Application Note: 41631

B_2O_3 Analysis in Glass

ARL ADVANT'X Series with IntelliPower™ Sequential X-Ray Fluorescence Spectrometer

Key Words

- ARL ADVANT'X
- IntelliPower™ - 4200 W
- XRF
- X-Ray Fluorescence
- Glass

Limit of detection

Introduction

The simplest form of glass is the single component fused silica (SiO_2). However it is both difficult to process and expensive. To reduce these difficulties, some other oxides are added giving specific properties. Most glasses used in construction are composed of 70 % of silica, which is a glass former, soda as a flux in the form of carbonate and sulfate (about 14 %), lime as a stabiliser in the form of limestone (about 10 %). Other types of oxides like alumina or magnesia improve the physical characteristics of glass, particularly the resistance to atmospheric conditions.

The replacement of alkali oxides by boric oxide (B_2O_3) in the silica glassy network produces a lower expansion, good thermal shock resistance and chemical stability glass. In addition it lowers considerably the softening point of the silica glass considerably. The B_2O_3 concentration may vary from about few percent in silica, alumino-silicate or lead silicate glasses to up to 30 % for low electrical loss glasses.

The wet chemical analysis of B_2O_3 in glass takes typically a couple of hours and involves hazardous chemicals. A powerful X-ray fluorescence spectrometer allows reduction in this measuring time down to a few minutes.

Instrumentation

An ARL Advant'X IntelliPower 4200 spectrometer has been used to derive the following results. The geometry of the instrument is optimized to provide the highest sensitivity. It is equipped with an end-window X-ray tube type 5GN fitted with a 50 µm Be window and a Rh anode. The AXBeB multilayer crystal, designed specifically for boron analysis, is used together with the extra-coarse collimator which exploits the full potential of this synthetic crystal. Generator settings are 30 kV/140 mA, which is beneficial for light element analysis.

Results

Quantitative analysis has been performed on a set of 4 samples using optimum analytical conditions. Figure 1 shows a scan over the boron Kα peak in one of the glass samples.

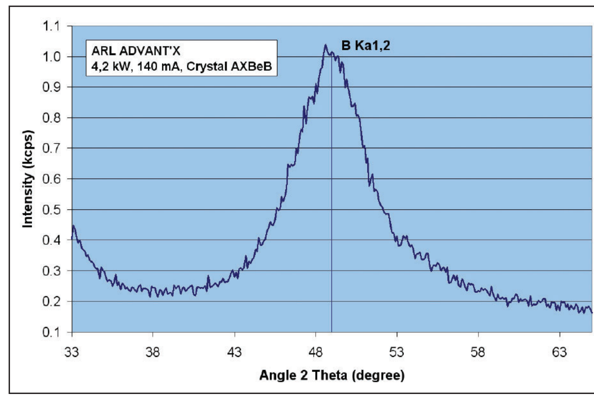

Figure 1: Scan of the boron Kα peak in a glass

The resulting calibration curve is illustrated in Figure 2. From the sensitivity (slope) and background level shown on this calibration curve, the limit of detection for boron in glass can be derived:

260 ppm in 100 s expressed as B_2

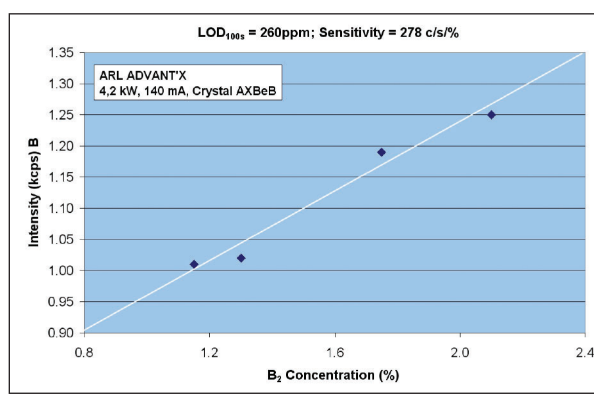

Figure 2: Calibration curve for boron in glass
Boron concentration expressed as B_2.
Analysis time : 100 s

Repeatability tests

A precision test has been performed in order to determine the repeatability of the boron analysis in glass. A pressed glass sample has been repeatedly analyzed 10 times with two different counting times: 120 s and 360 s.
The results are shown in Tables 1 and 2.

	B203	B203 (as B2)
Run 1	5.92	1.83
Run 2	6.01	1.86
Run 3	5.99	1.85
Run 4	6.07	1.87
Run 5	6.02	1.86
Run 6	6.10	1.88
Run 7	6.12	1.89
Run 8	6.06	1.87
Run 9	6.08	1.87
Run10	6.09	1.88
Average	6.05	1.87
Std dev	0.06	0.02

Table 1: Repeatability for boron analysis in glass (120 s - 30 kV/140 mA)

	B203	B203 (as B2)
Run 1	6.08	1.88
Run 2	6.08	1.88
Run 3	6.13	1.89
Run 4	6.08	1.88
Run 5	6.04	1.86
Run 6	6.02	1.86
Run 7	6.18	1.91
Run 8	6.11	1.89
Run 9	6.10	1.88
Run10	6.13	1.89
Average	6.09	1.88
Std dev	0.04	0.01

Table 2: Repeatability for boron analysis in glass (360 s - 30 kV/140 mA)

Conclusion

Limits of detection for ultra-light elements are dropping consistently with new spectrometer developments, allowing better analysis of difficult elements. This permits replacement of time consuming wet chemical analyses by faster determination using X-ray fluorescence.

It should be noted that in a glass sample, boron X-ray fluorescence radiation escapes from a depth of the order of 0.15 µm. This means that only the surface of the sample is analyzed for boron. Cleanliness of sample preparation, consistency in surface preparation methods and minimized risk of surface contamination are pre-requisites for this analysis. If this level of care is not taken, the results can be substantially in error as contamination will prevent the soft X-rays from escaping from the sample surface and hence they would not be measured by the spectrometer system.

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Australia
+61 2 8844 9500
Austria
+43 1 333 50340
Belgium
+32 2 482 30 30
Canada
+1 800 532 4752
China
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Denmark
+45 70 23 62 60
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Sweden/Norway/ Finland
+46 8 556 468 00
Switzerland
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UK
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USA
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