The Determination of Boron in Glass Using Radium-Beryllium Neutron Source (Neutron Absorption Technique)

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When a substance containing an element with a high absorption cross section for neutron is placed in a flux of neutrons, a reduction of the flux is caused. When the neutron absorption of other elements in the sample is negligibly small, the reduction degree of the neutron flux can be used as a measure of the quantity of the neutron-absorbing elements.

Such a neutron absorption method has been utilized as a means of determining the boron content of various substances[1-4], because of the high cross section of boron for thermal neutrons ($\sigma_{abs.}=755$ barn), of which the nuclear reaction is $^{10}B(n.\alpha)^{7}Li$. The author has applied this neutron absorption technique to the determination of boron in borosilicate glass with some interesting results.

Experimental and Discussion

The activation source consisted of a mixture of 84.2 mg. of radium bromide (49.3 mg. as radium element) with beryllium, which was sealed in a platinum tube (length: 40.0 mm., diameter: 5.0 mm., wall-thickness: 0.5 mm.). The source was surrounded by paraffin to moderate the fast neutrons to thermal velocities and to reflect these neutrons.

Studies on the Arrangements of Paraffin.—In order to obtain as much thermal neutron flux as possible, the following experiments were tried. In these experiments, the neutron detector was a silver plate (silver content, 75%; diameter, 23 mm.; weight, 4.6 g.). The time of neutron irradiation was 20 minutes and, after an interval of 30 seconds, the induced radioactivity (^{110}Ag [24.2 sec.], ^{108}Ag[2.3 min.]) was measured for 5 minutes by means of an end-window G. M. counter (thickness of mica window: 1.96 mg./cm^2) at a fixed position.

When a cadmium filter (thickness: 0.6 mm.) which absorbs all the neutrons with energies less than 0.4 eV. is used, the radioactivity produced by thermal neutrons only is given by the following equation.

$$I_{thermal}=I_0-I_{Cd}$$

$I_{thermal}$: the radioactivity obtained by thermal neutrons.

I_0: the radioactivity obtained without cadmium filter.

I_{Cd}: the radioactivity obtained with cadmium filter*.

In the first experiment the relationship between the thermal neutron density at the center of an upper surface of paraffin and its distance from the neutron source was studied. The maximum neutron flux was obtained at 2.5 cm. from the source as is shown Fig. 1.

In the second experiment, a cylindrical hole (diameter: 4.2 cm.) was bored through the paraffin block above the neutron source, as is shown by the dotted line in Fig. 2, and the relationship between the thermal neutron density at the outlet of the hole and the hole length was investigated.

The results obtained are shown in Fig. 3.

1) P. Süe and J. Martelly, *Bull. Soc. Chim. France*, **1946**, 103, 410.
2) J. Govaerts, *Exper.* **6**, 459 (1950).
3) M. Green and G. R. Martin, *Trans. Faraday Soc.* **48**, 416 (1950).
4) R. P. Hamlen and W. S. Koski, *Anal. Chem.* **28**, 1631 (1956).

* As shown on Fig. 2, a cadmium plate is put on the silver detector at every I_0 and I_{Cd} measurement to shut out thermal neutrons from the upper side of the detector. In the case of I_{Cd} measurement, two Cadmium plates must be put separately on and beneath the silver detector to shut out thermal neutrons from each side of the detector.

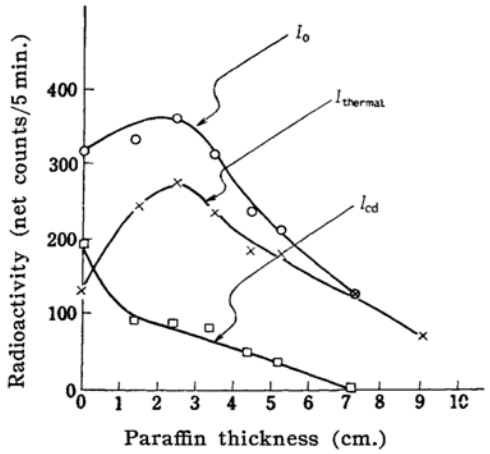

Fig. 1. Relationship between paraffin thickness and radioactivity induced in silver plate.

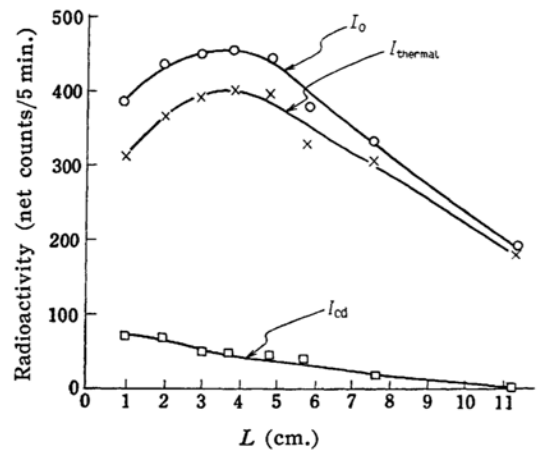

Fig. 3. Relationship between length of hole in paraffin ("L" in Fig. 2) and radioactivity induced in silver plate.

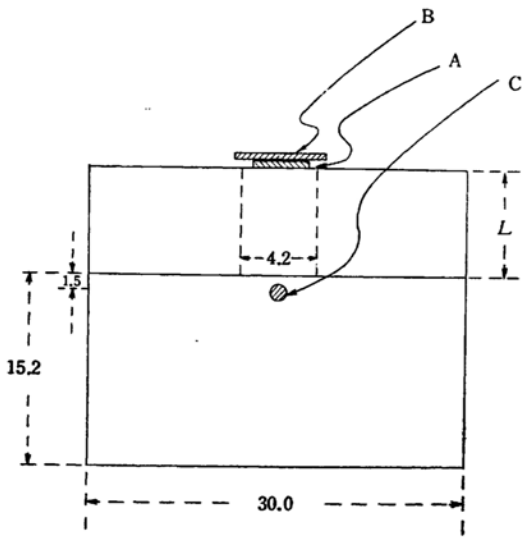

Fig. 2. Irradiation geometry (Dimensions in cm.)
A=silver plate. B=cadmium plate.
C=radium+beryllium neutron source.

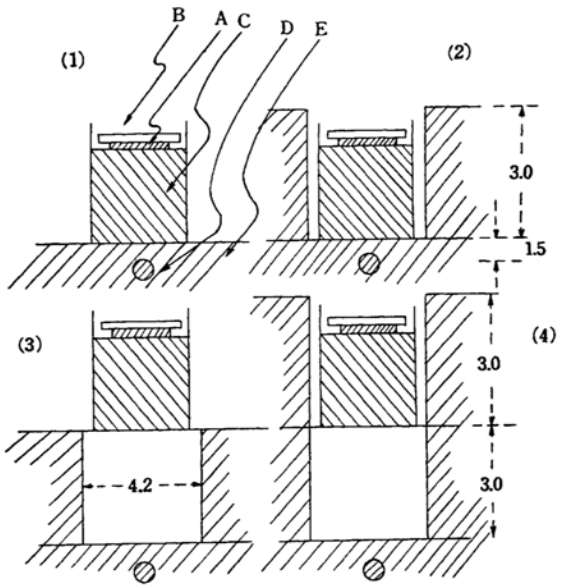

Fig. 4. Experiments for evaluating irradiation geometry. (Dimensions in cm.)
A=silver plate. B=cadmium plate.
C=sample powder. D=radium+beryllium neutron source. E=paraffin.

From these experimental results it is observed that a remarkable increase in neutron flux is caused by the use of a paraffin block with a hole in its centre. For example, the thermal neutron density at 5 cm. from the neutron source is about 2.2 times as great as that obtained by the use of a paraffin block without a hole. This phenomenon must be due to the "howitzer effect"[5,6], by which the collimation of neutrons is caused.

This effect was also observed in the following experiment. Ten grams of silicic oxide power (sieved through a 100 mesh screen) was placed in an aluminum cup (diameter: 3.0 cm., thickness: 0.05 cm.) and was irradiated with neutrons as shown in Fig. 4. The radioactivities of the silver detector in each case are shown in Table I. Then, in order to obtain stronger thermal

TABLE I
TESTS FOR IRRADIATION GEOMETRY

Geometry No. (in Fig. 4)	$I_{thermal}$ (net counts/5 min.)
1	82
2	477
3	90
4	439

5) F. L. Hopwood and T. A. Chalmers, *Nature*, **135**, 341 (1935).
6) G. A. Fink, *Phys. Rev.*, **50**, 738 (1936).

neutron flux, such geometrical arrangement as 2 or 4 in Fig. 4 would be more advantageous for neutron absorption analysis.

Studies on the Determination of Boron Content in Borosilicate Glass.—Relative cross section values of the various components in three practical examples of borosilicate glass were calculated[7] and summarized in Table II. As is shown in the table, the greater part of the cross section values (about 99% in σ_{abs} and about 95% in σ_t) in borosilicate glass is attributed to boron in the sample. Therefore it would be possible to apply the neutron absorption technique effectively to boron analysis of such samples.

TABLE II
RELATIVE CROSS SECTION VALUES OF VARIOUS COMPONENTS IN GLASS

Example 1
(Mean value of borosilicate glass[8])

Component	Composition (%)	σ_{abs} (%)	σ_t (%)
SiO_2	80	0.06	4.23
Na_2O	4	0.02	0.26
B_2O_3	14	99.92	95.41
Al_2O_3	2	—	0.10

Example 2

Component	Composition (%)	σ_{abs} (%)	σ_t (%)
SiO_2	68.60	0.03	2.37
Na_2O	3.84	0.01	0.17
K_2O	5.41	0.05	0.13
Al_2O_3	0.34	—	0.01
B_2O_3	21.81	99.91	97.32

Example 3
(BK-7 glass)

Component	Composition (%)	σ_{abs} (%)	σ_t (%)
SiO_2	69.58	0.07	5.09
Na_2O	8.44	0.06	0.77
K_2O	8.32	0.16	0.42
MgO	0.08	—	0.01
CaO	0.10	—	0.01
BaO	2.55	0.01	0.10
B_2O_3	9.91	99.69	93.54
Al_2O_3	0.44	—	0.03
As_2O_3	0.31	0.01	0.02

The geometrical arrangement of the paraffin, the analyzed sample and the neutron source in the experiments is shown in Fig. 5. In these cases, a round

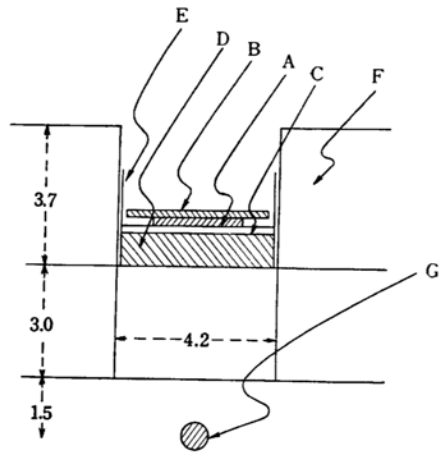

Fig. 5. Irradiation geometry for analysis (Dimensions in cm.)
A=indium plate. B=cadmium plate. C=plastic plate (thickness: 0.1 cm.). D=sample powder. E=plastic sample container. F=paraffin. G=radium+beryllium neutron source.

metallic plate of indium (diameter, 22 mm.; weight, 2.1 g.) was used as neutron detector. The irradiation samples were the mixed powder (100 mesh) of glass and aluminum oxide as the diluent (weight ratio=1:1.5). Ten grams of it were irradiated for 2.0 hours, in which case the saturation factor of ^{116m}In is 0.78. After an interval of one minute, the induced radioactivity of the indium detector was measured for 20 minutes. The method of determining the thermal neutron density is similar to that illustrated above.

By using a calibration curve, as shown

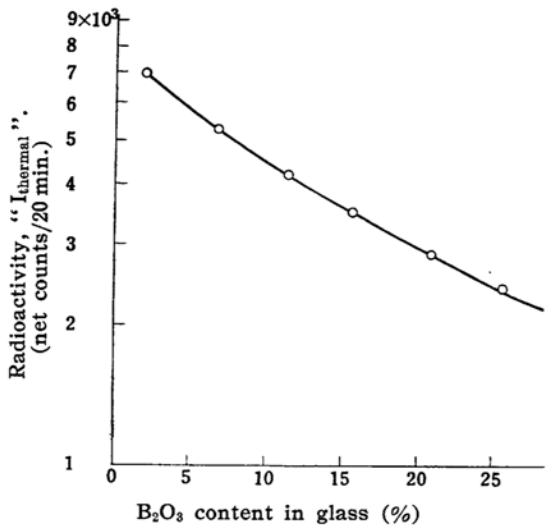

Fig. 6. Calibration curve in boron analysis.

7) The values in Table II were calculated from D. J. Hughes and J. A. Harvey's, "Neutron Cross Section", BNL 325 (1955), (U. S. A. E. C).
8) "Encyclopedia of Chemical Technology" Vol 7, Interscience Encyclopedia Inc., New York, p 181.

TABLE III
BORON ANALYSIS BY THIS METHOD AND COMPARISION WITH CHEMICAL METHOD

Sample*	Induced radioactivity (net counts/20 min.)				B_2O_3 content (%)	
	I_0.	I_{cd}.	$I_{thermal}$	Probable error	Neutron method	Chemical method
1	8159	2081	6078	68	4.3±0.2	4.44
2	6457	1994	4463	62	10.1±0.3	9.91
3	5593	1861	3732	58	14.4±0.4	13.15
4	5544	1930	3614	58	15.2±0.4	15.91
5	4390	1817	2573	53	23.9±0.6	23.28

* The samples used for this study were as follows;
No. 1: Optical glass containing BaO (19.7%), ZnO (11.9%) and PbO (3.2%), called as BaK-4.
No. 2: Optical glass containing BaO (2.5%), called as BK-7.
No. 3 and No. 4.: Commercial borosilicate glasses.

in Fig. 6, which is obtained from measurements on the glass of known boron content, it is possible to determine the boron content in any borosilicate glass.

Some experimental results by this method and the comparisons with the chemical method[9] are summarized in Table III.

The results given in Table III indicate the validity of the neutron absorption method. Fig. 7 shows the plots of the probable error in analysis versus the content of boric oxide in glass. This graph represents the degree of accuracy that can be expected from the method.

The most striking error involved in this method is due to the statistical uncertainty during the radioactivity measurement.

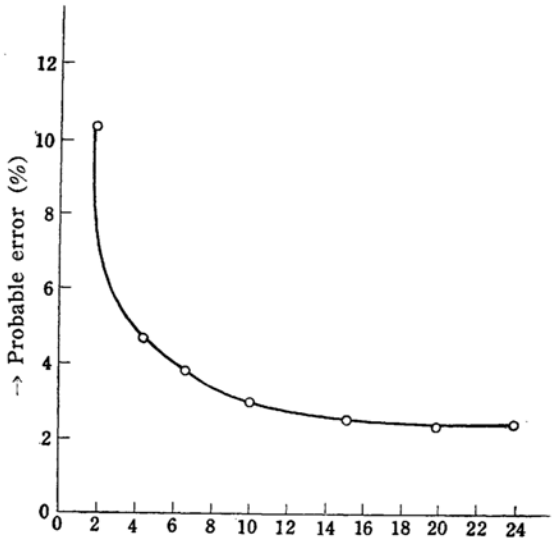

Fig. 7. Statistical error in boron analysis.

9) W. F. Hillebrand and G. E. F. Lundell, "Applied Inorg. Analysis", Wiley, New York, (1929), p 611.

Summary

The neutron absorption technique using radium-beryllium neutron source has been shown to be useful for rapid assay of boron in the samples containing other low cross-section elements. This method may also be useful for the analyses of other high cross-section elements.

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