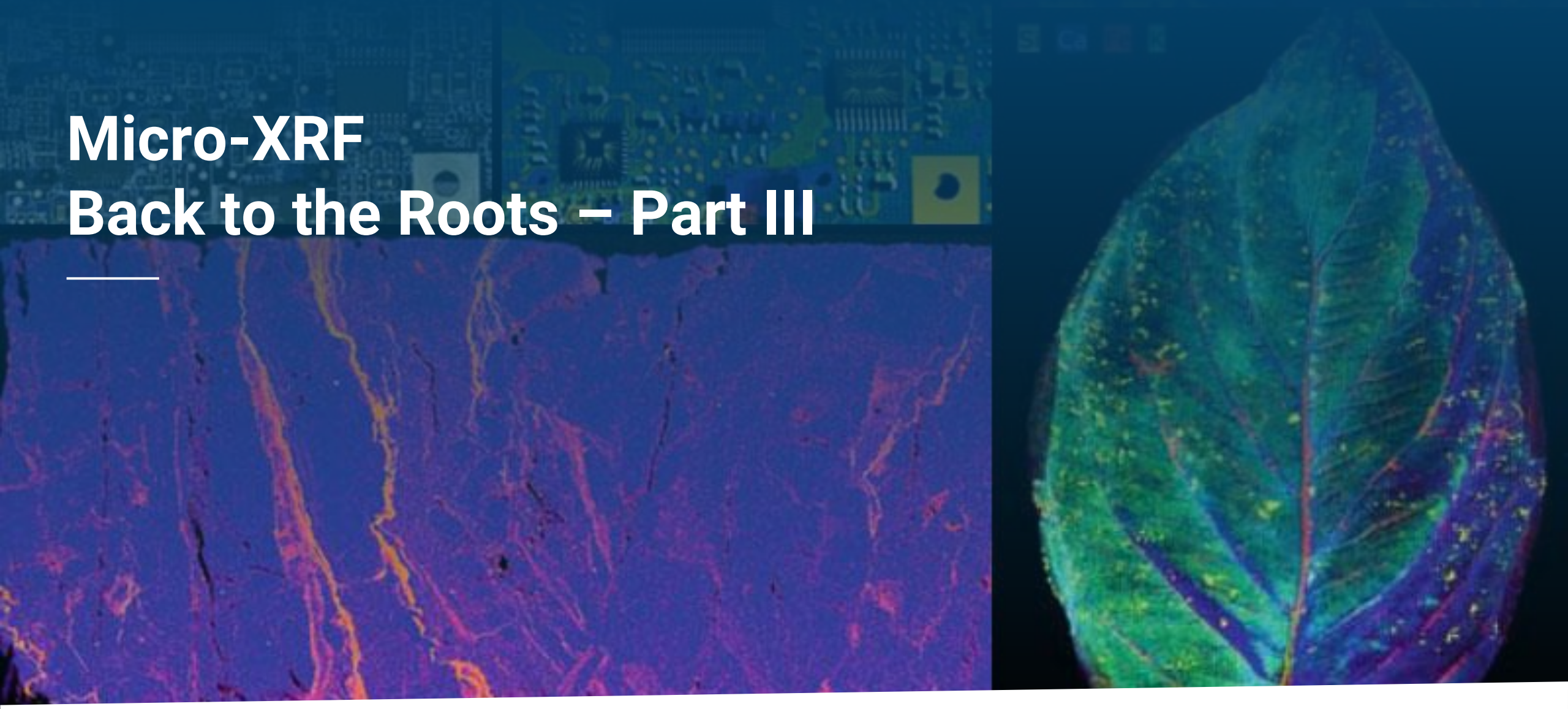
Micro-XRF
Back to the Roots – Part III



The speakers

- Falk Reinhardt
- Senior Application Scientist,
  Bruker Nano Analytics, Berlin, Germany

- Dr. Roald Tagle
- Head of XMP Application,
  Bruker Nano Analytics, Berlin, Germany

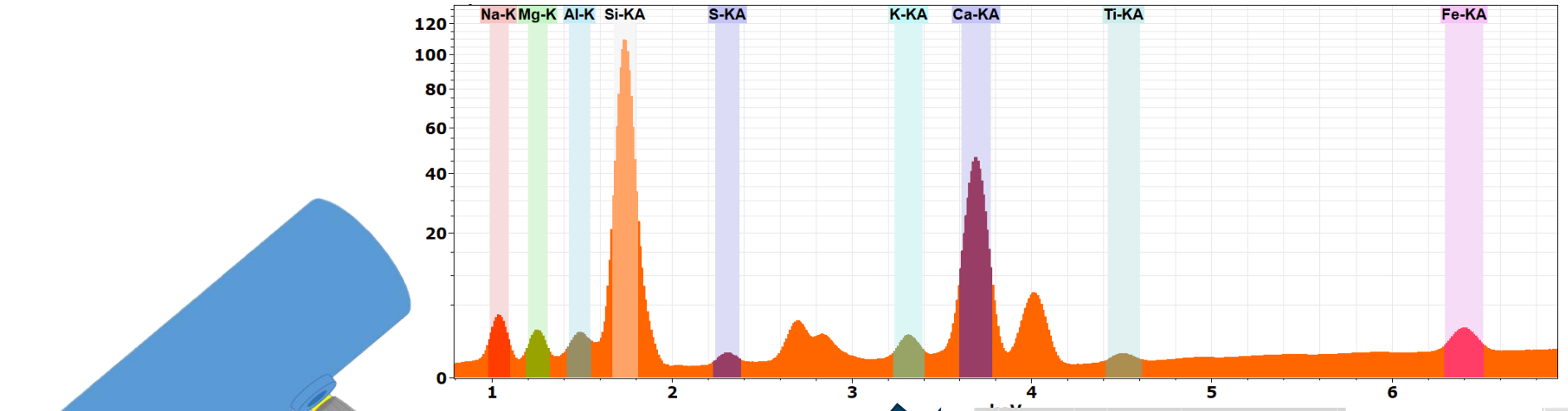
Overview

- Part I of this series aimed to introduce micro-XRF as a technique and give an overview of the individual components of a micro-XRF instrument and why they are they way they are, today.

- Part II focused on qualitative micro-XRF analysis.

- Part III will discuss quantitative XRF analysis.
  - What is quantification?
  - What are prerequisites for quantitative analysis?
  - Which samples can be quantified and how?

What is XRF quantification? The very basics



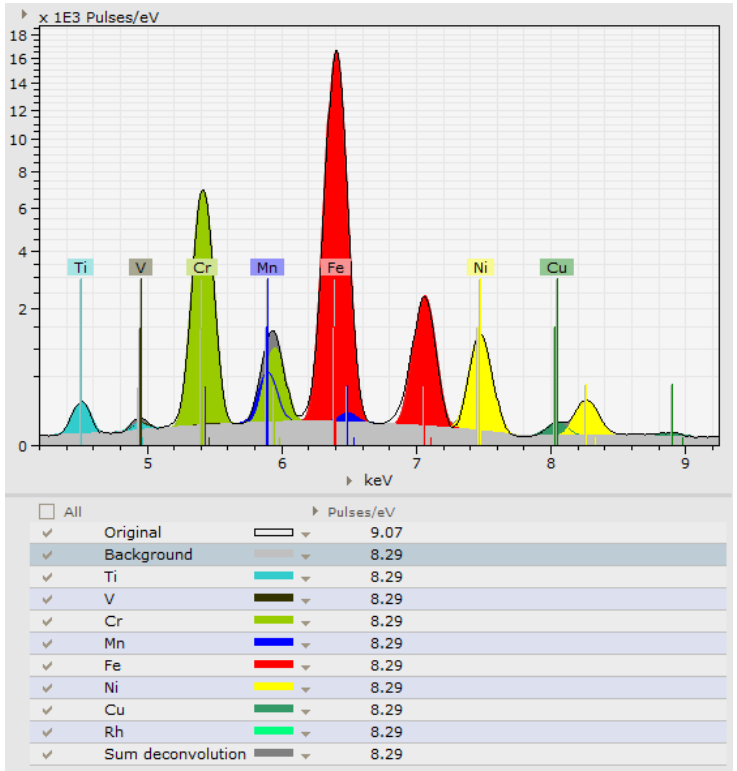
	Element	AN	Series	Net intensity [cps]
	O	8	K series	0.00
	Na	11	K series	340.84
	Mg	12	K series	172.30
	Al	13	K series	153.60
	Si	14	K series	12481.62
	S	16	K series	49.57
	K	19	K series	184.71
	Ca	20	K series	7147.86
	Ti	22	K series	56.83
	Mn	25	K series	10.70
	Fe	26	K series	327.96
	As	33	K series	6.21
	Rb	37	K series	17.30
	Sr	38	K series	32.57
	Zr	40	K series	23.02

	Element	AN	Series	Compound	norm. stoich. C [wt.%]
	O	8	K series		0.000
	Na	11	K series	Na2O	14.282
	Mg	12	K series	MgO	3.870
	Al	13	K series	Al2O3	1.521
	Si	14	K series	SiO2	71.338
	S	16	K series	SO3	0.175
	K	19	K series	K2O	0.351
	Ca	20	K series	CaO	8.331
	Ti	22	K series	TiO2	0.045
	Mn	25	K series	MnO	0.003
	Fe	26	K series	Fe2O3	0.076
	As	33	K series	As2O3	0.001
	Rb	37	K series	Rb2O	0.002
	Sr	38	K series	SrO	0.003
	Zr	40	K series	ZrO2	0.003

What is XRF quantification? Obtaining net peak intensities

- The spectrum is composed of fluorescence lines, some background, and different artifacts.
- The aim is to determine the net peak intensities of the fluorescence lines.
- There are two fundamentally different approaches:

1. “deconvolve” the spectrum, i.e. fit peaks into the spectrum.
   - Line overlap needs some thinking-about.
2. Assume a concentration and forward-calculate what the spectrum of this sample would look like.
   - The net peak intensities are the result of the quantification.



What are prerequisites for quantitative analysis? What is intensity?

- DIN ISO 22309 “Microbeam analysis - Quantitative analysis using energy-dispersive spectrometry (EDS) for elements with an atomic number of 11 (Na) or above” states:

> **3.21**
> **peak intensity**
> total number of X-rays (counts) under the profile of a characteristic X-ray peak after background subtraction
>
> NOTE This is sometimes referred to as the peak integral.

- Usually in physics, intensity is a time-normalized measure, i.e. a candle’s flame does not become more intense just because you look at it longer.

- We will be using counts per second as a measure for intensity, not counts.

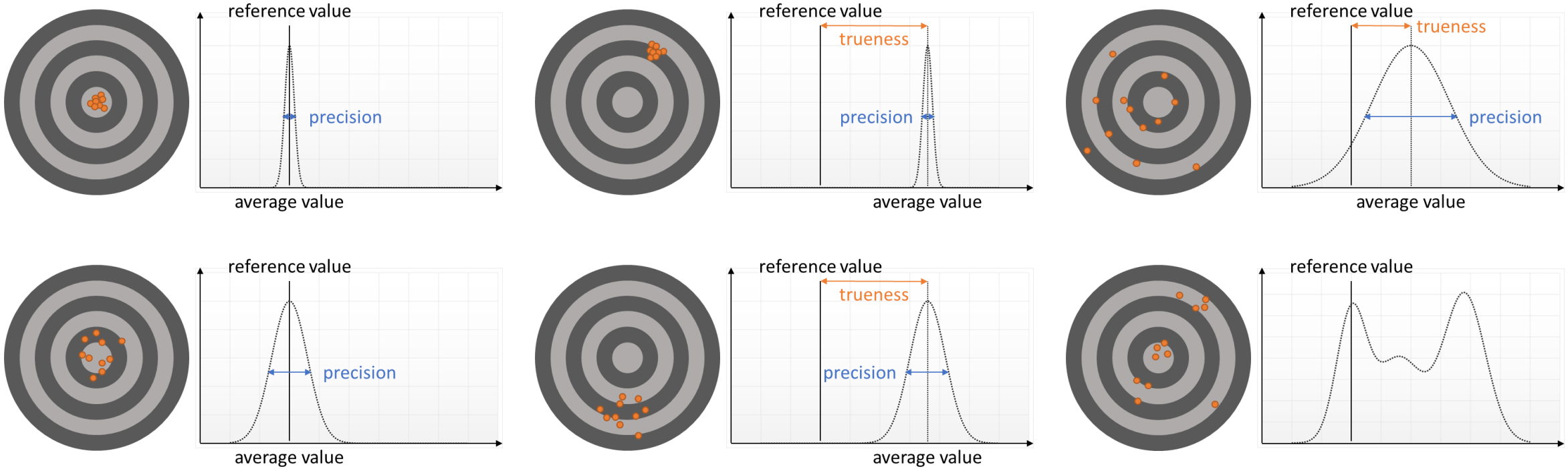
What are prerequisites for quantitative analysis?

- In order to obtain "good results" the expectations must be clear :
  - What is the analytical question? Can (and must) it be answered quantitatively?
  - What is the instrument’s capability to solve the task?
  - What measurement conditions are ideal?
  - What sort of sample can be quantified with the methods employed?

    Homogeneity, surface structure, dark matrix, crystallinity, concentration ranges, element combinations, orientation

What are prerequisites for quantitative analysis? Accuracy, Trueness and Precision

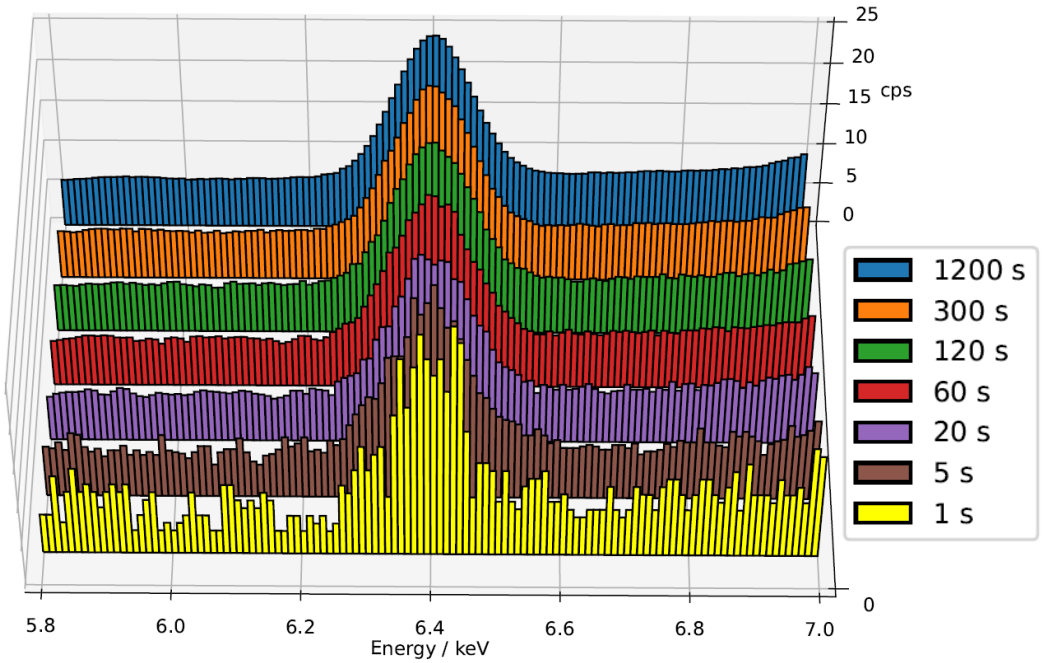
- Since 1994 ISO 5725-1 defines accuracy as a convolution of trueness and precision.



- This definition only works under the assumption that the reference value is the true value.

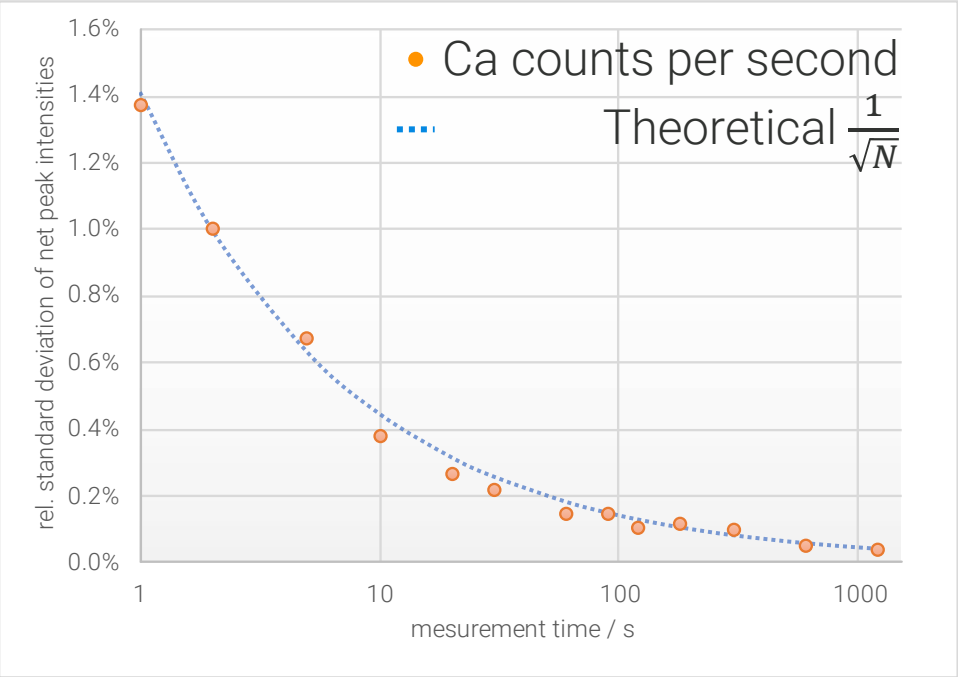
What are prerequisites for quantitative analysis? Uncertainty of the net peak intensity

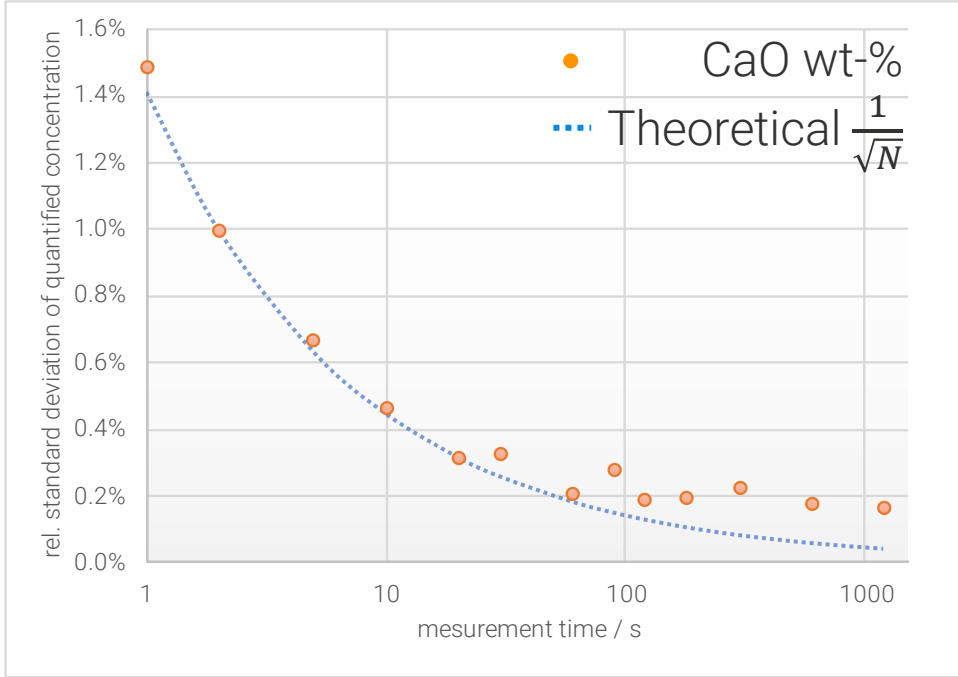
- XRF is a statistical measurement method, i.e. whether a photon is detected at a specific time is pure chance.
- With long enough time, several photons are detected and a peak becomes visible in the spectrum.
- This peak contains N counts and the statistical nature of this measurement dictates an uncertainty of $\sqrt{N}$.
- The relative error then is $\frac{\sqrt{N}}{N} = \frac{1}{\sqrt{N}}$
- The more counts, the smaller the relative uncertainty of the measurement → since the count rate is fixed, it needs time!
- This uncertainty is NOT reflected in the width of the fluorescence peak, it's how well we can determine it's area!



What are prerequisites for quantitative analysis? Net peak intensity and quantification

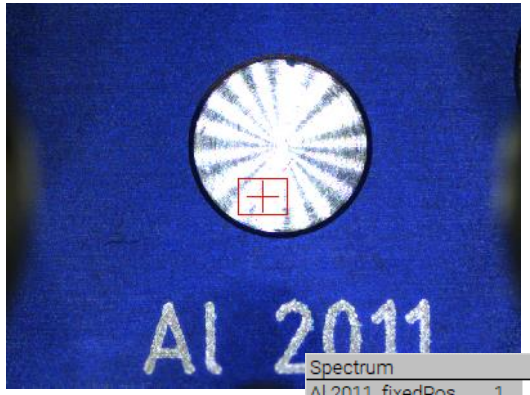
- Since all quantification is based on obtaining the net intensities of the fluorescence peaks, it is safe to assume that longer measurement time improves the precision of the quantification ...





- ... but there is a limit.

Which samples can be quantified? Homogeneity



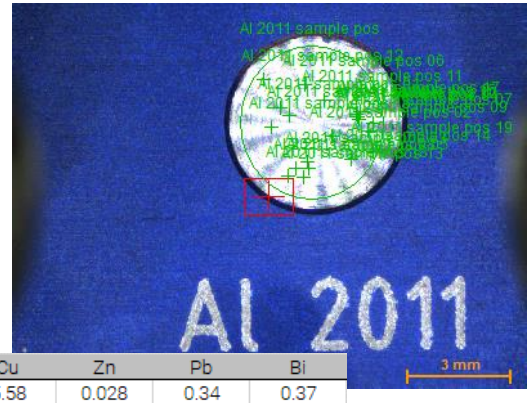
Instrument and analytical method performance

20 times same position
30 s each

Spectrum		Al	Mn	Fe	Cu	Zn	Pb	Bi
Al 2011 fixedPos	1	92.71	0.118	0.49	5.69	0.030	0.46	0.51
Al 2011 fixedPos	2	92.72	0.116	0.49	5.68	0.031	0.46	0.51
Al 2011 fixedPos	3	92.70	0.118	0.49	5.69	0.032	0.46	0.51
Al 2011 fixedPos	4	92.69	0.118	0.49	5.70	0.031	0.46	0.51
Al 2011 fixedPos	5	92.70	0.120	0.48	5.69	0.031	0.46	0.51
Al 2011 fixedPos	6	92.72	0.118	0.49	5.68	0.031	0.46	0.50
Al 2011 fixedPos	7	92.71	0.120	0.49	5.69	0.031	0.46	0.51
Al 2011 fixedPos	8	92.70	0.117	0.49	5.69	0.032	0.47	0.50
Al 2011 fixedPos	9	92.70	0.118	0.49	5.69	0.032	0.47	0.51
Al 2011 fixedPos	10	92.68	0.121	0.49	5.70	0.031	0.47	0.51
Al 2011 fixedPos	11	92.71	0.121	0.48	5.68	0.031	0.47	0.51
Al 2011 fixedPos	12	92.69	0.120	0.49	5.70	0.031	0.46	0.51
Al 2011 fixedPos	13	92.69	0.121	0.48	5.70	0.030	0.47	0.51
Al 2011 fixedPos	14	92.71	0.122	0.48	5.69	0.031	0.46	0.50
Al 2011 fixedPos	15	92.71	0.123	0.48	5.68	0.031	0.47	0.51
Al 2011 fixedPos	16	92.70	0.123	0.48	5.70	0.031	0.47	0.51
Al 2011 fixedPos	17	92.69	0.122	0.48	5.70	0.031	0.46	0.51
Al 2011 fixedPos	18	92.69	0.122	0.48	5.70	0.031	0.47	0.51
Al 2011 fixedPos	19	92.69	0.125	0.49	5.69	0.031	0.47	0.50
Al 2011 fixedPos	20	92.69	0.125	0.48	5.70	0.030	0.47	0.51
Mean value / wt.%:		92.70	0.120	0.48	5.69	0.031	0.47	0.51
Std. dev. / wt.%:		0.010	0.003	0.002	0.007	0.001	0.002	0.002
Std. dev. rel. / %:		**0.01**	**2.17**	**0.44**	**0.13**	**1.78**	**0.53**	**0.43**

Same as before plus sample homogeneity

20 different positions
30 s each

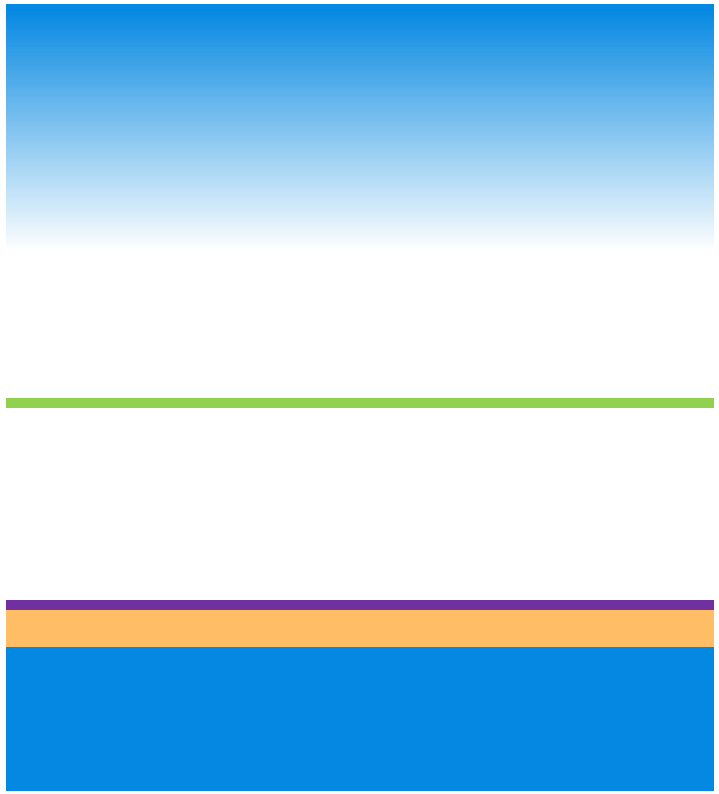


Spectrum		Al	Mn	Fe	Cu	Zn	Pb	Bi
Al 2011 sample pos	1	93.20	0.017	0.46	5.58	0.028	0.34	0.37
Al 2011 sample pos	2	92.52	0.065	0.47	5.98	0.034	0.44	0.49
Al 2011 sample pos	3	93.06	0.043	0.49	5.59	0.032	0.38	0.40
Al 2011 sample pos	4	92.83	0.066	0.46	5.61	0.032	0.49	0.52
Al 2011 sample pos	5	92.90	0.040	0.44	5.56	0.049	0.49	0.52
Al 2011 sample pos	6	92.44	0.030	0.51	6.11	0.035	0.42	0.46
Al 2011 sample pos	7	92.93	0.036	0.48	5.69	0.033	0.42	0.42
Al 2011 sample pos	8	92.85	0.019	0.49	5.66	0.046	0.47	0.48
Al 2011 sample pos	9	92.80	0.042	0.45	5.67	0.030	0.49	0.52
Al 2011 sample pos	10	93.20	0.144	0.44	5.58	0.032	0.27	0.32
Al 2011 sample pos	11	92.80	0.045	0.49	5.67	0.031	0.47	0.50
Al 2011 sample pos	12	92.96	0.092	0.43	5.52	0.026	0.48	0.49
Al 2011 sample pos	13	92.96	0.147	0.44	5.54	0.044	0.42	0.45
Al 2011 sample pos	14	93.04	0.034	0.47	5.63	0.027	0.38	0.42
Al 2011 sample pos	15	92.42	0.025	0.50	5.89	0.031	0.56	0.58
Al 2011 sample pos	16	92.84	0.021	0.49	5.60	0.027	0.49	0.53
Al 2011 sample pos	17	93.05	0.030	0.45	5.54	0.029	0.44	0.46
Al 2011 sample pos	18	92.92	0.035	0.44	5.51	0.028	0.53	0.55
Al 2011 sample pos	19	92.97	0.036	0.47	5.58	0.029	0.44	0.47
Al 2011 sample pos	20	92.91	0.017	0.48	5.60	0.027	0.47	0.49
Mean value / wt.%:		92.88	0.049	0.47	5.65	0.033	0.45	0.47
Std. dev. / wt.%:		0.214	0.038	0.023	0.158	0.007	0.066	0.061
Std. dev. rel. / %:		**0.23**	**76.59**	**4.87**	**2.79**	**19.96**	**14.72**	**13.02**

Which samples can be quantified? What “model” is implemented in the quantification algorithm?

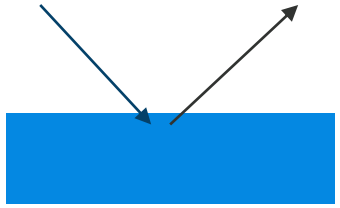
In XRF, samples are classified into 3 groups:

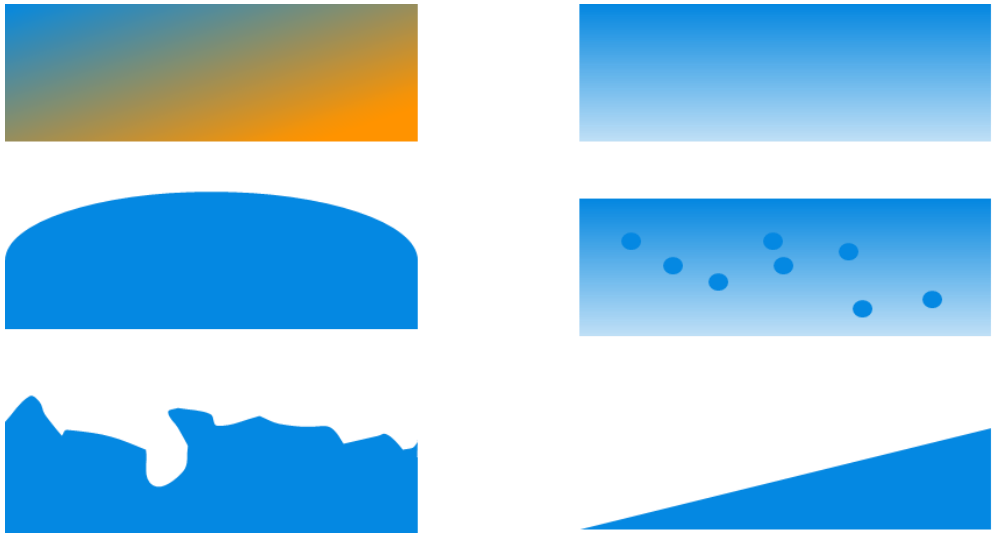
- Infinitely thick samples
  - A thicker sample would not change the XRF signal.
  - Bulk-XRF
- Thin samples
  - Attenuation and self-absorption effects can be neglected.
  - TXRF, some synchrotron science.
- Intermediate-thick samples
  - Not easy!
  - Almost all layer thickness analysis.
  - Makes a sample a “non-ideal” sample.



Which samples can be quantified? What is an ideal sample? What is not?

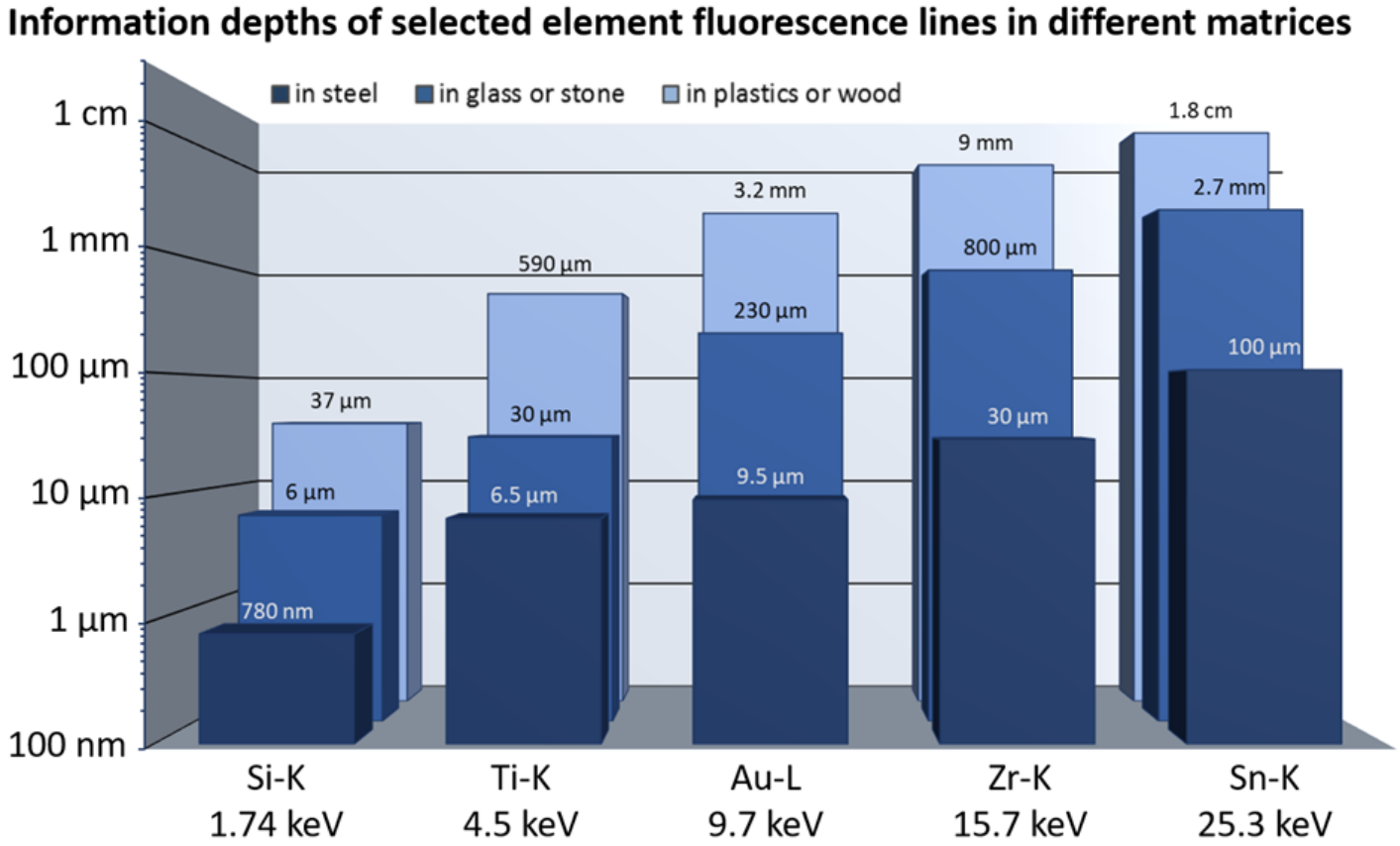
- Actually, anything that is not flat and homogeneous is not ideal.
- Homogenous, means **one composition** within the analytical volume!
- For bulk-XRF, in addition, the sample needs to be infinitely thick (for the analyzed elements).
- Defined sample geometry.





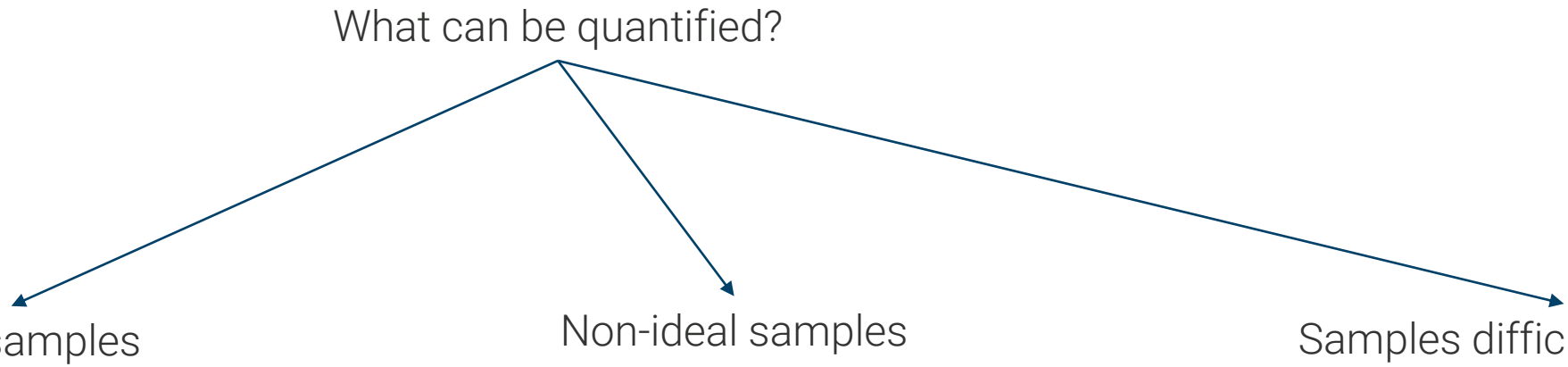
Which samples can be quantified? What “model” is implemented in the quantification algorithm?

- What is “infinitely thick”?
- The M4 TORNADO software comes with a Fundamental Parameter (FP) bulk quantification algorithm.
- For quantification of “non-ideal” samples XMethod software package can be used.



xrfcheck.bruker.com

Which samples can be quantified? ...and with what algorithm?

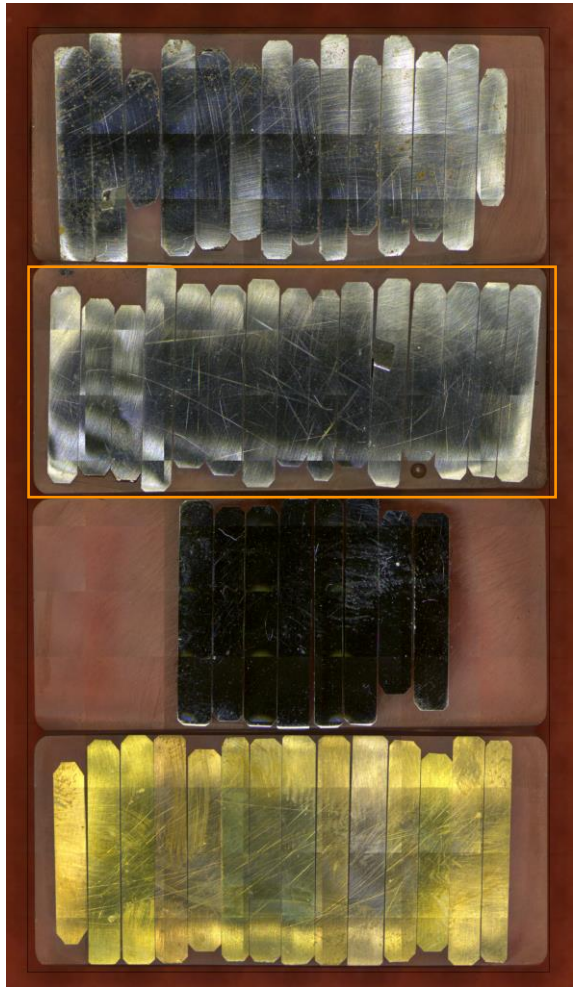


FP
- Flat, homogeneous bulk
- Flat layers

FP with larger uncertainties, i.e. lower accuracy
Empirical, if the standard matches the samples

empirical
- Wherever the standard matches the sample

Bulk-FP quantification of “ideal” samples
Steels

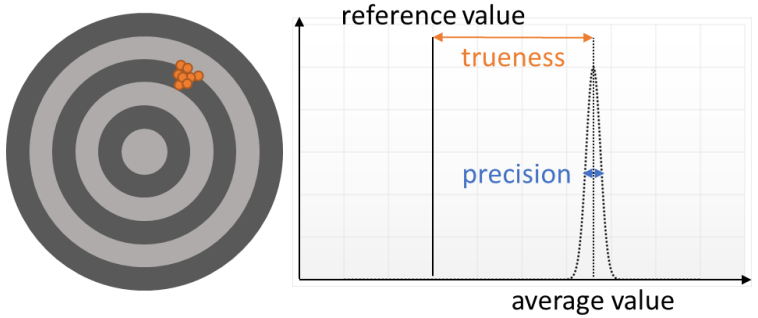


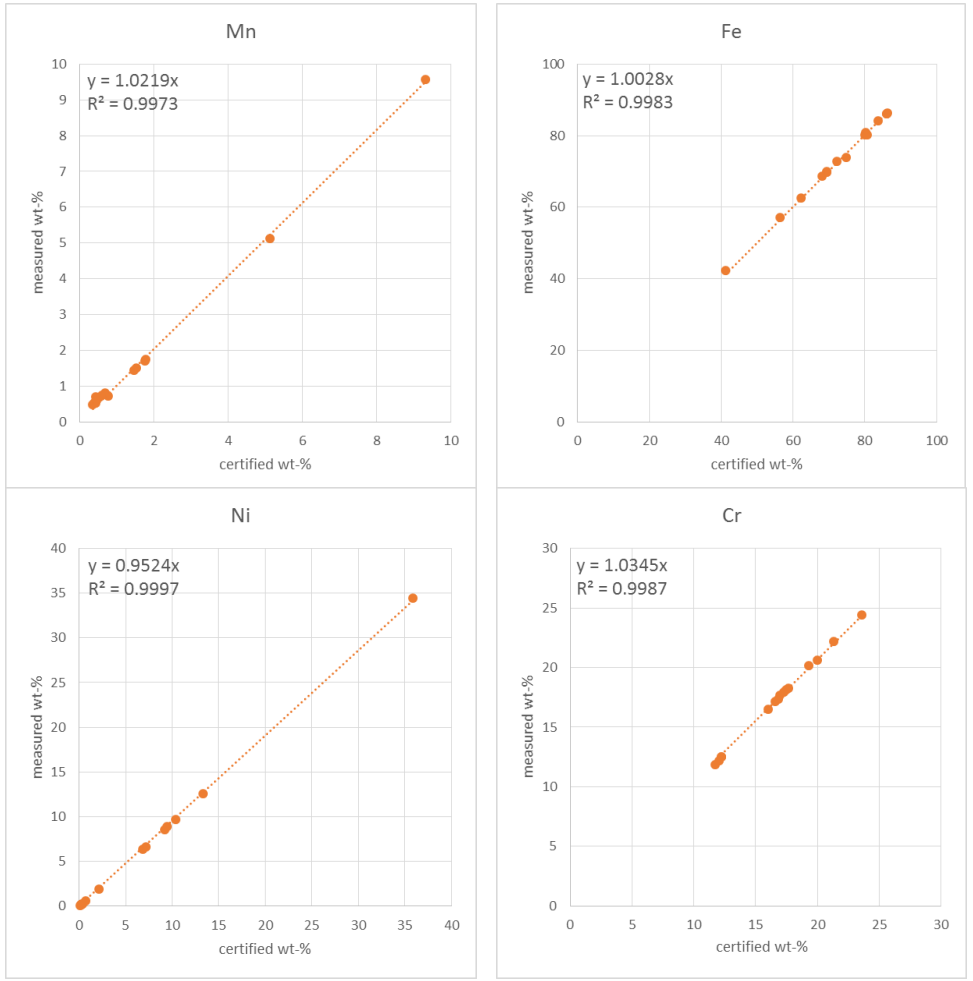
Grade-IARM	Al	Si	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Nb	Mo	W
AISI 316-5D	0.0050	0.5	0.0	0.0	16.6	1.8	68.2	0.1	10.4	0.17	0.004	2.1	0.0
AISI 321-6D	0.1100	0.3	0.6	0.1	17.5	1.5	69.4	0.2	9.4	0.30	0.039	0.4	0.1
AISI 330-7B	0.0230	1.4	0.0	0.0	19.3	1.5	41.3	0.1	35.8	0.21	0.023	0.2	0.0
AISI 347-8D	0.0040	0.4	0.0	0.1	17.3	1.8	69.3	0.1	9.2	0.47	0.720	0.4	0.1
AISI 410-9C	0.0140	0.4	0.0	0.1	12.0	0.4	86.2	0.0	0.3	0.06	0.005	0.2	0.1
AISI 416-10C	0.0030	0.4	0.0	0.0	12.3	0.4	86.0	0.0	0.2	0.16	0.003	0.1	0.0
AISI 420-154B	0.0020	0.5	0.0	0.1	12.2	0.4	86.1	0.0	0.2	0.09	0.003	0.1	0.0
AISI 422-205B	0.0090	0.4	0.0	0.3	11.7	0.7	83.7	0.0	0.7	0.15	0.018	1.0	1.1
AISI 430-11C	0.0100	0.5		0.0	17.7	0.5	80.8	0.0	0.2	0.07	0.005	0.1	
AISI 431-12B	0.0030	0.6	0.0	0.0	16.0	0.6	80.1	0.0	2.2	0.14	0.011	0.1	0.0
AISI 440C-13C	0.0030	0.7	0.0	0.1	16.8	0.4	80.2	0.0	0.1	0.03	0.004	0.5	
AISI 446-14B	0.0040	0.5	0.0	0.1	23.6	0.4	74.8	0.0	0.3	0.07	0.006	0.1	0.0
Nitronic 40-19B	0.0100	0.5	0.0	0.1	20.0	9.3	62.3	0.1	6.8	0.17	0.057	0.3	0.0
Nitronic 50-17B	0.0030	0.4	0.0	0.2	21.3	5.1	56.3	0.1	13.4	0.17	0.220	2.3	0.1
17-7PH-152B	1.1600	0.4	0.1	0.1	16.9	0.8	72.3		7.2	0.31	0.033	0.5	0.1

- Steels are usually homogeneous, they very quickly become infinitely thick, and they are easily made flat.
- Out of 51 ARMI reference alloys, we measured 15 stainless steels of certified composition.
- Average of 10 positions per sample with 10 s measurement time per point.

Bulk-FP quantification of “ideal” samples Steels

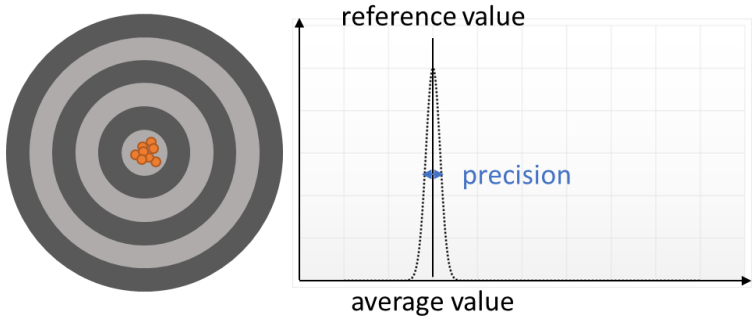
- Due to systematic physical effects (tertiary excitation in stainless steel samples) the quantification is not “spot-on”.
- The linearity of the regression shows a very stable recovery rate over the whole concentration range. → high precision
- The lack of trueness can be corrected for by calibration with one sample

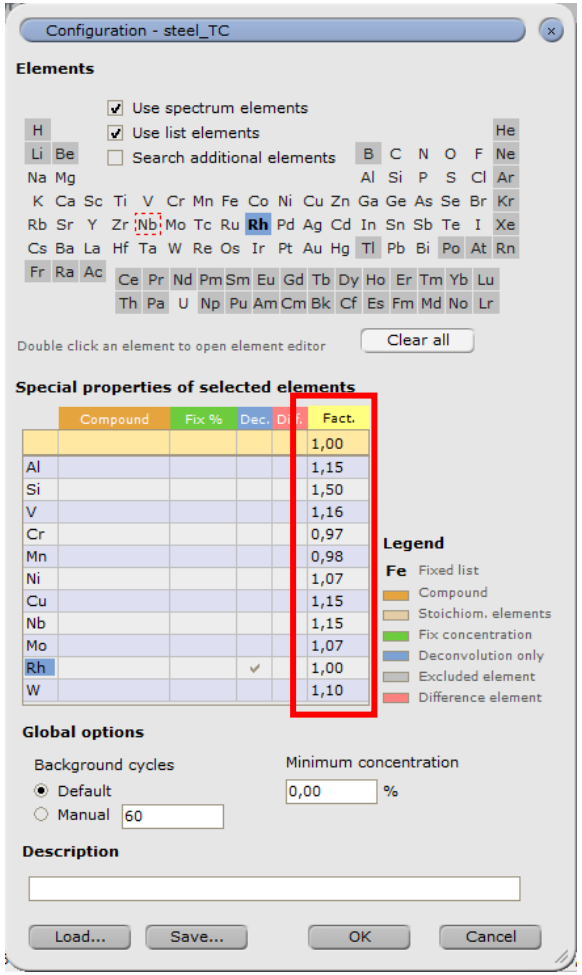


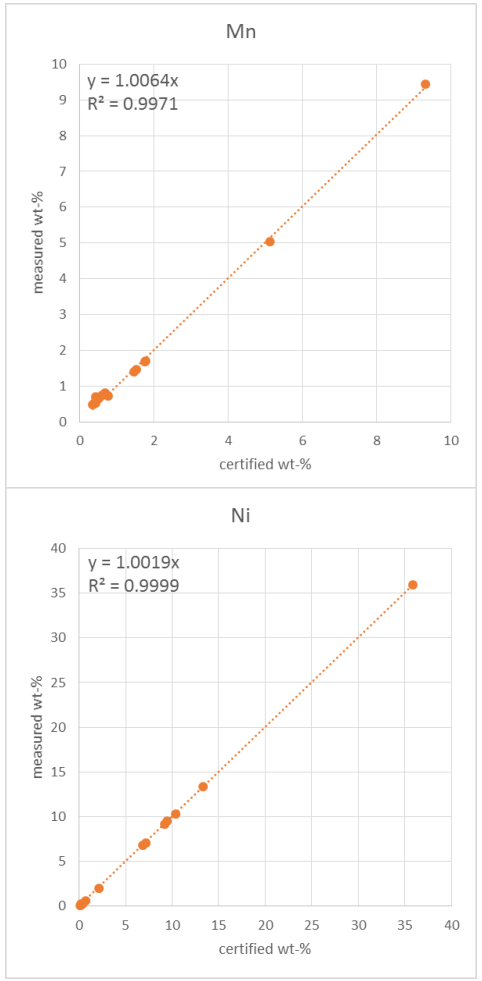


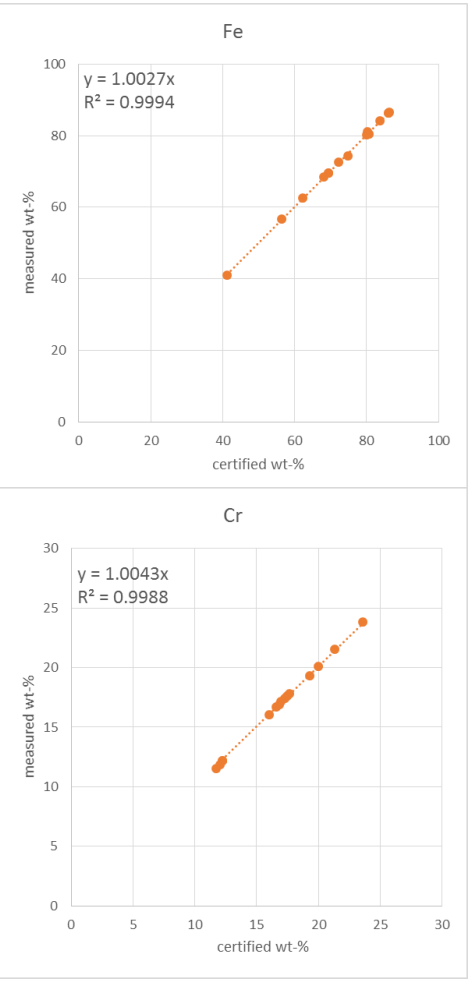
Bulk-FP quantification of “ideal” samples Steels

- Type-calibration: adapting the sensitivities of the FP algorithm for the elements to correct the recovery rate to 1

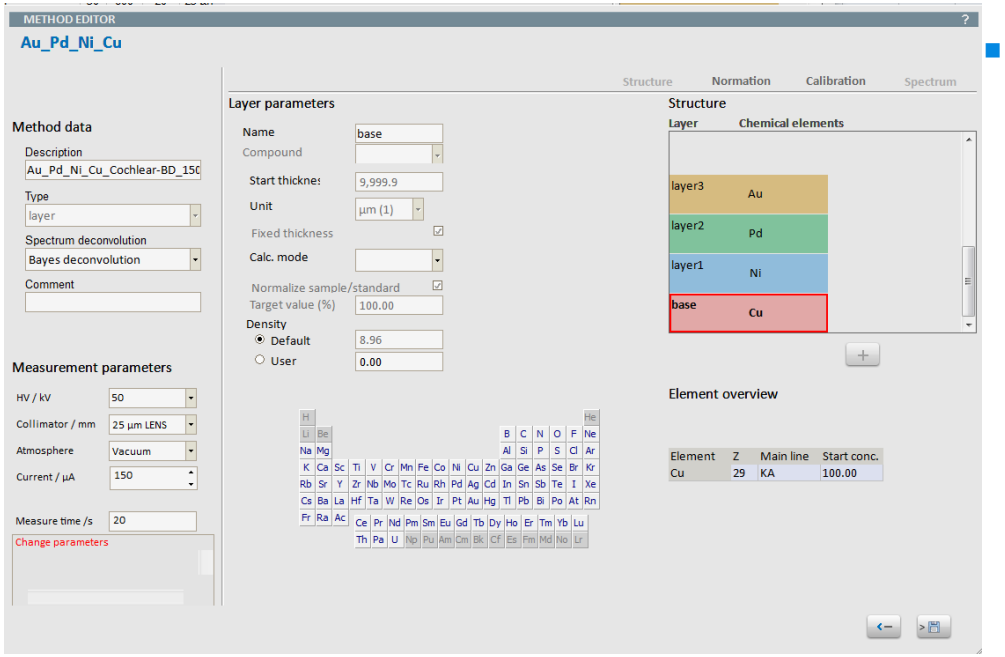




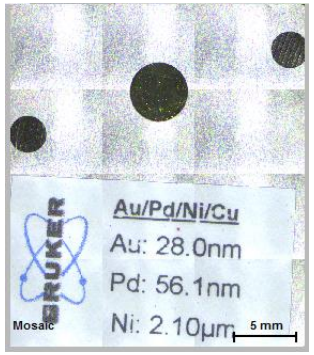




Layer-FP quantification of “ideal” samples
Metallic layers

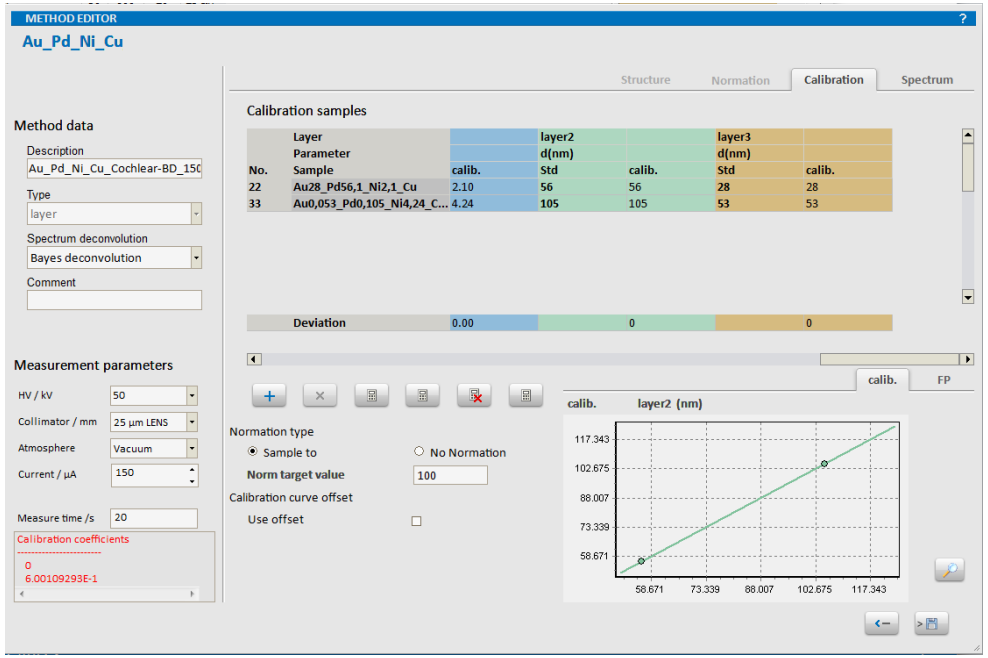


- To quantify layered samples, the quantification algorithm has to know the concentration of all the elements that are in each layer → appropriate ethod editor needed



- Even though FP methods work without calibration, sometimes (f.e. industrial applications) a calibration is required for highest accuracy (or proof thereof)

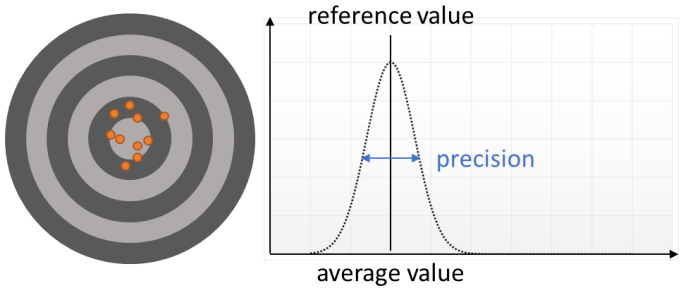
Calibration with two reference samples



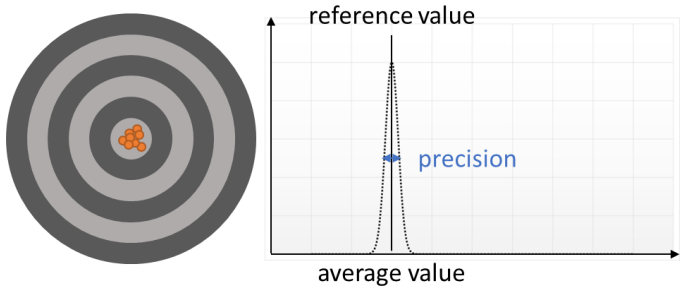
Layer-FP quantification of “ideal” samples Metallic layers

- The calibration corrects the trueness.
- The precision – if the sample allows – can be changed by different measurement times

Short measurements



long measurements



- For this sample system 20 s measurement time per point yielded sufficient stability.

10 points	Ni layer thickness	Pd layer thickness	Au layer thickness
1 s real time	/ µm	/ nm	/ nm
mean	2.09	50	28
sigma	0.05	14	3
sigma rel.	2.2	27.3	9.0

10 points	Ni layer thickness	Pd layer thickness	Au layer thickness
10 s real time	/ µm	/ nm	/ nm
mean	2.09	55	27
sigma	0.02	1.3	0.8
sigma rel.	0.7	2.3	2.8

10 points	Ni layer thickness	Pd layer thickness	Au layer thickness
60 s real time	/ µm	/ nm	/ nm
mean	2.10	54	27
sigma	0.01	0.7	0.3
sigma rel.	0.5	1.3	1.1

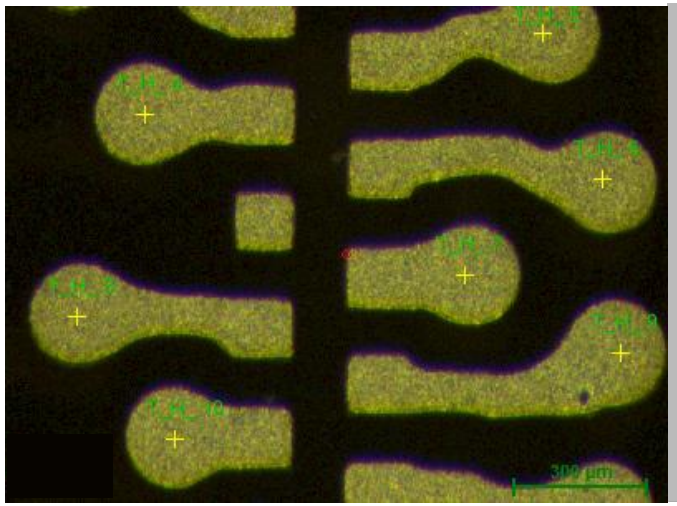
10 points	Ni layer thickness	Pd layer thickness	Au layer thickness
200 s real time	/ µm	/ nm	/ nm
mean	2.09	54	28
sigma	0.01	0.8	0.2
sigma rel.	0.4	1.5	0.8

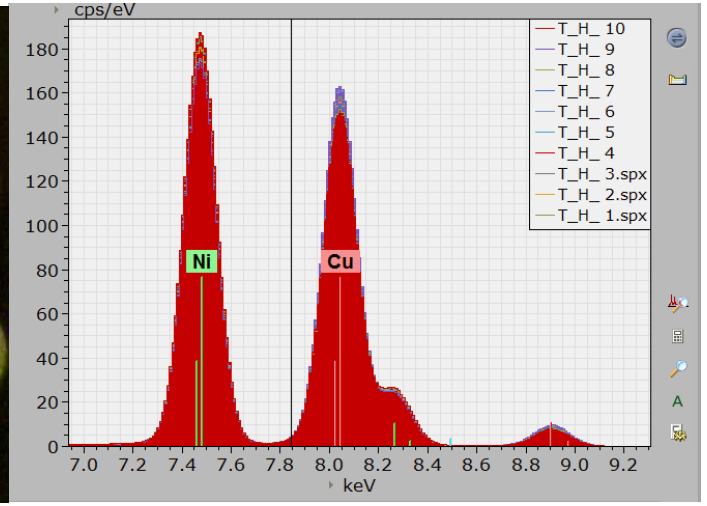
Layer-FP quantification of “ideal” samples
Metallic layers

- Verification on an independent layer thickness standard:

  Au: 53 nm
  Pd: 105 nm
  Ni: 4.02 µm

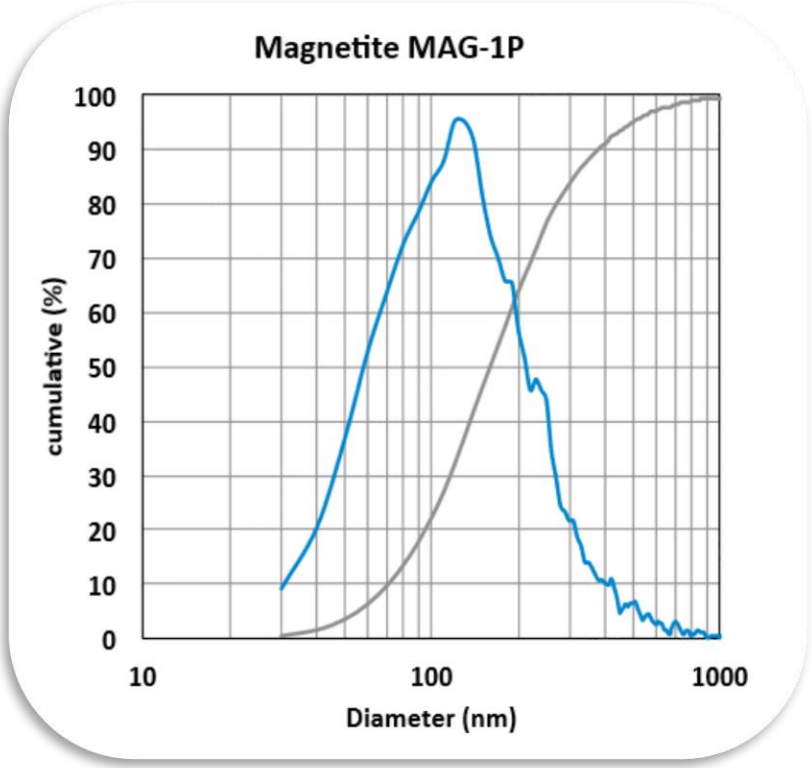
10 points	Ni layer thickness	Pd layer thickness	Au layer thickness
30 s real time	/ µm	/ nm	/ nm
mean	4.01	101	52
sigma	0.03	2	1
sigma rel.	0.7	2.1	1.2

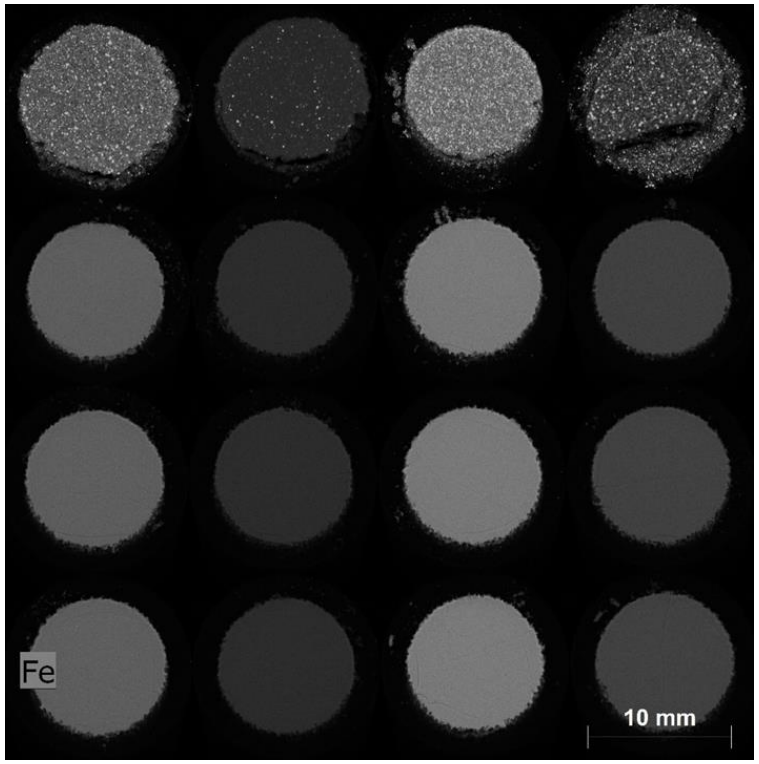


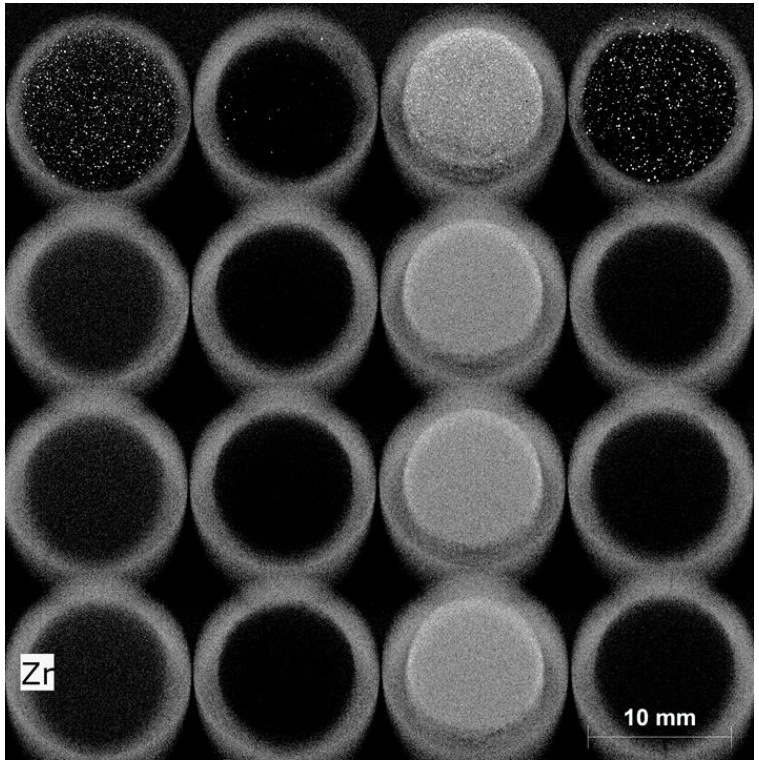


	Substrate	Layer 1		Layer 2		Layer 3	
Spectrum	Cu [%]	Thickn. [µm]	Ni [%]	Thickn. [µm]	Pd [%]	Thickn. [µm]	Au [%]
T_H_ 10	100.00	4.251	100.00	0.233	100.00	0.061	100.00
T_H_ 9	100.00	3.644	100.00	0.239	100.00	0.061	100.00
T_H_ 8	100.00	4.148	100.00	0.235	100.00	0.060	100.00
T_H_ 7	100.00	4.173	100.00	0.245	100.00	0.060	100.00
T_H_ 6	100.00	3.742	100.00	0.238	100.00	0.062	100.00
T_H_ 5	100.00	3.715	100.00	0.235	100.00	0.062	100.00
T_H_ 4	100.00	3.991	100.00	0.224	100.00	0.060	100.00

Bulk-FP of “ideal” samples
Nano-milled geological samples – major and minor elements



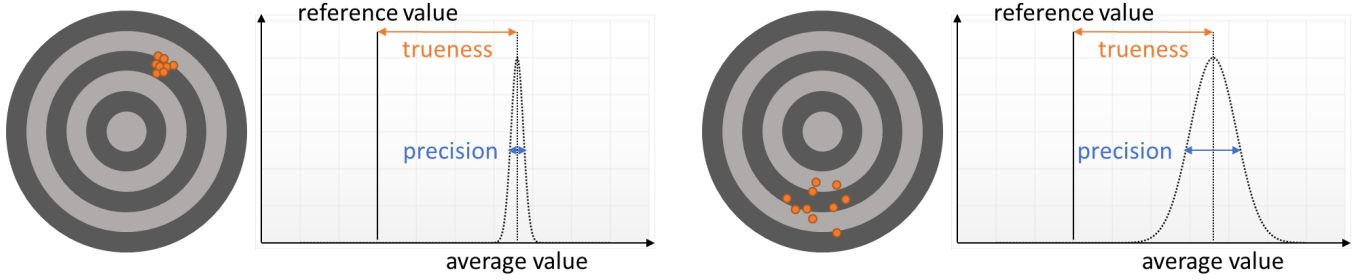


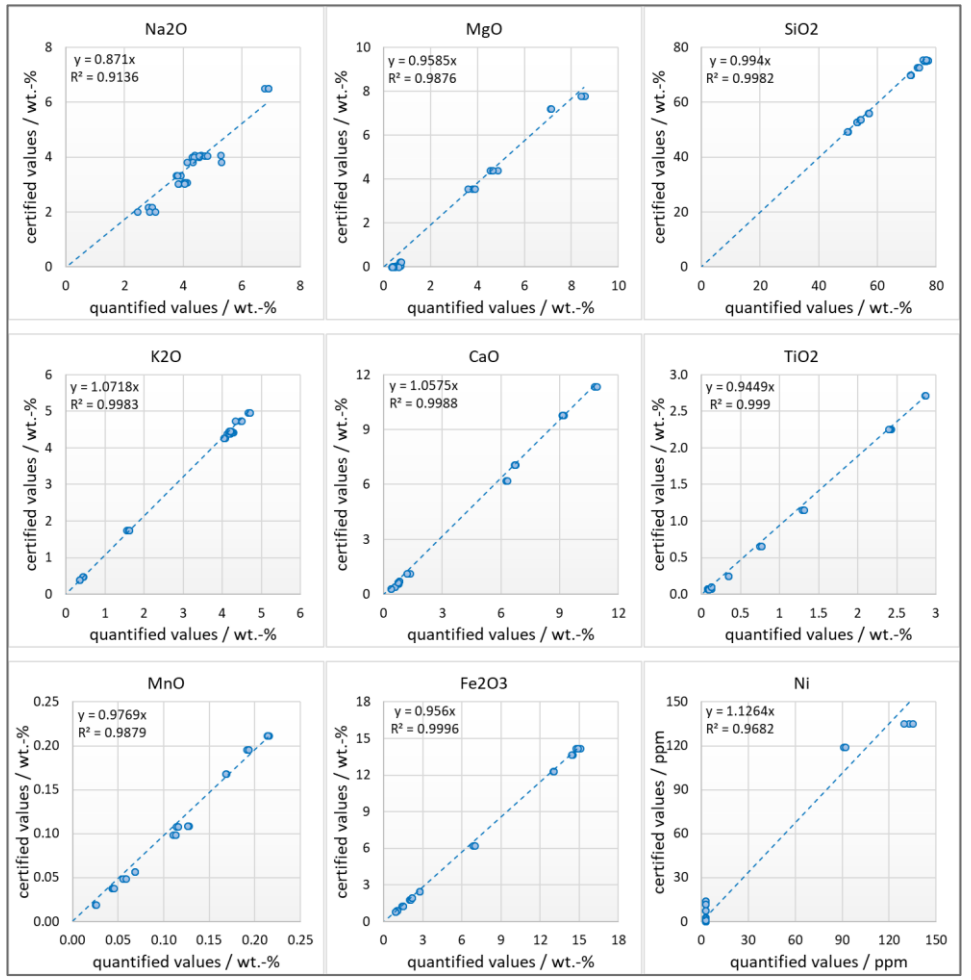


- Milling geo SRMs to < 200 nm gives the resulting powder a glass-like quality.
- There is no measurable inhomogeneity anymore. When pressed, they stick without binder. The grains are smaller than the information depth of the elements!

Bulk-FP of “ideal” samples
Nano-milled geological samples – major and minor elements

- Again, the recovery rate is very good over a wide concentration range. (especially when considering ~ 50 % “dark matrix”)
- Remember, the quantification assumes a sample composition, based on the selected elements and iteratively calculates all physical effects, like absorption and secondary excitation, and adapts the concentrations until the theoretical spectrum matches the measured one.





Empirical bulk-quantification
Nano-milled geological samples – trace elements

- When looking for trace elements, the signal-to-noise ratio of the measurement becomes crucial.
- To optimize the SNR for element like Rb, Sr, Y, Zr, Nb, a strong primary filter can be applied.
- BUT: using a strong filter reduces the excitation intensity for the light elements (which are major elements), therewith strongly afflicting the precision of the net peak intensity and, hence, the quantification.
- It is very ambitious to try trace element quantification like this.
- Solution: de-couple the quantification of the traces from the major elements.

http://georem.mpch-mainz.gwdg.de/sample_query.asp

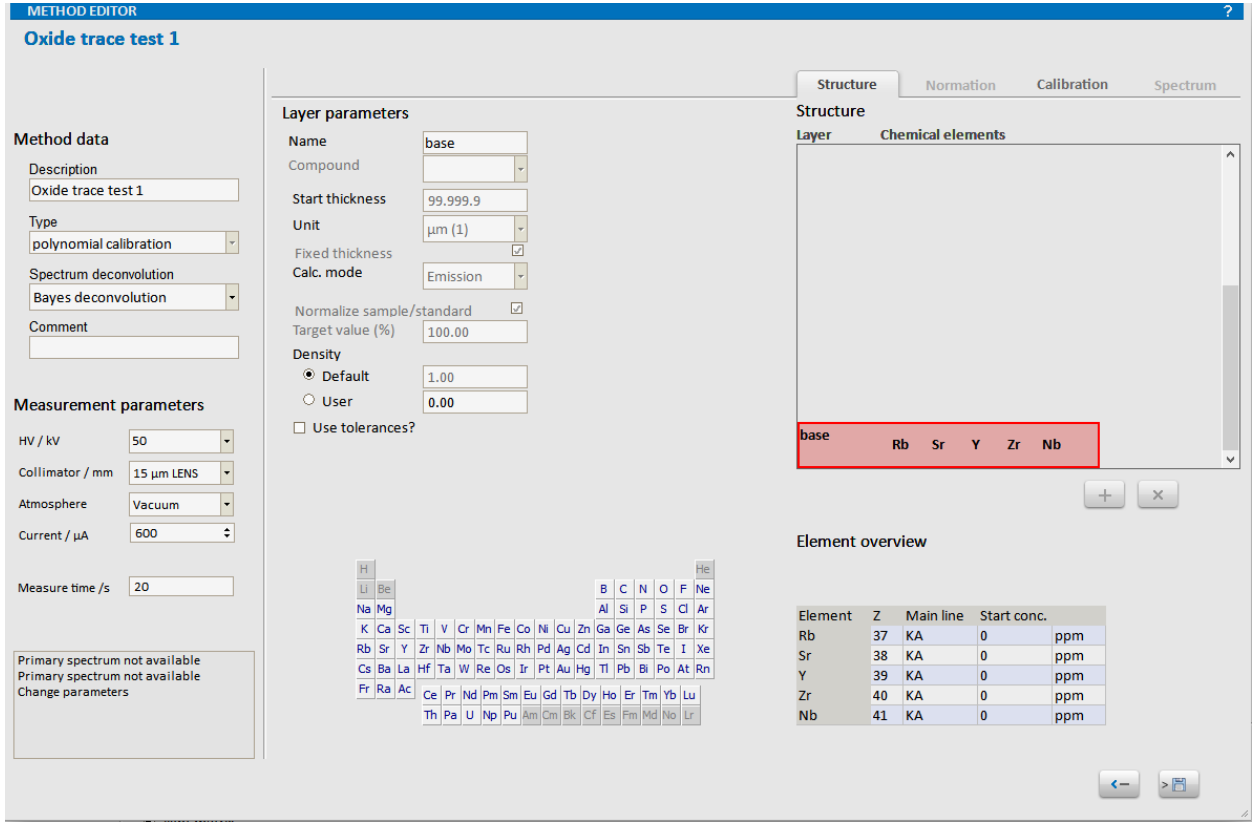
Elements Analyzed	Concentration Range / ppm
Rb	6 – 390
Sr	3 – 246
Y	17 – 75 (184*)
Zr	48 – 780
Nb	9 – 110
Pb	5 – 45
Th	5 – 87
U	2 – 18

Reference samples utilized:

GH, JR-2, AC-E, RGM-1, JA-2, JB-2, BHVO-2, SARM-1, NIST 620

Empirical bulk-quantification
Nano-milled geological samples – trace elements

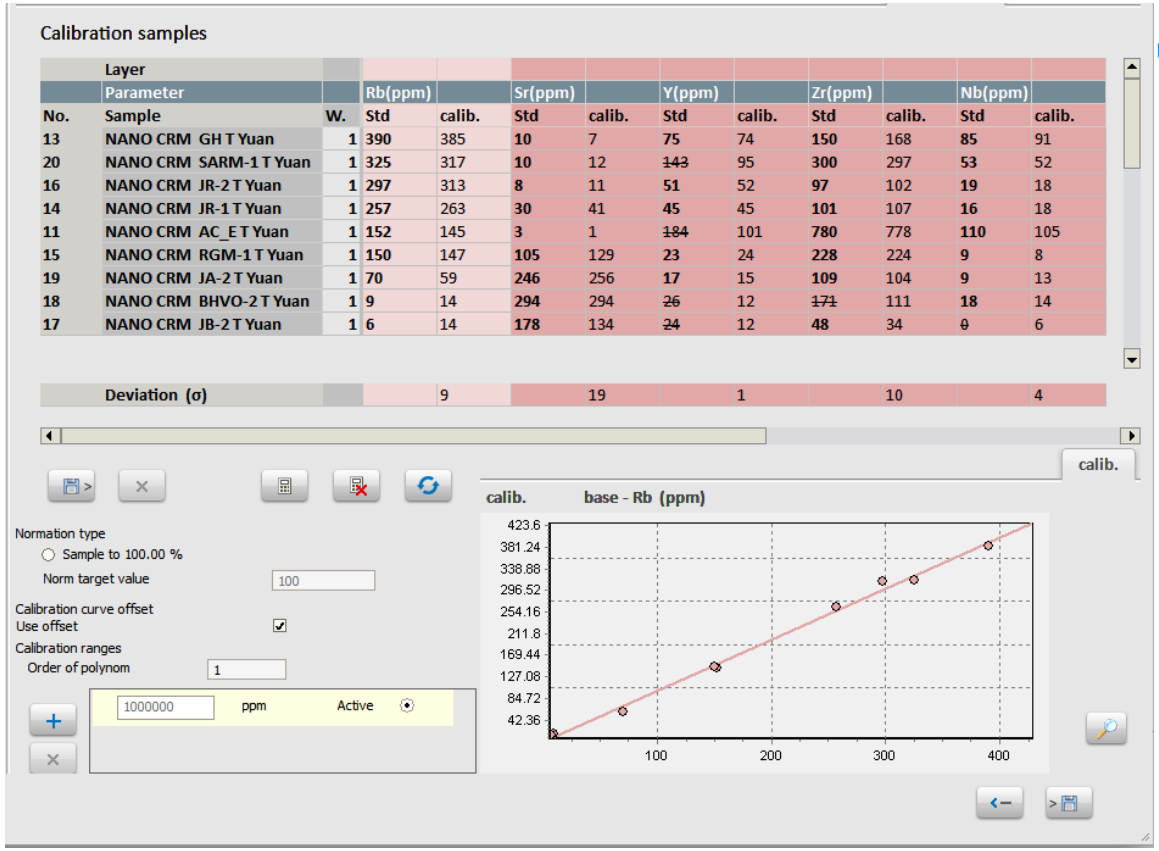
- The empirical method focuses solely on the elements of interest.
- This is a valid approach, because in oxidic matrix there are no relevant inter-element effects for these trace elements.
- So, ignoring 99.9 % of the sample, we basically determine the instrument's sensitivity (cps per ppm) for these elements.



Empirical bulk-quantification
Nano-milled geological samples – trace elements

Calibration samples

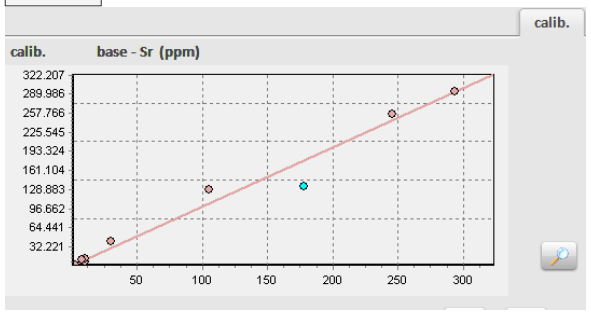
	Layer		Rb(ppm)		Sr(ppm)		Y(ppm)		Zr(ppm)		Nb(ppm)	
	Parameter		Std	calib.	Std	calib.	Std	calib.	Std	calib.	Std	calib.
No.	Sample	W.										
13	NANO CRM GH T Yuan	1	390	385	10	7	75	74	150	168	85	91
20	NANO CRM SARM-1 T Yuan	1	325	317	10	12	~~143~~	95	300	297	53	52
16	NANO CRM JR-2 T Yuan	1	297	313	8	11	51	52	97	102	19	18
14	NANO CRM JR-1 T Yuan	1	257	263	30	41	45	45	101	107	16	18
11	NANO CRM AC_E T Yuan	1	152	145	3	1	~~184~~	101	780	778	110	105
15	NANO CRM RGM-1 T Yuan	1	150	147	105	129	23	24	228	224	9	8
19	NANO CRM JA-2 T Yuan	1	70	59	246	256	17	15	109	104	9	13
18	NANO CRM BHVO-2 T Yuan	1	9	14	294	294	~~26~~	12	~~171~~	111	18	14
17	NANO CRM JB-2 T Yuan	1	6	14	178	134	~~24~~	12	48	34	~~0~~	6
	Deviation (σ)			9		19		1		10		4



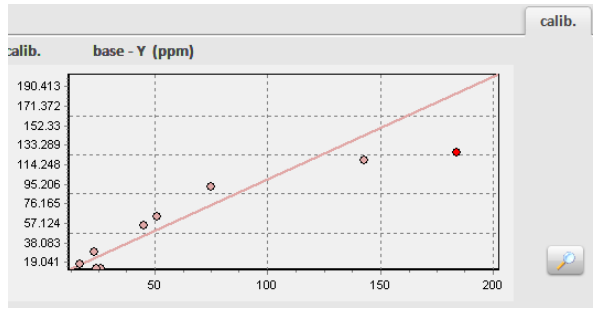
Rb average deviation = 9 ppm
Deviation of Rb in all samples ± 15 ppm

- Used calibration curve: linear correlation with offset

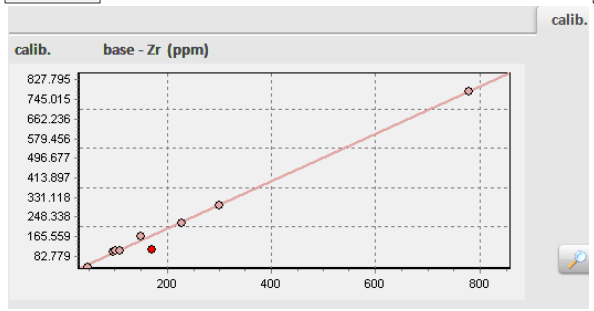
Sr



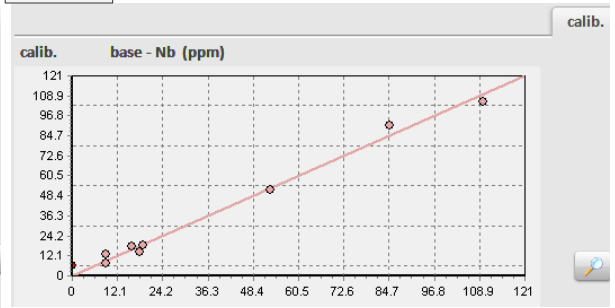
Y



Zr



Nb



Empirical bulk-quantification
Nano-milled geological samples – trace elements

- Detection limits in micro-XRF depend on instrument performance, samples used, and measurement conditions. In combination with a simple mathematical model allows fast and accurate quantification of geological materials or their derivates such as ceramics.

- The major uncertainty is the “known” values and (in)homogeneity for the chosen reference samples. Issues with sample inhomogeneity can be overcome in “ideal” samples such as the nano-powders described previously (or see presentation on nano-powders).

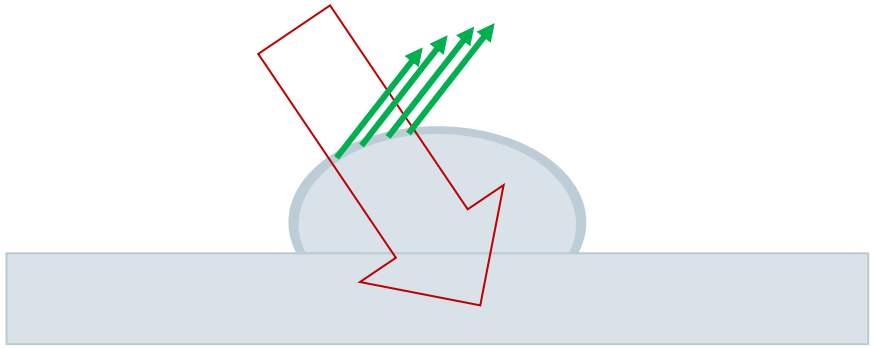
Element	Avg dev
Rb	9
Sr	19
Y	14
Zr	10
Nb	4

Values in ppm mg/kg sample	Rb	Sr	Y	Zr	Nb	Pb	Th	U
Estimated limit of detection*	1	1	1	3	2	1	1	1
Estimated limit of quantification**	3	3	3	5	5	3	3	4
* LOD= 3*concentration/SNR								
* LOQ= 9*concentration/SNR								

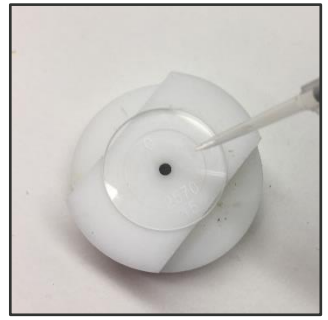
These limits represent method limits for oxide sample analysis with the M4 Tornado under the previously described condition.

Empirical bulk-quantification
Really adverse geometry

- Measuring sulfur in oil can be challenging, especially when only limited amounts of oil are available.
- Small amounts of oil for a droplet, which really does not fulfill the criteria of “ideal samples”.
- It’s round, most of the X-rays go through, wether the S is at the surface or homogenously distributed is difficult to assess, ...
- But: There is limited information depth for S and the self-absorption effects are negligible.
- Assumption: concentration of S oil is linear with measured intensity



Oil droplet

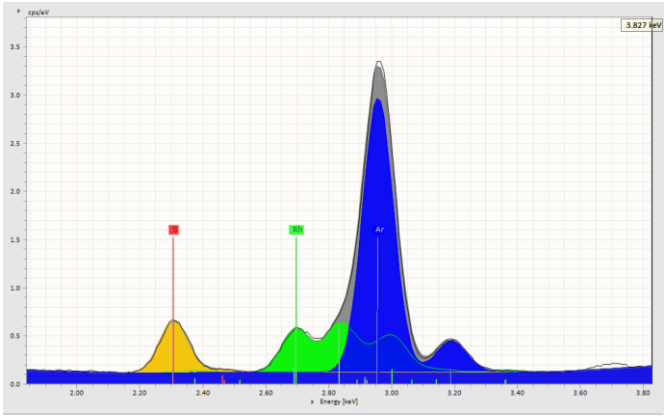


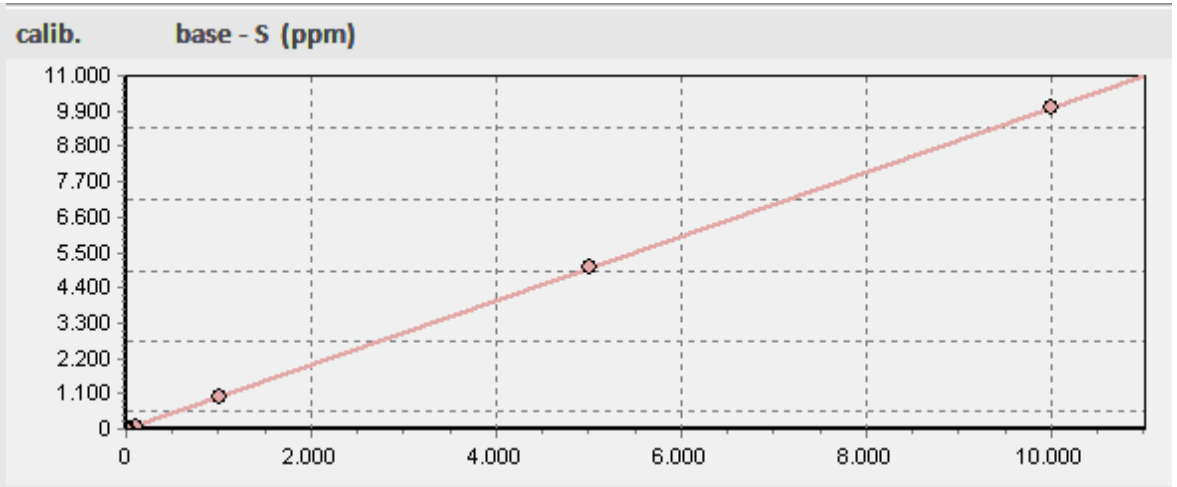
Empirical bulk-quantification
Really adverse geometry

- 10 µl of each reference sample (CONOSTAN) were deposited on a clean SiO_2 disc
- Concentration of S from 0 to 10000 ppm (1 %)
- The sample description merely accounts for the elements that interfere with the fitted S intensity

base S Rh Ar

- Still the approach yields very good quantification.



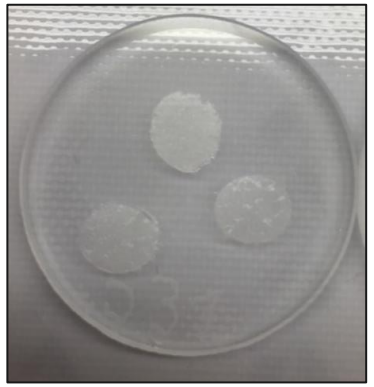


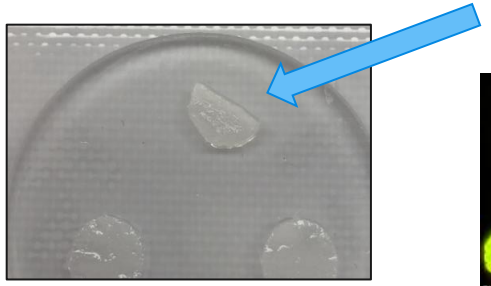
Calibration samples

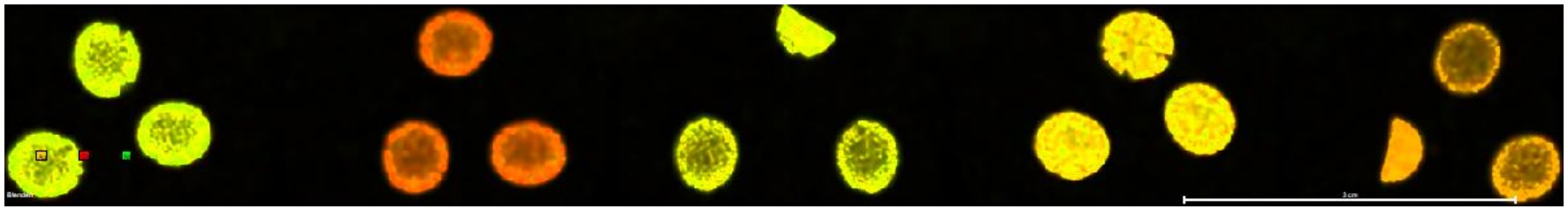
No.	Layer / Parameter / Sample	W.	S(ppm) Std	S(ppm) calib.	Rh(%) Std	Rh(%) calib.	Ar(%) Std	Ar(%) calib.
124	S_OIL_10000 ppm	1	10000	9993	~~0.00~~	0.00	~~0.00~~	0.00
123	S_OIL_5000 ppm	1	5000	5014	~~0.00~~	0.00	~~0.00~~	0.00
122	S_OIL_1000 ppm	1	1000	995	~~0.00~~	0.00	~~0.00~~	0.00
120	~~S_OIL_500 ppm~~	0	500	458	~~0.00~~	0.00	~~0.00~~	0.00
119	S_OIL_100 ppm	1	100	93	~~0.00~~	0.00	~~0.00~~	0.00
118	S_OIL_25 ppm	1	25	23	~~0.00~~	0.00	~~0.00~~	0.00
116	S_OIL_0 ppm	1	0	6	~~0.00~~	0.00	~~0.00~~	0.00

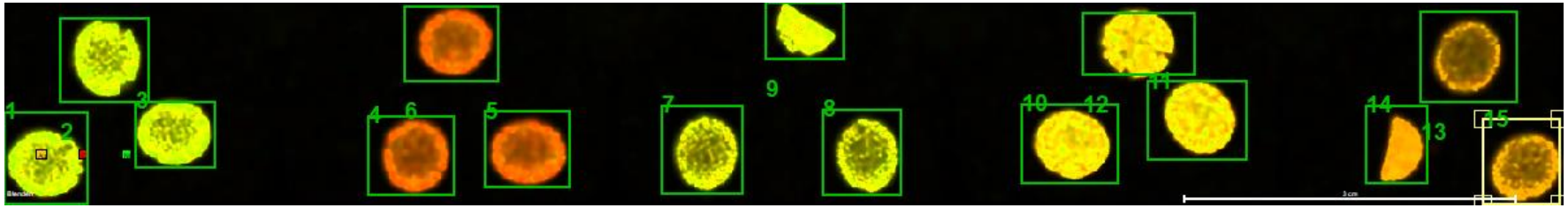
Empirical bulk-quantification
Soaked-up liquids

- Deposition of a **defined amount of solution in a defined area**, scanning, and integration the detected fluorescence intensity.
- For the analysis 5 µl-droplets were deposited on a ~ Ø 6 mm absorbent tissue.
- To evaluate impact of poor sample preparation, two samples were “folded”.





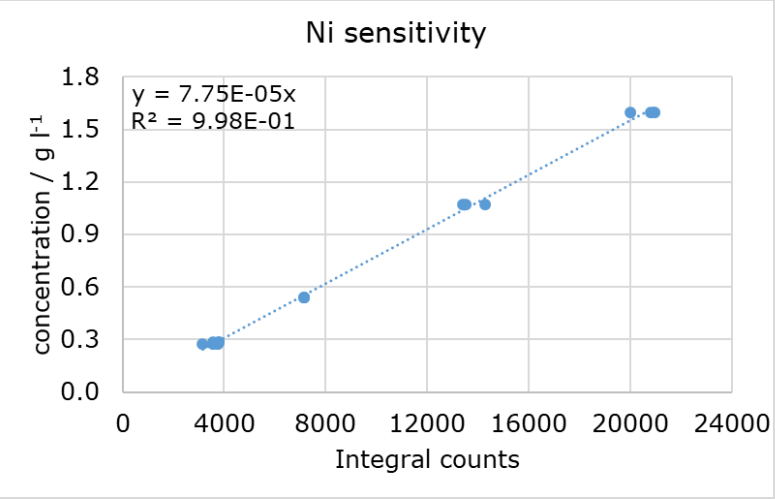


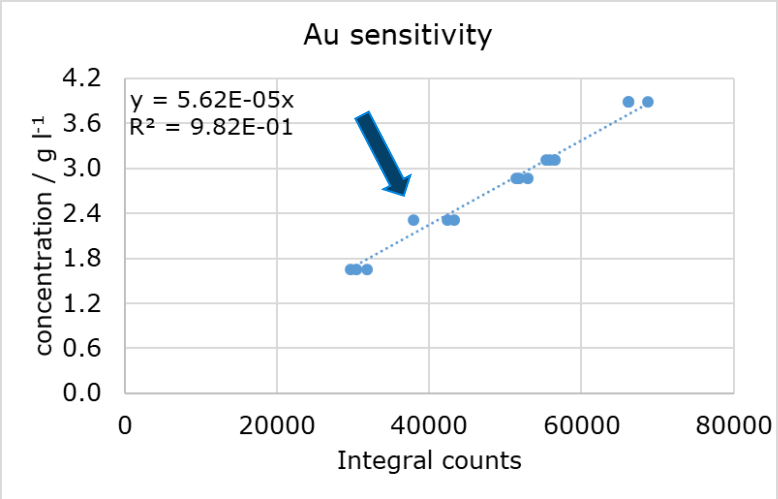


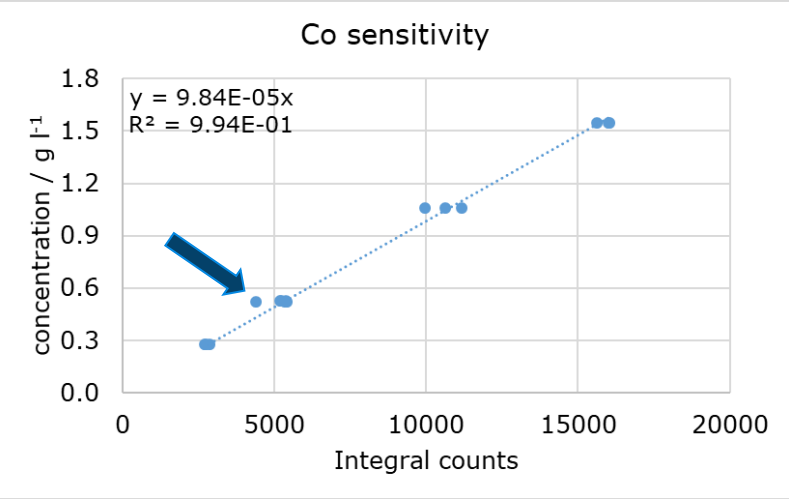
Empirical bulk-quantification Soaked-up liquids

- There is a very good linear correlation between the extracted intensity and the concentration. From the slope (sensitivity) the concentration in the sample can be derived. The main uncertainty results from the droplet preparation (see the deviation in the 3 dots for the same concentration).

- The folded samples also results in small deviations (see arrow).

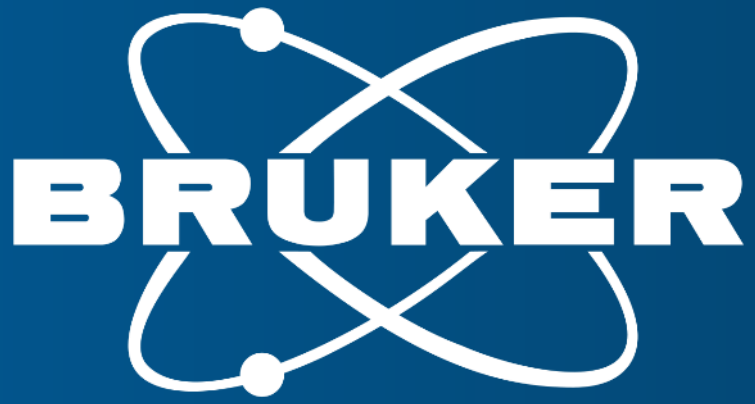






Summary

- Micro-XRF is versatile quantification tool for the analysis of multiple sample types from solid to liquids or powders.
- The combination of high element sensitivity from major to trace elements combined with the easy operation allows the use of multiple empirical and fundamental parameter-based models.
- Every sample with a defined composition within the analytical volume can be quantified as bulk.
- If the sample is not homogenous you can either make compromises in terms of trueness as the sample does not have a real or true “composition”, or you move to empirical approaches
- Layer samples can be quantified with high accuracy as long as the layer succession and composition is known.
- Liquid samples can be measured depending on the element of interest (low-Z, high-Z) or nature of the liquid (drying easily or not).



Innovation with Integrity