BRUKER NANO ANALYTICS

Handheld and Portable XRF in Cultural Heritage – Part II Paintings, Pigments, and surfaces with challenges

Maggi Loubser,
Senior Lecturer and Coordinator
Tangible Heritage Conservation Programme

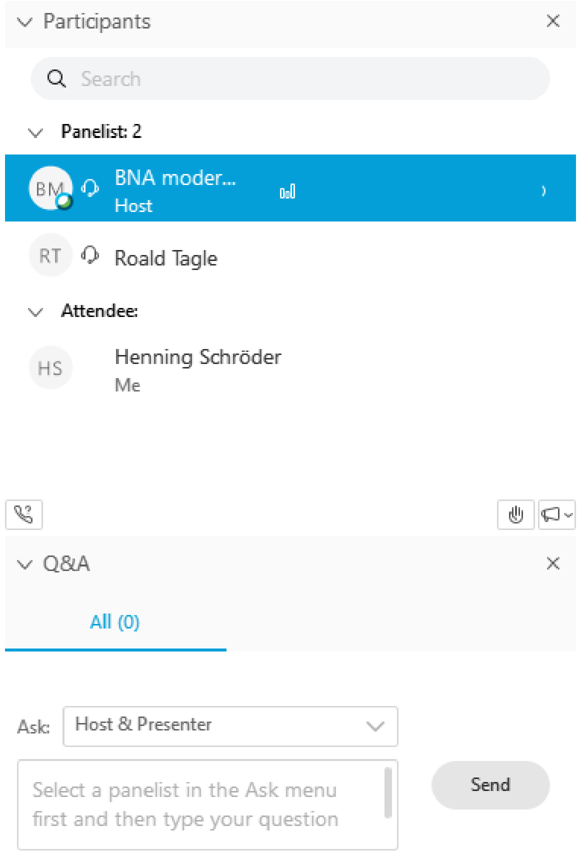
Dr. Kathrin Schneider,
Application Scientist HH-XRF
Bruker Nano Analytics

Art & Conservation Webinar Series
Handheld XRF in Cultural Heritage Studies

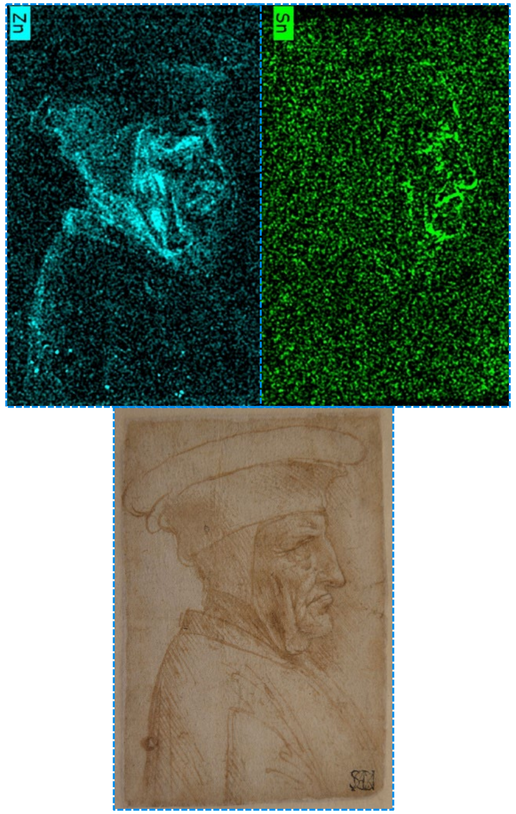
If you have questions during this webinar, please **type your questions**, thoughts, or comments in the **Q&A box** and **press Send**.

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

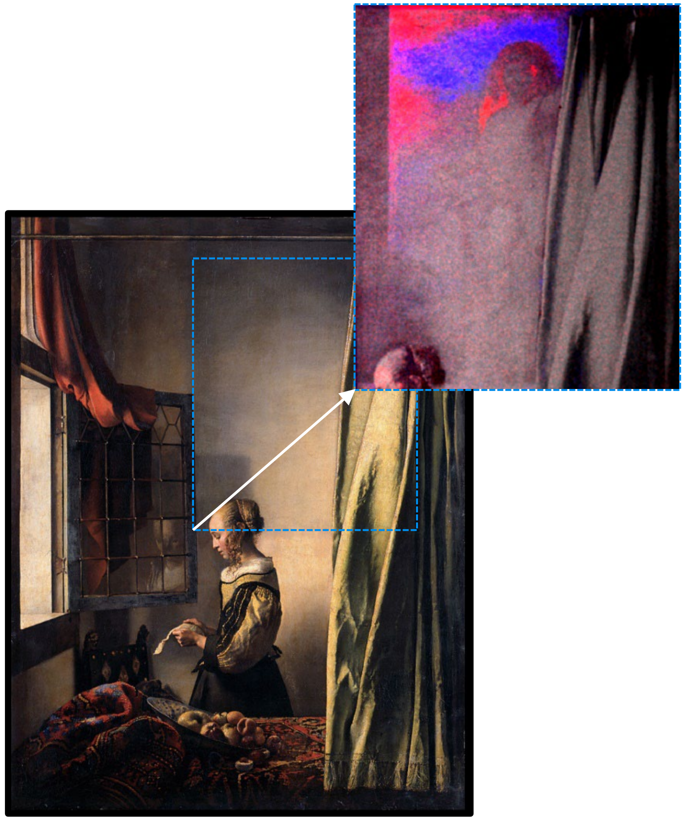
Any unanswered questions or comments will be answered and discussed by e-mail or in another WebEx session.



Art & Conservation Webinar Series
Handheld XRF in Cultural Heritage Studies



Trace element sensitive



Information from depth in the sample

No sample preparation

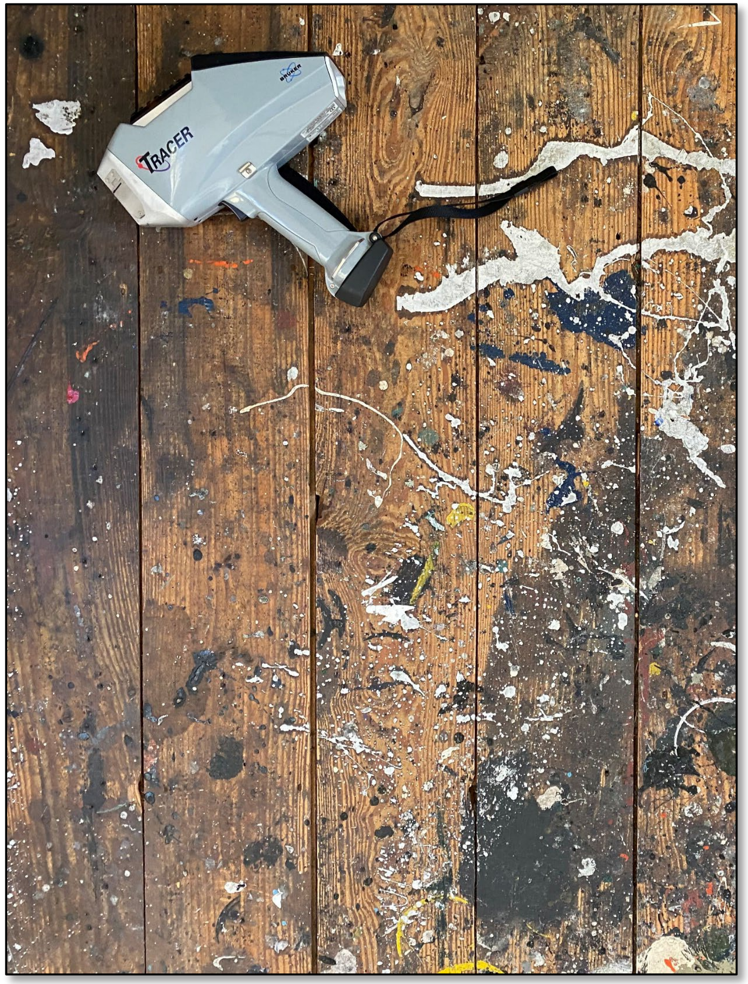
Art & Conservation Webinar Series
Handheld XRF in Cultural Heritage Studies

Handheld XRF in Cultural Heritage Studies
Webinar Series

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

BRUKER

- Three webinars specifically addressing use of handheld-XRF, presented as part of the ongoing series on elemental analysis in Cultural Heritage Studies
  - I. Back to basics - taking control of your path to meaningful information
  - II. Approaches to challenging measurements - Paintings, pigments and objects
  - III. Quantitative data - what do the numbers mean?



Back to basics with Handheld XRF
Our speakers

Maggi Loubser

- Programme Manager – Tangible Heritage Conservation University of Pretoria

Dr. Kathrin Schneider

- Application Specialist – Bruker Nano Analytics

Paintings and Pigments and surfaces with challenges
Presentation Outline

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

01 The sample (nonmathematical matrix effects)

02 Qualitative interpretations

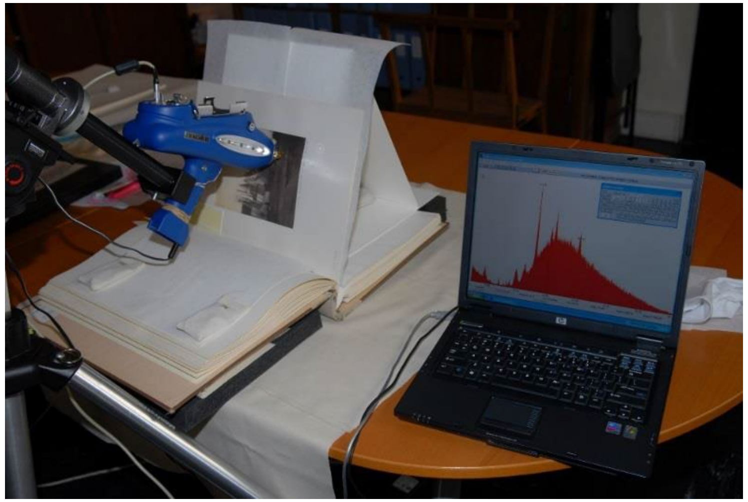
03 Ambiguity in results

04 Repeatability

05 Game plan – How do I approach a project to get the maximum out of my data?

01 The sample (non-mathematical matrix effects) Thickness

- Source of interferences:
  - Matboard
  - Paper substrate
  - Table
  - Underlying pages/materials



Cradle: bound photograph collection.

Thin Al sheet: bound illuminated manuscript.

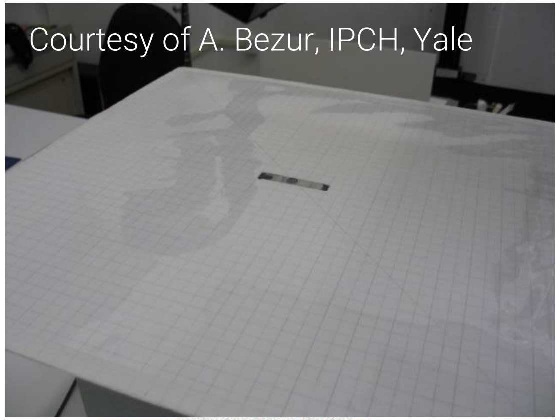
Foam to provide distance from tabletop.

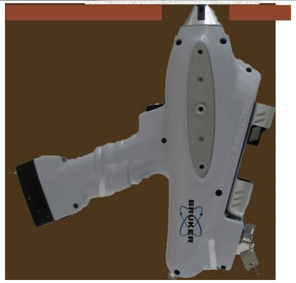
No backscatter – safety lock switch off X-ray Tube!

01 The sample (non-mathematical matrix effects)
Thickness

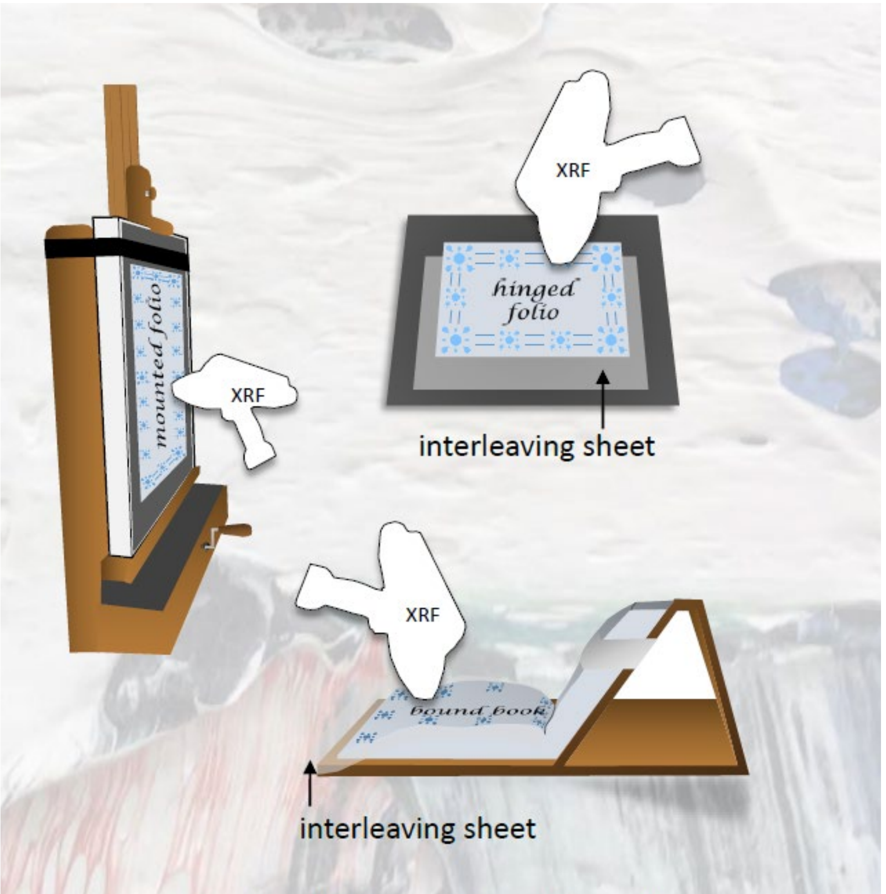
- Approach to reducing interferences:
  - Strategic selection of areas of analyses
  - Isolate area of analysis
  - Reduce contribution by using low-Z material under area of analysis
  - Analysis of background

Safety of object and user.





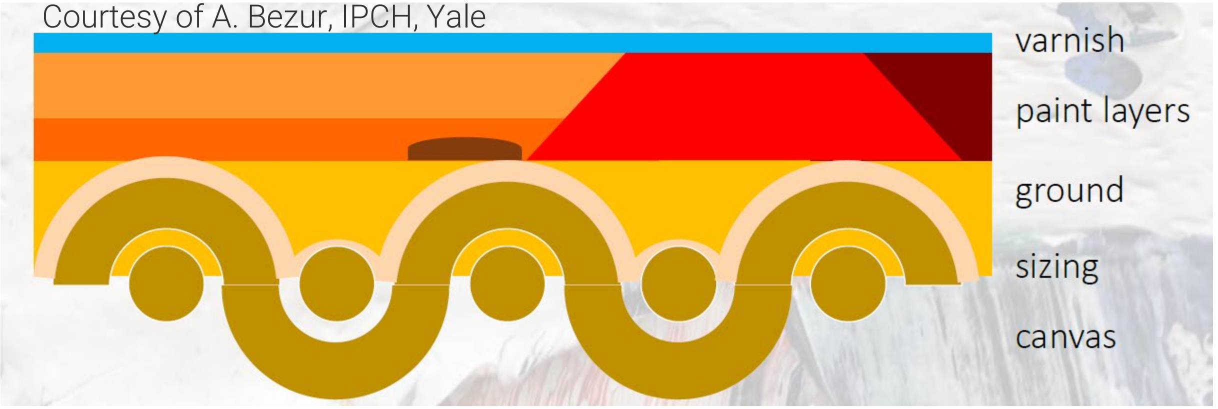
Setup to analyse from backside/ face down.



Trentelman, K., Patterson, C., and Turner, N. in Handheld XRF for Art and Archeology, edited by Shugar, AN and Mass, JL (2012)

01 The sample (non-mathematical matrix effects) Which layer am I looking at?

- Heterogeneity: Complex layering and /or mixtures
- Penetration depth
- Substrate vs. paint vs. conservation treatment
- Ceramics vs. glaze

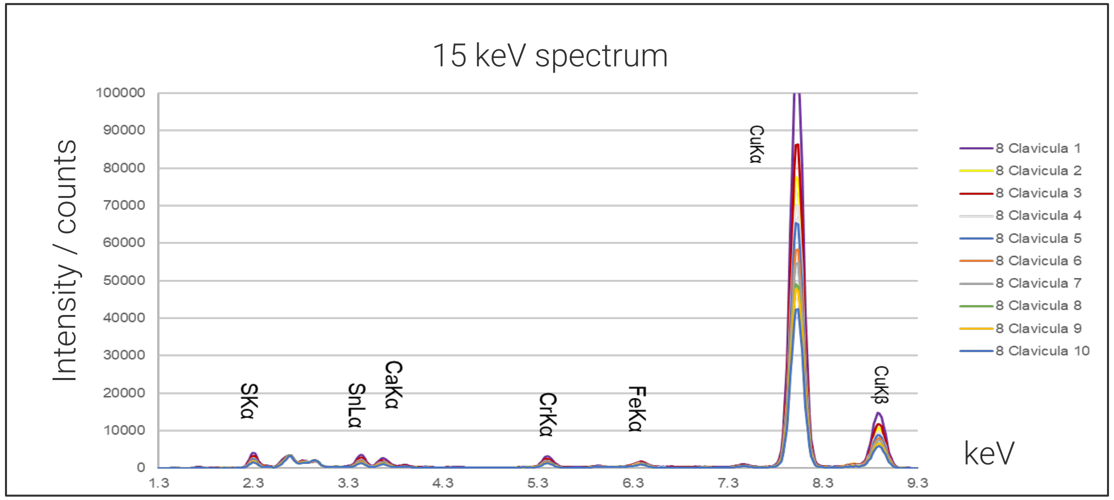


Hezekiel Ntuli	ElapsedTime										
GeoExploration	90 seconds										
Oxide3phase	Bushman										
ID	MgO	Al2O3	SiO2	P	S	K2O	Ca	Ti	Cr	Mn	Fe
Mid chest	1.62	26.13	64.13	0.02	0.43	1.65	0.20	0.58	0.0102	0.01	2.86
Right Shoulder	1.75	24.36	65.66	0.03	0.66	1.58	0.20	0.61	0.0079	0.01	2.95
Left scapula	2.24	22.52	61.22	0.04	0.59	1.51	0.18	0.58	0.0087	0.01	2.86
Back of head	1.41	21.89	61.49	< LOD	0.65	1.50	0.16	0.62	0.0073	0.02	2.87
Average	1.75	23.73	63.12	0.03	0.58	1.56	0.19	0.60	0.01	0.01	2.88
Standard deviation	0.35	1.92	2.14	0.01	0.11	0.07	0.02	0.02	0.001	0.001	0.04
underside no slip	1.37	18.76	50.03	< LOD	0.12	1.33	0.14	0.49	0.0071	0.01	3.01

01 The sample (non-mathematical matrix effects)
Uneven surfaces: Curves, complex artifacts

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

BRUKER



relative concentrations	Al	Si	P	S	Cr	Mn	Fe	Ni	Cu	Zn	As	Sn	Sb	Pb
8 clavicula 1	0.64	1.32	0.08	0.39	0.46	0.04	0.28	0.31	85.36	0.17	0.02	7.40	0.18	3.31
8 clavicula 2	< LOD	0.72	0.07	0.31	0.36	0.03	0.32	0.28	85.83	0.42	< LOD	7.75	0.19	3.44
8 clavicula 3	< LOD	1.14	0.09	0.36	0.43	0.04	0.38	0.30	85.54	0.25	0.05	7.54	0.12	3.21
8 clavicula 4	< LOD	1.32	0.09	0.36	0.50	< LOD	0.23	0.29	86.48	< LOD	0.04	7.39	0.19	2.74
8 clavicula 5	< LOD	1.28	0.09	0.36	0.51	< LOD	0.23	0.28	86.17	< LOD	0.05	7.48	0.22	2.74
8 clavicula 6	0.97	1.23	0.09	0.36	0.43	0.03	0.44	0.26	85.49	< LOD	< LOD	7.38	0.18	3.07
8 clavicula 7	0.99	1.09	0.10	0.35	0.41	0.03	0.41	0.31	85.32	< LOD	0.05	7.55	0.21	3.14
8 clavicula 8	< LOD	1.08	0.08	0.34	0.42	0.02	0.42	0.29	85.38	< LOD	0.03	7.45	0.25	3.30
8 clavicula 9	1.17	0.98	0.11	0.36	0.46	0.04	0.45	0.28	84.60	< LOD	0.05	7.96	0.20	3.28
8 clavicula 10	< LOD	1.03	0.08	0.33	0.42	0.04	0.42	0.31	84.75	< LOD	0.05	7.99	0.29	3.31
Average	**0.94**	**1.12**	**0.09**	**0.35**	**0.44**	**0.03**	**0.36**	**0.29**	**85.49**	**0.28**	**0.04**	**7.59**	**0.20**	**3.15**
Standard deviation	**0.22**	**0.18**	**0.01**	**0.02**	**0.04**	**0.01**	**0.08**	**0.02**	**0.57**	**0.13**	**0.01**	**0.23**	**0.05**	**0.24**
Relative Standard deviation	**23.30**	**16.53**	**14.92**	**5.95**	**9.69**	**16.63**	**23.26**	**5.61**	**0.67**	**46.00**	**22.38**	**3.05**	**22.51**	**7.57**
min	**0.64**	**0.72**	**0.07**	**0.31**	**0.36**	**0.02**	**0.23**	**0.26**	**84.60**	**0.17**	**0.02**	**7.38**	**0.12**	**2.74**
max	**1.17**	**1.32**	**0.11**	**0.39**	**0.51**	**0.04**	**0.45**	**0.31**	**86.48**	**0.42**	**0.05**	**7.99**	**0.29**	**3.44**

01 The sample (non-mathematical matrix effects) Heterogeneity: Glass, Alloy vs. Patina

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

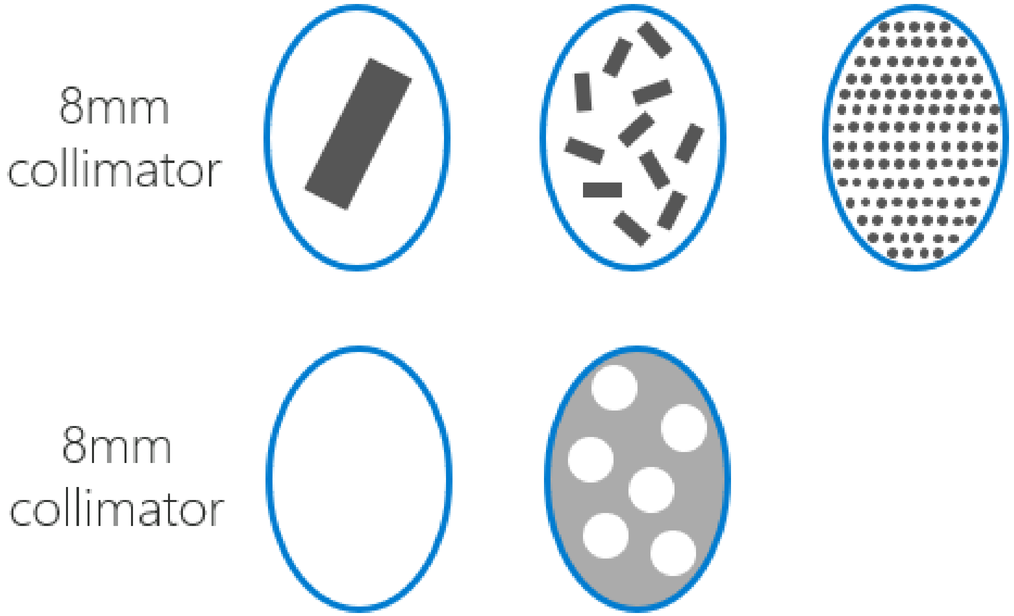
BRUKER

ID	MgO	Al2O3	SiO2	P	S	K2O	Ca	Ti	Cr	Mn	Fe
Central bust	1.79	18.22	45.84	0.06	0.70	1.23	1.77	0.48	0.0072	0.01	2.67
Right shoulder under signature	1.22	11.39	32.95	0.03	1.37	0.86	2.13	0.37	0.0058	0.01	2.15
Back of neck	1.90	18.87	46.09	0.04	1.09	1.25	2.45	0.46	0.0079	0.01	2.73
middle of back	2.15	15.79	39.28	0.75	0.71	1.25	1.37	0.49	0.0072	0.01	2.99
back of hair	1.82	18.15	43.20	0.03	0.75	1.25	2.18	0.46	0.0087	0.01	3.00
left shoulder back	1.48	22.77	53.27	0.04	1.24	1.55	2.38	0.53	0.0109	0.01	3.27
left clavicula	1.75	19.46	48.35	0.07	0.57	1.28	1.60	0.46	0.0090	0.01	2.57
right clavicula	< LOD	19.76	48.28	0.09	0.70	1.29	1.96	0.48	0.0080	0.01	2.72
right eyebank	< LOD	23.97	62.45	0.06	1.18	1.62	3.20	0.53	0.0088	0.01	3.23
left forehead	1.59	25.80	56.30	0.04	1.20	1.68	2.71	0.58	0.0095	0.01	3.07
Average	**1.71**	**19.42**	**47.60**	**0.12**	**0.95**	**1.33**	**2.18**	**0.48**	**0.008**	**0.01**	**2.84**
Standard deviation	**0.28**	**4.13**	**8.42**	**0.22**	**0.29**	**0.24**	**0.54**	**0.06**	**0.001**	**0.002**	**0.34**

Hezekiel Ntuli,
Large Zulu Bust

01 The sample (non-mathematical matrix effects)
Beam size vs. analytical area

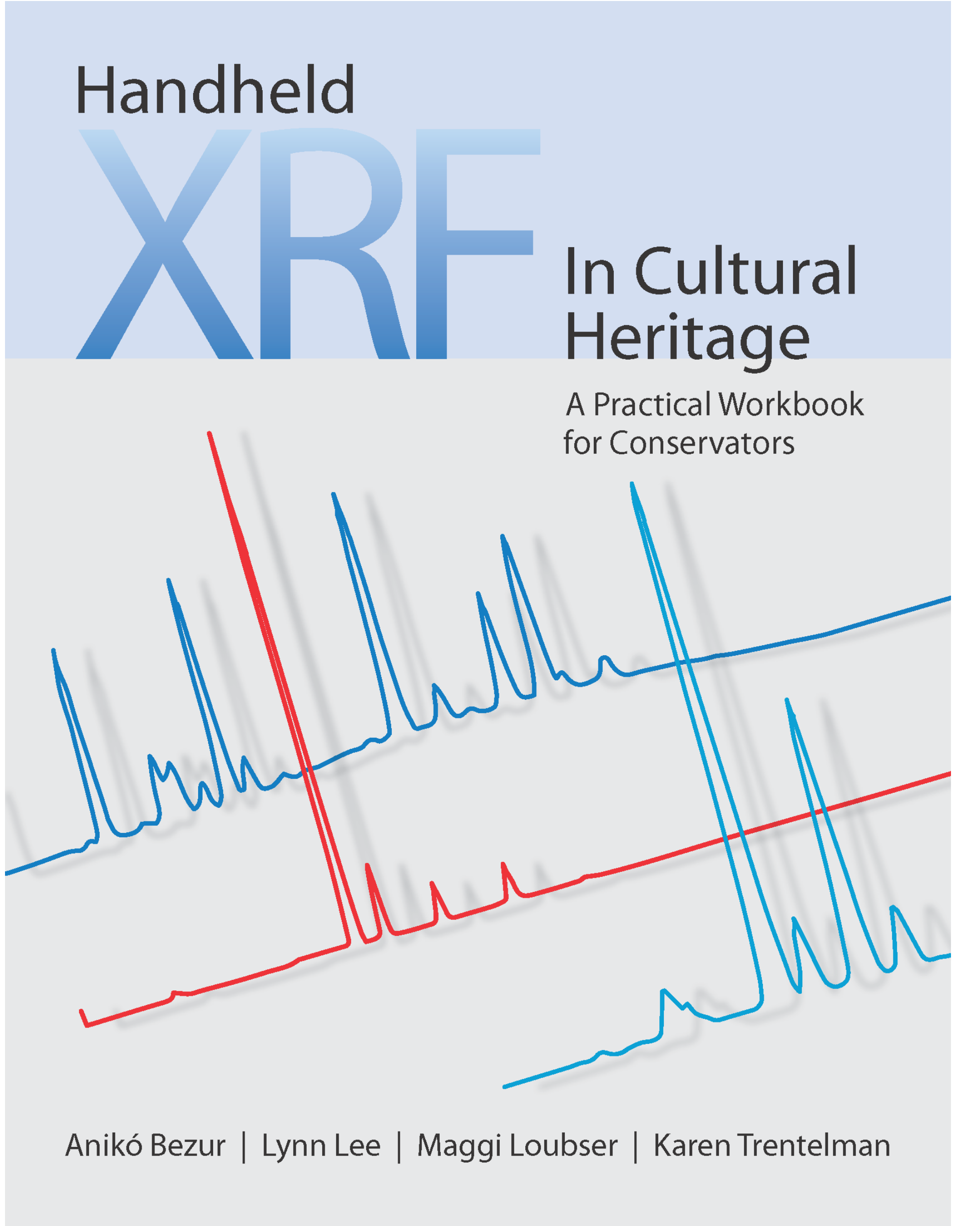
- Beam size vs Analytical target



Quick Advertisement Break!

Quick Advertisement Break!

The book is available from the GCI website as a free, downloadable pdf: https://gty.art/3a7Mjaa



UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

BRUKER

Practical exercises for students

TABLE 3.1. Suggested mock-up samples for use in the exercises, cross-referenced by concept to be explored and class of material.

	Detection of high vs. low Z elements	Detection of elements in different layers	Detection of elements in substrate	Detection of elements in fine mixtures (inhomogeneity on micro scale)	Detection of elements in rough/uneven surfaces	Detection of elements in coarse mixtures (inhomogeneity on macro scale)	Inference of presence of organic matter
Painted surfaces	UM Blue-CoBlue (3.1b) Bone Black (3.1c) Alizarin (3.1d)	Ti White-Lithopone layers (3.2c) Ti White-PG7 layers (3.2e)	UM Blue-CoBlue (3.1b) Bone Black (3.1c)	Ti White-Lithopone mixtures (3.2c) Prussian Blue serial dilutions (3.3a)	Accordion pleat (3.4a)	Cd Red lines (3.2a)	"Varnish" layer (3.1b) PMMA backing (3.3b, 3.3d)
Manuscripts/ Drawings	UM Blue-CoBlue (3.1b)	Interleaving material (3.2d, 3.2e)	Drawing materials on paper (3.3d)	Ti White-Lithopone mixtures (3.2c) Prussian Blue serial dilutions (3.3a)	Accordion pleat (3.4a)	Cd Red lines (3.2a)	"Varnish" layer (3.1b) PMMA backing (3.3b, 3.3d)
Photographs	Bone Black (3.1c) Alizarin (3.1d)	B&W photograph (3.3b)	B&W photograph (3.3b)				
Metals	Copper alloy (3.1e) Lead-tin solder (3.1f)	"Corroded" surface (3.2b) Lead-tin solder with Al sheets (3.2d)	Copper alloy (3.1e) Lead-tin solder (3.1f)	Copper alloy (3.1e) Lead-tin solder (3.1f)	"Corroded" surface (3.2b)	Lead-tin solder (3.1f)	"Varnish" layer (3.1b) PMMA backing (3.3b, 3.3d)
Ethnographic/ Archaeological		Ti White-Lithopone layers (3.2c) Ti White-PG7 layers (3.2e)	Drawing materials on paper (3.3d)	Ti White-Lithopone mixtures (3.2c) Prussian Blue serial dilutions (3.3a)	Accordion pleat (3.4a)	Cd Red lines (3.2a) Lead-tin solder (3.1f)	"Varnish" layer (3.1b) PMMA backing (3.3b, 3.3d)
Ceramics/ Glass	Glass (3.1a)		Glass (3.1a)		Accordion pleat (3.4a)		

Practical exercises for students
Exercise 3.2a: Example for beam size vs. analytical area

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

BRUKER

EXERCISE 3.2a:

Cadmium Red Lines on Titanium White Ground with Various Line Widths

In this exercise, you will study the effect of the spot size of the instrument relative to the scale of the feature under examination. XRF spectra are collected from a sample on which lines of cadmium red paint of different widths are painted over a titanium white ground, and the degree to which the spot size of the instrument allows for the individual elements to be isolated is explored. This exercise exemplifies the situation of analyzing fine details, such as might be found in a manuscript or drawing.

SUGGESTED SAMPLE

Titanium white applied to bare, unprimed canvas. Lines of cadmium red of different widths (1 mm, 3 mm, and 7 mm) are subsequently painted on top of the titanium white ground (fig. 3.7). See appendix 2 for instructions.

EXPERIMENT

Collect spectra from the sample using conditions A–D (table 3.9). Position the sample (or instrument) so that the painted surface is in front of the XRF nose. The sample must lie flat, with no air gap. If needed, place weights at the edge of the canvas but not in the direct beam path of the X-rays.

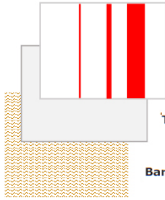


FIGURE 3.7. Diagram showing layers of sample used to study effect of spot size. Titanium white is applied to bare canvas, followed by lines of cadmium red of different widths.

TABLE 3.9. Conditions A–D for effect of spot size on cadmium red (CR) lines on titanium white ground with various line widths for exercise 3.2a.

Instrument:							
	Sample / Condition	**Mode**	**Voltage (V)**	**Current (I)**	**Atm.**	**Filter**	**Time**
A	Titanium white on canvas						
	High V, low I, air, no filter, 30s						
	Spectrum name:						
B	Thin CR line (1 mm width)						
	High V, low I, air, no filter, 30s						
	Spectrum name:						
C	Medium CR line (3 mm width)						
	High V, low I, air, no filter, 30s						
	Spectrum name:						
D	Thick CR line (7 mm width)						
	High V, low I, air, no filter, 30s						
	Spectrum name:						

Handheld XRF in Cultural Heritage 73

EXERCISE 3.2a:

Cadmium Red Lines on Titanium White Ground with Various Line Widths

In this exercise, you will study the effect of the spot size of the instrument relative to the scale of the feature under examination. XRF spectra are collected from a sample on which lines of cadmium red paint of different widths are painted over a titanium white ground, and the degree to which the spot size of the instrument allows for the individual elements to be isolated is explored. This exercise exemplifies the situation of analyzing fine details, such as might be found in a manuscript or drawing.

Practical exercises for students
Exercise 3.2a: Example for beam size vs. analytical area

SUGGESTED SAMPLE

Titanium white applied to bare, unprimed canvas. Lines of Cadmium red of different widths (1 mm, 3 mm, and 7 mm) are subsequently painted on top of the titanium white ground (fig. 3.7). See appendix 2 for instructions

EXPERIMENT

Collect spectra from the sample using conditions A-D (table 3.9). Position the sample (or instrument) so that the painted surface is in front of the XRF nose. The sample must lie flat, with no air gap. If needed, place weights at the edge of the canvas but notin the direct beam path of the X-rays.

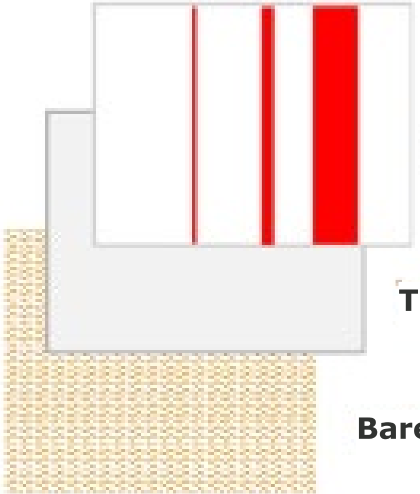


Figure 3.7. Diagram showing layers of sample used to study effect of spot size. Titanium white is applied to bare canvas, followed by lines of Cadmium red of different widths.

Practical exercises for students
Exercise 3.2a: Example for beam size vs. analytical area

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

BRUKER

Table 3.9. Conditions A–D for effect of spot size on cadmium red (CR) lines on titanium white ground with various line widths for exercise 3.2a.

Instrument:							
	Sample / Condition	Mode	Voltage (V)	Current (I)	Atm.	Filter	Time
A	Titanium on white canvas						
	High V, low I, air, no filter, 30 s						
	Spectrum name:						
B	Thin CR line (1 mm width)						
	High V, low I, air, no filter, 30 s						
	Spectrum name:						
C	Medium CR line (3 mm width)						
	High V, low I, air, no filter, 30 s						
	Spectrum name:						
D	Thick CR line (7 mm width)						
	High V, low I, air, no filter, 30 s						
	Spectrum name:						

Practical exercises for students
Exercise 3.2a: Example for beam size vs. analytical area

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

EXPECTED OUTCOMES TO EXERCISE QUESTIONS

Effect of line width

Spectra used in addressing the following questions from exercise 3.2a are shown in figure 3.29.

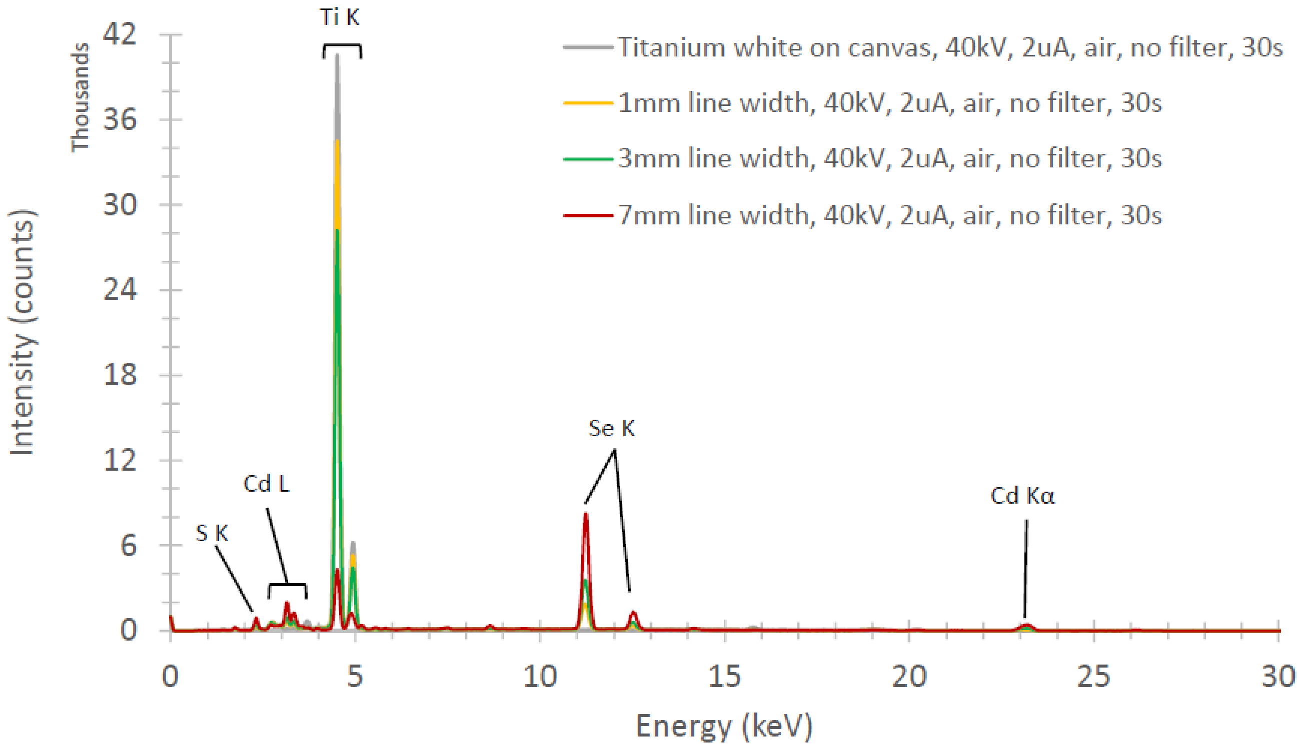
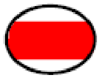
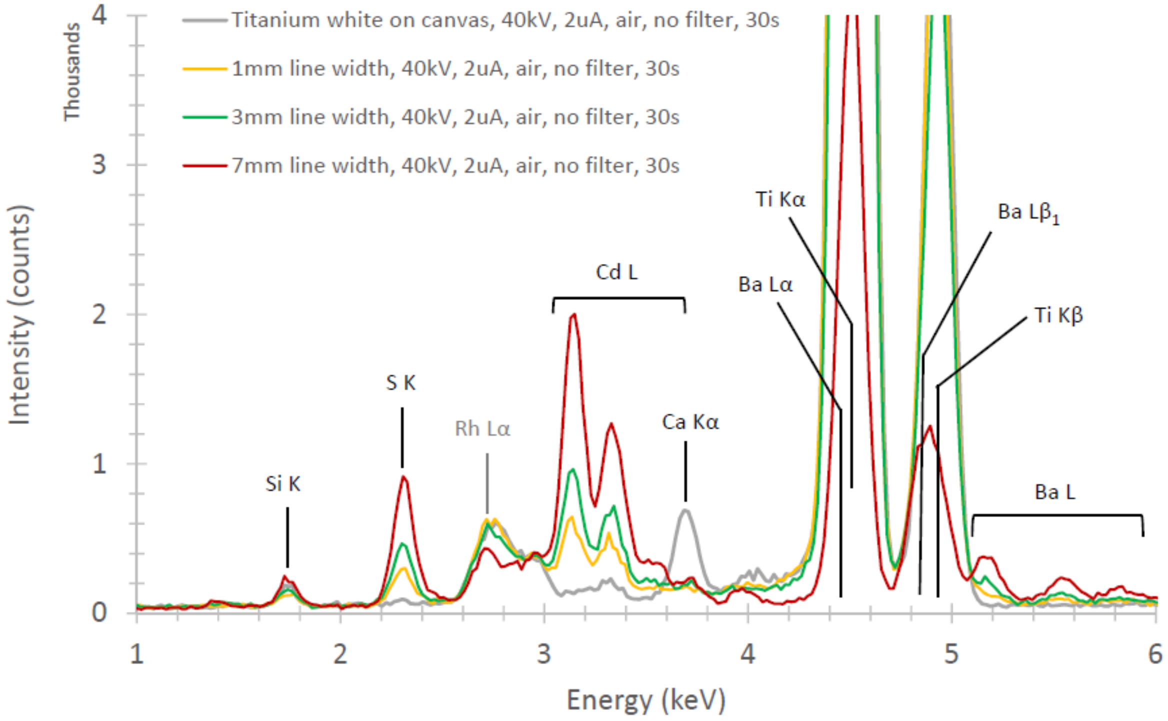


FIGURE 3.29. Spectra (with detail, bottom) for analysis of cadmium red lines on titanium white ground on canvas (effect of line width) in exercise 3.2a. Data collected using a Bruker Tracer III-SD, Rh tube.

Practical exercises for students
Exercise 3.2a: Example for beam size vs. analytical area

Analysis of thin lines with large apertures means that both red and white surfaces are included in the analysis region.





- Thinner red lines do not completely cover the instrument’s aperture (when analysed with a Bruker Tracer III-SD).
- When analysing 1 mm and 3 mm wide lines, the spectrum represents a combination of results for the following layer sequences:
  - cadmium red, titanium white, canvas support
  - titanium white and canvas support.
- The 7 mm wide line completely fills the instrument’s aperture. The spectrum represents analysis of the following layer sequence only: cadmium red, titanium white, canvas support.

Practical exercises for students
Exercise 3.2a: Example for beam size vs. analytical area

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

BRUKER

Does the detected Ti in each of the three line widths come from the underlying paint or from the exposed sides?

- To understand which peaks relate to which layers, compare the spectra
  - of the widest red line with those of titanium white on canvas and
  - the bare canvas.
- Elements unique to titanium white on canvas are Ti, Ca, Zr, Nb. Titanium white contains TiO2.
- Elements unique to cadmium red paint are S, Cd, Ba, Se. These elements relate to the presence of: CdS, CdSe, $BaSO_4$ (likely with traces of $SrSO_4$).
- Elements that may be present in both titanium white paint and cadmium red paint are Si, Zn, Sr.
- As the width of the red lines increases, the proportion of cadmium red relative to titanium white in the analysed area increases.
- There is an increase in the intensity of peaks related to the cadmium red paint: S, Cd, Ba, Zn, Se, Sr.
- There is a decrease in the intensity of peaks related only to the titanium white paint: Ti, Ca.

Let´s go back to the presentation!

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

02 Qualitative Interpretations

A Typical spectrum is a combination of

- Background from tube lines and atmosphere
- Analyte lines
- Sum Peaks and Escape Peaks
- Spectral line overlaps

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

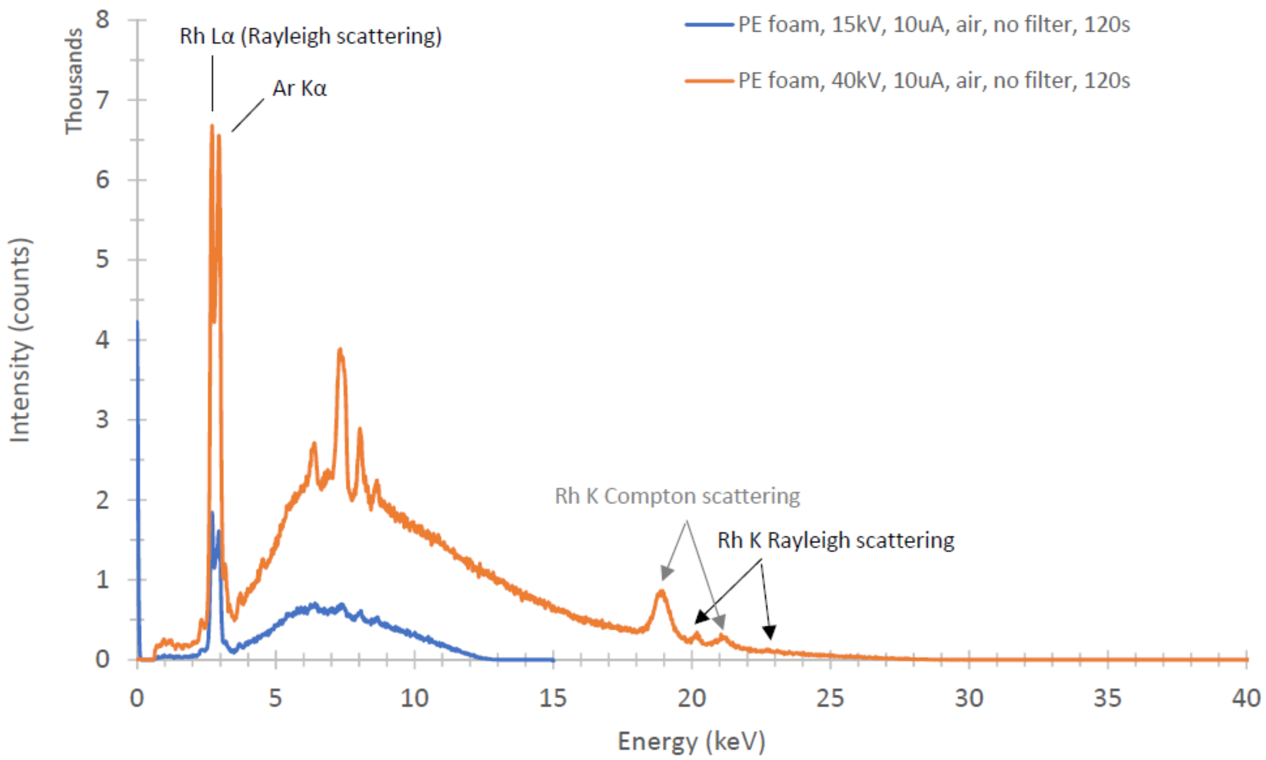
BRUKER

02 Qualitative Interpretations

A Typical spectrum is a combination of

- Background from tube lines and atmosphere
- Analyte lines
- Sum Peaks and Escape Peaks
- Spectral line overlaps

Tube lines and atmosphere



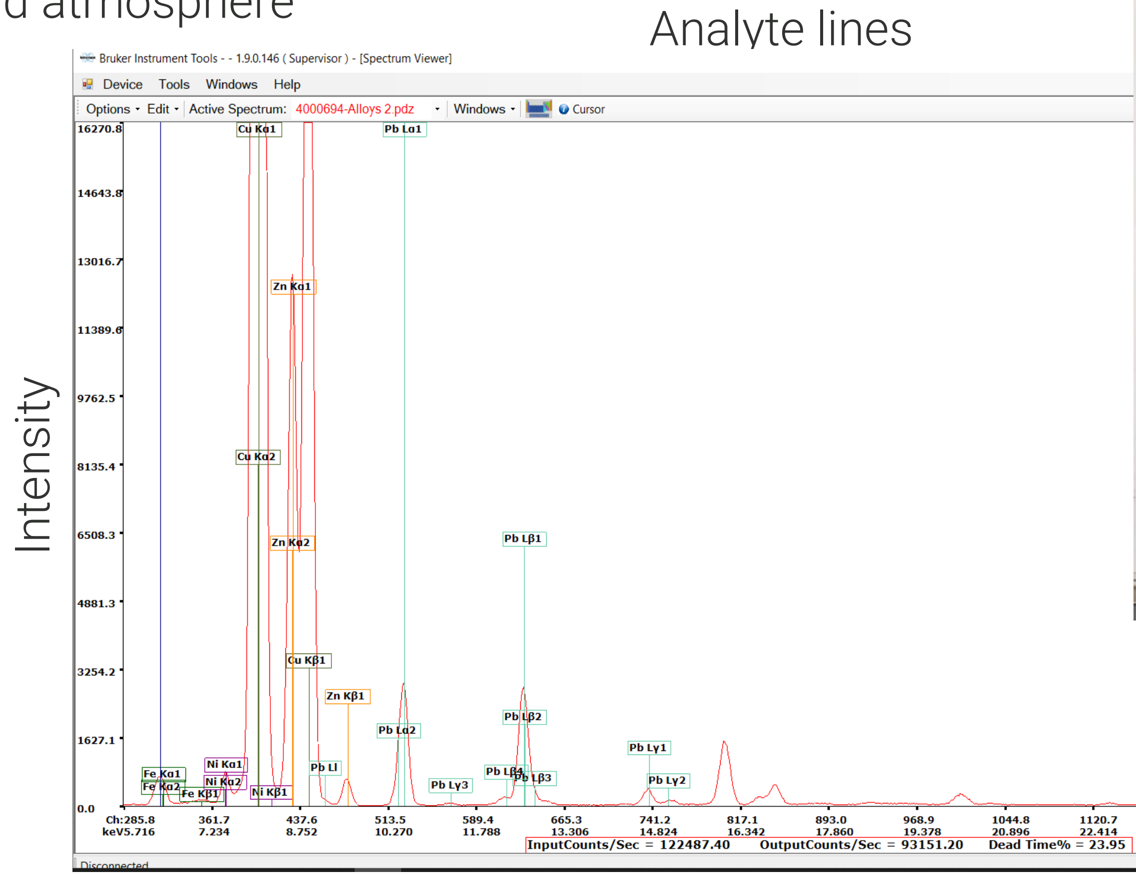
UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

BRUKER

02 Qualitative Interpretations

A Typical spectrum is a combination of

- Background from tube lines and atmosphere
- Analyte lines
- Sum Peaks and Escape Peaks
- Spectral line overlaps

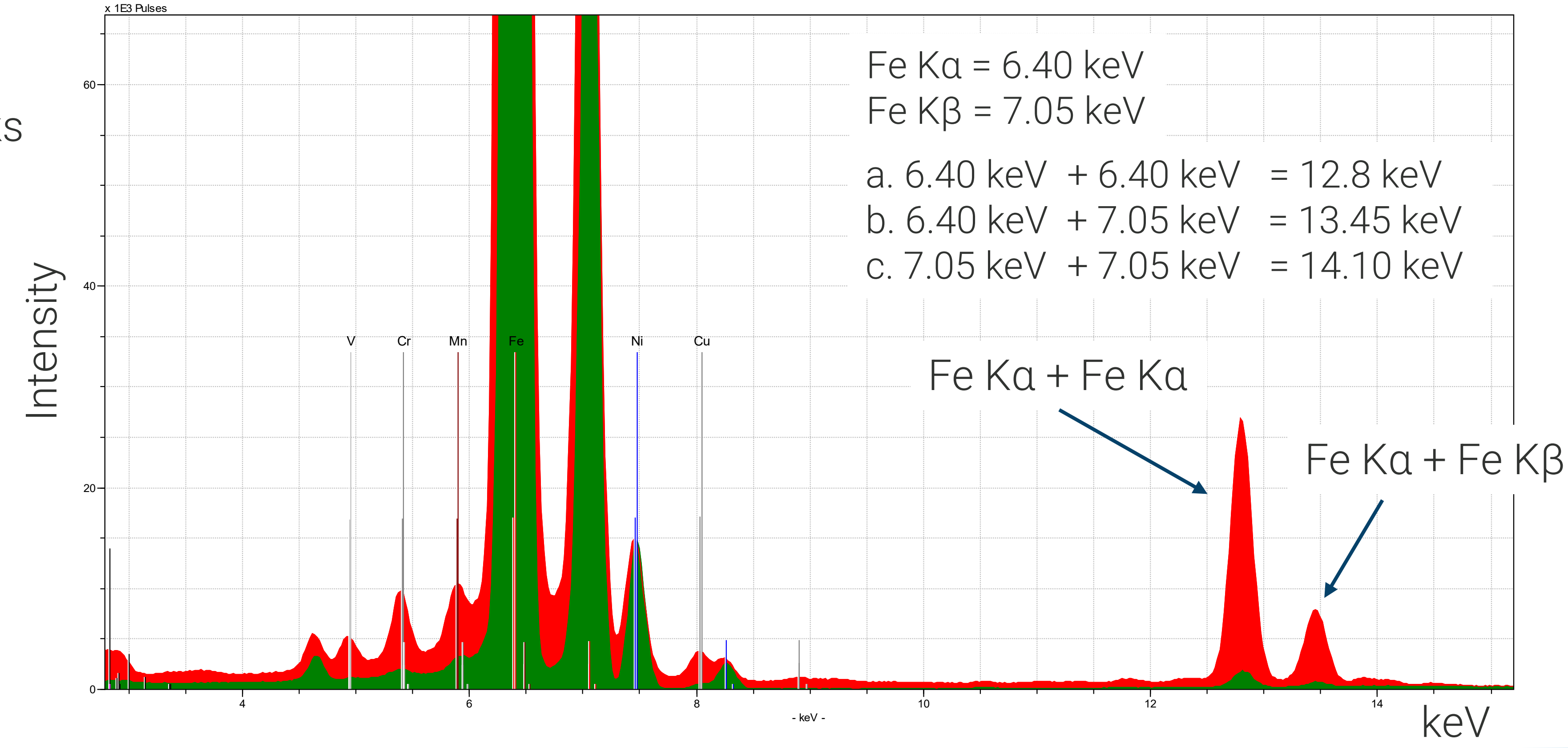


02 Qualitative Interpretations

A Typical spectrum is a combination of

- Background from tube lines and atmosphere
- Analyte lines
- Sum Peaks and Escape Peaks
- Spectral line overlaps

Sum Peaks



UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

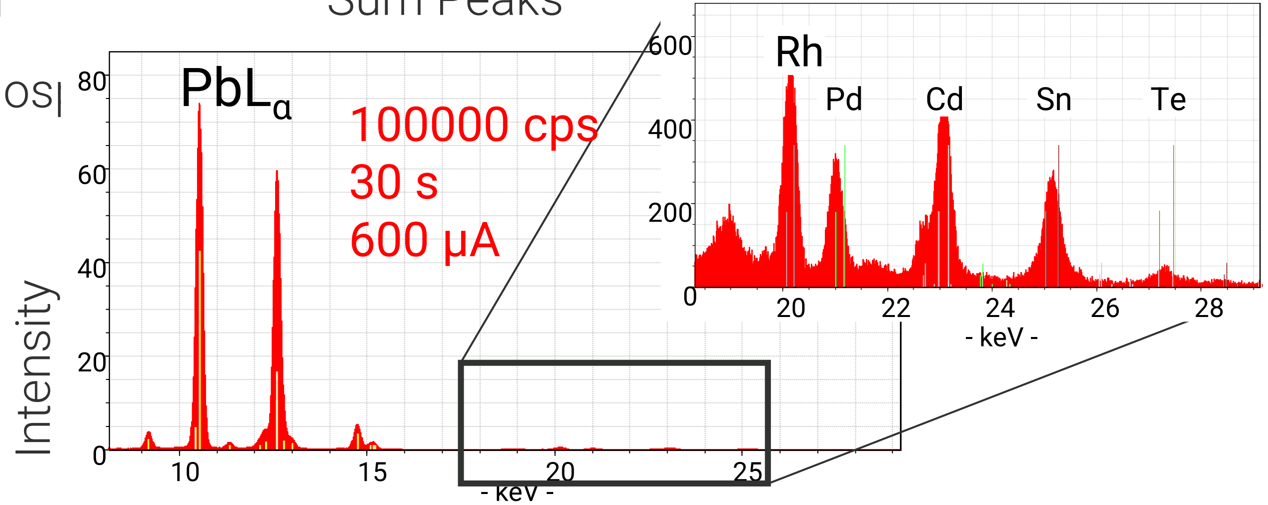
BRUKER

02 Qualitative Interpretations

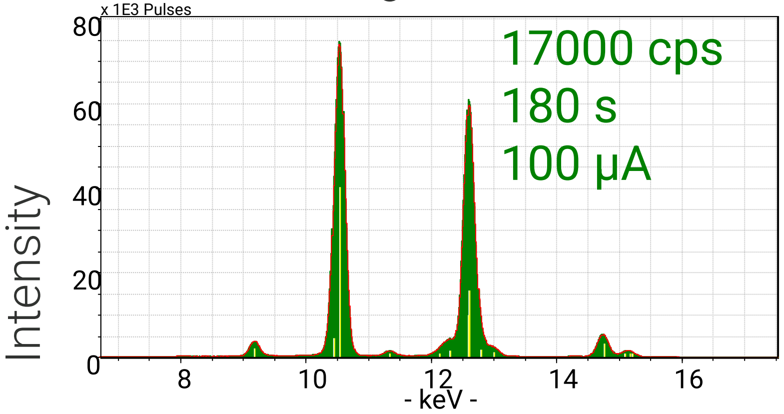
A Typical spectrum is a combination of

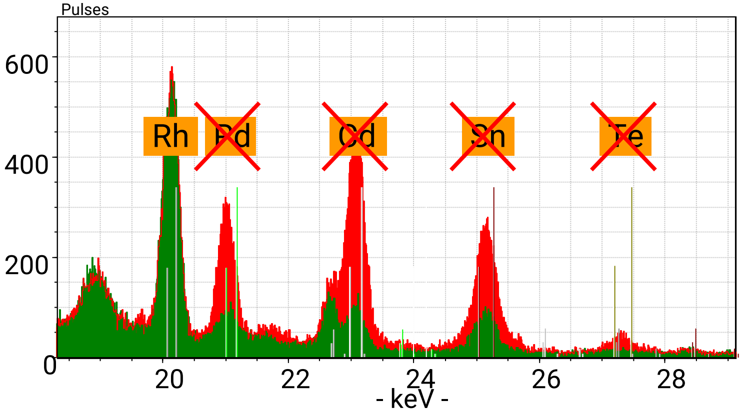
- Background from tube lines and atmos|
- Analyte lines
- Sum Peaks and Escape Peaks
- Spectral line overlaps

Sum Peaks



After reducing the current:





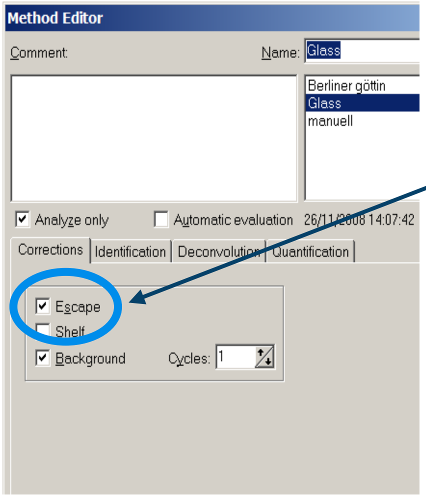
"Sum Peaks" affect detection of trace elements!

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

02 Qualitative Interpretations

A Typical spectrum is a combination of

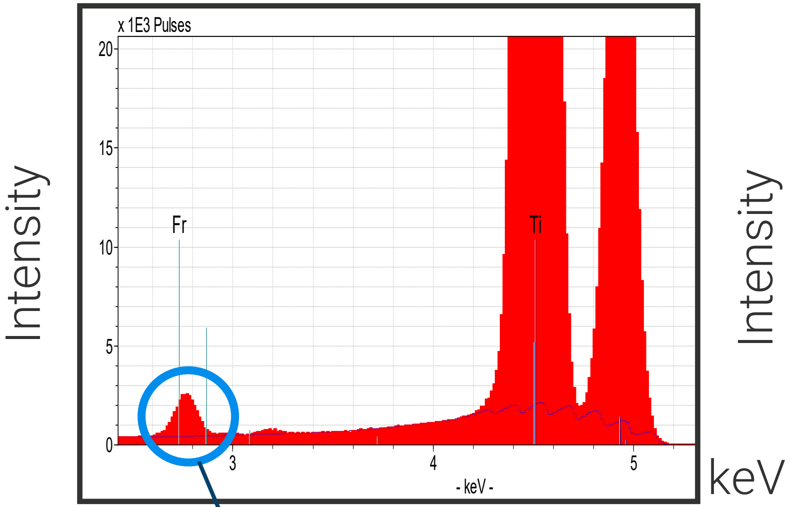
- Background from tube lines and atmosphere
- Analyte lines
- Sum Peaks and Escape Peaks
- Spectral line overlaps



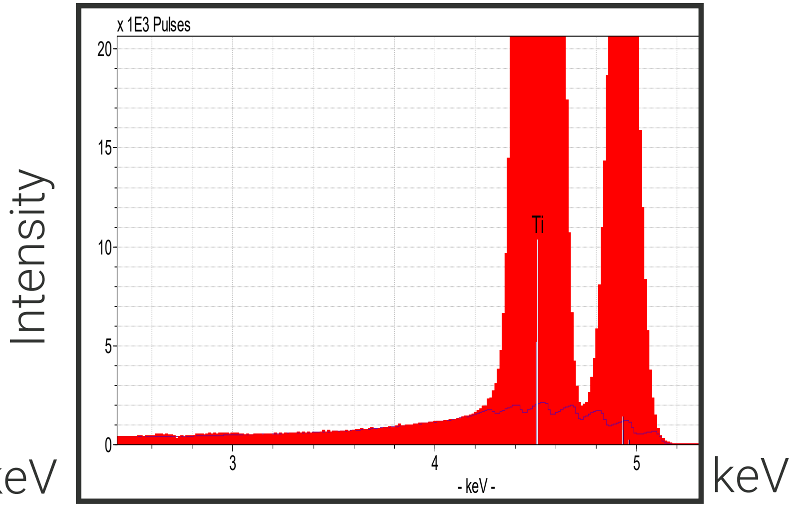
To eliminate the "Escape Peak" is needed to select the function!

Escape Peaks

Measured Spectrum



Corrected spectrum



Ti Ka	4.512 keV
Si Ka	1.7398 keV
Escape peak	2.77 keV

Escape peak = 4.5 keV-1.74 keV

02 Qualitative Interpretations

A Typical spectrum is a combination of

- Background from tube lines and atmosphere
- Analyte lines
- Sum Peaks and Escape Peaks
- Spectral line overlaps

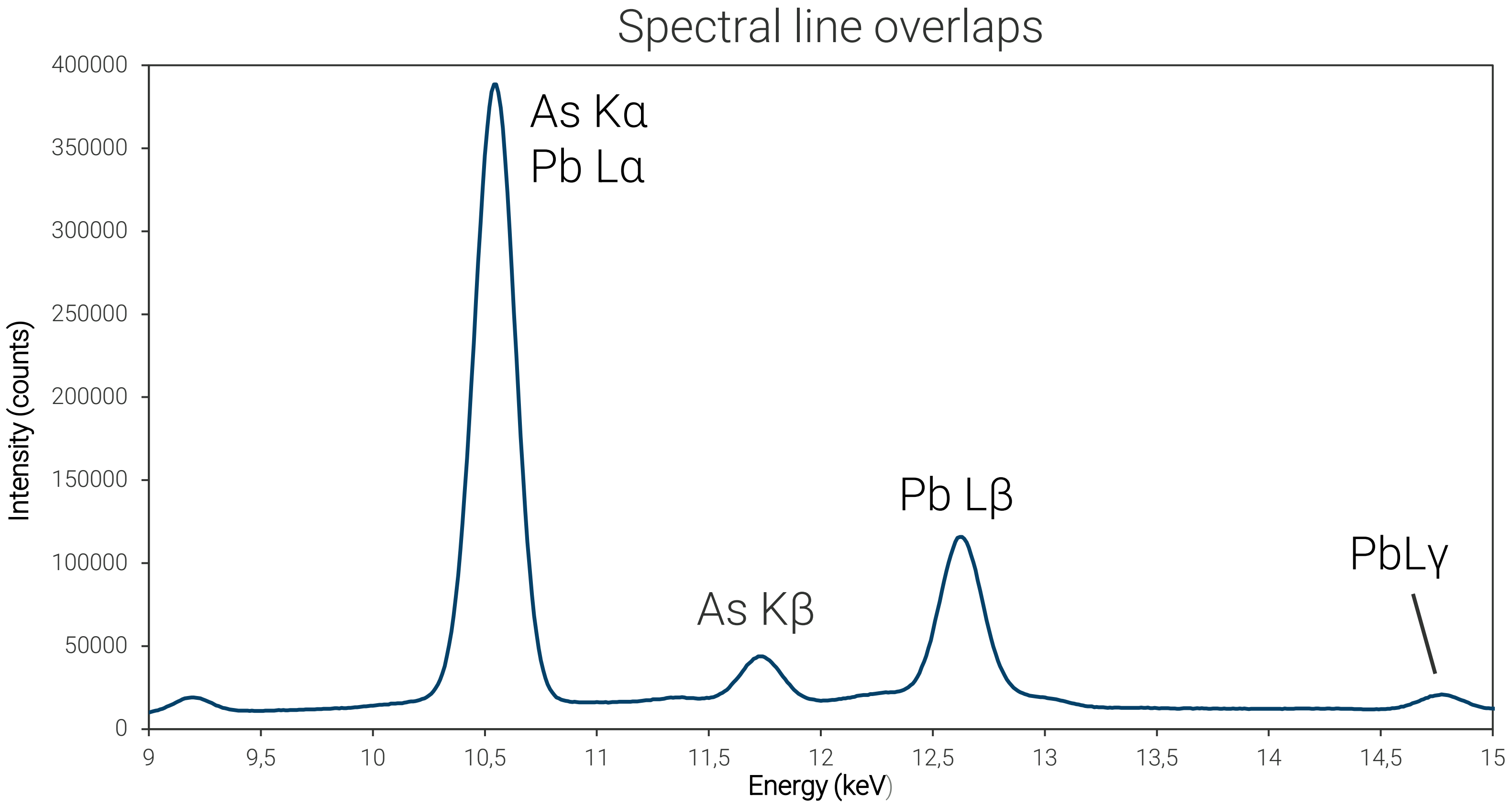
Spectral line overlaps

Base Metal Element	Ka1	Kb1	REE Element	La1	Lb1	Heavy Elements	La1	Lb1
Ti	4.51	4.93	La	4.65	5.04	Ba	4.47	4.83
V	4.95	5.43	Ce	4.84	5.26	Hf	7.9	9.02
Cr	5.41	5.95	Pr	5.03	5.49	Ta	8.15	9.34
Mn	5.9	6.49	Nd	5.23	5.72	W	8.4	9.67
Fe	6.4	7.06	Sm	5.64	6.21			
Co	6.93	7.65	Eu	5.85	6.46			
Ni	7.48	8.26	Gd	6.06	6.71			
Cu	8.05	8.9	Tb	6.28	6.98			
Zn	8.64	9.57	Dy	6.5	7.25			
			Ho	6.72	7.53			
			Er	6.95	7.81			
			Tm	7.18	8.1			
			Yb	7.41	8.4			
			Lu	7.65	8.71			

02 Qualitative Interpretations

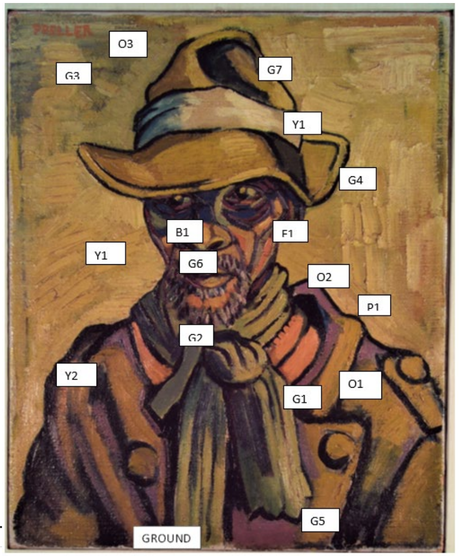
A Typical spectrum is a combination of

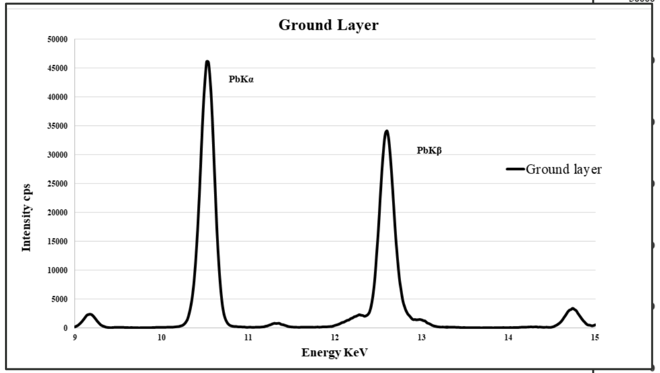
- Background from tube lines and atmosphere
- Analyte lines
- Sum Peaks and Escape Peaks
- Spectral line overlaps

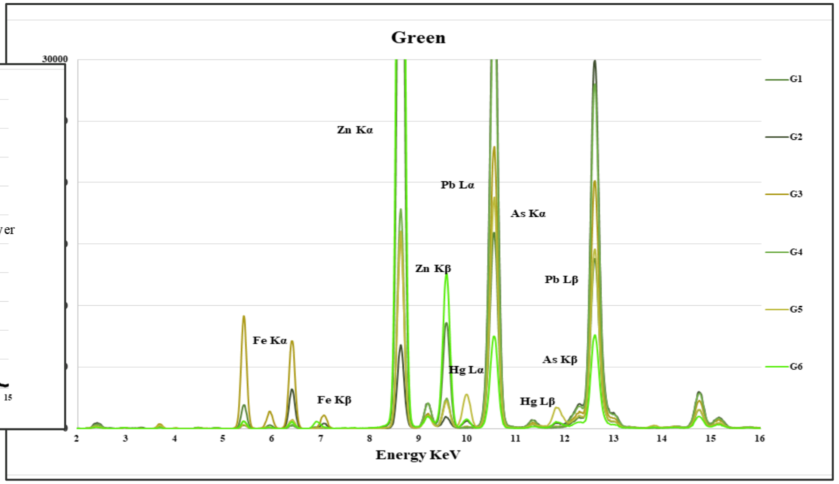


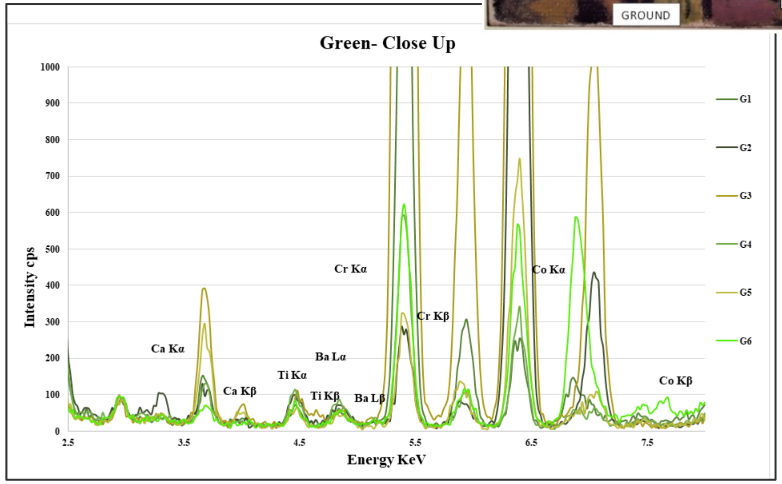
02 Qualitative Interpretations
Summary

- The data from an artwork can often be very overwhelming, and it is important to plan the analyses, but also plan how to represent the data.
- In this example, colour fields were collected.
- First, the entire spectra were evaluated, however, for a more detailed evaluation it is recommended to focus on specific areas.
- It is crucial to consider sum peaks, escape peaks, and spectral overlap when interpreting spectra.

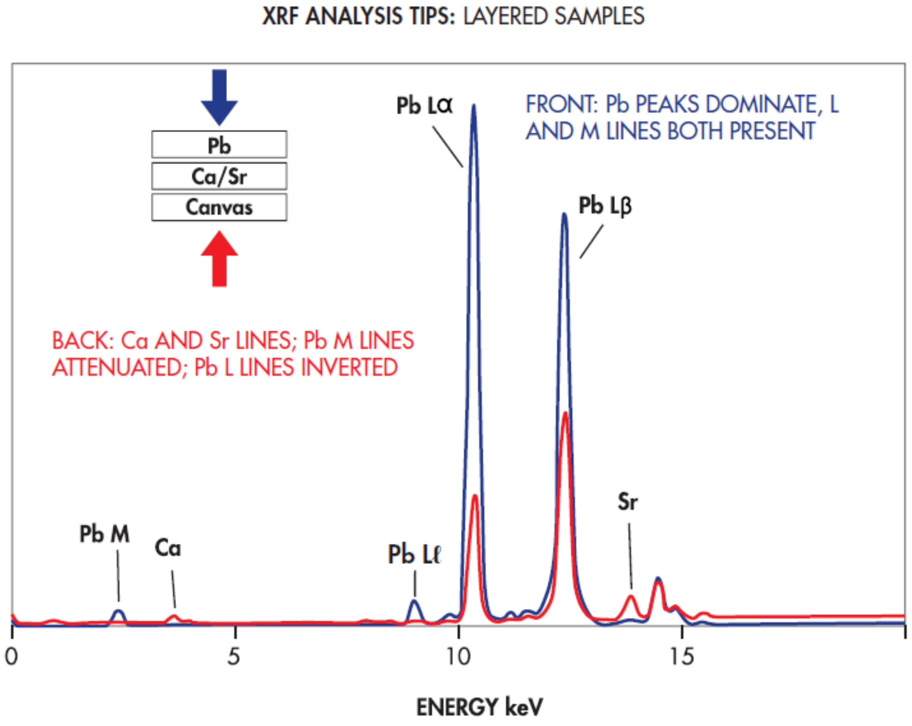


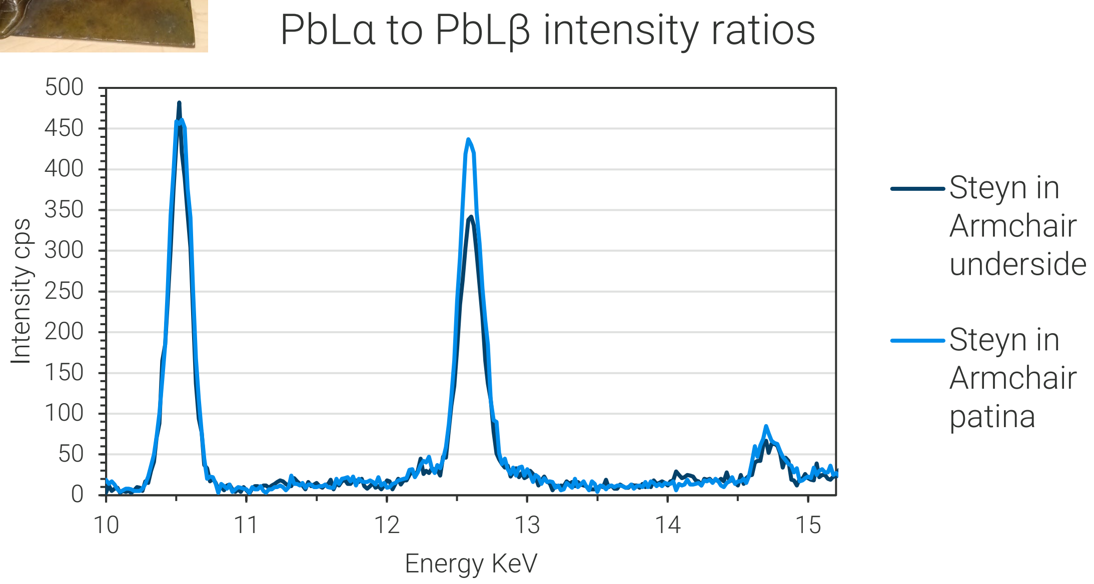






03 Ambiguity in results
Mixtures: Layers and mixed pigments

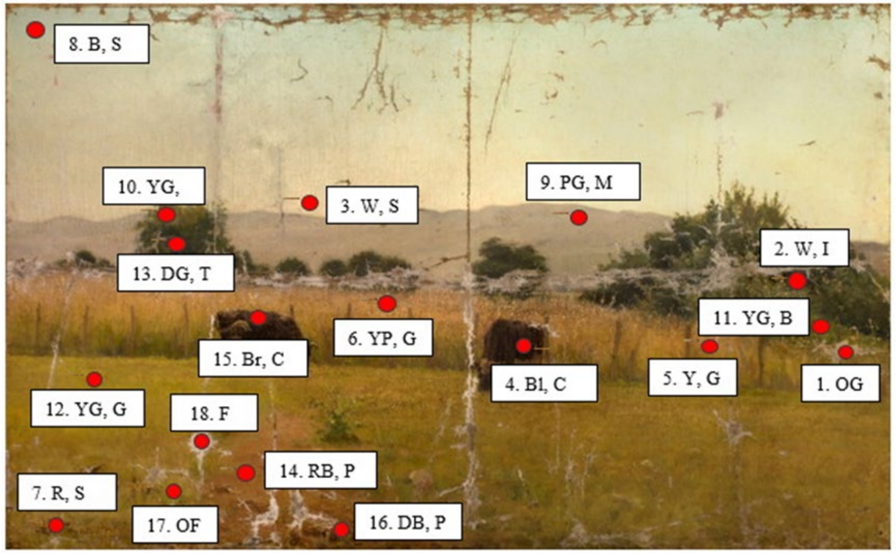




03 Ambiguity in results
Same elements, different pigments

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

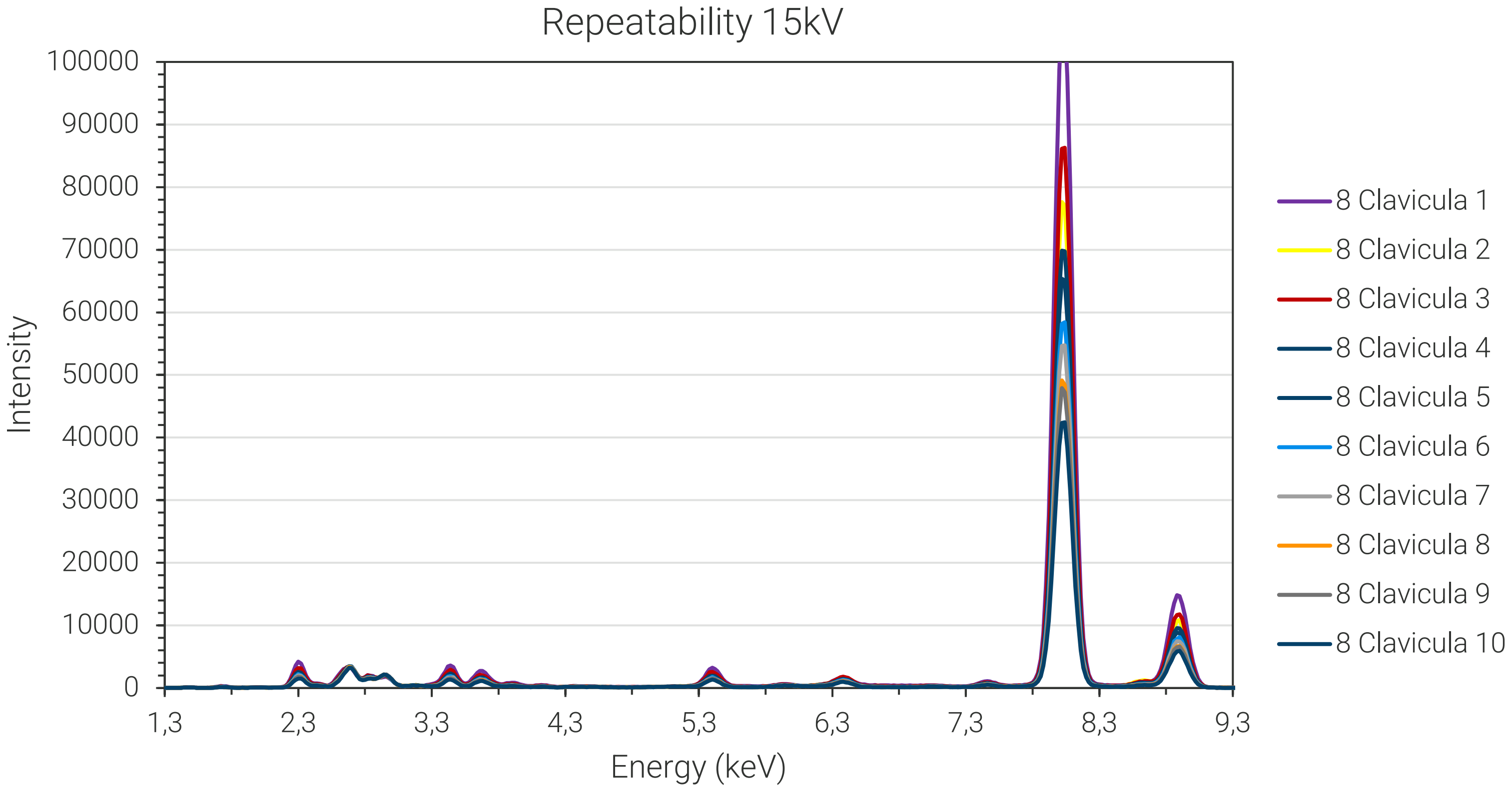
BRUKER



No	Code	Description	Elements detected	Possible pigments
4	Bl, C	Black, cow	Pb, Si, Fe, S, Ca, P, Ba, Hg, *Cr, Zn*	Bone black, ivory black, mars black, vermillion, cinnabar, lead white, chalk, gypsum
5	Y, G	Yellow, grass	Pb, S, Si, *Ba, Hg, Cr, Fe, Ca, P, Zn*	Yellow lead, lemon yellow, yellow ochre, raw sienna, barium chromate yellow, chrome yellow, zinc yellow, mars yellow, lead white, chalk, gypsum
6	YP, G	Yellow-pink, grass	Pb, Si, S, Hg, *Fe, Ca, Ba, Zn, Cd*	Lead white, chalk, gypsum, lemon yellow, yellow ochre,
7	R, S	Red, signature	Pb, Si, Fe, S, Ca, *Ba, Hg, Cr, Zn*	Vermillion, mars red, red lead, cinnabar, lead white, chalk, gypsum
8	B, S	Blue, sky	Pb, S, Si, P, Ca, Fe, *Ba, Zn*	Prussian blue, vivianite, indigo, lead white, chalk, gypsum

04 Repeatability

- Nose protection
- Positioning: Consistent vs non-consistent distance between nose and sample

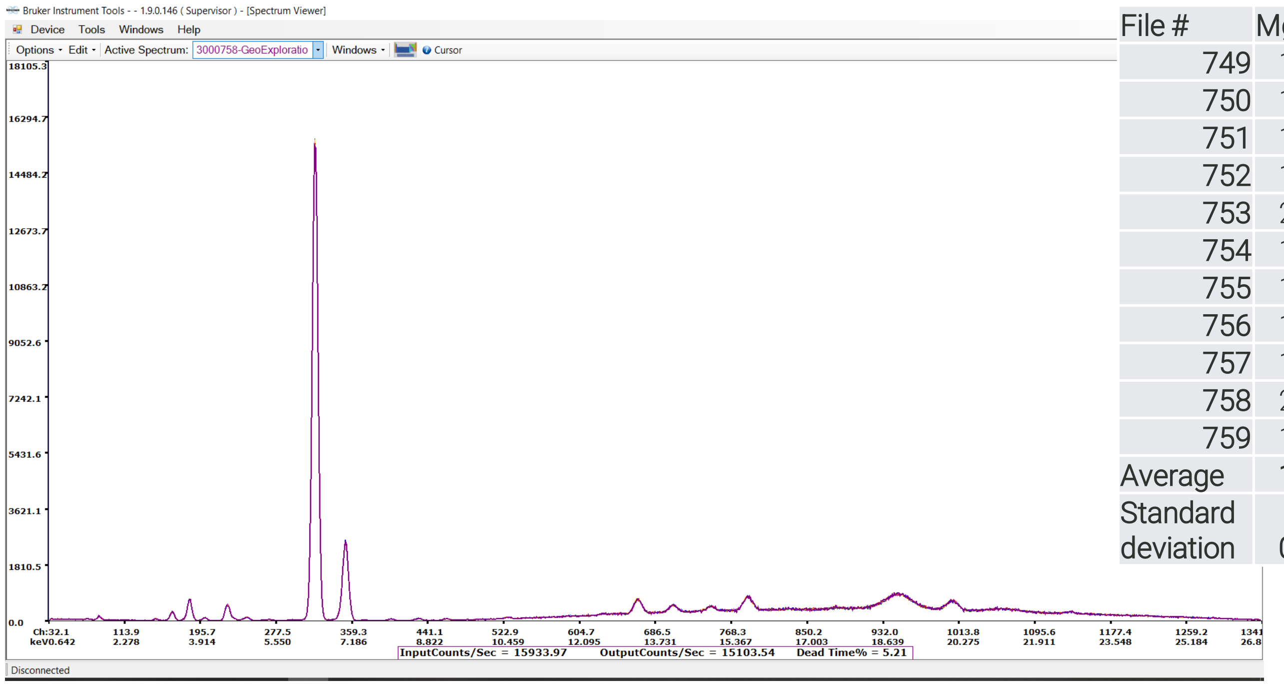


05 Game Plan – How to approach a project to get the maximum out of the data?

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

BRUKER

- Determine that your instrument is running correctly – using a check sample.
- Then check your instrument repeatability: With the tripod, the spectrometer was carefully set up to analyse one spot 10 times.
- The standard deviation is the average deviation from the average value of the ten measurements.

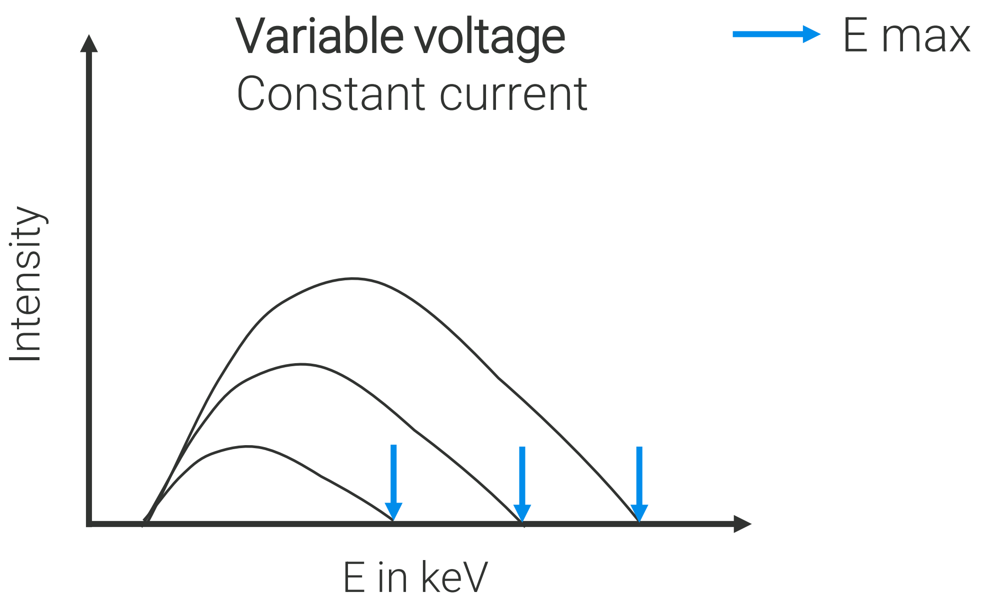


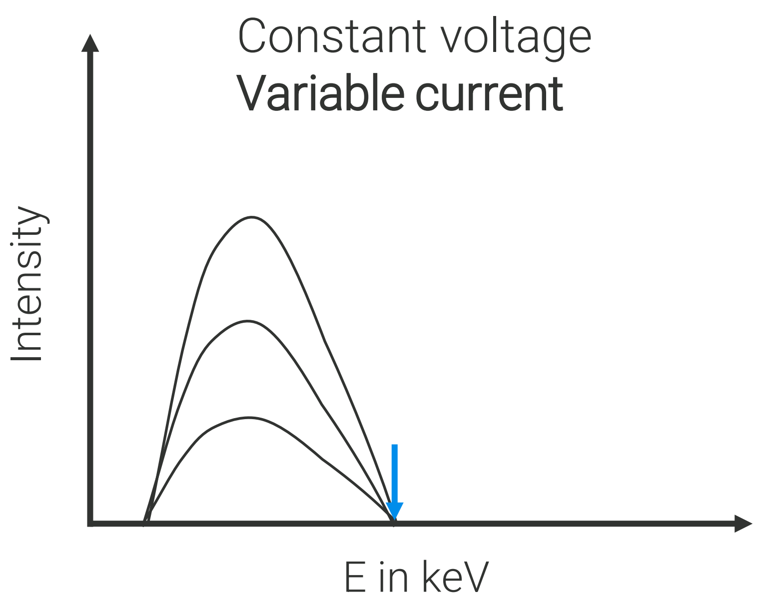
File #	MgO %	Al_2O_3 %	SiO_2 %	P %	S %	K_2O %	Ca %	Ti %	Cr %	Mn %	Fe %
749	1.79	18.22	45.84	0.06	0.70	1.23	1.77	0.48	0.0072	0.01	2.67
750	1.65	18.39	45.75	0.06	0.71	1.25	1.77	0.48	0.0084	0.01	2.67
751	1.54	18.17	45.90	0.07	0.70	1.24	1.78	0.48	0.0079	0.01	2.70
752	1.50	18.39	46.30	0.07	0.72	1.24	1.78	0.47	0.0069	0.01	2.69
753	2.53	18.57	46.13	0.07	0.71	1.24	1.77	0.48	0.0083	0.01	2.69
754	1.93	18.06	46.04	0.06	0.69	1.25	1.77	0.48	0.0077	0.01	2.70
755	1.73	18.48	46.28	0.06	0.70	1.25	1.79	0.48	0.0120	0.01	2.68
756	1.30	18.56	46.56	0.07	0.72	1.26	1.78	0.47	0.0088	0.01	2.68
757	1.42	18.41	46.38	0.07	0.70	1.25	1.80	0.47	0.0074	0.01	2.69
758	2.02	18.51	46.57	0.07	0.69	1.25	1.79	0.48	0.0099	0.01	2.68
759	1.60	18.33	46.44	0.06	0.73	1.26	1.79	0.48	0.0074	0.01	2.71
Average	1.73	18.37	46.20	0.07	0.71	1.25	1.78	0.48	0.01	0.01	2.69
Standard deviation	0.34	0.16	0.29	0.00	0.01	0.01	0.01	0.00	0.00	0.00	0.01

05 Game Plan – How to approach a project to get the maximum out of the data?

- Optimise settings to cover the entire range:
  - Voltage
  - Current
  - Filter
  - Time

Optimal voltage: 2-3 times the energy of the heaviest element.

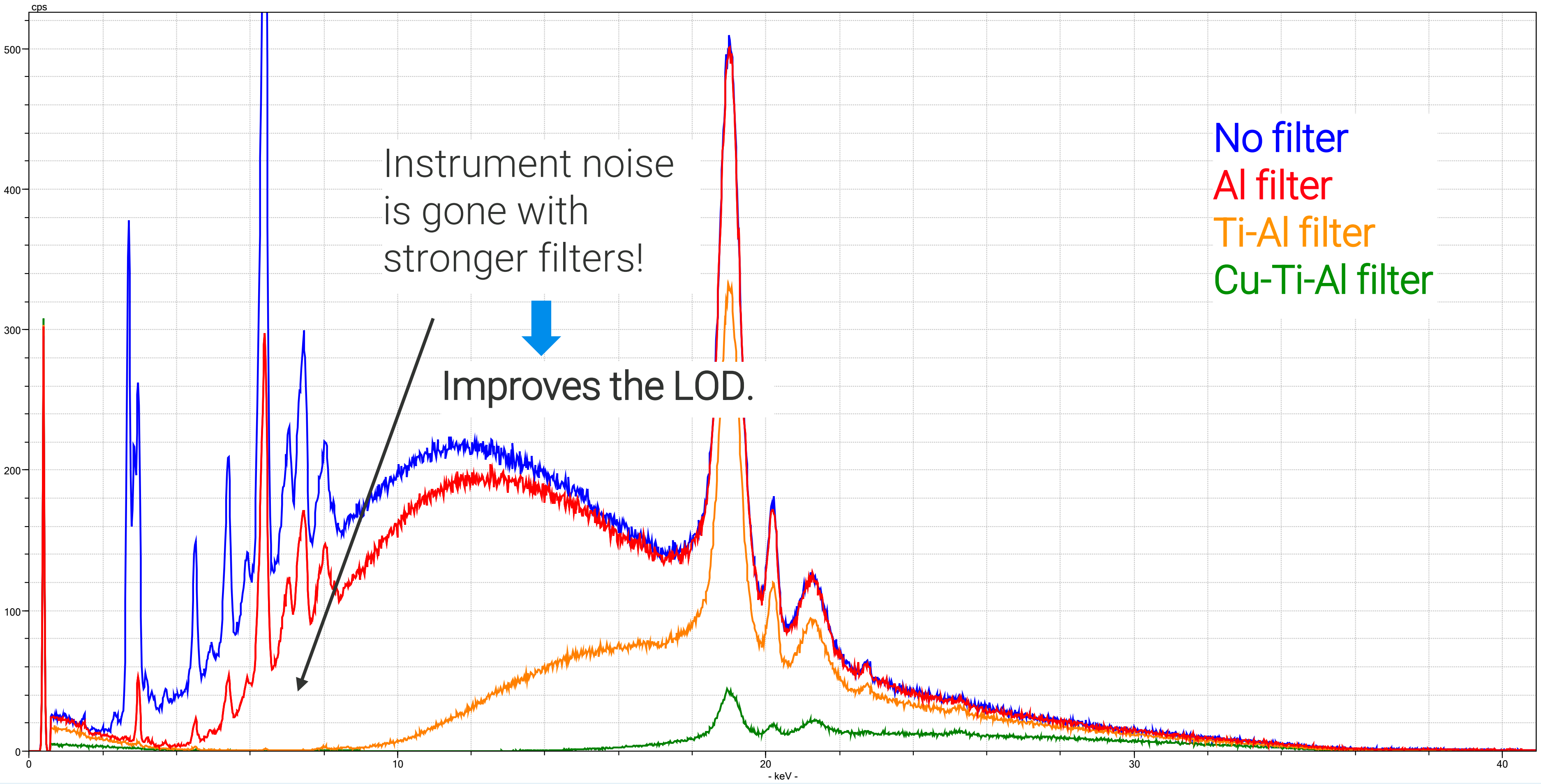




05 Game Plan – How to approach a project to get the maximum out of the data?

- Optimise settings to cover the entire range:
  - Voltage
  - Current
  - Filter
  - Time

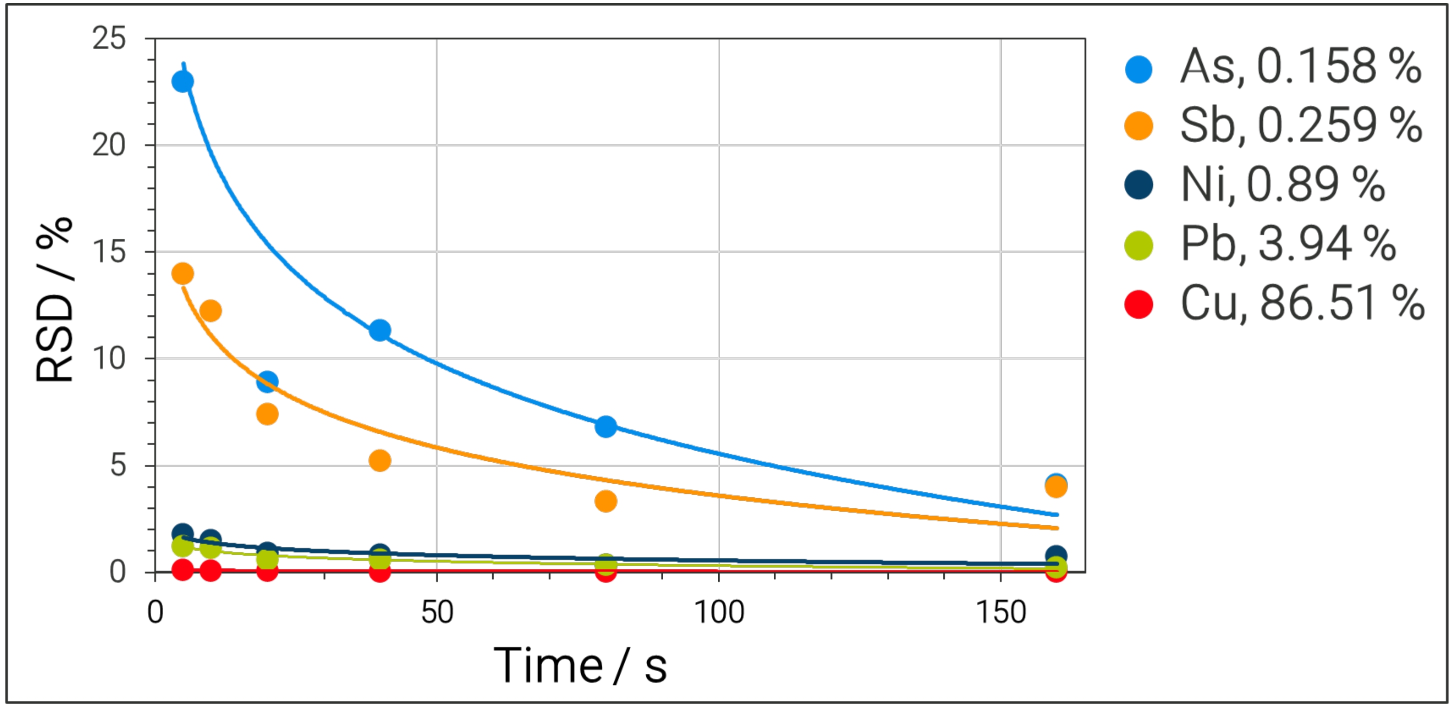
Fixed voltage 40 kV
Fixed Current: 15 µA
Fixed time: 30 s



05 Game Plan – How to approach a project to get the maximum out of the data?

- Optimise settings to cover the entire range:
  - Voltage
  - Current
  - Filter
  - Time

Measure longer for low concentrations: An increase in measurement time by a factor of 4, reduces the standard deviation by a factor of 2!



05 Game Plan – How to approach a project to get the maximum out of the data?

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

- If you are unsure how to start, choose one of the pre-set illuminations from the drop-down menu in the Spectrometer Mode.
- Run it and look at the spectrum to ensure that you excited all the elements that you are interested in.
- Look at the spectra while data collection and allow enough time to return to the object and re-collect the spectra under different conditions.

Add On One heartfelt wish:
Hazardous substances from preservation chemicals

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

BRUKER

ID	Species	S	Cr	As	Hg	Pb
51630	Trachyphonus margaritatus	9.13	0.0000	3.071	0.032	0.000
34530	Accipiter nisus linn	13.17	0.0000	0.428	0.048	0.008
34532	Accipiter nisus linn	10.06	0.0000	6.375	0.058	0.006
34531	Accipiter nisus linn	11.07	0.0000	5.434	0.074	0.014
34534	Scelospizias brevipes (sen)	7.34	0.0000	2.530	0.088	0.008
86148	Accipiter nisus	8.30	0.0000	4.811	0.044	0.000

Wear PPE and protect yourself!

Summary
Part II Paintings, Pigments, and surfaces with challenges

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

BRUKER

- Particularly caution must be taken when we deal with
  - non-infinite thick samples,
  - heterogeneity and layering,
  - uneven surfaces,
  - samples that do not fully cover the measurement spot.
- For qualitative interpretations of a spectrum
  - the background from tube lines and atmosphere,
  - the analyte lines,
  - Sum Peaks and Escape Peaks,
  - spectral line overlaps need to be considered.
- Ambiguity in data interpretation: Some pigments contain the same elements, but in different proportions.
- We presented a game plan for approaching a project to get the maximum out of the data.

Art & Conservation Webinar Series
Handheld XRF in Cultural Heritage Studies

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

BRUKER

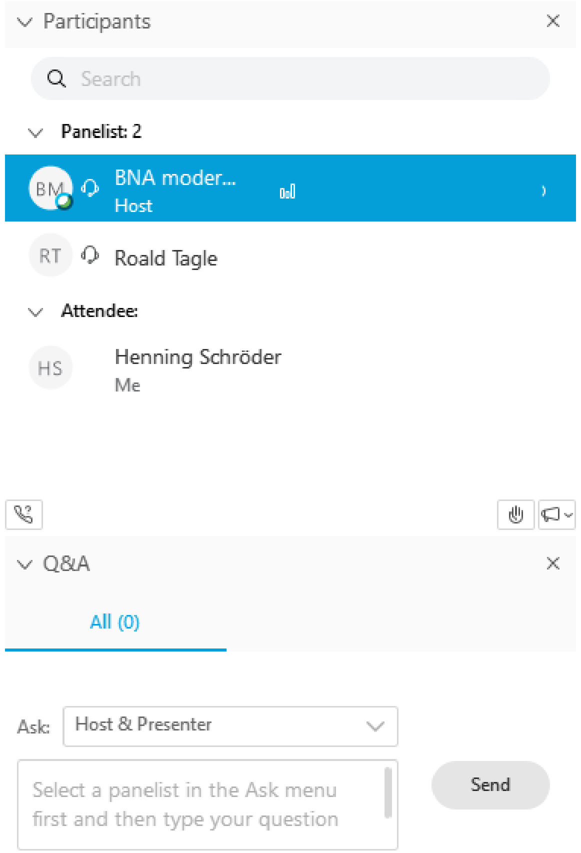
If you have questions during this webinar,

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.



Thank you!

Maggi Loubser
Kathrin Schneider

info@bna.bruker.com
www.bruker.com/bna

BRUKER

Innovation with Integrity