MASTER

COMPARATIVE BORON ISOTOPIC ANALYSIS

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A B S T R A C T

Surface ionization results for natural boron isotopic analysis are in agreement with other recent investigations indicating a B^{11}/B^{10} atom ratio nearer to 4.00 than the more commonly accepted value of 4.31 based on BF_3 analysis.

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INTRODUCTION

The isotopic abundance of natural boron is of interest because of its wide use as a standard neutron absorber. There has not been recent general agreement in the field concerning the B^{11}/B^{10} ratio of natural boron. Inghram⁽¹⁾ reported a value of 4.31 based on mass spectrometric analysis of boron trifluoride, a compound noted for its adsorption characteristics resulting in "instrument memory". Since publication of that work a number of attempts to verify the abundance ratio with boron trifluoride have resulted in a wide range of values. More recent work with gaseous boron hydrides, trimethylboroxine⁽²⁾, and methyl borate⁽³⁾ as well as surface ionization work at KAPL^(10,11) indicate that a B^{11}/B^{10} atom ratio of 4.00 approaches the true natural abundance ratio more closely than the accepted value of 4.31.

Thode and his co-workers detected small variations in the relative abundance of the boron isotopes B^{10} and B^{11} in natural materials.⁽⁴⁾

Variations in natural lithium isotopes have been reported by Cameron.⁽⁵⁾ In this report the hypothesis is given that variations in both natural boron and lithium isotopic abundance may be due to neutron exposure since both elements possess isotopes of large capture cross sections. However, because of demonstrated changes from precipitation reactions in the isotopic ratio of lithium, these variations may well be ascribed to chemical effects according to Cameron.

A more recent study on variations in relative abundance of boron isotopes in nature indicated constancy in the isotopic composition of boron.⁽⁶⁾ These workers suggested that chemical preparation of boron trifluoride in the work of Thode et. al. may account for their observed variations.

In view of the wide range of values reported for the B^{11}/B^{10} ratio, and the remaining uncertainty as to variation of boron isotopes in nature, the authors are of the opinion that more work in boron isotopic analysis by surface ionization mass spectrometry is worthwhile. The technique is based on $Na_2BO_2^+$ emission from a borax bead on the filament.⁽⁷⁾ Tendency toward less "memory effects" in surface ionization instruments offers an important advantage.

The work described in this report was initiated on behalf of Reactor Technologists at the NRTS in connection with their uses of natural boron as a standard neutron absorber.⁽⁸⁾ Objectives of the program with emphasis on surface ionization mass spectrometry were as follows:

1. To obtain samples of material other laboratories have analyzed and are using as standards in order to compare their results with ICPP results.
2. To calibrate an appropriate boron-containing material for use as standard at the NRTS.

SAMPLE PREPARATION

Boron materials were obtained for this study in the form of sodium tetraborate and boric acid crystals. Boric acid was converted to the tetraborate with sodium carbonate of less than 0.0002 wt. % boron according to spectrographic analysis. It was found that impure borax beads

give emission of short duration, possibly because of poor bonding with the filament or low ionization efficiency. Stoichiometric amounts of boric acid and sodium carbonate were therefore mixed followed by several recrystallizations in water to minimize foreign materials in prepared borax. Emission is especially stable and lasting if the borax bead has a clear, glassy appearance after heating on the filament.

MASS ANALYSIS

Fundamentally the mass spectrometer used in this investigation is a 12-inch radius, 60 degree, direction-focusing instrument equipped with a vacuum lock for the introduction of solid samples without undue loss of vacuum. The instrument is described by Stevens and Inghram.⁽⁹⁾ The signal from the ion collector is amplified by use of an Applied Physics vibrating reed electrometer and Brown-Electronik recorder.

The Na_2BO_2^+ spectrum from borax is observed at comparatively low filament temperatures. Tantalum ribbon 0.001" x 0.030" is channeled with a special jig, and pure borax on the filament is heated openly to form a clear, glassy bead in the channel. Emission from a pure borax bead is ideal for accurate results. Beam intensity can be maintained at high levels for long periods. A small correction for O^{17} on the 89 peak due to the ion $(\text{Na}_2\text{B}^{10}\text{O}^{16}\text{O}^{17})^+$ can be applied if necessary.

RESULTS

Natural boron standards analyzed by surface ionization techniques were obtained from KAPL and Hanford. A very accurate blend prepared at KAPL and used to investigate bias in their surface ionization technique⁽¹⁰⁾ was also obtained. An abundant supply of boric acid (Baker-Analyzed,

Lot No. 92308) was obtained as the NRTS standard. All results are summarized in Table I.

TABLE I. COMPARATIVE RESULTS OF BORON ISOTOPIC ANALYSIS
BY SURFACE IONIZATION

(Precision limits for average values are at the
95% confidence level)

<u>SAMPLE</u>	<u>AVE. ATOM RATIO B^{11}/B^{10}</u>	
	<u>KAPL Anal.</u>	<u>ICPP Anal.</u>
KAPL-Normal Boron	* 4.041 ± 0.06	4.044 ± 0.06
	<u>Hanford Anal.</u>	<u>ICPP Anal.</u>
Hanford-Normal Boron	** 4.173 ± 0.04	4.017 ± 0.04
	<u>KAPL Anal.</u>	<u>ICPP Anal.</u>
KAPL-*Enriched Boron $B^{11}/B^{10}=3.661 \pm 0.007$ by blending	* 3.657 ± 0.08	3.666 ± 0.05
		<u>ICPP Anal.</u>
Normal Boric Acid-"Baker- Analyzed" Lot No. 92308		4.000 ± 0.06
* Ref. (10)		
** Ref. (12)		

SUMMARY AND CONCLUSIONS

Table I shows that surface ionization techniques at KAPL and ICPP agree very well within experimental limits on natural and blended boron standards.

It is particularly significant that the blended standards mass data from both locations agree with the known values and that both sets of values for a natural boron sample indicate a B^{11}/B^{10} ratio of nearly 4.00 in agreement with several recent studies. The use of 99.9% B^{10} and 98.5% B^{11} in blending the standards at KAPL resulted in nearly absolute standards, with known uncertainties in B^{11}/B^{10} ratios.

The ICPP results for the Hanford boron standard are not significantly different from other normal results listed in Table I, although no explanation is offered for the significant difference between Hanford and ICPP results.

No conclusions regarding variation of isotopic abundance in nature are made for this report because of the limited number of samples analyzed. However, the authors are of the opinion that an extensive investigation should be carried out with surface ionization techniques. Results give some assurance that a B^{11}/B^{10} atom ratio of approximately 4.00 can be expected from randomly selected natural boron samples; therefore, the experimental value of 4.00 ± 0.06 is recommended to the Reactor Technologists at NRTS for the normal boron standard available from this laboratory.

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