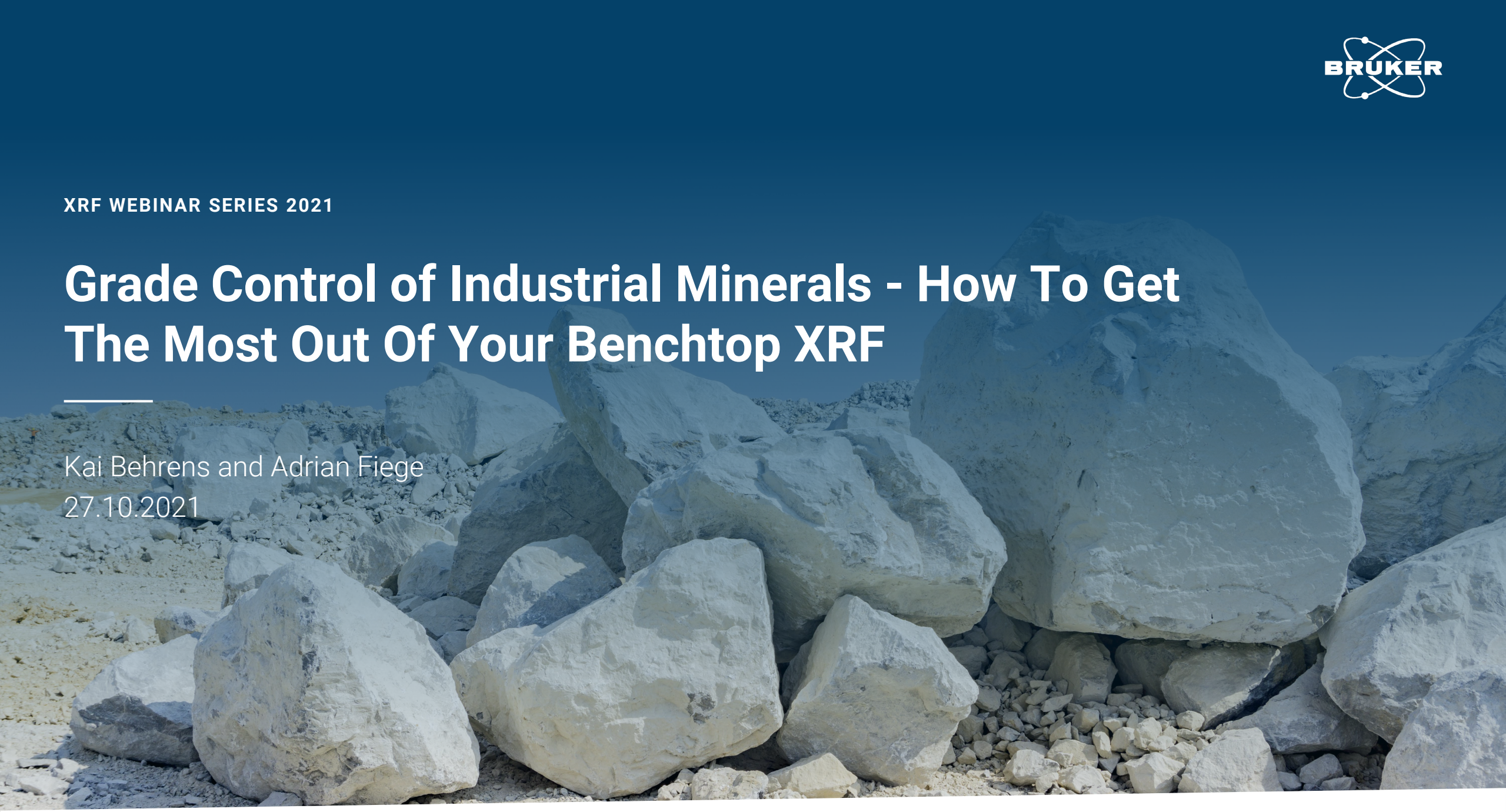
XRF WEBINAR SERIES 2021

Grade Control of Industrial Minerals - How To Get The Most Out Of Your Benchtop XRF



Kai Behrens and Adrian Fiege

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Grade Control of Industrial Minerals - How To Get The Most Out Of Your Benchtop XRF

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Ph D, Analytical Chemistry, Hamburg

2001 Method Development Bruker AXS

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2019 Product Management Bruker AXS

Product Management XRF

Karlsruhe, Germany

Outline

New XRF Technology – Advances in Components for Better Data

The production of most materials starts with or involves industrial minerals from ceramics and glass to metals and polymers.

XRF Spectrometry

- Established analytical tool for process and quality control in the industry
- Meet changing quality requirements based on excellent data quality
- Analysis defines material value based on grade control (mining)
- Analysis adds value and can often determine the suitability of a product for specific applications (high tech functional material)
- Each application defines the final quality criteria regarding **elemental and phase composition, layer thickness, impurities**, grain size and other chemical and structural properties

Mature Technology in XRF?
Are there any Changes to Address Future Requirements

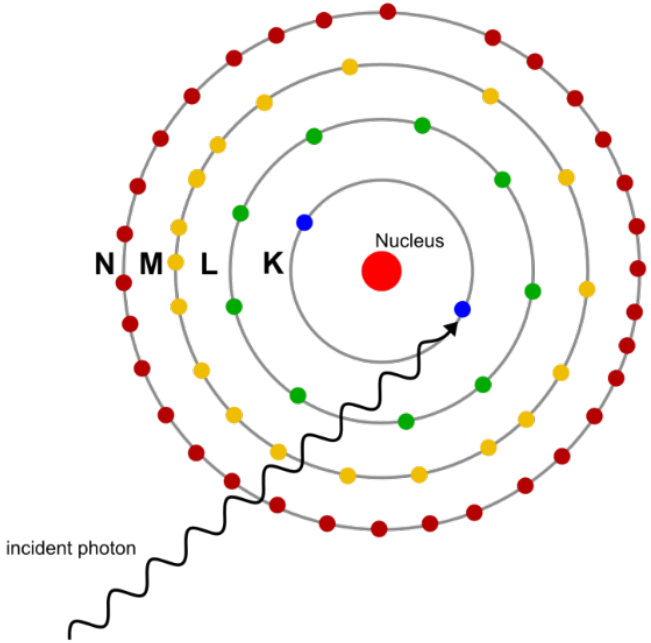
Applications are getting more demanding for higher accuracy, better precision, better detection of traces

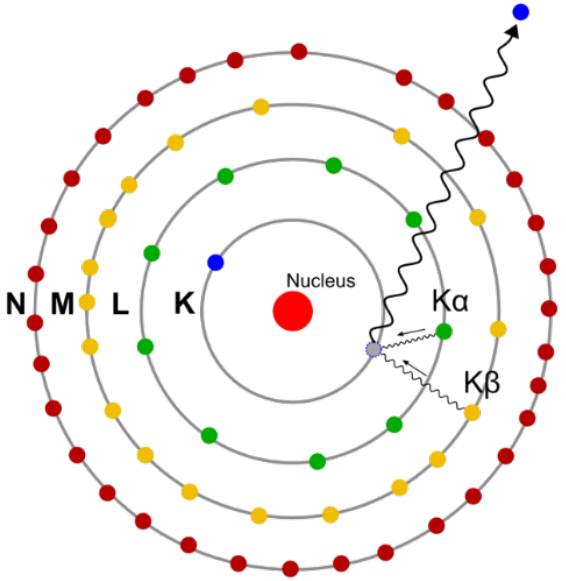
New legislation is enforcing extended analysis – more elements, lower detection limits, closer monitoring

Examples:

- Sand/Quartz: Construction, playgrounds, glass or solar cells, semicon wafers (Fe content)
- Coal: heating, power generation, electrodes for aluminum, electrodes for batteries (S, Cl, trace elements)
- Limestone: application defines the purity requirements: Cement, Feed (mineral nutrients), chemicals
- Bauxite: Bauxite useable in the Bayer process is containing 50 to 55 % Al_2O_3, max. 1.5 % SiO_2 and up to 30% Fe_2O_3.

X-ray Fluorescence Analysis (XRF) Principle – Photoelectric Effect





- Sample excited with an X-ray beam causing fluorescence
- Electron ejected from an inner shell of its atom
- Electron from a shell farther out falls into the vacancy

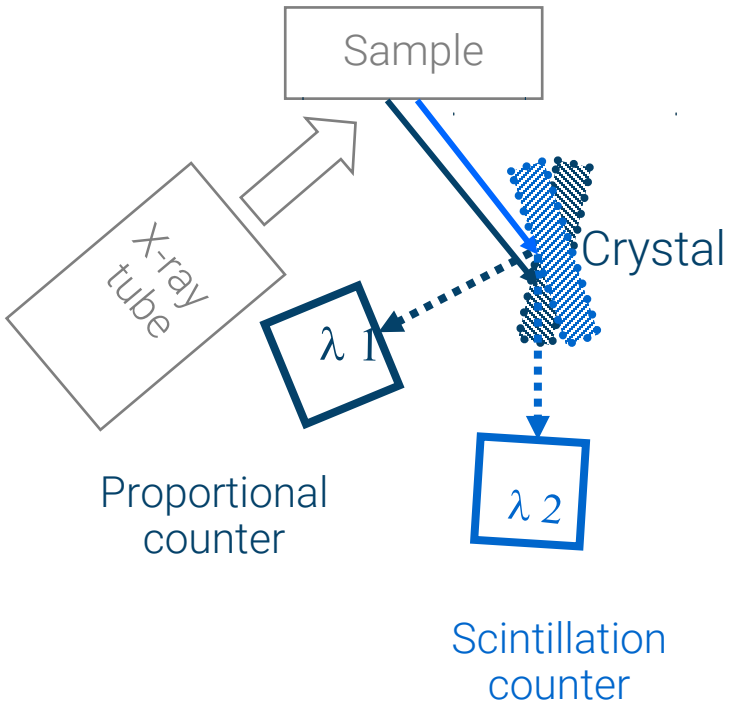
- Energy difference is emitted as an X-ray photon
- Discrete energy or wavelength is characteristic for the emitting element / transition
- Intensity of characteristic radiation is proportional to concentration of the element in the sample

X-ray Fluorescence Analysis (XRF) Capabilities

- Qualitative Analysis
  - Identification of elements
  - “What’s inside?”
- Quantitative Analysis
  - Determination of concentrations
  - “How much is inside?”
- Semi-Quantitative Analysis
  - Estimation of concentration
  - “About how much?”

- Solid and liquid samples can be analyzed directly:
  - Little or no sample preparation required
- Analysis is non-destructive to the sample
- Sampling-to-analysis result time is relatively short (~3 to ~10 min)
- Quantitative and qualitative analyses are possible
- Accuracy and long-term stability
- Elemental range: (Be) Na to U
- Linearity from ppm to 100%

Wavelength-dispersive X-ray Fluorescence (WDXRF)

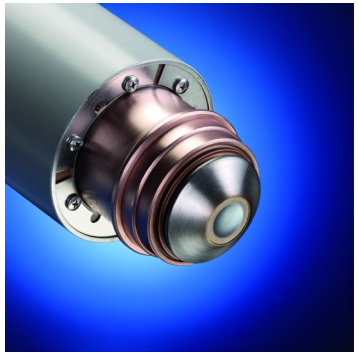


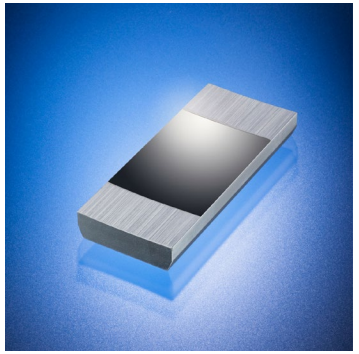
An analyzer crystal is used to separate the different wavelengths λ (energies)

The detector records the number N of X-ray photons at a given wavelength (energy)

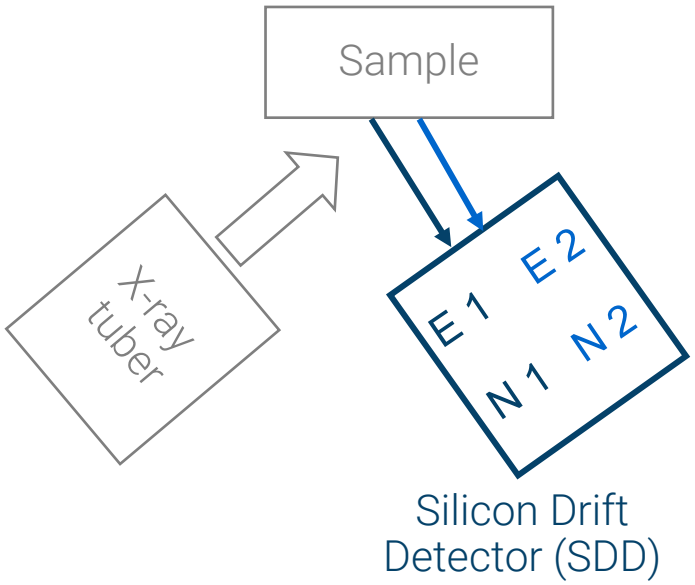
Two detectors are used to cover the entire element area

- Proportional counter: B to Cr
- Scintillation counter: Mn to U





Energy-dispersive X-ray Fluorescence (EDXRF)



The detector is used to detect both

- the energy E
- record the number N of X-ray photons at a given energy

- No collimators (as used in WDXRF), no crystals are required
- No moving components

X-ray Fluorescence Analysis (XRF)
The Comparison of Energy and Wavelength Dispersive Spectrometers

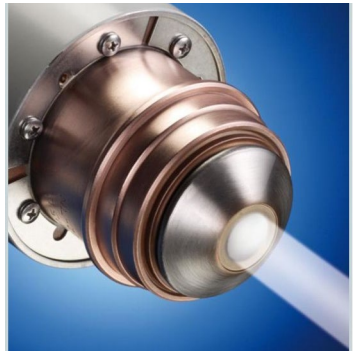
EDXRF

- Mechanical simplicity
- Cheaper
- Sensitivities: down to the ppm level
- Easy operation
- Smaller, “can be brought to the sample”

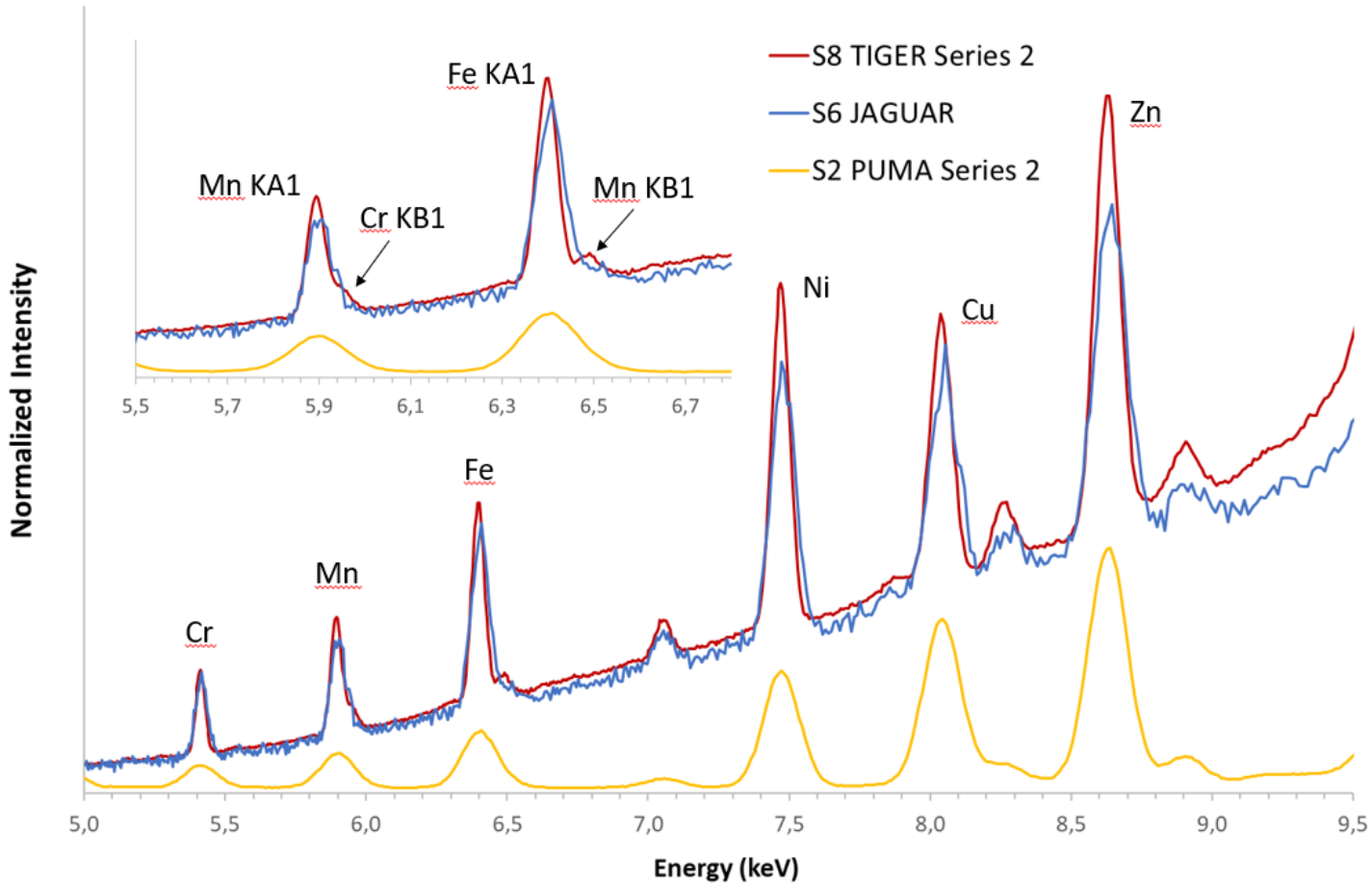
WDXRF

- High precision mechanics
- Higher capital
- Precision: <0.05%
- Higher resolution
- Sensitivities: down to the ppm level, but roughly one to two orders more sensitive
- Very fast analysis
- Highest sample throughput






EDXRF & WDXRF - Intensity vs. Resolution

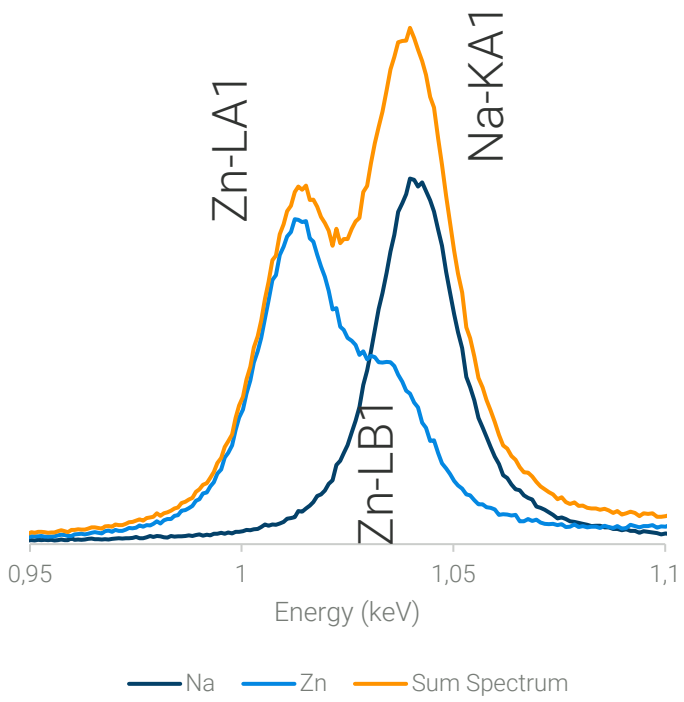


- Intensity and resolution comparison
- Fused bead with 100 ppm of several elements
- Measured on EDXRF and WDXRF instruments
  - S2 PUMA
    40 kV, 500 µm Al
  - S6 JAGUAR
    50 kV, LiF200
  - S8 TIGER
    50 kV, LiF200
- Intensities normalized to tube current

New Multilayer Optics for Better Separation Enhancing WDXRF for Specific Applications

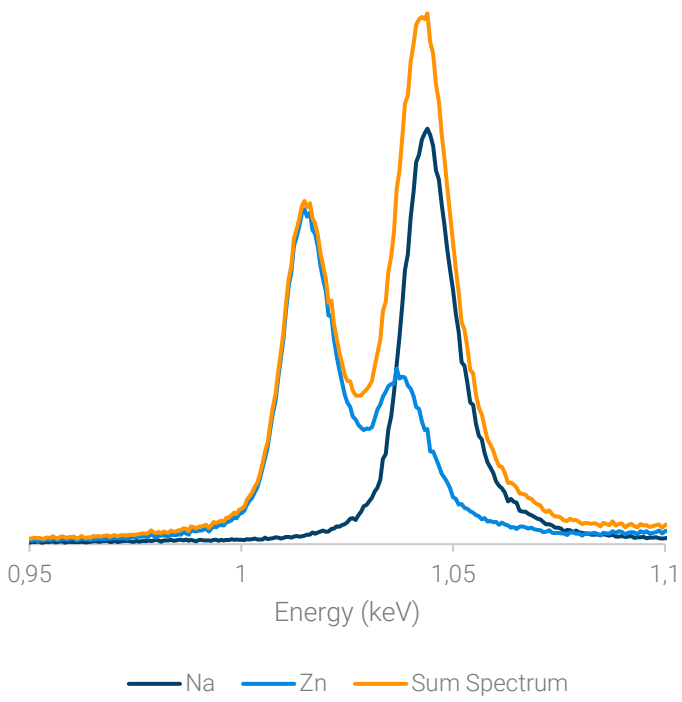
Na-KA1 / Zn-KA1
30kV, 0.23-degr, XS-55, Flow Counter

XS-55 (2d = 5.47nm)



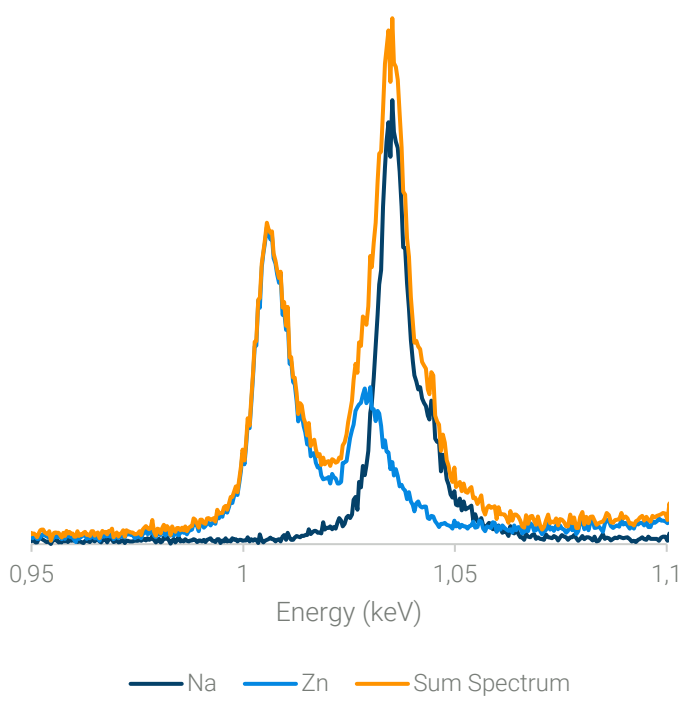
Na-KA1 / Zn-KA1
30kV, 0.23-degr, XS-100, Flow Counter

XS-100 (2d = 3.95nm)



Na-KA1 / Zn-KA1
30kV, 0.23-degr, XS-CEM, Flow Counter

XS-CEM (2d = 2.75nm)



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

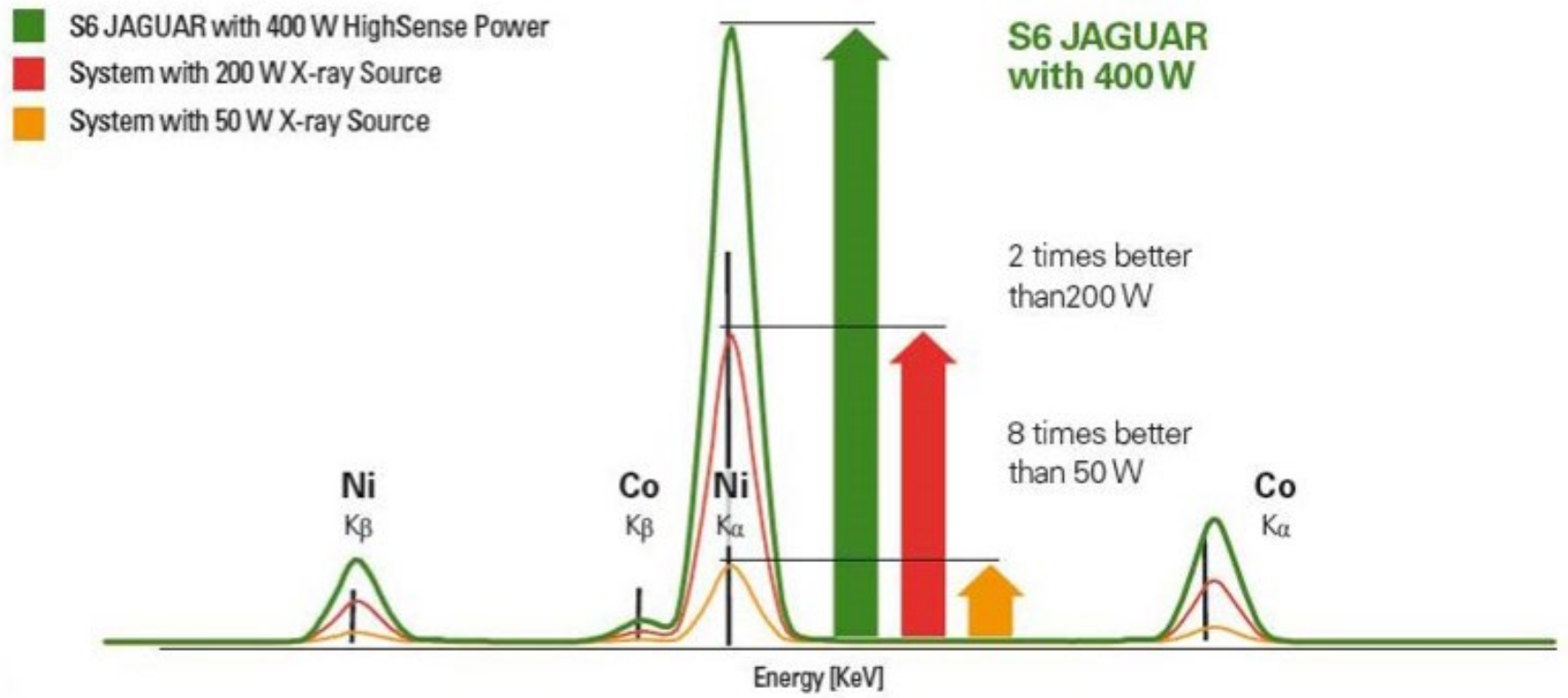
WDXRF and OES are established in central labs, but is there a place for affordable instruments?

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: 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

Silica Sands Analysis
S6 JAGUAR (benchtop WDXRF)

- Analysis of Silica Sands
- Determination of low concentrations of Fe (value determining element: < 50 ppm for clear glass)
- Analysis of minor elements, such as Ca, Al
- Quick preparation as pressed pellets

Grade Control at mining sites

Pressed powders

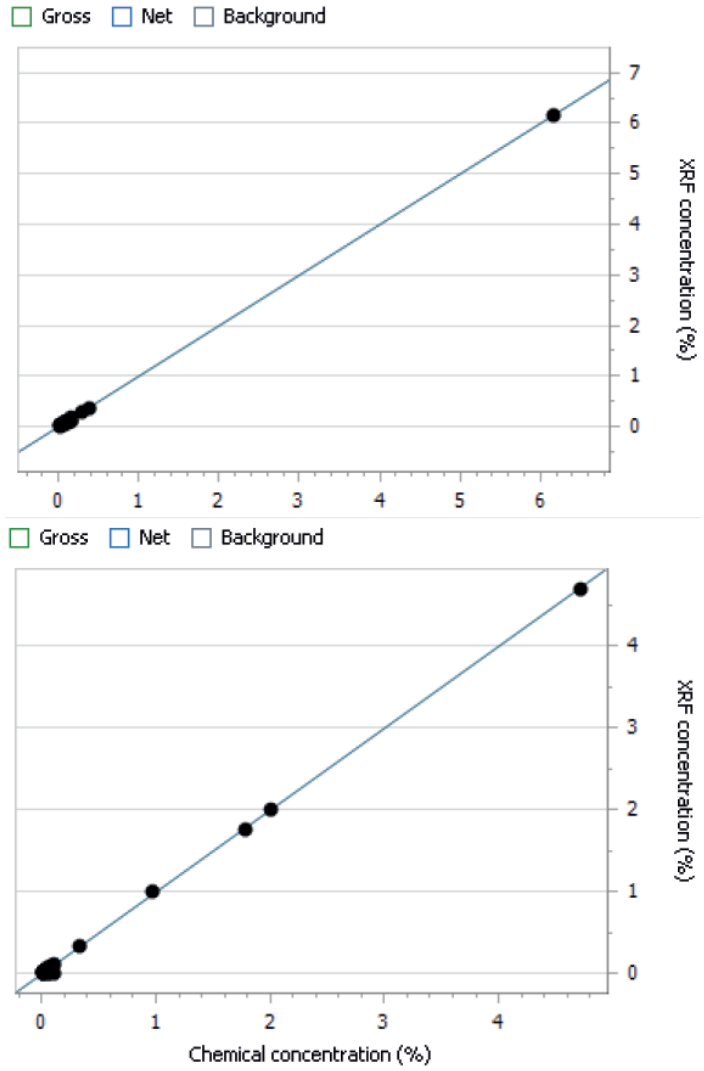
Loose powders

Silica Sands Analysis
S6 JAGUAR (benchtop WDXRF)

Compound	Calibration Range [wt.%]
Na_2O	0.001 - 1.00
MgO	0.001 - 0.89
Al_2O_3	0.018 - 6.12
SiO_2	Matrix
K_2O	0.004 - 9.99
CaO	0.004 - 0.25
TiO_2	0.002 - 0.25
Fe_2O_3	0.007 - 4.72

- Separation of major element peaks SI Ka from Al Ka with S6 JAGUAR's
- Optimal detection limit for Fe
- High accuracy and precision of Fe

Specific calibration based on CRM's, alternatively with fused bead preparation and GEO-QUANT BASIC calibration



Silica Sands Analysis
S6 JAGUAR (benchtop WDXRF)

Rep.	Fe_2O_3 [%]	Al_2O_3 [%]	TiO_2 [%]	CaO [%]	MgO [%]	Na_2O [%]	TiO_2 [%]	SiO_2 [%]
Average Tab. 4	0.011	0.035	0.018	0.007	0.003	0.002	0.005	99.91
Certified	0.012	0.036	0.017	0.006	0.001	0.002	0.005	99.92
Deviation	0.001	0.001	0.001	0.001	0.002	0.001	0.000	0.007

- Very close match between certified values and analytical data
- Less than 1 % RSD. for major element, SI
- Less than 10% RSD. for traces (e.g., Fe)
- Excellent light element detection (Traces of Na and Mg)

- Better data compared to EDXRF instruments, comparable to floor-standing high power WDXRF, just slightly longer measurement time

Silica Sands Analysis
S6 JAGUAR (benchtop WDXRF)

Rep.	Fe_2O_3 [%]	Al_2O_3 [%]	TiO_2 [%]	CaO [%]	MgO [%]	Na_2O [%]	TiO_2 [%]	SiO_2 [%]
#01	0.010	0.038	0.017	0.007	0.004	0.002	0.005	99.91
#02	0.011	0.037	0.016	0.007	0.007	0.002	0.004	99.91
#03	0.010	0.035	0.018	0.007	0.003	0.002	0.005	99.92
#04	0.011	0.033	0.017	0.007	0.004	0.002	0.005	99.91
#29	0.011	0.035	0.018	0.006	0.003	0.002	0.005	99.92
#30	0.011	0.036	0.017	0.008	0.001	0.002	0.005	99.91
Min.	0.010	0.032	0.016	0.006	0.001	0.001	0.004	99.91
Max.	0.011	0.038	0.019	0.009	0.007	0.002	0.006	99.92

- High analytical precision within 30 rep. Measurements
- Less than 0.5% R.S.D. variantion for major element Si
- High precision even for traces

Limestone Analysis
S6 JAGUAR (benchtop WDXRF)

- Analysis of Limestone
- Later use in paints, polymers, pharma, paper, toothpaste
  - Color important (Mn, Fe)
  - Hazardous elements (Cr)
  - Hardness (SiO2)
  - Quick sample preparation with 8 g sample and 2 g binder (wax)

Grade Control at mining sites

Pressed powders

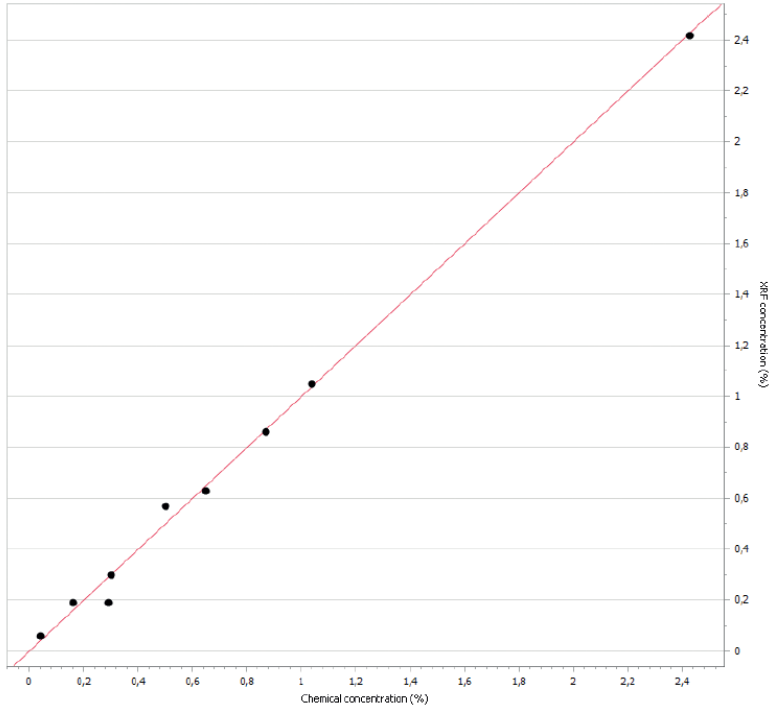
Loose powders

Limestone Analysis
S6 JAGUAR (benchtop WDXRF)

[wt.%]	Concentration Range	R^2
Na_2O	0.003 – 0.46	0.991
MgO	0.24 – 17.88	0.999
Al_2O_3	0.05 – 2.4	0.972
SiO_2	0.45 – 12.4	0.999
P_2O_5	0.013 – 0.117	0.970
SO_3	0.016 – 0.13	0.981
K_2O	0.001 – 0.96	0.993
CaO	38.46 – 55.15	0.984
TiO_2	0.03 – 0.15	0.992
MnO	0.007 – 0.28	0.999
Fe_2O_3	0.04 – 2.43	0.996

- Optimal detection limit for Fe
- High accuracy and precision of Fe

Specific calibration based on CRM's, alternatively with fused bead preparation and GEO-QUANT BASIC calibration



[wt.%]	Na_2O	MgO	MnO	Fe_2O_3
Certified	0.460	5,97	0.280	2,43
Measured	0.461	5.87	0.286	2.47
Abs. Deviation	0.001	0.10	0.006	0.04

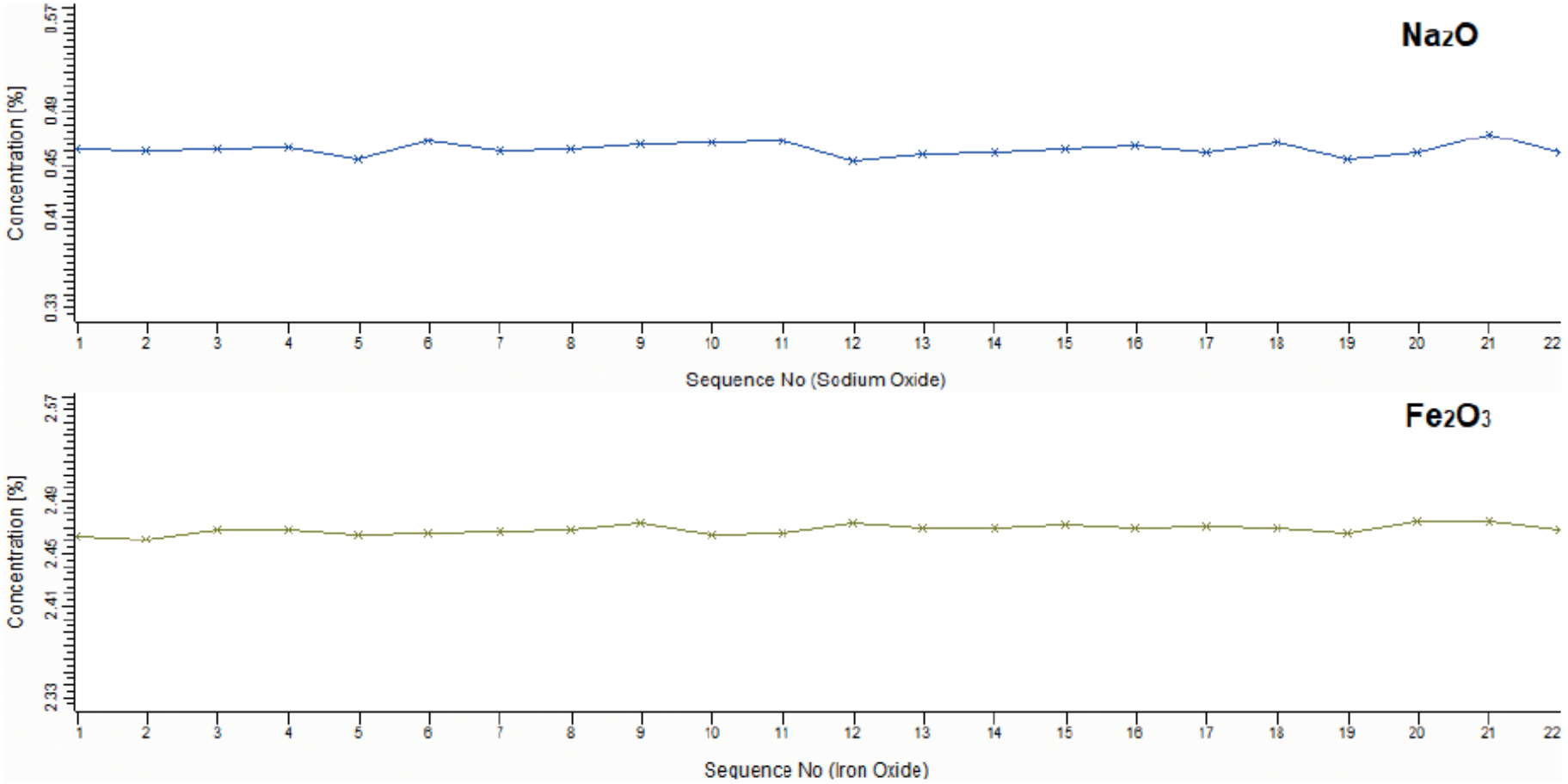
Limestone Analysis
S6 JAGUAR (benchtop WDXRF)

[wt.%]	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	Fe_2O_3
Rep-1	0.327	2.08	5.75	19.72	0.057	0.252	0.964	36.65	0.281	0.046	3.23
Rep-22	0.326	2.05	5.72	19.54	0.055	0.245	0.959	36.37	0.278	0.045	3.21
Average	0.326	2.07	5.73	19.64	0.056	0.248	0.962	36.56	0.281	0.045	3.22
Abs. Std. Dev.	0.003	0.010	0.015	0.040	0.001	0.002	0.002	0.063	0.001	0.001	0.006
Rel. Std. Dev. [%]	1.03	0.48	0.25	0.20	1.14	0.76	0.22	0.17	0.47	1.14	0.18

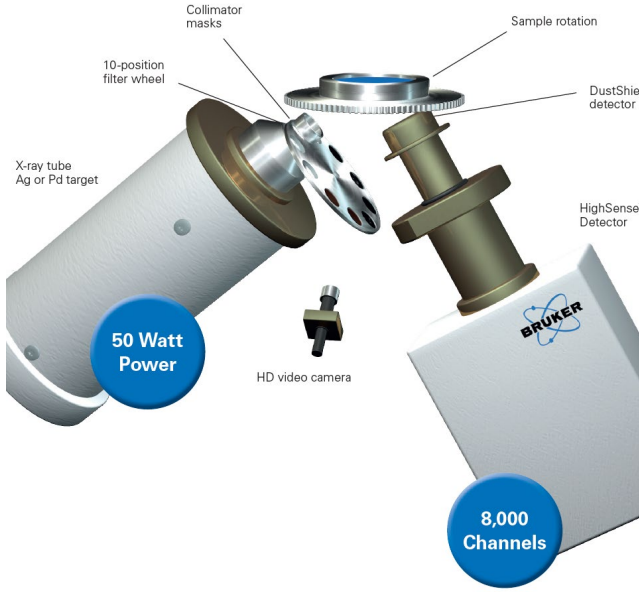
- Very close match between certified values and analytical data
- Less than 1 % RSD. for major element, SI
- Less than 10% RSD. for traces (e.g., Fe)
- **Excellent light element detection** (Traces of Na and Mg)

Limestone Analysis
S6 JAGUAR (benchtop WDXRF)

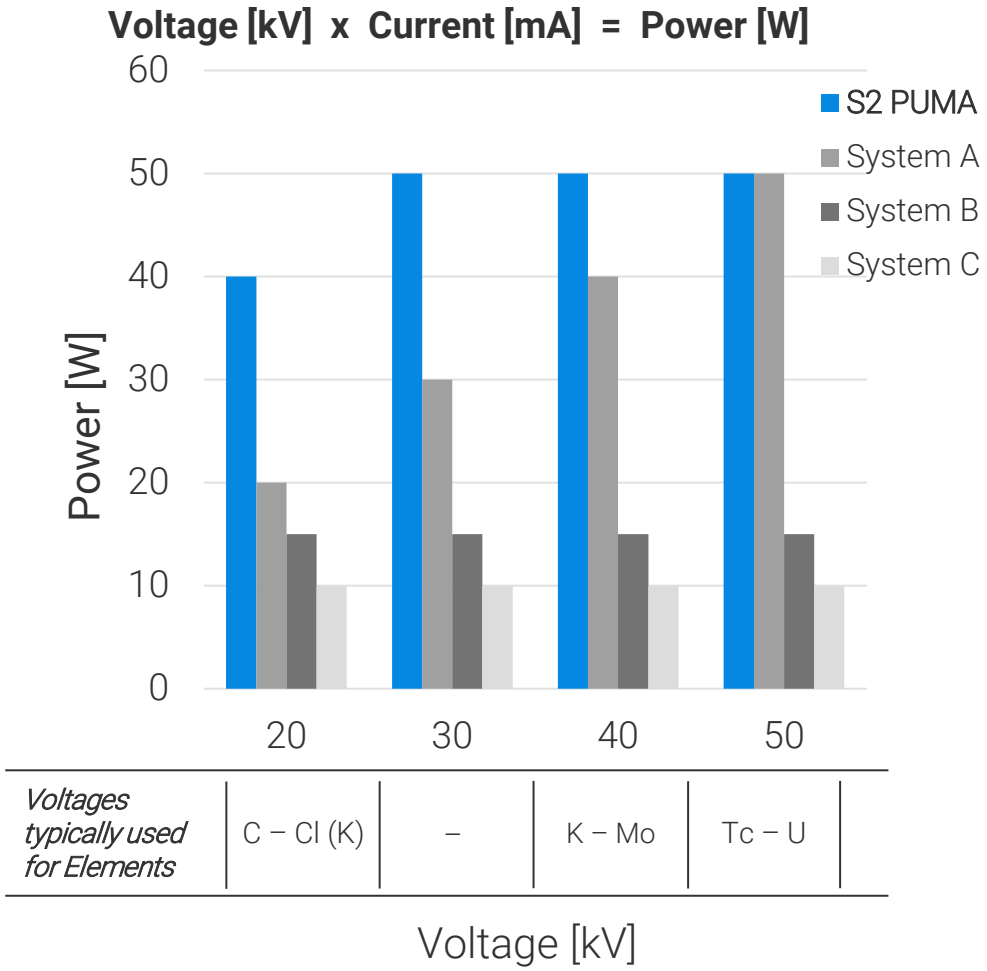
- High long term stabity for traces of light elements (Na) and elements of discoloring (Fe)



When to use EDXRF?
S2 PUMA Series 2 – Next Generation EDXRF

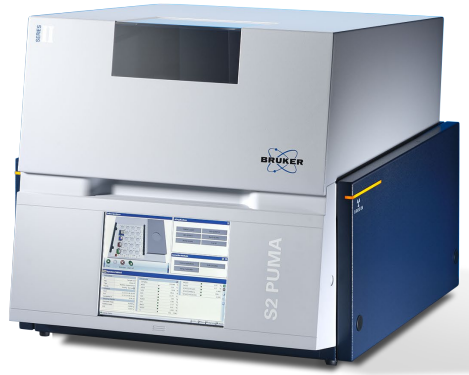


HighSense turns power into performance



Optimal excitation of the sample is ensured by:

- High power 50 Watt X-ray tube
- Up to 2 mA and 50 kV (30 kV)
- 10-position primary beam filter
- Direct excitation
- Closely coupled optics

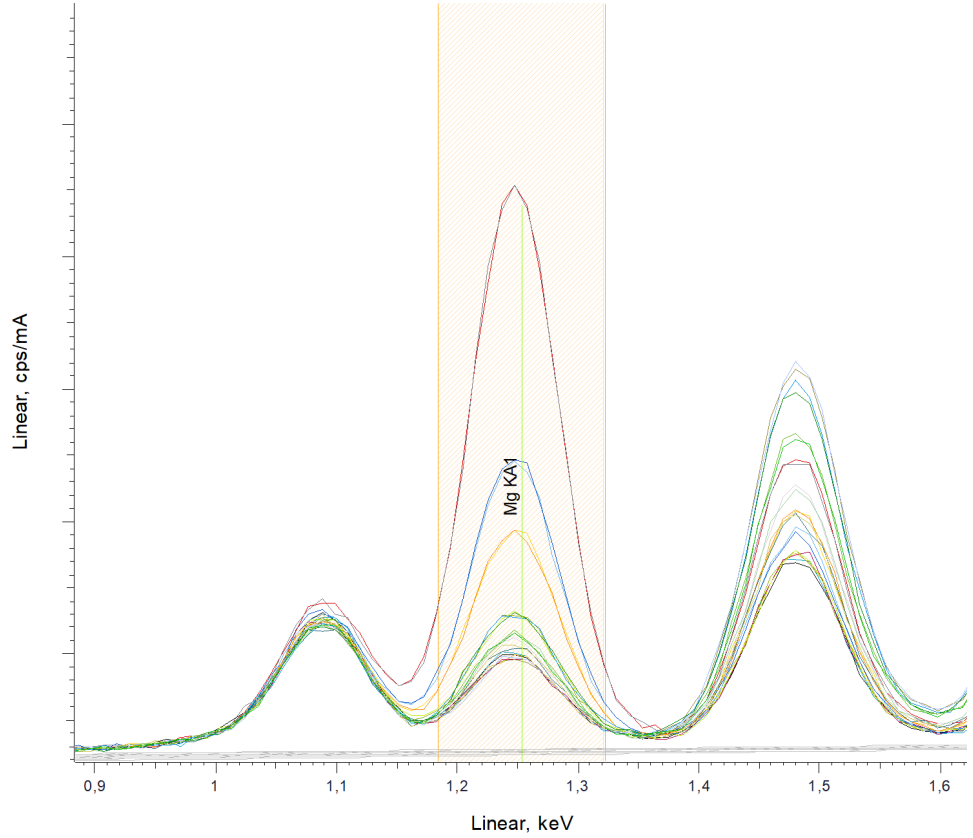


Analysis of Limestone S2 PUMA Series 2

- Pressed powder pellets, no wax
- Mg, Al, Si, S: 20 kV, no filter
- Ca, Mn, Fe: 40 kV, Al filter
- Time-to-result ~7 min. (incl. loading and unloading)

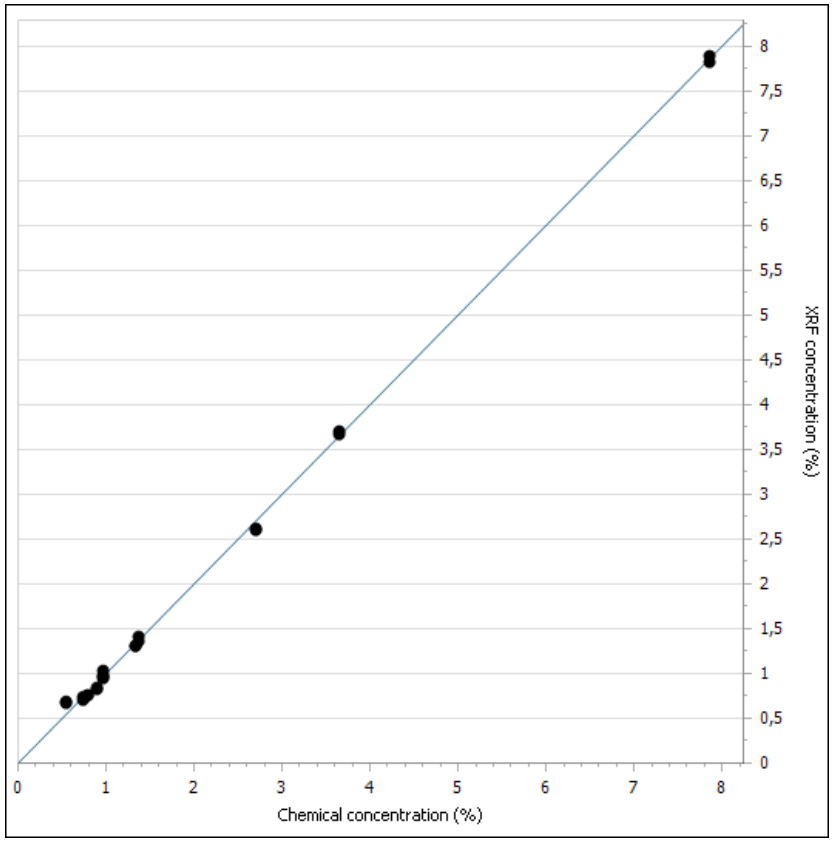
Analysis of Limestone S2 PUMA Series 2

- Mg KA1, 20 kV, auto-current, 0.5 – 7.8wt%
- Excellent signal to noise ratio



Analysis of Limestone S2 PUMA Series 2

- Mg KA1, 20 kV, auto-current, 0.5 – 7.8wt%
- R^2: 0.99917



Analysis of Limestone S2 PUMA Series 2

- S2 PUMA: Very good precision!

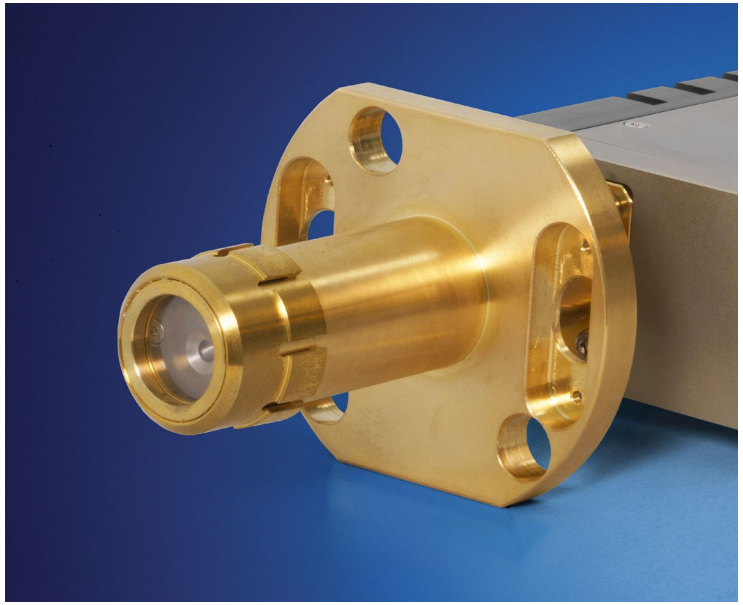
wt.%	CaO ($CaCO_3$)	MgO ($MgCO_3$)	SiO_2	Al_2O_3	Fe_2O_3	Mn_3O_4	SO_3	Sum
1	96.88	1.07	1.17	0.454	0.298	0.047	0.068	99.99
2	96.86	1.07	1.16	0.441	0.298	0.047	0.069	99.94
3	96.84	1.09	1.16	0.445	0.299	0.046	0.070	99.96
...	...	...	...	...	...	...	...	...
10	96.98	1.08	1.17	0.444	0.300	0.048	0.069	100.09
Min	96.84	1.07	1.16	0.441	0.298	0.045	0.066	99.94
Max	96.98	1.09	1.17	0.454	0.300	0.048	0.070	100.09
Average	96.91	1.08	1.16	0.448	0.299	0.046	0.068	100.01
SD	0.044	0.007	0.005	0.004	0.001	0.001	0.001	0.045
RSD(%)	0.05	0.69	0.39	0.96	0.30	2.17	2.05	0.05

- S6 JAGUAR (same samples): Similar precision in 5 min (instead of 7 min on the S2 PUMA)
- S6 JAGUAR: Better performance for F and Na (and low Mg)

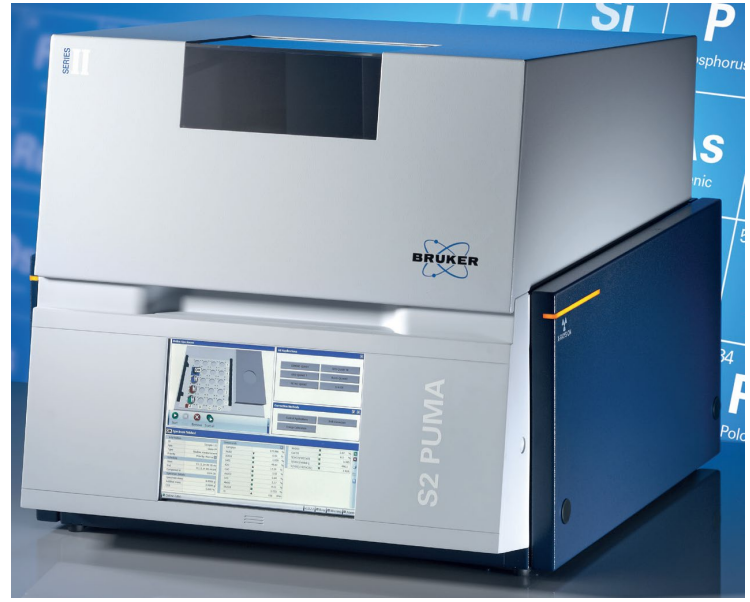
S2 PUMA Series 2 with HighSense™ XP Detector

State-of-the-Art Hardware & Next Generation Software

- New premium detector for all elements (C to Am)
- Robust, high transmission Graphene window (non-toxic)
- Bruker's detector chip technology
- Further enhanced cooling (Peltier) performance



New HighSense XP Detector



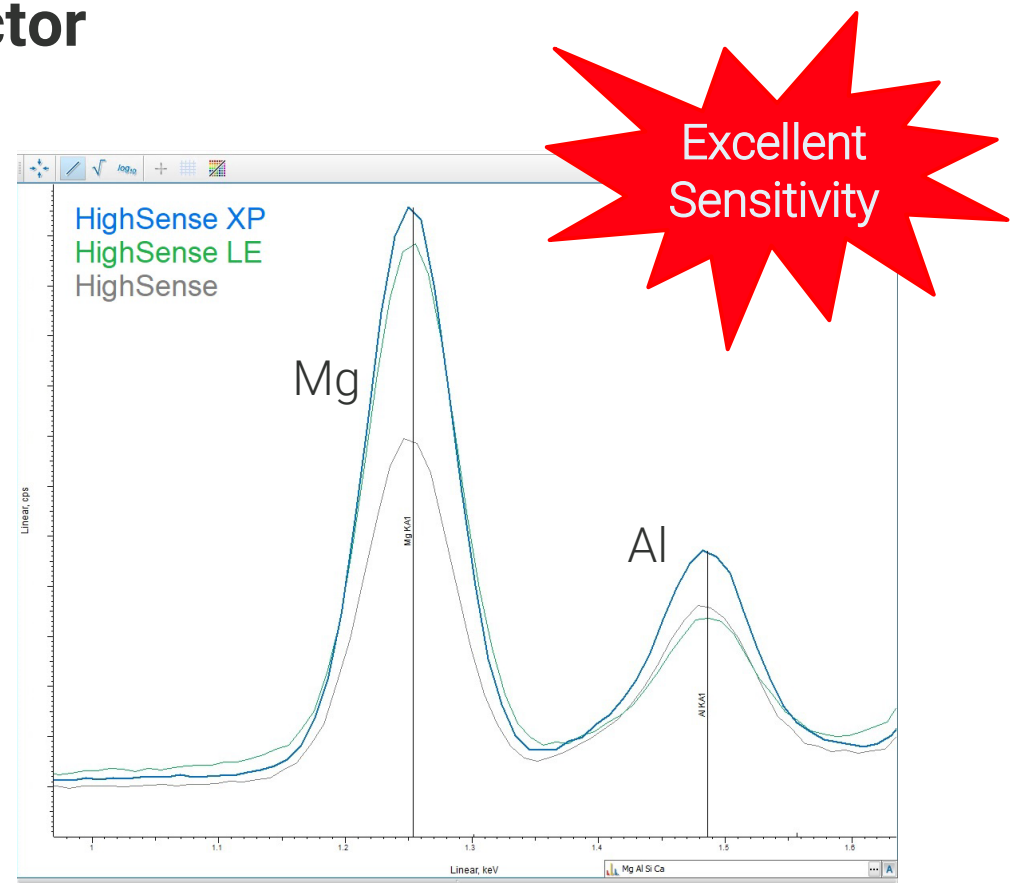
S2 PUMA Series 2 with XY Autochanger

- New SPECTRA.ELEMENTS with **Dynamic Detector Profiling**

S2 PUMA Series 2 with HighSense™ XP Detector

Ultra-high transmission

- The 0.9 µm Graphene Window offers ultra high transmission
- Sensitivity improved by typically 30% compared to the HighSense LE
- A Si-grid is not required, minimizing diffraction peaks and improving the performance for Si



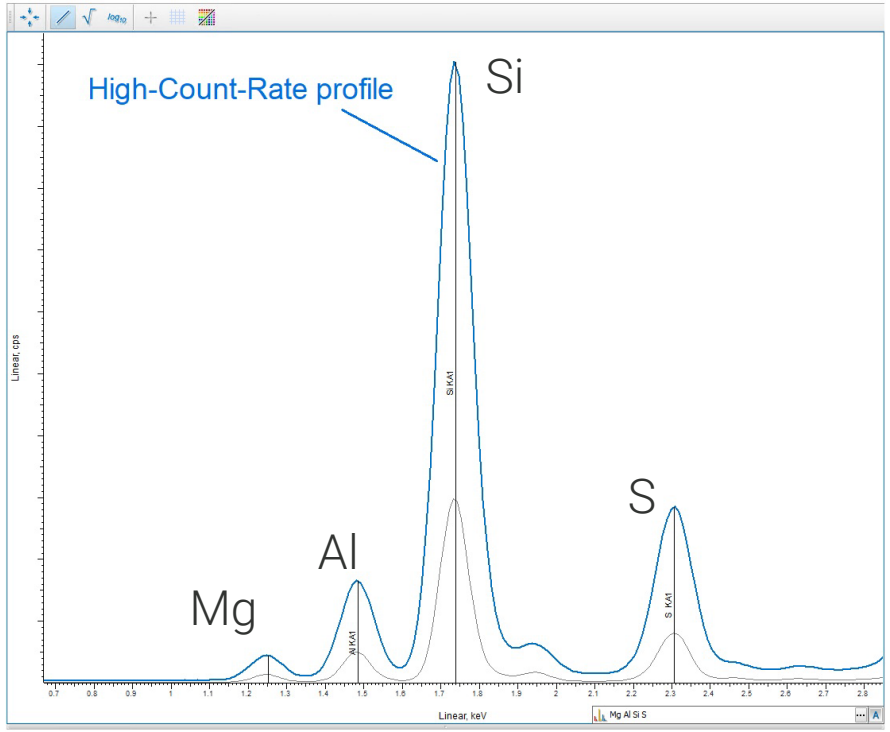
HighSense XP: Best performance for all element!

The Benefits of Detector Profiling

- New, fully integrated feature in SPECTRA.ELEMENTS
- Flexible: Make your selection each analytical range
  - **High Resolution** for optimal peak separation of neighboring elements
  - **High-Count-Rate** to boost the throughout or decrease LLD

SPECTRA.ELEMENTS

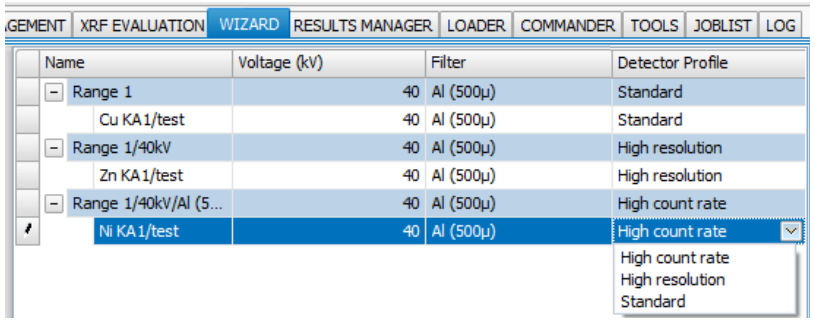
Easy. Fast. Smart. Powerful.



Example: Achieve 4 x higher net intensity for **Cement** samples with the High-Count-Rate profile

The Benefits of Detector Profiling

- New, fully integrated feature in SPECTRA.ELEMENTS
- Flexible: Make your selection each analytical range
  - **High Resolution** for optimal peak separation of neighboring elements
  - **High-Count-Rate** to boost the throughout or decrease LLD



Select the profile for each range when you setup your method.

SPECTRA.ELEMENTS

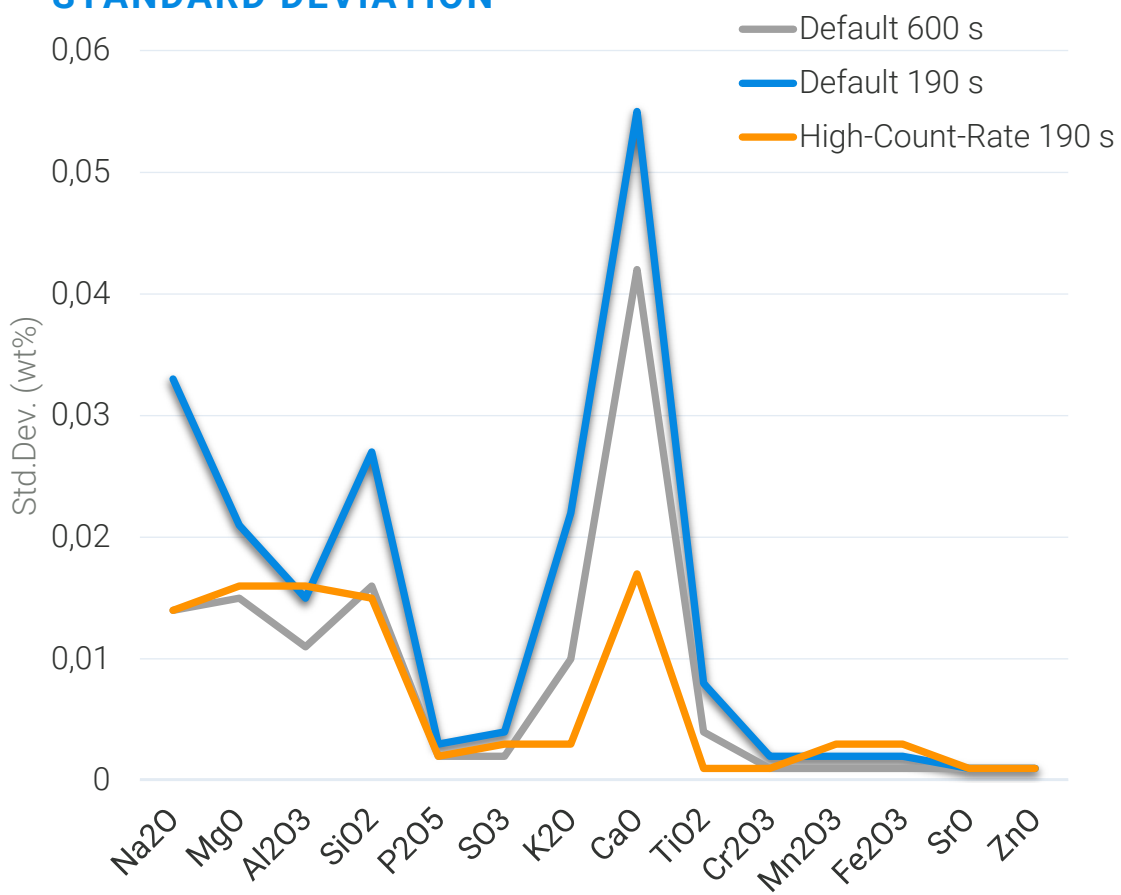
Easy. Fast. Smart. Powerful.

The Benefits of Detector Profiling

Precision Test

- Cement QC sample
- 3 ranges, 190 or 600 s total counting time, 25 repetitions

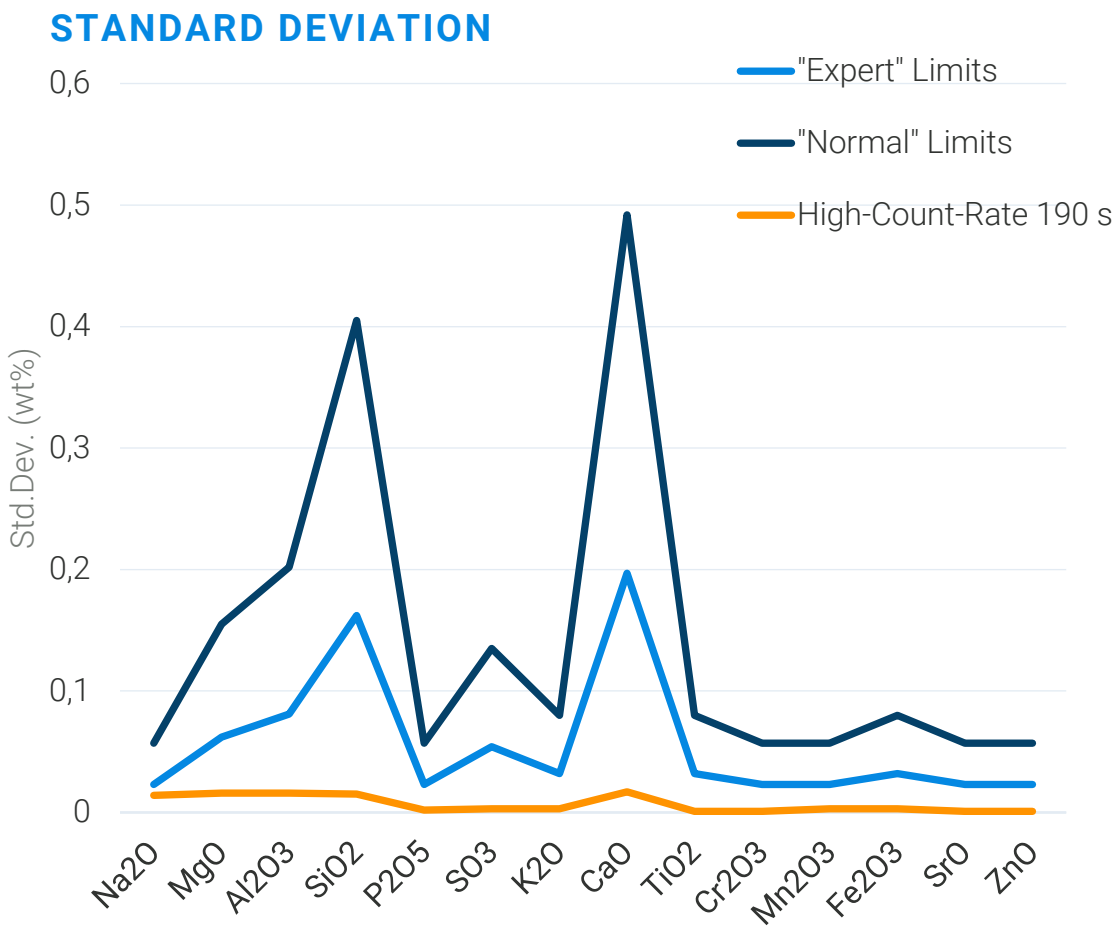
The High-Count-Rate setting allows to reduce the counting time factor of ~3



The Benefits of Detector Profiling

ISO 29581 / EN 196-2:
A Cement norm which defines two categories of precision (repeatability) :
“Normal” and “Expert”

- The S2 PUMA Series 2 with HighSense XP meets standard deviation limits easily.



GEO-QUANT Basic
Out-of-the-box solution for Industrial Minerals Applications

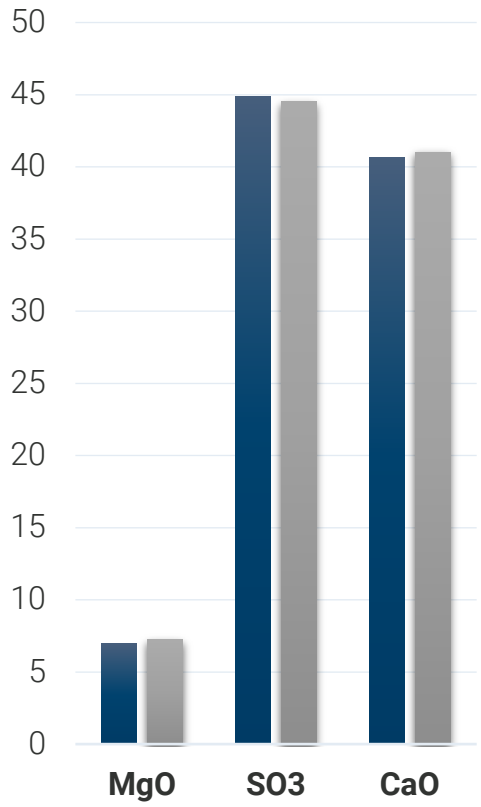
Ready for various applications

- Industrial minerals grade control
- Raw materials for cement and building materials
- Mining operations
- Refractories
- Ceramics and glass
- Geochemical research
- Archaeology
- Environmental studies

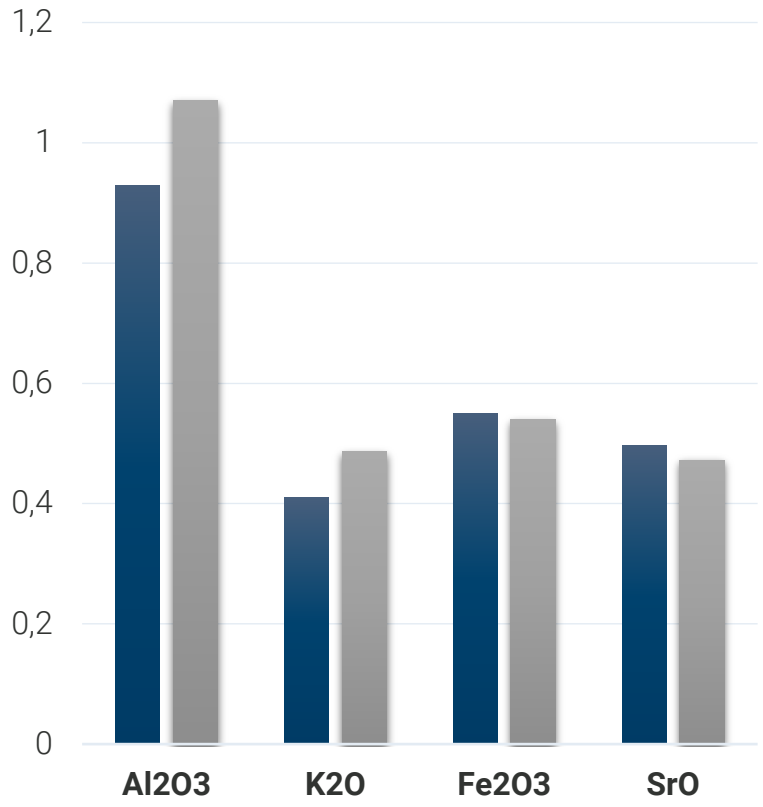
Compound	Number of CRMs	Max. conc. (wt%)
Na_2O	16	11
MgO	18	40
Al_2O_3	19	85
SiO_2	19	100
P_2O_5	16	7.5
SO_3	13	58
K_2O	18	12
CaO	18	100
TiO_2	19	4
Cr_2O_3	14	0.1
MnO	16	0.9
Fe_2O_3	20	100
ZnO	11	0.2
SrO	13	0.3

GEO-QUANT Basic
Out-of-the-box solution for Industrial Minerals Applications

MAJOR ELEMENTS



MINOR ELEMENTS



Gypsum – concentrations in wt%

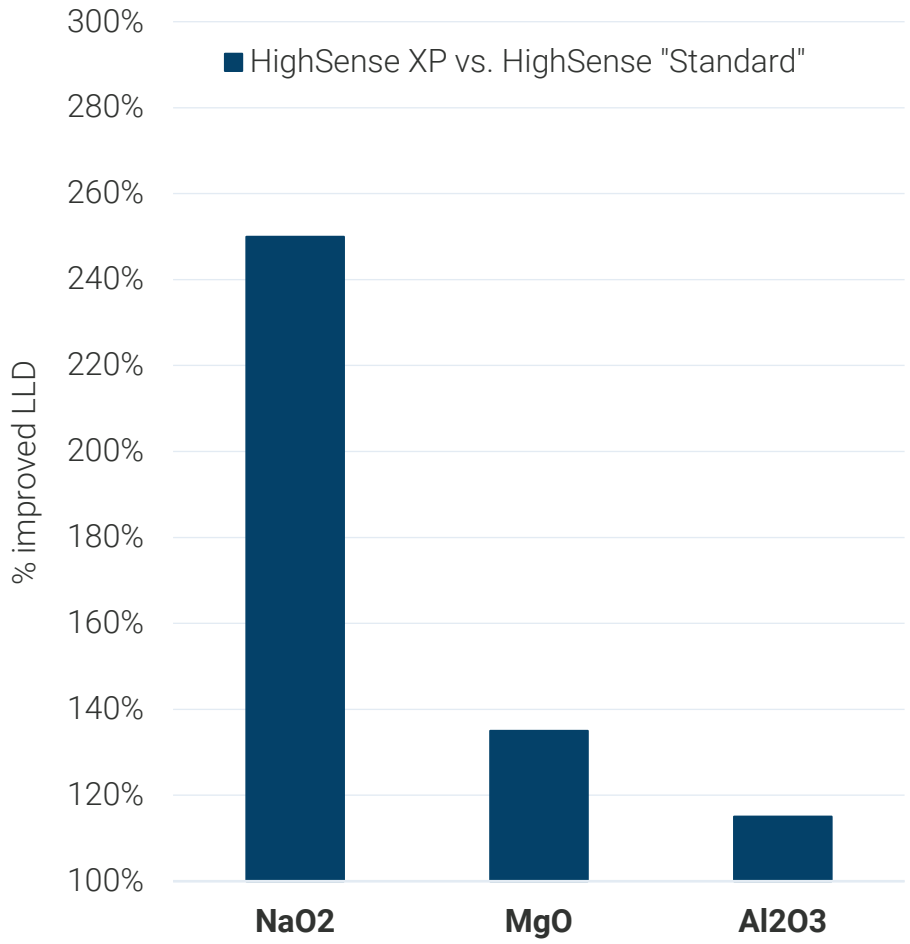
GEO-QUANT Basic
Out-of-the-box solution for Industrial Minerals Applications

Detector Comparison

HighSense XP vs. HighSense

- QC samples tested:
  - Gypsum
  - Iron Ore Sinter
  - Limestone
  - Cement

- HighSense XP: Advantages for low levels of Na, Mg, and Al.



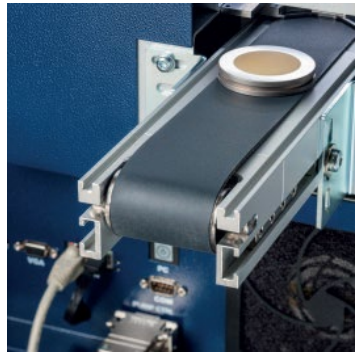
S2 PUMA Series 2 - S6 JAGUAR
Taylor-made Solution for all Applications

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

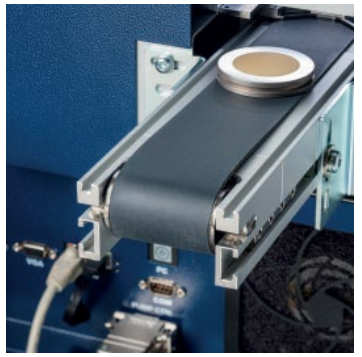
- Samples are fed directly, via robot or belt, from the automated sample preparation system
- LIMS-compatible: The AXSCOM interface connects to the process control software



Powerful Benchtop units for dedicated tasks

- Compact X-ray fluorescence devices are now very powerful due to new detector technologies
  - Improved spectral resolution
  - High analytical precision thanks to higher counting rates
  - Simple operation and therefore quick integration into quality control
- Decision about the technology (ED or WD) by:
  - Elements of interest and concentration ranges
  - Required precision and LLDs
  - Required sample throughput

S6 JAGUAR Benchtop WDXRF



S2 PUMA Series 2 EDXRF

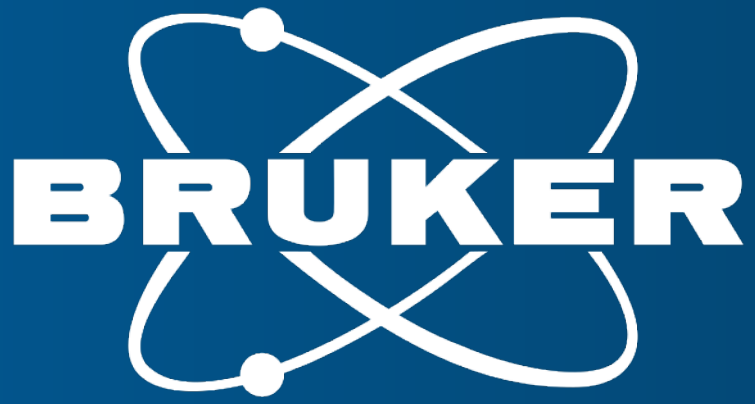
Any Questions?

Learn more about Ore Grade Control with XRF: Key For Successful Metal Production

In our 10.11.2021 webinar – Sign Up Now!

Thank you!

Kai Behrens, Frank Portala, Adrian Fiege



Innovation with Integrity