Application Note XRF 701

Ensuring Food Quality and Safety with Elemental Analysis

X-Ray Fluorescence (XRF) spectrometers provide fast and reliable results required for QC/QA

The globalized food supply chain poses serious challenges to producers, brands, consumers, and governments. An ever-intensifying worldwide competition, a growing number of contaminants and a strong demand for food quality control (QC) are constantly increasing the number of stringent regulations. Here, safety concepts such as HACCP (Hazard analysis and critical control points) are implemented to ensure safe edibles at any point in the production chain.

The compliance with these directives requires continuous product quality monitoring in all steps of food production. This includes quality checks

- at customs
- when receiving raw materials
- during processing
- before packaging
- and spot tests of the final product.

S2 PUMA Series 2 with XY Autochanger

Elemental analysis of food guarantees that contaminants are monitored and that foreign particles within the food matrix are detected. Other quality control applications include the inspection of mineral supplements and other vital mineral elements. For those and other food industry-related applications, modern X-ray fluorescence (XRF) spectrometers offer several key advantages when compared to conventional, wet-chemical techniques, such as ICP-OES (Inductively Coupled Plasma Optical Emission Spectrometry):

- no or minimal sample preparation required
- short time to result
- low operation costs
- wide range of concentrations and elements
- no re-calibration necessary
- ease-of-use (no dedicate lab-staff required)

In this report, we present analytical results for several food products and food components, highlighting the versatility of Bruker's X-ray fluorescence spectrometers and their suitability for numerous applications in the food industry and research.

Raw material identification (ID)

Starting at the beginning, the quality of a final product depends significantly on the raw material being used for its production. Here, XRF can provide rapid and reliable information, allowing not only the identification of a material (e.g., NaCl vs. KCl) but also the determination of its purity (e.g., traces of toxic metals). The S2 PUMA Series 2, an energy dispersive XRF (EDXRF) spectrometer, comes with a powerful and versatile standardless solution, SMART-QUANT FP, enabling fast qualitative and quantitative screening of raw materials. The 22-postion XY Autochanger allows to load large batches and individual priority samples at any time, enabling high throughput and flexibility.

Process monitoring and quality assurance (QA)

During food production, many factors can influence the final quality. For quality assurance (QA), process monitoring is often inevitable. It doesn't matter if you need to know whether the Fe content in your rice or wheat grains comes from fortification or hazardous metal parts during production (foreign body identification); Or, if the nutritional additives (e.g., Mg, Ca, Zn) are within the desired thresholds; Or, if the heavy metals (e.g., As, Cd, Pb) are kept well below the strictly regulated limits: XRF provides you the information you need!

S6 JAGUAR – a 400 Watt benchtop WDXRF

Application Example 1

Dairy and Infant Formula

Like all processed human food, dairy products must fulfill certain quality standards. When it comes to healthy and safe food for our youngest (e.g. infant formula), these regulations are even more stringent. Here, XRF can handle many analytical tasks.

For dairy products, elemental compositions are required for various components such as lactose, milk fats, and milk proteins. Here, the new S2 PUMA Series 2 is an excellent solution to meet the analytical requirements; e.g., precise measurement of Na in milk protein (Fig. 1), and of Ca in milk lactose (Fig. 2). The sample preparation is easy and fast: The dried powder samples are either measured directly in a powder cup or pressed into pellets; i.e., no time-consuming dissolution and dilution is required!

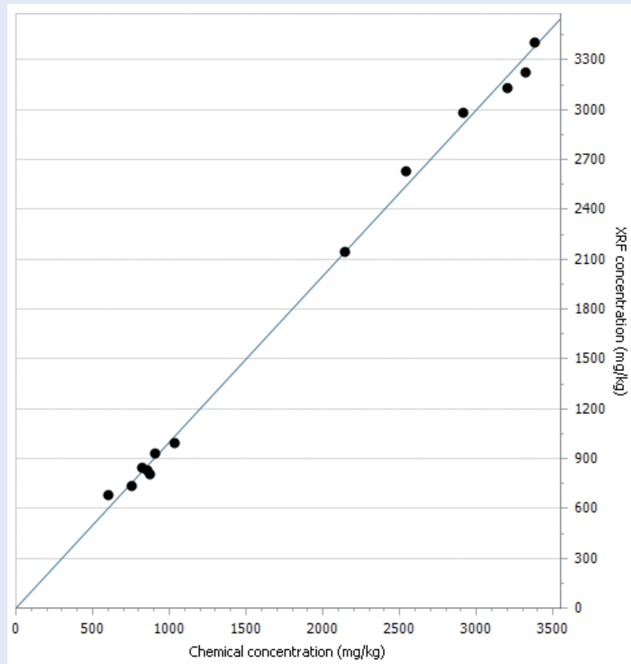

Fig. 1: Calibration curve for Na (KA1) measured in milk protein by using the S2 PUMA (EDXRF); sample preparation: pressed pellets.

Depending on the analytical requirements (e.g., sample throughput per day, precision, detection limits), a EDXRF (S2 PUMA) or a benchtop wavelength dispersive XRF (WDXRF) is most suitable for dairy and infant formula applications. The results listed in Table 1 highlight the excellent performance of Bruker's 400 Watt benchtop WDXRF spectrometer, the S6 JAGUAR, for the analysis of milk powder (relative standard deviation is typically < 1%).

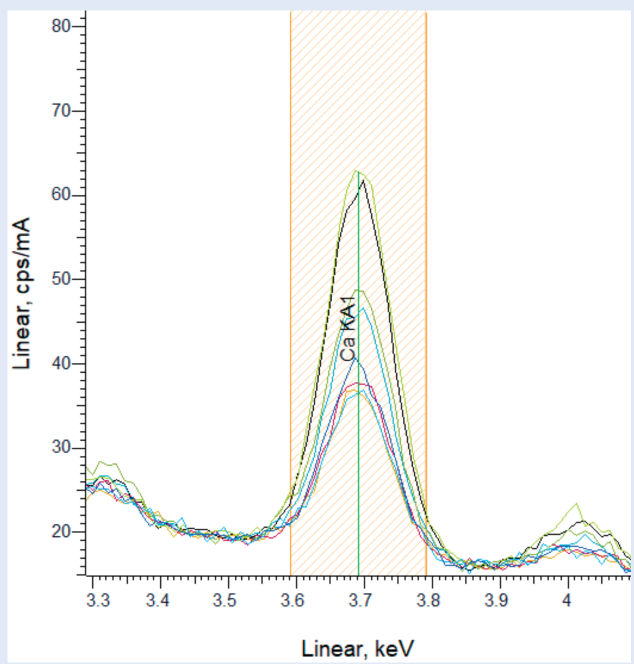

Fig. 2: Ca signal in milk lactose used for the determination of the ash content. The samples were prepared as pressed pellets and measured via EDXRF.

	Na [mg/100g]	Mg [mg/100g]	P [mg/100g]	Cl [mg/100g]	K [mg/100g]	Ca [mg/100g]
1	455.5	83.8	263.9	472.5	582.7	436.0
2	455.1	83.0	266.3	473.1	584.7	434.7
3	458.7	84.4	265.7	473.4	585.0	436.2
4	451.6	82.0	266.6	470.7	586.9	437.9
5	450.7	82.6	266.6	471.8	586.9	437.1
7	460.0	85.3	267.3	473.3	596.7	436.7
6	460.6	84.1	266.0	472.8	588.5	438.4
8	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**

Table 1: Results of a repeatability test performed on pressed milk powder pellets by using the S6 JAGUAR.

An important parameter for milk products is also the ash content. Traditionally, the ash content is determined gravimetrically, by ashing the sample at 550°C. This procedure is time consuming and the sample is lost after the measurement. By accurately measuring the inorganic components (e.g., Na, Mg, P, Cl, Ca, K, Zn), the S2 PUMA allows to determine the ash content in minutes and without destroying the sample (see also Fig. 2).

Application Example 2

Agricultural products

Even for agriculture products which are produced in enormous quantities and require minor processing; like corn, wheat, soy or rice, quality and safety need to be controlled. We calibrated the S2 PUMA by using 23 secondary reference materials to analyze multiple elements in maize kernel, including Mg, P, S, Cl, K, and Zn. The measurements reveal an excellent precision and accuracy (see Table 2), proving the suitability of the S2 PUMA for this and related applications.

	Mg [mg/kg]	P [mg/kg]	S [mg/kg]	Cl [mg/kg]	K [mg/kg]	Zn [mg/kg]
ICP	1140	3254	1152	380	3232	20.9
S2 PUMA Series 2	1207	3279	1149	384	3295	21.0
Abs. Std. Dev.	**13**	**13**	**8**	**4**	**17**	**0.1**
Rel. Std. Dev. (%)	**1.07**	**0.40**	**0.74**	**1.02**	**0.52**	**0.39**
Abs. diff.	**67**	**25**	**3**	**4**	**63**	**0.1**

Table 2: Precision and accuracy of maize kernel analysis. The samples were prepared as pressed pellets and measured by using the S2 PUMA.

Application Example 3

Edible Oils

In edible oils, concentrations of elements such as Si, P, S, and Cl are being monitored for quality assurance. For instance, the total phosphorus content is often measured to optimize the oil refining process because phospholipids can have a negative impact at various process steps. Used cooking oil (UCO) from fast-food chains and restaurants can be recycled to produce biodiesel. In biodiesel, P, as well as S and Cl, must not exceed certain limits and need to be monitored.

These elements in edible oils have one thing common: Their concentration and the thresholds are low, typically < 10 mg/kg. This demands for a dedicated analytical tool. The S2 POLAR with its polarized beam path was developed for trace element analysis in oil matrices, achieving excellent signal to noise ratios in edible oil (see Fig. 3). This results in lower limits of detection of about 1 mg/kg or even better for most elements of interest in edible oil, e.g., 0.4 mg/kg P at a precision of 0.1 mg/kg P.

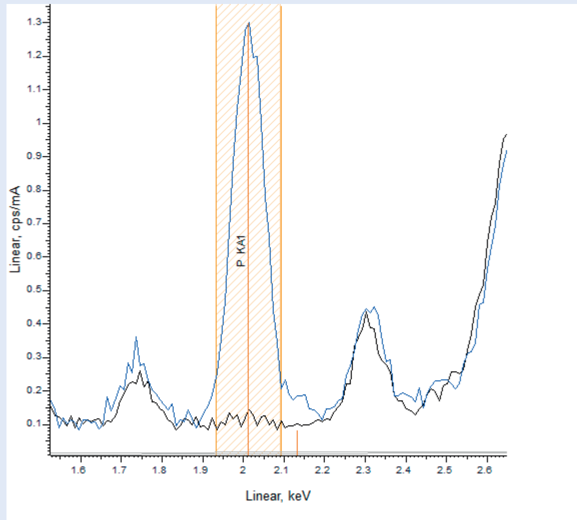

Figure 3: Phosphorous (KA1) signal of virgin coconut oil (23 mg/kg P) (blue) compared to a blank measurement (black). The analyses were performed by using the S2 POLAR, a polarized EDXRF spectrometer.

Conclusion

Modern EDXRF and benchtop WDXRF spectrometers are suitable for many QC and QA tasks in the food industry and food research. XRF has several advantages when compared to traditional methods such as ICP-OES and AAS (Atomic Absorption Spectroscopy), including ease-of-use and low cost of operation. In this application note we show the suitability of Bruker's XRF spectrometers for various food application. Clearly, the application (e.g., liquid or solid) and analytical requirements (e.g., throughput and LLDs) define the type of spectrometer (EDXRF, polarized EDXRF, benchtop WDXRF) and the configuration (e.g., XY-autochanger for highest throughput and flexibility).

Bruker's XRF portfolio for the Food Industry: S2 POLAR, S2 PUMA Series 2 and S6 JAGUAR

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