

S4 EXPLORER

DETERMINATION OF BORON IN BOROPHOSPHOSILICATE-GLASSES

Introduction

Since the early 80s Boron analysis by X-ray fluorescence spectrometry (XRF) is appreciated as a fast, reliable process and quality control tool in the Semiconductor, Glass and Ceramic Industries. This report aims to demonstrate the superb light element performance of the Bruker AXS S4 EXPLORER.



Figure 1. The plug 'n analyse S4 EXPLORER

Instrumental

The S4 EXPLORER (Figure 1.) is an innovative step forward in the development of sequential wavelength dispersive XRF spectrometers with a 1kW end window Rhodium X-ray tube. It comprises all of the usual components, 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 Boron, the sealed proportional counter Pro4 with the innovative, unique Super High Transmission window, the OVO-B analyzer crystal and the very coarse 2.0° collimator was used.

Calibration

A measurement method was developed by carefully selecting the peak and two background positions for Boron. The total measuring time was 5 minutes. The B K α 1/2 line was calibrated from a set of 10 BoroPhosphoSilicate-Glass reference samples, covering a Boron concentration range from 0 – 2.6 %, using the comprehensive calibration tools of SPECTRA^{plus}. The high accuracy, shown in Table 1, was achieved applying the uncorrected gross intensities. The LLD lower limit of detection is in the range of 550 ppm (3 σ , 100 sec.).

Std.	Chem. Conc. (%)	XRF Conc. (%)	A.S.D. (%)	R.S.D (%)
1	1.65	1.61	-0.04	-2.54
2	2.50	2.50	0.003	0.13
3	2.60	2.50	-0.10	-3.79
4	1.00	1.03	0.03	2.95
5	2.60	2.51	-0.09	-3.47
6	1.00	1.09	0.09	9.08
7	1.65	1.64	-0.01	-0.84
8	2.50	2.52	0.02	0.70
9	1.05	1.02	-0.03	-2.51
10	0.00	0.00	0.00	

Figure 2. Details of Boron calibration data

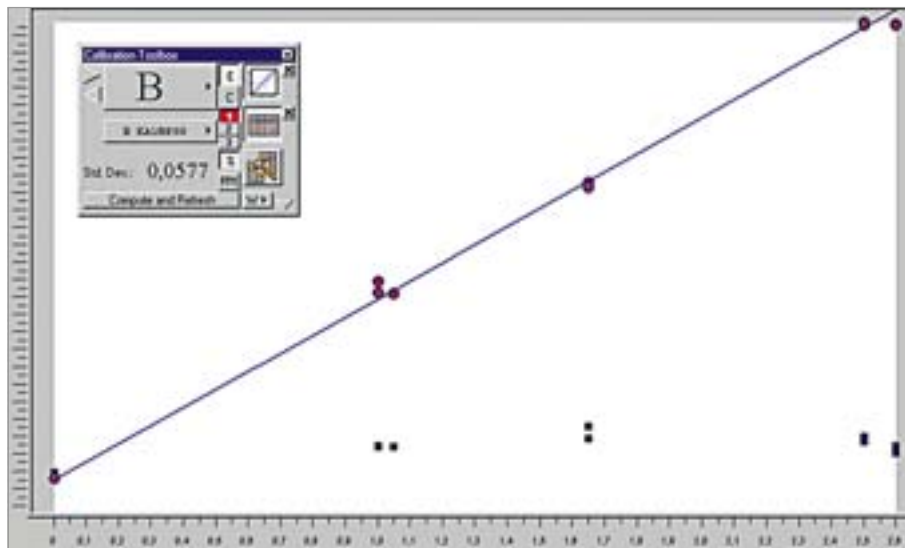


Figure 2. Boron calibration curve

Figure 2 presents the regression line for the Boron calibration. The measured background intensities are shown by the blue squares. The uncorrected gross intensities are given by the pink dots. Table 2 shows the test results of 6 short term reproducibility measurements.

Conclusion

This application note has summarised the outstanding performance of the S4 EXPLORER for the analysis of Boron based on 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.

	Concentration (%)
repetition 1	2.698
repetition 2	2.710
repetition 3	2.689
repetition 4	2.720
repetition 5	2.708
repetition 6	2.688
Average	2.69
Std. Dev. abs.	0.007
Std. Dev. rel.	0.27

Table 2. Short term reproducibility of Boron measurements

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