

Precision Determination of the Slow Neutron Absorption Cross Section of B^{10} [†]

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The total neutron cross section of B^{10} has been measured at neutron energies between 0.00291 ev and 0.1 ev with the Columbia University crystal spectrometer. The B^{10} absorption cross section was obtained by subtracting the relatively small, known, scattering cross section from the measured total cross section. Two samples enriched to 92.84 ± 0.06 atom % and 99.88 ± 0.01 atom % B^{10} were used for these measurements. The B^{10} absorption cross section obtained with the 92.84% B^{10} sample followed the relation $E^{1/2}\sigma_a = 612.3 \pm 2.4$ barns $ev^{1/2}$ and yielded a value of 3849 ± 15 barns at 0.0253 ev. For the 99.88% B^{10} sample, the B^{10} absorption cross section obeyed the relation $E^{1/2}\sigma_a = 608.9 \pm 2.3$ barns $ev^{1/2}$ and

gave a value of 3828 ± 15 barns at 0.0253 ev. A sample of Argonne-Brookhaven standard normal boron, $19.8 \pm 0.1\%$ B^{10} , was measured simultaneously. The resulting absorption cross section for normal boron was $\sigma_a(0.0253 \text{ ev}) = 764 \pm 3$ barns and obeyed the relation $E^{1/2}\sigma_a = 121.5 \pm 0.5$ barns $ev^{1/2}$. The average of the results for the two enriched samples yields $\sigma_a(B^{10}) = 3838 \pm 11$ barns at 0.0253 ev and $E^{1/2}\sigma_a(B^{10}) = 610.5 \pm 1.7$ barns $ev^{1/2}$. The ratio of the above values of the absorption cross sections for B^{10} and normal boron yields a value of $19.9 \pm 0.1\%$ B^{10} for the normal sample which is in good agreement with mass spectrometer determinations.

I. INTRODUCTION

THE neutron absorption cross section of B^{10} has been obtained in the past from the measured neutron cross section of natural boron together with a mass-spectrometer determination of the B^{10} content of the sample. The absorption cross section of normal boron is almost entirely due to the $B^{10}(n,\alpha)$ reaction and is obtained from a measured value of the total cross section by subtraction of the relatively small and known, scattering cross section. Recently published values of the neutron absorption cross section of natural boron are summarized in Table I and are in good agreement with the quoted errors of about $\pm 0.7\%$. The major uncertainties in these measurements have arisen from the determination of the amount of boron in the sample and from contamination of a sample by

small quantities of water.¹ Mass spectrometer measurements of the isotopic ratio of natural boron are summarized in Table II. The values range between the extremes of 18.4% to 20.0% and give rise to a variation of $\pm 4\%$ on the calculated B^{10} cross section.

The availability of highly enriched B^{10} samples, improvements in the reliability of mass spectrometric determinations of the boron isotopic abundance, and the high degree of precision that has been attained for neutron transmission measurements in the thermal region, now make possible direct and precise measurements of the B^{10} cross section. The need for reducing the uncertainties in the boron cross section arises from its widespread use as a standard for measurement of other cross sections and from its use in boron loading specifications for reactors and in reactor physics calculations.

In the present experiment, the total neutron cross section of B^{10} was measured over the neutron energy range from 0.00291 ev to 0.1 ev. Two samples enriched in B^{10} content (92.8 atom % and 99.88 atom % B^{10} , respectively) were used. The total neutron cross section of a sample of Argonne-Brookhaven standard natural boron was also measured simultaneously with the above two samples as a check on these measurements and on the sample preparation.

II. EXPERIMENTAL PROCEDURE AND APPARATUS

The boron samples for this measurement were solutions of $NaBO_2$ dissolved in D_2O , which were contained in boron-free quartz cells of known thickness. A separate cell of identical thickness (matched to 0.1% in thickness) contained Na_2SO_4 in a D_2O solution. This cell was used to determine the effective transmissions of the elements other than boron in the cell containing the $NaBO_2$. The concentrations of the $NaBO_2$ and

TABLE I. Recent measurements of the 2200-m/sec absorption cross section for natural boron.

Source	σ_a (barns)	Type of measurement
Argonne ^a	755 ± 3	Transmission
Brookhaven ^b	749 ± 4	Transmission
Oxford ^c	752 ± 3	Neutron lifetime
Harwell ^d	762 ± 5	Transmission
Columbia University ^e	756 ± 6	Transmission
Stockholm ^f	760 ± 4	Neutron lifetime
U. S. S. R. ^g	757.5 ± 3.5	Transmission

^a B. Hamermesh, G. R. Ringo, and S. Wexler, Phys. Rev. **90**, 603 (1953).

^b R. S. Carter, H. Palevsky, V. W. Myers, and D. J. Hughes, Phys. Rev. **92**, 716 (1953).

^c C. H. Collie, R. E. Meads, and E. E. Lockett, Proc. Phys. Soc. (London) **A69**, 464 (1956).

^d P. A. Egelstaff, J. Nuclear Energy **5**, 41 (1957).

^e W. W. Havens, Jr., E. Melkonian, and B. M. Rustad, Columbia University Report CU-160, 1957 (unpublished).

^f G. Von Dardel and N. G. Sjöstrand, Progress in Nuclear Energy, edited by O. J. Hughes, J. E. Sanders, and J. Horowitz (Pergamon Press, New York, 1958), Vol. II, p. 183.

^g Inginatev, Kirpichikov, and Sukhoruchkin, Proceedings of the Second United Nations International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958 (United Nations, Geneva, 1958), Vol. 16, p. 209.

[†] Work performed under the auspices of the U. S. Atomic Energy Commission.

¹ D. J. Hughes, Proceedings of the Second United Nations International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958 (United Nations, Geneva, 1958), Vol. 16, p. 8.

TABLE II. Mass spectrometric determinations of the isotopic composition of natural boron.

Source	Atomic % B ¹⁰
Thode <i>et al.</i> ^a	19.0–18.5%. Reported a 3% geographic variation in the B ¹¹ /B ¹⁰ isotope ratio.
Carter <i>et al.</i> ^b	A measurement at Oak Ridge National Laboratory on Argonne-Brookhaven standard material yielded 18.5% (systematic error not estimated). It was found to be the same within $\frac{1}{2}\%$ as B ¹⁰ content of "California boron" measured at Oak Ridge National Laboratory.
M. G. Ingram ^c	(18.83±0.2)%
V. Shiuttse ^d	Italian boron (19.6±0.3)%.
	Central Asian boron (19.0±0.3)%.
Panchenkov <i>et al.</i> ^e	(18.4±0.2)%
Melton <i>et al.</i> ^f	20.0%. "Run of mine material" at Oak Ridge National Laboratory.

^a H. G. Thode, J. Macnamara, F. P. Lossing, and C. B. Collins, J. Am. Chem. Soc. **70**, 3008 (1948).

^b R. S. Carter, H. Palevsky, V. W. Myers and O. J. Hughes, Phys. Rev. **92**, 716 (1953).

^c M. G. Ingram, Phys. Rev. **70**, 653 (1946).

^d V. Shiuttse, J. Exptl. Theoret. Phys. (U.S.S.R.) **29**, 486 (1955) [translation: Soviet Phys. JETP **2**, 402 (1956)].

^e G. M. Panchenkov and V. D. Motsseev, Zh. Fiz. Khim. **30**, 1118 (1956).

^f C. E. Melton, L. O. Gilpatrick, R. Baldock, and R. M. Healy, Anal. Chem. **28**, 1049 (1956).

Na₂SO₄ were adjusted such that a BO₂⁻ ion is compared with $\frac{1}{2}$ SO₄⁻ and the number of Na⁺ ions per cc was the same for the two cells. When the ratio of the transmission of the two cells is taken, the effects of the Na⁺ cancel as do the effects of the D₂O except for a small, measured D₂O off-balance, for which a correction is made, due to the slightly different volumes occupied by the BO₂⁻ and $\frac{1}{2}$ (SO₄⁻) ions in solution. In addition, the above balancing technique reduces the size of the relatively small correction for the effective scattering cross sections of the BO₂⁻ and the $\frac{1}{2}$ SO₄⁻ ions, including the interference effects in the coherent scattering cross sections of these ions.

The Columbia University neutron crystal spectrometer at the Brookhaven National Laboratory reactor was used for these measurements. Similar types of high precision spectrometers have been described previously in the literature.² In the energy range from 0.0253 ev to 0.1 ev, a germanium single crystal oriented to the (111) plane, for which second order reflections is forbidden, is used for the neutron monochromator together with a neutron filter composed of large quartz crystals.³ The measured, fractional higher-order contamination is less than 0.05% in this energy range. From 0.00291 to 0.0253 ev, a mechanical neutron velocity selector was used to select only those neutrons from first order reflections.⁴ At energies above 0.006 ev, a germanium crystal was used as a spectrometer monochromator. At lower energies, a mica crystal having a

larger lattice spacing was used. The measured total higher-order contamination in the beam after the velocity selector is less than 0.2%.⁵

The data for the transmission measurements were taken at each energy in the sequence, beam open - sample in - sample in - beam open, to minimize the effect of intensity drifts in the neutron beam from the reactor. More than 300 such cycles were taken at each energy to obtain the desired statistical accuracy of about 0.1%. The background was measured by turning the crystal 2° from the peak reflection and repeating the above sample sequence. The transmission data at each energy consisted of four or five individual measurements spaced as much as two weeks apart. In all cases, the resulting cross sections were found to agree with the statistical accuracy and showed that the data were reproducible.

Possible systematic errors in the measurements, such as energy determination, resolution, background, and detector dead time have been investigated and were found to be negligible.

III. PREPARATION OF THE SAMPLES

The B¹⁰ isotopic contents of the samples, as determined by mass spectrometer measurements, are given in Table III. As a check on the reliability of mass spectrometer determinations, two independent measurements of the B¹⁰ isotopic abundance of each sample were made; one at Knolls Atomic Power Laboratory and the other at Oak Ridge National Laboratory. The results are in excellent agreement.

A major difficulty in producing boron samples for neutron transmission measurements has been the preparation of a weighed portion of a known boron compound which is free from water. In most of the reported measurements,⁶ a weighed portion of the fused B₂O₃ was dissolved in D₂O. Recent experiments have shown, however, that it is very difficult to remove the last traces of interstitial water in preparing the

TABLE III. Isotopic purity of boron samples (mass spectrometer determinations).

Sample	Atomic % B ¹⁰	
	Knolls Atomic Power Laboratory ^a	Oak Ridge National Laboratory
No. 1	(99.88±0.01)%	(99.89±0.05)%
No. 2	(92.84±0.06)%	(92.8±0.1)%
No. 3 (Argonne-Brookhaven standard boron)	(19.8±0.2)%	(19.8±0.1)% ^b

^a Errors quoted are at the 95% confidence level.

^b Oak Ridge National Laboratory reports a measurement of (19.9±0.1)% B¹⁰ for a separate batch of Argonne-Brookhaven standard boron. This number was obtained from four determinations in two spectrometers with Na₂B₄O₇ on the sample filament [private communication from J. R. Sites (ORNL) to G. J. Safford, January, 1960.]

⁵ J. Moore, J. Rush, and B. M. Rustad, Bull. Am. Phys. Soc. **4**, 245 (1959).

⁶ R. S. Carter, H. Palevsky, V. W. Myers, and D. J. Hughes, Phys. Rev. **92**, 716 (1953).

² V. L. Sailor, H. L. Foote, H. H. Landon and R. E. Wood, Rev. Sci. Instr. **27**, 26 (1956).

³ B. M. Rustad, T. Wajima, and E. Melkonian, Bull. Am. Phys. Soc. **4**, 245 (1959).

⁴ N. Holt, Rev. Sci. Instr. **28**, 11 (1957).

fused boron oxide from boric acid.⁷ In the present experiment, the preparation of the boron sample followed a modification of a procedure used to prepare samples of B₂O₃ for the precise determination of the molar heats of solution of boric oxide.⁸ The desired weight of B₂O₃ was prepared directly in a calibrated volumetric flask by dehydration of boric acid at a controlled low pressure. The boric acid was heated at a temperature of 275°C and at a pressure of 1–4 mm of Hg for 10 hours. During this period, dry nitrogen at the rate of 1–2 cc/minute was flowed through the flask. The neck of the flask was cooled by an ice bath to condense any H₃BO₃ that may have volatilized. The flask neck was later separately heated to 285° to convert the boric acid deposited on the neck to B₂O₃, and the nitrogen flow was reversed to carry it back to the flask. At the temperatures involved, the B₂O₃ is not volatile. Prolonged heating did not give rise to any change in weight of the B₂O₃ sample. Amorphous boric oxide prepared in this manner is extremely porous and has a large effective surface area which facilitates dehydration. A weighed portion of B₂O₃ produced by this technique was dissolved in a known volume of H₂O and titrated using 0.1N NaOH with α naphtholphthalein in the presence of mannitol as an end point indicator. The result showed that the dehydrated sample corresponded to B₂O₃ within $\pm 0.1\%$ experimental error. In addition, the loss in weight of the initial samples boric acid dehydrated by this technique according to the reaction $2\text{H}_3\text{BO}_3 \rightarrow \text{B}_2\text{O}_3 + 3\text{H}_2\text{O}$ was determined. A careful titration of the initial H₃BO₃ determined its possible B₂O₃ content. The experimental loss of weight was equal to the computed one to within 0.1%.

The NaBO₂ sample was prepared by adding D₂O directly into the volumetric flask to dissolve the weighed charge of B₂O₃. Pure metallic sodium was added gravimetrically to form NaBO₂. Heavy water was then added to dilute the solution to the final calibrated volume. The solutions were then placed in identical boron-free quartz cells whose effective thicknesses were 10 ± 0.01 mm. The number of grams of boron per cc was calculated from the weight of the known compound, B₂O₃, and the calibrated volume of the flask and yielded the results: 0.004210 ± 0.000010 g/cc for the 92.84% B¹⁰ sample, 0.02425 ± 0.00061 g/cc for the natural boron sample, and 0.004180 ± 0.000011 g/cc for the 99.88% B¹⁰ sample. At the conclusion of the experiment, quantitative analyses for the boron concentration of each of the three samples agreed with the above values to within 0.5%, the accuracy of this analysis.

⁷ W. O. Haas, F. M. Rourke, J. L. Mewherter, and P. H. McDonald, Atomic Energy Commission Research and Development Report KAPL-2062 Office of Technical Services, Department of Commerce, Washington 25, D. C. (1959).

⁸ E. R. Van Artsdalen and K. P. Anderson, J. Am. Chem. Soc. **73**, 579 (1951).

The use of NaBO₂ and the above chemical procedure also avoided other difficulties previously encountered in the determination of the B¹⁰ cross section. When B₂O₃ dissolved in D₂O is used for a sample, it has been necessary in certain cases to heat the sample to an evaluated temperature during the measurements to keep the desired amount of boron in solution.⁶ As NaBO₂ is highly soluble in D₂O no such heating was necessary. In addition, all the chemical preparation of the standard solution of NaBO₂ was performed directly in the calibrated flask. This avoided possible errors due to losses of boron during transfer procedures.

The Na₂SO₄ solution was prepared by the vacuum dehydration of a chemically pure sample of sodium sulfate in a calibrate flask in a similar manner to the above preparation of the B₂O₃. Heavy water was then added to the desired weight of Na₂SO₄ to bring the solution to the standard volume.

The small difference in the number of D₂O molecules per cc arising from the different volumes occupied by the two compounds in solution was determined by subtracting the known weights of Na₂SO₄ and NaBO₂ from the weights of the final contents of each volumetric flask.

Spectrochemical analyses of the final Na₂SO₄ and NaBO₂ solution showed chemical impurities including rare earths were small, giving rise to about a 0.1% correction of the final values of the cross sections.

IV. RESULTS AND CONCLUSIONS

The final value of the B¹⁰ neutron absorption cross section was calculated from the ratio of transmissions of the Na₂SO₄ and NaBO₂ cells; the measured thicknesses of the quartz cells; the isotopic B¹⁰ content of the sample; and the weight of the B₂O₃ in the standard volume of solution.

The Remington Rand 409.2R computer at Brookhaven National Laboratory was used to calculate the sample transmissions for each data collection cycle, the statistical variation in the data based on the counting statistics, and the standard deviation of each set of measurements. All sets of data were satisfactory on the basis of Pearson's Chi-squared test. All errors quoted unless otherwise stated are one standard deviation.

The ratio of the measured transmissions of the NaBO₂ and the Na₂SO₄ sample cells may be expressed as:

$$\frac{T(\text{NaBO}_2)}{T(\frac{1}{2}\text{Na}_2\text{SO}_4)} = \exp\{-n f \sigma_a [1 + C_1 + C_2 + C_3]\},$$

where f is the fractional isotopic B¹⁰ content of the enriched samples; n is the number of atoms per cm² of total boron in the sample;

$$C_1 = \frac{[(1-f)\sigma_a(\text{B}^{11}) - \frac{1}{2}\sigma_a(S)]}{f\sigma_a(\text{B}^{10})}$$

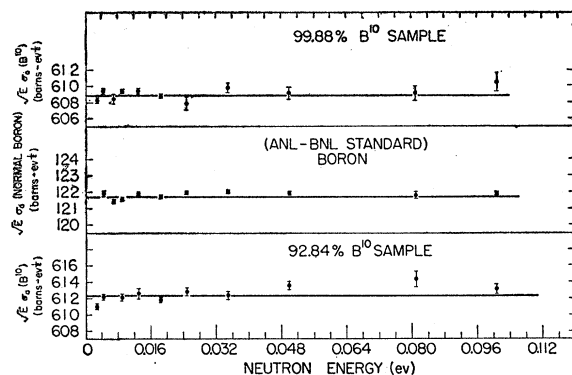


FIG. 1. The variation of $E^{1/2}\sigma_a$ for B^{10} as obtained with the 99.88% B^{10} , the 92.84% B^{10} samples, and the Argonne-Brookhaven standard boron. The solid lines are weighted least-squares fits to a straight line for each of the three sets of data shown.

is a correction term for the relatively small, absorption cross sections of B^{11} and sulphur in the samples;

$$C_2 = \frac{\sigma_s(BO_2^-) - \frac{1}{2}\sigma_s(SO_4^{--})}{f\sigma_a(BO_2^-)}$$

is a term giving the fractional correction for the effects of scattering by the BO_2^- and SO_4^{--} ions; and $C_3 = [\Delta n\sigma(D_2O)]/fn\sigma_a(B^{10})$ is a correction for a small off-balance in number of D_2O molecules per cc between the two cells due to an BO_2^- ion occupying a slightly different volume in solution than a $\frac{1}{2}SO_4^{--}$ ion.

The values of f , the isotopic content of B^{10} in the enriched samples are given in Table III. In the case of the boron sample, f is effectively 1 if the absorption cross section for normal boron is being considered.

The final values of n the effective sample thicknesses used to obtain the cross sections, were $(2.5162 \pm 0.0072) \times 10^{+20}$ atoms/cm² for the 99.88% B^{10} sample, $(2.5141 \pm 0.0072) \times 10^{+20}$ atoms/cm² for the 92.84% B^{10} sample, and $(13.505 \pm 0.039) \times 10^{+20}$ atom/cm² for the normal boron sample.

In the correction term C_1 the absorption cross section for oxygen does not enter the calculation due to the balancing of the two cells contain the same number of atoms of oxygen atoms per cc. The absorption cross sections of sulphur and B^{11} were assumed to obey a $(1/v)$ behavior and were normalized to the 2200-m/sec values; of $\sigma_a(B^{11}) < 50$ mb and $\sigma_a(S) = 0.52 \pm 0.02$ barn, respectively.⁹ The magnitude of the correction was -0.028% for the enriched samples and -0.13% for the normal boron sample.

The term, C_2 includes a calculation of the interference terms in the coherent scattering cross sections of the BO_2^- and SO_4^{--} ions.

The scattering cross sections¹⁰ which were used in

⁹ *Neutron Cross Sections*, compiled by D. J. Hughes and R. B. Schwartz, Brookhaven National Laboratory Report BNL-325 (Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C., 1958), 2nd ed., p. 3 and 5.

¹⁰ D. J. Hughes and R. B. Schwartz, see reference 9, p. 3-5.

the calculation of this term were $\sigma_s(B^{10}) = 4.0 \pm 0.5$ barns, $\sigma_s(B^{11}) = 4.4 \pm 0.3$ barns, $\sigma_s(O) = 4.24 \pm 0.02$ barns, and $\sigma_s(S) = 1.2 \pm 0.2$ barns. This term corresponds to a correction of $+0.20\%$ for the enriched samples and to $+0.98\%$ for the normal boron sample at 0.0253 ev.

In the correction C_3 , the term $\Delta n(D_2O)$, the difference in the number of D_2O molecules per cm², was obtained by the weighing technique described previously. The total cross section of D_2O versus neutron energy was obtained from the literature.¹¹ This correction was $+0.48\%$ for the enriched samples and $+1.87\%$ for the normal boron sample at thermal energy.

The values of $E^{1/2}\sigma_a$ vs E for each of the three samples are plotted in Fig. 1. The errors shown are those for counting statistics only. The solid lines in Fig. 1 are the weighted least-squares-fit to a straight line for the function $E^{1/2}\sigma_a = a + bE$. For all three samples, the coefficient (b) was less than 2.5 times the standard deviation. The resulting 2200-m/sec values of σ_a and

TABLE IV. The neutron absorption cross sections of B^{10} and natural boron.

Sample	σ_a (2200 m/sec) (barns)	$E^{1/2}\sigma_a$ (2200 m/sec) (barns ev ^{1/2})
No. 1	$\sigma_a(B^{10}) = 3828 \pm 15$	608.9 ± 2.3
No. 2	$\sigma_a(B^{10}) = 3849 \pm 15$	612.3 ± 2.4
No. 3 Argonne-Brookhaven standard boron	σ_a (normal boron) $= 764 \pm 3$	121.5 ± 0.5

$E^{1/2}\sigma_a$ are given in Table IV for the three samples. The B^{10} cross section obtained from weighted average of the above measurements is 3838 ± 11 barns at 0.0253 ev and follows the relation $E^{1/2}\sigma_a = 610.5 \pm 1.7$ barns ev^{1/2}. The ratio of the thermal values of the B^{10} and the normal boron absorption cross sections gives $(19.9 \pm 0.1)\%$ for the atomic % B^{10} for Argonne-Brookhaven normal boron. This value is in good agreement with the mass spectrometer measurements given in Table III.

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¹¹ D. J. Hughes and R. B. Schwartz, see reference 9, p. 78.