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Analysis of mineral content in milk powder

Epsilon 3^X

Introduction
This datasheet investigates the capabilities of the Epsilon 3^X, a benchtop energy dispersive X-ray fluorescence spectrometer as a tool for analyzing P, Cl, K, Ca, Mn, Fe, Cu and Zn in milk powder.

Application background
The simple sample preparation along with quick and simultaneous measurement of elements, make the XRF technique an interesting analytical method for the food industry. It allows analysis close to production lines.

Instrumentation
Measurements were performed using PANalytical Epsilon 3^X EDXRF spectrometers, equipped with a 50 kV silver anode X-ray tube, 6 beam filters, a helium purge facility, a high-resolution silicon drift detector, a sample spinner and a 10-position removable sample changer.

Sample preparation
Pressed pellets were prepared using 6.5 g of milk powder compressed at 2-3 tons with a hardened stainless steel die.

Measurement procedure
Secondary milk powder standards containing P, Cl, K, Ca, Mn, Fe, Cu and Zn, were used to set up the calibration. Four different measurement conditions were used, each one optimizing the excitation of a group of elements (Table 1). The analyses were performed in either helium or air atmosphere and the total measurement time was 10 minutes per sample. Example spectra for the measuring conditions <P-Cl>, and <Ni-Mo>, respectively, are given in Figures 1 and 2.

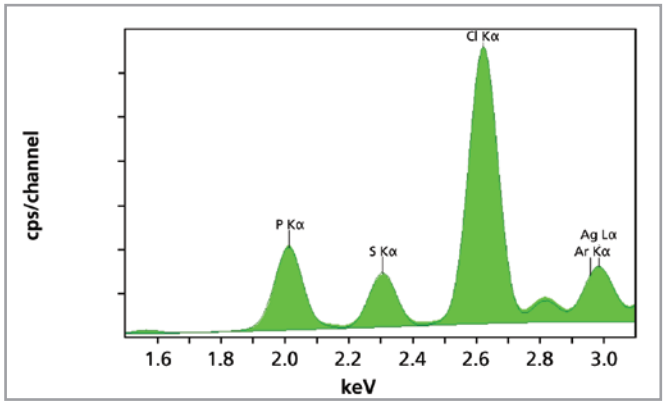

Figure 1. The spectrum obtained using condition <P-Cl>, for P and Cl in milk powder

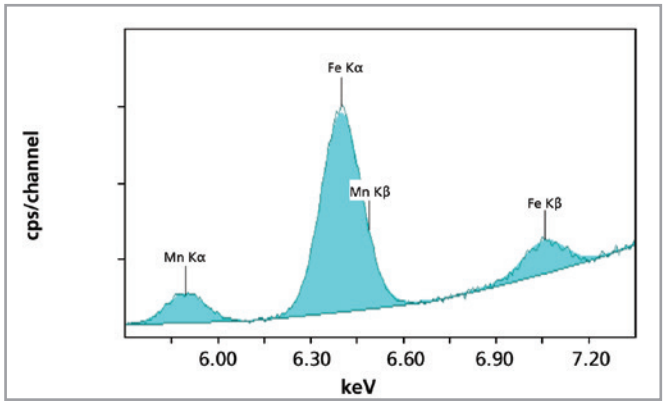

Figure 2. The spectrum obtained using condition <Ni-Mo>, for Mn and Fe in milk powder

Condition	kV	µA	Medium	Filter	Meas. time (s)	Elements
<P-Cl>	9	850	Helium	Ti	300	P, Cl
<K-V>	12	750	Air	Al-thin	60	K, Ca
<Cr-Co>	20	360	Air	Al-thick	180	Mn, Fe
<Ni-Mo>	50	180	Air	Ag	60	Cu, Zn

Table 1. Measurement conditions

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Calibration results

Figures 3 and 4 show the calibration plots for Ca, and Mn in milk powder applying the conditions listed in Table 1. Note the very low Mn concentration (µg/100g) in the calibration graph (Figure 4). The calibration plots demonstrate a good correlation between the chemical concentrations and the measured intensities. Table 2 summarizes the calibration data for the milk powder samples.

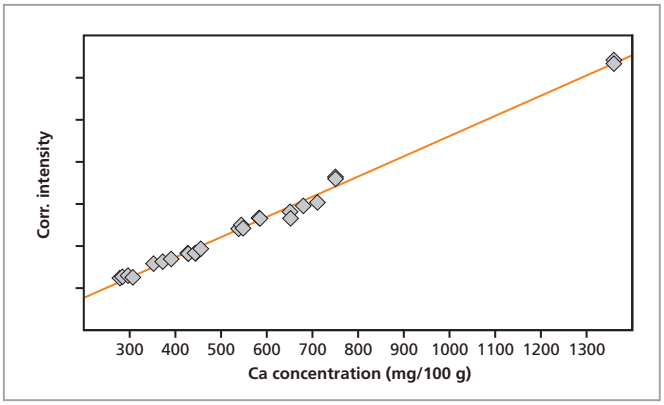

Figure 3. Calibration graph for Ca in pressed pellets

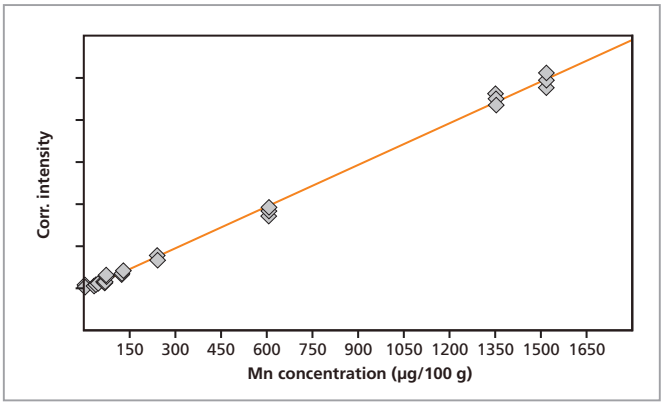

Figure 4. Calibration graph for Mn in pressed pellets

Element	Concentration range (mg/100g)	RMS* (mg/100g)	Correlation	LLD (mg/100g)
P	176 – 1020	23.2	0.9899	0.5
Cl	276 – 984	11.4	0.9971	0.2
K	416 – 1670	15.8	0.9978	0.6
Ca	278 – 1360	21.3	0.9959	0.3
Mn	0.0063 – 1.515	0.03	0.9981	0.05
Fe	4.90 – 9.16	0.26	0.9794	0.04
Cu	0.3 – 0.85	0.05	0.9411	0.06
Zn	3.66 – 7.69	0.15	0.9866	0.06

Table 2.Calibration details (RMS: The more accurate calibrations have the smaller RMS values).*

Precision

To test the repeatability, one pressed sample was measured twice daily for six days. The average concentration, RMS (1 sigma standard deviation) and the relative RMS of the repeat measurements for all the elements are presented in Table 3. The results for most elements demonstrate excellent precision. However, the high RMS values for Mn and Cu are mainly due to low concentrations in the sample.

Element	Average concentration (mg/100g)	RMS (wt%)	Relative RMS (%)
P	234.8	2.1	0.9
Cl	292.3	2.9	0.9
K	442.3	2.2	0.5
Ca	368.3	2.1	0.6
Mn	0.06	0.02	28.7
Fe	5.42	0.10	2.6
Cu	0.40	0.04	9.3
Zn	3.75	0.06	1.7

Table 3. Results of the repeatability test of milk powder sample prepared as pressed pellet

Conclusion

The data presented in this datasheet clearly demonstrate that an Epsilon 3^X EDXRF spectrometer is well suited for the analysis of a wide range of minerals in milk powder with an analysis time of only 10 minutes.

The repeatability results illustrate the stability and robustness of the Epsilon 3^X. The combination of excellent detector resolution, high sensitivity and powerful software deconvolution models contribute to the accuracy and precision of the results.

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