S4 PIONEER
THE ANALYSIS OF BORON IN COLEMANITES

Introduction

Due to its ability to reduce the melting temperature for mineral mixtures, Boron Oxide is becoming an important compound in the glass and ceramic industry. Additionally, Boron Oxide increases the thermal stability, acid resistance and hardness of ceramic products. X-ray fluorescence analysis (XRF) is an important tool for any modern analytical process and quality control of Boron in the ceramic, glass and even in the semiconductor industry. The excellent performance of the S4 PIONEER for the analysis of Boron is demonstrated in this report with the mineral colemanite ($Ca[B_3O_4(OH)_3]\cdot H_2O$) given as an example.

Instrument

The innovative concept of the S4 PIONEER is a new milestone in the development of wavelength dispersive XRF (WD-XRF) instruments. It combines the flexibility of a modern spectrometer with up to 10 primary beam filters, up to 4 collimators, up to 8 analyzer crystals and the advantage of 4kW excitation with an ultrathin Be-endwindow tube, but, despite all of this, requires only a very small amount of floor-space (~0,8m^2). Consequently, this intensity-optimized compact design with the excitation power of 27kV and 150mA (real 4kW) leads to very high sensitivities, even for ultra-light elements like Boron. Therefore, superb Lower Limits of Detection (LLD) can be reached within a very short measuring time. For the determination of Boron in colemanites the analyzer crystal OVO-B was combined with the very coarse 2°collimator.

Figure 1. The S4 PIONEER

Method

This measurement required the careful adjustment of the Boron Kα1/2-Line and one background position. The calibration was performed by measuring 7 colemanites, prepared as pressed pellets, with Boron concentrations covering a range from 2,45 to 13,97% using the unique comprehensive calibration tools of SPECTRA^{plus}. A total measuring time of 130 seconds was chosen.

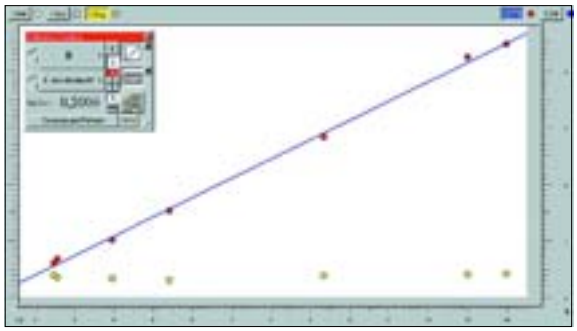

Figure 2. Calibration curve for Boron Kα1/2

Results

Figure 2 presents the calibration curve. The background intensities are displayed with yellow dots, the corrected intensities with red dots. The details of the Boron calibration are shown in Table 1. The LLD is 180ppm (3sec., 100sec.).

Std.	Chem. Conc. (%)	XRF Conc. (%)	A.S.D. (%)	R.S.D (%)
Cm 1	2.45	2.56	-0.11	4.2
Cm 2	2.53	2.78	0.25	9.7
Cm 3	3.94	3.75	-0.19	4.9
Cm 4	5.40	5.27	-0.13	2.4
Cm 5	9.32	9.11	-0.21	2.2
Cm 6	12.99	13.25	0.26	1.9
Cm 7	13.97	13.90	-0.07	0.5

Table 1. Details of Boron calibration

To demonstrate the light element reproducibility of the S4 PIONEER two samples, one with a high and one with a low Boron concentration, were measured ten times. The samples were alternately loaded to ensure that all instrument parameters were changed. The reproducibility is shown in Table 2.

Std.	Colemanite 1 Conc. (%)	Colemanite 7 Conc. (%)
repetition 1	2.55	13.98
repetition 2	2.57	14.03
repetition 3	2.57	13.98
repetition 4	2.56	13.97
repetition 5	2.57	13.97
repetition 6	2.55	13.99
repetition 7	2.57	13.95
repetition 8	2.58	13.95
repitition 9	2.59	13.98
repitition 10	2.55	13.98
Average	2.57	13.98
Std. Dev. abs.	0.01	0.02
Std. Dev. rel.	0.53	0.16

Table 2. Short term reproducibility of Boron analysis of two colemanite samples

Conclusions

This lab report demonstrates the outstanding performance of the S4 PIONEER, especially for the determination of light elements like Boron.

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