**APPLICATION NOTE**
**# XE-2011-3162**
**Quantitative analysis of Na, Mg, P, K, and Ca in Milk Powder by X-Calibur SDD**


## ABSTRACT
Quantitative analysis of Na, Mg, P, K, and Ca in milk powder were performed by Xenemetrix's X-Calibur with Silicon Drift Detector (SDD).


## OBJECTIVE
1. To create a simple and fast non-destructive quantitative method for the following elements in milk powder: Na, Mg, P, K and Ca.
2. To analyze an unknown sample with the presently developed calibration.

## BACKGROUND
EDXRF is an ideal method for a quick and simple elemental analysis for industrial control purposes mainly for the analysis of elements in solids powders and liquids. It has the following advantages: 1) Fast and minimal sample preparation, 2) an automated analysis process, 3) Limited or no exposure to corrosive reagents used by other analytical techniques, 4) Ease of operation by non-technical or non-specialized personnel. The instrument's high resolution detector, enhanced peak to background ratio and superior flux and ability to handle higher count rates provide superior sensitivity for low molecular weight elements.

## ANALYTICAL CONFIGURATION
**Table 1:** Analytical Configuration of X-Calibur SDD

| | |
|---|---|
| **Instrument** | X-Calibur SDD EDXRF Bench top Spectrometer System. |
| **Excitation** | Rh-Anode X-ray Tube, 50W |
| **Detector** | High Performance Silicon Drift Detector (SDD) |
| **Analysis Time** | 600 seconds |
| **Type of analysis** | Quantitative linear regression analysis |
| **Excitation** | Direct excitation. |
| **Environment** | Helium |
| **Sample Preparation** | Samples were measured without any sample preparation, as obtained from the customer, in XRF sample cups with prolene (4µm) XRF support film. |

## EXPERIMENTALS and RESULTS
Five calibration standards and one unknown sample were provided by the customer. The elemental content of the standards are listed in Table 2.

**Table2:** Concentrations contents in ppm of the STD series

| Standards | Na (ppm) | Mg (ppm) | P (ppm) | K (ppm) | Ca (ppm) |
|---|---|---|---|---|---|
| Bag 1 | 6220 | 1020 | 7700 | 24600 | 11400 |
| Bag 16 | 6070 | 9800 | 7400 | 24700 | 11000 |
| Bag 1479 | 710 | 240 | 1070 | 2040 | 190 |
| Bag 1480 | 610 | 230 | 1090 | 2080 | 180 |
| Bag 161 | 170 | 640 | 10900 | 3380 | 16800 |

Quantitative method for analyzing Na, Mg, P, K and Ca in milk powder was performed using X-Calibur EDXRF analyzer equipped with High Performance Silicon Drift Detector. The analysis was performed using helium purge at low energy to enhance the low energy Na and Mg signals. P, K and Ca were also analyzed at this instance.

The standards and sample were analyzed as obtained from the customer in XRF sample cups with prolene (4µm) XRF support film. In order to enhance technique sensitivity, the powder was compacted into the cups with a clean plastic rod. Each standard was acquired at a time, immediately after opening the pack; it was assured that the samples did not adsorb any moisture.

A calibration curve was built for each element, by the nEXT software, based on the extracted peak intensities from the standards spectra. A representative spectrum is shown in figure 1, the calibration data is summarized in Table 3 and correlation plots per each element components are shown in Figures 1-5. Repeatability studies were also performed to evaluate the instrument static precision (table 4).

**Table 3:** Summary of the calibration data of Na, Mg, P, K and Ca. The spectra was acquired at low energy in helium purge.

| Element | Std.Dev. (ppm) | Correlation |
|---|---|---|
| Na | 217.9 | 0.9971 |
| Ca | 192.7 | 0.9995 |
| K | 570.7 | 0.9986 |
| P | 27.2 | 0.9999 |
| Mg | 19.1 | 0.9988 |

| Element | Standard ID | Given Conc. (ppm) | Calc. Conc. (ppm) | Abs.Dev. | Rel.Dev. |
|---|---|---|---|---|---|
| Na | Bag 1 | 6220 | 5906 | 314 | -0.050 |
| | Bag 16 | 6070 | 6362 | 292 | -0.048 |
| | Bag 1479 | 710 | Omitted | N/A | N/A |
| | Bag 1480 | 610 | 568 | 42 | -0.069 |
| | Bag 161 | 170 | 232 | 62 | -0.365 |
| Ca | Bag 1 | 11400 | 11130 | 270 | -0.024 |
| | Bag 16 | 11000 | 11258 | 258 | -0.023 |
| | Bag 1479 | 190 | 345 | 155 | -0.816 |
| | Bag 1480 | 180 | 32 | 148 | -0.822 |
| | Bag 161 | 16800 | 16802 | 2 | 0.000 |
| P | Bag 1 | 7700 | 7676 | 24 | -0.003 |
| | Bag 16 | 7400 | 7414 | 14 | -0.002 |
| | Bag 1479 | 1070 | 1039 | 31 | -0.029 |
| | Bag 1480 | 1090 | 1119 | 29 | -0.027 |
| | Bag 161 | 10900 | 10899 | 1 | 0.000 |
| K | Bag 1 | 24600 | 24419 | 181 | -0.007 |
| | Bag 16 | 24700 | 24860 | 160 | -0.006 |
| | Bag 1479 | 2040 | 2917 | 877 | -0.430 |
| | Bag 1480 | 2080 | 2116 | 36 | -0.017 |
| | Bag 161 | 3380 | 2486 | 894 | -0.264 |
| Mg | Bag 1 | 1020 | 1010 | 10 | -0.010 |

| | | | | | |
|---|---|---|---|---|---|
| | Bag 16 | 9800 | omitted | N/A | N/A |
| | Bag 1479 | 240 | 232 | 8 | -0.033 |
| | Bag 1480 | 230 | 266 | 36 | -0.157 |
| | Bag 161 | 640 | 642 | 2 | -0.003 |

**Figure 1:** Regression plot for Na

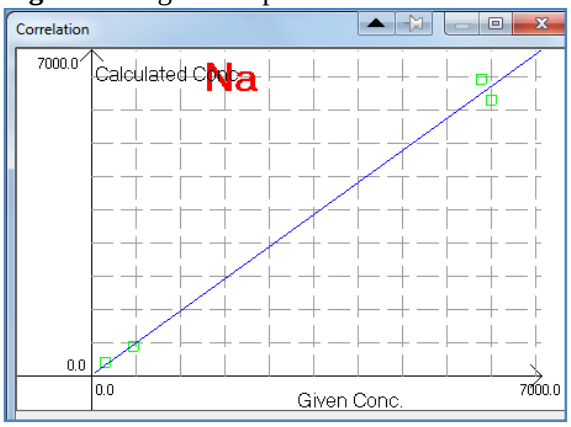



**Figure 2:** Regression plot for Mg

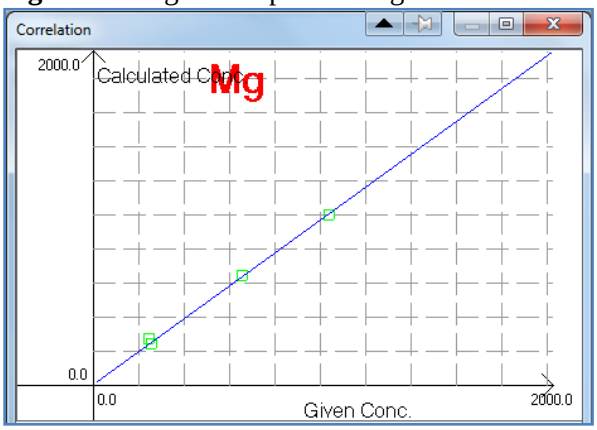



**Figure 3:** Regression plot of P

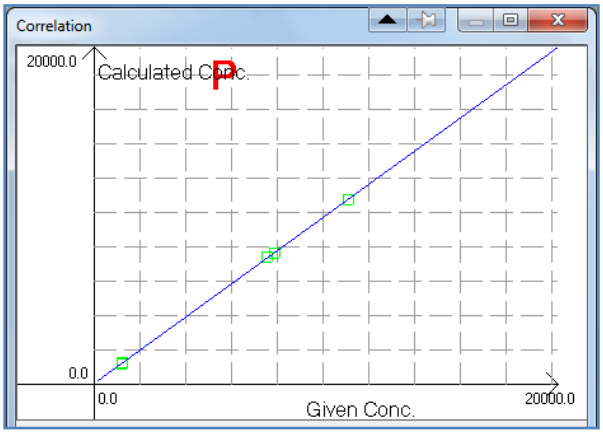



**Figure 4:** Regression plot of K

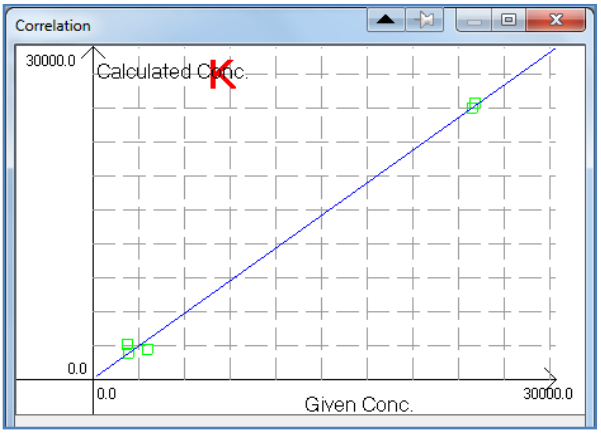
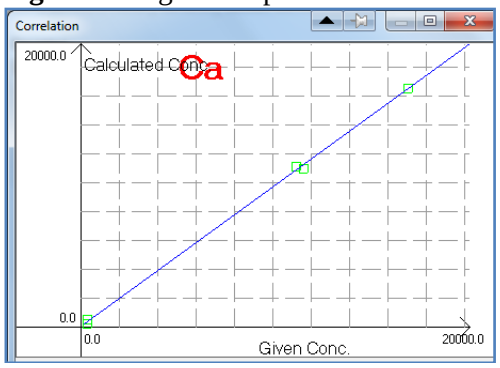


**Figure 5:** Regression plot of Ca




## ANALYSIS OF THER UNKNOWN SAMPLE

The results for the analysis of the unknown sample, "Bag 8" are listed in table 4.

**Table 4**: **summary of standard composition and calibration results**

| STD ID | Na (ppm) | Mg (ppm) | P (ppm) | K (ppm) | Ca (ppm) |
|---|---|---|---|---|---|
| **Bag- 8** | 6910 | 1073 | 7362 | 23594 (2.35%) | 10795 (1.07%) |

## PRECISION STUDY

Static precision was performed by performing ten consecutive acquisitions on one of the samples without moving it between the acquisitions. The results from the low energy acquisition are shown in Table 4.

**Table 3: Precision** (relative standard deviation at one sigma) for the different elements in Bag 16 standard

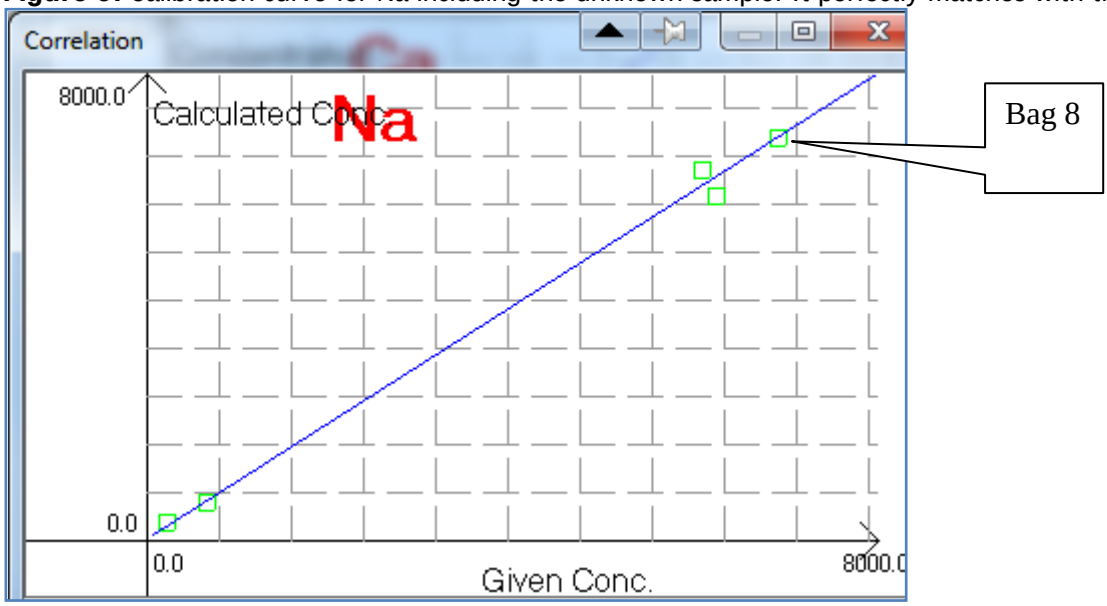
| Element | Average | Standard deviation |
|---|---|---|
| Na | 2743 | 413.5 |
| Mg | 809 | 33.5 |
| P | 7818 | 25.0 |
| K | 25341 | 46.7 |
| Ca | 1148 | 74.7 |

## RESULTS and DISCUSSIONS

The main goal of this work was to develop a quantitative method for five elements (Na, Mg, P, K, and Ca) in milk powder by EDXRF technology with the 4 standards provided. All the elements in the standards were analyzed by single acquisition at low energy with Helium purge. This makes the analysis of the unknown ever simpler, since minimal spectra acquisitions are necessary for the analysis of all the elements present.
The calibrations results show very good correlations with software treatments of the inter-elements effects.
Precision studies for all elements except for Na was satisfactory. In the latter case, there were inconsistencies in the Na peaks. It could be seen as the Na was unevenly distributed throughout the sample. More samples with improved grinding and mixing (Vortex or powder mixer) may be a good solution.
The analysis of the unknown sample, Bag 8, was spectrally compared to the other known standards spectra, the peak intensities of the standard match those of the other spectra.

This can be shown by adding the unknown sample to the calibration curve with the appropriate elemental values found by the calibration method. Curve 6 shows a good match between the additional Na figure arriving from the unknown analysis to the overall curve.

**Figure 6:** Calibration curve for Na including the unknown sample. It perfectly matches with the curve.




## Conclusions

This work shows the performance of X-Calibur with SDD detector for the analysis of Na, Mg, P, K, and Ca. Despite differences in particle size of the powders, good correlations was achieved for all elements.

Upgrading this method with additional representative standards could significantly improve the robustness and accuracy of the method.

In summary, this study shows that X-Calibur SDD Bench top Spectrometer system is capable of providing a good quantitative analysis for light elements in milk powder.