WEBINAR SERIES 2021

Elemental Analysis in the Food Industry

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From raw material to final product: Typical XRF application in the Food Industry

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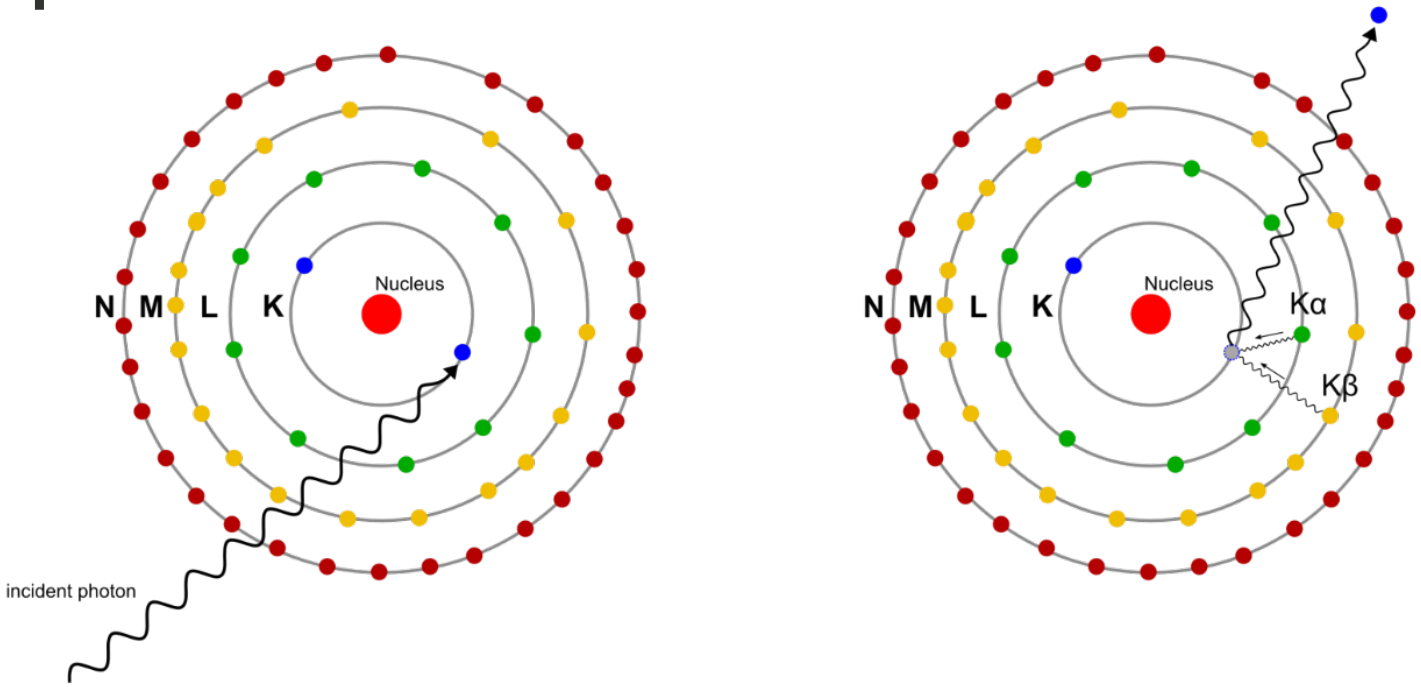
From raw material to final product: Typical XRF application in the Food Industry

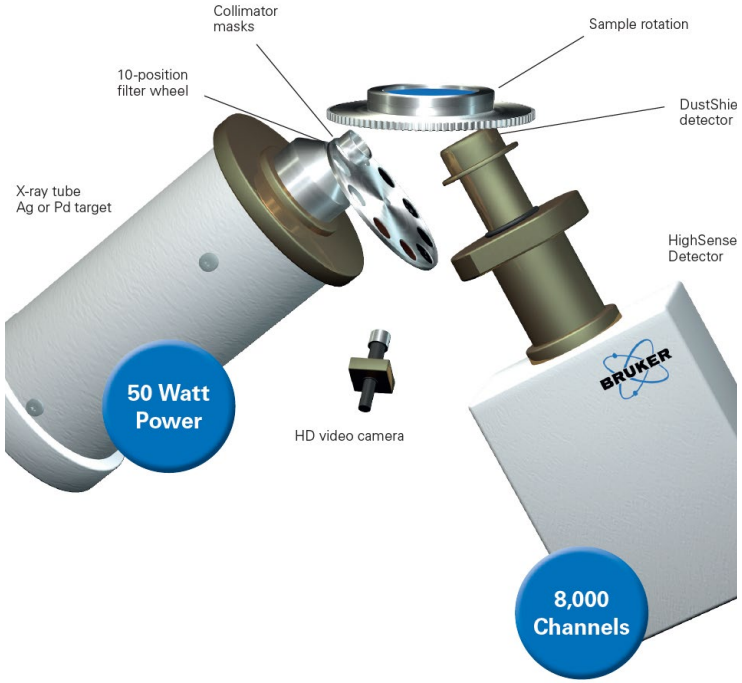
X-ray Fluorescence (XRF) Analysis Principle – Photoelectric Effect



- 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

Energy-dispersive XRF (EDXRF)
Wavelength-dispersive XRF (WDXRF)

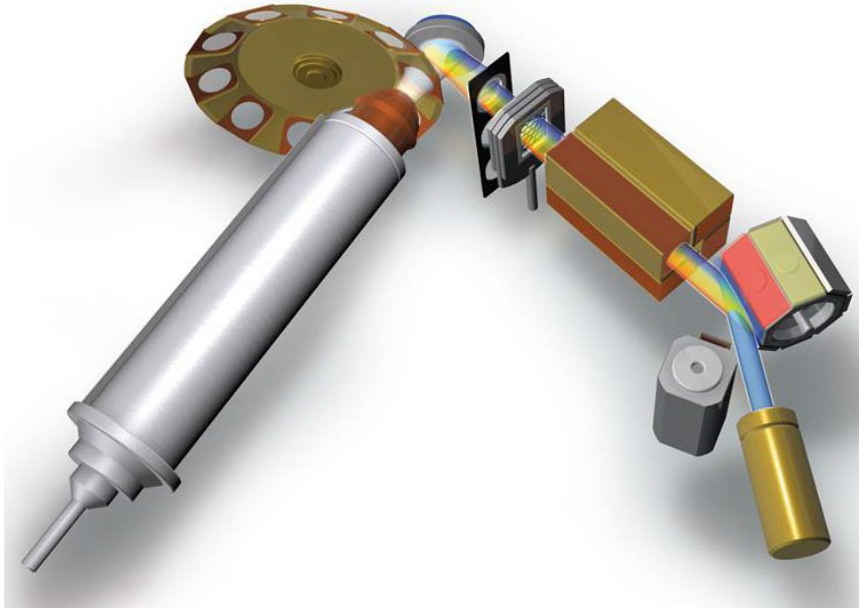
EDXRF



Direct or Polarized excitation

- Detects intensity & energy

WDXRF



Benchtop (400 W) or floor standing (up to 4 kW)

- Detects intensity at a given wavelength

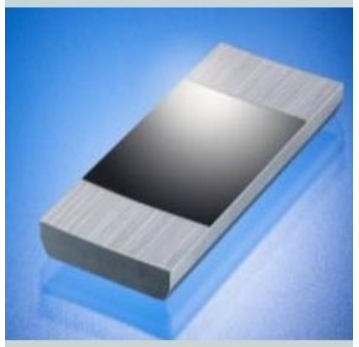
X-ray Fluorescence Analysis (XRF)
The Comparison of Energy and Wavelength Dispersive Spectrometers

EDXRF

- Mechanical simplicity
- Cheaper
- Sensitivities: down to the ppm level
- Records the entire spectrum
- 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
- Records the counts at a given wavelength
- Very fast analysis
- Highest sample throughput




Energy-dispersive XRF (EDXRF)
Wavelength-dispersive XRF (WDXRF)

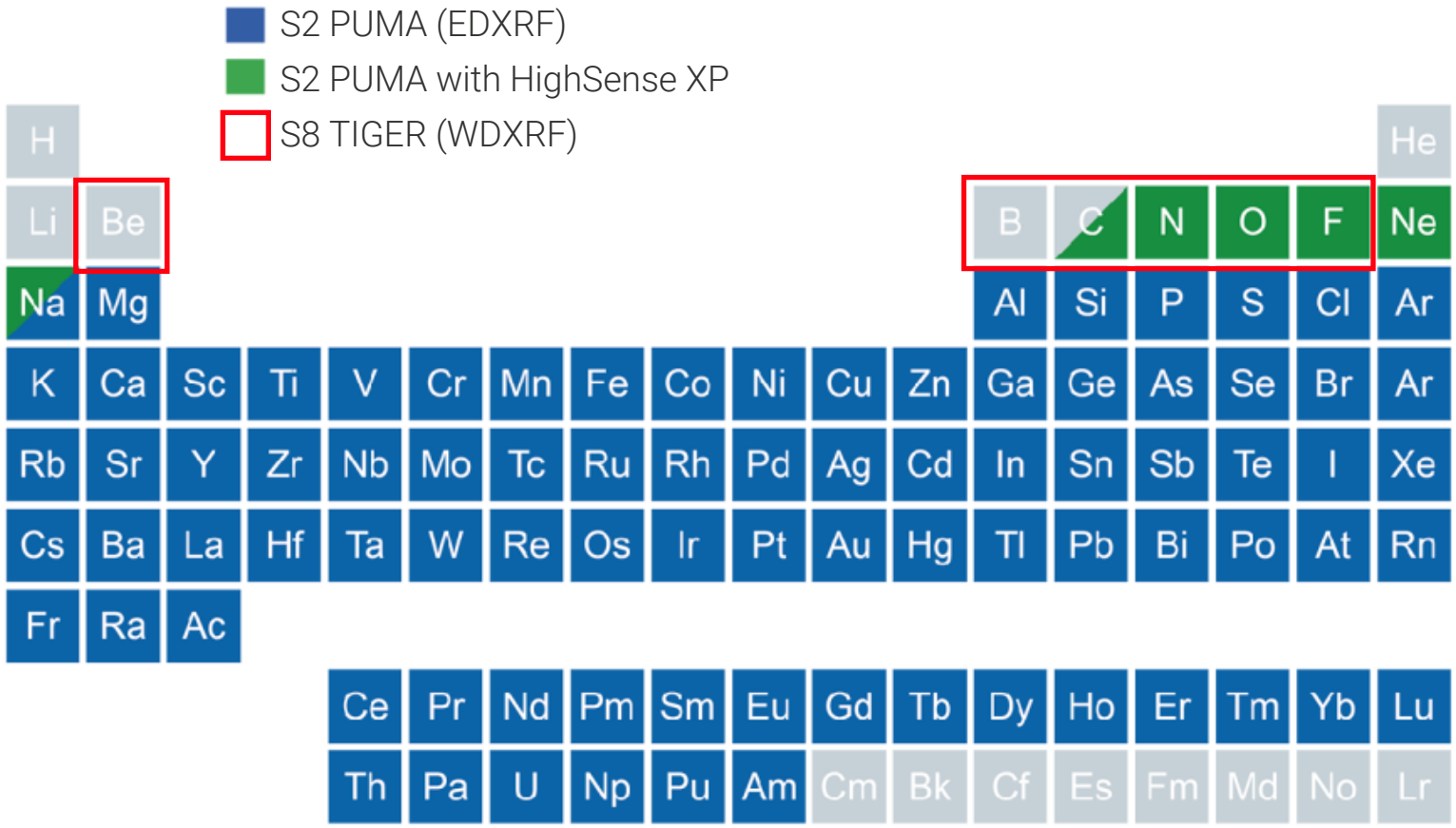
Elemental Ranges:

EDXRF: (C) F to Am

- Food/Feed: Na to U

WDXRF: (B) Be to Am

- Food/Feed: F to U



Sample Types

XRF can be used for all sample types and applications

- Solids, liquids, powders, pressed pellets, fused beads, bulk

A selection of atmosphere modes ensures optimal performance – even for light elements

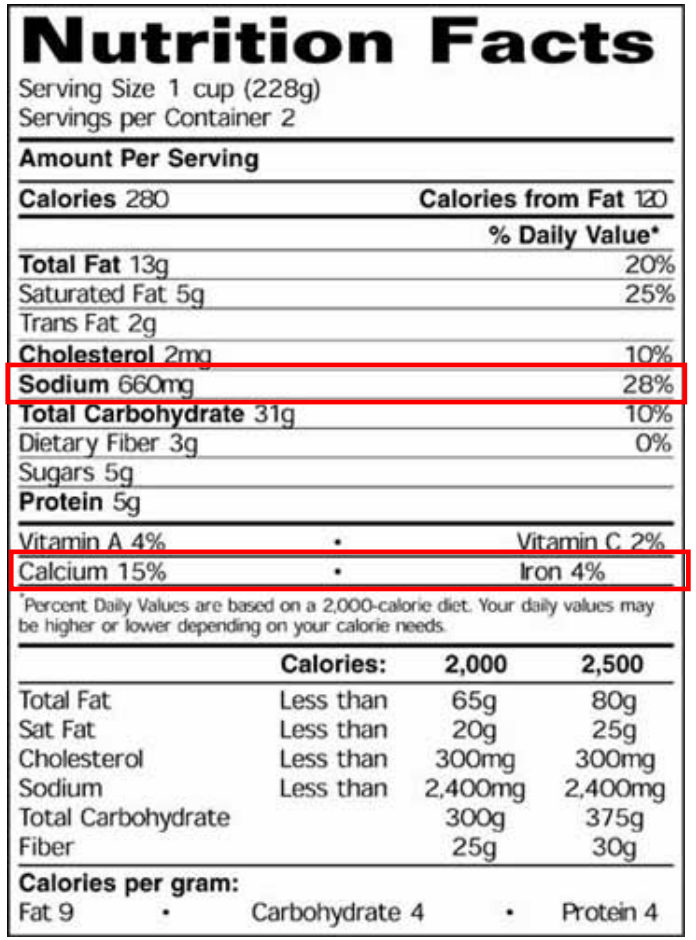
- Vacuum, helium, air, nitrogen






Reasons for Elemental Analysis in Feed & Food

- Legal regulations and recommendations
  - *Standards of Identity* specify type and amount of ingredients of certain foods
  - *Nutritional Labels* state e.g., contained Ca and Fe, and may inform about nutrient content claims ("low sodium")
  - *Food Inspection* ensures food stuffs meeting the appropriate laws and regulations



Nutrition Facts

Serving Size 1 cup (228g)
Servings per Container 2

Amount Per Serving	
Calories 280	**Calories from Fat** 120
	% Daily Value*
Total Fat 13g	20%
Saturated Fat 5g	25%
Trans Fat 2g	
Cholesterol 2mg	10%
Sodium 660mg	28%
Total Carbohydrate 31g	10%
Dietary Fiber 3g	0%
Sugars 5g	
Protein 5g	

Vitamin A 4% • Vitamin C 2%
Calcium 15% • Iron 4%

*Percent Daily Values are based on a 2,000-calorie diet. Your daily values may be higher or lower depending on your calorie needs.

	Calories:	**2,000**	**2,500**
Total Fat	Less than	65g	80g
Sat Fat	Less than	20g	25g
Cholesterol	Less than	300mg	300mg
Sodium	Less than	2,400mg	2,400mg
Total Carbohydrate		300g	375g
Fiber		25g	30g

Calories per gram:
Fat 9 • Carbohydrate 4 • Protein 4

Reasons for Elemental Analysis in Food

- Food safety
  - Avoid toxic chemicals (e.g. Ni, Pb), foreign material (e.g. metal, plastic particles)
- Quality control
  - Characterization of raw materials
  - Monitoring food during processing
  - Analysis of the final product
- Research & development
  - Constant need for optimization: healthier, cheaper, longer lasting etc.

Examples for Elemental Analysis in Food & Animal Feed

Dairy production

- Animal feed / feed supplement quality control
  Ensure the correct contents and ratio of Ca, P, Mg, Na, K, Cl and S
  - Critical for the lactation cycle and, thus, for high quantity and quality milk
- Monitor Urine and Milk
  - Analyze Ca, P, Mg, Na, K, and Cl.
  - Monitor the healthy of the cow and the feedback to changes in diet
- Use the speed of XRF to drive immediate actions at the farms

Examples for Elemental Analysis in Feed & Food

Agriculture

- Soil: Analyze nutrients and toxic elements in soils from major (Al, Si) to minor (Na, Mg, K, P) to trace element (As, Pb) levels
  - Use to results to determine actions such as the selection of the right type and amount of fertilizer
- Fertilizer quality and production control
  - Precise analysis of major active components (P, K, Mg) and trace elements (Zn)
  - Monitor toxic components such as As, Cd, and U

Examples for Elemental Analysis in Feed & Food

Food

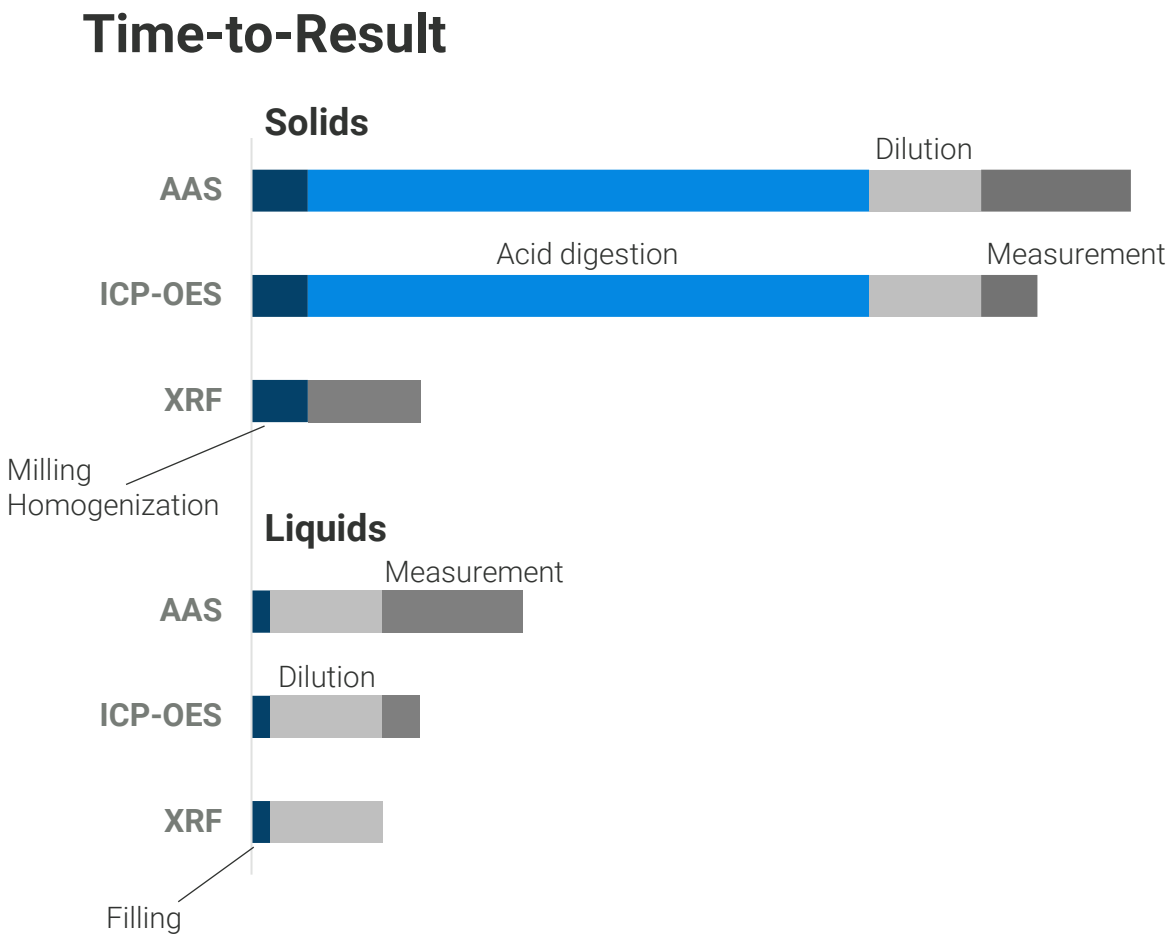
- **Milk powder:** Fe fortification, Ca and Mg monitoring
- **Salty Food/Snacks:** Cl / Cl_2 compliance for salt labeling requirements (NaCl, KCl or $CaCl_2$)
- **Rice and grains:** Fe fortification or trace process metal contamination?
- **Bakeries:** TiO_2 as product brightener
- **Chocolate:** Toxic trace elements (Pb, Al)

Pet Food

- Ash content (Na, Mg, K, and Ca oxides) as regulatory requirement, nutritional additives (e.g., Mn, Fe, Zn)

Advantages of XRF
Time-to-Result: XRF vs. ICP-OES

- Effective quality and process control requires the shortest time-to-result possible
- This is the time needed from sampling to the final quantitative result
- Any advantage results in:
  - Higher sample throughput
  - Stable industrial processes due to immediate feedback
  - Constantly high product quality



Advantages of XRF
Cost of Ownership: XRF vs. AAS and ICP

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/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)

ELEMENTAL ANALYSIS IN THE FOOD INDUSTRY

Food & Animal Feed Application Examples

Process Control in Edible Oil Refineries

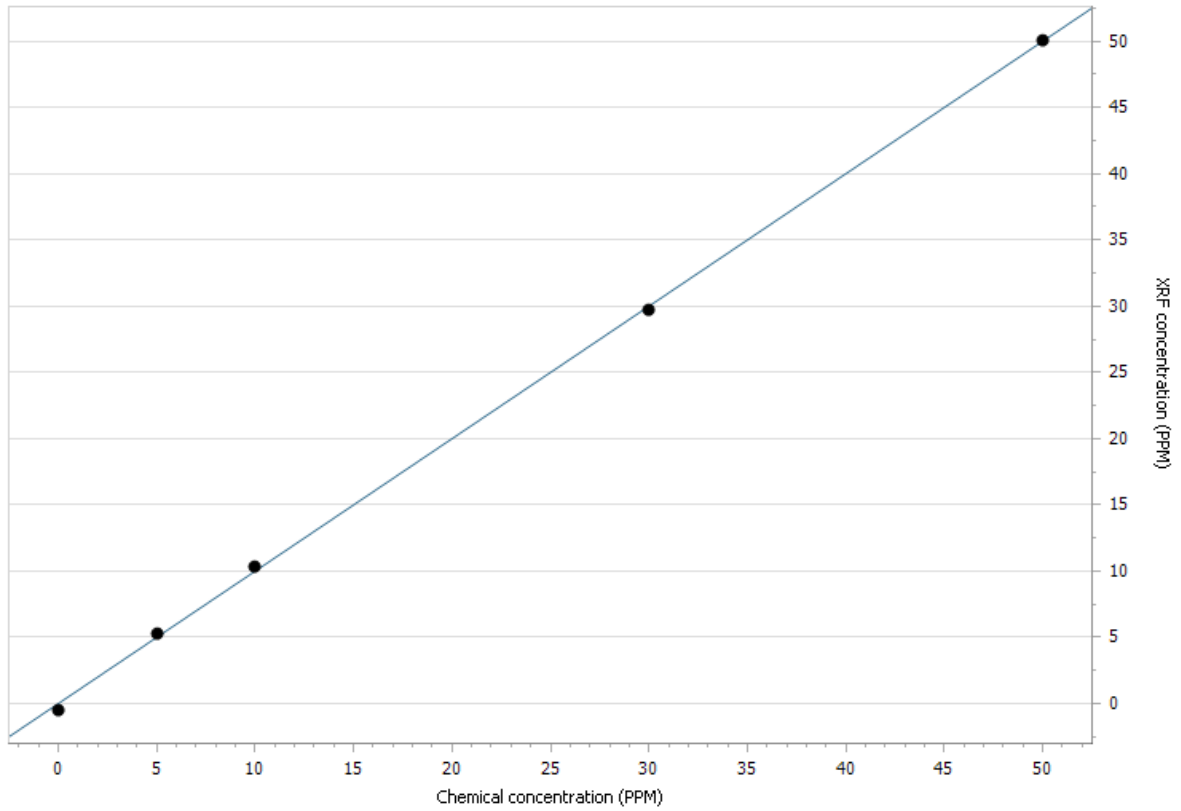
- In edible oil **refinery process**, ease of use and immediate feedback of results is more important
- Time-consuming ICP-OES analysis is a disadvantage
- Clear **advantage of XRF** for process control
- **P** is one of the key elements for proper **process control**
- Phospholipids do have a **negative impact** at some process steps and on the **final product**, e.g. on shelf lifetime
- Total P is measured **to monitor** the **phospholipids removal**

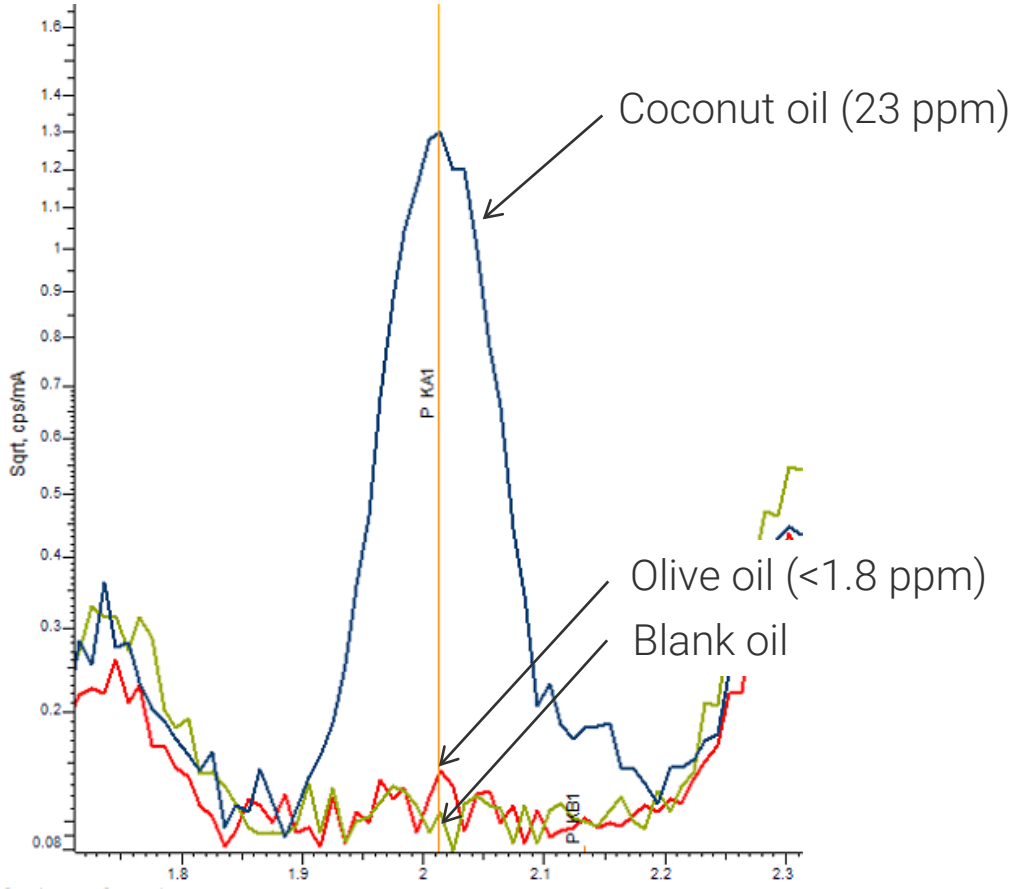
Used Cooking Oil (UCO) and Biofuels

- **Cooking oil** used in fast-food chains and restaurants is usually recycled
- Later the UCO is used to produce other products, quite often for **biofuels production**
- In biofuels, elements such as **P, S and Cl** must not exceed certain limits
- For **fast and easy process control** in biofuels refineries
- **Fast time-to-result** analysis when truck or vessel is waiting for its approval in a biofuel refinery

P in Cooking Oil
S2 POLAR

- Lower concentration range (0 - 50 ppm) of P calibration (LLD: 0.4 ppm)

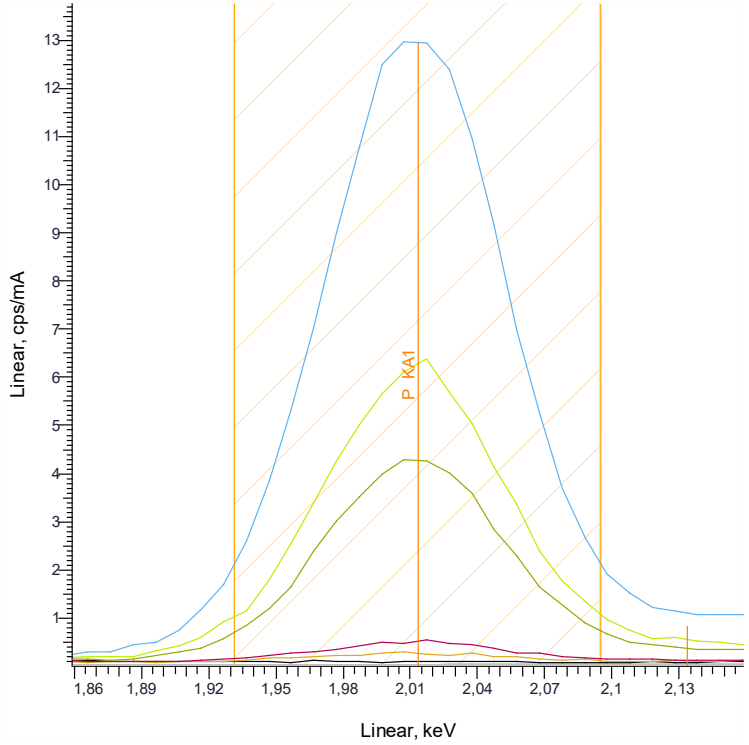




P in Edible Oil - Repeatability S2 POLAR

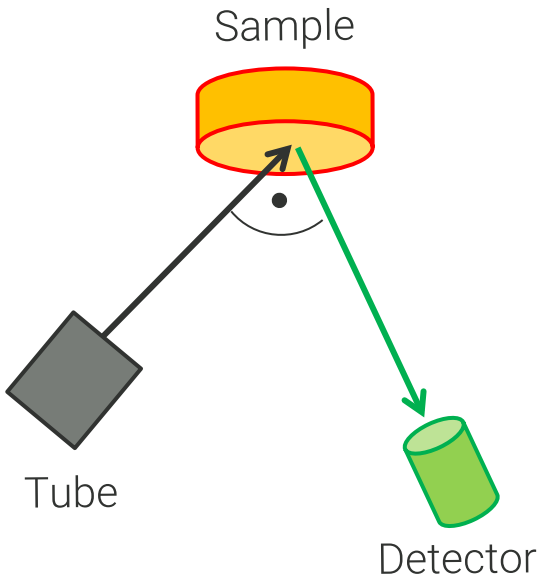
- 10 ppm QC sample - **Excellent repeatability** for such low P concentrations
- Simple and straight forward, without extensive sample preparation
- LLD: 0.4 ppm P (300 s, with SampleCare™ cup)

Measurement #	P [ppm]
1	10.4
2	10.4
3	10.2
4	10.2
5	10.2
6	10.4
7	10.2
8	10.2
9	10.1
10	10.3
Average	10.22
Abs. Std Dev.	0.11
Re. Std. Dev.	1.10%

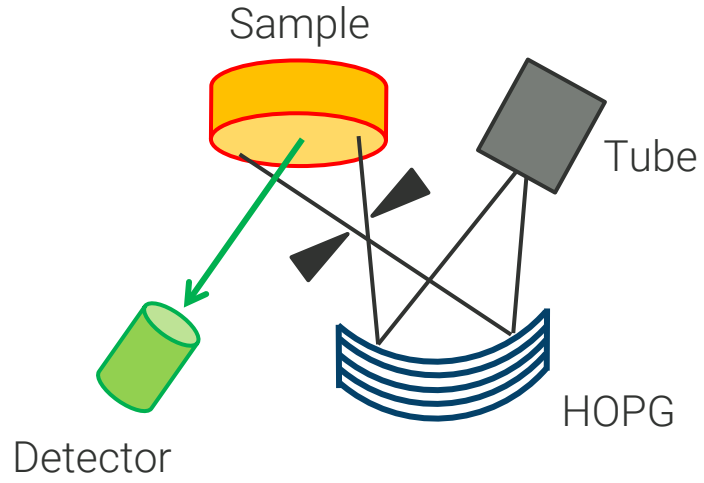


EDXRF Excitation Techniques
Direct Excitation vs. Polarized Excitation

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)

Edible Oils and Biofuels
S2 POLAR

- For fast and easy **process control**
- For fast, incoming crude oil or raw material analysis
- **At edible oil refineries**
- **At biofuel refineries**
- Quick and simple sample preparation, much faster than ICP-OES
- Reliable **results within minutes**
- **P, S and Cl** are the most important elements, others of interest are K, Ca, Fe, Ni, and Cu
- **The S2 POLAR offers excellent analytical performance and fast, reliable results within minutes.**

Quality Control of Maize Kernel

- Use of Maize / Corn: Food (vegetable, corn flakes, flour, …), starch, animal feed (corn silage), biofuels
- 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)

Quality Control of Maize Kernel S2 PUMA Series 2

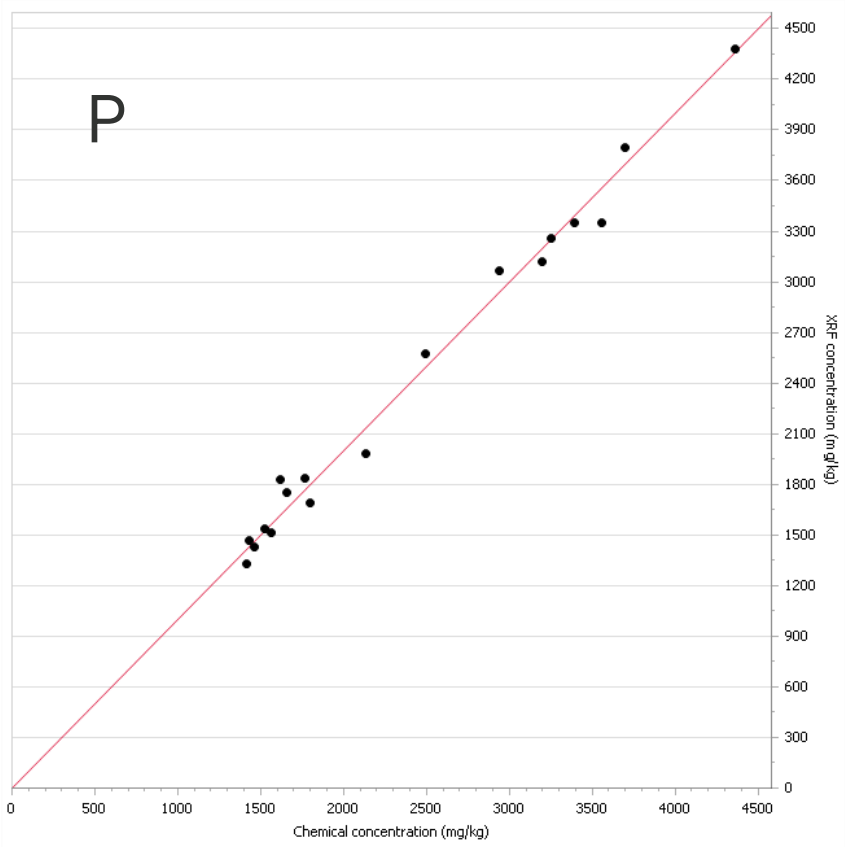
- Calibration with 23 secondary reference materials (measured by ICP)
- Covering 9 elements with concentrations ranging from few ppm up to ~ 1 wt% (Mg, P, S, Cl, K, Ca, Mg, Fe, Zn)
- Selected preparation: Pressed powder pellets with wax

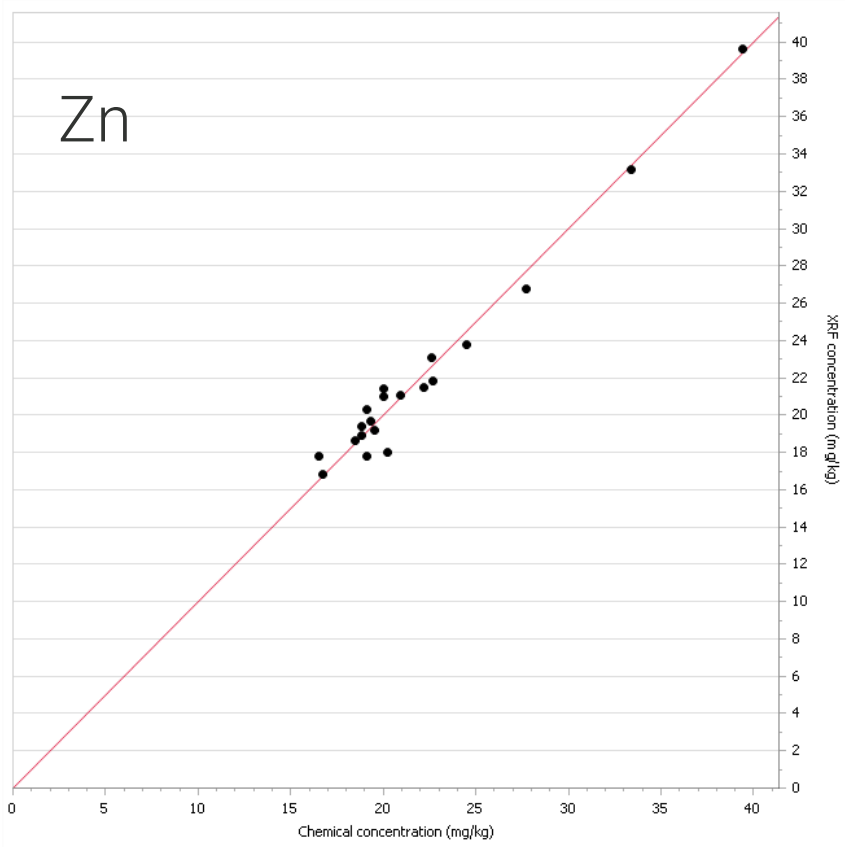
Quality Control of Maize Kernel S2 PUMA Series 2

- Exemplary line P KA1 and Zn KA1

Line	P KA1
Conditions	20 kV, auto. current, no filter
Concentration range	1412 – 4363 mg/kg
Std. dev.	105 mg/kg
R^2	0.9881

Line	Zn KA1
Conditions	40 kV, auto current, 500 µm Al-Filter
Concentration range	15 – 40 mg/kg
Std. dev.	1.9 mg/kg
R^2	0.9731





Quality Control of Maize Kernel S2 PUMA Series 2

- Excellent precision and accuracy for macro and micro minerals.

	Mg (mg/kg)	P (mg/kg)	S (mg/kg)	Cl (mg/kg)	K (mg/kg)	Ca (mg/kg)	Mn (mg/kg)	Fe (mg/kg)	Zn (mg/kg)
ICP	1140	3254	1152	380	3232	43	6	15.4	20.9
S2 PUMA	**1207**	**3279**	**1149**	**384**	**3295**	**58**	**5.6**	**16.3**	**21.0**
SD	13	13	8	4	17	3	0.7	0.7	0.1
rel. SD (%)	1.07	0.40	0.74	1.02	0.52	5.00	12.42	4.00	0.39
Abs. diff.	**67**	**25**	**3**	**4**	**63**	**15**	**0.4**	**0.9**	**0.1**

How to make Food?
How to best analyze raw material quality?

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

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

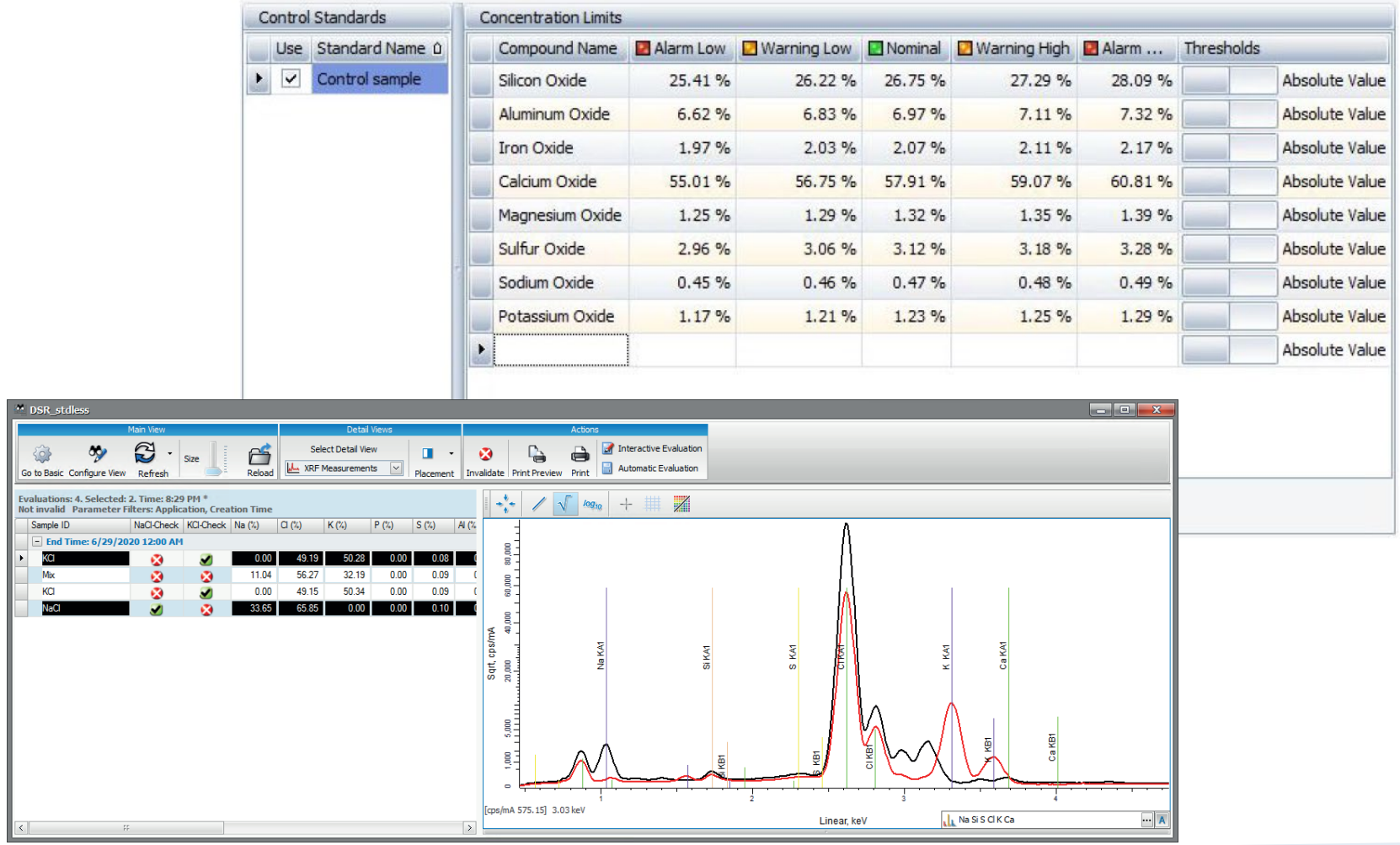
SMART-QUANT: Push-button solution for quick and reliable analysis of unknown samples

S6 JAGUAR: Full WDXRF performance in a benchtop unit

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 / Cl_2 compliance for salt labeling requirements (NaCl, KCl or $CaCl_2$)

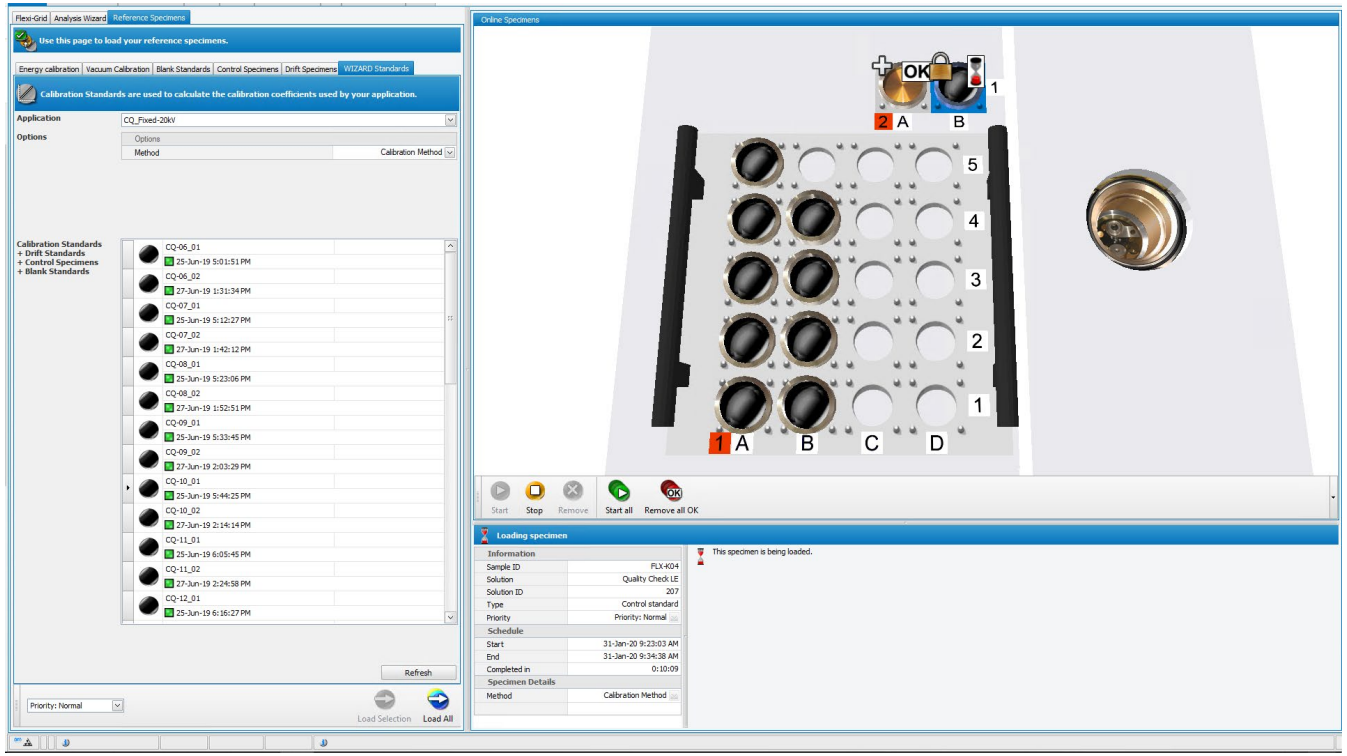


Routine operation made easy
Thanks to SPECTRA.ELEMENTS

Faster. Smarter. Easier.

Easier to use

- Quick learning with new User Interface
- Hints provide help when needed
- WIZARD: the proven tree-structure guide you through the calibration process
- LOADER: Intuitive interface for routine operation
- RESULT MANAGER: Access all your data quickly and get extended reporting



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

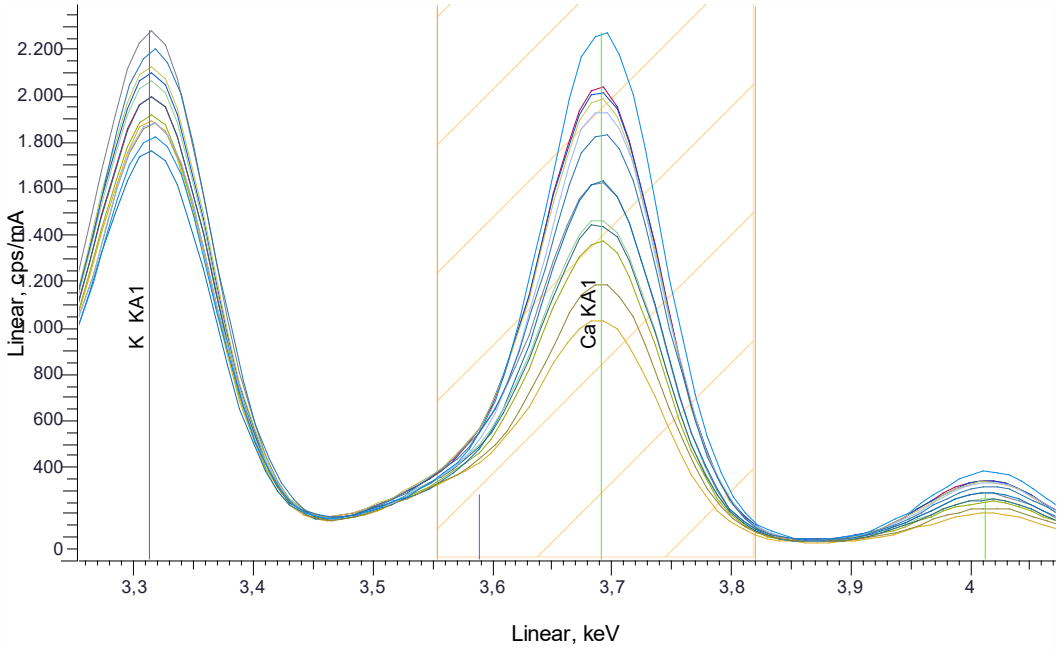
Quality Control of Infant Formula S2 PUMA Series 2

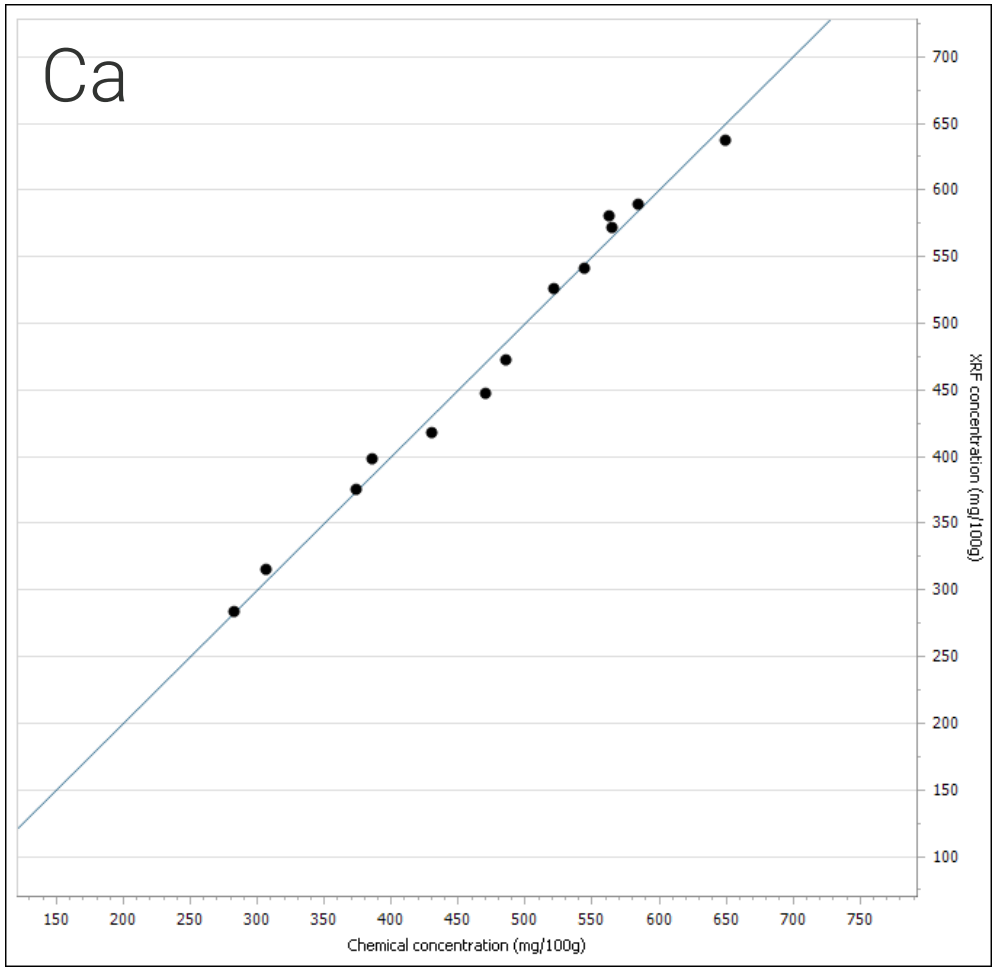
- Milk powder is essential for baby nutrition and the accurate control especially of vital elements is critical
- Since milk powder is the only food source over a long period of time, children's health strongly depends on the correct concentration of Na, Mg, P, K, Ca and Fe.
- Additional elements needs monitoring since they could possibly contaminate the milk powder such as Cl, e.g., from ion exchange resins.

- EDXRF and benchtop WDXRF are optimal solutions for quick and reliable raw material ID, process monitoring and quality control

Quality Control of Infant Formula S2 PUMA Series 2

- Milk powder measured as pressed pellets
- Elements covered:
  Na, Mg, P, Cl, K, Ca, Fe
- ~ 5 min per sample





Quality Control of Infant Formula S2 PUMA Series 2

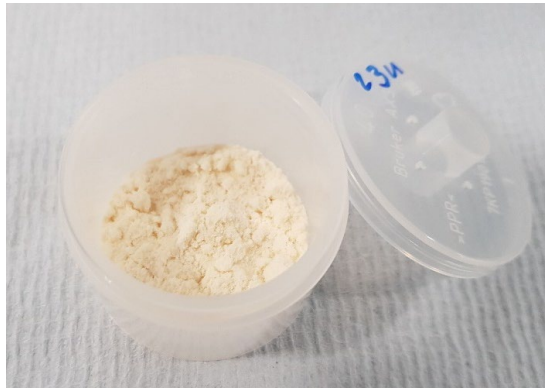
- Excellent precision!
- Low detection limits, even for light elements (~7.5 mg/100g Na, ~3.0 mg/100g Mg)

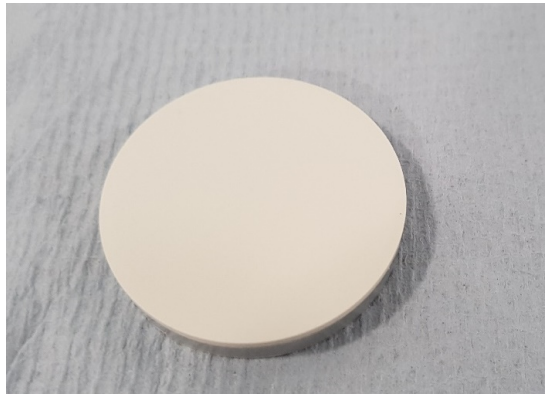
Element [mg/100 g]	Na	Mg	P	Cl	K	Ca	Fe
Average	240	61	257	418	597	381	4.86
Std.Dev.	2.5	1.4	0.8	0.8	4.5	2.1	0.05
Rel.Std.Dev (%)	**1.04**	**2.34**	**0.33**	**0.20**	**0.75**	**0.55**	**1.06**

Ash content in milk components
S2 PUMA Series 2

Gravimetric ash determination is very time consuming (ashing at 550°C), while the measurement on a S2 PUMA Series 2 takes only a few minutes

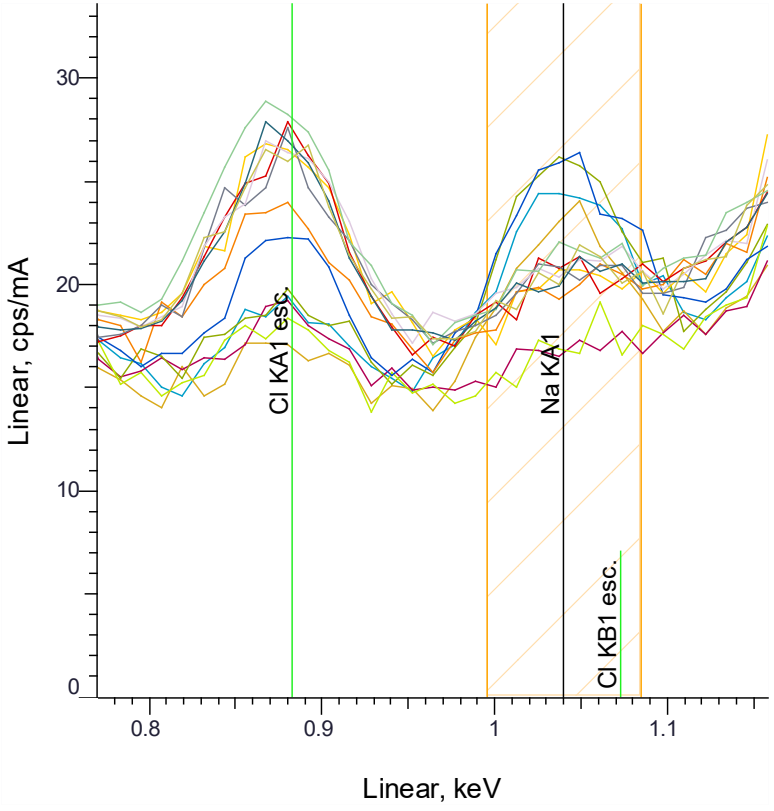
- Calibration with 8 secondary reference materials (ICP, titration, gravimetric)
- Components tested: Lactose, milk protein, whey protein, milk-calcium
- Covering up to 9 elements with concentrations ranging from few ppm (e.g., Fe, Zn) up to couple wt% (e.g. Ca, P)
  (Na, Mg, P, Cl, K, Ca, Mg, Fe, Zn)
- **Preparation test:** Pressed pellets (no wax) and loose powder



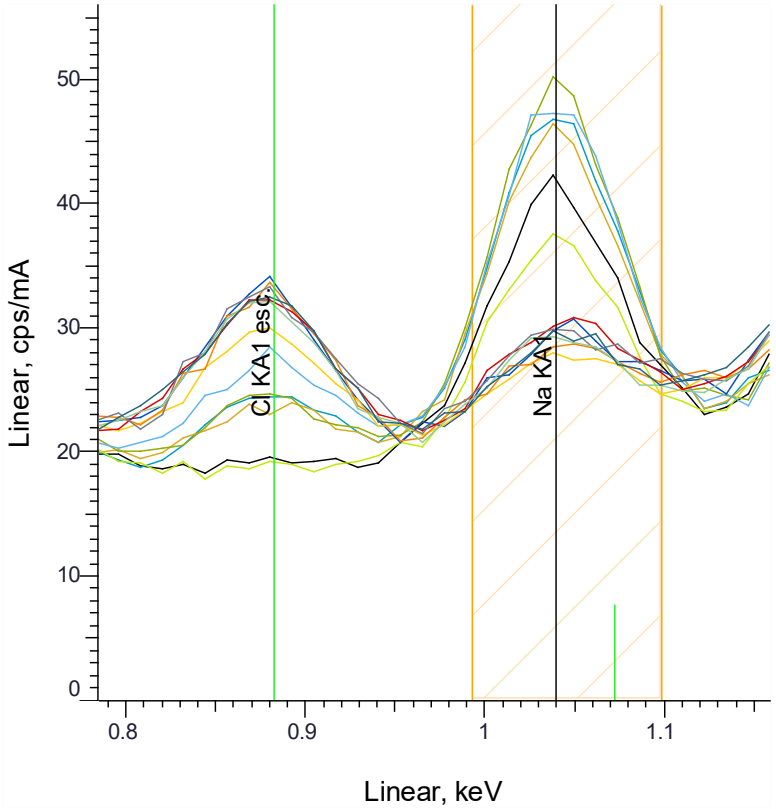


Ash content in milk components S2 PUMA Series 2

- Milk protein sample preparation test



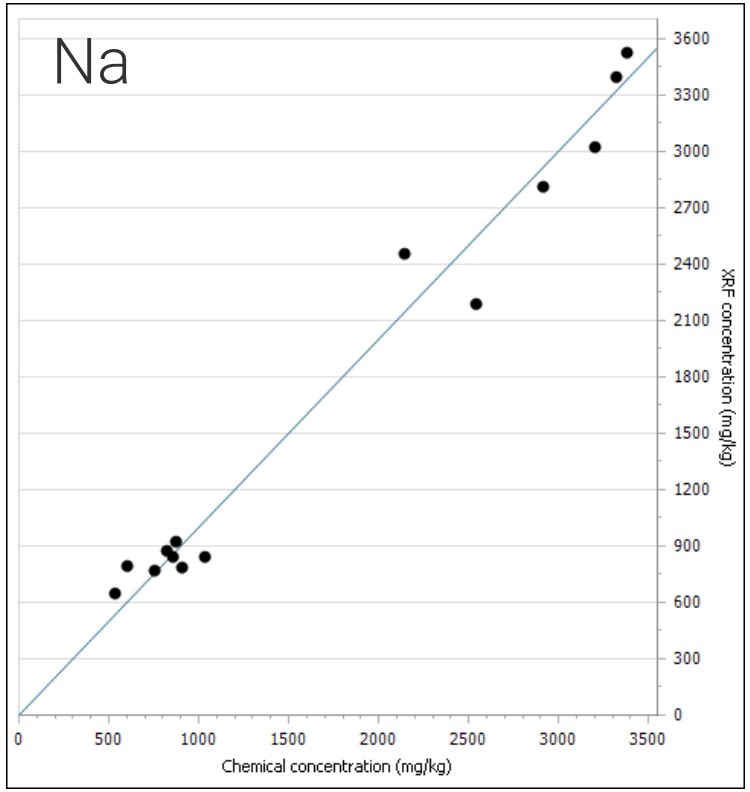
- Loose powder
- He, 4 µm Prolene® foil
- ~27 cps/mA



- Pressed pellet
- Vacuum
- ~50 cps/mA

Ash content in milk components S2 PUMA Series 2

- Milk protein sample preparation test



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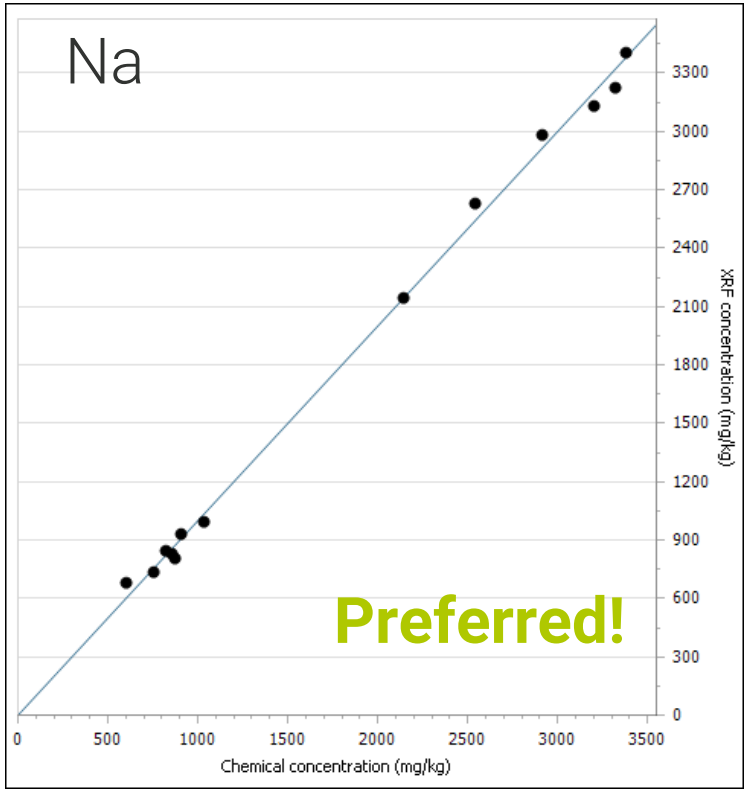


- Pressed pellet
- Vacuum
- ~50 cps/mA

Bruker provides tailor-made XRF Solution for your applications S2 PUMA Series 2

State-of-the-art Software and Hardware

- Powerful 50 W X-Ray Tube
- HighSense™ Technology
- SampleCare™ Technology
- SPECTRA.ELEMENTS
- And more…

Ready for all sample types, sizes and throughput requirements

- Single, Carousel, Mapping-Stage, XY-Autochanger, XY-Automation

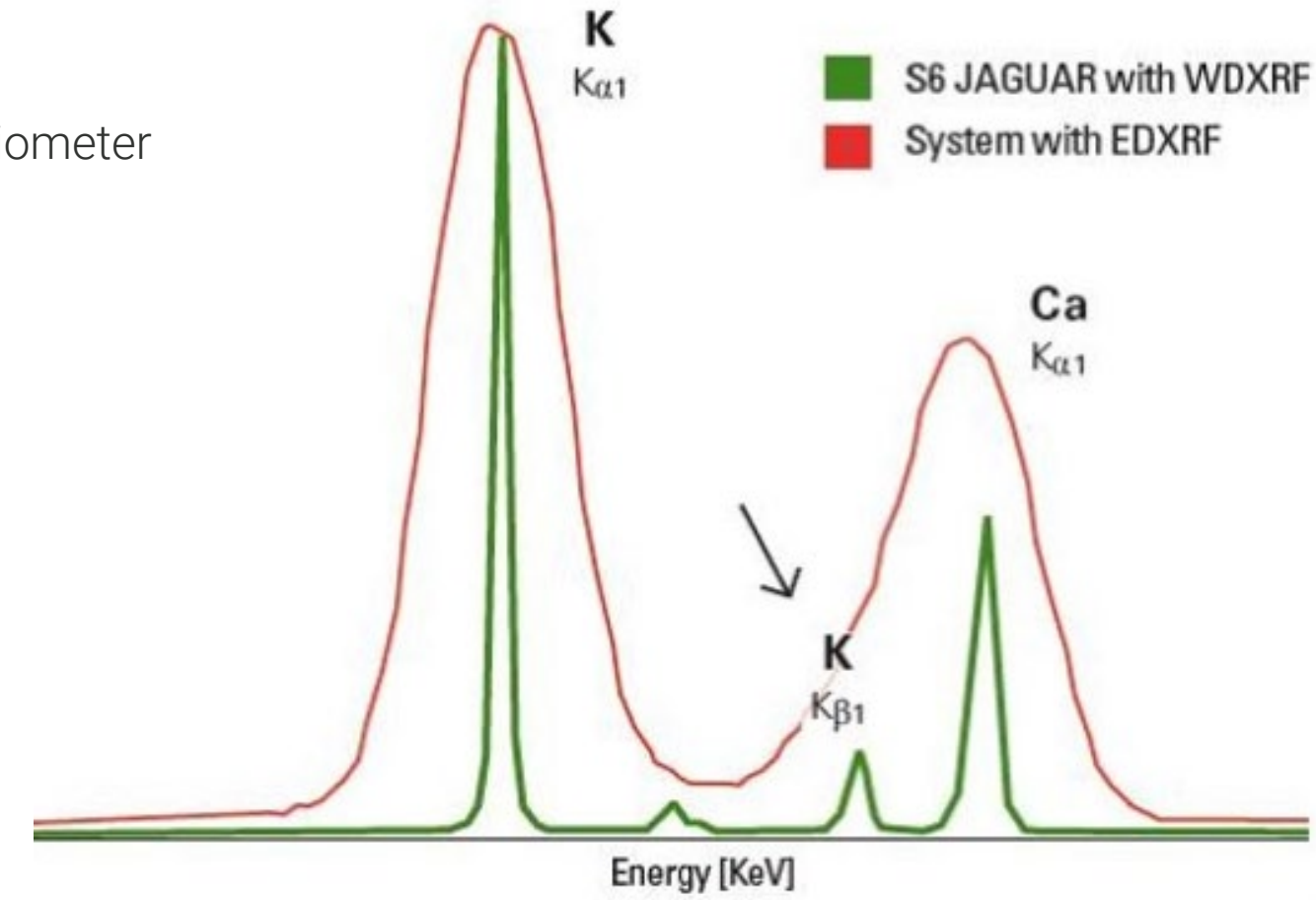
Various options to ensure optimal performance

- Light element configuration
- Vacuum, He, N_2, and Air mode
- Detector profiling
- TouchControl™
- Sample Rotation
- SMART-QUANT FP
- QUANT Solutions
- 21 CFR Part 11 compliance
- IQ-OQ Documentation
- And more ….

Enhance the Performance for Light and Trace Elements S6 JAGUAR: Benchtop WDXRF

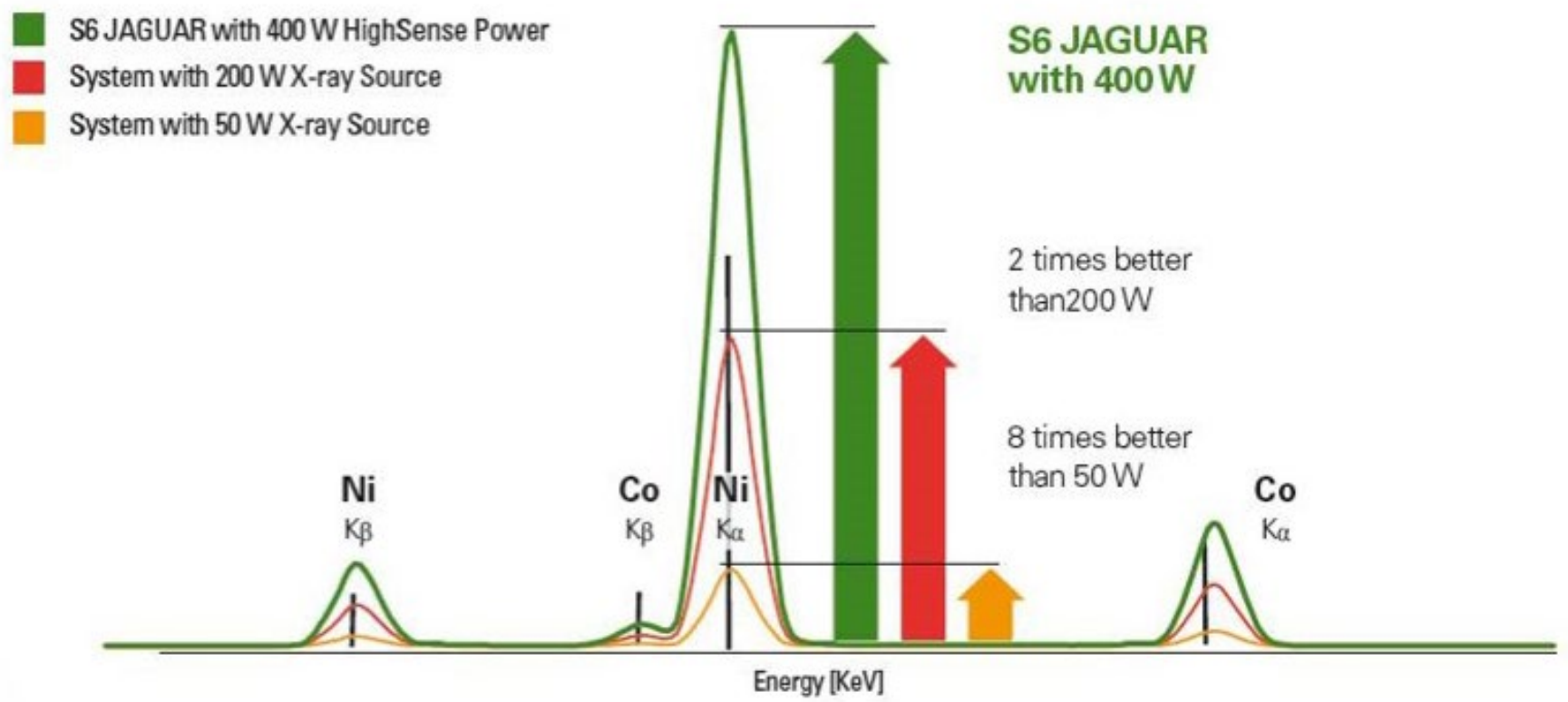
HighSense Goniometer: High Resolution

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



Enhance the Performance for Light and Trace Elements 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



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 countrates for high calibration ranges

Food Analysis
What are the challenges?

Requirements for forage and feed analysis

Many elements:

- Na, Mg, Si, P, S, Cl, K, Ca, Mn, Fe, Cu, Zn, Se, Mo, …

Wide range of concentrations:

- Low ppm to several wt%

Minimal sample preparation:

- Loose powder, 7 g of material placed in liquid cup with Prolene® thin film 4 µm

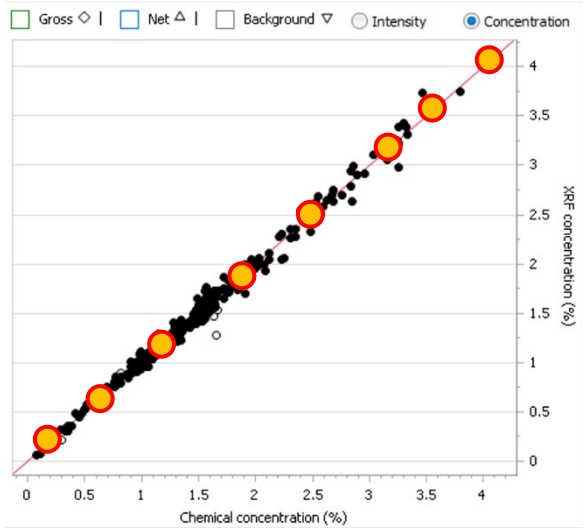
Several calibrations for different matrices

- Typically using large sets of reference materials and secondary standards which may be not very stable

What is Forage? How to prepare it for analysis?

- Plant based material (mainly leaves and grasses) eaten by grazing livestock
  - Includes hay and silage
- Often used as the ‘base’ of animal feed for livestock animals
  - Can be mixed with premix / additives to give a particular nutrition content
- Preparation should include some form of homogenization
  - Special mills (e.g. WC ring mill) reduce the grain size and provide a homogeneous sample within minutes

Forage Analysis
S2 PUMA Series 2



Calibration curve K Kα1

FORAGE QUANT transfer kit

- To transfer customer “forage” solution from central lab to plant
- 8 synthetic glass standards
- 1 drift correction standard
- USB Stick containing all method files

	Compositional Ranges [wt%]
Na	0 – 1.06
Mg	0.07 – 0.75
Si	0.16 – 4.75
P	0.06 – 0.74
S	0.06 – 0.56
Cl	0.04 – 2.67
K	0.11 – 6.07
Ca	0.01 – 2.65
Mn	3.9 – 288.6 ppm
Fe	20.9 – 2853.1 ppm
Cu	1.2 – 38.7 ppm
Zn	6.5 – 150.4 ppm
Se	0 – 586.5 ppm
Mo	0 – 27.7 ppm

Forage Analysis
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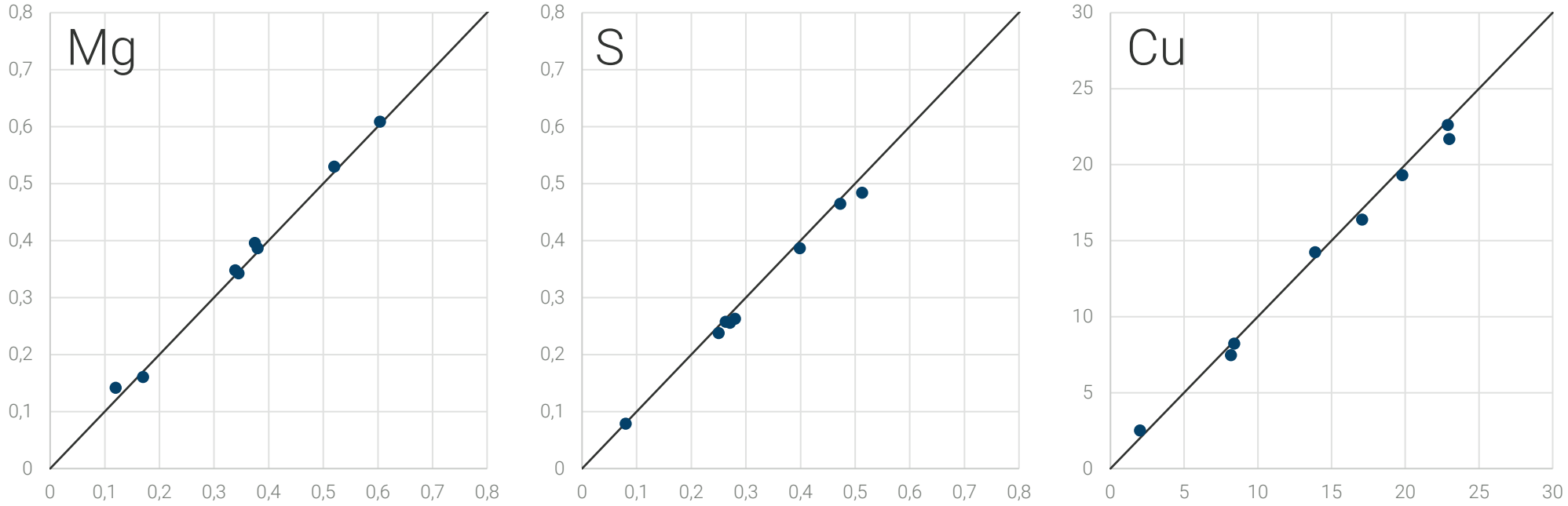


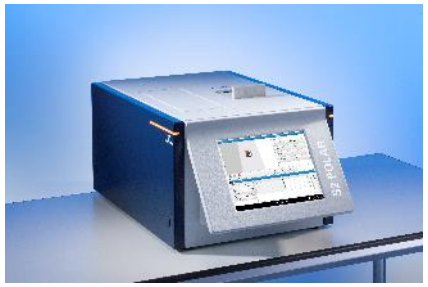
Exemplary results of a Forage solution transfer:

- **Excellent correlation** between Master (central lab) and Client (plant) ensuring lab-to-lab comparability

ELEMENTAL ANALYSIS IN THE FOOD INDUSTRY

Summary

Summary
XRF: An excellent technique for Food elemental analysis



- 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.
  - S2 POLAR is used for traces in liquids (e.g., P, Cl, S)
  - S6 JAGUAR is used for very low trace element contents (<10 ppm) and for (low) F

Perfect Team-Work for Food Applications
FT-NIR & XRF combine speed with performance

Grinding

NIR & XRF: Powder Analysis

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

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

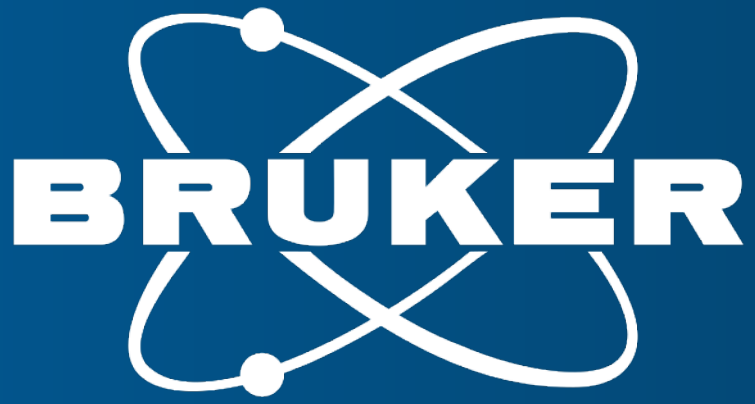
Any Questions?

Learn more about ‘Pharma & Cosmetics’

In our 13.10.2021 webinar – Sign Up Now!

Thank you!

Kai Behrens, Frank Portala, Adrian Fiege



Innovation with Integrity