WEBINAR 2021

New XRF Technology – Advances in Components for Better Data

Kai Behrens, Adrian Fiege and Frank Portala

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New XRF Technology – Advances in Components for Better Data

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Ph D of Analytical Chemistry, Hamburg

2001 Method Development Bruker AXS

Now Head of Product Management XRF

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Ph D, Geology

2019 Product Management Bruker AXS

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Outline

New XRF Technology – Advances in Components for Better Data

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:

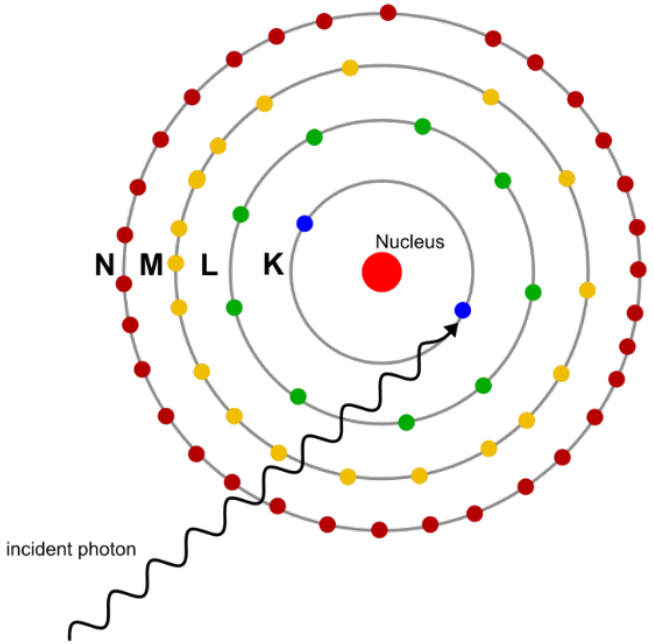
- Sand/Quartz: Construction, playgrounds, glass or solar cells, semicon wafers (Fe content)
- Coal: heating, power generation, electrodes for aluminium, electrodes for batteries (S, Cl, trace elements)
- Pt/Rh – jewelry, car catalysts, electrodes in fuel cells (Traces, thickness)
- Additives in lubricants and polymers – use the expensive additives sparsely while meeting the desired performance

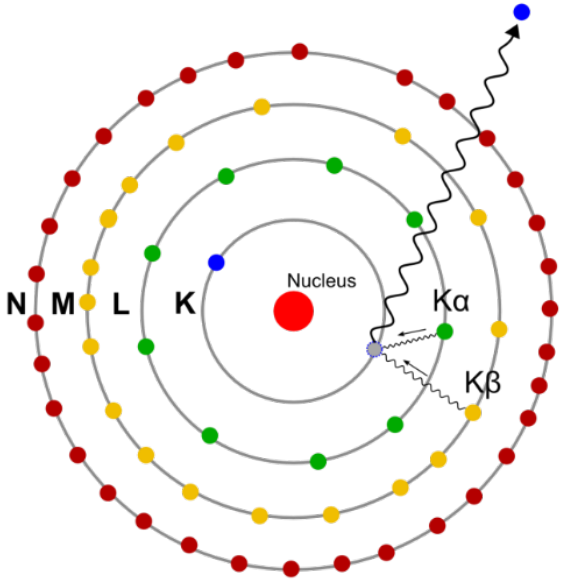
XRF X-ray Fluorescence Analysis X-ray Spectrometry

- ... is the method to do qualitative and quantitative analysis of elemental
- composition by excitation of atoms and detection of their characteristic
- X-rays: one form of "Electromagnetic Radiation"

Energy [keV]	Wavelength	Description
< 10^{-7}	cm to km	Radio waves
< 10^{-3}	μm to cm	Microwaves
< 10^{-3}	μm to mm	Infrared light
0.0017 - 0.0033	380 to 750 nm	Visible light
0.0033 - 0.1	10 to 380 nm	Ultraviolet light
0.1 - 100	**0.01 to 10 nm**	**X-rays**
10 - 5000	0.0002 to 0.12 nm	Gamma radiation

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

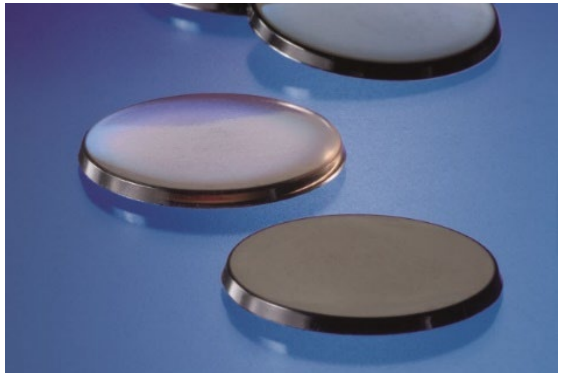
X-ray Fluorescence Analysis (XRF) Capabilities

- 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
- Quantitative and qualitative analyses are possible
- Accuracy and long-term stability
- Elemental range: (Be) Na to U
- Linearity from ppm to 100%

Preparation – Driven by Application

Fused beads – Best results for Major Element QC



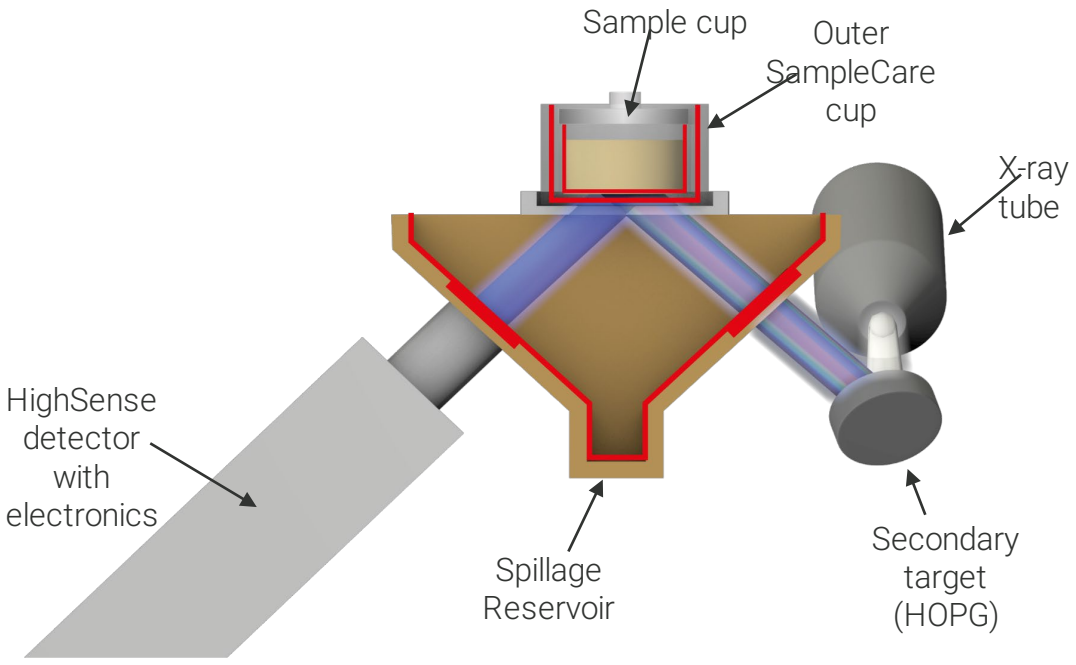
Powder – Fastest preparation, less precise

Pressed Pellets – Simple, fast, great for traces

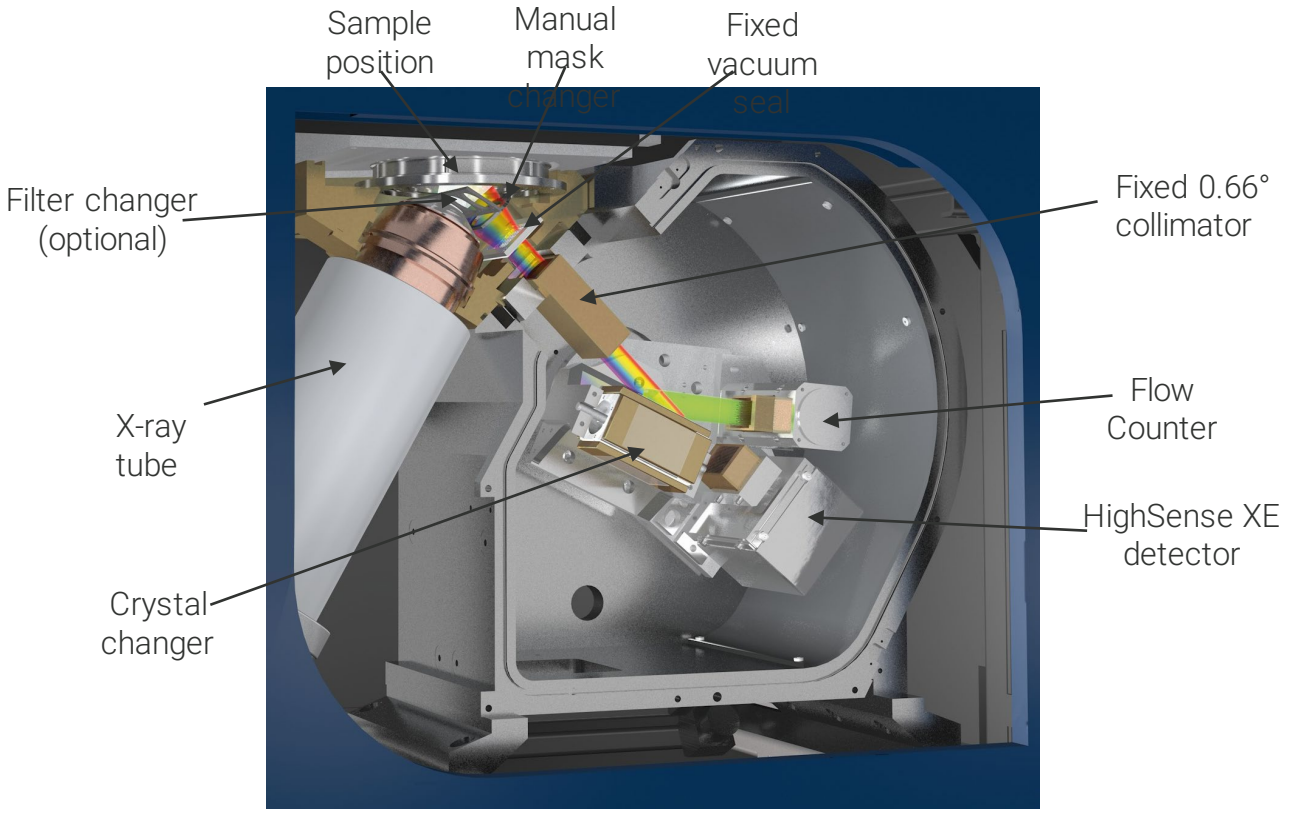
Liquid – Simple and reproducible

Energy-Dispersive XRF (EDXRF) vs. Wavelength-Dispersive XRF (WDXRF)

EDXRF

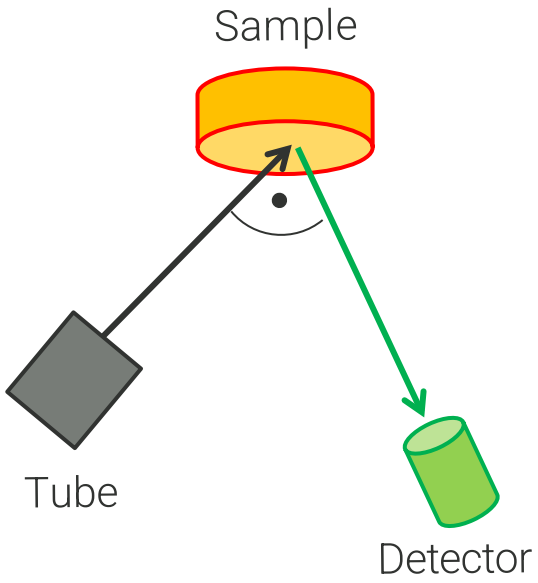


WDXRF

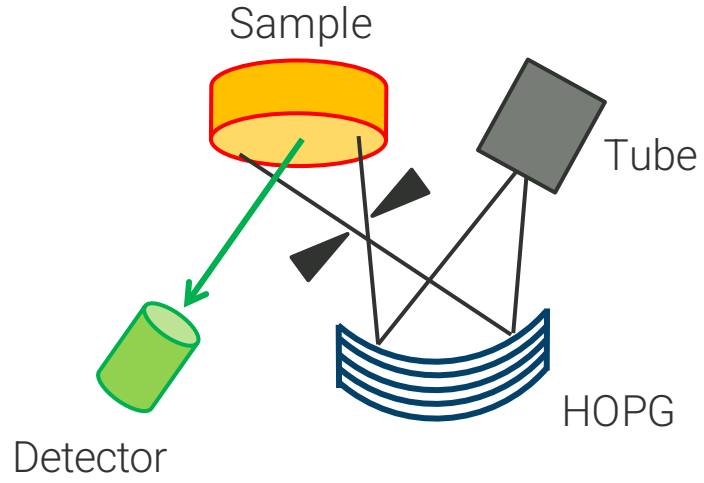


EDXRF Excitation Techniques
Direct Excitation vs. Polarized Excitation

S2 PUMA



S2 POLAR



Direct excitation:

- Leads to very high intensity
- But also to higher background intensity

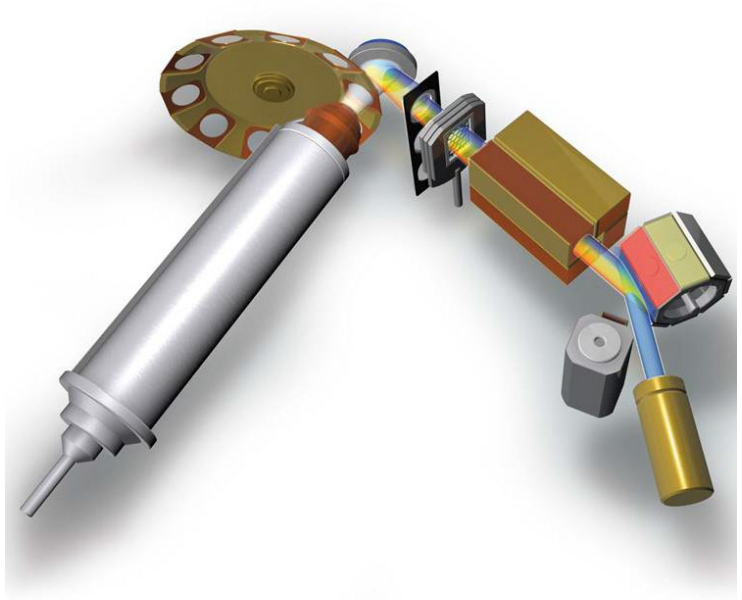
Polarized excitation:

- Reduced background due to polarization
- Improved signal to background ratio (better LLD)

Multi Purpose Sequential WDXRF Spectrometer S8 TIGER for the Central Lab

Analytical flexibility and high performance for sub-ppm traces

- 4 kW excitation
  20 – 60 kV
- 5 – 170 mA
- 10 beam filters
- 4 collimators
- 8 crystals
- 2 detectors



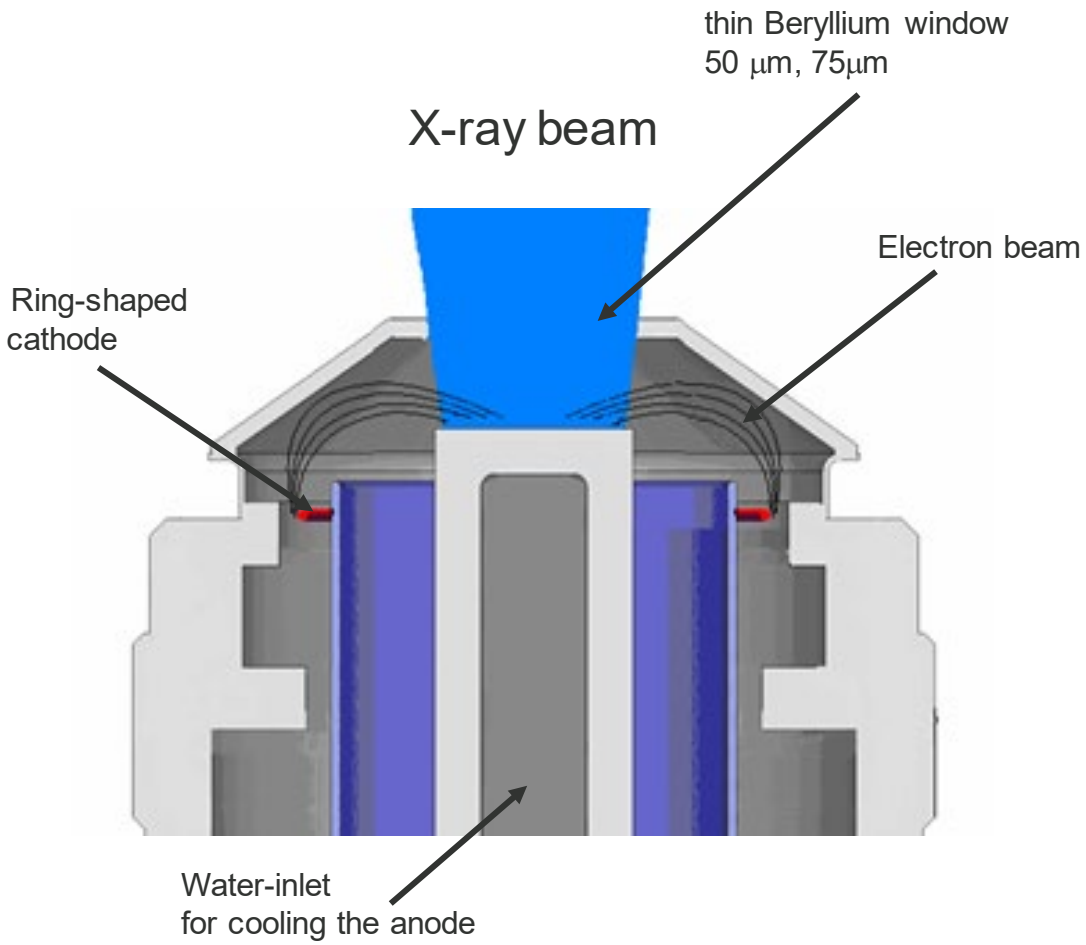
S8 TIGER WDXRF Beam path

Floorstanding WDXRF S8 TIGER

With 1, 3 or 4 kW

Instrument Parameters
The X-ray Tube

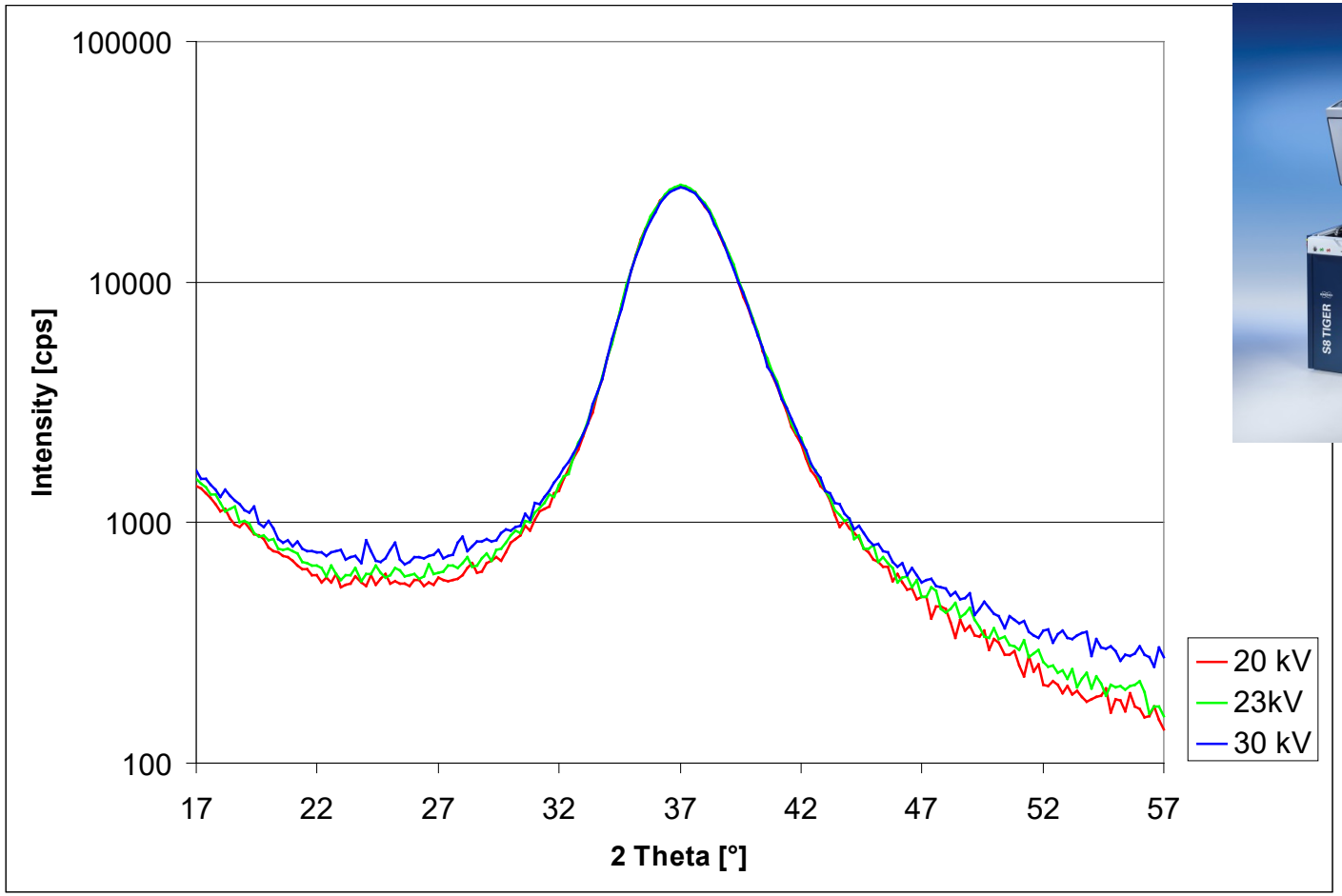
- Window material: Be 50 – 75 µm
- Tube power; excitation potential (kV) and the current (mA)
  - Production of primary X-rays
  - Depends on the elements binding energy. General:
    - High energy: high kV
    - Light energy: high mA
- Cooling by internal and/or external water circuit
  - 400 W, 1kW, 3kW and 4kW



Light Elements
S8 TIGER

Improve Peak to Background Ratio by low kV

- Low kV to maintain the sensitivity but increase Peak to Background ratio
- Lowering scattered primary beam
- High intensity X-ray tube – max. 170mA - 4 kW for best light element analysis e.g. B_2O_3
- P/BKG: 20 kV – 45
  23 kV – 42
  30 kV - 36

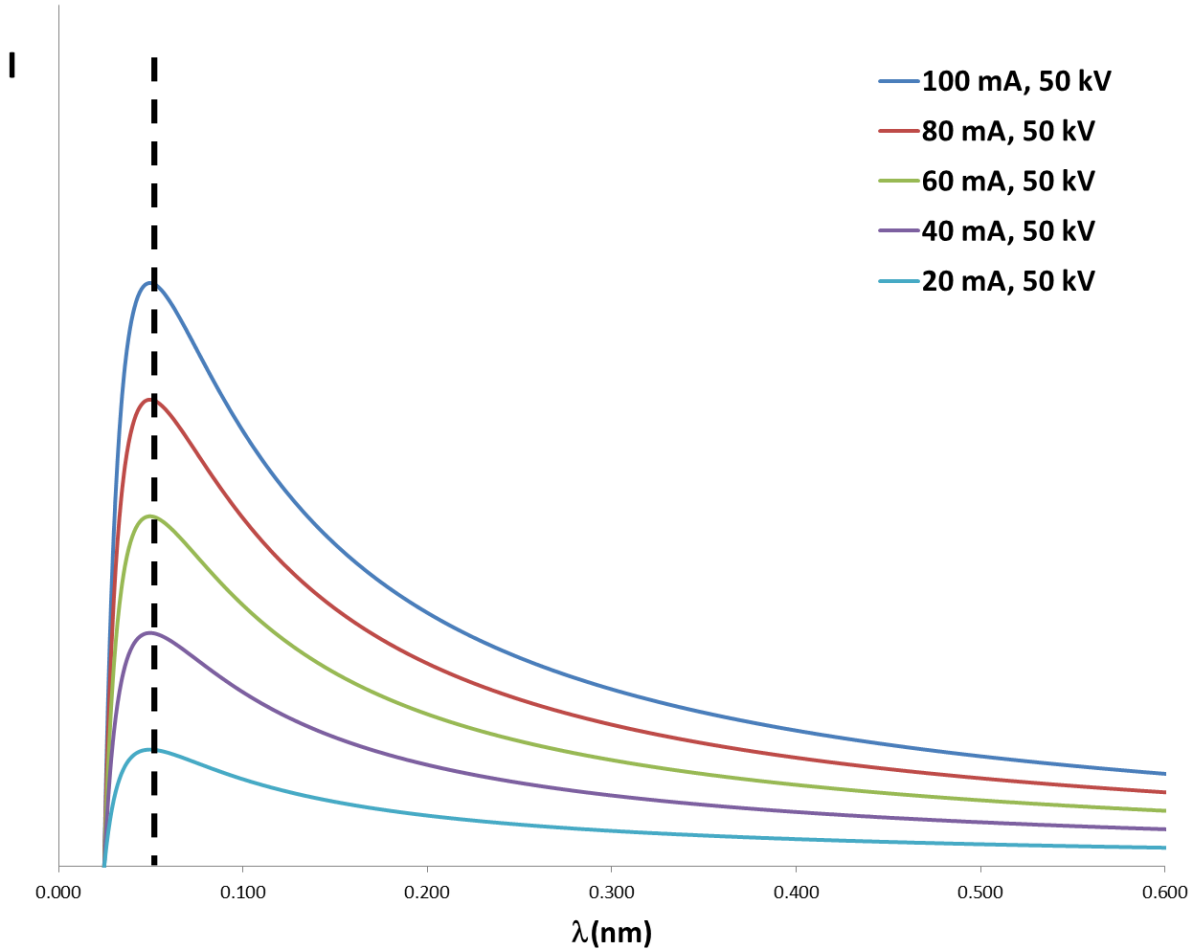


Instrument Parameters
The X-ray Tube – Power [mA]

Influence of power

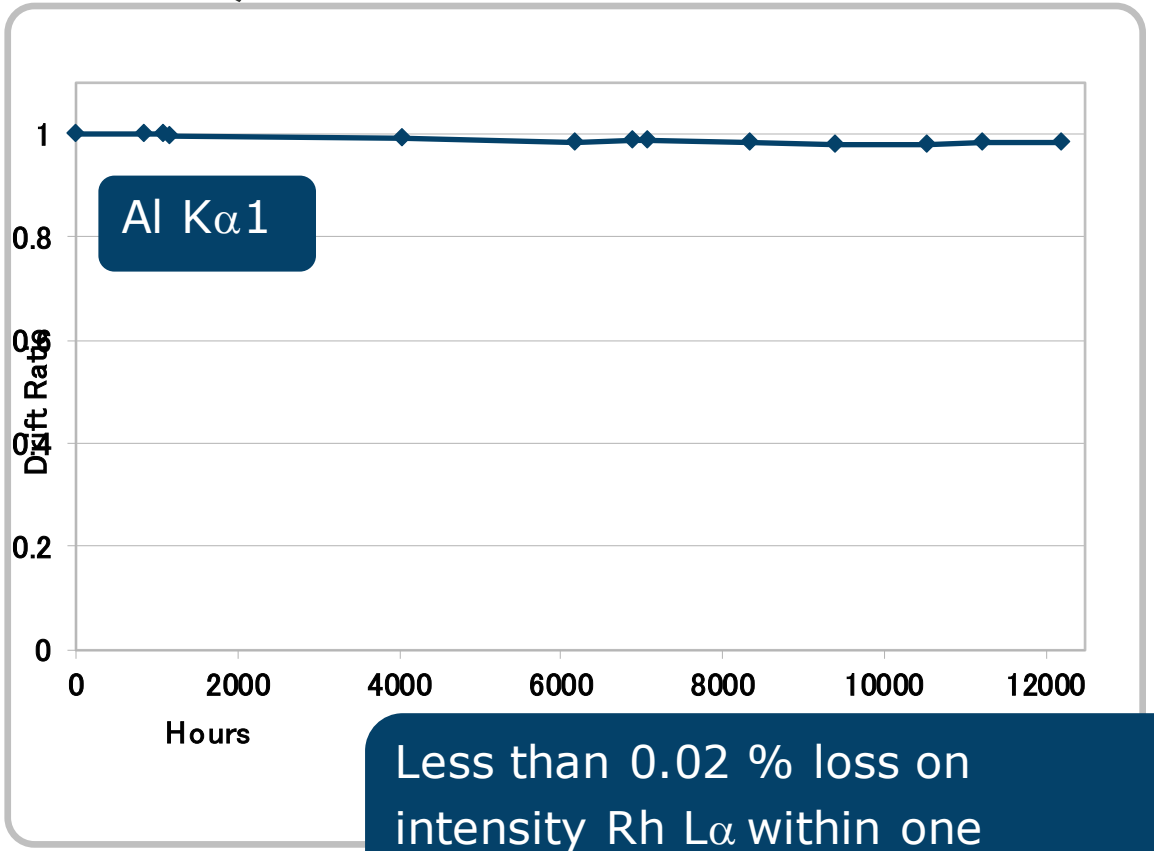
- Intensity scales up with linear with power increase (if geometry and components stay the same)
- Same counting statistics will lead to similar precision, so lower power will only require SQRT additional amount of measurement time
- Accuracy will not change at all
- LLD requires longer measurement time

$$I(\square) = KiZ\left(\frac{\lambda}{\lambda_{min}} - 1\right)\left(\frac{1}{\lambda^2}\right)$$



S6 JAGUAR - S8 TIGER
The Zero Drift Instrument with new X-ray Tube Technology

Observation: Zero Drift over one year for light elements, such as Al

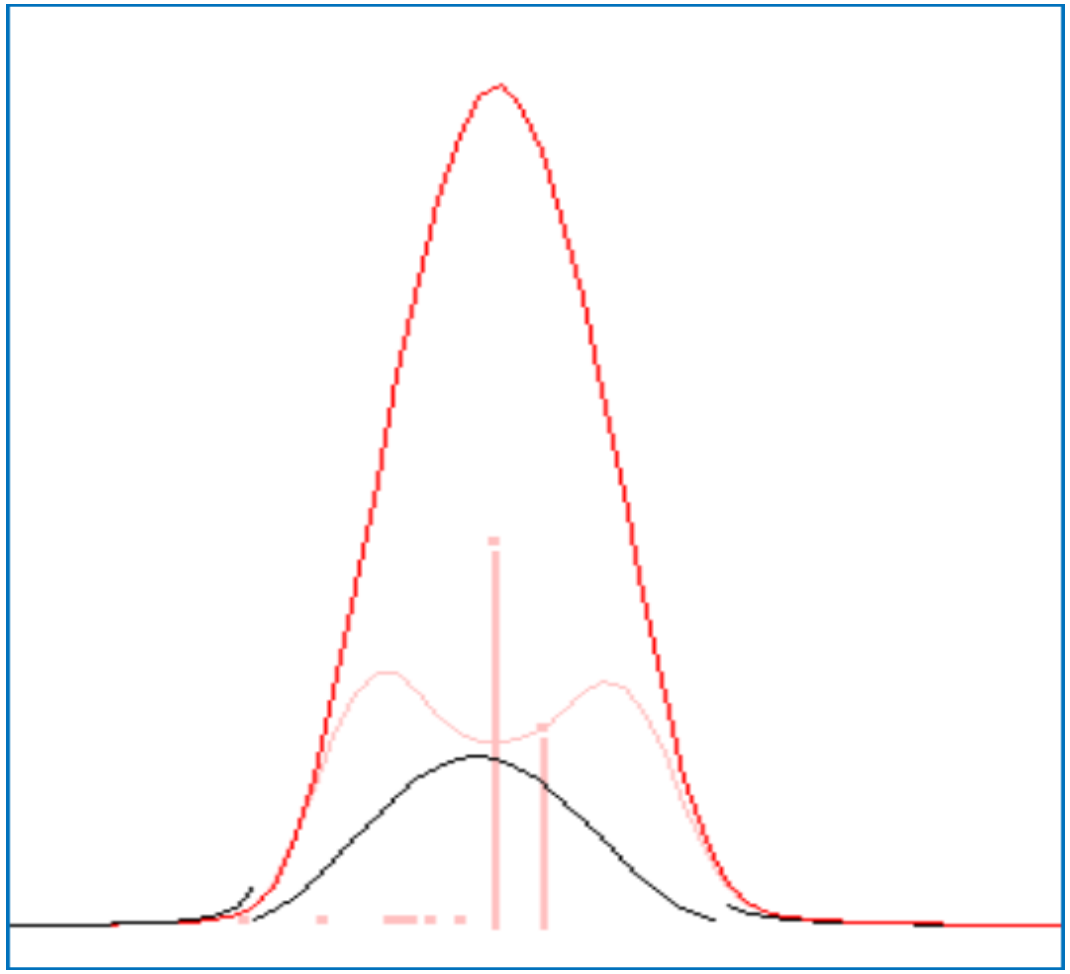


Less than 0.02 % loss on intensity Rh Lα within one year (Tube, Crystals, Detector)

Influence parameters on tube drift and recent changes

- Instrument drift depends on aging of components, such as tube, crystals, counters
  - Tube aging was biggest contributor due to W deposition on window (absorption), release from the W filament
  - Change of tube geometry (internal) shadows from free W to the tube window – no deposits, no aging
  - Non aging multilayer optics:
    - O, Na, Mg, F: XS-55
    - Al, SI: XS-CEM
    - P,S,Cl: Ge or XS-Ge-curved

DynaMatch™ Technology S8 TIGER

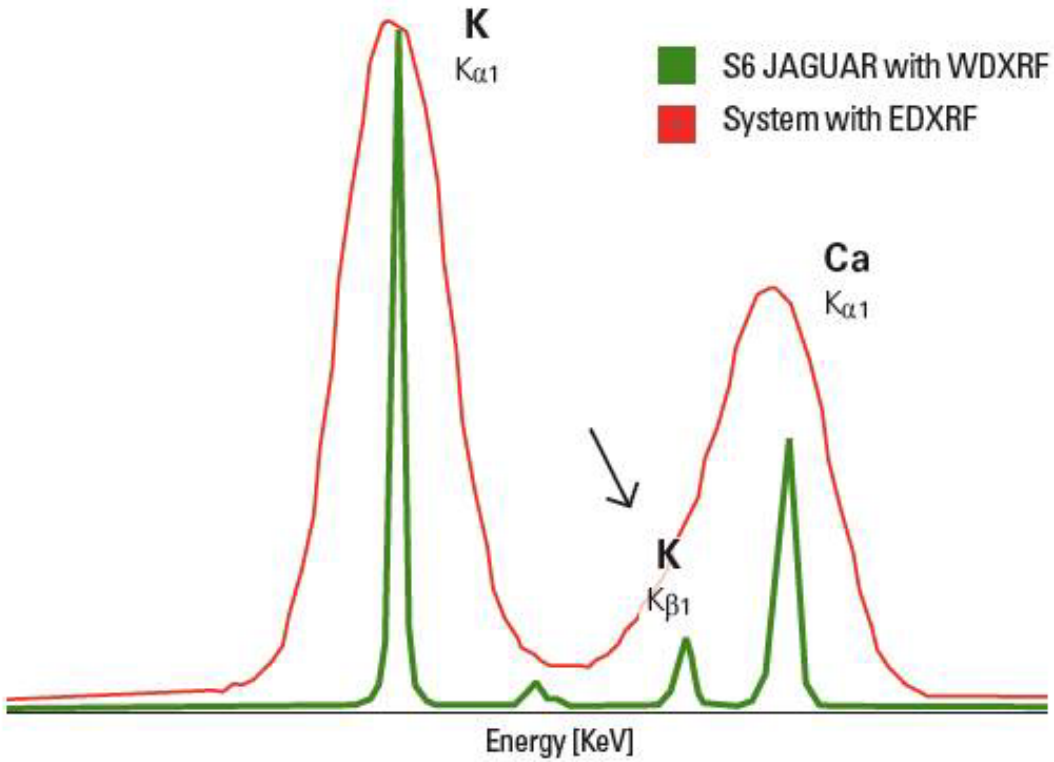


What to do in case of high intensities?

- Smaller mask, reduce power manually?

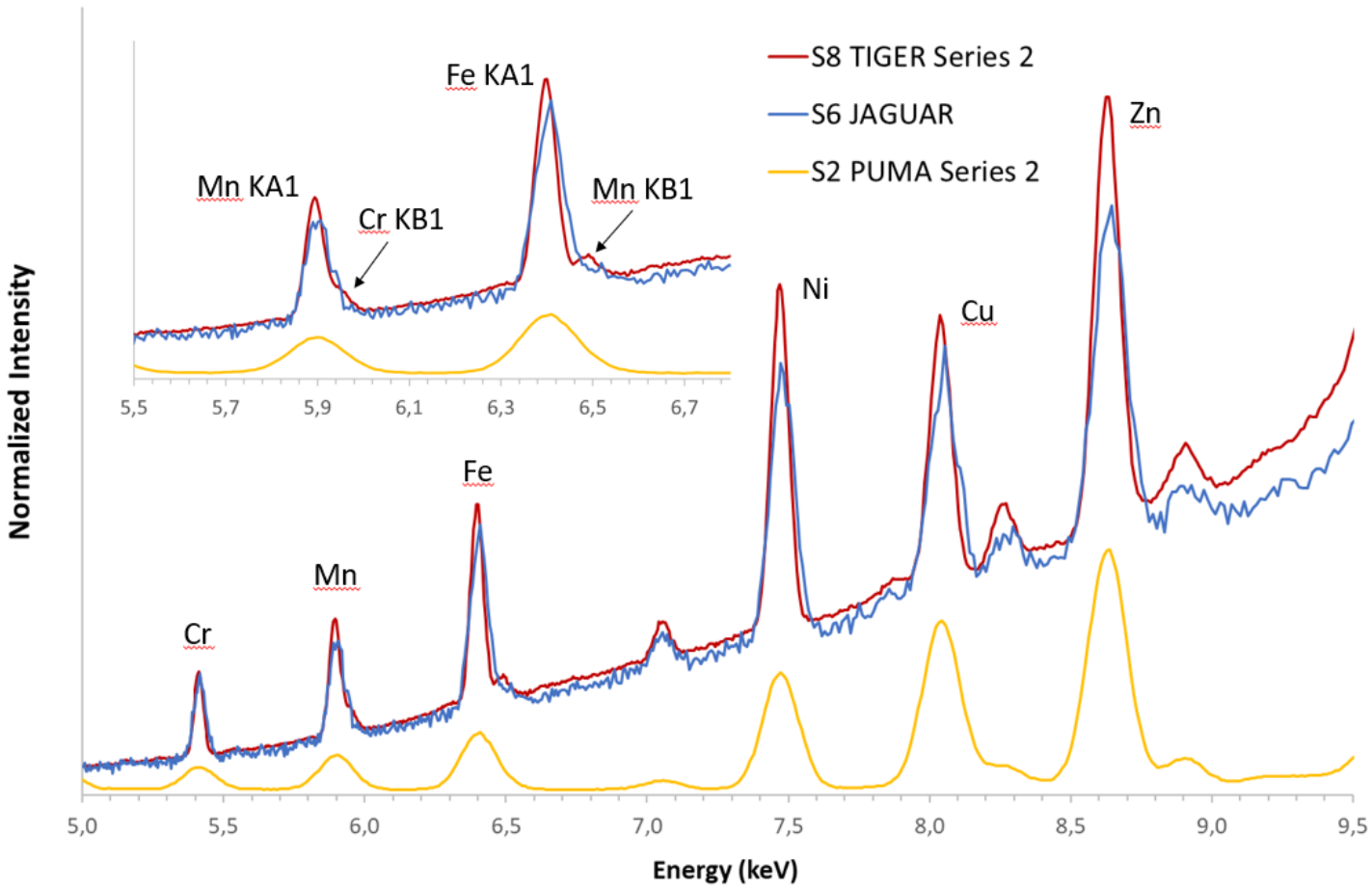
- Automatic Current Reduction:
  Best Sensitivity - No Detector Overflow
- Unlimited intensity range by automatic current reduction
- Best sensitivity without risk of detector overflow
- Physical limit of detector: up to two million counts per second
- Corrected intensity: 10 million counts per second

HighSense™ Goniometer: High Resolution S6 JAGUAR



- The S6 JAGUAR with WDXRF HighSense Goniometer excels
- ED based systems in resolution and analytical precision
  - Better peak to background signal ratio
  - Higher intensity (signal in left picture is scaled)
  - Better separation of neighboring signals

EDXRF & WDXRF - Intensity vs. Resolution

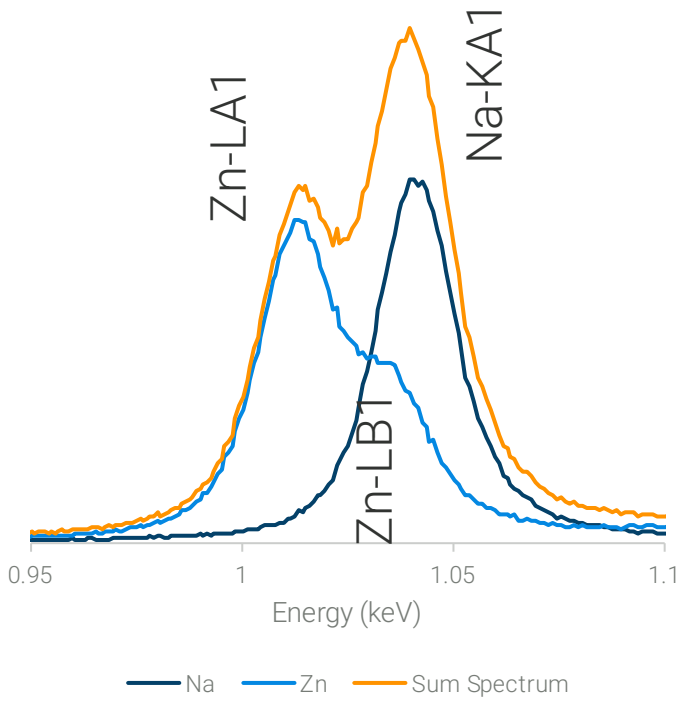


- Intensity and resolution comparison
- Fused bead with 100 ppm of several elements
- Measured on EDXRF and WDXRF instruments
  - S2 PUMA
    40 kV, 500 µm Al
  - S6 JAGUAR
    50 kV, LiF200
  - S8 TIGER
    50 kV, LiF200
- Intensities normalized to tube current

New Multilayer Optics for Better Separation Enhancing WDXRF for Specific Applications

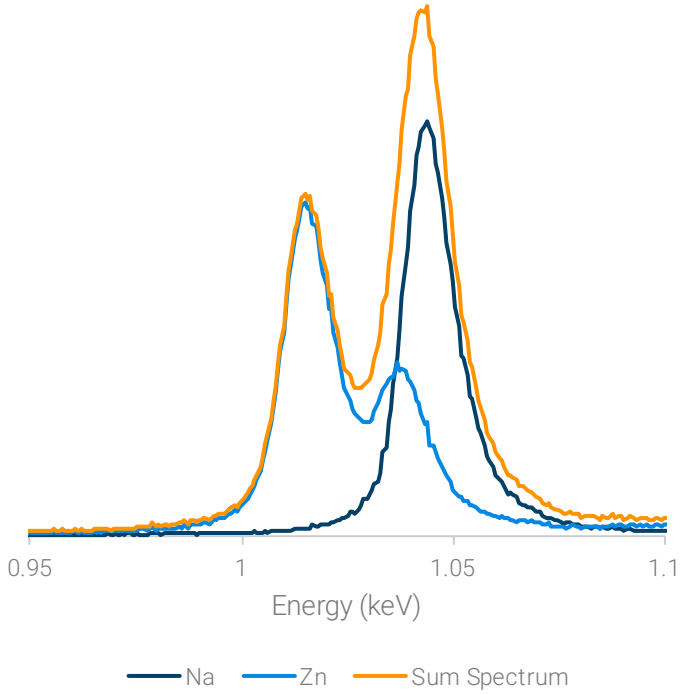
Na-KA1 / Zn-KA1
30kV, 0.23-degr, XS-55, Flow Counter

XS-55 (2d = 5.47nm)



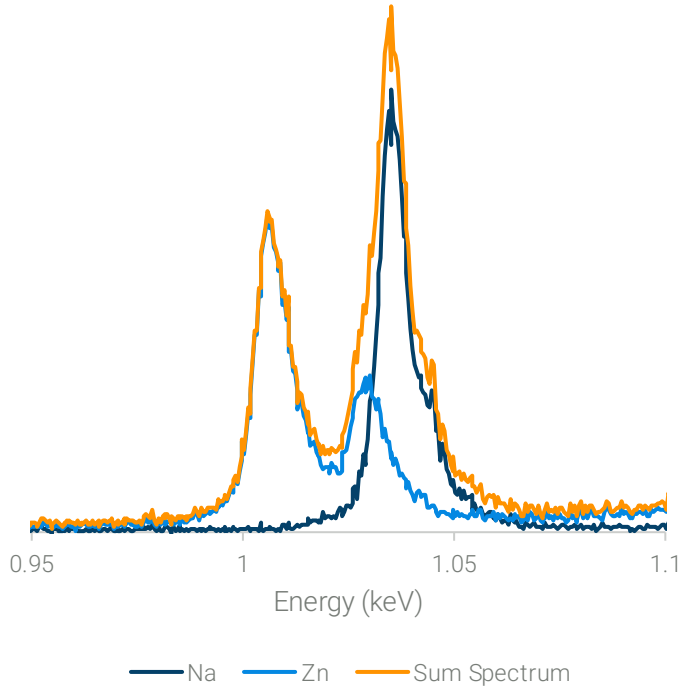
Na-KA1 / Zn-KA1
30kV, 0.23-degr, XS-100, Flow Counter

XS-100 (2d = 3.95nm)



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

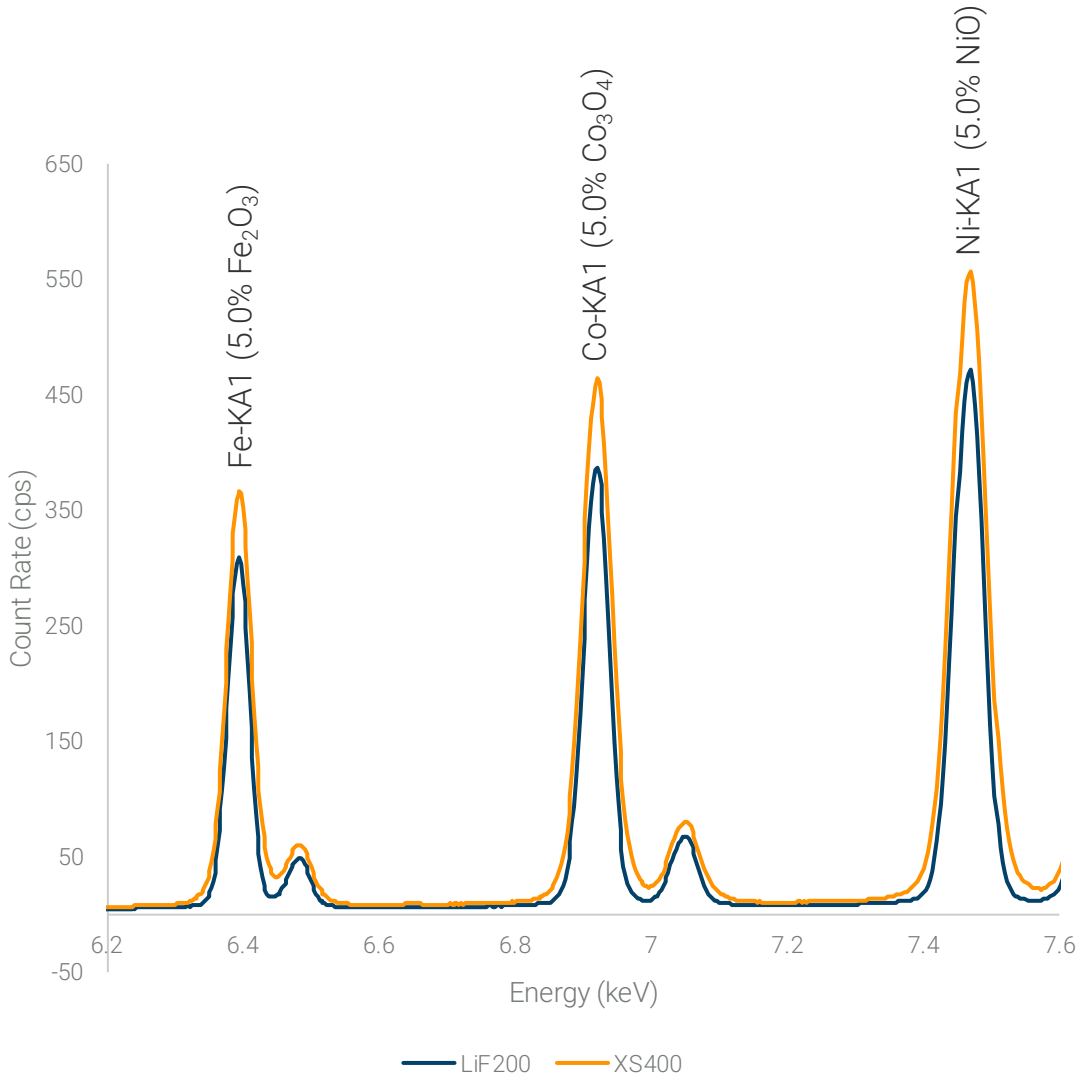
XS-CEM (2d = 2.75nm)



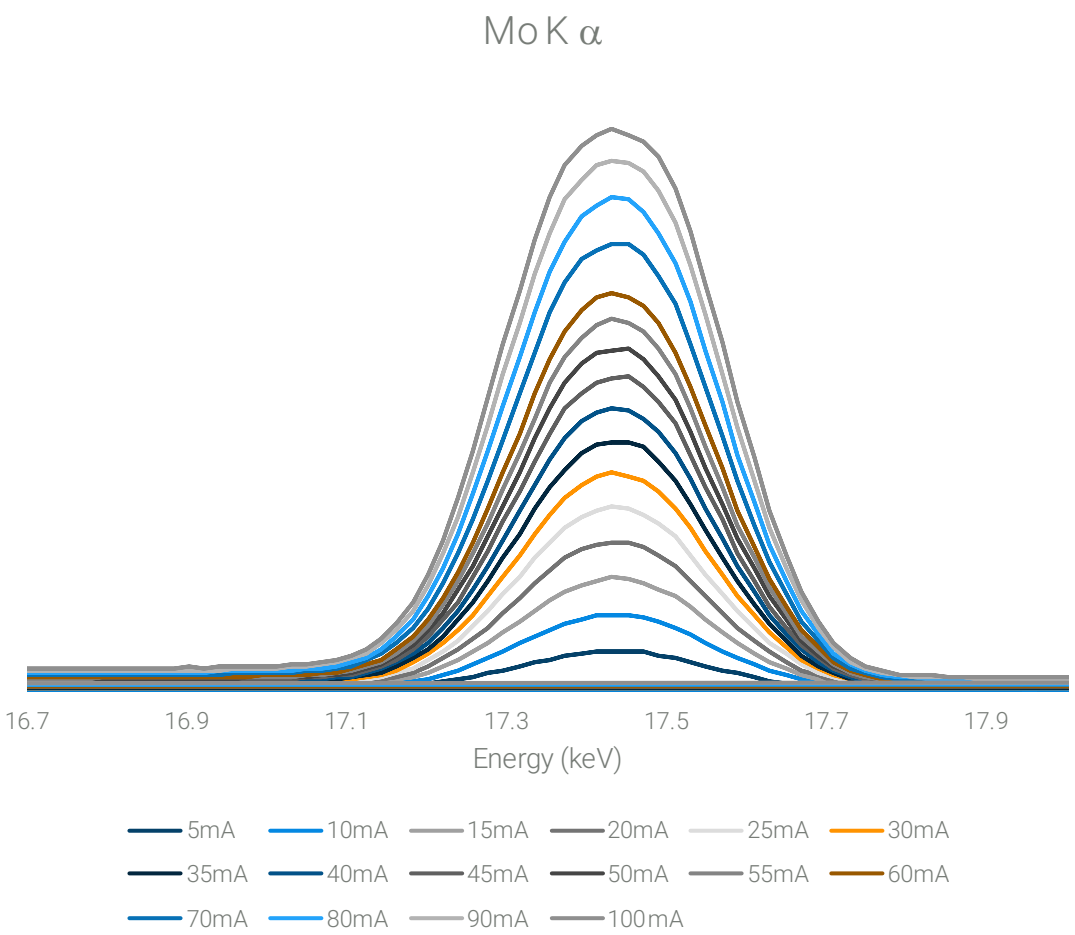
Boost Sensitivity with New Analyzer Crystals Simply Better Precision at the Same Time

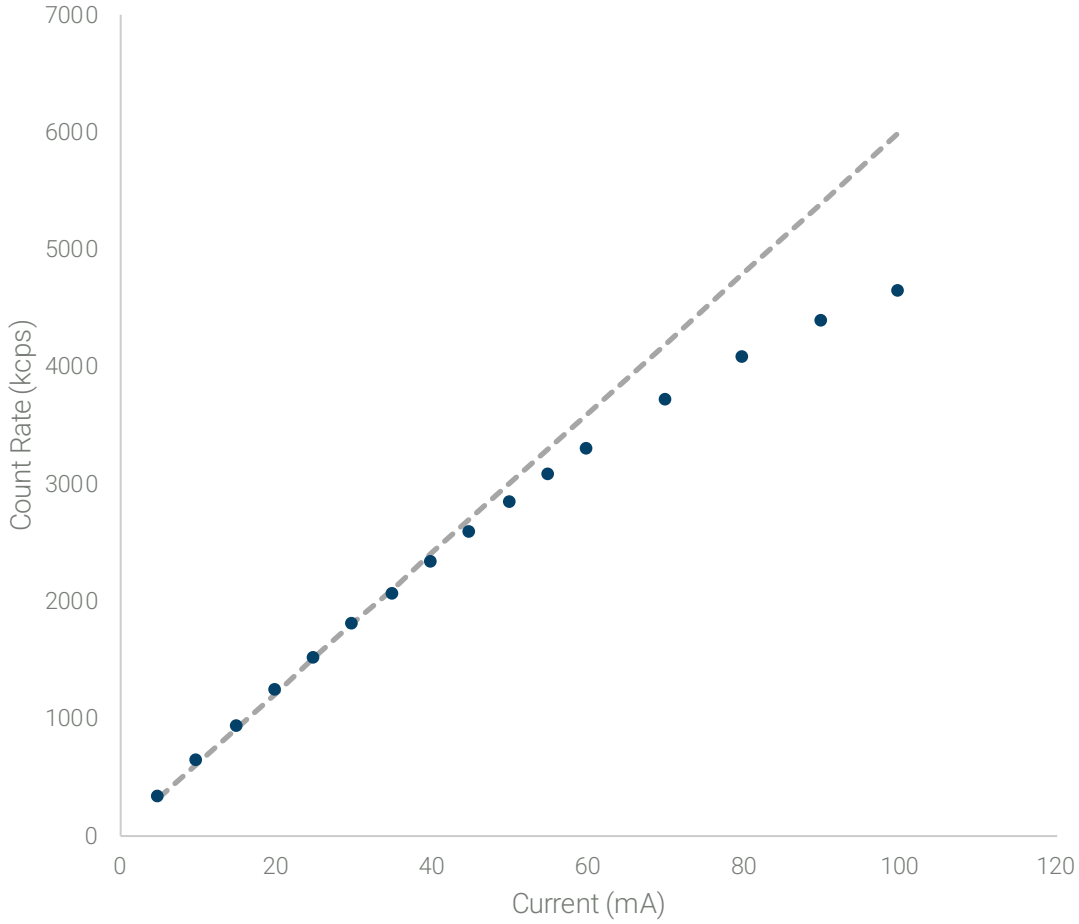
- New geometry and change of crystal composition adds up to 35 % intensity compared to standard LiF200 crystal
  - For elements from K – U
  - Reducing measurement time

  Or
  - Enhancing precision due to higher counts



Linearity of S8 TIGER Series II
Multi-Channel Analyzer, Dynamic Dead Time Compensation





High Performance Benchtop WDXRF S6 JAGUAR

Configurable from single element analyzer for S to multi-purpose unit for the periodic table

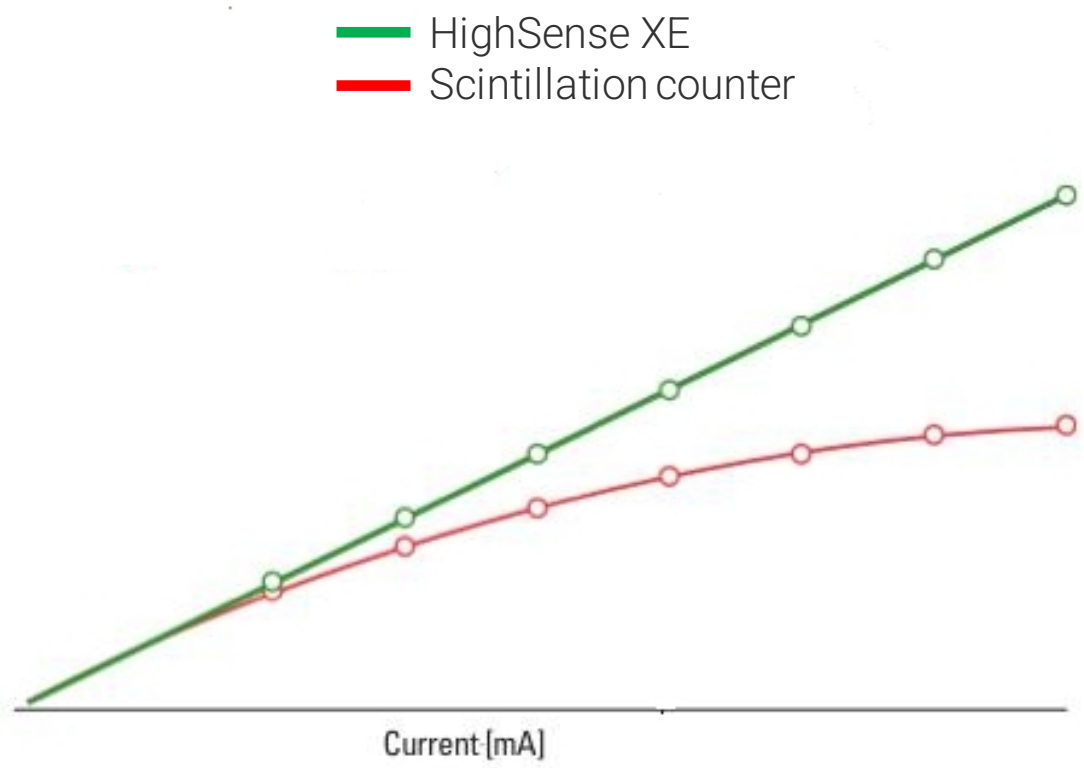
All-new technology and software:

- Long lifetime X-ray tube
- Compact goniometer with high precision gears and closely coupled X-ray beam path
- Optimized analyzer crystals for the entire element range and special applications
- HighSense detection with 2 Mcps countrate
- HighSense XE detector for medium and heavy elements
- SPECTRA.Elements analytical SW
- SMART-QUANT WD with new FP algorythms

S6 JAGUAR Benchtop WDXRF

400 W excitation

HighSense XE™: Unrivalled Linearity S6 JAGUAR



- 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

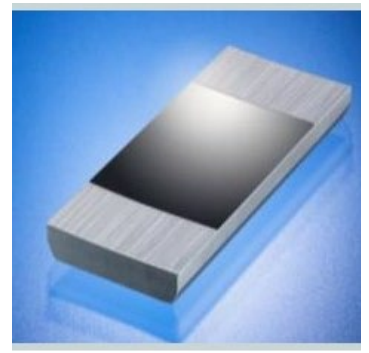
X-ray Fluorescence Analysis (XRF)
The Comparison of Energy and Wavelength Dispersive Spectrometers

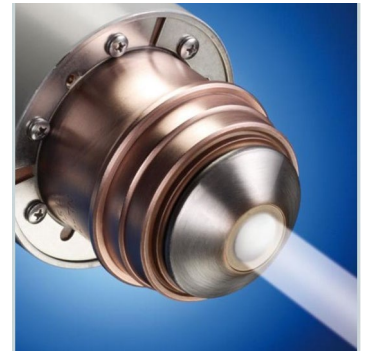
EDXRF

- 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





Petrochemical Applications Refineries

Refineries:

- Monitoring **sulfur** content from **crude oil** feedstock and cracking products to final **Ultra-Low Sulfur (ULS)** levels in **automotive fuels**
- Monitoring important elements in refinery processes such as
  Ni, V, and Fe, Si, Cl,…

Downstream sector:

- **S** analysis along the downstream supply chain at **tank terminals**, storage tanks, **depots**, pipelines, petrol stations, custom control at harbors, and **service** and **inspection labs**

High Performance Benchtop EDXRF for Petro Market S2 POLAR

- Process control in the refinery
- Quality Control in downstream at tank farms or shipping stations

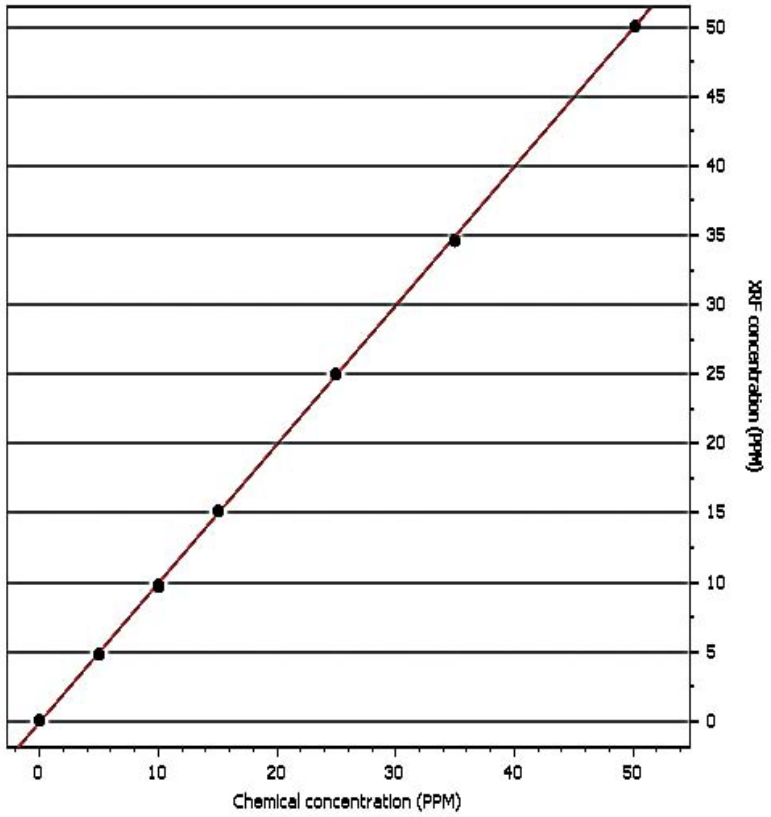
- Small size for small labs, low cost of ownership
- Quick sample preparation w/o any chemicals involved
- Ease-of-use with TouchControl for shift workers
- Robust technology with little maintenance
- One-time calibration
- Norm compliant, for various matrices

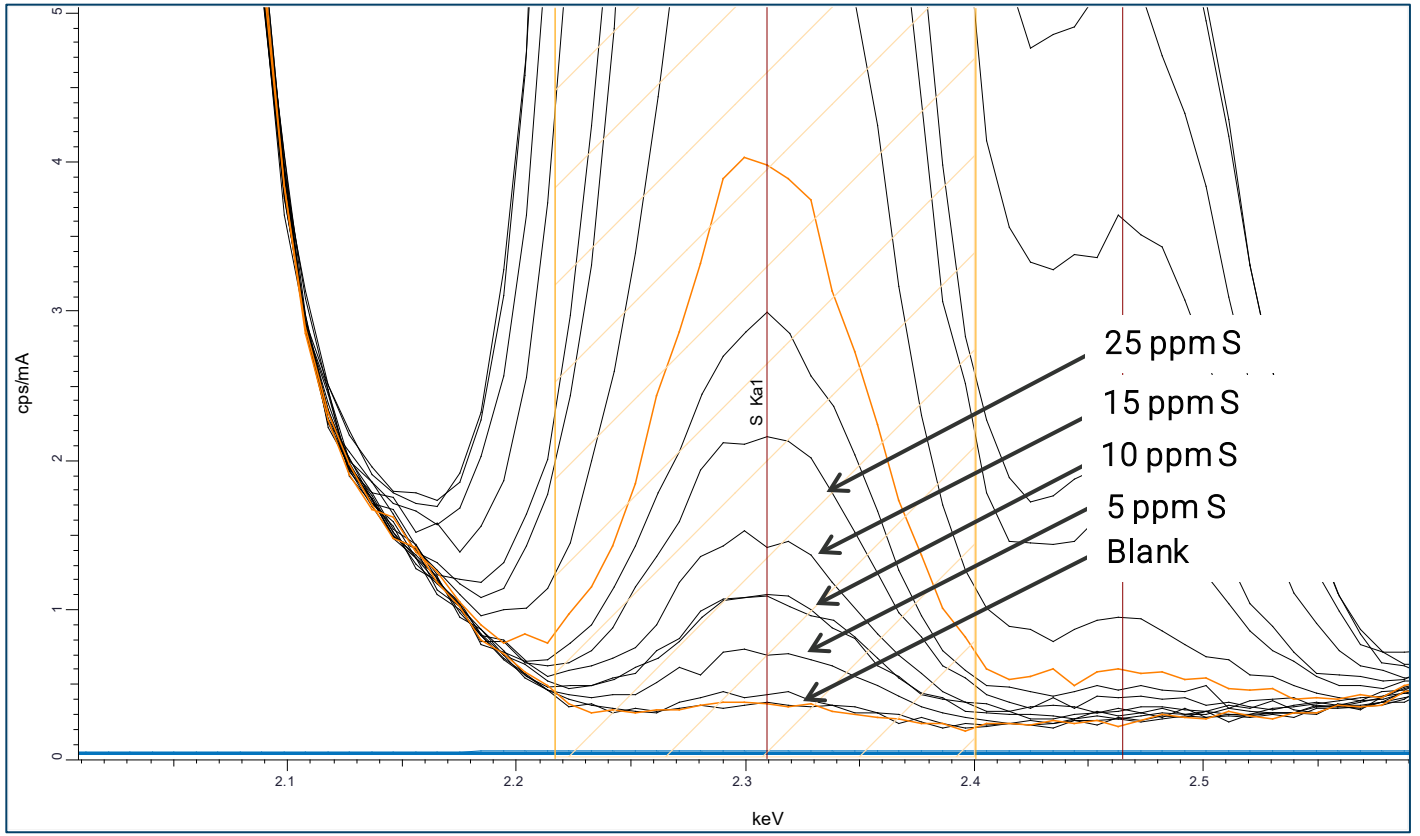
Sample Preparation in XRF
Easy and straight forward

- First step: **Prepare the sample cup**
- Second step: **Fill the sample cup**
- Place the cup on the balance
- Fill with approx. 7 g of sample
- Third step: **Load the sample**
- Place the cup in the chamber of the instrument
- Done! **No dilution, no digestion!**
- **Low costs of ownership with standard liquid cups!**

ASTM D7220 – Ultra-Low Sulfur in Gasoline S2 POLAR

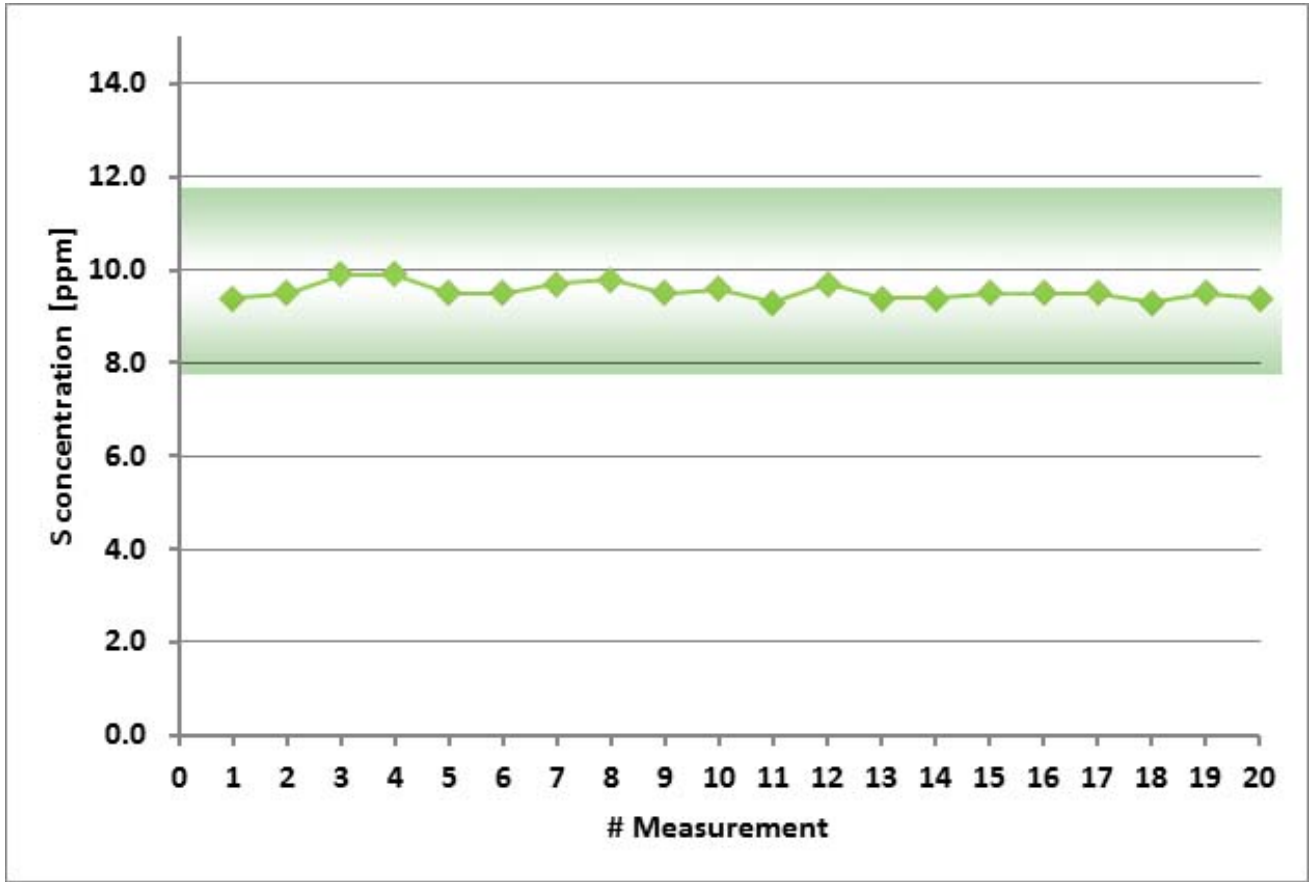
- Excellent calibration curve for ultra-low sulfur in the range of 0 to 50 ppm with a clear separation of different sulfur levels





ASTM D7220 – Ultra-Low Sulfur in Gasoline S2 POLAR

- Gasoline sample (9.8 ppm S) with excellent repeatability at such low concentration level



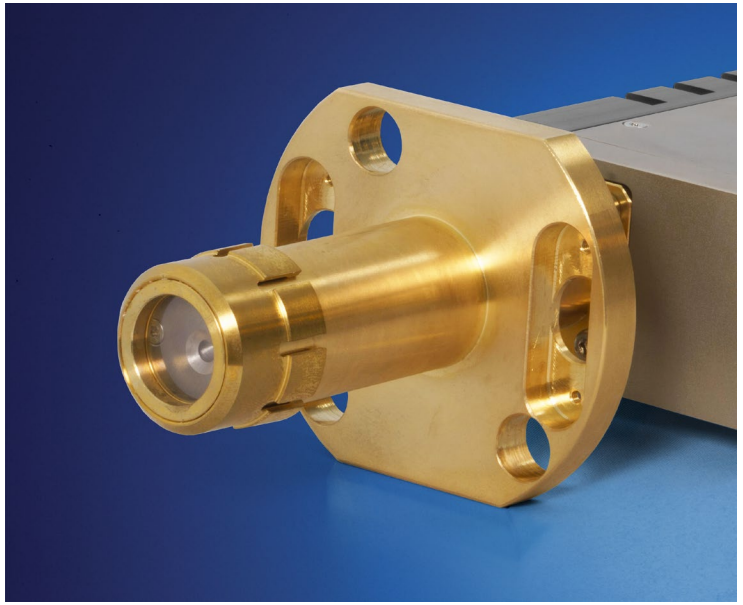
✓ Passed

Accepted difference between 2 measurements: ±2.0 ppm S

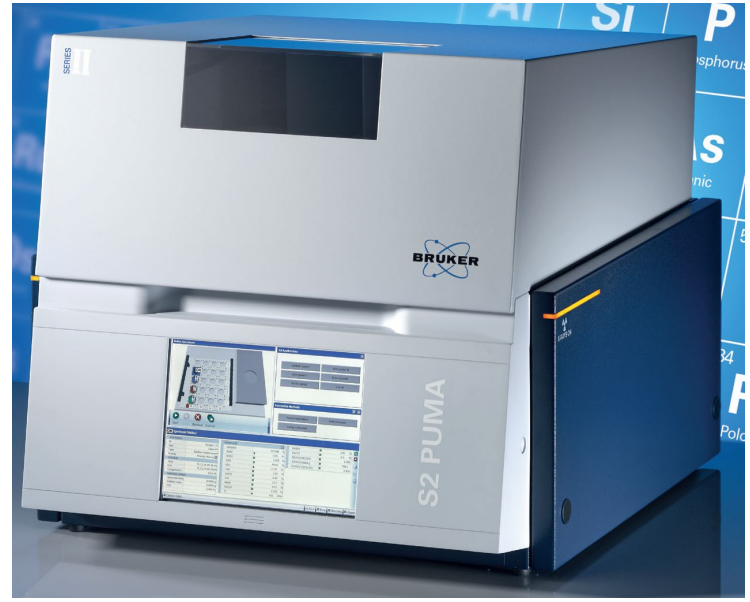
HighSense™ XP Detector
Now available for the S2 PUMA Series 2

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



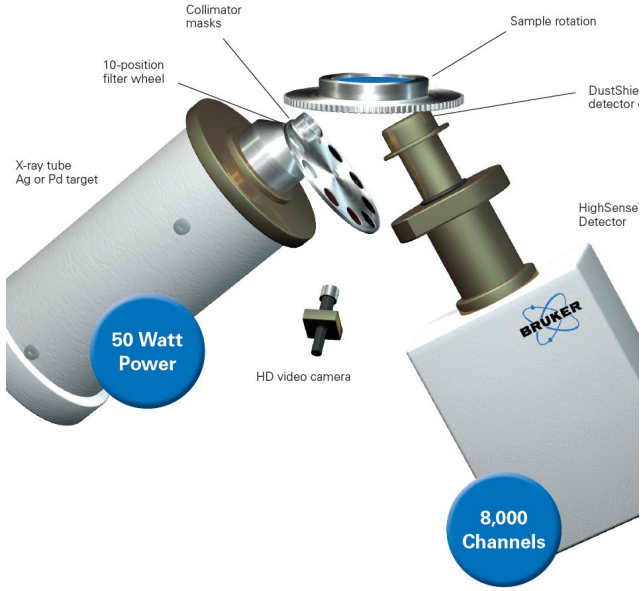
New HighSense XP Detector



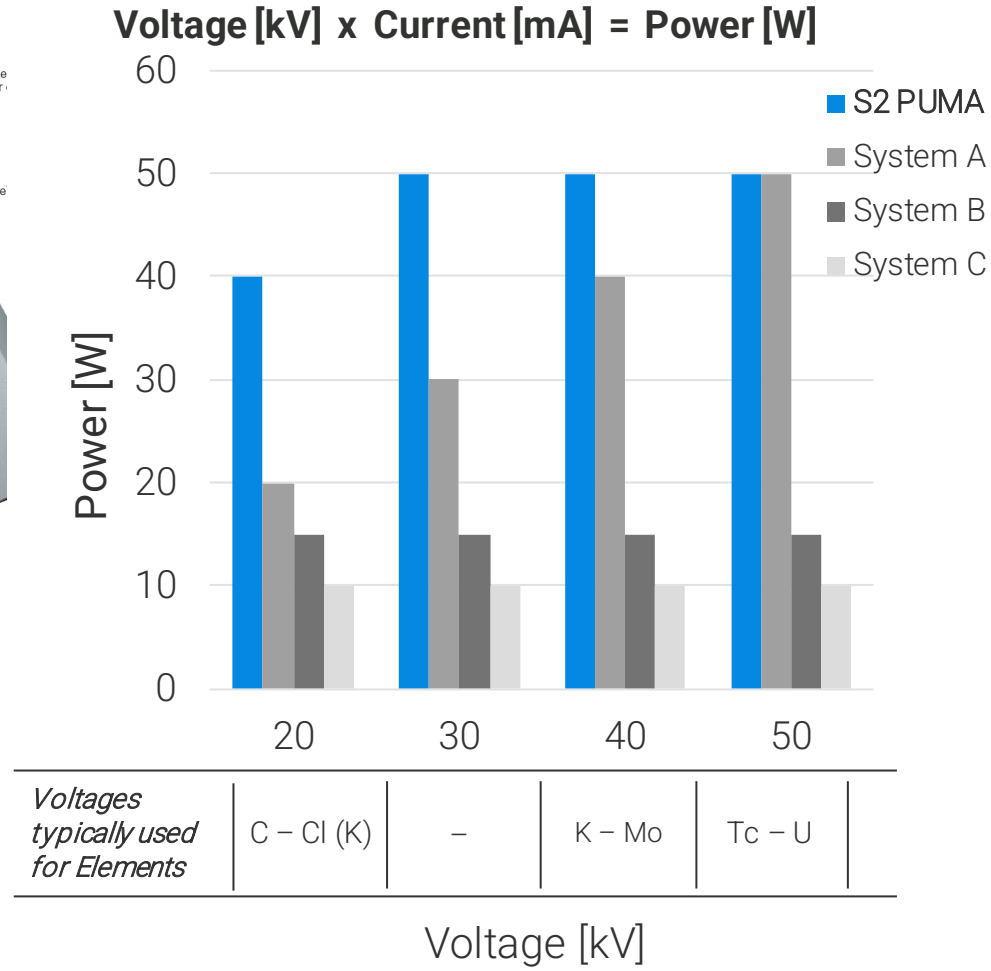
S2 PUMA Series 2 with XY Autochanger

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

HighSense™ XP Detector
Now available for the S2 PUMA Series 2



HighSense turns power into performance



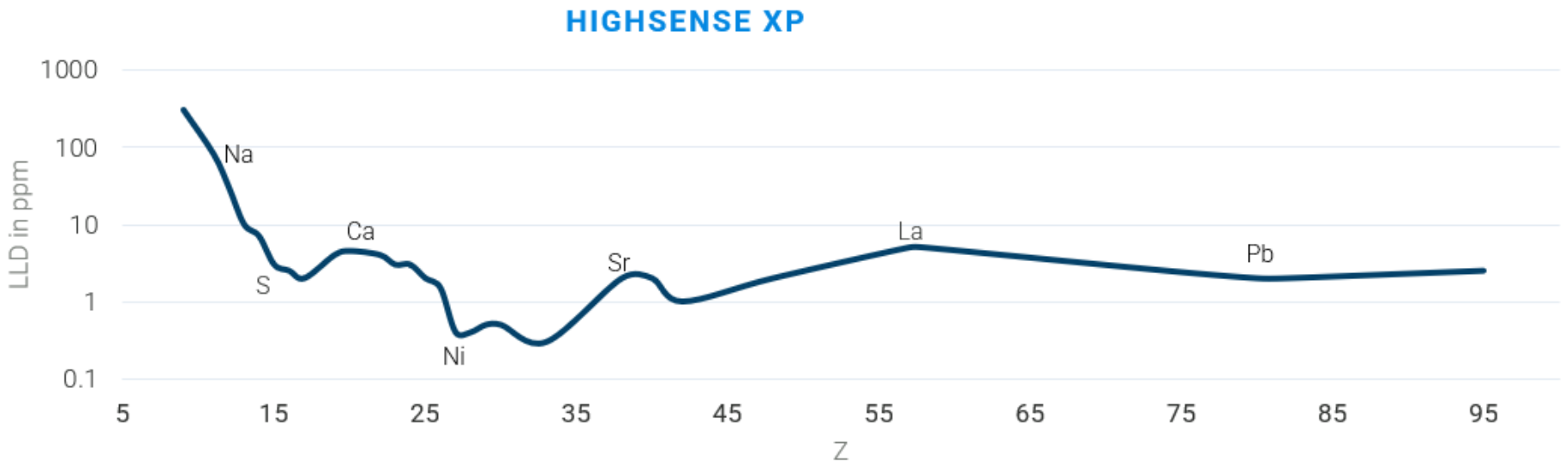
Optimal excitation of the sample is ensured by:

- 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

HighSense XP
Optimal Performance even for Trace Elements

Lower Limit of Detection (LLD)

- Determined for optimal conditions
- LLD = 3 x Standard Deviation

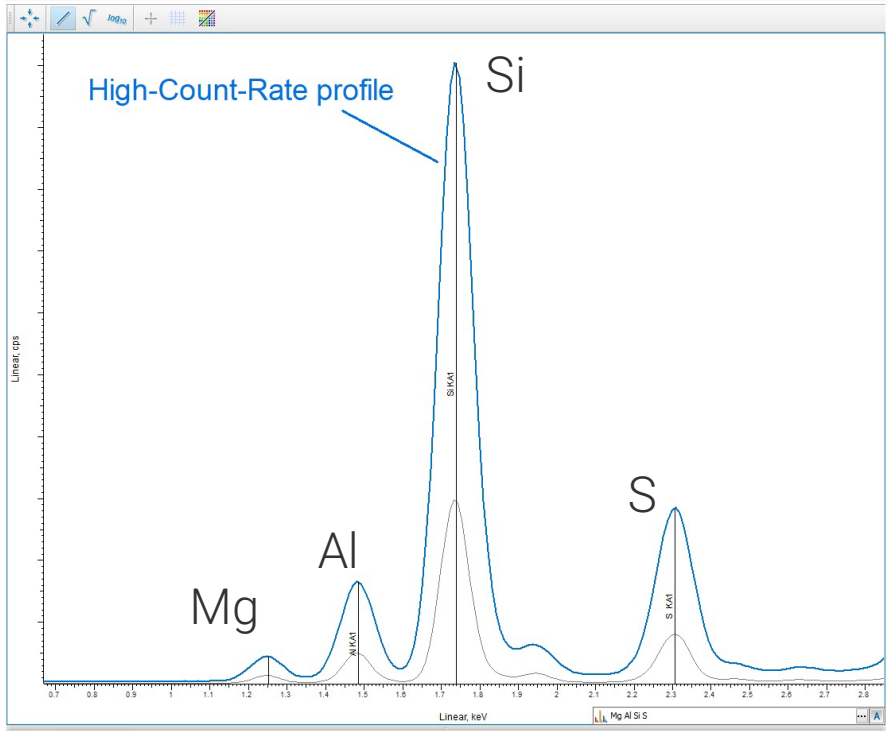


The Benefits of Detector Profiling

- 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.



Achieve up to 4 x higher net intensity with the High-Count-Rate profile

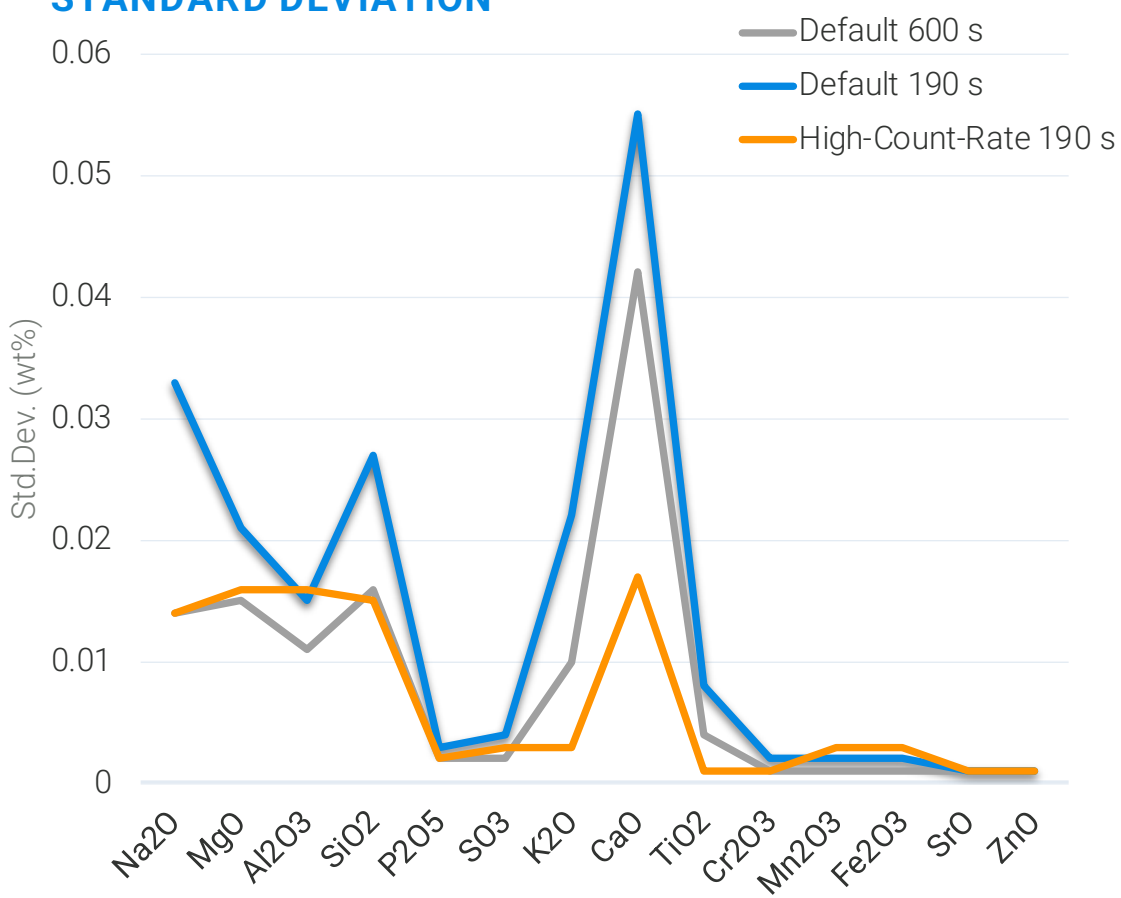
→ Enhance the performance for heavy trace elements in polymers

The Benefits of Detector Profiling

Precision Test

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

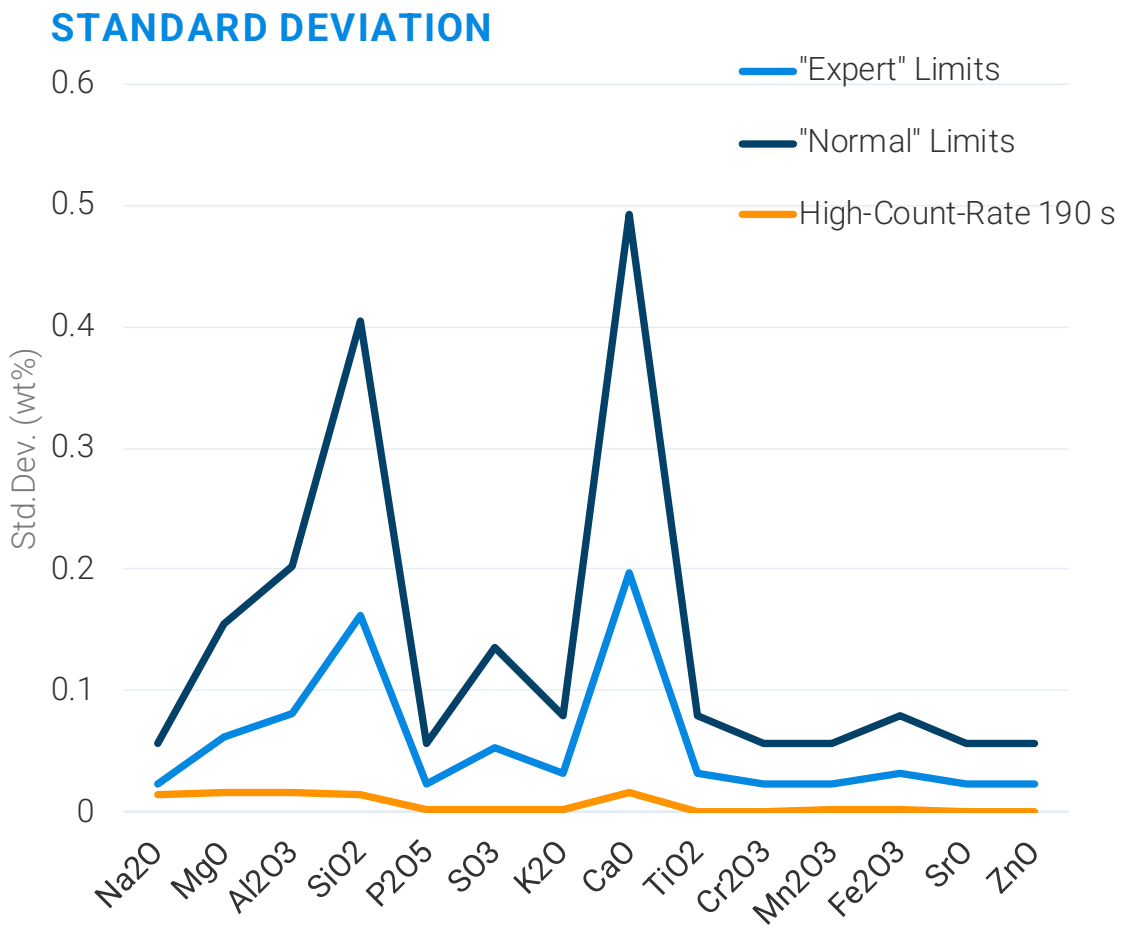
The High-Count-Rate setting allows to reduce the counting time factor of ~3



The Benefits of Detector Profiling

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

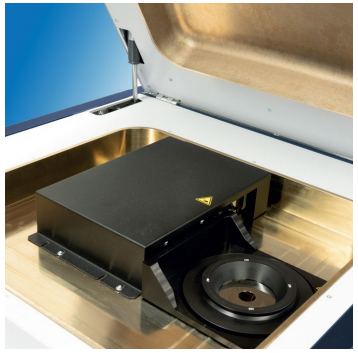
- The S2 PUMA Series 2 with HighSense XP meets standard deviation limits easily.



S2 PUMA Series 2
Taylor-made Solution for all Applications

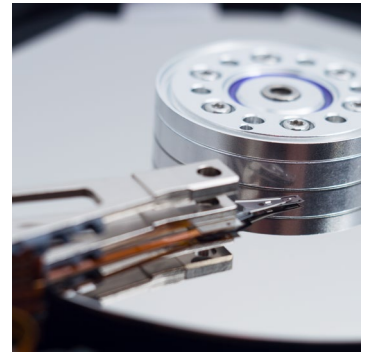
Benefits of the S2 PUMA

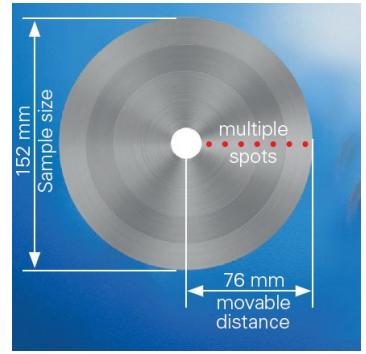
- Sample Handling for all needs:
  - Single
  - XY-Autochanger
  - XY-Automation
  - Carousel
  - Mapping-Stage



Elemental Analysis Coatings and More Easy and Reproducible S2 PUMA Series 2

- Production processes are not always yielding homogenous material in two dimensions.
- Coatings or paint may vary in thickness between the center and the rim of a material.
- Production parameters such as temperature and time can cause compositional variations.
- For other products, local coatings and local differences in alloying composition are used to achieve certain material properties, while using expensive compounds and additives sparsely, such as Ti-coatings of sawblades.



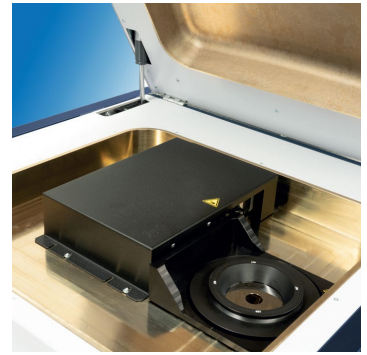


Elemental Analysis Coatings and More Easy and Reproducible S2 PUMA Series 2

- The Mapping-Stage of the S2 PUMA Series 2 fills the gap between bulk measurements and more expensive and time consuming, high-end 2D µ-XRF Mapping.
- It is the ideal tool whenever multiple locations need to be measured to properly monitor and evaluate production processes and product quality.

Technical Details

- Max. sample diameter: 152 mm
- Movable Distance: 0 mm to +76 mm
- Spot size: 1 mm to 34 mm
- Spatial precision: <0.1 mm

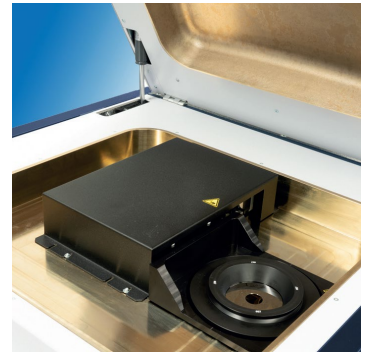


Elemental Analysis Coatings and More Easy and Reproducible S2 PUMA Series 2

- Measure the thickness and/or homogeneity of a Co, W or Zn layer on a hard-disk.
- Determine the homogeneity of alloys such as W, Ni and Mo in tool steel or Pb in brass.
- Check the Ti-coating quality of drill bits and sawblades.

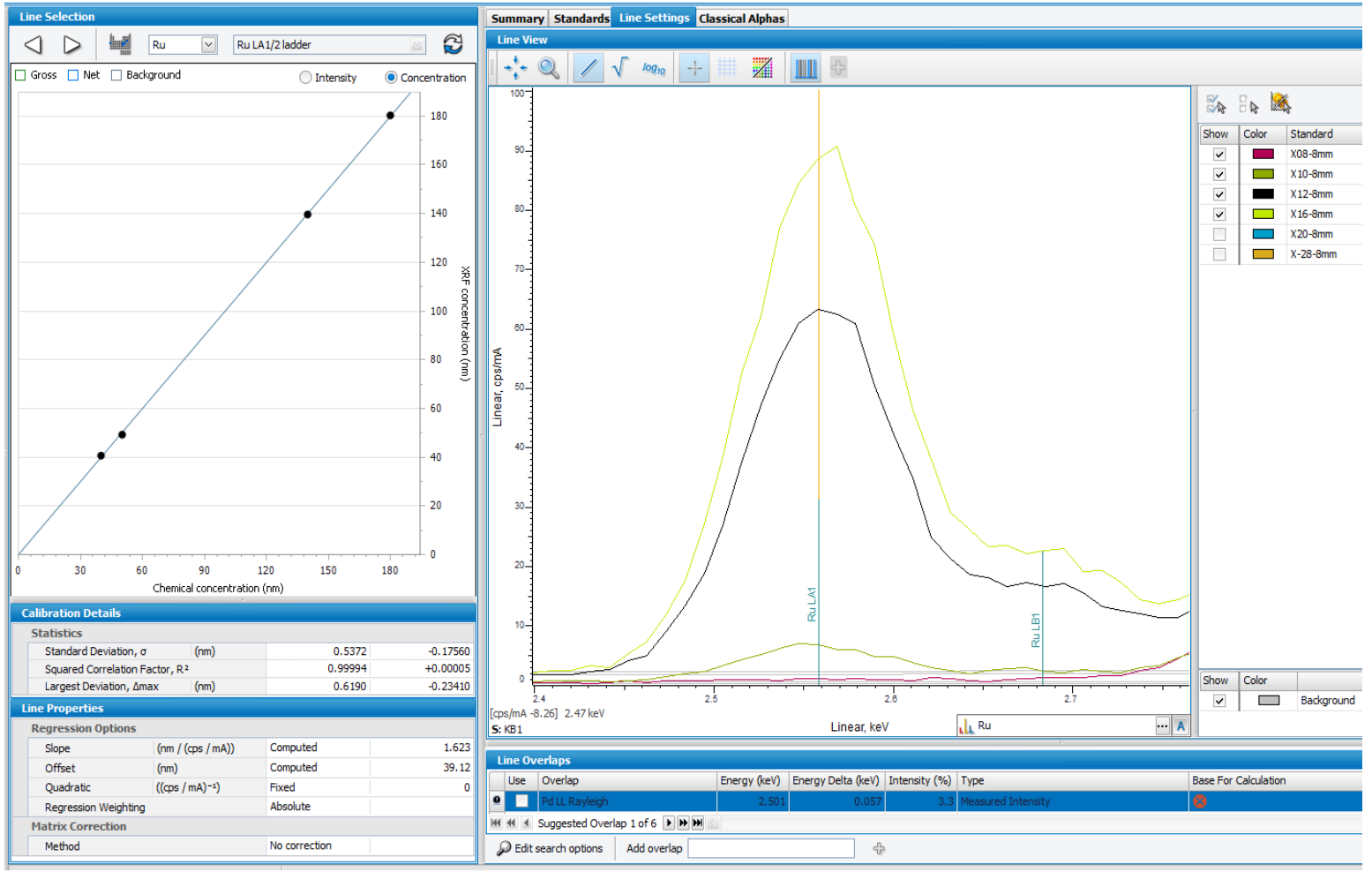
Applications include

- Coating homogeneity
- Coating thickness (one-layer)
- Paint homogeneity
- Material homogeneity
- Chemical composition



Elemental Analysis Coatings and More Easy and Reproducible S2 PUMA Series 2

Repetitions	Co (nm)	Ru (nm)	Fe (nm)
Rep-1	232.6	157.8	64.2
Rep-2	232.5	159.9	66.7
Rep-3	232.5	158.5	66.8
Rep-4	233.3	158.3	64.7
Rep-5	234.1	159.6	65.0
Rep-6	233.2	159.2	64.3
Rep-7	235.4	159.2	65.0
Rep-8	234.9	160.1	66.9
Rep-9	236.0	161.9	66.5
Rep-10	238.0	162.5	66.6
Average	**234.3**	**159.7**	**65.7**
Std.Dev.	**1.7**	**1.4**	**1.1**
Rel.Std.Dev.	**0.7%**	**0.9%**	**1.6%**



BRUKER's Strength
Complete Product Range for Advanced XRF Analysis

From Dedicated Performer to a full versatile XRF for all

S2 POLAR:

- Compact instrument with very low LLD for lighter elements

S2 PUMA:

- Versatile Benchtop XRF for broader applications

S6 JAGUAR:

- Benchtop WDXRF with high resolution for difficult applications, but few samples per day

S8 TIGER:

- Versatile instrument for all applications, incl traces

S6 JAGUAR
Benchtop WDXRF

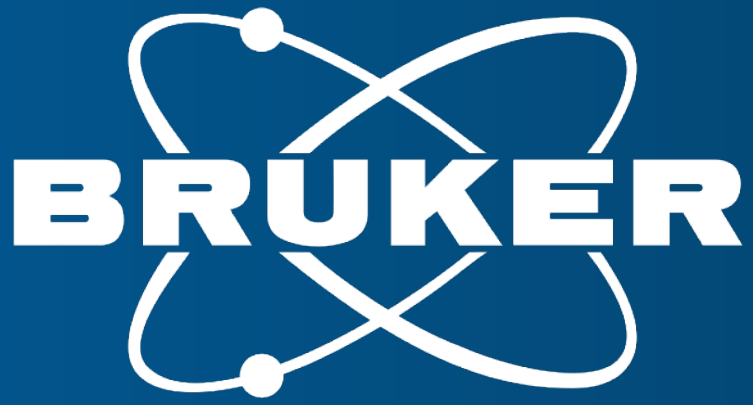
Any Questions?

Learn more about ‘New Software Functionality’

In our 7.7.2021 webinar – Sign Up Now!

Thank you!

Kai Behrens Frank Portala Adrian Fiege



Innovation with Integrity