

S4 EXPLORER

DETERMINATION OF MAJOR AND MINOR ELEMENTS IN MINERAL SALT

Introduction

For many years X-ray fluorescence spectrometry (XRF) has been used as a fast, reliable process and quality control tool in the refining of mineral salt products.

This report aims to demonstrate the superb light element performance of the Bruker AXS S4 EXPLORER and the power of SPECTRA^{plus} Variable Alphas based matrix corrections.

Major and minor elements were determined in mineral salts applying X-ray fluorescence analysis with the S4 EXPLORER. Calibrations for the individual elements were established from a selection of 21 mineral salt standards. Calibration curves and calibration data are presented for Na, Mg, P, S, K and Ca.

Instrumental

The Bruker AXS S4 EXPLORER (Figure 1.) is a revolutionary step forward in the development of Sequential Wavelength Dispersive XRF spectrometers. It comprises all of the usual components - a 1kW end window Rhodium X-ray tube, up to ten primary beam filters, up to four collimators and up to eight analysing crystals. For highly sensitive analysis of light elements analysis, such as Sodium and Magnesium, the sealed proportional counter Pro4 with the innovative, unique Super High Transmission window was used.



Figure 1: S4 EXPLORER

Software

Perhaps the most outstanding feature of the S4 EXPLORER analytical system is the inclusion of the SPECTRA^{plus} software package. This is a fully interactive program that uses the Windows NT platform for maximum speed and stability.

It comes complete with a "standardless" XRF routine, the Fundamental Parameter based Variable Alpha matrix correction routine, a rapid data retrieval system and full networking capability.

Simple and rapid specimen preparation

The samples were ground 60 seconds in a swing mill with a tungsten carbide vessel. After grinding the pellets were pressed directly from the dried sample material (105°C, 12 h). Pelletizing of 40 mm discs occurred at a pressure of 200 kN (20t) during a period of 7 seconds.

Calibration

A measurement method was developed by carefully selecting the peak and background positions for six elements. The total measuring time was less than 3 minutes. The $K\alpha_{1/2}$ lines of six analytes were calibrated from a set of 21 mineral salt standards using the comprehensive calibration tools of SPECTRA^{plus}. The high accuracy, shown in Table 1, was achieved applying the Variable Alpha matrix correction model.

Analyte	Range of Conc. [%]	Accuracy [%]
Na	0.46 - 4.1	0.05
Mg	1.5 - 3.6	0.04
P	3.7 - 6.6	0.05
S	1.8 - 10	0.07
K	10 - 23	0.2
Ca	10 - 17	0.2

Table 1: Details of calibration data

The Variable Alpha matrix correction model requires the complete information on the sample composition. In some applications this includes elements which cannot be determined by XRF analysis, such as H in (OH) bearing minerals, or elements which are not certified with the calibration reference standards. In the last case the "standardless" XRF routine of SPECTRA^{plus} is the right tool to get an acceptable estimation of the trace elements and further matrix elements (for example Chlorine and Oxygen) to be used for the Variable Alpha matrix correction. Table 2 summarizes these analytes.

Calibration curves

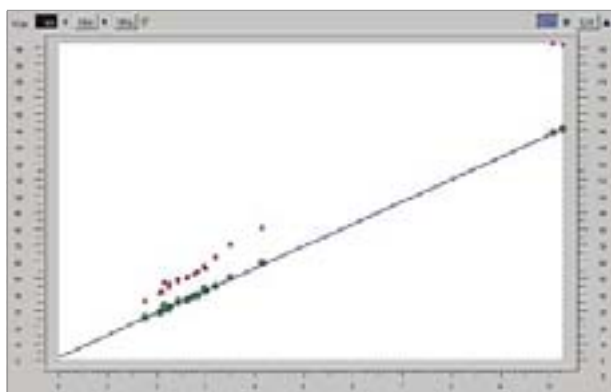


Figure 2: Calibration curve for Sulphur

The preceding figures present the regression lines for some of the analytes. The measured net intensities are shown by the red dots. The corrected intensities (Variable Alpha matrix correction method) are given by the green dots.

Conclusion

This application note has summarised the outstanding performance of the BRUKER AXS S4 EXPLORER, in combination with the effective evaluation tools of

Analyte	Range of Conc. [%]	Analyte	Range of Conc. [%]
O	22 - 43	V	LLD - 0.1
F	0.2 - 1.2	Fe	0.16 - 2.9
Al	0.08 - 0.9	Mn	0.01 - 0.4
Si	0.6 - 1.8	Sr	0.007 - 0.03
Cl	0.78 - 28	Zn	0.1 - 0.5
Ti	LLD - 0.09	Br	LLD - 0.06

Table 2: Analytes determined by "standardless" XRF routine

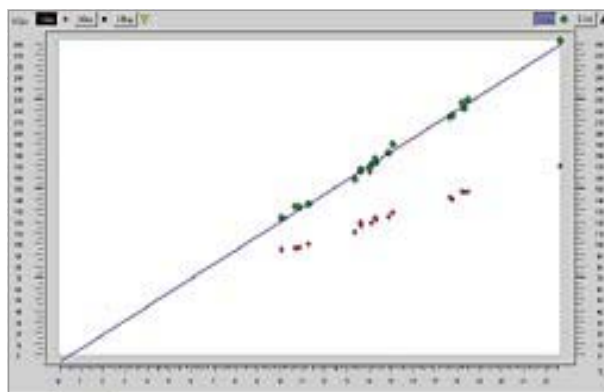


Figure 3: Calibration curve for Potassium

SPECTRA^{plus}, for the analysis of major and minor elements in mineral analysis.

It has also demonstrated the general good quality of light element analysis using the S4 EXPLORER with the highly sensitive Pro4 sealed proportional counter and excitation by the intensity optimized AG22 end-window X-ray tube with its 75 μm thin tube window.

BRUKER AXS GMBH

D-76187 KARLSRUHE
GERMANY

TEL. (+49) (721) 595-2888
FAX (+49) (721) 595-4587
<http://www.bruker-axs.de>
Email: info@bruker-axs.de

BRUKER AXS, INC.

5465 EAST CHERYL PARKWAY
MADISON, WI 53711-5373
USA

TEL. (+1) (800) 234-XRAY
TEL. (+1) (608) 276-3000
FAX (+1) (608) 276-3006
<http://www.bruker-axs.com>
Email: info@bruker-axs.com

BRUKER AXS LTD

MEADOWSIDE
MOUNTBATTEN WAY, CONGLETON
CHESHIRE CW12 1DN
GREAT BRITAIN

TEL. (+44) (1260) 296-900
FAX (+44) (1260) 296-909
Email: info@bruker-axs.co.uk