00	0.01	0.41	0.95	2.07	0.02

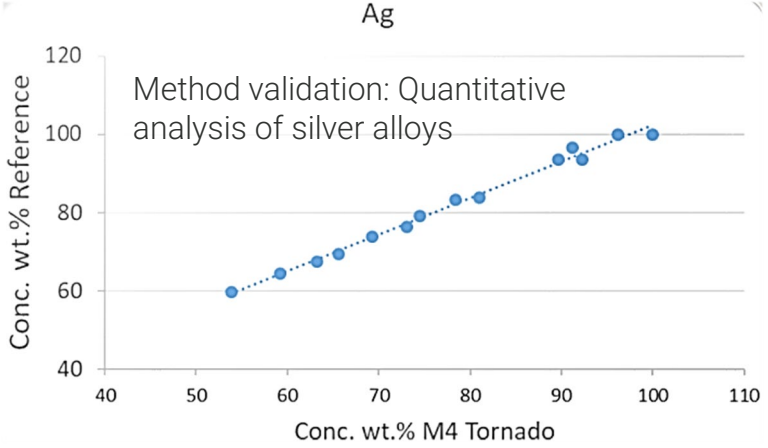
Quantification of Metals
Case study: A Roman silver coin

The composition of silver coins varied significantly, depending on time and manufacturing location. For Roman denarii, the silver content changed over time due to debasement of the currency.

M4 TORNADO Ag-intensity map of a Roman silver denarius, Severus Alexander 208 – 235 AC.



However, Ag may enrich on the surface!



wt. %	Ag	Cu	Zn	As	Au	Pb	Bi
Coin point 1 core	34.5	65.2	0.01	0.13	0.13	0.42	0.01
Coin point 2 core	35.1	64.6	0.01	0.13	0.12	0.42	0.01
Coin point 3 core	35.4	64.3	0.01	0.11	0.12	0.48	0.01

wt. %	Ag	Cu	Zn	As	Au	Pb	Bi
Coin point 1 surface	94.1	6.4	0.03	0.03	0.38	0.92	0.02
Coin point 2 surface	95.0	5.5	0.03	0.02	0.37	0.94	0.02
Coin point 3 surface	95.7	5.1	0.03	0.01	0.34	0.63	0.02

Quantification of Metals
Case study: A Roman silver coin

Depending on which values you look at, the coin can be assigned to different time periods.

Luckily, we know the exact date of the coin in this case…

M4 TORNADO Ag-intensity map of a Roman silver denarius, Severus Alexander 208 – 235 AC.

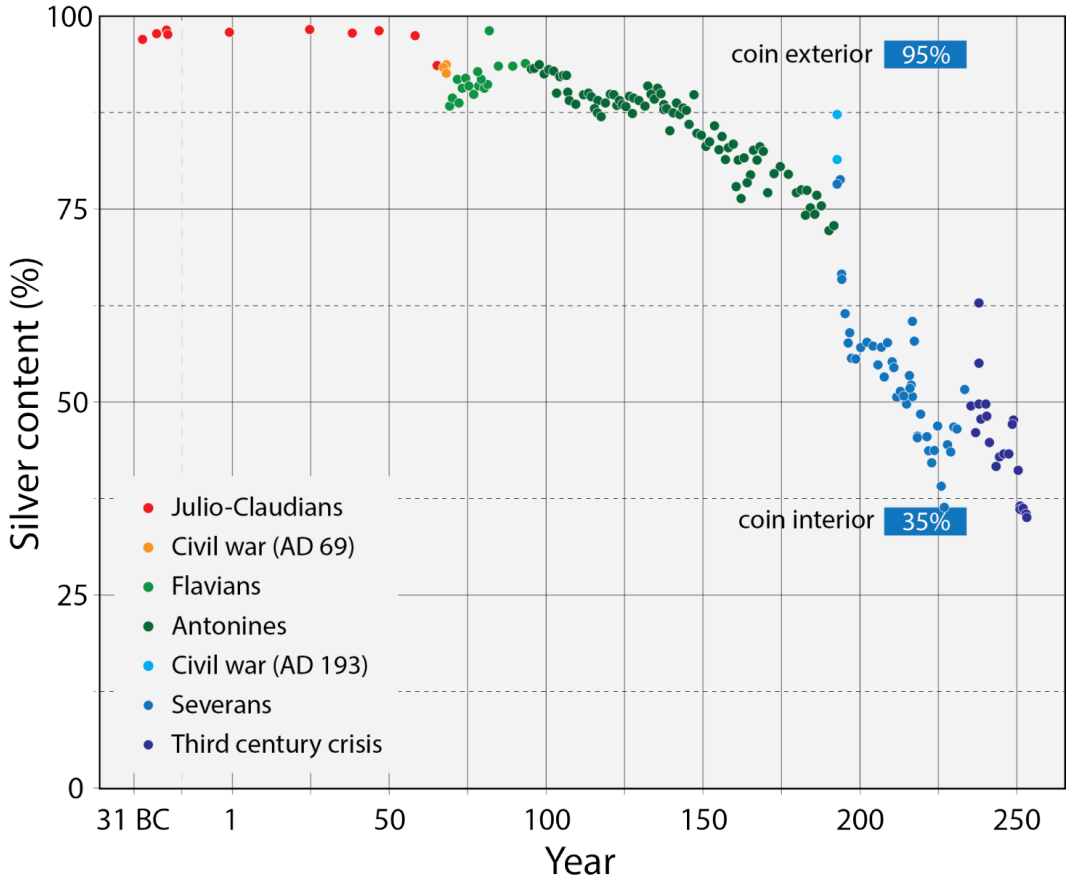


Image modified from Nicolas Perrault III, Wikipedia.

The silver content of the core at 35 ± 0.35 wt. % is in alignment with similar coins from the same period!

Quantification of Metals
Case study: A Lydian coin

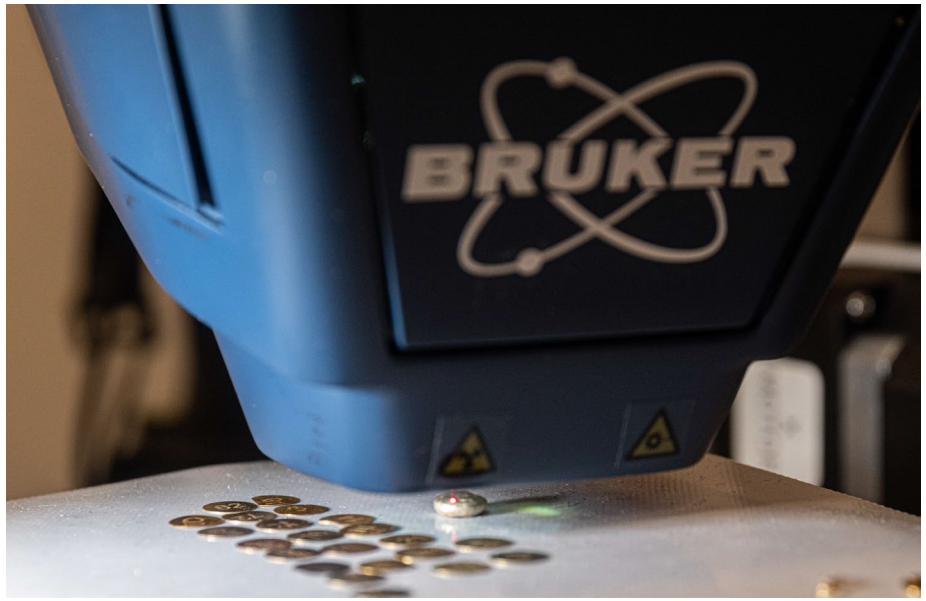
- Lydian Stater are the world's first coin currency.
- The Lydian Stater was the official coin of the Lydian Empire, introduced before Lydia fell to the Persian Empire.
- The earliest Staters probably date from the second half of the 7th century BC during the reign of King Alyattes (r. 619–560 BC).
- The Lydian Stater is thought be have been composed of electrum, a naturally-occurring gold-silver alloy. But was the alloy deliberately casted?

Lydia, Alyattes,
610–561 BC
1/12th Stater
⌀ 8 mm
1.16 g

*Millman, E. (2015, March 27). The Importance of the Lydian Stater as the World's First Coin. World History Encyclopedia. Retrieved from https://www.worldhistory. org/article/797/the-importance-of-the-lydian-stater-as-the-worlds/

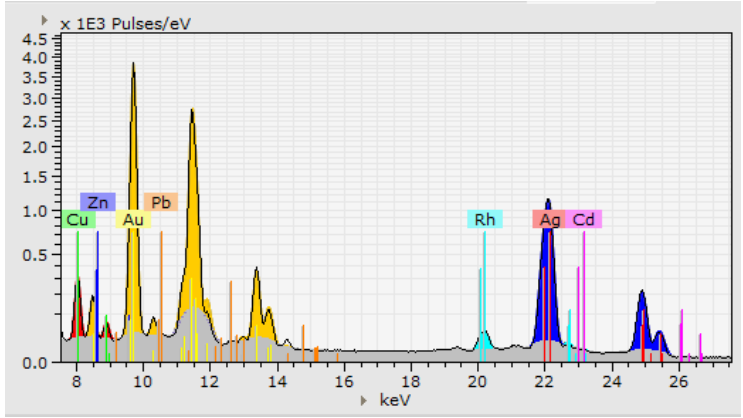
Quantification of Metals
Case study: A Lydian coin

Elio measuring the coin and gold reference samples.



Laser beam for focusing

Forward-calculated spectrum



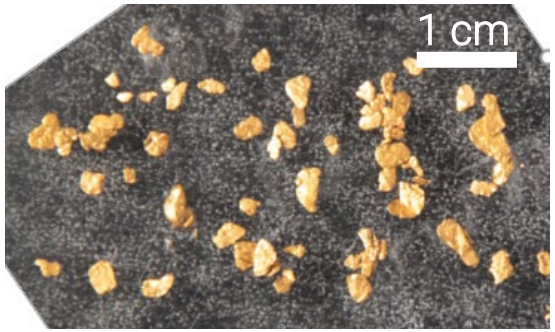
Results of the coin measurement using Esprit Reveal FP and reference samples.
Conditions: 50 kV, 80 µA, 120 s real time.

	Fe	Cu	Zn	Rh	Ag	Cd	Au	Pb	**Sum**
Au_120 s II	0.13	1.70	0.00	0.00	45.47	0.00	52.65	0.06	**100.00**
Au_120 s I	0.12	1.69	0.00	0.00	45.71	0.00	52.42	0.06	**100.00**

Quantification of Metals
Case study: A Lydian coin

Was the coin made from electrum, a naturally-occurring gold-silver alloy?

Coin: Au: 52.5 wt.%
Ag: 45.6 wt.%
Cu: 1.69 wt.%

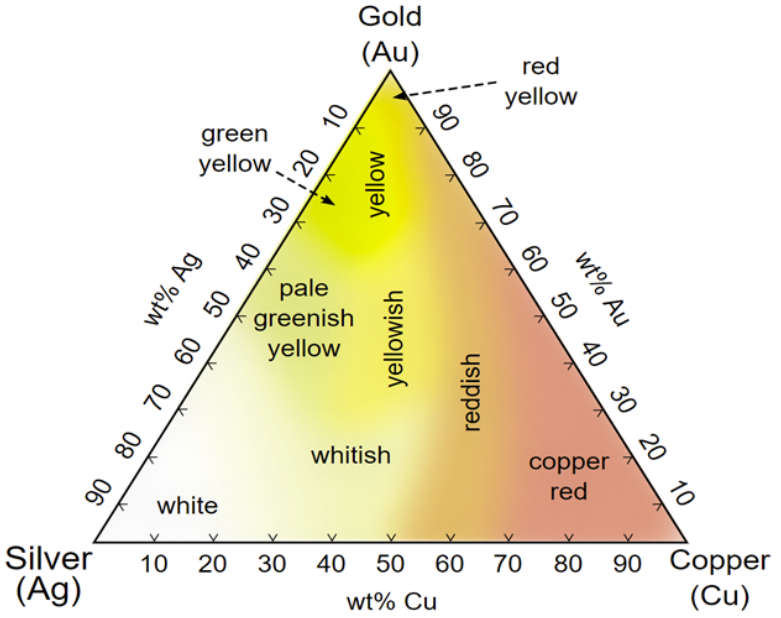


There has been a long discussion whether the coin alloy was of a natural origin or created on purpose.

Currently it is mostly accepted that the alloy was casted on purpose.

Measured values of the coin analyzed are in agreement with literature* at 52.5 ± 2 wt. %

Interestingly the composition of the coin is very close to the lowest possible Au-Ag alloy with a yellow appearance.



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https://commons.wikimedia.org/wiki/File:Ag-Au-Cu-colours-english.svgc

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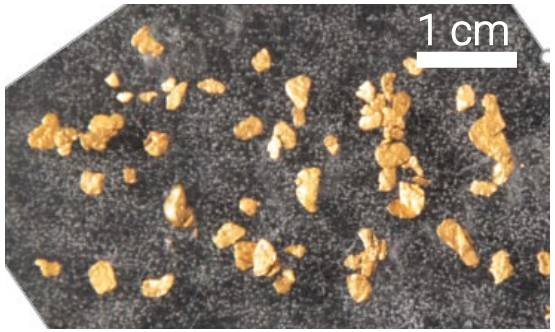

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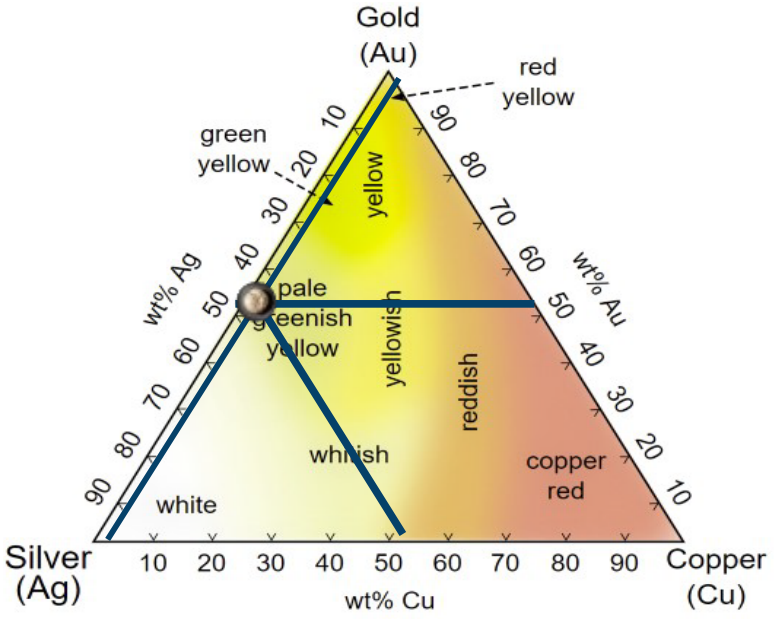

Image modified from:
https://commons.wikimedia.org/wiki/File:Ag-Au-Cu-colours-english.svgc

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Navigating around pitfalls in Quantitative Analysis
A “What-to-do”

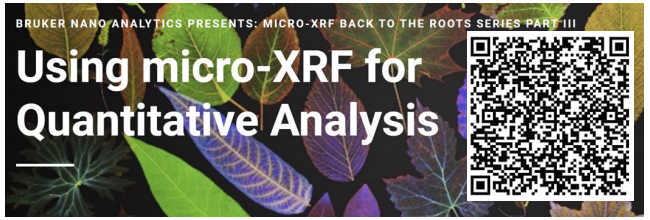
Sample characteristics	Work-around
2D Heterogeneity, e.g. segregation, exsolution	▪ Sufficient number of point measurements or XRF mappings ▪ Use large spot size
3D Heterogeneity, e.g. layer systems (patina or corrosion)	▪ Sample preparation ▪ Only considering heavy elements
Poor signal to noise ratio of trace elements	▪ Use filter ▪ For light elements below Sulfur: use Helium ▪ Optimize measurement time
Diffraction peaks	▪ Use filter
Matrix effects of „heavy“ glass (Pb)	▪ Standard-supported FP quantification
Attenuation of elemental signals by glazes	▪ Find an uncovered position ▪ Creation of a set of standards for an empirical approach ▪ Only consider high-Z elements

Conclusion

- The multi-elemental sensitivity and non-invasive nature of XRF make it an ideal investigative tool for both qualitative or semi-quantitative analysis of Cultural Heritage objects.
- Quantification is often hampered by the fact that many materials do not have a defined composition within the analytical volume of the technique.
- Likewise, each instrument has its own specifications that equal its performance.

However, knowledge on both the sample, as well as the instrument capabilities combined with a well-defined research question allows to refine the methodological approach leading to accurate and reliable results!

For more general information see:

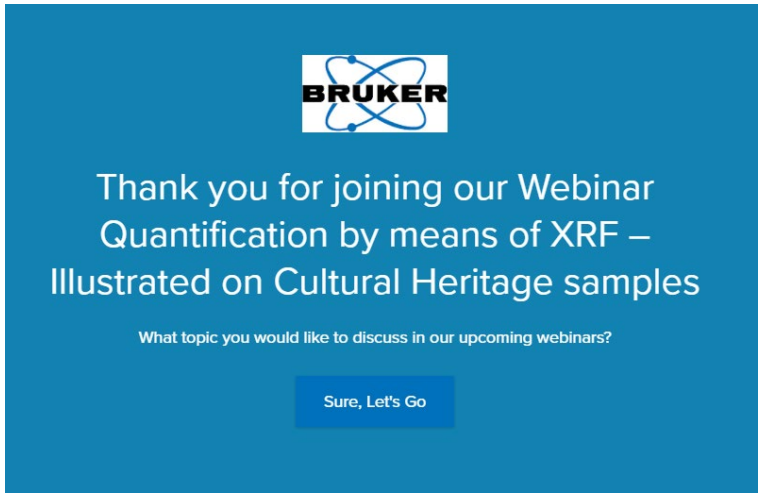


Art & Conservation Webinar Series
Quantification by means of XRF in Cultural Heritage Studies

There are many topics involving XRF in Cultural Heritage!

We are interested to know which topic(s) you would like to learn more about in upcoming webinars?

- For live sessions: At the end of this webinar, you will be automatically redirected to a short survey via the Bruker Webex side.
- For on-demand: You can find a link in the video description or follow the QR code on the right.

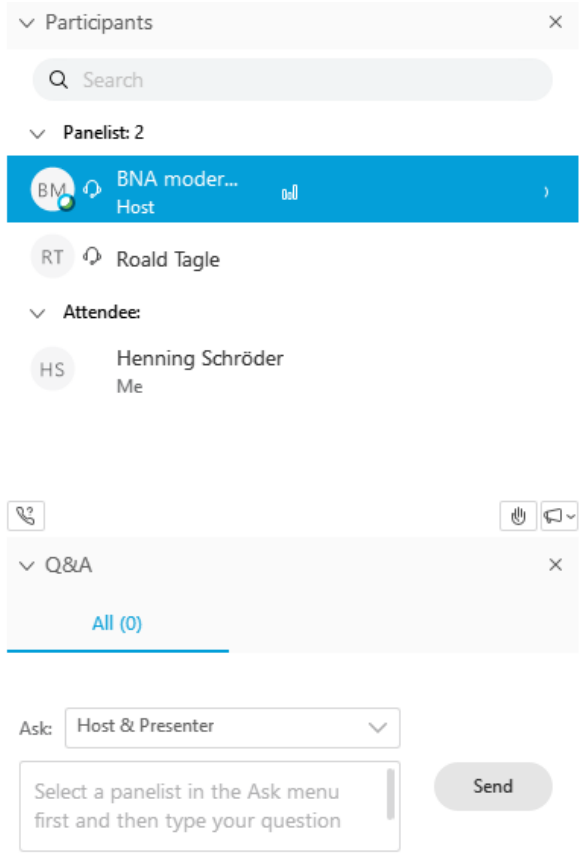


Art & Conservation Webinar Series
Quantification by means of XRF in Cultural Heritage Studies

If you have questions during this webinar,

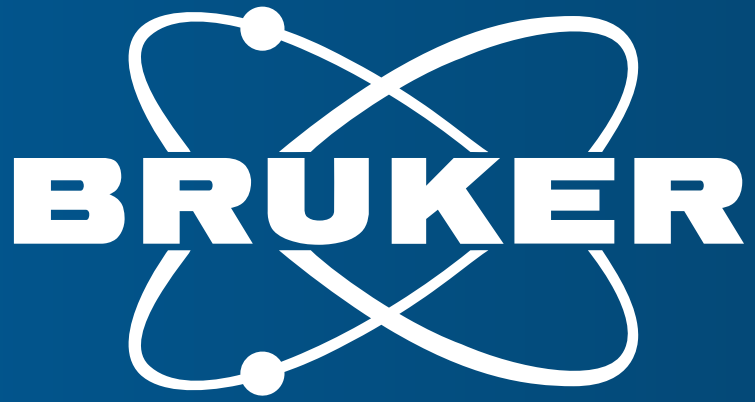
please **type your questions**, thoughts, or comments in the

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



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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