

WEBINAR SERIES 2021

Fast & Easy Analysis of Macro- and Micro-Nutrients: Applications of XRF in the Animal Feed Industry

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Fast & Easy Analysis of Macro- and Micro-Nutrients: Applications of XRF in the Animal Feed Industry

01

Basics about XRF

02

Benefits of XRF
Where to use XRF in Feed & Agriculture

03

The importance of Sample Preparation

05

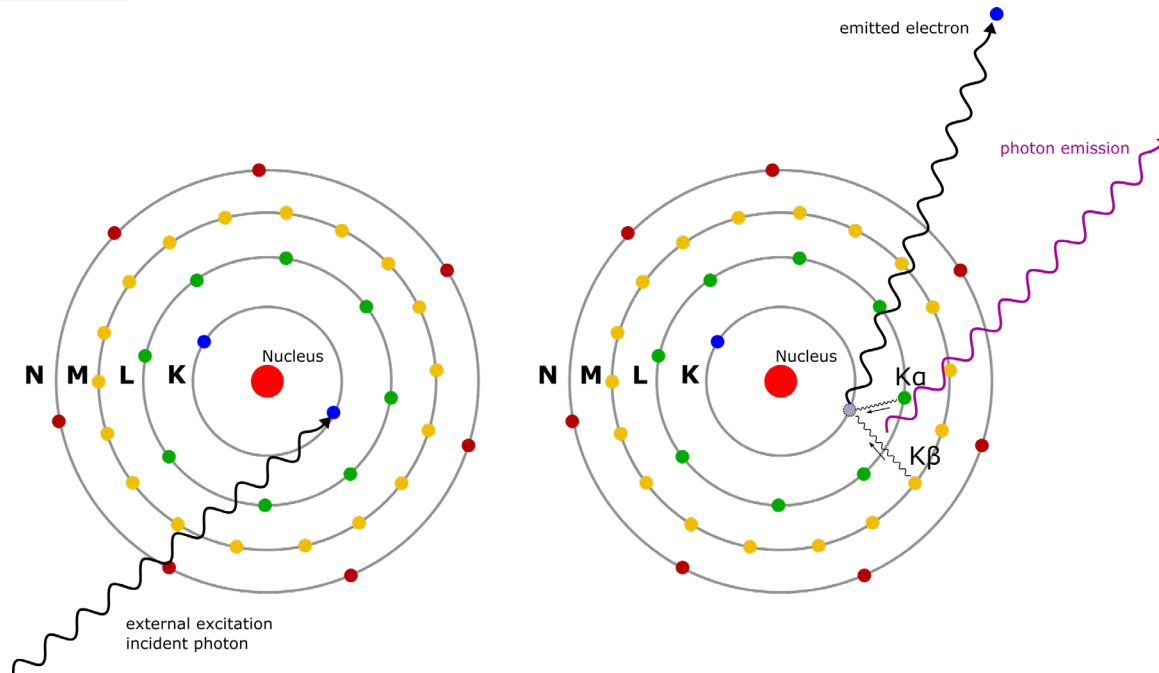
Application examples: From Raw Material to final feed

06

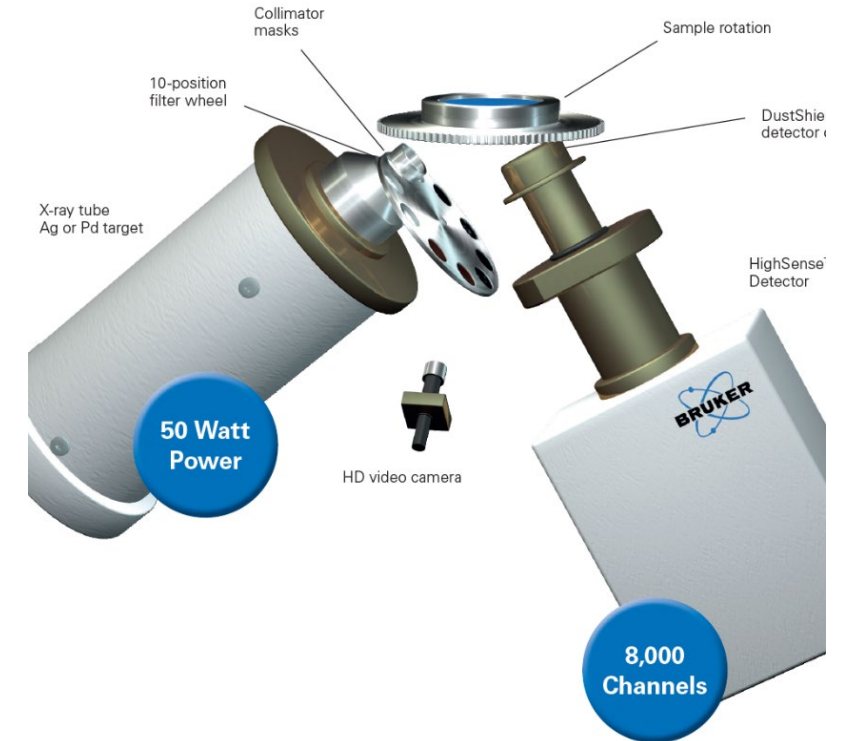
Summary and Q&A

X-Ray Fluorescence (XRF)

Analysis 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

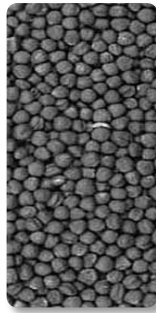
Quality control in feed and agriculture by XRF



Grains
whole/ground



Oil Seeds
whole/ground/expeller



Legumes
ground



Animal proteins
ground



Feed
ground/pellets



Forages
wet/dry

Elemental Ranges

- Wide coverage of the periodic table: F to U

■ S2 PUMA (EDXRF)
■ S2 PUMA with HighSense XP
□ S8 TIGER (WDXRF)

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Concentrations & Time

- Marco-, Micro- and Trace Mineral Nutrients
 - µg/g (ppm) to 100%
- Time-to-result: 3 to 15 min

Benefits of XRF vs. AAS and ICP

Cost of Ownership

The investment for the analytical instrument is only one part of the total cost of ownership. Expenses for laboratory equipment, labour and consumables add to that cost.

ICP-OES/AAS

- Use of expensive accessories (AAS: graphite tubes)
- Consumption of noble gases (ICP: Argon)
- Need for hazardous chemicals (compliance with high-level safety regulations)
- Complicated sample preparation equipment (training and time)

S2 PUMA Series 2 EDXRF Spectrometer:

- No hazardous consumables
- No highly trained lab-technician needed
- No gas or very little gas consumption (Helium for volatile liquids)

Example: ICP vs. S2 PUMA Series 2

- Final Feed measure as pressed powder pellet
 - XRF: <\$2000/year for binding material
 - ICP: >\$20.000/year for gas, references and other consumables

Benefits of XRF vs. AAS and ICP

Ease of use and Time to Result

- XRF makes Quality Control Fast and Simple!

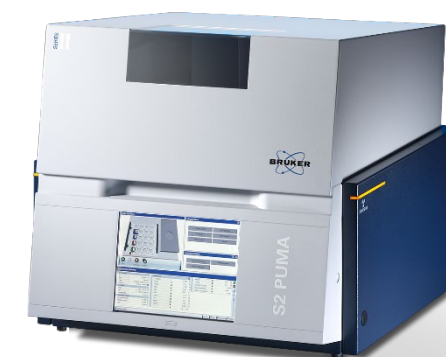
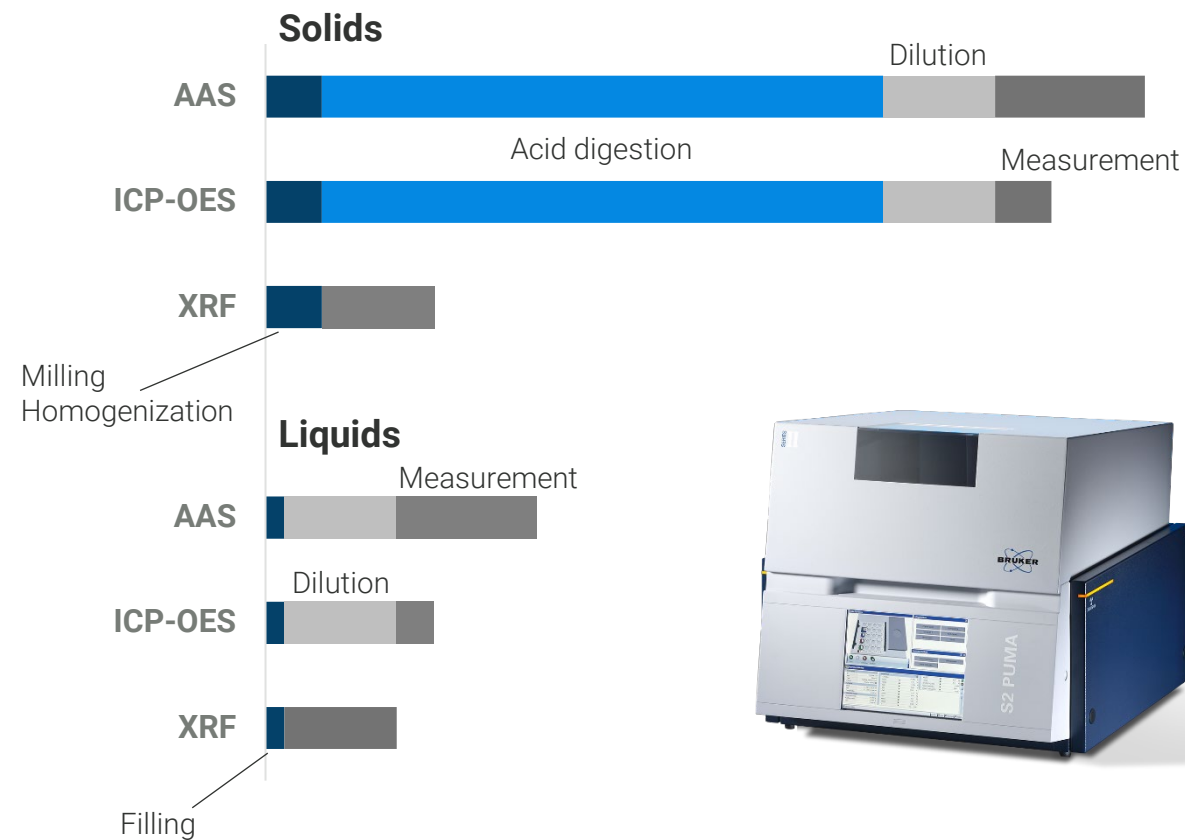


ICP/AAS



XRF

Time to Result

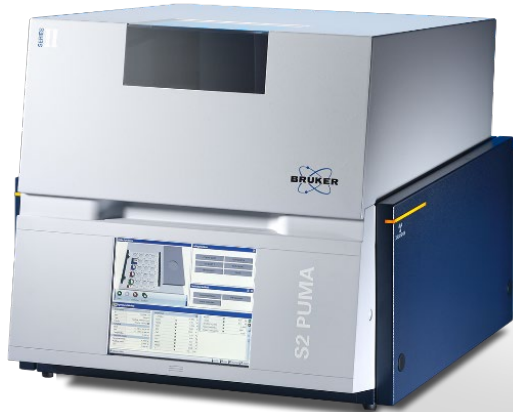


Highest Safety Standards

S2 PUMA Series 2



- Fully radiation-protected system
- German Type Approval and Vollschutz according to BfS RöV
- NF C 74-100 Approval (France)
- GHOST Certificate (Russia)
- 30 kV option, to reduce regulatory efforts in certain countries:
 - Austria/Italy/France
 - Belgium/Luxembourg
 - Egypt/Taiwan



Examples for Quality Assurance (QA) & Quality Control (QC) in Feed & Agriculture

- Macro & Micro mineral nutrients, and trace level contaminant in feed and feed supplement / pre-mixes
- Raw Material ID, Production Monitoring, Quality Control
- **Safety:** Avoid toxic chemicals (e.g., Ni, Pb), foreign materials (e.g., metal, plastic)
- **Dairy cows:** Ensure the correct contents & ratio of Ca, P, Mg, Na, K, Cl, and S
 - Urine Analysis: Monitor the health of the cow → Use the speed of XRF to drive immediate actions at the farms
- **Soils:** Analyze nutrients and toxic elements from major (Al, Si) to minor (Na, Mg, K, P) to trace (As, Pb) levels
 - Use to results for actions like type selection and amount of fertilizer
- **Fertilizer** quality and production control
 - Precise analysis of active components (P, K, Mg, Zn) and toxic traces (As, Cd, U...)



How much preparation is needed for XRF?

Depends on the Application: raw material, pre-mix, forages, final feed,...

- 1. Loose powder or grains
 - Benefit: Fastest and easiest sample preparation
 - Challenges: Matrix sensitive, grain size effects, heterogeneities.
→ Grinding to improve homogeneity and reduce grain size effects
- 2. Pressed powder pellet
 - Benefit: Simple, quick and reproducible sample preparation
 - Challenges: Matrix sensitive
→ for high accuracy samples can be grouped
- 3. Fused glass bead
 - Benefit: Global calibration for many materials possible
 - Challenges: More sophisticated sample preparation



Quality control in grain and oil seed sector

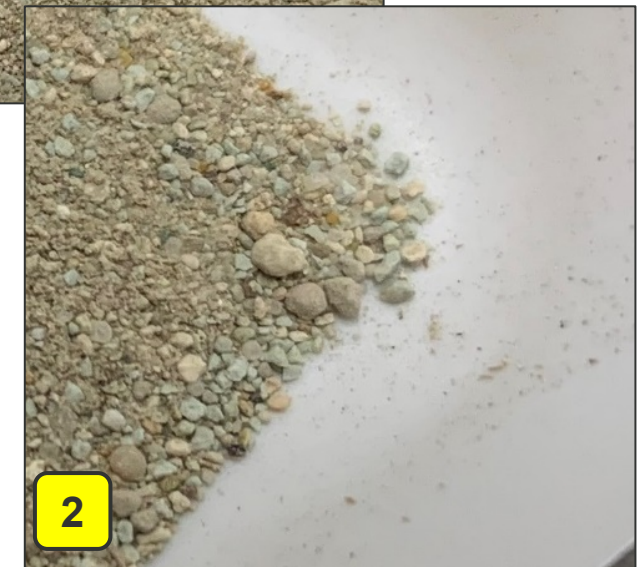


Sample pre-processing

- Pre-processing can be essential, especially grinding
- Homogeneity of solid samples is a key point for gaining best accuracy and repeatability
- Grinding takes few minutes and pays back especially for rapeseed, sunflower seed and corn.

The importance of grinding

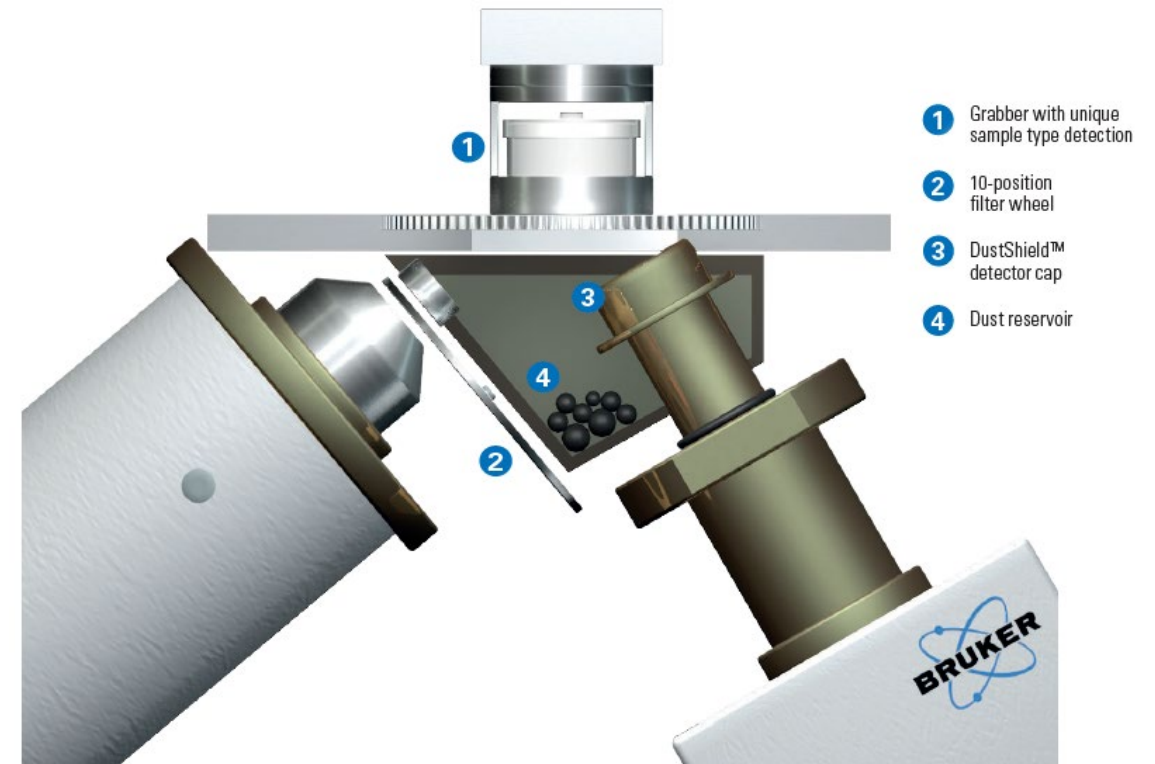
Example: Pre-mix heterogeneity



Low Maintenance thanks to SampleCare™ S2 PUMA Series 2

Worry-free operation, even for dusty feed samples

- SampleCare is a unique, multi-layer system, protecting vital spectrometer components
- XY Grabber with liquid cup recognition ensures He mode for liquids
- 10-position filter wheel protect the X-ray tube head
- DustShield detector cap avoids spillage of liquids and contamination with small sample particles on detector head
- Dust reservoir enable easy cleaning



ELEMENTAL ANALYSIS IN THE ANIMAL FEED INDUSTRY

Animal Feed Application Examples

Standardless analysis with EDXRF and WDXRF

Great choice for Raw Material ID and Verification

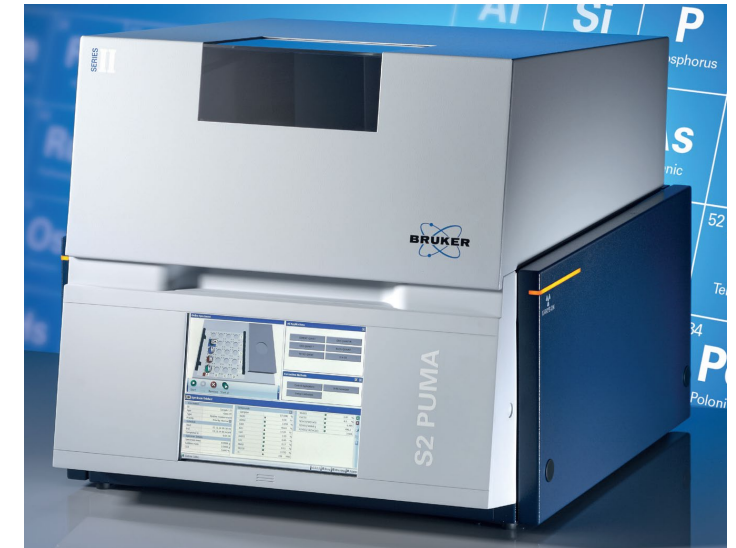
SMART-QUANT is set up to work in full Fundamental Parameter (FP) mode – this means no calibrations necessary!

Excellent for raw material testing and whenever special samples outside the analytical routing need to be measured.

- F to Am (FP) / Na to Am (WD)
- ppm to 100%
- Air, Helium, Vacuum
- 30 and 50 kV



SMART-QUANT: Push-button solution for quick and reliable analysis of unknown samples

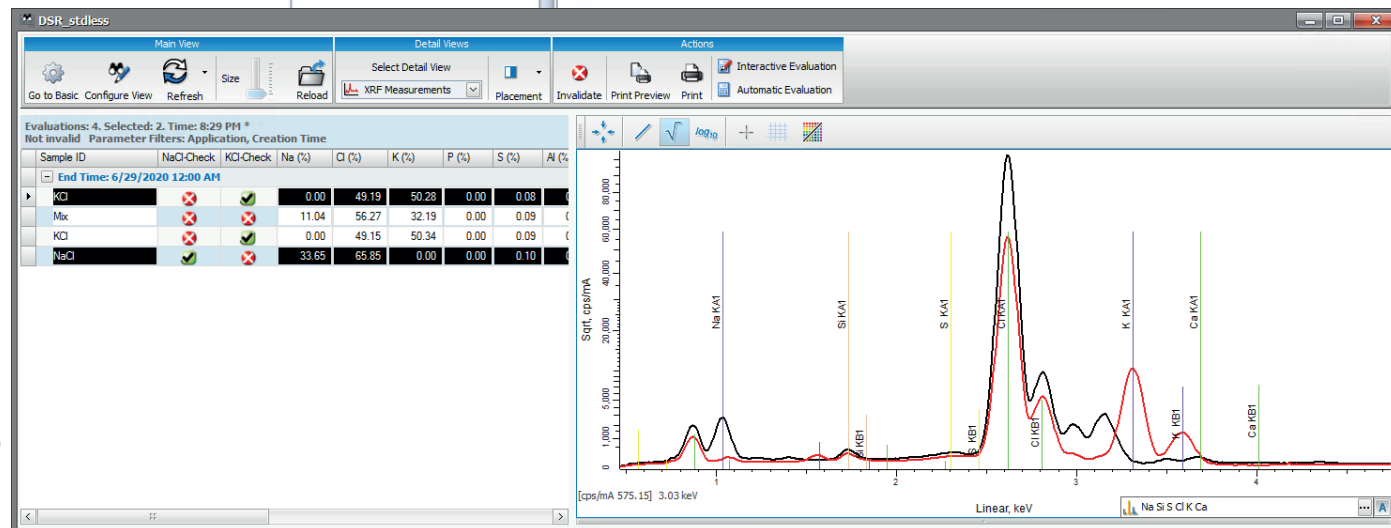


S2 PUMA: High-performance EDXRF

Raw Material ID and Quantification Setting Control Limits

- Setup color-coded control limits for allow a quick decision during production, e.g.:
 - **Nominal** → all good
 - **Warning** → production can continue but a closer look or re-analysis may be needed
 - **Alarm** → immediate production stop
- **Example:** Cl / Cl₂ compliance for salt labeling requirements (NaCl, KCl or CaCl₂)

Control Standards		Concentration Limits							
<div><div></div><div>Use</div></div>	Standard Name ▾		<div><div></div><div>Alarm Low</div></div>	<div><div></div><div>Warning Low</div></div>	<div><div></div><div>Nominal</div></div>	<div><div></div><div>Warning High</div></div>	<div><div></div><div>Alarm ...</div></div>	Thresholds	
<div><div></div><div>▶</div></div>	<div><div><div>✓</div></div>Control sample</div>	Silicon Oxide	25.41 %	26.22 %	26.75 %	27.29 %	28.09 %	<div><div></div><div></div></div>	Absolute Value
		Aluminum Oxide	6.62 %	6.83 %	6.97 %	7.11 %	7.32 %	<div><div></div><div></div></div>	Absolute Value
		Iron Oxide	1.97 %	2.03 %	2.07 %	2.11 %	2.17 %	<div><div></div><div></div></div>	Absolute Value
		Calcium Oxide	55.01 %	56.75 %	57.91 %	59.07 %	60.81 %	<div><div></div><div></div></div>	Absolute Value
		Magnesium Oxide	1.25 %	1.29 %	1.32 %	1.35 %	1.39 %	<div><div></div><div></div></div>	Absolute Value
		Sulfur Oxide	2.96 %	3.06 %	3.12 %	3.18 %	3.28 %	<div><div></div><div></div></div>	Absolute Value
		Sodium Oxide	0.45 %	0.46 %	0.47 %	0.48 %	0.49 %	<div><div></div><div></div></div>	Absolute Value
		Potassium Oxide	1.17 %	1.21 %	1.23 %	1.25 %	1.29 %	<div><div></div><div></div></div>	Absolute Value
<div><div></div><div>▶</div></div>	<div></div>							<div><div></div><div></div></div>	Absolute Value



How to calibrate a XRF spectrometer

Example: Pig feed

Standard Material for calibration

- 3 BIPEA, 5 AAFCO standards with known composition
- Covering 12 key Macro- and Micronutrients
 - Na, Mg, Al, P, S, Cl, K, Ca, Mn, Fe, Cu, Zn

Sample Preparation

- Ground to a fine powder
- Pressed to a pellet
- Run in one go on the XY Autochanger



Why pressed pellet preparation?

- Foil used for liquid/powder analysis mainly affects light elements
- A smooth surface is of particular importance for the measurement of light elements

Thickness of the sample from which 90% of the measured intensity is derived

Element	Line	Energy (KeV)	Graphite	Glass	Iron	Lead
Cd	KA1	23.17	14.46	8.2	0.7	77.3
Mo	KA1	17.48	6.06	3.6	0.31	36.7
Cu	KA1	8.05	5.51	0.38	36.4	20
Ni	KA1	7.48	4.39	0.31	29.8	16.6
Fe	KA1	6.4	2.72	0.2	164	11.1
Cr	KA1	5.41	1.62	0.12	104	7.23
S	KA1	2.31	116	14.8	10.1	4.83
Mg	KA1	1.25	20	7.08	1.92	1.13

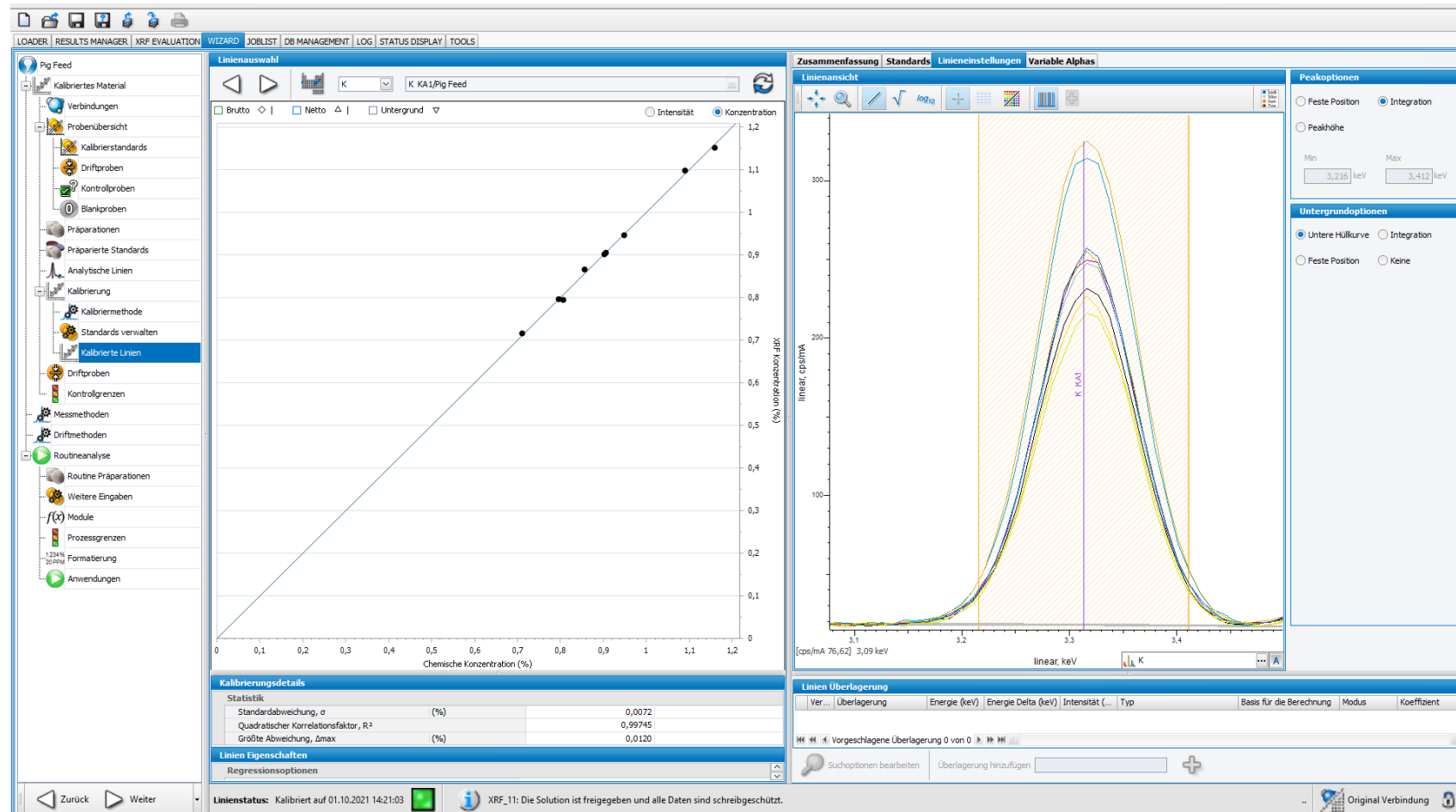
■ μm -range

■ mm-range

■ cm-range

How to calibrate a XRF spectrometer

Example: Pig feed

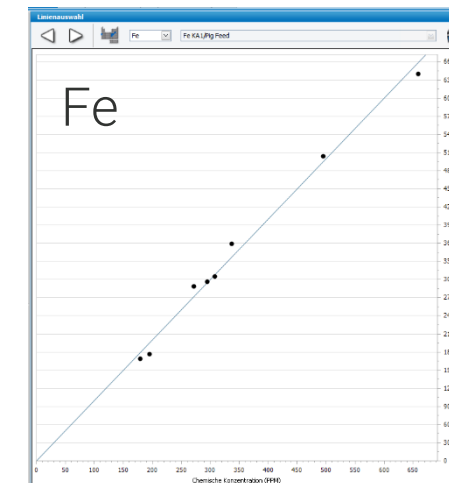
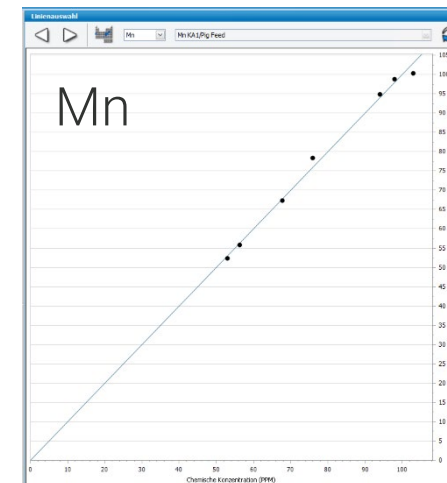
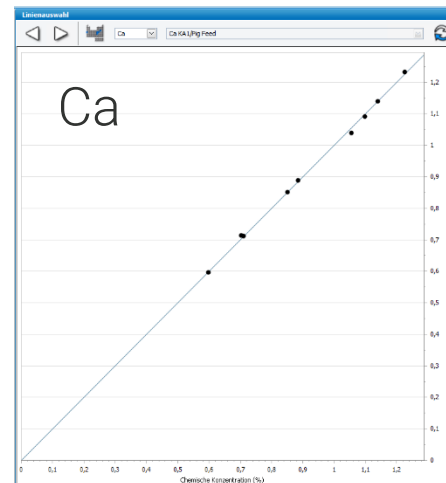
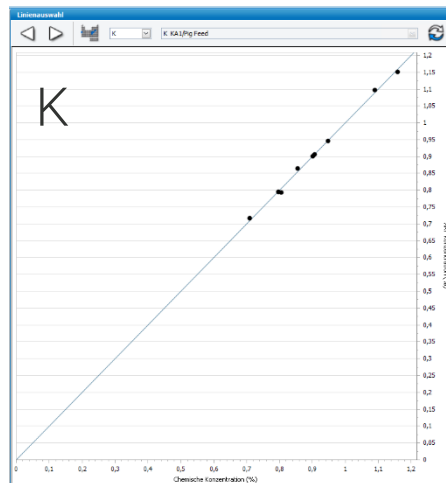
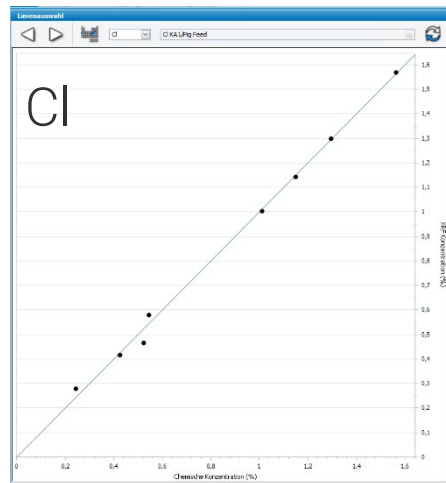
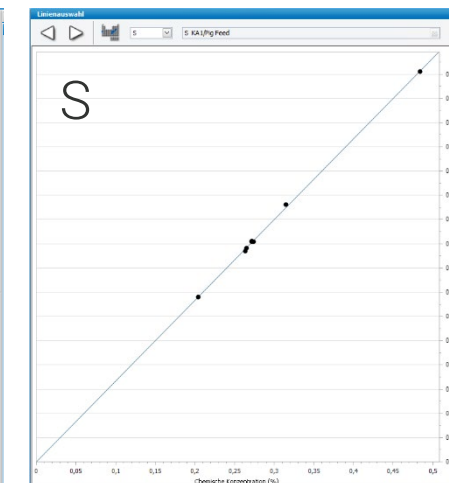
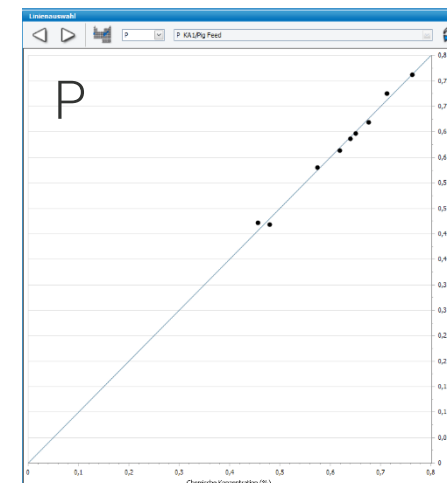
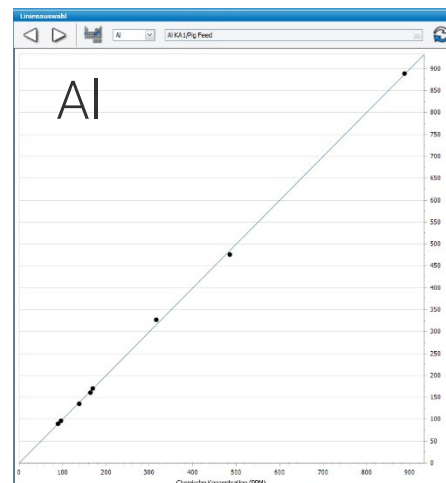
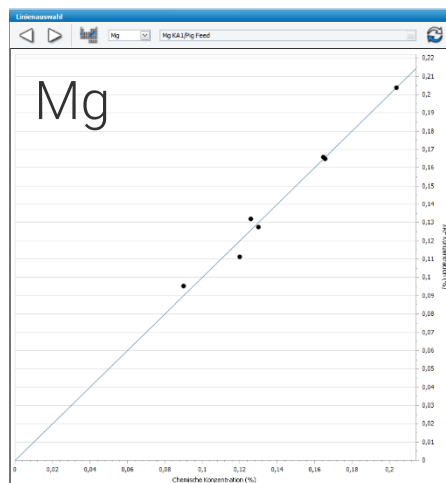
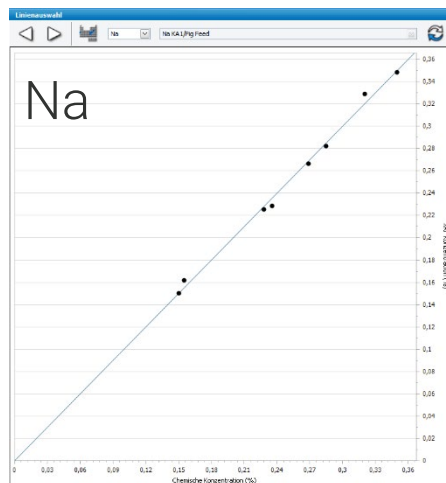


SPECTRA.ELEMENTS

- **WIZARD:** the proven tree-structure guide you through the calibration process
- Quick learning with intuitive User Interface. Hints provide help when needed

How to calibrate a XRF spectrometer

Example: Pig feed



Quality control with XRF

Example: Pig feed

Repeatability test

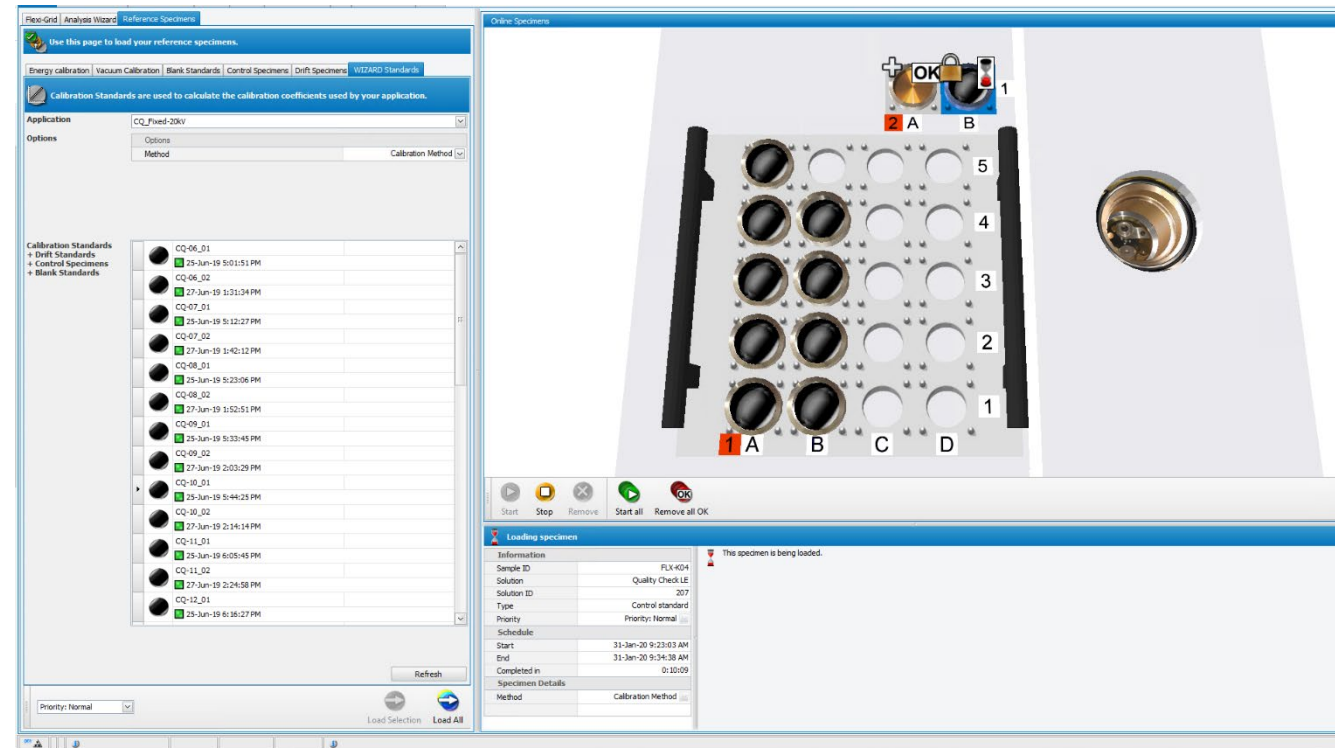
- Measuring the same sample multiple times
- Loading/unloading the sample between the measurements
- Precision: 2-3% for light element, <1% for medium and heavier element

Sample ID	Na (%)	Mg (%)	Al (PPM)	P (%)	S (%)	Cl (%)	K (%)	Ca (%)	Mn (PPM)	Fe (PPM)	Cu (PPM)	Zn (PPM)
Rep-1	0.298	0.120	160.9	0.654	0.279	1.67	0.980	0.931	89.9	312.9	140.9	175.8
Rep-2	0.302	0.119	160.7	0.654	0.280	1.66	0.978	0.930	88.8	312.4	141.2	176.4
Rep-3	0.302	0.115	157.3	0.652	0.279	1.66	0.972	0.931	90.2	312.7	140.6	174.7
Rep-4	0.297	0.116	165.0	0.650	0.278	1.64	0.972	0.912	89.7	312.3	139.8	174.5
Rep-5	0.297	0.116	165.0	0.650	0.278	1.64	0.972	0.912	89.7	312.3	139.8	174.5
Rep-6	0.283	0.115	166.8	0.647	0.277	1.63	0.959	0.923	88.3	309.6	139.1	173.7
Rep-7	0.283	0.115	166.8	0.647	0.277	1.63	0.959	0.923	88.3	309.6	139.1	173.7
Rep-8	0.283	0.115	166.8	0.647	0.277	1.63	0.959	0.923	88.3	309.6	139.1	173.7
Average	0.293	0.117	163.7	0.650	0.278	1.64	0.969	0.925	89.2	311.4	140.0	174.6
Std.Dev.	0.009	0.002	3.6	0.0031	0.001	0.01	0.009	0.005	0.8	1.5	0.9	1.0
Rel.Std.Dev.	3.0%	1.7%	2.2%	0.5%	0.3%	0.9%	0.9%	0.5%	0.9%	0.5%	0.6%	0.6%

Routine operation made easy Thanks to SPECTRA.ELEMENTS

Faster. Smarter. Easier.

- **Intuitive:** Quick learning with new User Interface
- **Tab Structure** enables easy navigation. No floating windows.
- **LOADER:** Intuitive interface for routine operation
- **RESULT MANAGER:** Access all your data quickly and get extended reporting
- **LIMS compatible:** For automatic reporting



The LOADER: load / unload samples; adjust priorities; start / stop your measurements; view your results.

Forage Analysis

What are the challenges?

Different matrices:

- Corn silage, grass silage, haylage, hay, small grain silage, total-mixed ratio (TMR).

Many elements:

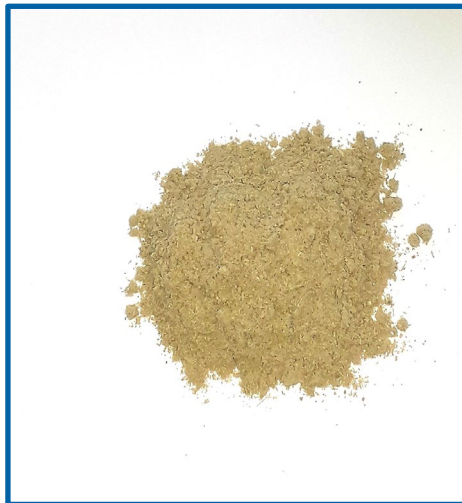
- Na, Mg, Si, P, S, Cl, K, Ca, Mn, Fe, Cu, Zn, ...

Wide concentration range:

- Low ppm to several wt%

Minimal sample preparation:

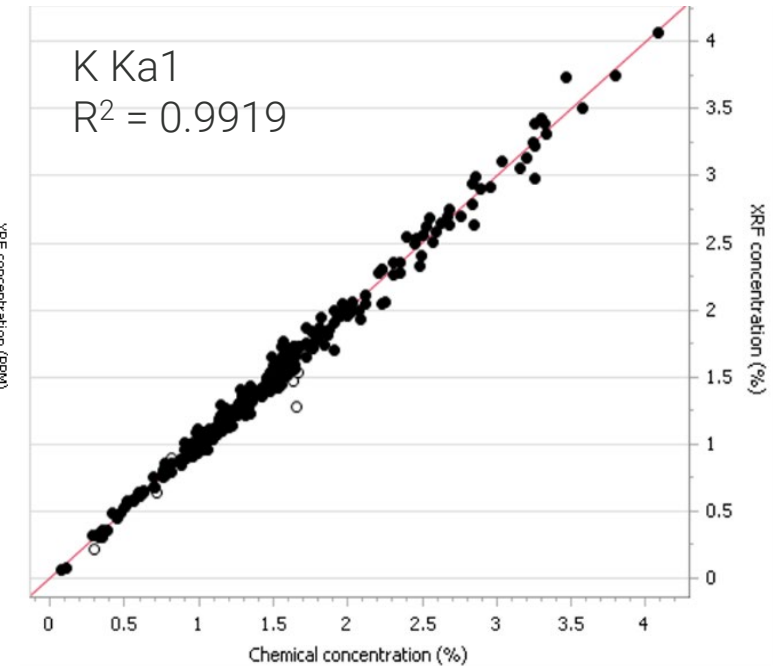
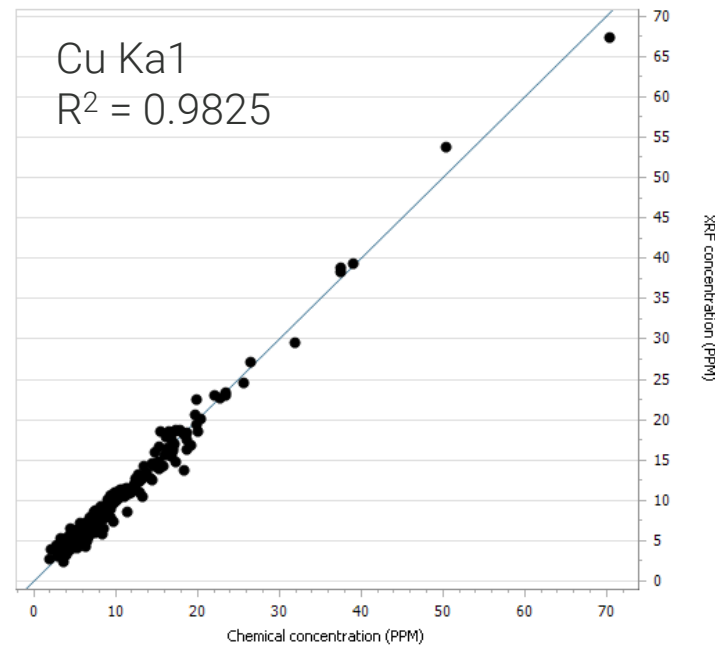
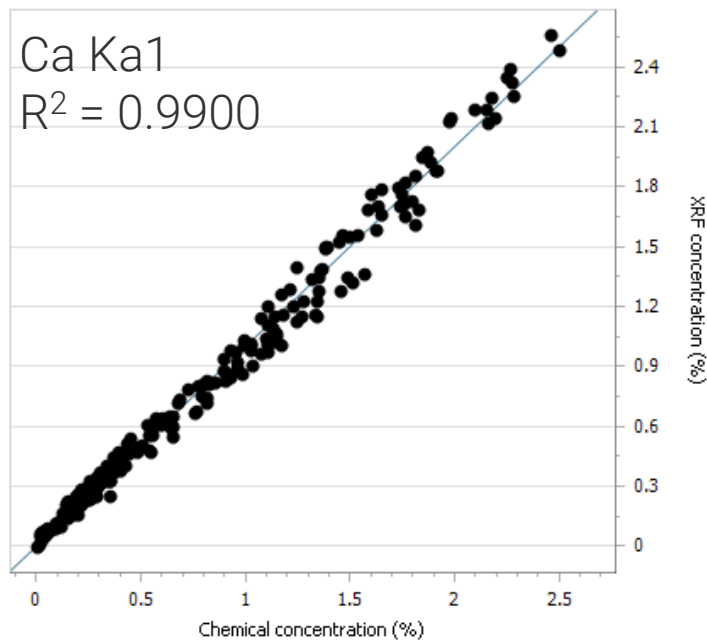
- Drying & grinding (like for FT-NIR)
- Measure as loose powder in a cup



Forage Analysis

What are the challenges?

- 270 secondary reference materials were used for the calibration
- All 7 material types can be covered by a single applications
- Time-to-result: 7 min



Forage Analysis

What are the challenges?

Repeatability test

- Excellent precision except for Na (due to Prolene® thin film)
- Comparable precision: Well ground loose powder vs. pressed pellet without grinding
- Accuracy: Will always be better with dedicated, matrix-based calibrations

TMR	Ca (%)	P (%)	Mg (%)	K (%)	S (%)	Na (%)	Cl (%)	Fe (PPM)	Zn (PPM)	Cu (PPM)	Mn (PPM)
Rep-1	0.519	0.339	0.385	1.432	0.197	0.076	0.379	376	316	69	131
Rep-2	0.513	0.340	0.384	1.429	0.197	0.081	0.378	373	315	69	129
Rep-3	0.515	0.339	0.381	1.437	0.197	0.086	0.378	375	315	70	130
Rep-4	0.521	0.338	0.387	1.431	0.197	0.093	0.378	374	315	69	129
Rep-5	0.517	0.340	0.397	1.435	0.197	0.100	0.378	375	316	70	130
...
Rep-20	0.510	0.340	0.386	1.427	0.197	0.094	0.379	373	316	69	128
Average	0.513	0.338	0.386	1.428	0.197	0.090	0.378	374	315	69	128
Std.Dev.	0.003	0.001	0.005	0.007	<0.001	0.011	0.001	1.4	1.0	0.3	1.5
Rel.Std.Dev.	0.6%	0.3%	1.2%	0.5%	0.2%	12.2%	0.2%	0.4%	0.3%	0.5%	1.1%



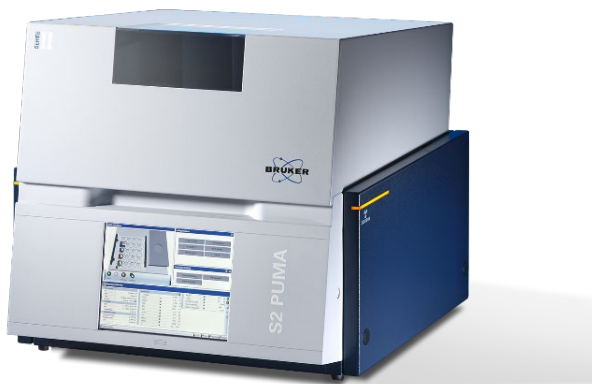
“EDXRF (energy dispersive X-ray fluorescence) is a newer technology that has evolved to be a cost-effective solution for the rapid determination of mineral content in various forage and feed materials. Where NIR is a rapid method for organic constituents, it is not valid for mineral determination. EDXRF is a complementary technology that allows for rapid (4 to 8 min) determination of multiple minerals (+10) relevant to animal nutrition. It can be run on the same ground material used for NIR determinations.”

Ralph Ward, CEO, Cumberland Valley Analytical Services (CVAS), *LinkedIn*

CVAS uses the S2 PUMA in several countries across the globe.

Quality Control of Maize Kernel

- Use of Maize / Corn: Animal feed (corn silage), biofuels, food (vegetable, flour, ...), starch, ...
- Analytical questions:
 - Macro and micro mineral contents and ratios (e.g., for dedicated animal feed diet)
 - Contaminant & safety (e.g., heavy metals)
 - Contents of P, Cl, and S (biofuel production)



Analyzing Maize Seedlings to investigate the effect of Fertilization

S2 PUMA Series 2

Research & Development Application

- Understand the effect of fertilization on the growth rate & resistance of different maize types
- Understand the nutritional uptake of based on the timing, amount and type of fertilizer
- GOAL (1): Optimize fertilization process
- GOAL (2): Determine/develop the optimal seeds



Analyzing Maize Seedlings to investigate the effect of Fertilization

S2 PUMA Series 2

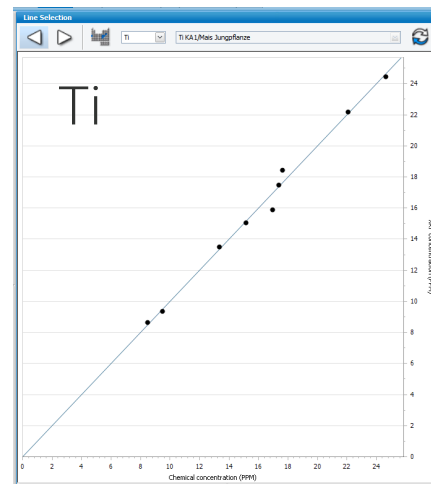
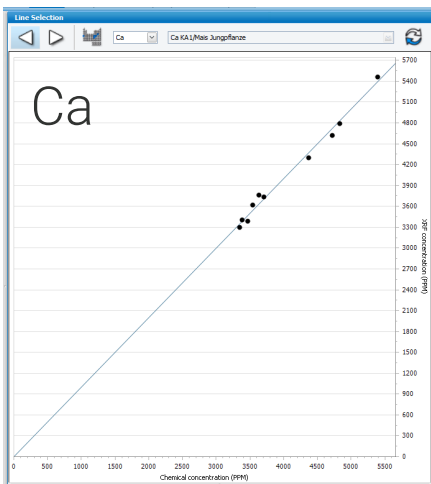
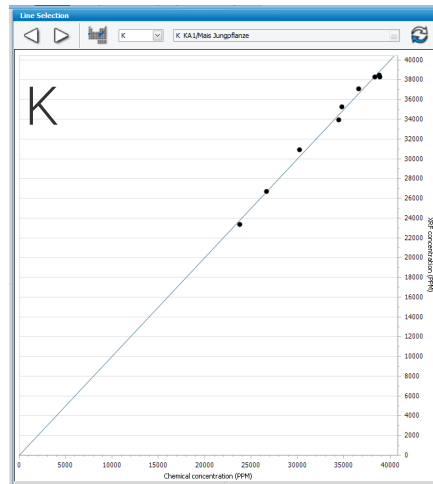
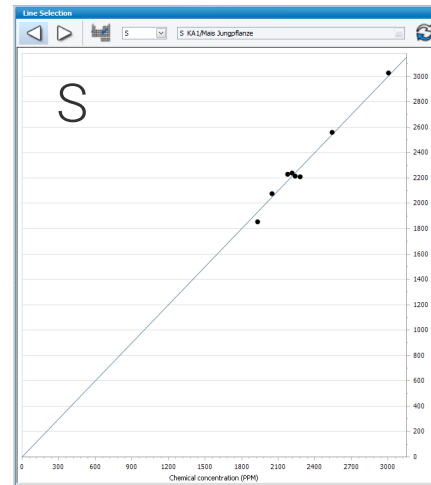
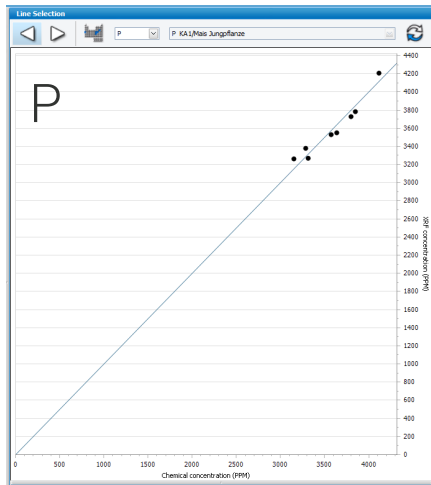
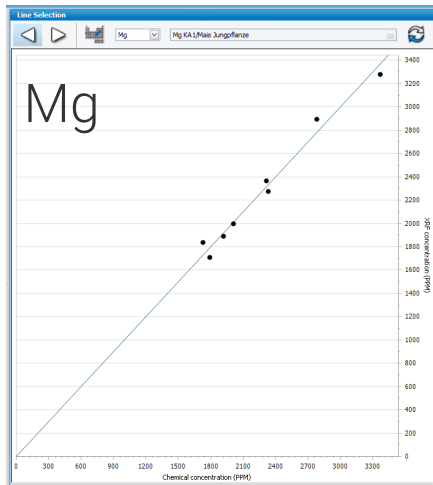
Exemplary Calibration

- Maize seedlings: Calibration with 10 secondary reference materials (measured by ICP)
- Covering 9 elements with concentrations ranging from few ppm up to ~ 4 wt% (Mg, P, S, Cl, K, Ca, Mg, Fe, Zn)
- Preparation options:
 - Loose powder: coarse or ground?
 - Pressed pellet: with or without wax?
- **Best performance:** Pressed pellet with ground material (no wax needed)
- **Time-to-result optimized:** Pressed pellet with ground powder and wax

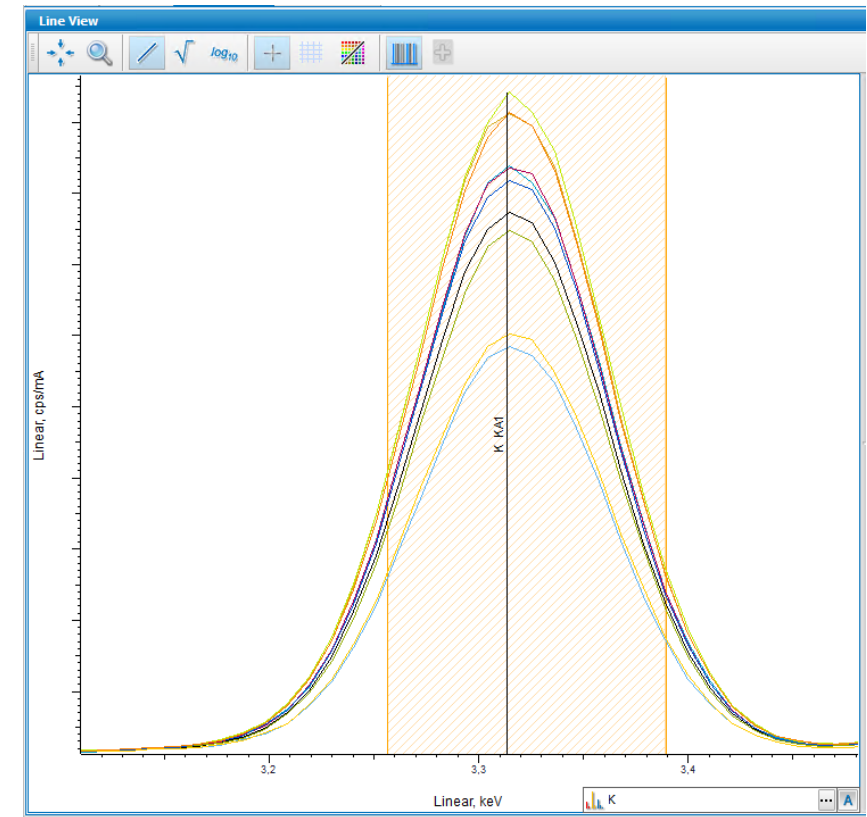


Analyzing Maize Seedlings to investigate the effect of Fertilization

S2 PUMA Series 2



- Exemplary calibration curves
- Peak for K (KA1)



Analyzing Maize Seedlings to investigate the effect of Fertilization

S2 PUMA Series 2

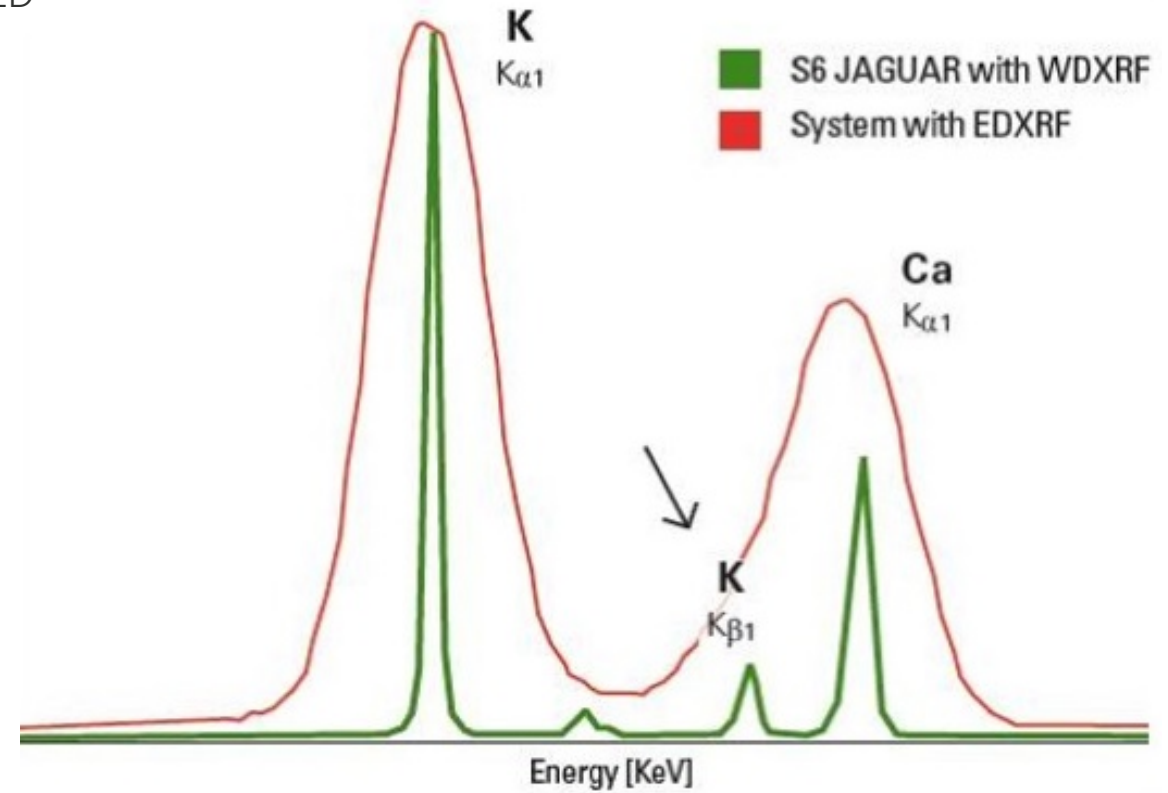
- Excellent precision macro and micro minerals.

	Mg (PPM)	P (PPM)	S (PPM)	K (%)	Ca (PPM)	Ti (PPM)	Mn (PPM)	Fe (PPM)	Zn (PPM)
Rep-1	1767	3809	2379	3.91	3366	10.6	26.8	329	27.7
Rep-2	1749	3802	2372	3.90	3369	10.5	26.7	328	27.7
Rep-3	1733	3798	2377	3.91	3384	10.5	26.2	327	27.7
Rep-4	1769	3791	2372	3.89	3388	10.5	27.0	328	27.9
Rep-5	1759	3795	2374	3.90	3380	10.5	25.9	327	27.4
Rep-6	1760	3798	2376	3.90	3357	10.5	26.8	328	27.4
Rep-7	1758	3789	2364	3.89	3392	10.5	26.8	328	27.4
Rep-8	1762	3792	2367	3.88	3392	10.6	26.3	329	27.5
Rep-9	1748	3787	2365	3.88	3369	10.5	27.0	327	27.8
Rep-10	1766	3778	2366	3.88	3391	10.4	26.8	325	27.4
Average	1757	3794	2371	3.89	3379	10.5	26.6	328	27.6
Std.Dev.	11	9	5	0.01	13	0.1	0.4	1	0.2
Rel.Std.Dev.	0.6%	0.2%	0.2%	0.3%	0.4%	0.5%	1.4%	0.4%	0.7%

Enhance the Performance for Light and Trace Elements

S6 JAGUAR: Benchtop WDXRF

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





“The XRF is mostly used to analyse TMR, forages, milk and urine, and allows us to gather accurate information about the concentrations of the minerals measured by the instrument. This is a very useful insight as it means we can not only determine what the cow needs for her own health and wellbeing, but also what minerals she will need to produce better quality milk.”

Dr. Rita Fornaciari, Nutristar
(*New Food Magazin*)

S6 JAGUAR enables high performance also for low concentrations of light elements in urine samples



Enhance the Performance for Light and Trace Elements

HighSense™ Goniometer: Impressive versatility

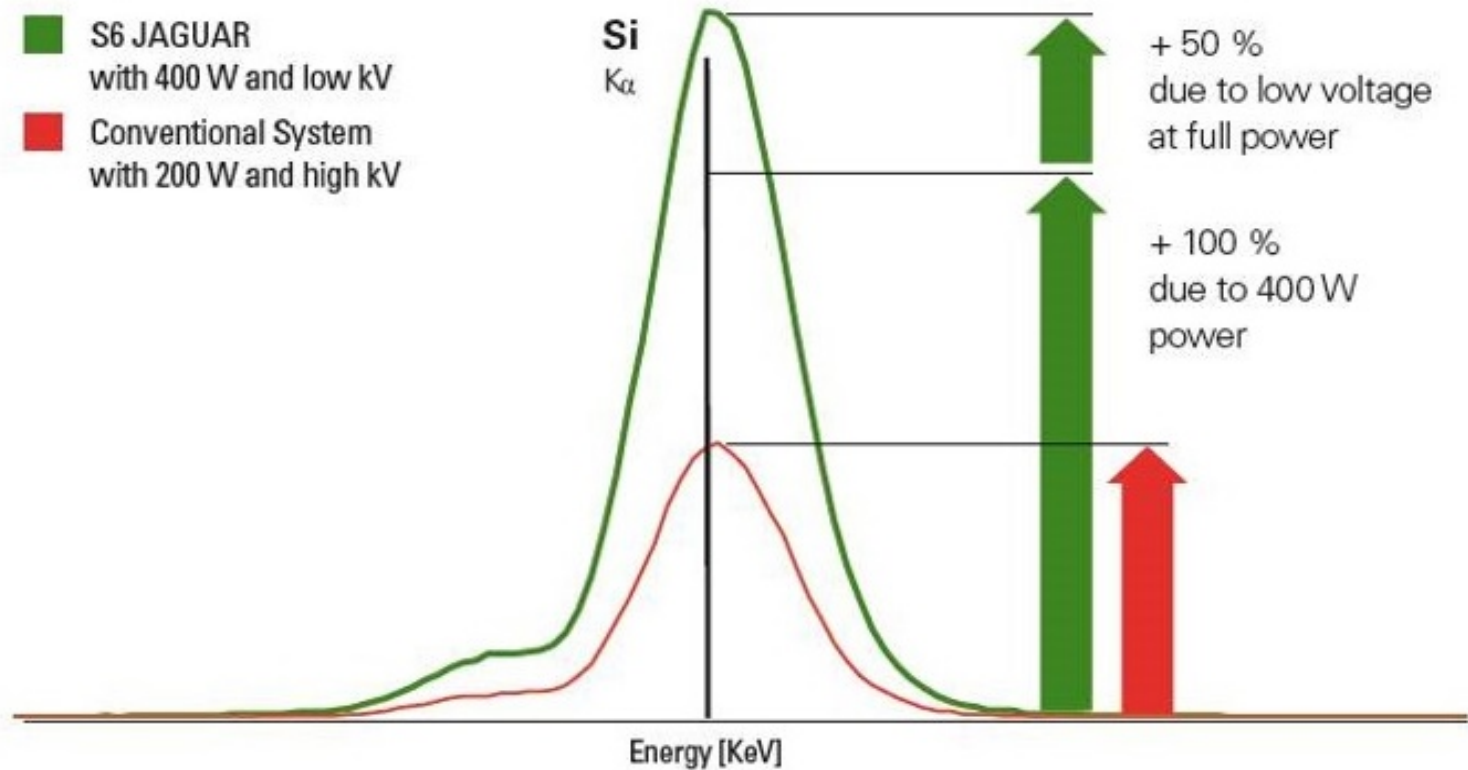


- 400 W excitation
 - 20 – 50 kV
 - 1 – 17 mA
 - Optimal settings for every single element at full power
- 5 position beam filter (optional) for improved peak-to-background ratio
- 4 sample masks (optional for different sample sizes)
- Vacuum seal for low-cost-of-operation
- Up to 4 analyzer crystals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

Enhance the Performance for Light and Trace Elements

HighSense™: Full 400 W excitation power

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





“This machine can carry up to 20 samples and takes approximately 10 minutes (per sample) to turn around solid sample results, and seven minutes for liquid samples. We achieve an excellent precision and accuracy with lower costs and in a shorter time than ICP.”

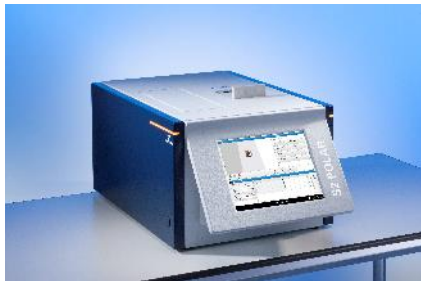
Simone Blotta, Lab Technician, Nutristar
(*New Food Magazin*)

S6 JAGUAR with XY Autochanger enables the required sample throughput

ELEMENTAL ANALYSIS IN THE FEED INDUSTRY

Summary

XRF: An excellent technique for rapid and reliable quantification of macro and micro mineral nutrients in animal feed and more!



- XRF is used for a wide range of applications in the food industry:
 - Macro and micro nutrition, toxic traces, foreign particles, raw material ID, quality control, process control, and more
- **Key benefits of XRF:** Wide range of elements and concentrations, simple sample preparation, ease-of-use, low operation costs, high accuracy & precision, high throughput.
- Bruker offers a full portfolio of laboratory equipment for food and feed applications:
 - **S2 PUMA Series 2** is the ideal choice for many food and feed applications – including milk powder, forage/feed premix, etc.
 - **S6 JAGUAR** for enhanced performance for low contents of light elements
 - **S2 POLAR** is used for traces in liquids/oils (e.g., P, Cl, S)



FT-NIR & XRF: Perfect Team-Work for Feed Applications

Combine speed with performance

FT-NIR for Organics

Composition analysis of ingredients and finished feeds

- Moisture, Fat, Protein, Fiber, Ash, NDF, ADF, and other parameters

Conformity testing

- Check for product conformity and uniformity
- Screening of organic contaminants

Material ID for ingredients and premixes

- Identification of pure materials
- Verification of components in solid, semi-solid and liquid ingredients



Grinding



NIR & XRF: Powder Analysis

XRF for Inorganics

Mineral nutrients composition

- Ingredients, premixes, and finished formulations, e.g. Na, Ca and P in feed, ash content

Premixes and additives

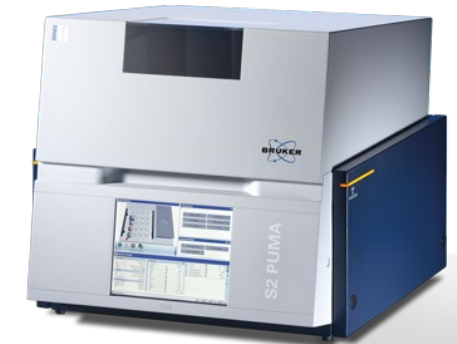
- Material ID
- Fe, Zn, and Co analysis

Contaminants

- Toxic metals taken up by plant in local soils e.g., As, Cd, and Pb
- Detection of fraud

Foreign Body Analysis

- Analyzing foreign materials in production

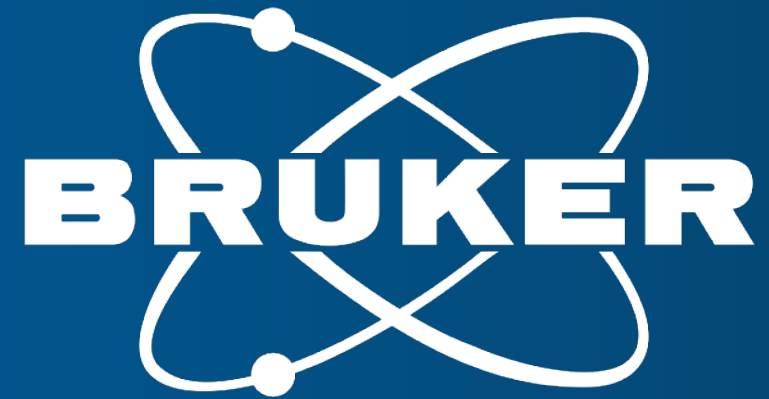


Any Questions?

Thank you!

Kai Behrens, Frank Portala, & Adrian Fiege





Innovation with Integrity