XRF WEBINAR SERIES 2021

When Precision Counts – How new XRF technology masters challenges in Quality Control in Metals

Kai Behrens and Adrian Fiege

15.09.2021

When Precision Counts – How new XRF technology masters challenges in Quality Control in Metals

Kai Behrens
Head of XRF Product Management

PhD of Analytical Chemistry, Hamburg

2001 Method Development Bruker AXS

Now Head of Product Management XRF

Karlsruhe, Germany

Adrian Fiege
Product Manager XRF

PhD, Geology, Hannover

2019 Product Management Bruker AXS

Product Management XRF

Karlsruhe, Germany

Outline

01 Backgrounds on Metal Production

02 Typical applications of high-end WDXRF

03 When to use benchtop WDXRF and EDXRF

04 Failure Analysis using XRF-Mapping

05 Coatings and Multi-Layer Analysis

06 Applications in Additive Manufacturing

07 Summary and Q&A

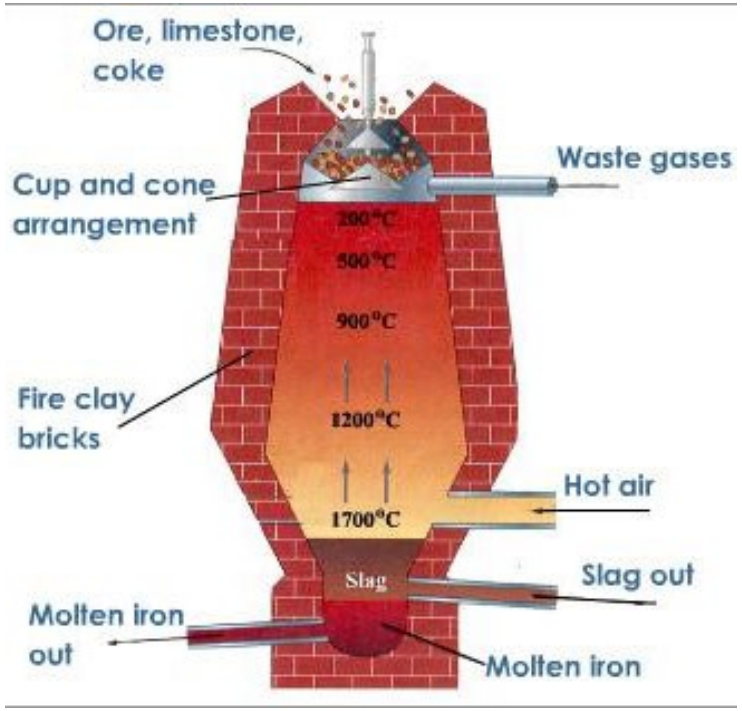
Why XRF analysis is key in today‘s technology change for better environment and why precison and speed count!

- Metals play a vital role in today‘s life, despite any change in technology – Aluminum is required to reduce weight for energy saving, copper will be needed for the new e-mobility and steel and still on high demand for housing
- New initiatives are driving the demand for extensive analysis:
  - Durability: traces of copper are harmful for corrosion resistance of steel – this is key parameter for any use of scrap
  - Energy saving: blast furnace slags are providing insight information about the kiln status in steel making
  - High purity: Copper for energy transportation requires high purity copper to reduce the electrical resistance
- Just a few examples where XRF is a great help!

WDXRF Analysis
Blast Furnace Slags

- Furnace condition critical when extracting iron from the ore
- Slags are formed from metal oxides during the process
  - Removal of “waste” from the molten metal
  - Protect against re-oxidation of molten iron and steel
  - Reduce energy consumption
  - Analysis is vital in blast furnace operation to access furnace conditions
  - Dosing of flux, limestone, coke and sinter
- Analysis speed
- Few elements, but light ones

S8 TIGER Sequential WDXRF for high performance and full analytical flexibilty



Blast Furnace operation

Slag Analysis
Excellent Precision

Sample	SiO2 (%)	CaO (%)	Al2O3 (%)	MgO (%)	TiO2 (%)	S (%)	CaO/SiO2
SLAG	37,41	39,49	18,04	4,01	0,903	0,751	1,056
SLAG/1	37,46	39,53	18,01	4,01	0,905	0,751	1,055
SLAG/2	37,34	39,44	17,99	3,99	0,903	0,754	1,056
SLAG/3	37,29	39,47	18,01	4,00	0,902	0,752	1,058
SLAG/4	37,44	39,50	18,00	4,00	0,903	0,751	1,055
SLAG/5	37,49	39,49	17,99	4,00	0,902	0,751	1,053
SLAG/6	37,43	39,48	18,00	4,00	0,903	0,751	1,055
SLAG/7	37,39	39,48	18,00	4,00	0,902	0,752	1,056
SLAG/8	37,40	39,46	18,00	4,00	0,902	0,751	1,055
SLAG/9	37,40	39,45	17,99	4,00	0,901	0,753	1,055
SLAG/10	37,40	39,47	18,00	3,99	0,901	0,753	1,055
SLAG/11	37,36	39,47	17,99	4,00	0,902	0,754	1,056
Mean	**37,40**	**39,48**	**18,00**	**4,00**	**0,902**	**0,752**	**1,055**
Std Dev	0,05	0,02	0,01	0,01	0,001	0,001	0,001
RSD	**0,14**	**0,06**	**0,07**	**0,15**	**0,125**	**0,143**	**0,114**

Central Lab
Analytical work

- Grade control of incoming materials:
  - Iron ore
  - Coke
  - Ferro-alloys, binary alloys
  - Scrap (Handheld-XRF)
  - Limestone, flux

  → Accuracy determines margin!

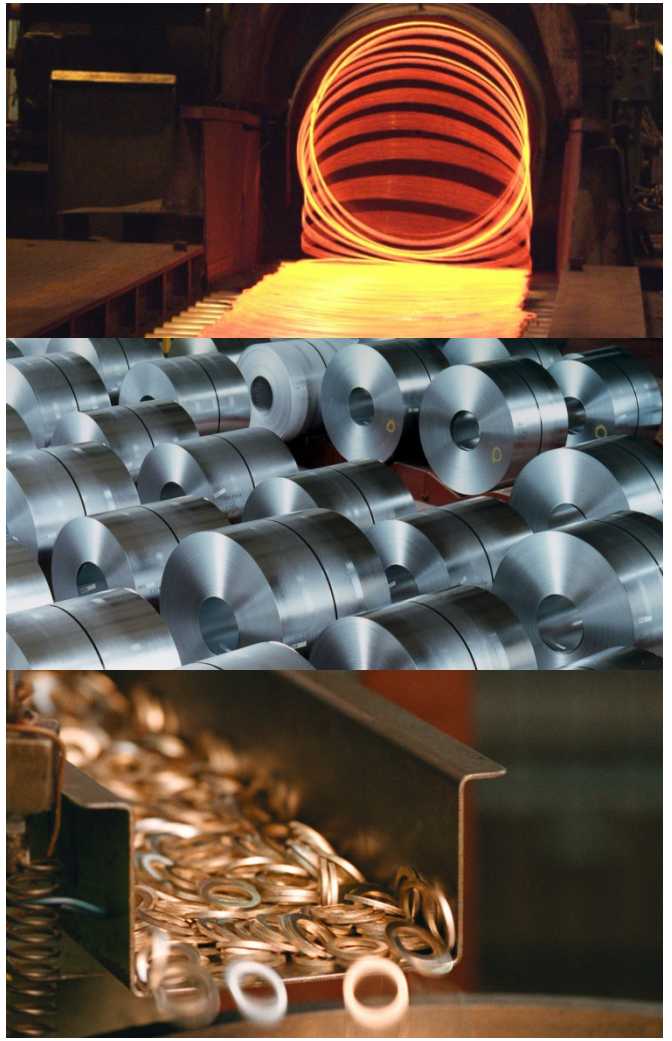
- Quality Control
  - Final determination of product composition
  - Layer analysis (protective coatings – thickness and composition – Zn, P, Polymers …)

Definition of Ferro Alloys

- Ferro Alloys
  are widely used in low and high alloy steel production in the final step
- To achieve the final specified melt composition
- Group of mostly binary mixtures, such as:

  Fe-Cr, Fe-Si, Fe-Mo, Fe-W, Fe- Ni,…
- Rarely mixture of three or four elements:

  Fe-Cr-Mg, Fe-Si-Cr, Fe-Si-Mn,…
- Hard material, produced and delivered typically in lump form



Analytical Requirements

Producer and Customer:

- Determination of commercial value of ferro-alloys
  - Major and minor components with high accuracy, homogenous composition of the material
- Determination of traces and impurities
  - Avoiding the contamination of produced steel products

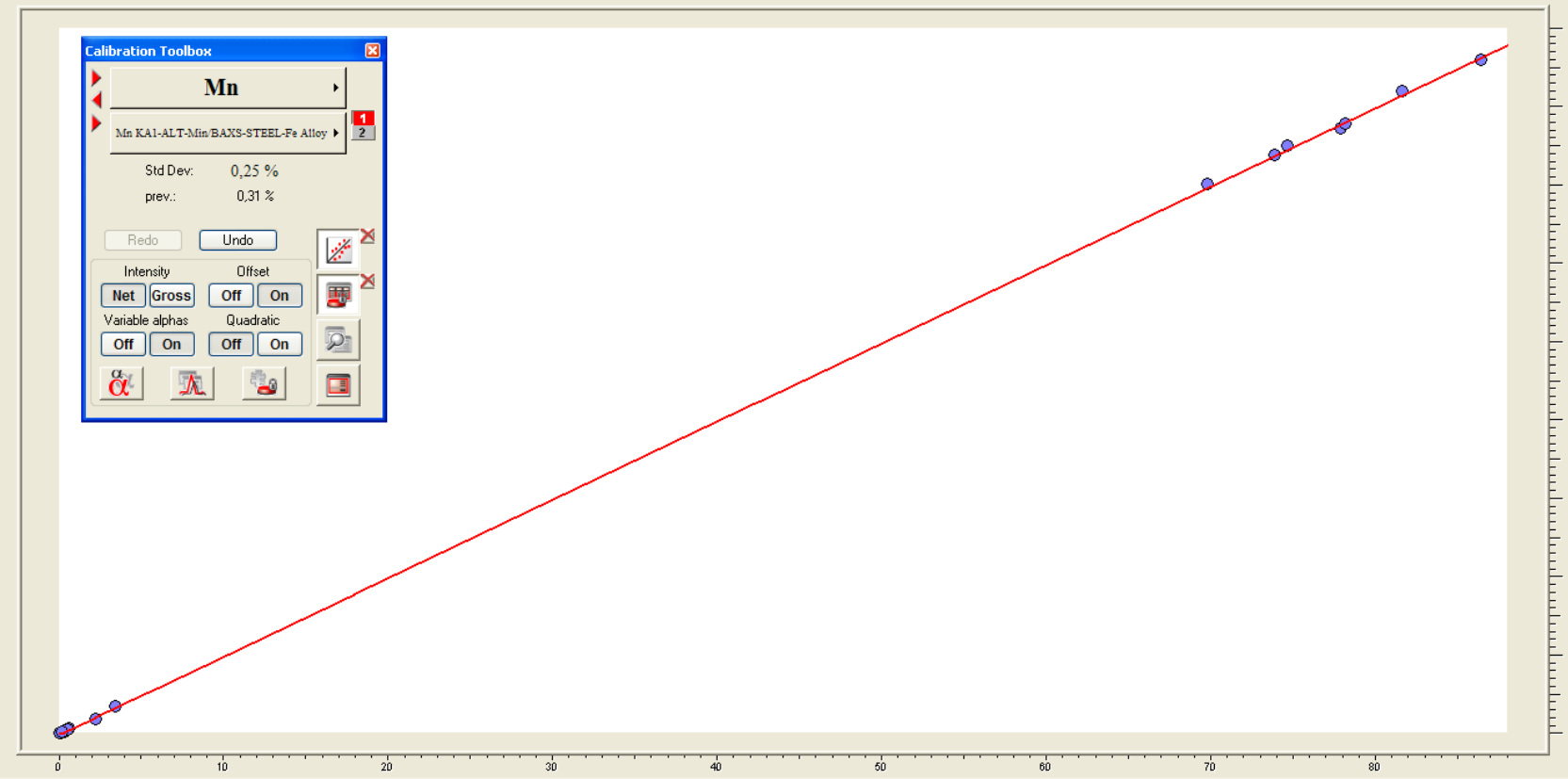
Analytical Requirements

- Quick and reliable sample preparation in order to produce high quality results:
  - Pressed pellets not possible due to particle size effects
  - Material is not sticky – difficult for reliable sample preparation

- Feasible for production control at manufacturer site due to homogenous material and narrow concentration range
  - Will fail at steelmakers lab and central laboratory

Global Calibration
Fe – Mg – Si – Cr – Mn – V – Mo – W - Nb

- One global calibration method based on variable alphas covers in one function a wide concentration range
- Mn at trace level/impurity, medium and high concentration range
- Calibration standard deviation kept in the same range: 0.25 % instead of 0.76% for theoretical FP model



Precision Test

Fe – Mg – Si – Cr – Mn – V – Mo – W - Nb

Sample	Al (%)	Si (%)	Mg (%)	Ca (%)	P (%)	Ti (%)	V (%)	Cr (%)	Mn (%)	Fe (%)	Co (%)	Ni (%)	Ce (%)
FeCrMg 1	7.781	1.177	31,930	0,844	0.006	0.086	0.053	12.036	0.091	7.530	0.013	0.101	0.180
FeCrMg 2	7.759	1.179	32,065	0,842	0.005	0.083	0.054	12.029	0.087	7.520	0.007	0.101	0.180
FeCrMg 3	7.790	1.174	31,998	0,835	0.004	0.087	0.049	12.039	0.088	7.520	0.010	0.099	0.180
FeCrMg 4	7.785	1.179	32,080	0,832	0.004	0.079	0.052	12.055	0.088	7.520	0.010	0.099	0.180
FeCrMg 5	7.787	1.173	32,013	0,838	0.005	0.082	0.053	12.033	0.091	7.520	0.008	0.101	0.180
FeCrMg 6	7.804	1.170	31,995	0,838	0.005	0.081	0.053	12.030	0.088	7.520	0.008	0.101	0.180
FeCrMg 7	7.784	1.180	32,046	0,838	0.006	0.087	0.055	12.043	0.087	7.530	0.008	0.100	0.190
FeCrMg 8	7.820	1.165	32,099	0,840	0.006	0.081	0.048	12.034	0.091	7.530	0.011	0.099	0.180
FeCrMg 9	7.797	1.162	32,070	0,837	0.006	0.078	0.051	12.039	0.088	7.540	0.013	0.097	0.180
Mean Value	7.790	1.173	32,033	0,838	0.005	0.083	0.052	12.038	0.089	7.526	0.010	0.100	0.181
Abs Std Dev.	0.017	0.006	0,053	0,004	0.001	0.003	0.002	0.008	0.002	0.007	0.002	0.001	0.003
Rel Std Dev.	0.216	0.549	0,167	0,425	15.957	4.057	4.406	0.066	1.933	0.097	22.741	1.398	1.840

- Excellent precision applying the global calibration model based on variable alpha model:
- Mg: 31.930 +/- 0.053 %
- Al: 7.790 +/- 0.017 %
- Cr: 12.038 +/- 0.008 %
- Fe: 7.526 +/- 0.007 %

S6 JAGUAR – What Are The Benefits in Metals
A New Class of High Performance Benchtop WDXRF

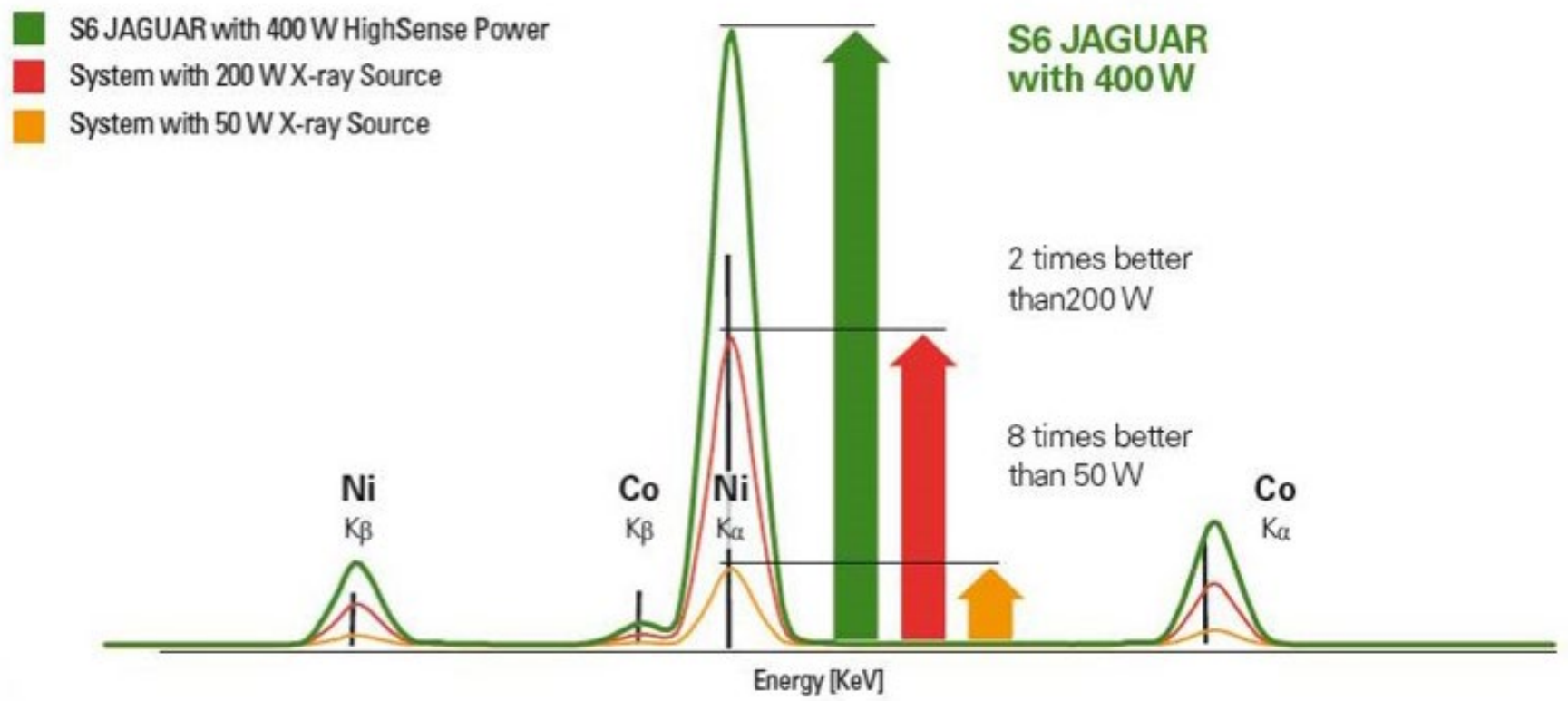
WDXRF and OES are established in central labs, but is there a place for affordable instruments?

The all-new S6 JAGUAR combines WDXRF performance with compact size:

- Analytical precision based on high sensitivity
- Excellent accuracy due to HighSense detection and state-of-the-art FP software
- Optimal versatility for wide ranges of samples, concentrations and applications
- Best ease-of-use and reliability with „plug & analyze“

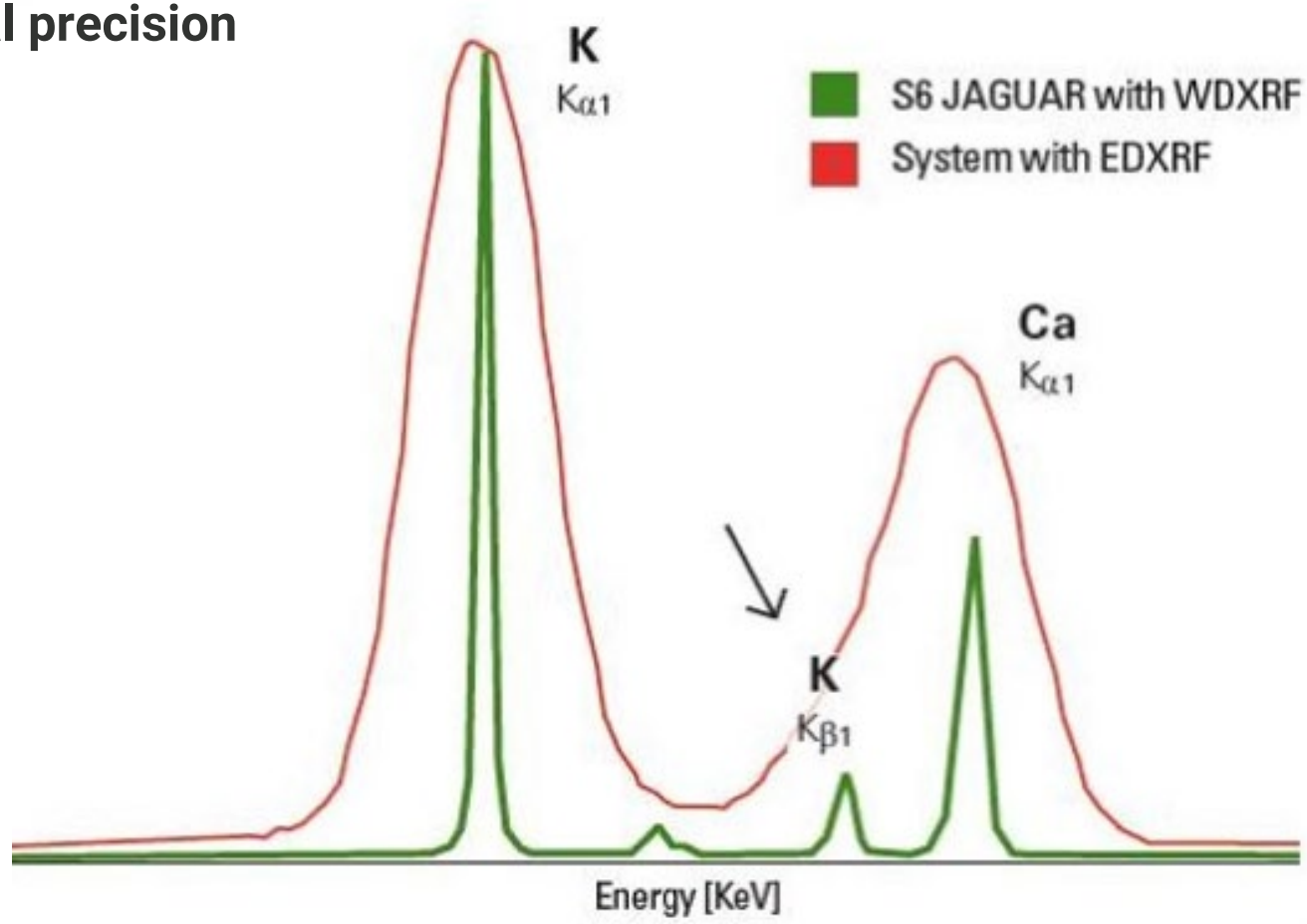
S6 JAGUAR
HighSense™: Full 400 W excitation power

S6 JAGUAR is twice as powerful as a 200 W system and 8 times more powerful than a 50 W system



S6 JAGUAR
HighSense™ Goniometer: High Resolution

The S6 JAGUAR with WDXRF HighSense Goniometer excels ED based systems in resolution and analytical precision



S6 JAGUAR
HighSense™ Goniometer: Impressive versatility

- 400 W excitation
  - 20 – 50 kV
  - 1 – 17 mA
  - Optimal settings for every single element at full power
- 5 position beam filter (optional) for improved peak-to-background ratio
- 4 sample masks (optional for different sample sizes)
- Vacuum seal for low-cost-of-operation
- Up to 4 analyzer crystals for the entire element range and specific demands
- Flow counter and HighSense XE detector for 2 Mcps count rates for high calibration ranges

“Based on the results of the S6 JAGUAR with the standardless SW SMART-QUANT WD we are able to optimize our products for the minerals industry. Longer uptime, better separation, higher output are benefits for our customers.”

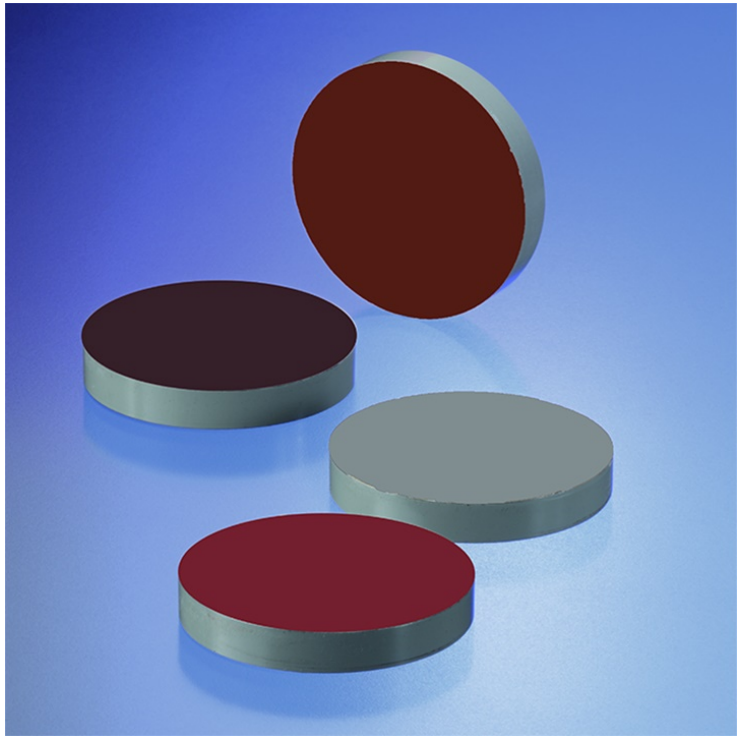
S6 JAGUAR installed to minerals and mining, incoming inspection labs:

- Smaller mining sites and mineral beneficiation w/o budget for big units, e.g., silicates

S6 JAGUAR in Minerals & Mining
Nickel Ore

- Analysis of Nickel Laterite (Low grade nickel ore) for grade control in mining operations and smelters
- Determination of valuable high Ni
- Analysis of waste rock elements:
  - Ca, Mg, Fe, Mn
- Evaluation of traces
  - Ti, Cr, Co, Cu, Zn
- Quick preparation as pressed pellets

Grade Control at mining sites

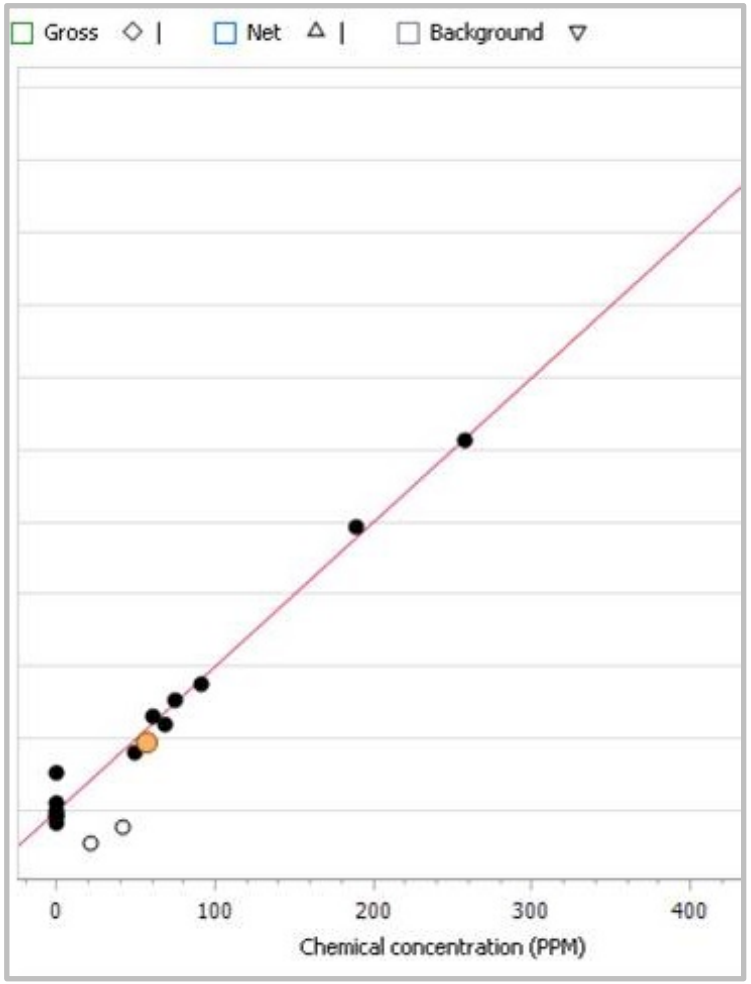


Pressed powders

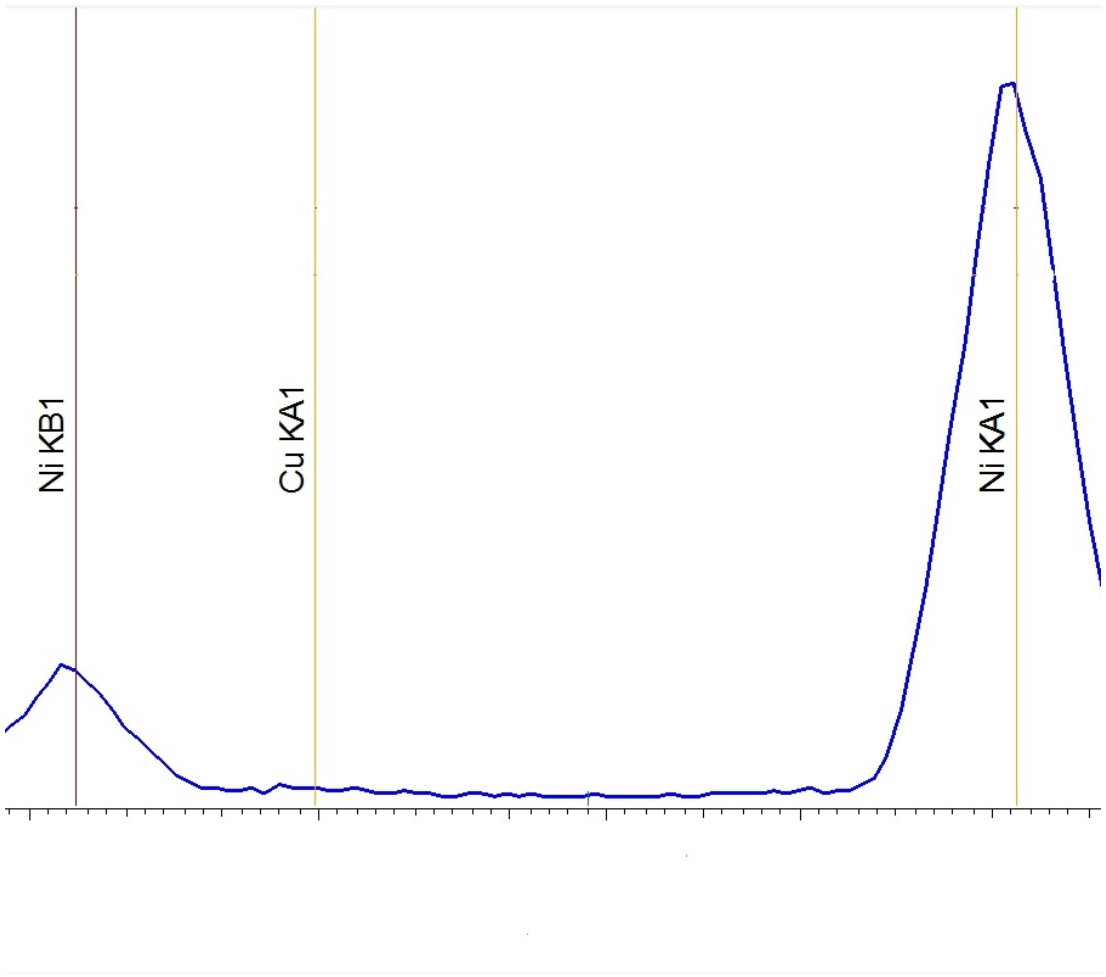
Loose powders

Fused beads

S6 JAGUAR in Minerals & Mining
Nickel Ore



- Separation of Cu Ka from Ni Kb with S6 JAGUAR‘s
- HighSense WDXRF goniometer
- Excellent Cu trace calibration: LOD 3 ppm
- Option: Even better resolution with 4th crystal: LiF220



S6 JAGUAR in Minerals & Mining
Nickel Ore

	MgO (%)	Al_2O_3 (%)	SiO_2 (%)	CaO (%)	TiO_2 (%)	Cr_2O_3 (%)	MnO (%)	Fe_2O_3 (%)	Co (PPM)	Ni (%)	Zn (PPM)
1	18.65	2.87	43.36	0.34	0.04	0.94	0.29	18.56	477	2.97	297
2	18.63	2.88	43.21	0.35	0.03	0.93	0.29	18.57	488	2.98	297
	...	...	...	...	...	...	...	...	...	...	...
19	18.74	2.91	43.62	0.35	0.04	0.93	0.29	18.48	478	2.96	301
20	18.78	2.91	43.65	0.35	0.04	0.92	0.28	18.42	476	2.95	289
Mean [%]	**18.69**	**2.90**	**43.45**	**0.35**	**0.04**	**0.93**	**0.29**	**18.53**	**477**	**2.97**	**302**
Std. Dev. [%]	**0.05**	**0.01**	**0.15**	**0.005**	**0.004**	**0.005**	**0.005**	**0.04**	**7**	**0.01**	**5**
Rel Std. Dev.	**0.24**	**0.43**	**0.34**	**1.35**	**10.80**	**0.49**	**1.76**	**0.23**	**1.47**	**0.27**	**1.65**

- Optimal analytical precision of Nickel for better grade control:
  - less than 0.3% relative @ 3 %
- Accurate analysis of elements from waste rocks
  - lower cost for mineral beneficiation

“Fluorine is a critical parameter in our foundry products. The results for Fluorine, but also other light elements, of the S6 JAGUAR are excellent and far better than any other compact spectrometer based on sensitivity and resolution we have tested”

S6 JAGUAR is successful in glass & ceramics:

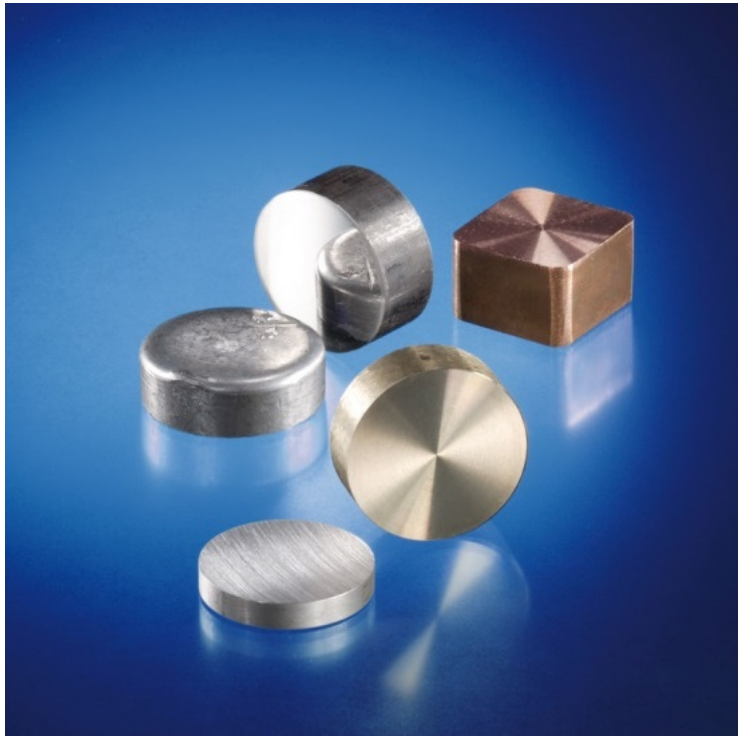
- Better light element performance than EDXRF, non-educated workforce

S6 JAGUAR in Metals & Slags
Low Alloy Steels

- Quality Control of low alloy and mild steels:
- Analysis of Fe, Ti, V, Cr, Mn, Co, Ni, Cu, Mo, W

- Impurities with negative impact on steel quaiily:
- Al, Si, P, S, Cu, As, Sb, Pb

- Incoming inspection of raw material for manufacturing
- Specifying alloy types



Huge variety of different materials

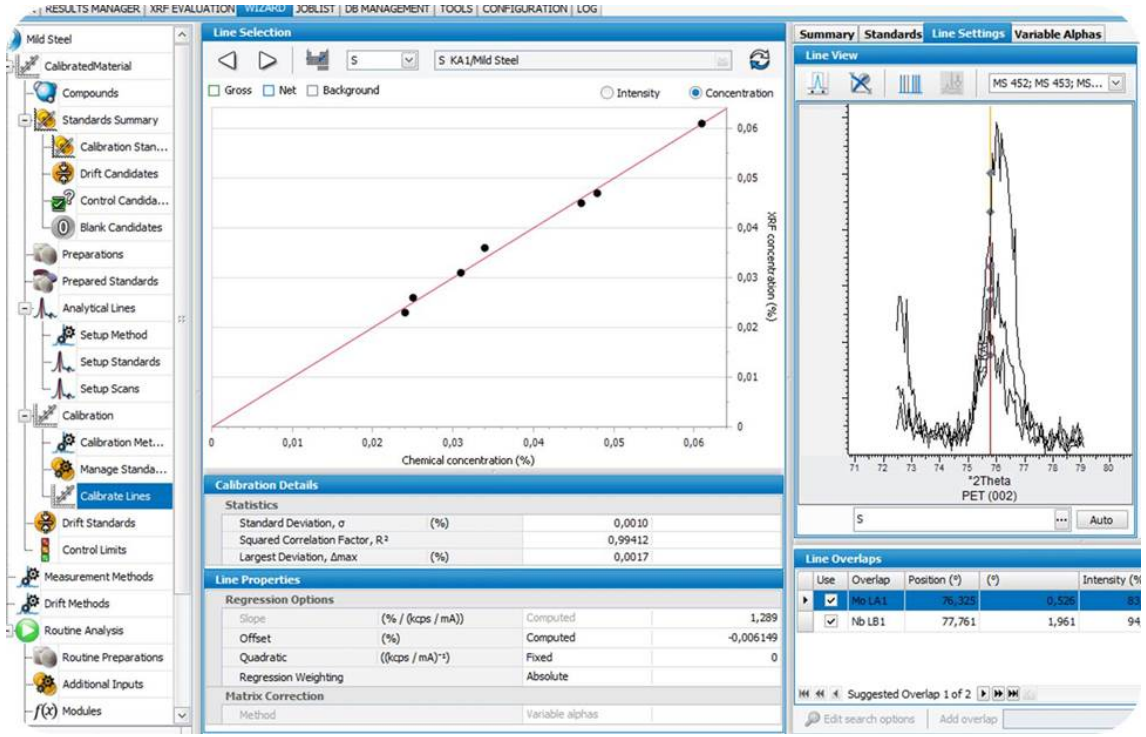
Aluminium, Iron % Steel, Tool Steel,

Copper, Brass and Bronze, Super Aloys

Quality control in Industry

S6 JAGUAR in Metals & Slags
Low Alloy Steels

Straight calibration curves
High spectral resolution



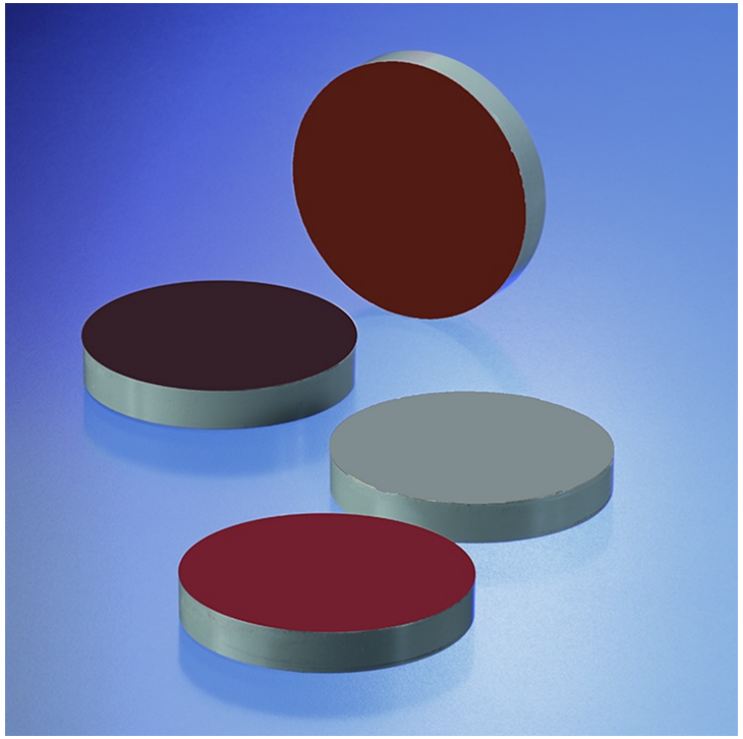
Excellent results based on advanced evaluation (Handling of line overlays and Matrix)

- Separation of S Ka from Mo La
  - with high resolution WDXRF
- Handling of line overlays and
  - FP matrix correction with
  - variable alphas

S6 JAGUAR in Minerals & Mining
Coal, Coke and Carbon

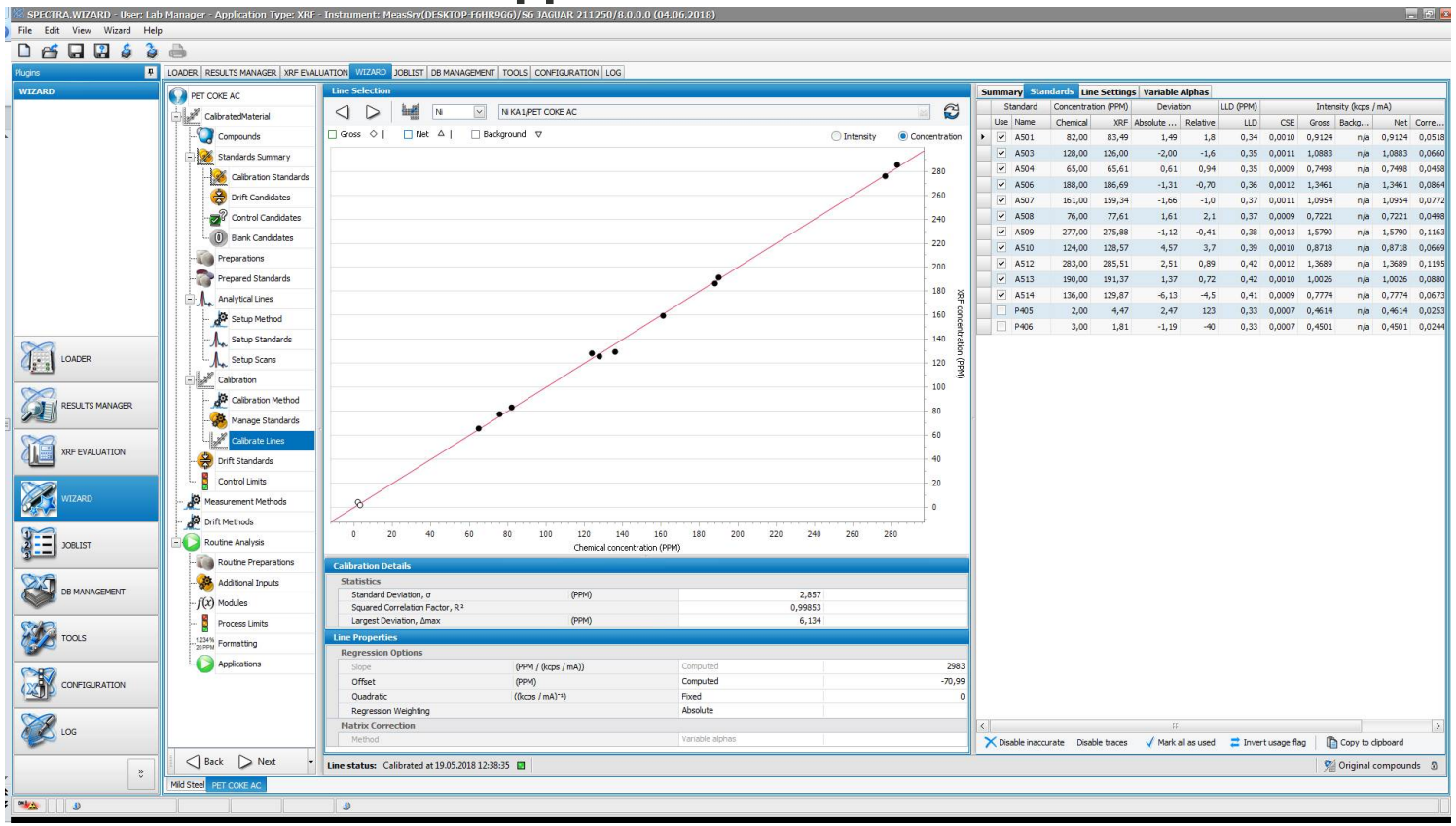
- Coal is widely used in metal making (aluminium, steel) and power generation (electricity)

- Analysis of coal, coke and carbon products is vital:
- Prevent contamination of metals (impurities)
- Inhibit corrosion (monitoring of Cl)
- Reduce environmental impact (reducing S conc.)



S6 JAGUAR in Minerals & Mining
Coal, Coke and Carbon

Nickel trace calibration:
Squared. Corr. Coeff: 0.99853 LOD: 0.4 ppm

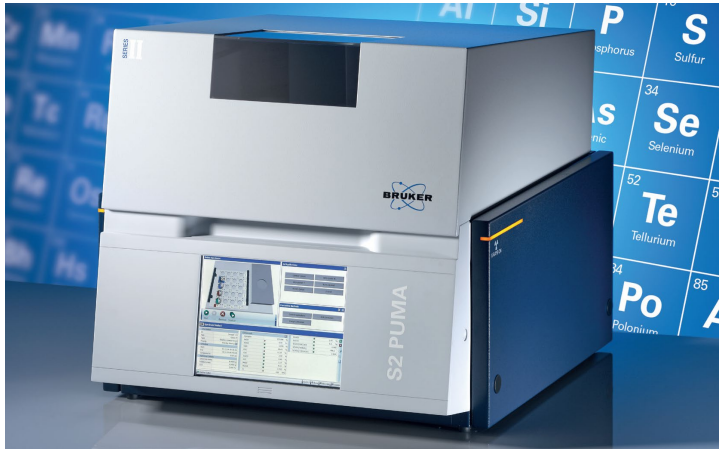


S6 JAGUAR in Minerals & Mining
Coal, Coke and Carbon

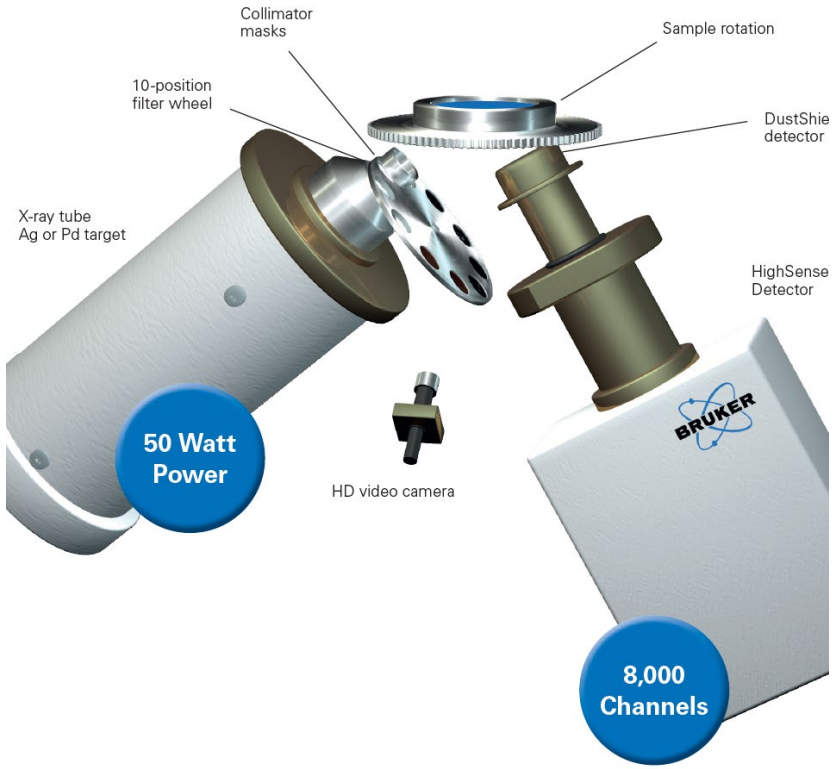
Optimal accuracy for minor elements, such as S
And for traces, e.g., Si, Cl, Fe

Element	XRF conc.	Cert. Conc.	Abs Std Dev.	Rel Std Dev.
S [%]	3.40	3.30	0.02	0.72
Ni [ppm]	128.00	124.00	4.48	3.50
Si [ppm]	24.00	28.00	1.15	4.80
Fe [ppm]	276.00	266.00	2.65	0.96
Na [ppm]	623.00	645.00	43.61	7.00
Al [ppm]	153.00	150.00	5.66	3.70
Ca [ppm]	112.00	107.00	2.35	2.10
K [ppm]	17.00	17.00	1.84	10.80
Cl [ppm]	100.00	n.a.		
Ti [ppm]	5.00	4.00	0.31	6.10
Zn [ppm]	40.00	41.00	0.10	0.25
V [ppm]	302.00	300.00	2.33	0.77

S2 PUMA Series 2
HighSense™ XP Technology



HighSense turns power into performance



Optimal excitation of the sample is ensured by:

- High power 50 Watt X-ray tube
- Up to 2 mA and 50 kV
- 10-position primary beam filter
- Direct excitation
- Large illuminated sample area of 34 mm
- Closely coupled optics

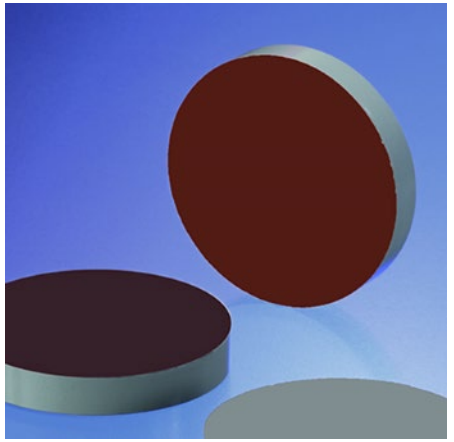
Elemental Analysis of Slags
Fast and reliable with the S2 PUMA Series 2

Sample Preparation

- Samples were crushed and the remaining metallic iron was removed with a magnet before grinding.
- Pressed to pellets by using 15 g of sample material and 1 g grinding aid.

The key advantage of pressed pellets when compared to fused beads is the fast and simple procedure.

Careful pellet preparation enables high repeatability and reliability, in particular for minor and trace element analysis.



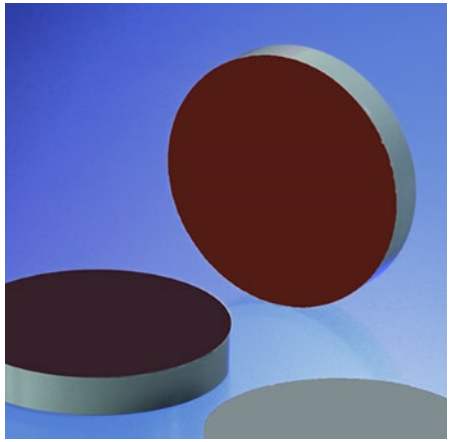
Elemental Analysis of Slags
Fast and reliable with the S2 PUMA Series 2

Analytical Conditions and Calibration

Elements	Voltage [kV]	Current [mA]	Measurement time [s]	Beam Filter	Mode
F, Mg, Al, Si, P, S, Ca, Ti, Mn, Fe	20	automatic*	120	none	Vacuum

*Current is maximized automatically for best count statistics.

- A set of 30 certified and secondary reference materials



Elemental Analysis of Slags
Fast and reliable with the S2 PUMA Series 2

EAF Slags
Compositional range

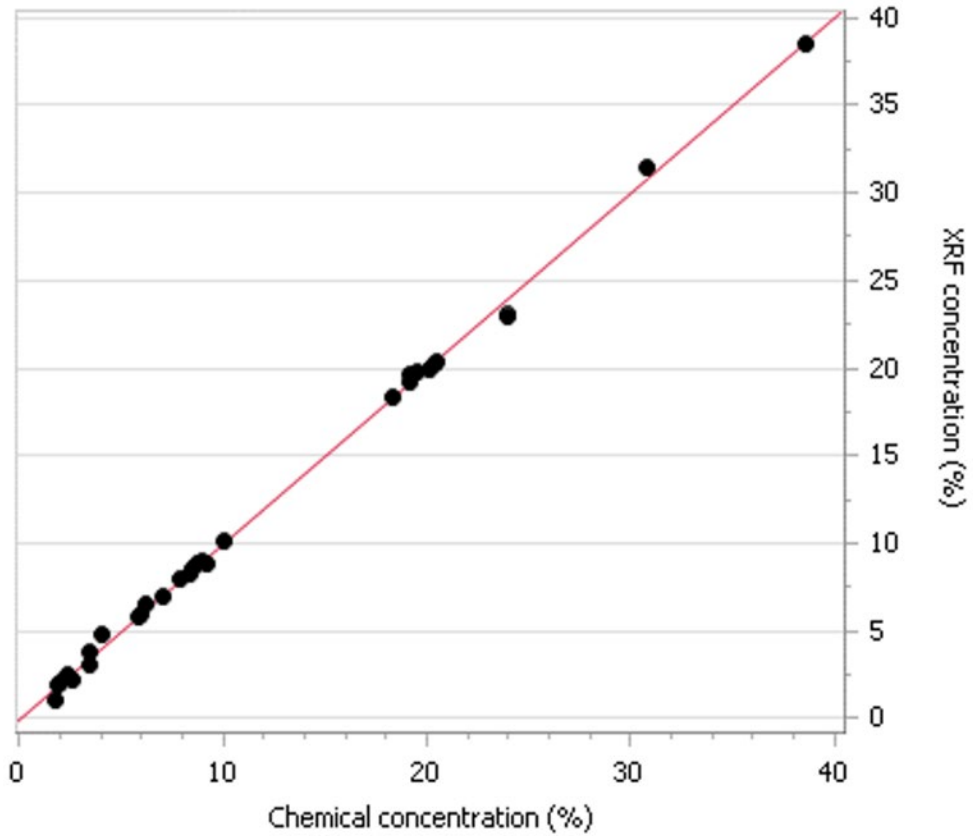
Compound	EAF Slags [wt%]
MgO	2.4 – 23.5
Al_2O_3	0.5 – 10.2
SiO_2	4.7 – 48.7
P_2O_5	0.01 – 16.7
S	0.03 – 0.2
CaO	1.2 – 42.9
TiO_2	0.15 – 2.3
Cr_2O_3	0.5 – 53.8
MnO	2.0 – 28
FeO	9.1 – 48.1

LMF Slags
Compositional range

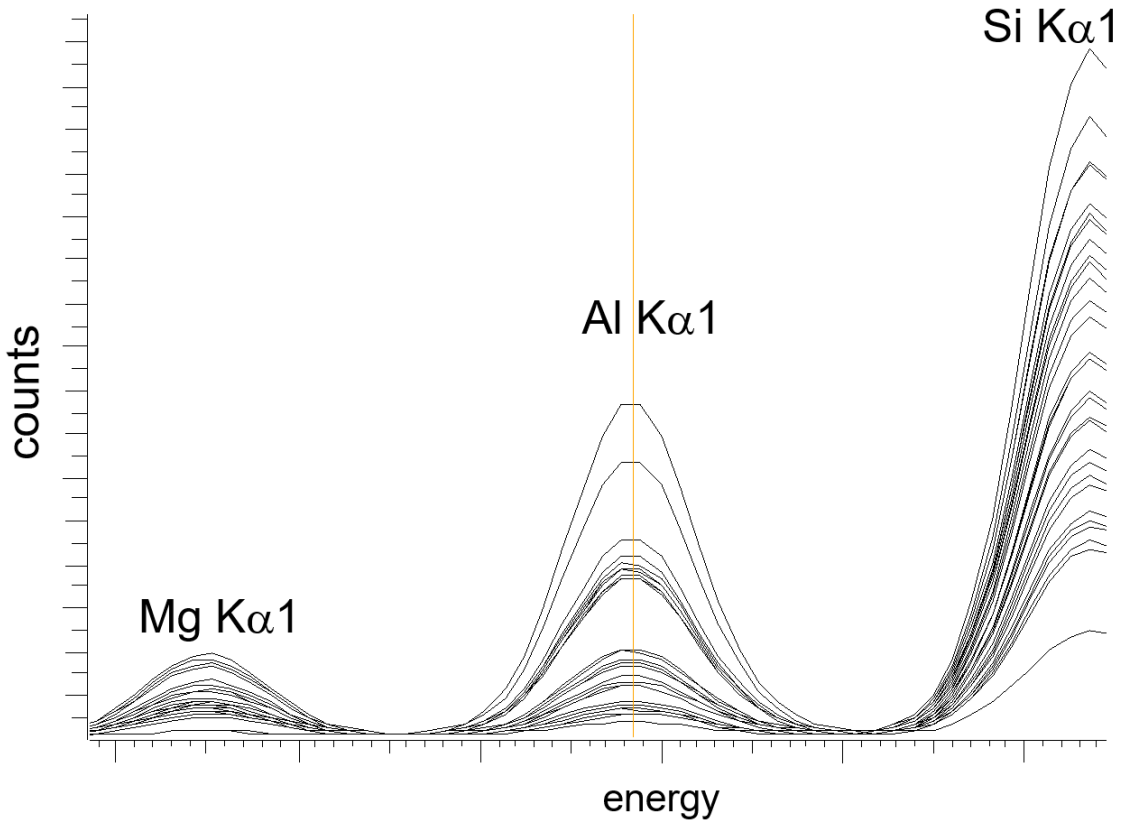
Compound	LMF Slags [wt%]
F	0.03 – 7.9
MgO	0.2 – 21.2
Al_2O_3	1.8 – 38.6
SiO_2	7.4 – 51.4
P_2O_5	0 – 1.6
S	0 – 1.2
CaO	0.6 – 60.4
TiO_2	0.01 – 2.2
MnO	0.06 – 14.9
FeO	0.1 – 17.2

Elemental Analysis of Slags
Fast and reliable with the S2 PUMA Series 2

Calibration curve for MgO



Calibration peaks for Mg Kα1, Al Kα1 and Si Kα1



Elemental Analysis of Slags
Fast and reliable with the S2 PUMA Series 2

[wt.%]	MgO	Al_2O_3	SiO_2	P_2O_5	S	CaO	TiO_2	Cr_2O_3	MnO	FeO	V-Ratio	B3-Ratio
Rep-1	13.53	6.36	14.20	0.240	0.130	26.97	0.36	2.16	5.59	29.84	1.90	1.29
Rep-2	13.61	6.38	14.20	0.240	0.130	26.97	0.37	2.16	5.59	29.84	1.90	1.29
Rep-3	13.63	6.37	14.27	0.240	0.130	27.01	0.37	2.16	5.59	29.78	1.89	1.29
Rep-4	13.56	6.36	14.21	0.240	0.120	27.00	0.37	2.17	5.58	29.85	1.90	1.29
Rep-5	13.61	6.36	14.26	0.240	0.120	27.01	0.37	2.16	5.56	29.79	1.89	1.29
Rep-6	13.55	6.38	14.18	0.240	0.130	27.00	0.37	2.17	5.58	29.85	1.90	1.29
Rep-7	13.56	6.38	14.27	0.230	0.130	27.01	0.37	2.16	5.58	29.79	1.89	1.28
Rep-8	13.55	6.37	14.24	0.240	0.130	27.01	0.37	2.17	5.60	29.81	1.90	1.29
Rep-9	13.69	6.39	14.24	0.240	0.120	27.04	0.37	2.16	5.56	29.76	1.90	1.29
Rep-10-101	…	…	…	…	…	…	…	…	…	…	…	…
Rep-102	13.75	6.54	14.41	0.240	0.130	27.03	0.37	2.17	5.56	29.62	1.88	1.26
Average [wt.%]	13.69	6.44	14.34	0.244	0.126	27.02	0.37	2.17	5.57	29.70	1.89	1.28
Abs. Std. Dev. [wt.%]	0.10	0.05	0.07	0.005	0.005	0.03	<0.01	0.01	0.01	0.07	0.01	0.01
Rel. Std. Dev. [%]	0.7	0.7	0.5	2.3	3.8	0.1	0.9	0.3	0.2	0.2	0.5	0.5
Certified	13.40	6.58	14.24	0.250	0.13	26.66	0.35	2.17	5.63	29.49	1.87	1.26
Difference	0.29	0.14	0.10	0.006	0.004	0.36	0.02	<0.01	0.06	0.21	0.02	0.02

Viscosity (V) Ratio: CaO / SiO_2; Basicity (B3) Ratio = $CaO / (SiO_2 + Al_2O_3 + TiO_2)$

Repetition test (samples were loaded and unloaded between the analyses)

- Results for EAF slag
- Vicosity (V) and Basicity (B) are critical monitoring parameters

- Excellent precision and accuracy for Slag analysis thanks to the HighSense Technology of the S2 PUMA Series 2

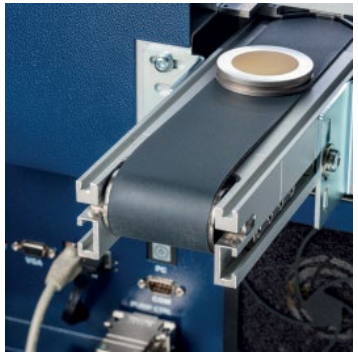
S2 PUMA Series 2
Taylor-made Solution for all Applications

The XY Autochanger – Add Efficiency and Flexibility to your lab

- EasyLoad™ XY tray with 20 sample positions
- 2 fixed positions for QC and Drift samples
- Load mixed batches
- New samples can be loaded at any time

The XY Automation – No Compromise on Productivity

- Samples are fed directly, via robot or belt, from the automated sample preparation system
- LIMS-compatible: The AXSCOM interface connects to the process control software

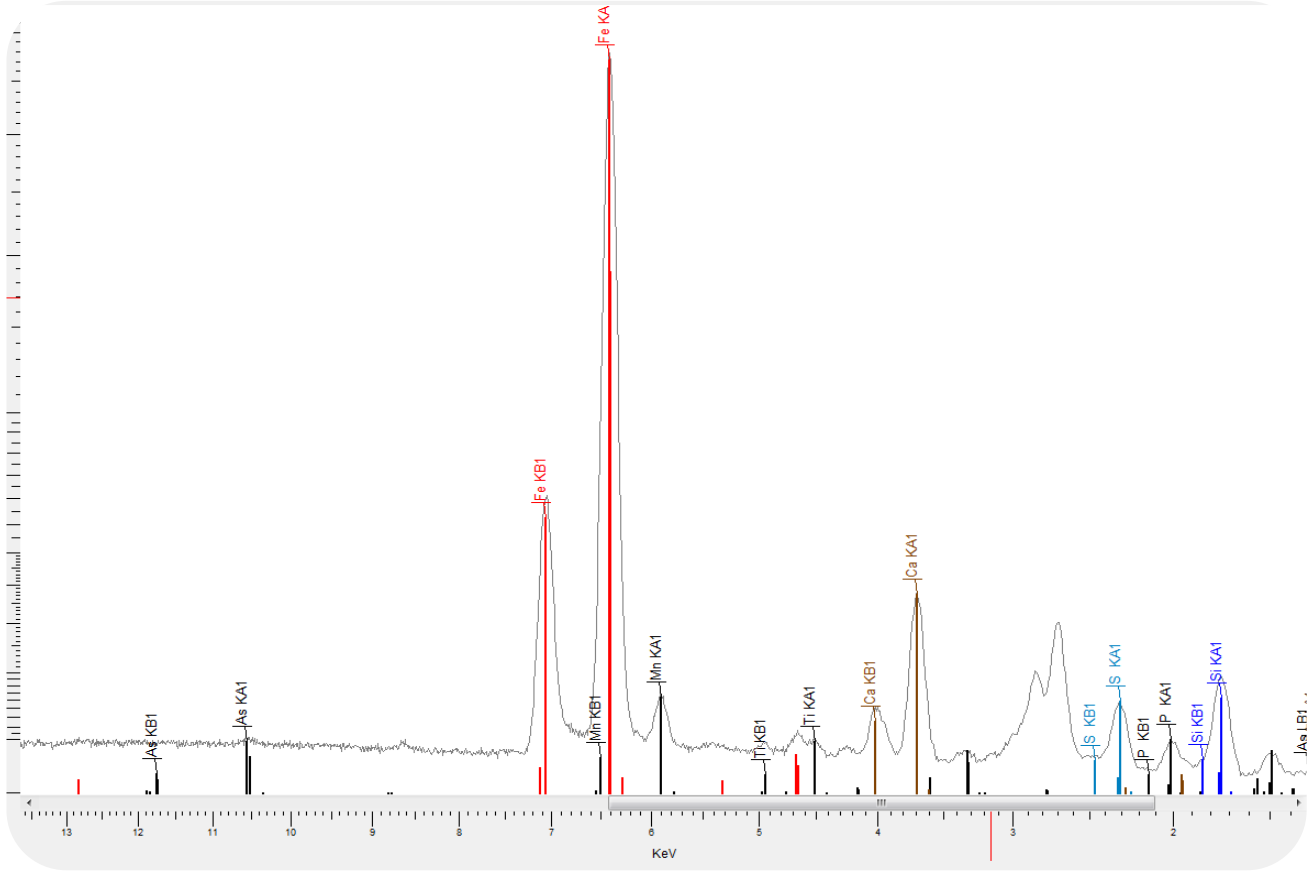
Iron Ore - Rapid Grade Control
When speed and sample throughput is vital!

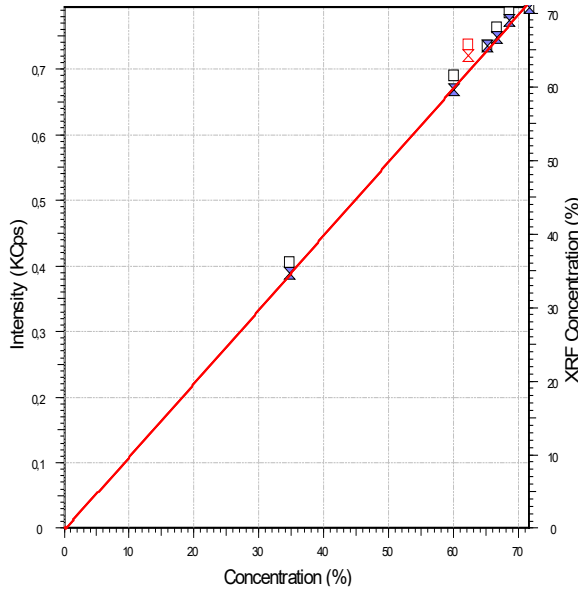
- Rapid control of element concentrations for 21 elements (ISO 9516)
- Quality check
  - at the mining site
  - at shipping stations

Narrow concentration range and similar matrix allows pressed pellet preparation

- Quickest sample preparation
- Crushing, milling, pressing with cellulose as binder (15 g sample plus 2 g binder)
- Maximum 60 s measurement time
- Simple automation path, high sample throughput, easy operation, fast feedback

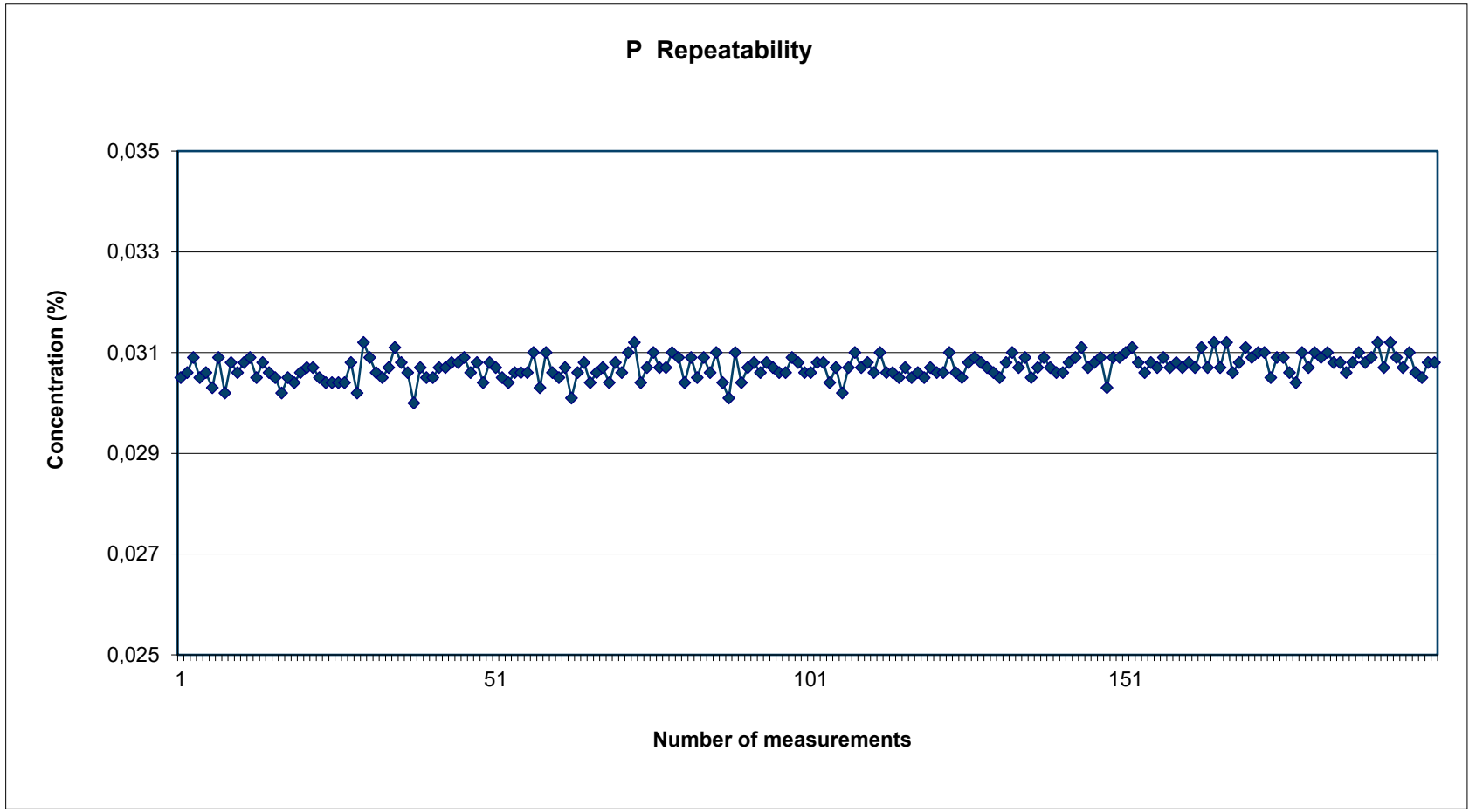
Spectrum Iron Ore - Dual Mode Data Acquisition S8 LION





- Parallel measurement of SEC (WDX) and Multi Element Channel
- Identification of trace elements, internal backup (second information source)

SEC: Precision Test on P over One Week
S8 LION



- 200 measurements: 0.031 % +/- 0.001 % P

WD Element Channels
S8 LION

Up to 24 WD element channels with

Dual monochromators (2 channels per port)

For fast process control applications in metals:

- Integrated steel mills
- Copper base alloys

For high throughput applications in minerals and mining

- Iron ore acc. to ISO 9516
- Base metal ores acc. to ISO 18855

Steel
S8 LION

- Elements analyzed: Al, Si, P, S, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Zr, Nb, Mo, Sn, Sb, Ta, W, Pb, Bi (24)
- Measurement time = 40 s, plus handling
- Dual Mode acquisition with Multielement Channel

Precision Test for Steel
40 s Measurement Time Dual Mode

Element line	Region [%]	RSM [%]	LLD [PPM]	Comment
Si KA1	0 – 4.1	0.056	350	Multielement Channel
P KA1	0 – 0.1	0.004	2	
Ti KA1	0 – 2.2	0.08	3	
V KA1	0 – 9.7	0.15	3	
Cr KA1	0 – 30	0.07	2	
Mn KA1	0 – 2	0.01	3	Two ranges
	0 – 20	0.07		
Fe KA1	50 – 100	0.8		Multielement Channel
Co KA1	0 – 2	0.02	2	Two ranges
	0 -10	0.02		
Ni KA1	0 – 6,2	0.008	2	Two ranges
	0 – 35	0.06		

Precision Test for Steel
40 s Measurement Time Dual Mode

Element line	Region [%]	RSM [%]	LLD [PPM]	Comment
Cu KA1	0 – 3.3	0.02	2	
Zn KA1	0 – 0.1	0.004	16	Multielement Channel
As KB1	0 – 0.1	0.002	6	
Zr KA1	0 – 0.1	0.007	10	
Nb KA1	0 – 1.1	0.01	75	Multielement Channel
Mo KA1	0 – 0.3	0.003	1	Two ranges
	0 – 3.2	0.013		
Sn KA1	0 – 0.2	0.002	4	
Ta LB1	0 – 0.3	0.008	12	
W LA1	0 – 11	0.06	6	Two ranges
	0 – 20	0.10		
Pb LA1	0 – 1.1	0.009	3	

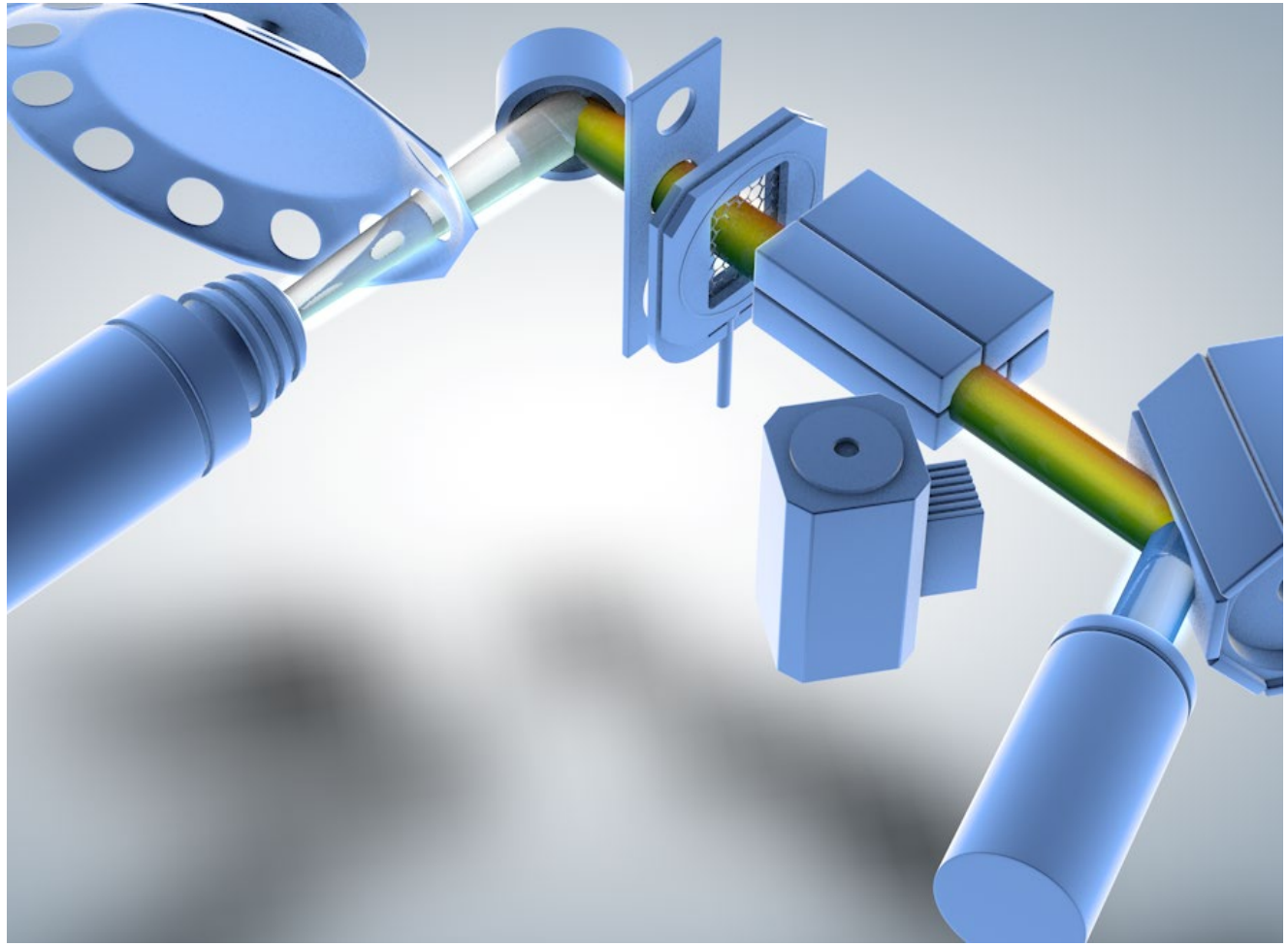
Analytical Flexibility paired with high performance for all applications in the central lab and development

- S8 TIGER Series 2 sequential WDXRF with HighSense Technology for
  - Ultimate detection limits
  - Optimal sensitivity
  - Best precision
  - Enhanced light element sensitivity
  - Shortest measurement times

Fulfills demands for low detection limits and precision for routine applications, but also serves for R&D -> layer thickness, small spot analysis or metal powder for additive manufacturing applications

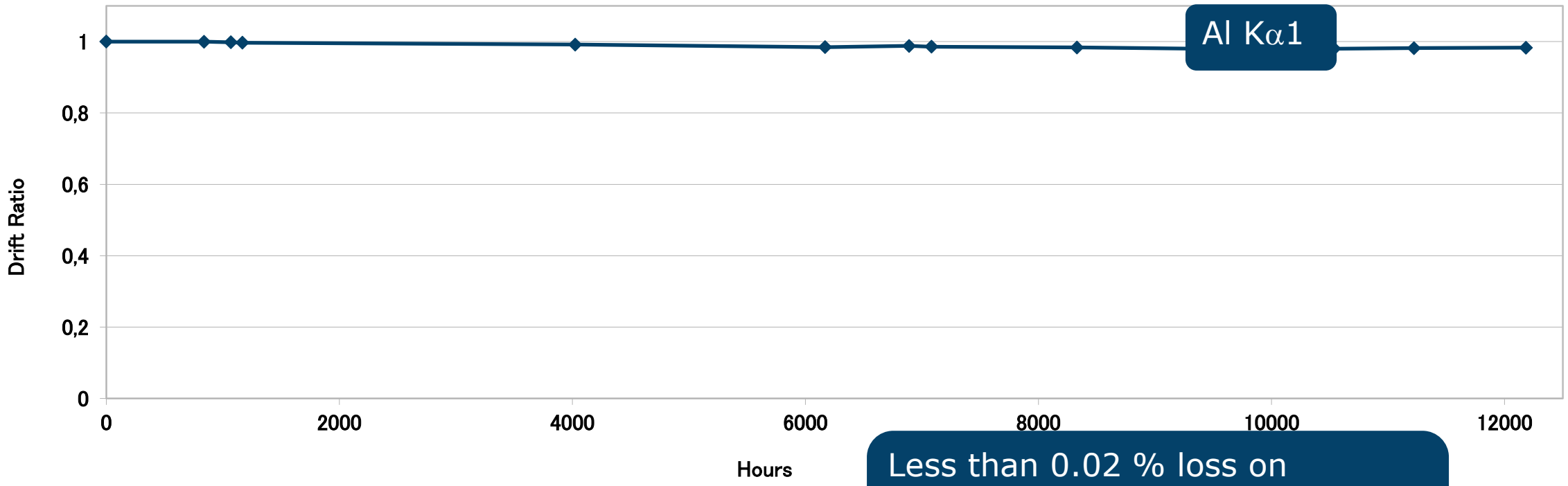
Optimal Settings for each element and concentration range by sequential WDXRF

- Optimal detection for every element
- from Be – Am, ppm to 100%:
- 20 – 60 kV, 5 – 170 mA for 4 kW
- 10 primary beam filters
- 8 collimator masks
- HighSense vacuum seal
- 8 analyzer crystals out of 18 available
- Two detectors with HighSense counting electronics



S8 TIGER
The Zero Drift Instrument

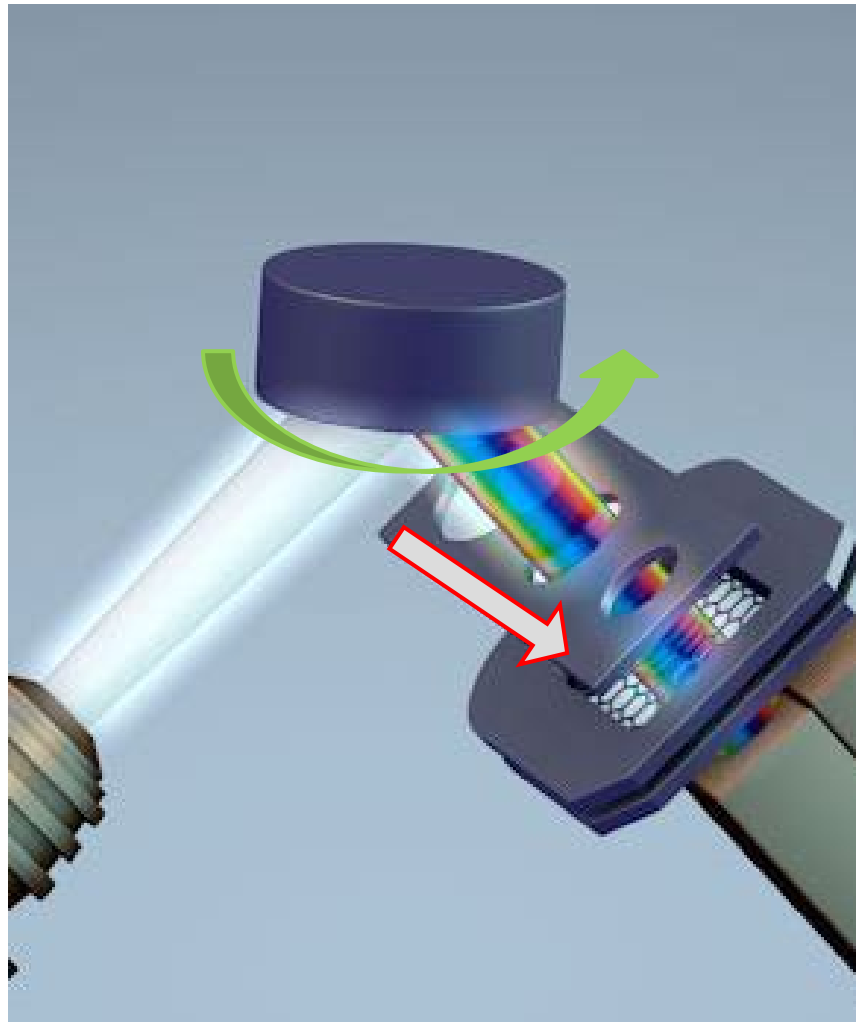
The S8 TIGER Series 2 provides high precision due to the precise performance of all involved parts: Tube, detectors, generator, crystals, drives



Less than 0.02 % loss on intensity Rh Lα within one year (Tube, Crystals, Detector)

Mapping and small spot analysis in bulk-WDXRF systems

- Mapping is realized as P, Φ stage with a combination of high precision rotation of the sample and movement of the mask changer
- Other solution:
  - Movement of sample in X,Y direction
    - Less mechanical precision, bulk analysis is suffering
    - Mechanism tends to failures and higher service costs
- Using WDXRF goniometer:
  - Maintaining high resolution
  - Optimized measurement settings for each particular element



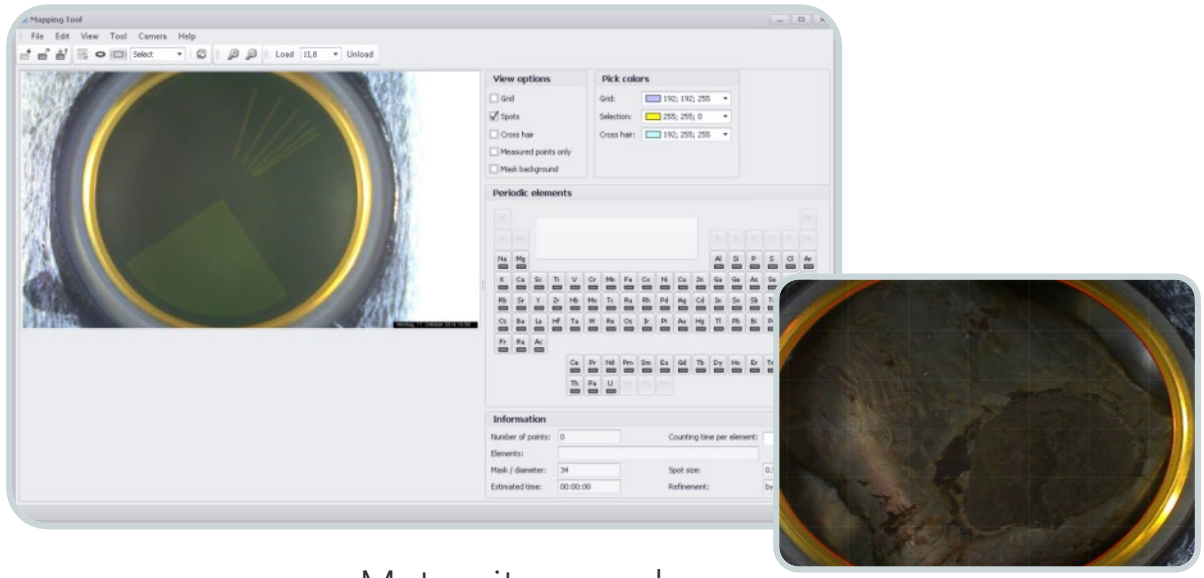
The all-new S8 TIGER Series 2 XRF² – Metorite „Canyon Diablo“

- Meteorites are delivering information from outer space
  - Material science
  - Cosmic science
  - Information on formation of Earth and Space

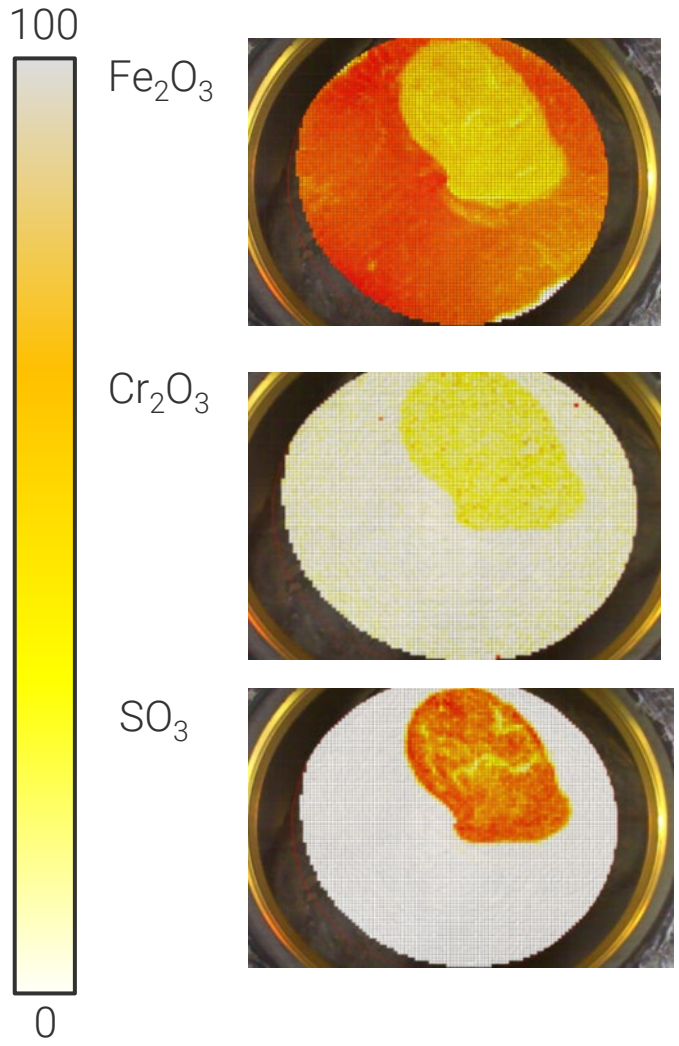
- Iron-Nickel meteorite „Canyon Diablo“ from the Barringer crater in Arizona, US
- Part with inclusion

The all-new S8 TIGER Series 2 XRF² – Metorite „Canyon Diablo“

- Mapping of points, lines, several areas and complete sample
- Mapping client for easy setup
- Standardless evaluation and user calibration



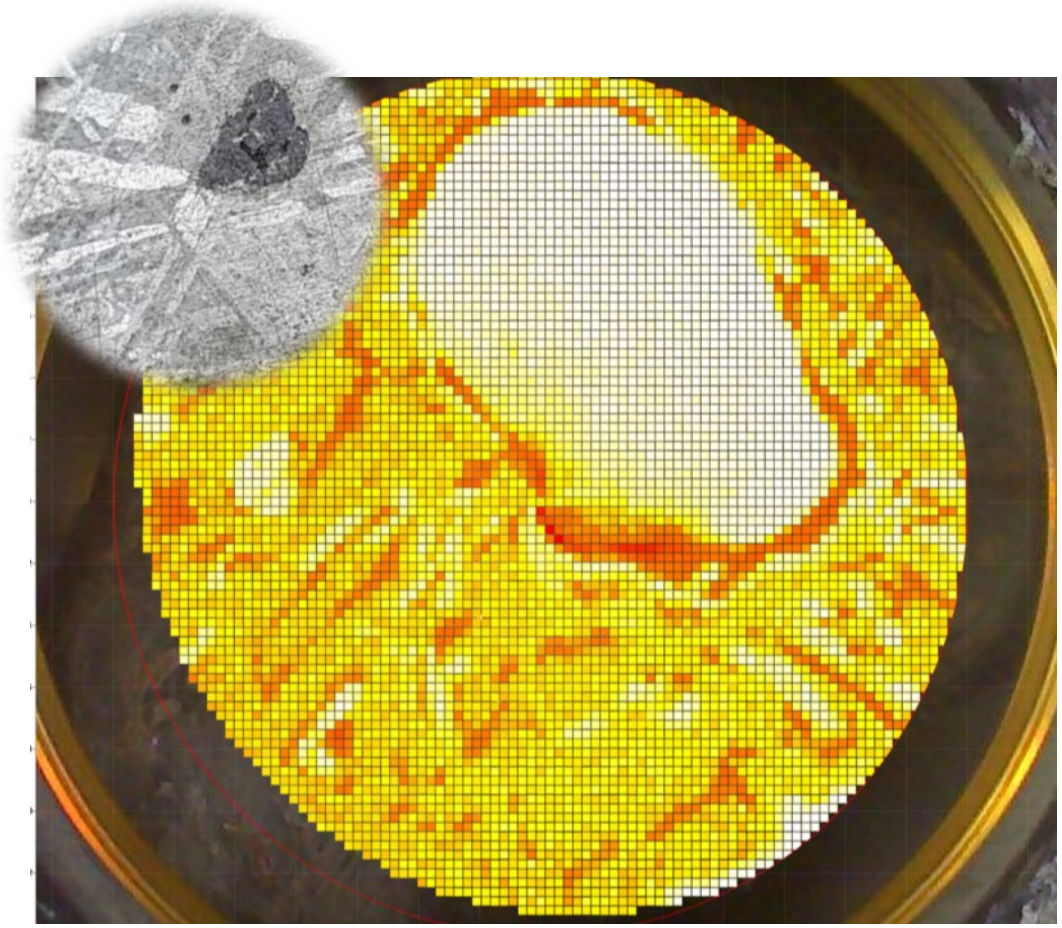
Meteorite sample
Canyon Diablo, Arizona



The all-new S8 TIGER Series 2 XRF² – Metorite „Canyon Diablo“

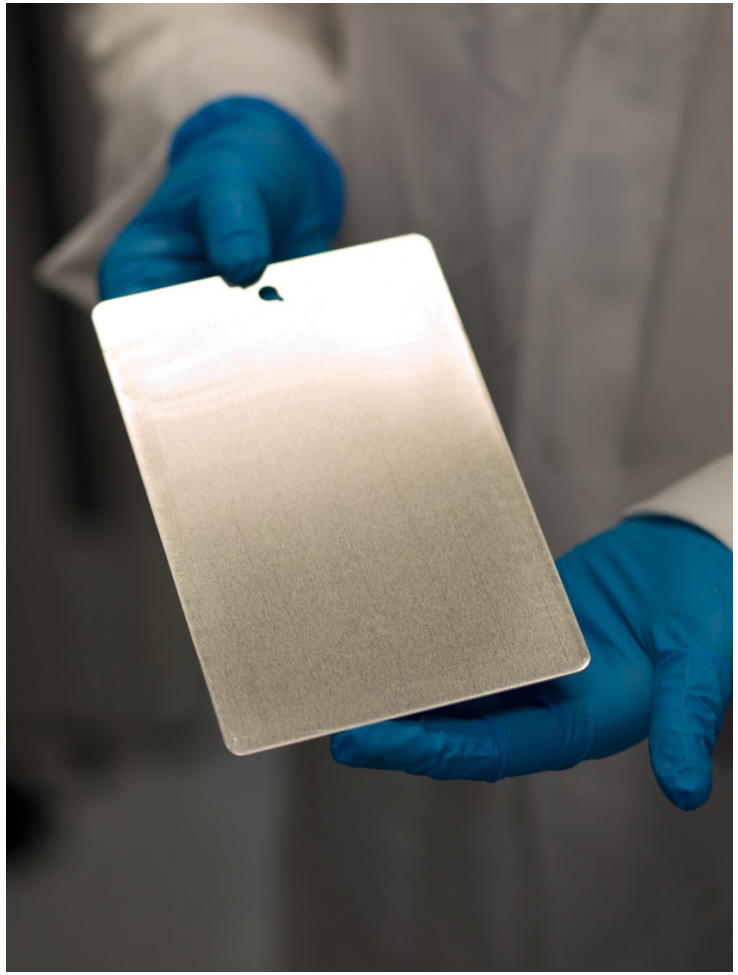
- Nickel distribution in Meteorite Canycon Diablo“, Arizona, US, above: example for Widmannstätten figures

- High spatial resolution
- High sensitivity to resolve different Nickel phases kamacite and taenite
- Structures only seen by leaching experiments (destructive) or
- S8 TIGER Series 2 (non-destructive)



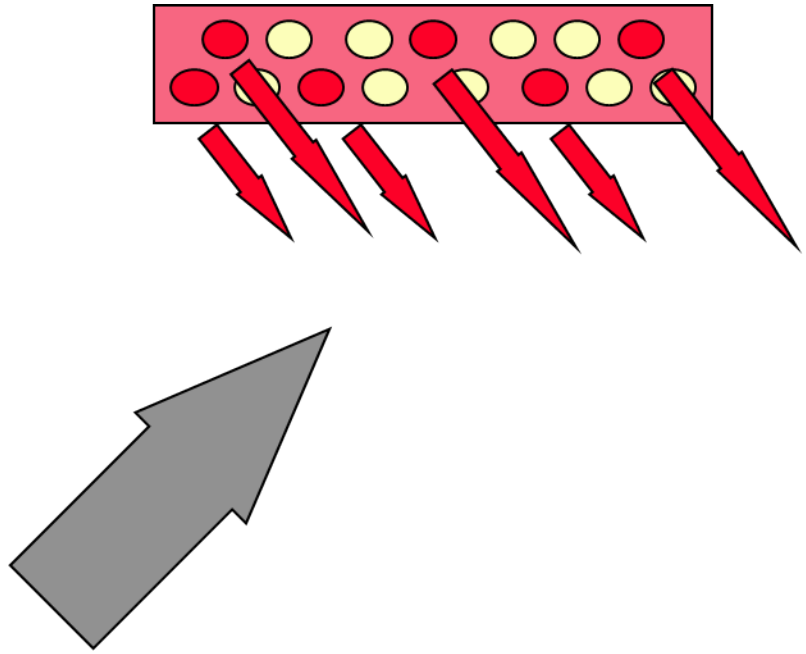
X-ray fluorescence has multiple layers at-line, on-line and inline

- Commercial value of accurate process control is obvious
  - Expensive chemicals and/or processing steps to built
  - Thin
  - Stable
  - Complete
  - Single or multiple layers
- Chemical analytical methods (ICP,AAS) depend on leaching:
  - Non complete
  - Time consuming (delayed feedback)
- Physical analytical methods (electro magnetic, beta backscatter, SEM)
  - Not general, not specific
  - Not for multiple layers
  - Non-routine methods (SEM)



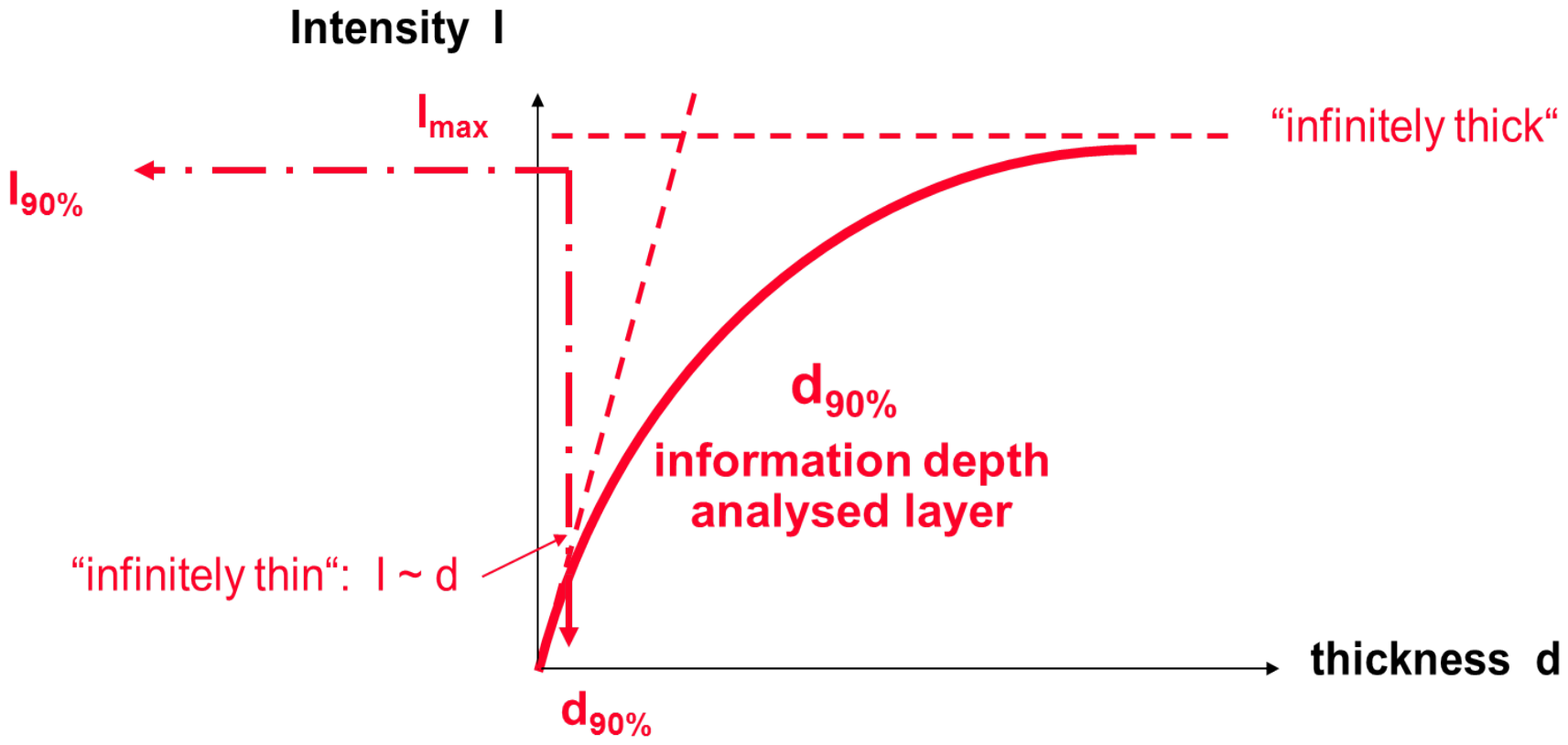
X-ray Fluorescence Analysis “Infinitely Thin Sample”

- the absorption of the exciting and the fluorescence radiation can be neglected
- the intensity of a characteristic line is proportional
- to the number of atoms of this element per area
- to the mass per area (g/cm²) of this element
- to the thickness (for constant density and composition)



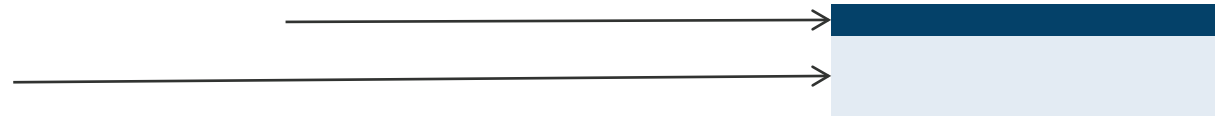
X-ray Fluorescence Analysis
Influence of Sample Thickness

- Information Depth / Analyzed layer with XRF based on 90% saturation



S8 TIGER
Thin layer analysis of Ferroelectrics

- $BaTiO_3$-$CoFe_2O_4$ (BTCF)
- $SrTiO_3$ (STO)
- Multi-ferroic sample was measured and evaluated.



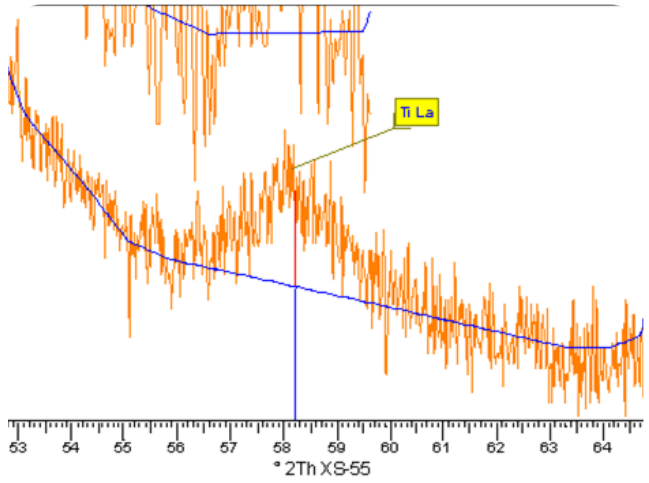
- The layer structure was BTCF on STO substrate.

S8 TIGER
Thin layer analysis of Ferroelectrics

- Measurement conditions (5mm diameter)

BaTiCoFe										
Line Name	tube KV	Filter	Collimator	Crystal	Detector	2Th-Peak	2Th-Bkg	LLD	ULD	M.T.(s)
Ti KA1	50	None	0.23	LiF200	Gas	86.157*	84.600; 89.800	50	150	300
Ti LA1	30	None	0.23	XS-55	Gas	58.194*	55.000; 62.500	50	150	1000
Fe KA1	60	None	0.23	LiF220	Sci.	85.753*	87.5	50	150	300
Co KA1	60	None	0.23	LiF220	Sci.	77.900*	79.5	60	140	300
Ba LB1	50	None	0.23	LiF200	Gas	79.273*	75.000; 83.000	50	150	300

- Ti is present in both substrate and layer. Therefore, Ti K lines are difficult to use for calculating Ti concentration and thickness.
- Ti La is used for this calculation, but very low intensity with small aperture size (5mm) with conventional WDXRF instruments



S8 TIGER
Thin layer analysis of Ferroelectrics

- Calculated composition and layer thickness by Ti Ka line

  → yields false results for Ba:Ti

Ti K lines			
Thickness	0.37um	5.189	g/cm3
Composition			
Ba:Ti	1.00:1.54		
Co:Fe	1.00:2.03		

- Calculated composition and layer thickness by Ti La line

  → yields accurate results!

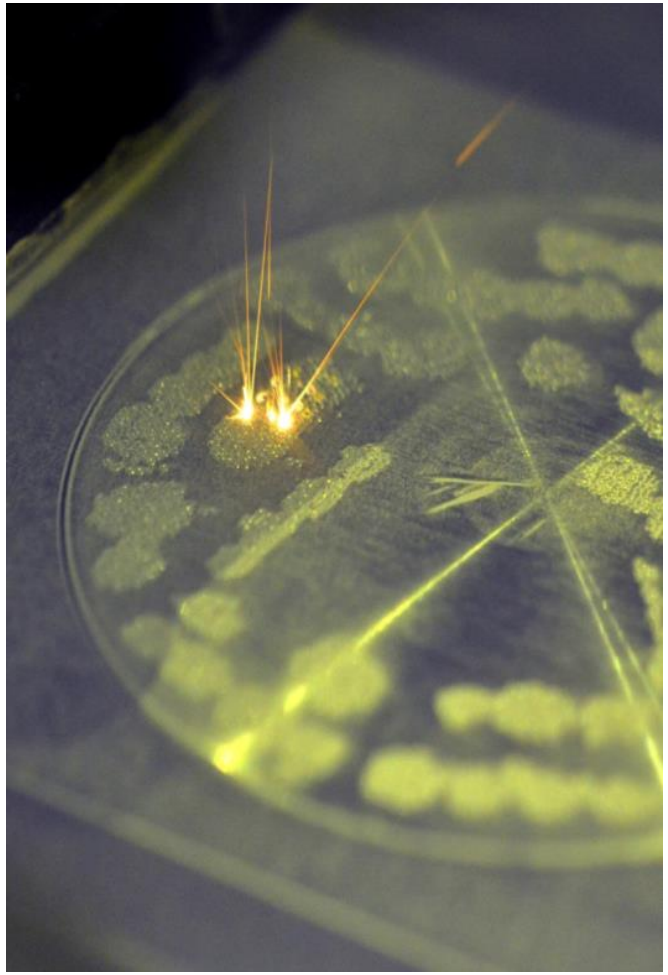
Ti La line			
Thickness	0.29um	5.31	g/cm3
Composition			
Ba:Ti	1.00:0.92		
Co:Fe	1.00:2.05		

New applications
XRF + OES + CGA

- 3D-printing (additive manufacturing) will revolutionize the industry:
  - bionic thin structures with extreme stability and low weight
    → aircrafts, cars, construction
  - Complex parts in one manufacturing step
    → jet turbine wheel
  - Print-on-demand, when needed and where needed (worldwide)
    → replace spare part supply chain and make transportation obsolete
  - Medical implants and jewelry
- replacing casting, cutting, milling

3D – printing (additive manufacturing) XRF applications

- Additive Manufacturing:
  - A laser is melting specific spots in a thin layer out of metal powder
  - After hardening a new layer of powder is added on top
  - Again, A laser melts up specific spots
  - again, again,…
- Requirements for the metal powder:
  - Homogenous grain size
  - High purity
  - Absolute alloy grades



XRF is the ideal method

- Direct analysis of metal powders

S8 TIGER Series 2 is the perfect instrument with:

- Direct loading
- Low helium consumption for loose powders
- XRF^2 mapping for failure analysis

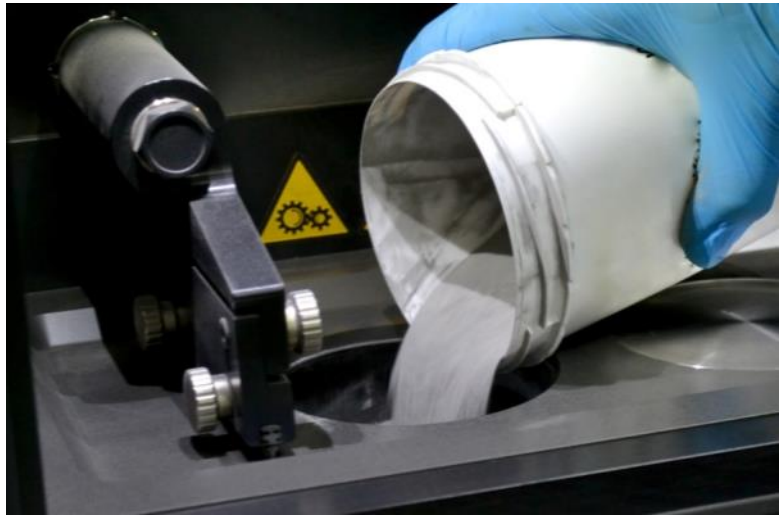
3D – printing (additive manufacturing) XRF applications: Preparation

Element	Powder	Pellet
	[%]	[%]
Fe	74.90	74.83
Cr	16.29	16.32
Ni	3.98	4.03
Cu	3.66	3.66
Si	0.53	0.52
Mn	0.34	0.33
Nb	0.303	0.297
V	0.017	0.017

- Quick material screening with QUANT-EXPRESS
- as powder or pellet

3D – printing (additive manufacturing) XRF applications: METAL-QUANT

Formula	Concentration
Al	0,31%
Si	0,02%
P	0,00%
S	0,00%
Ti	0,98%
V	---
Cr	17,19%
Mn	0,01%
Fe	18,56%
Co	0,02%
Ni	54,01%
Cu	0,00%
Zn	---
As	---
Zr	---
Nb	5,44%
Mo	3,44%
Sn	---
Sb	---
Ta	0,02%
W	---
Pb	---



- Quality control and incoming inspection
  - Alloy grades
  - Impurities/contaminations
- METAL-QUANT customized to 5:13 min. measurement time max.

Metal Applications with S8 TIGER Series 2 Flexibility and Performance on Demand

- Quality control at production side
- Incoming material inspection at buyers side
- Failure analysis with mapping in parts

- S8 TIGER Series 2:
  - HighSense technology for short measurement times and high analytical precision
  - XRF² mapping for failure analysis with 300 µm spot size
  - WDXRF high spectral resolution, e.g. resolving line overlays
    - Mo Lα on S Kα
    - Fe Kβ on Co Kα
  - Lowest helium consumption for loose powders
  - SampleCare when running loose powders
  - METAL-QUANT for excellent results
  - QUANT-EXPRESS for quick screening

Powerful Benchtop units for dedicated tasks

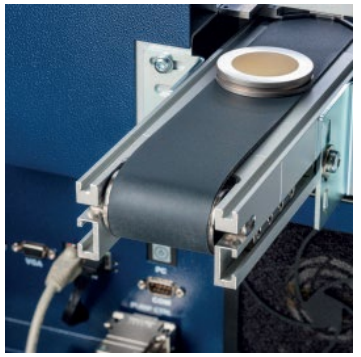
S6 JAGUAR

- High precision analysis of metals, alloys and slags
- Excellent performance even for light elements

S2 PUMA Series

- Optimal backup unit
- Great performance for slag analysis
- Excellent choice for smaller mining sites

S6 JAGUAR
Benchtop WDXRF



S2 PUMA Series 2
EDXRF

Launch of the Q4 POLO
The „Little Giant“ in Metals Analysis

Compact spark spectrometer with superior performance

- High precision metal analysis
- Incredible long-term stability
- Full elemental range, covering a multitude of applications
- Low cost of ownership
- Small footprint, high uptime
- Ready-to-analyze

Q4 POLO – compact and tidy all-in-one workstation when combined with optional multi purpose tray and notebook PC

Tray serving as secure storage for samples

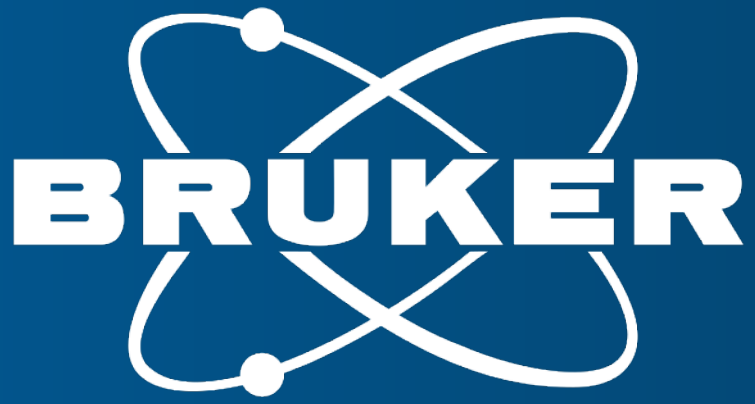
Any Questions?

Learn more about Elemental Analysis in the Food Industry: Where to use X-ray Fluorescence (XRF)

In our 29.9.2021 webinar – Sign Up Now!

Thank you!

Kai Behrens, Frank Portala, Adrian Fiege



Innovation with Integrity