Industrial Quality Control with the S6 JAGUAR

Welcome

How to accomplish your quality control applications with new powerful benchtop WDXRF S6 JAGUAR in a cost efficient way!

- What is XRF – a quick tour
- XRF technologies – a comparison
- What is important to set up a quality control application
- Instrument parameters and their influence on data quality
- Application examples for industrial QC
- Summary

- Q&A session

Dr. Adrian Fiege
Product Manager XRF
Karlsruhe, Germany

Dr. Kai Behrens
Head of Product Management XRF
Karlsruhe, Germany

Industrial Quality Control Applications by X-ray fluorescence (XRF)

- Concentrations of industrial products are vital:
  - Define commercial value of the final product (ores)
  - Monitor an efficient process (clinker)
  - Establish product performance (additives)
  - Protect humans, environment (hazardous elements in food)

- Application requires precise and accurate determination of concentrations in liquids and solids:
  - Majors for grade control in the range of 10 - 100 %: ores, metals, minerals
  - Minors from 0.1 - 10 %: additives, minerals in feed, sulfur in oil
  - Trace elements from sub-ppm - 1000 ppm: Toxic elements in soil, RoHS, Si and P in iron ore

X-ray fluorescence (XRF) analysis
Element Ranges

X-ray fluorescence analysis
or X-ray spectrometry

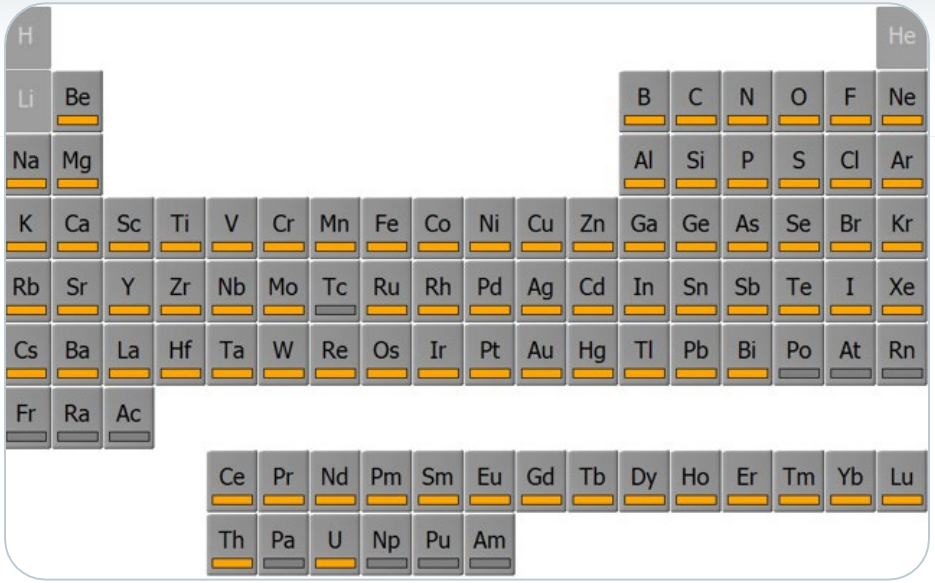
- A method to do qualitative and quantitative analysis of the elemental composition by excitation of atoms and detection of their characteristic X-rays

- High power WDXRF:
  Be (B) – Am

- Mid-range power WDXRF:
  O (F) – Am

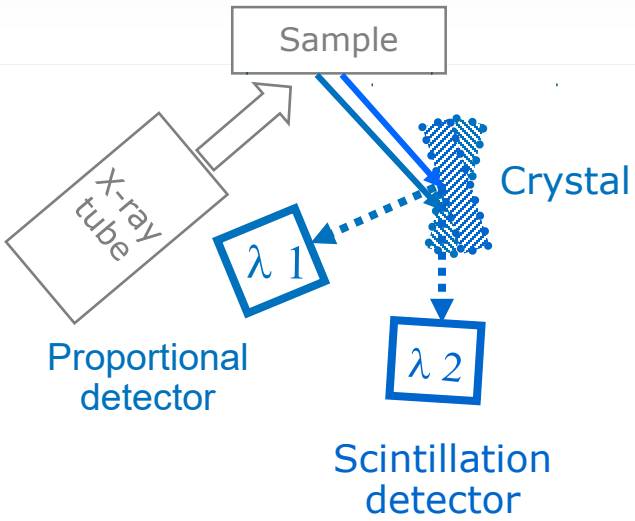
- EDXRF:
  C (F) – Am

- Low performance EDXRF:
  Na (Mg) – Am



- Elemental range:
  (Be) B to U
- Concentration range:
  Sub-ppm - 100 %

Instrumentation for X-ray Spectrometry
Wavelength Dispersive Spectrometers

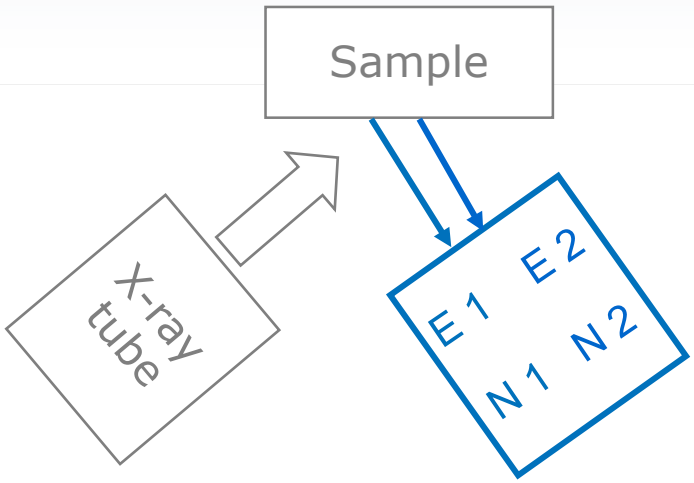


- An analyzer crystal is used to separate the various wavelengths λ (energies)
- The detector records the number N of X-ray photons at a given wavelength (energy)
- Two detectors are used to cover the whole elemental range

  Proportional detector → B to Cr

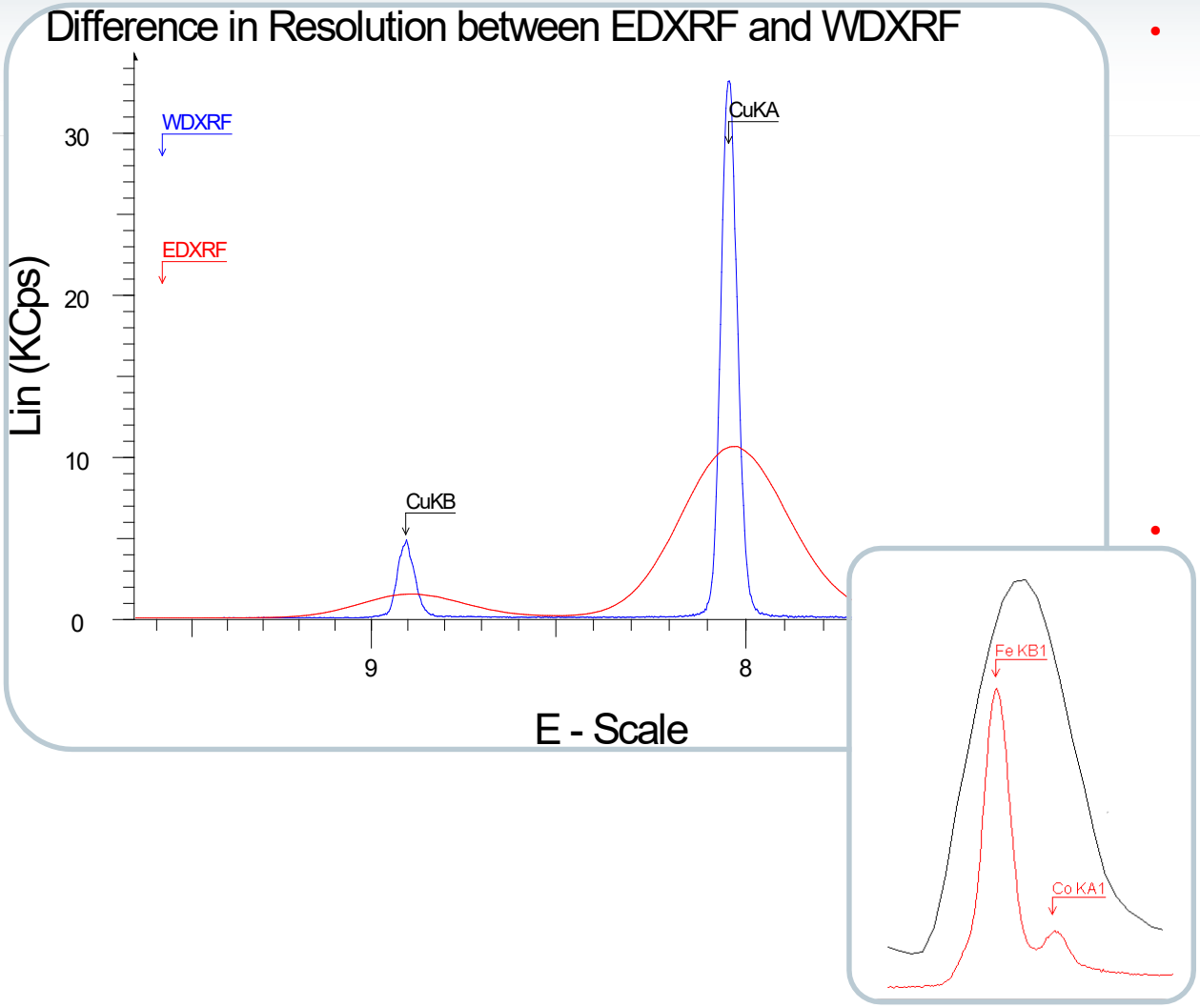
  Scintillation detector → Mn to U

Instrumentation for X-ray Spectrometry
Energy Dispersive Spectrometers



- The detector is used to record both
  - the energy E
  - the number N of X-ray photons at a given energy
- No Soller slits (collimators as used in WDXRF) and no crystals are required

X-ray fluorescence analysis (XRF)
EDXRF vs. WDXRF



- Large difference in resolution between WDXRF and EDXRF
  - In WDXRF the combination of crystal, collimator, and detector gives us a much higher resolution
  - In EDXRF the resolution only depends on the detector

- Steel sample with 0.31% Co:

  Co $K\alpha_1$ line is overlapped by Fe $K\beta_1$

  Metal applications → WDXRF

X-ray fluorescence analysis (XRF)
EDXRF vs. WDXRF

WDXRF	EDXRF
• High precision mechanics • Higher cost • Precision: <0.05% • Higher resolution • Sensitivity: down to the ppm level, but roughly one to two orders more sensitive • Very fast analysis • Highest sample throughput	• Mechanical simplicity • Cheaper • Sensitivity: down to the ppm level • Easy handling • Smaller, “can be brought to the sample”

X-ray fluorescence analysis (XRF)
Which instrument?

The analytical performance of an X-ray spectrometer is determined by:

- the range of elements
- the separation of elements ("resolution")
- the sensitivity (kcps/%, cps/ppm)
- the peak to background ratio
- the lower limits of detection
- the reproducibility

How to address the gap between affordable compact EDXRF and high-performance floor-standing WDXRF?

Without too much compromise on performance?

S6 JAGUAR
High Performance Benchtop WDXRF

Maintain WDXRF resolution:

- Compact goniometer with high precision gears and closely coupled X-ray beam path
- Optimized analyzer crystals for the entire element range and special applications

Maintain analytical precision and sensitivity:

- Higher power X-ray tube compared to EDXRF (there is no saturation due to single element detection)
- HighSense detection with 2 Mcps count rate
- HighSense XE detector for medium and heavy elements

S6 JAGUAR
Application Examples


Glass & Ceramics


Minerals & Mining


Metals & Slags


Academia


Pharma


Food & Feed


Cement & Building Materials


Material Research

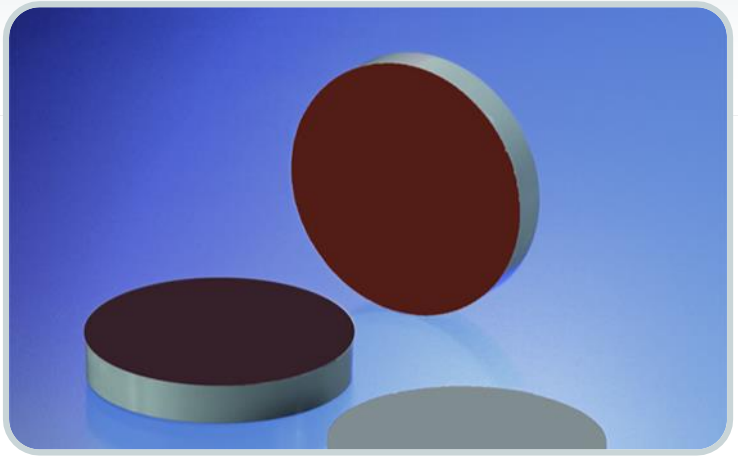

Petrochemistry

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

High intensity is required for best precision in order to control the important Ni and Cu concentrations



XRF - X-ray Fluorescence Analysis
Precision and Counting Statistics

Precision limited by counting statistical error

$$\Delta c / c = \text{SQRT}(N) / N = 1 / \text{SQRT}(N)$$

N =	100	SQRT (N) =	10	3*SQRT(N) / N =	30 %
N =	1000	SQRT (N) =	30	3*SQRT(N) / N =	10 %
N =	10 000	SQRT (N) =	100	3*SQRT(N) / N =	3 %
N =	100 000	SQRT (N) =	300	3*SQRT(N) / N =	1 %
N =	1000 000	SQRT (N) =	1000	3*SQRT(N) / N =	0.3 %
N =	10 000 000	SQRT (N) =	3000	3*SQRT(N) / N =	0.1 %

The S6 JAGUAR delivers more precise results:

- 400 W power, closely coupled optics
- Optimal excitation
- Enhanced sensitivity with optimal analyzer crystals
- HighSense detectors and counting electronics with up to 2 Mcps

S6 JAGUAR
HighSense™: Full 400 W Excitation Power

400 W HighSense™ power*:

- Twice the sample throughput
- 50% shorter measurement times
- 30% enhanced analytical precision

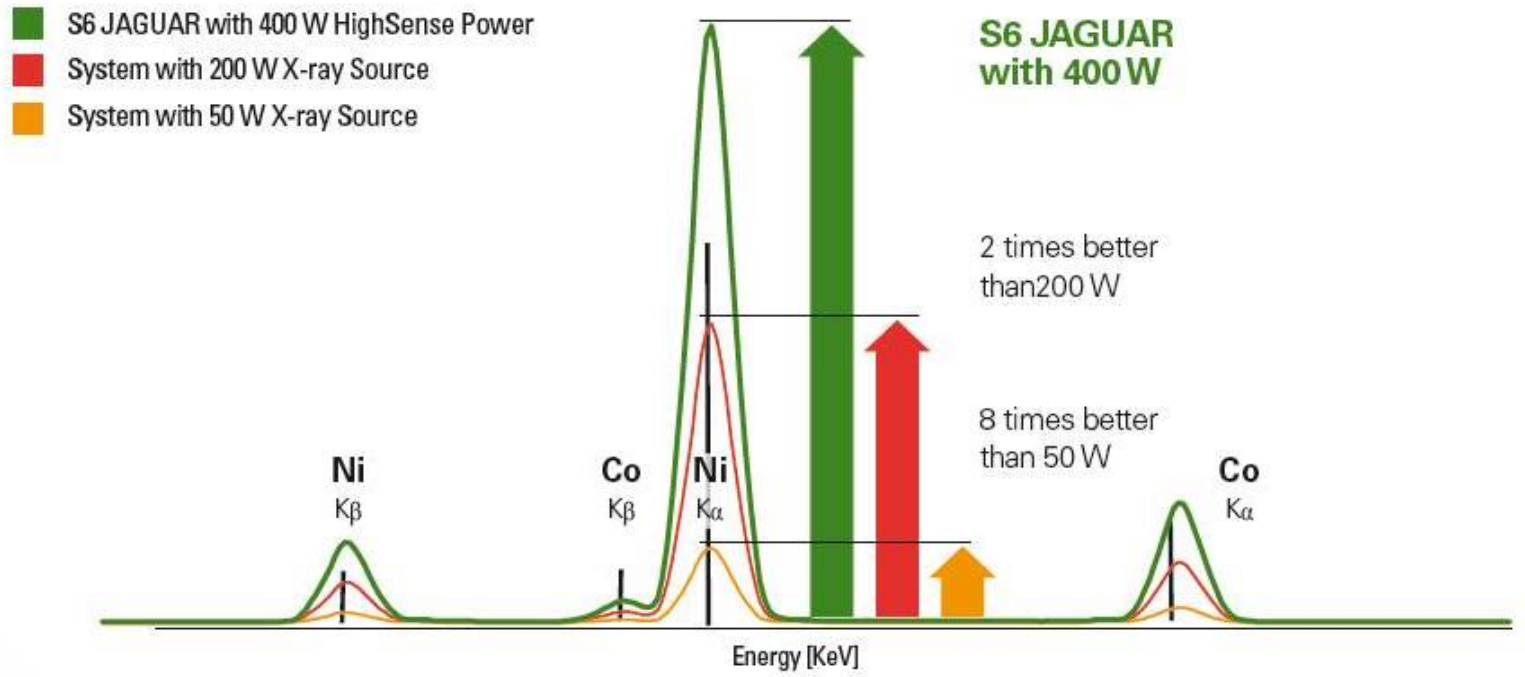
The significantly higher sensitivity leads to more than

- 30% better detection limits
- 50% enhanced light element analysis

→ full 400 W power at low kV

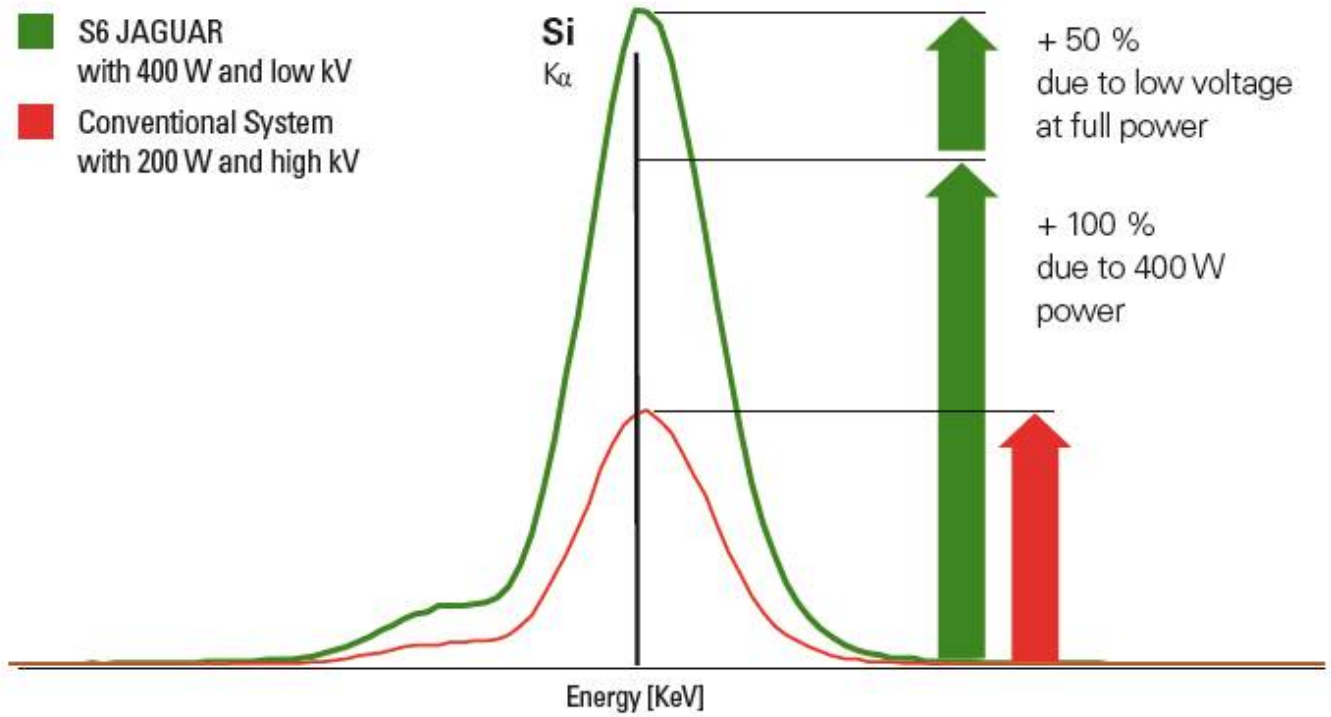
*compared to 200 W

S6 JAGUAR
HighSense™: Full 400 W Excitation Power



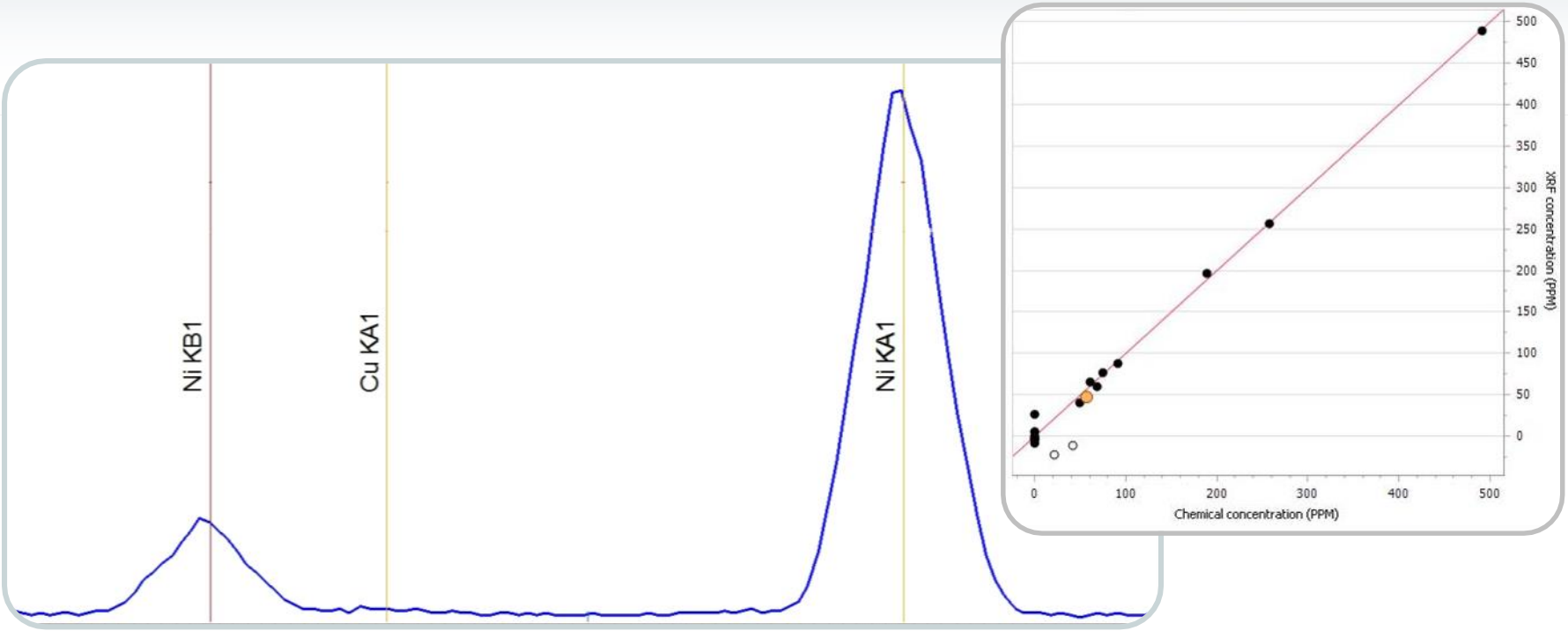
S6 JAGUAR is twice as powerful as a 200 W system and 8 times more powerful than a 50 W system

S6 JAGUAR
HighSense™: Light Element Performance



S6 JAGUAR analyzes light elements with optimal low voltage and full 400 W power settings!

S6 JAGUAR in Minerals & Mining
Nickel Ore – Resolution by Crystals



Separation of Cu Kα from Ni Kβ 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

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

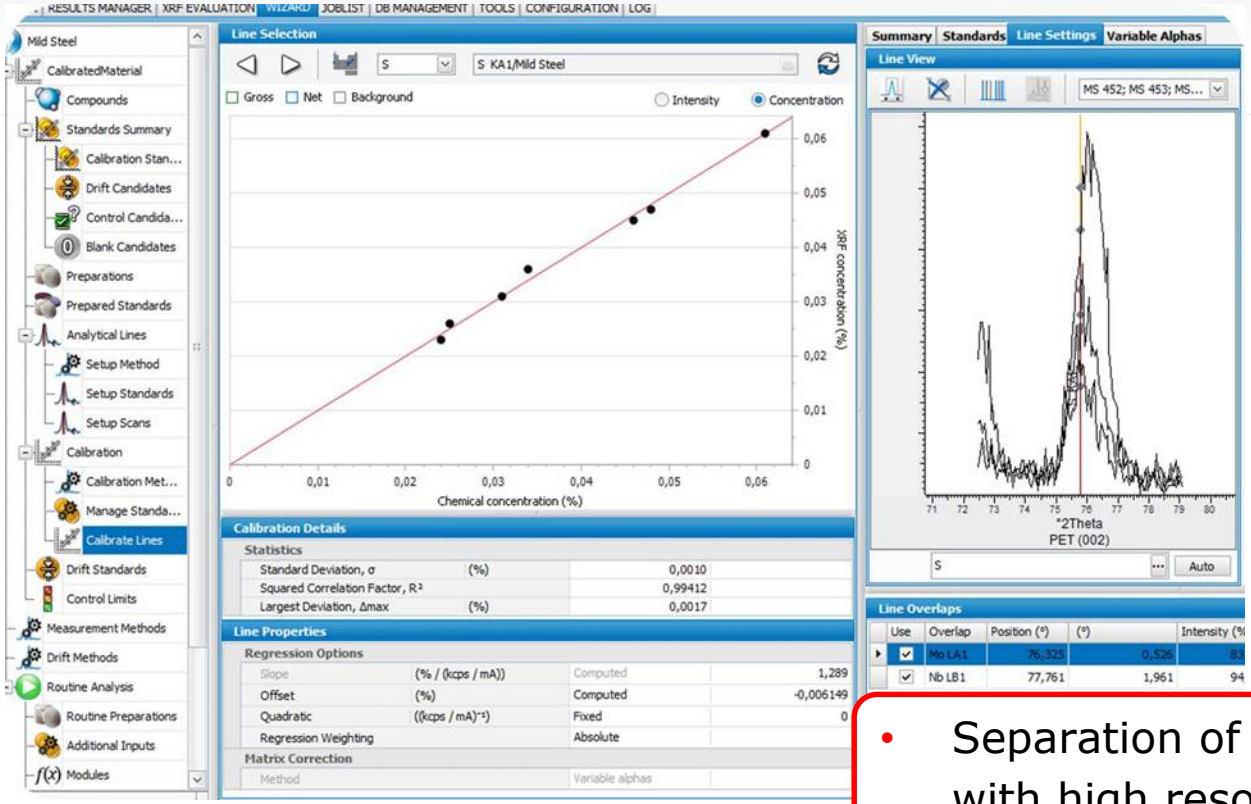
Impurities with negative impact on steel quaiily:

Al, Si, P, S, Cu, As, Sb, Pb

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

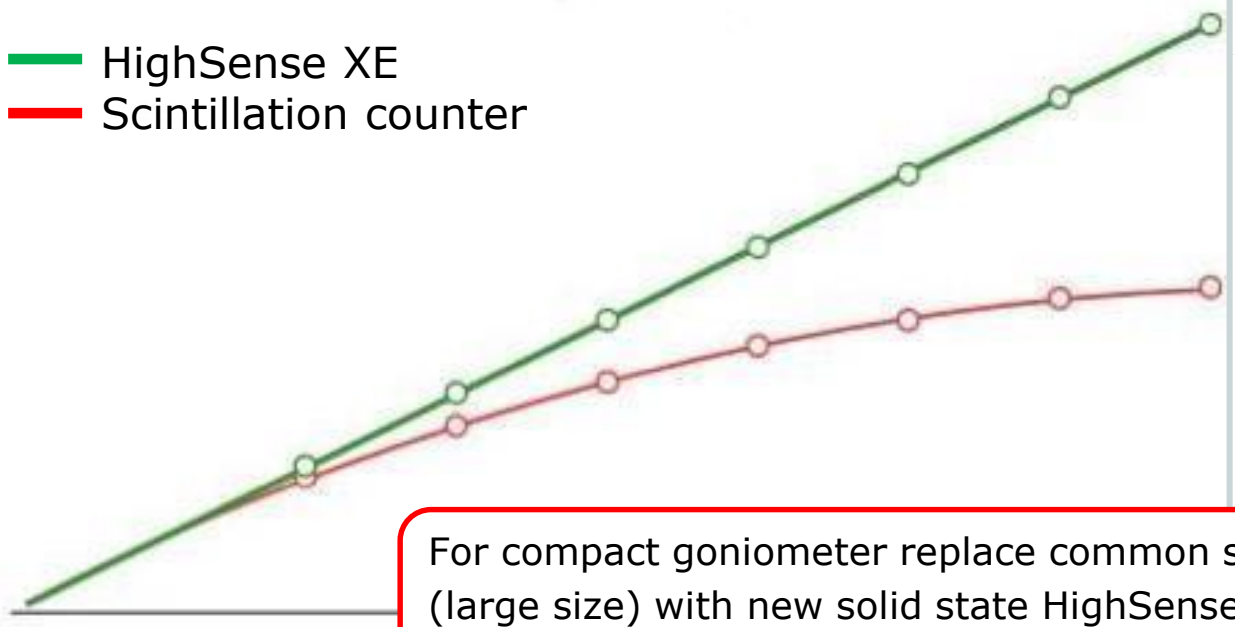
Current WDXRF spectrometers are suffering for major elements due to saturation of the scintillation counter. Sum, escape peaks and crystal artefacts will even worsen the situation (Pulse Height Distribution)

S6 JAGUAR in Metals & Slags
Low Alloy Steels



- Separation of S Kα from Mo Lα with high resolution WDXRF
- Handling of line overlaps and FP matrix correction with variable alphas

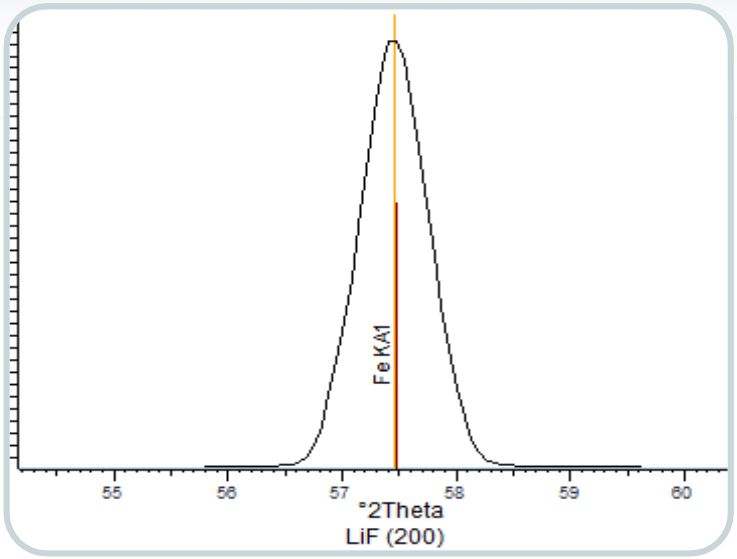
S6 JAGUAR
HighSense XE™: Unrivalled Linearity

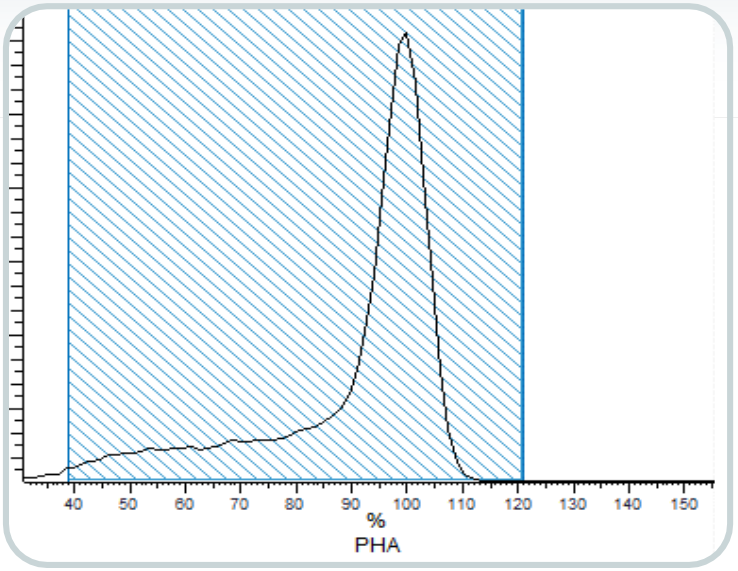


For compact goniometer replace common scintillation detector (large size) with new solid state HighSense XE detector:

- State-of-the-art technology
- Medium and heavy element detection
- Linear range of 2 Mcps for wide range calibrations
- 2 times better energy resolution than scintillation counters
- Significant reduction of element interferences and crystal fluorescense

S6 JAGUAR with HighSense XE
Advances in Detector Technology





HighSense XE detector technology offers unique advantages especially for high concentrations of medium and heavy elements:

- High linearity of more than 10 Mcps due to no dead time – no saturation of the detector like any scintillation counter (typically 400 kcps, with dead time compensation 1.5 Mcps)
- Two times better energy resolution overcomes interferences with sum and escape peaks, as well as crystal fluorescense signal in the PHA

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

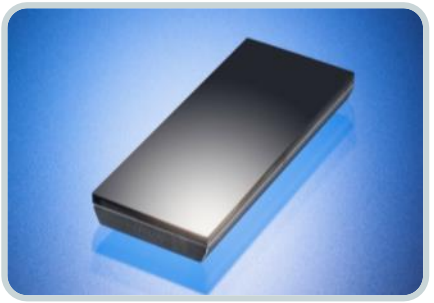
S6 JAGUAR
HighSense™ Goniometer: Up to 4 Crystals

The entire element range is covered with 3 analyzer crystals:

- XS-55 for F –Mg
- PET for Al – Cl
- LiF200 for K – Am

Oil, Refineries, Automotive

- Germanium or curved Germanium crystal to boost sample throughput for P and S, e.g. for ASTM D 2622

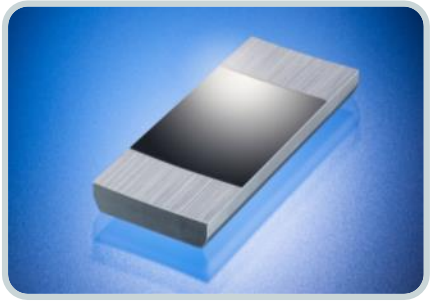


Minerals, Mining, Metals

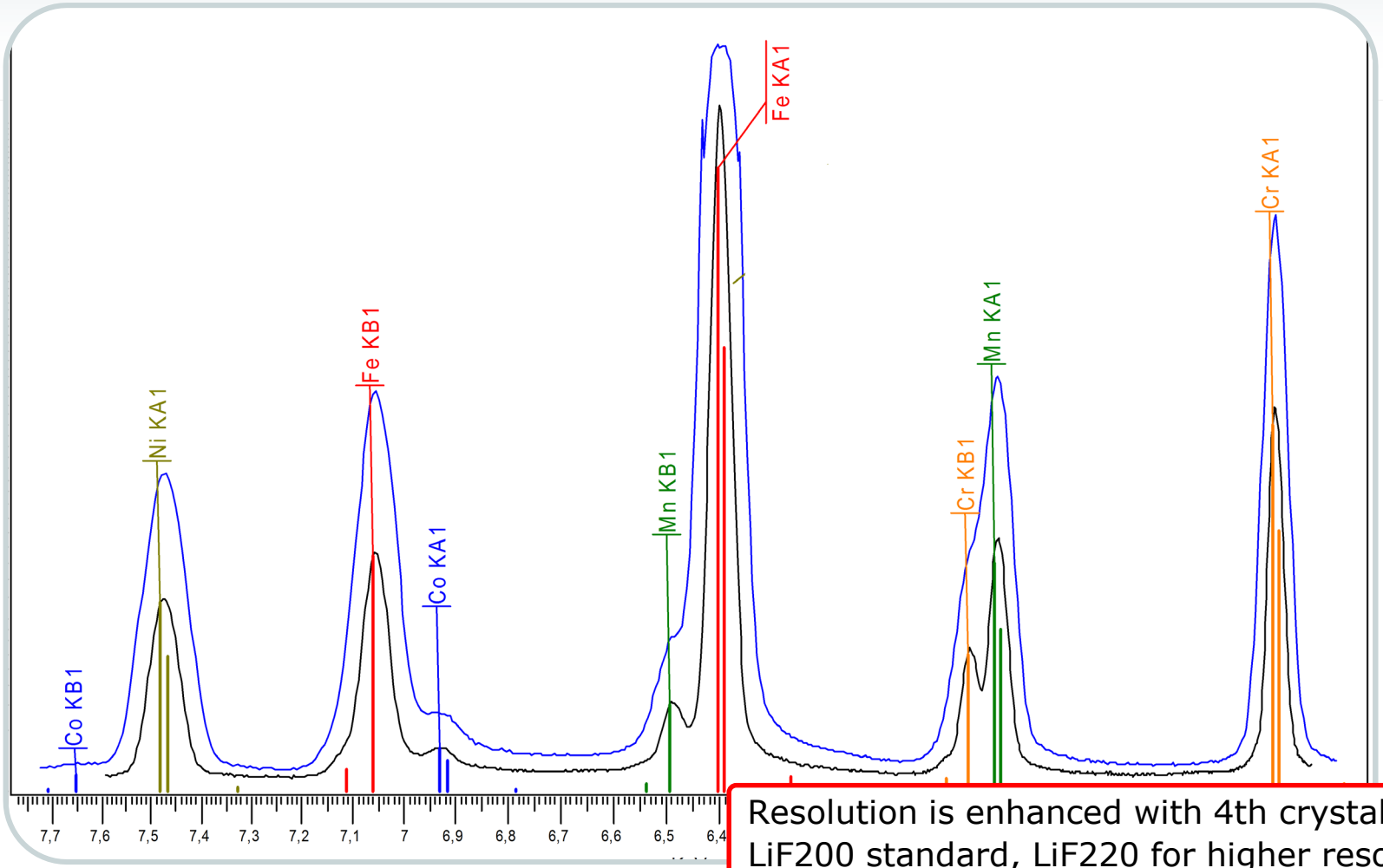
- XS-400 to add 35% more intensity for elements from K – Am

Geology, Academia, Research, Metals

- LiF 220 for better resolution and detection for trace elements from V – Am

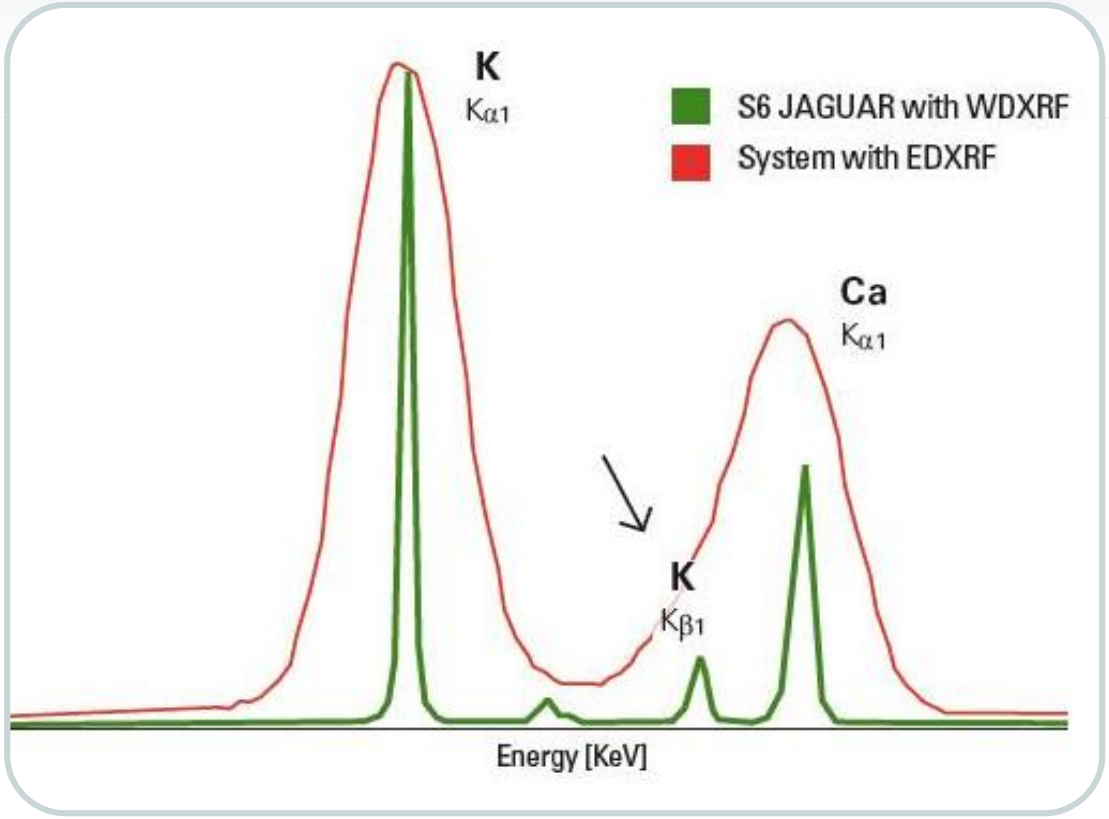


S6 JAGUAR
HighSense™ Goniometer: Up to 4 Crystals



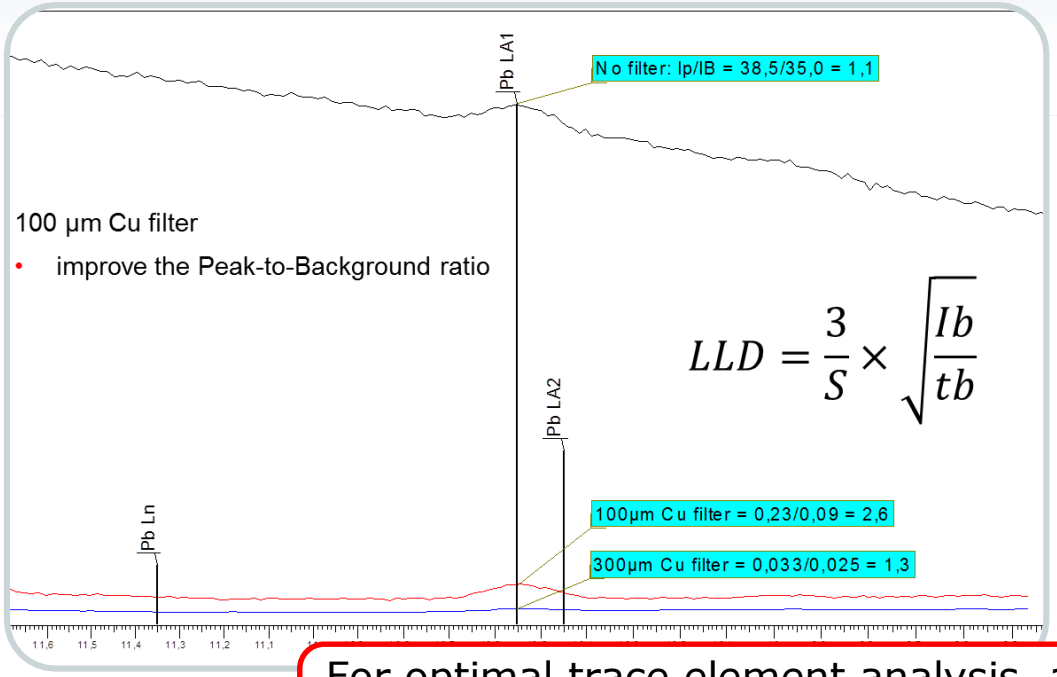
Resolution is enhanced with 4th crystal:
LiF200 standard, LiF220 for higher resolution

S6 JAGUAR
HighSense™ Goniometer: High Resolution



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

S6 JAGUAR
HighSense™ Goniometer: Primary Beam Filters



For optimal trace element analysis, a set of different primary beam filters is required:

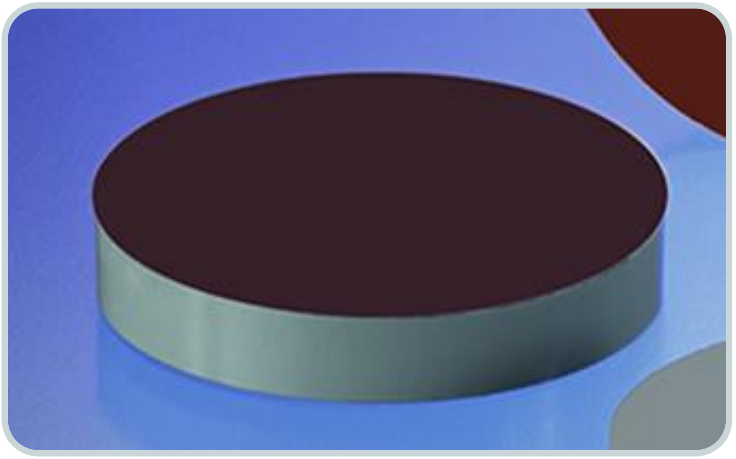
- No filter -> High background leads to bad LLD‘s
- 100 µm Cu filter -> ideal for Pb, good ratio between reduction of background and maintaining peak sensitivity
- 300 µm Cu filter -> works for Cd, but worse situation for Pb -> lowest background, but no peak left

S6 JAGUAR in Minerals & Mining
Coal, Coke and Carbon

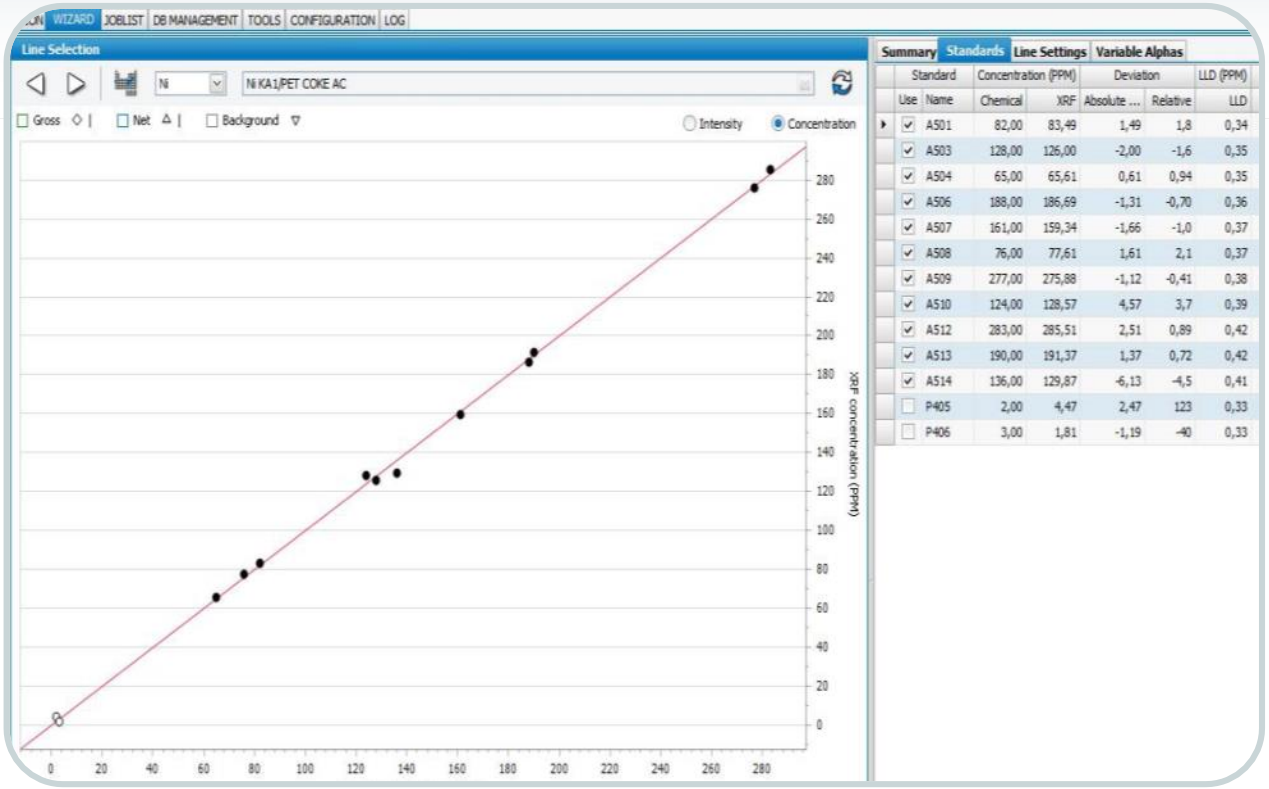
Coal is widely used in metal making (aluminium, steel) and power generation (electricity)

Analysis of coal, coke and carbon products is vital:

- Prevent contamination of metals (impurities)
- Inhibit corrosion (monitoring of Cl)
- Reduce environmental impact (reducing S concentration)



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

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

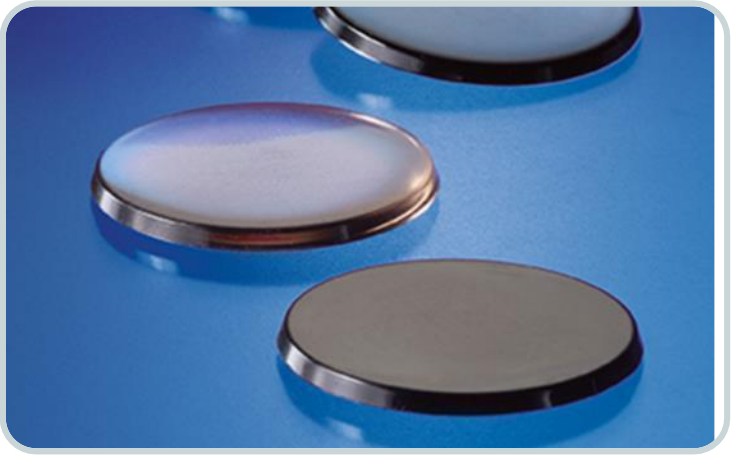
Optimal accuracy for
- minor elements, such as S
- traces, such as Si, Cl, Fe

S6 JAGUAR in Cement ASTM C114

Quality control of cements according to ASTM C114 and ISO 29581-2 / DIN EN 196-2

- Determination of the elemental composition
- Analysis of major elements:
  - Ca, Al, Si, S, Fe as oxides
- Evaluation of minors
  - Na, Mg, P, K, Ti, Cr, Mn, Zn, Sr

- Fused bead preparation for optimal accuracy eliminating mineralogical effects



S6 JAGUAR in Cement ASTM C114

Compound	Average [%]	Difference between Duplicates [%]	Max. Difference acc ASTM C 114 [%]		Certified Concentration [%]	Difference between average of duplicates and certified concentration [%]	Max. Difference acc ASTM C 114 [%]	
Na_2O	0.09	0.01	0.03	√	0.093	0.00	0.05	√
MgO	1.19	-0.01	0.16	√	1.196	-0.01	0.20	√
Al_2O_3	5.25	-0.01	0.20	√	5.271	-0.02	0.20	√
SiO_2	20.69	0.10	0.16	√	20.766	-0.07	0.20	√
P_2O_5	0.25	0.00	0.03	√	0.248	0.00	0.03	√
SO_3	27.39	0.02	0.10	√	2.756	-0.02	0.10	√
K_2O	0.66	0.00	0.03	√	0.657	0.00	0.05	√
CaO	65.30	0.05	0.20	√	65.247	0.05	0.30	√
TiO_2	0.24	0.00	0.02	√	0.240	0.00	0.03	√
Cr_2O_3	0.02	0.00			0.020	0.00		
Mn_2O_3	0.20	0.00	0.03	√				
Fe_2O_3	3.75	0.01	0.10	√				
ZnO	0.01	0.00	0.03	√				
SrO	0.03	0.00						

S6 JAGUAR easily passes precision and accuracy test for ASTM C114, ISO 29581-2 / DIN EN 196-2

- 400 W excitation power
- Closely coupled HighSense goniometer: optimal sensitivity

S6 JAGUAR
CEMENT-QUANT

- CEMENT-QUANT
  - A complete solution for cement industry for
    - Raw meal mix
    - Cement
    - Clinker
    - Deliveries from quarry
  - 20 Reference Materials
  - Covers the 12 most important elements
- In accordance with ASTM C114 and ISO 29581-2 / DIN EN 196-2

S6 JAGUAR in Food & Feed
Milk Powder

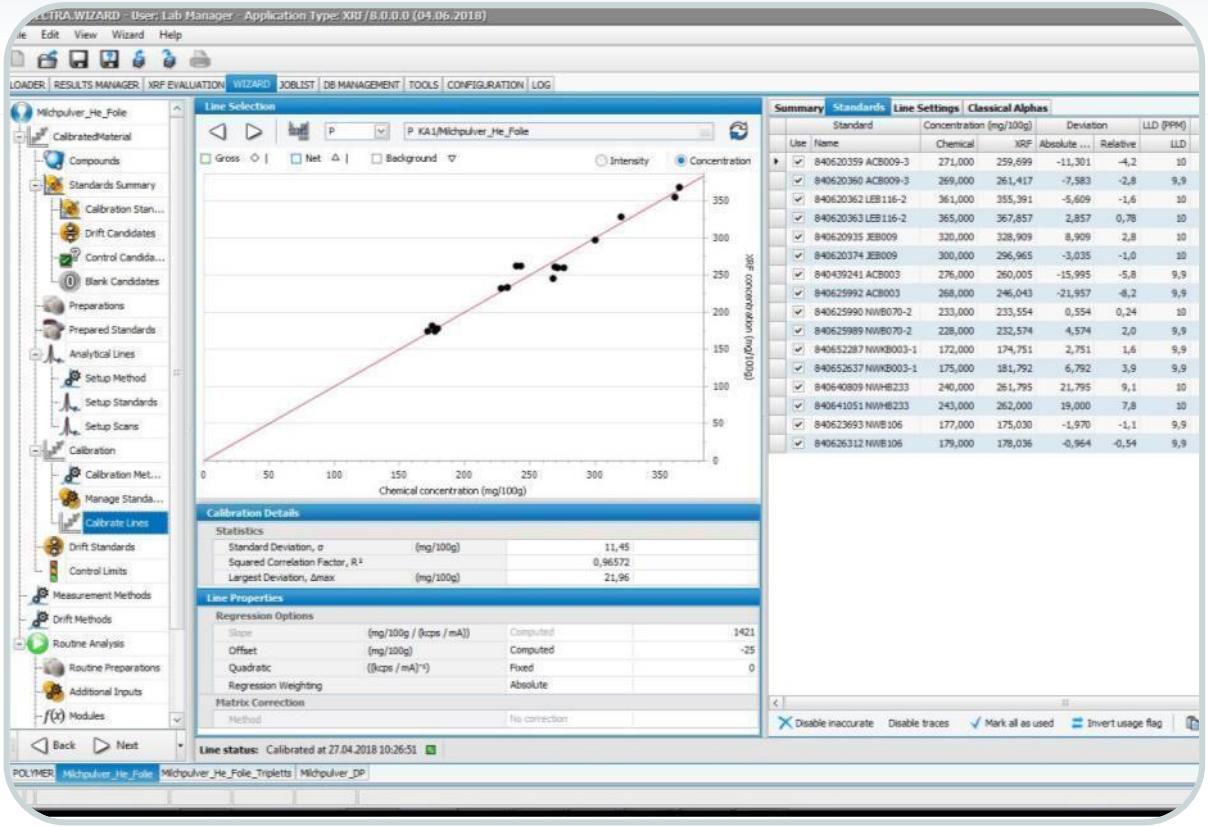
Quality control of milk powder for vital elements

- Product qualification for infant nutrition
- Critical -> single nutrients source for a longer period

- Vital elements: Na, Mg, P, Cl, K, Ca, Fe

- Quick sample preparation as pressed pellet
- Quick analysis ~ 5-6 minutes

S6 JAGUAR in Food & Feed
Milk Powder



Linear calibration for traces of P in milk powder

S6 JAGUAR in Food & Feed
Milk Powder

	Na [mg/100 g]	Mg [mg/100 g]	P [mg/100 g]	Cl [mg/100 g]	K [mg/100 g]	Ca [mg/100 g]
	455.5	83.8	263.9	472.5	582.7	436.0
	455.1	83.0	266.3	473.1	584.7	434.7
	458.7	84.4	265.7	473.4	585.0	436.2
	451.6	82.0	266.6	470.7	586.9	437.9
	450.7	82.6	266.6	471.8	586.9	437.1
	460.0	85.3	267.3	473.3	586.7	436.7
	460.6	84.1	266.0	472.8	588.5	438.4
	460.4	85.2	268.7	476.7	587.6	438.4
Mean Value [mg/100g]	456.6	83.8	266.4	473.0	586.1	436.9
Abs. Std. Dev. [mg/100g]	3.9	1.2	1.4	1.7	1.8	1.3
Rel. Std. Dev. [%]	0.86	1.46	0.51	0.37	0.31	0.29

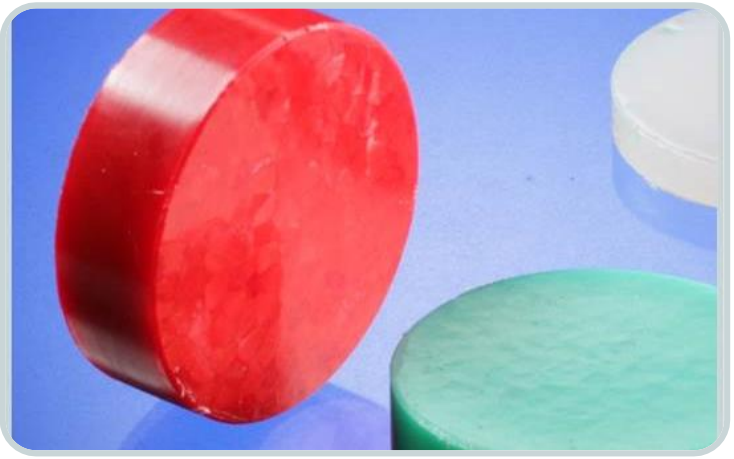
- Accurate and precise analysis of milk powder for quick quality control with S6 JAGUAR
- Less than 2% rel std deviation for all elements

S6 JAGUAR in Petrochemistry
Additives in Polymers

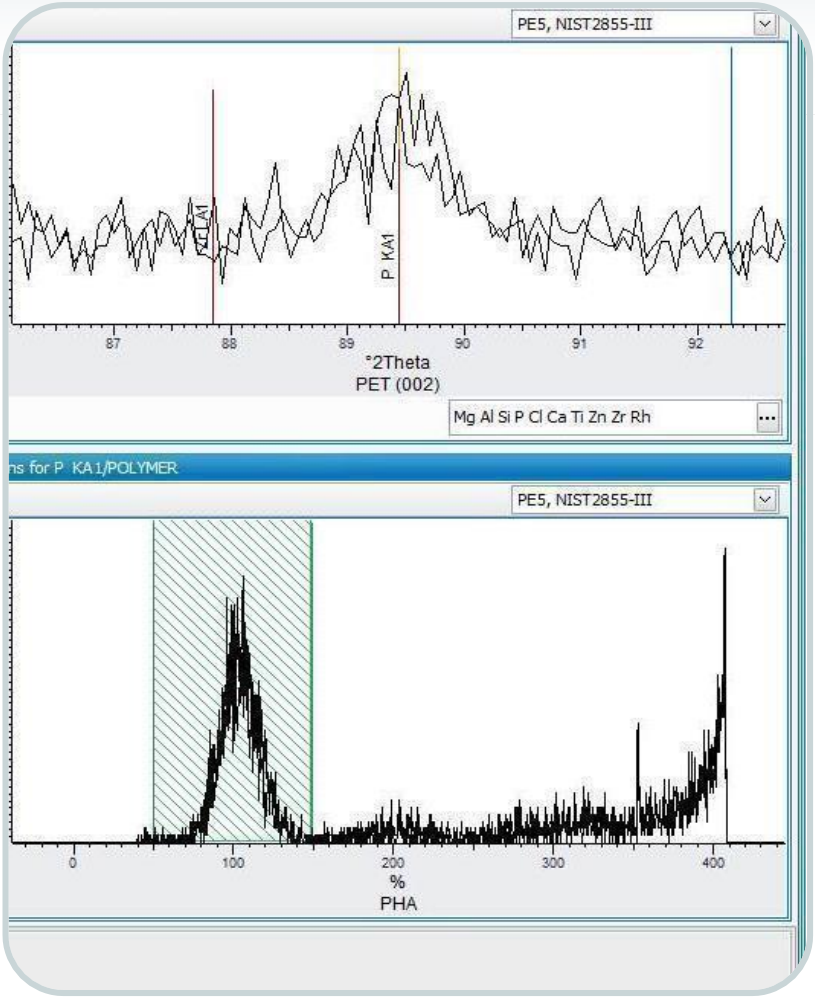
Analysis of additives and impurities in virgin polymers:

- Mg, Al, Si, P, Cl, Ca, Ti, Zn Zr in the lower ppm range
- Aim: High accuracy in the trace element region

- Direct analysis of solid specimen (PUK) after hot pressing
- Analysis of granules in liquid cups



S6 JAGUAR in Petrochemistry
Additives in Polymers



Set up scans for P Kα in polymers

- High resolving WDXRF separates Zr Lα from P Kα
- High power setup with closely coupled beam path leads to optimal detection limits
- Low temperature tube head maintains samples (no decomposition)

S6 JAGUAR in Petrochemistry
Additives in Lubricating Oils

Analysis of additives in lubricating oils to ensure proper oil performance

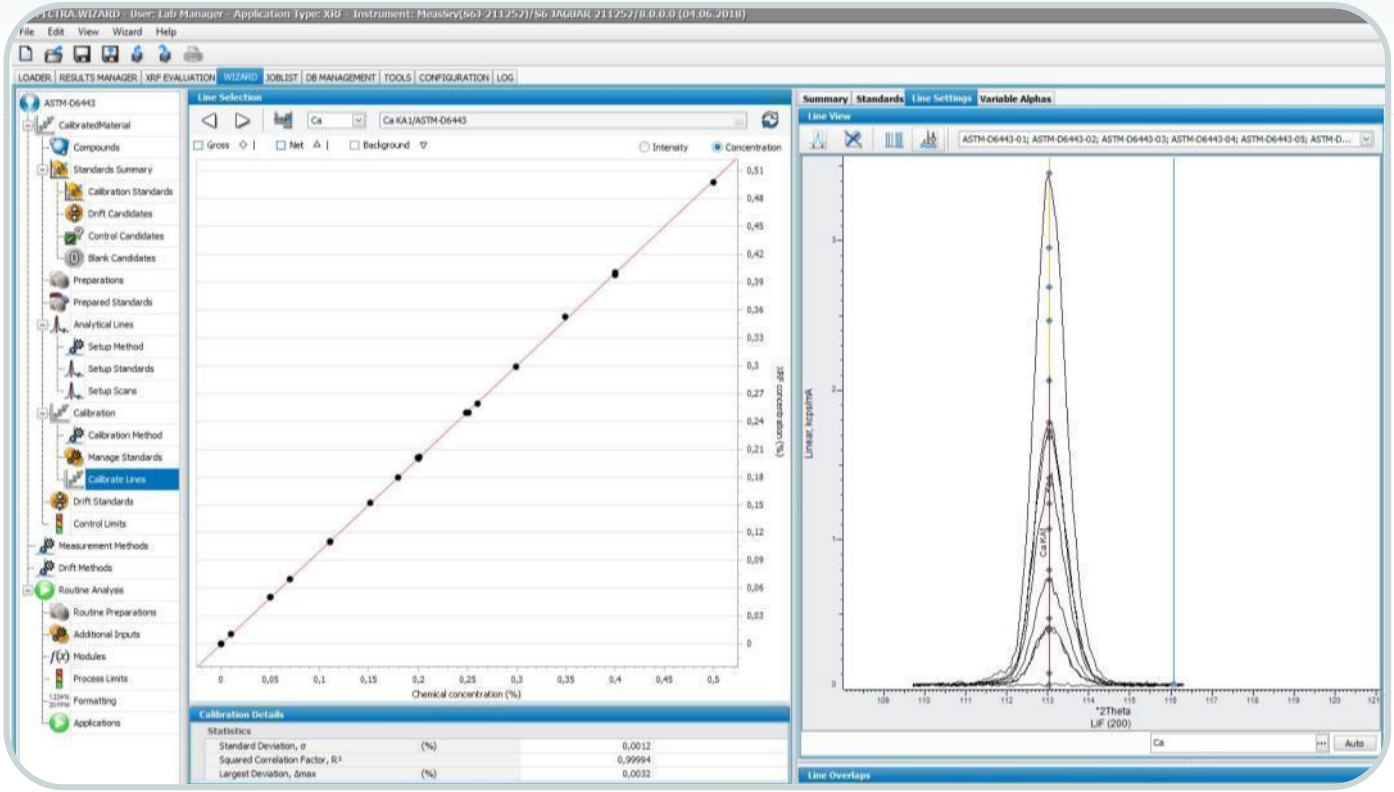
According to ASTM D 6443 for Ca, Cl, Cu, Mg, P, S, and Zn

Cost reduction possible with highly accurate and precise analysis

Blending of oils close to specifications

Cost savings: expensive additives

S6 JAGUAR in Petrochemistry
Additives in Lubricating Oils



Ca calibration curve with setup scans
Squared. Corr. Coeff: 0.99994
LOD: 2.4 ppm

S6 JAGUAR in Petrochemistry
Additives in Lubricating Oils

	Mg (%)	P (%)	Cl (%)	Ca (%)	Cu (%)	Zn (%)
# 1	0,077	0,049	0,050	0,201	0,020	0,050
# 2	0,077	0,050	0,051	0,201	0,020	0,050
# 3	0,079	0,050	0,050	0,200	0,019	0,050
# 4	0,078	0,049	0,050	0,200	0,020	0,050
# 5	0,078	0,050	0,051	0,201	0,020	0,050
# 6	0,078	0,050	0,050	0,201	0,019	0,050
# 7	0,078	0,050	0,050	0,202	0,019	0,050
# 8	0,080	0,050	0,050	0,202	0,019	0,050
# 9	0,078	0,050	0,051	0,201	0,020	0,050
Mean Value	**0,078**	**0,050**	**0,050**	**0,201**	**0,020**	**0,050**
Std. Dev.	**0,0009**	**0,0003**	**0,0004**	**0,0006**	**0,0004**	**0,0001**
Rel. Std. Dev.	**1,1**	**0,5**	**0,8**	**0,3**	**1,9**	**0,3**

Excellent reproducibility and high analytical precision
- Less than 1% rel. per element
- Light elements about 1% rel.
- Traces at 2% rel dev.

X-ray fluorescence analysis (XRF)
Capabilities

Samples measured as

- Liquids
  - directly
- Powders
  - directly
  - as pressed pellets
  - as fused beads
- Bulks
  - Directly, after fitting into sample cups

S6 JAGUAR
Manual: Quick Sample Change

- Easy access to large sample chamber
- For all kinds of samples:
  - Solids
  - Pressed powders
  - Fused beads
  - Liquids with helium mode
  - Loose powders in cups with helium
- Economical option for lower sample throughput

S6 JAGUAR
EasyLoad™: High Productivity

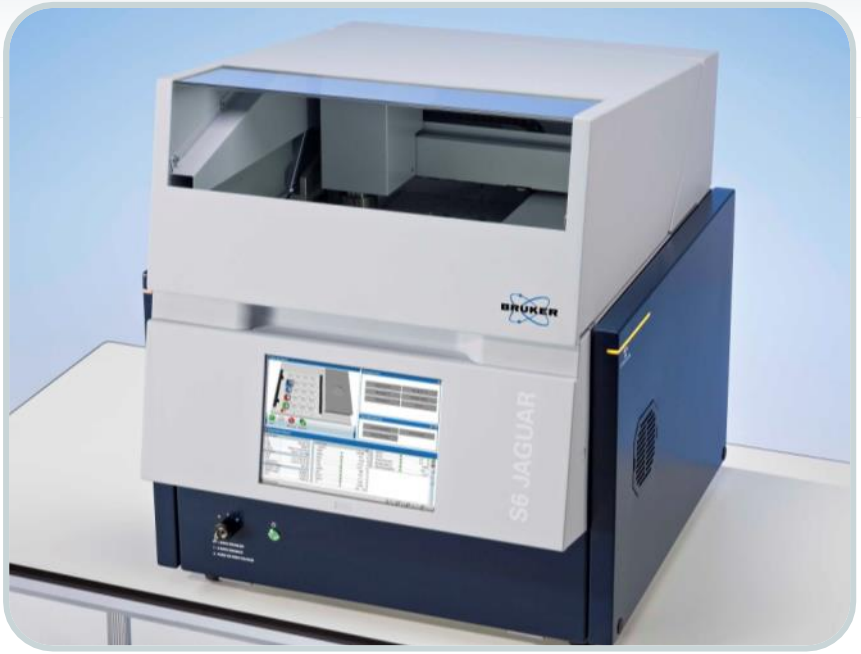
Unattended operation of sample batches for high throughput

- Loading of new samples at any time
- Quick analysis of high priority samples
- Loading of entire batches just by exchanging complete trays

- EasyLoad 24 Positions
  - 20 positions on a tray (51.5 mm rings)
  - 4 fixed positions for QC samples
  - Automatic liquid sample recognition

- EasyLoad ONLINE 22 Positions
  - 20 positions on a tray (51.5 mm rings)
  - 2 fixed positions for QC samples
  - Automatic liquid sample recognition
  - Automation for robot and belt

- XY Autochanger 15 Positions
  - 15 positions for two-part sample cups



S6 JAGUAR
TouchControl™: Ease-of-use

Intuitive interface: Three steps to accurate results

1) Select sample position and application
2) Enter sample ID
3) Press “Measure”

- No operator training required
- Standalone operation (no PC): IslandMode
- Remote access via TCP/IP:
  - Routine with TouchControl: Calibration, evaluation, and reporting remotely
- Languages: English, German, French, Spanish, Italian, Portuguese, Russian, Chinese, Japanese, Bahasa

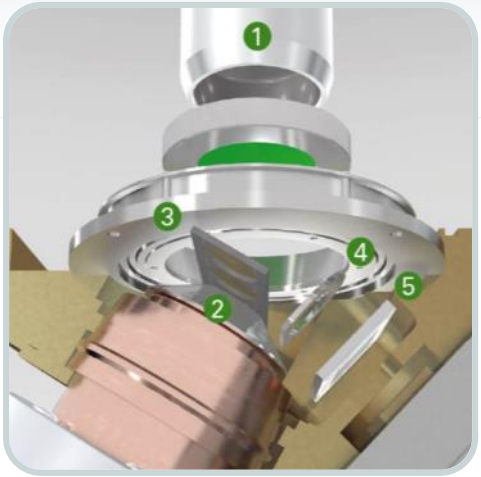
S6 JAGUAR
SampleCare™: High Instrument Uptime

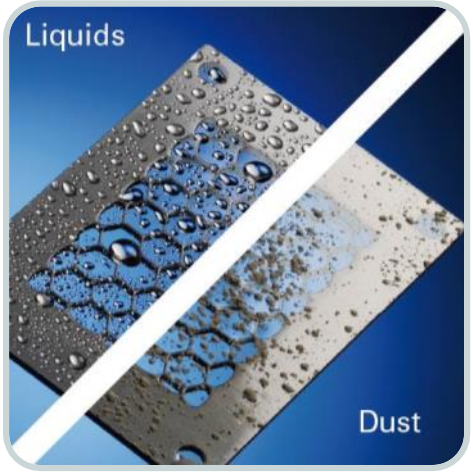
Low cost of operation

- High instrument uptime due to unique protection during loading and unloading
- Two contamination shields to protect tube window and goniometer
- Unique Vacuum Seal with high transmission window for goniometer protection

- Low helium consumption
  - Flushing of sample chamber only
  - Goniometer chamber remains in vacuum all the time

1. Grabber with automatic sample detection
2. Tube shield
3. Filter changer
4. Mask holder
5. Vacuum seal





Unique High Transmission Vacuum Seal

S6 JAGUAR
Recent Developments in Benchtop WDXRF

The all-new S6 JAGUAR combines WDXRF performance with compact size and overcomes cost issues, being an affordable WDXRF:

- Maintain WDXRF resolution, especially for applications in minerals and metals with elements of interest with neighboring element lines
  (Mo Lα/S Kα; Co Kα/Fe Kβ)
- Work with higher power and better detectors for better counting statistics and higher precision for tighter process and quality control
- Maintain analytical flexibility with choice of analyzer crystals and sample masks

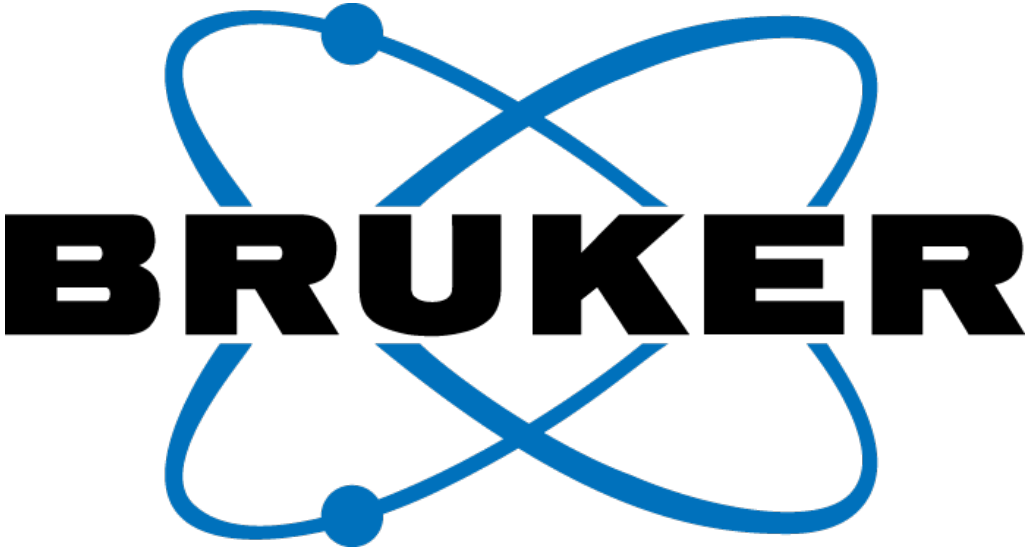
for

- Industry laboratories
- Research and academia

Q&A

Any questions?

Thank you!



Innovation with Integrity