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Determining the Atomic Fraction of Boron Isotopes in Various Boron-Containing Inorganic Compounds

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Abstract

The article discusses a method for determining the atomic fraction of isotopes in boron trifluoride, boron carbide, boron anhydride, ferroboron and boric acid, by converting them into potassium tetrafluoroborate. This compound is then analyzed using the spectrometric electron bombardment method. This technique is known for its high accuracy and "self-verifying" capabilities, meaning that the mass fraction of isotopes can be calculated using different mass lines, allowing for the identification of any potential errors.

Keywords

Boron, Mass Spectrometer, Isotopic Analysis, Atomic Share, Electronic Bombardment, Boron-Containing Substances

1. Introduction

Isotopes of chemical elements vary from one another based on the number of neutrons they contain, which affects their atomic mass. This difference leads to slight variations in their chemical and physical properties. These distinctions determine their specific applications. Boron isotopes, in particular, exhibit significant differences in physical and chemical properties, making them valuable in scientific research, the nuclear industry, and various other fields.

The boron-10 isotope is known for its high probability of absorbing thermal neutrons, making it versatile in the nuclear industry. Its significant neutron absorption rate allows it to be used in nuclear chain reactors to help regulate reactions and ensure safety. Additionally, boron-10 isotope-enriched materials are employed to shield nuclear waste and to manage processes involving neutron

radiation.

The boron-11 isotope is utilized in thermonuclear energy research due to its clean and safe reaction with protons, which provides a promising source of energy. Enriched boron-11 solid materials are employed in various sectors of the chemical compound manufacturing industry, including the production of high-precision materials and ceramics. Additionally, these materials are used to manufacture sensors, detectors, and semiconductors. Neutron detectors are also used to monitor nuclear installations effectively.

The unique neutron properties of boron isotopes are leading to an expanding range of industrial applications, particularly in areas where the specific characteristics of nuclear reactions are crucial. As demand for these isotopes increases, so does the performance of boron isotope separation units. To effectively monitor the separation process and certify the final products, high-precision isotopic analysis methods and tools are essential.

The working substance in the boron isotope separation unit is gaseous boron trifluoride; however, this compound is characterized by high adsorption properties. During isotopic analysis, boron trifluoride that has adsorbed onto surfaces can alter the isotopic composition of the sample being measured, particularly if the samples differ significantly in their isotopic content. To eliminate the "memory effect", mechanical cleaning or chemical treatment of the ion source components can be employed. Additionally, the ceramic parts of the ion source should be cleaned using an acid solution. If the difference in the atomic fraction of isotopes between the samples is minimal, flushing with an inert gas stream, heating, or performing multiple flushes with the analytic sample may effectively remove this effect.

Thermal ionization and inductively coupled plasma ionization methods are utilized to measure the atomic fractions of boron isotopes in solid compounds modified with boron isotopes. Additionally, for air analysis, electronic bombardment can be employed.

In the literature [1]-[5], attention primarily focuses on determining the atomic composition of boron isotopes in boron carbide, which requires additional processing. Measuring high concentrations of boron-11 with these methods is challenging.

Boron trifluoride is a gaseous compound with high absorption properties. Excluding the "memory effect" from previous samples is a challenging process.

The mass spectrometric determination of boron isotope fractionation using electrical bombardment is known for its high accuracy, stable ion current, and informative results. However, boron-containing solid compounds tend to vaporize only at high temperatures. When the evaporator is heated to these high temperatures, it can disrupt the electronic optics, leading to a decline in the adjustment conditions of the mass spectrometer and the accuracy of measurements. Therefore, it is preferable to conduct isotopic analysis using thermal ionization or inductively coupled plasma ionization methods.

2. Experimental

This article presents a mass spectrometric method for determining the atomic fraction of boron isotopes in boron trifluoride, boron carbide, boron anhydride, ferroboron and boric acid that have been modified with boron isotopes. The compounds are first converted into potassium tetrafluoroborate. Subsequently, the atomic fraction of boron isotopes is measured using an electron deposition mass spectrometric method.

МИ-1201 mass spectrometer was utilized for isotope analysis, which is designed to permit only gaseous samples for introduction into the mass spectrometer. To enable isotopic analysis of solid-phase samples, a gas ion source-based sample introduction system was developed [6]. The design of the ion source has undergone significant changes. By employing electronic bombardment with this system, the accuracy of measurements has improved; however, eliminating the "memory effect" has proven to be quite challenging.

To accurately determine the atomic share of the boron isotope and release it from substances adsorbed on the surface, a new construction was developed based on the ion source (**Figure 1**):

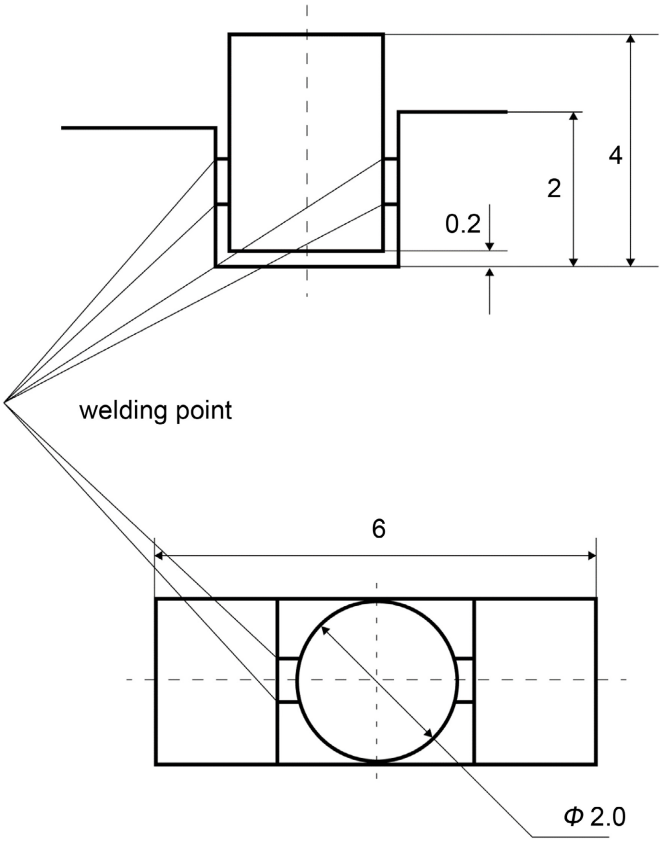

Figure 1. Evaporator.

The bottom cathode of the source has been replaced with a specialized cylindrical tantalum evaporator. The standard hole in the ionization cell, which has a

diameter of 1 mm, has been expanded into a rectangular shape measuring 3.5 mm by 2.0 mm. This shape is then formed into a cylinder with a height of 4 mm and a lateral overlap of 0.5 mm. Spot welding is applied at three or four points using a spot welder.

A cylinder holder, measuring 100 mm in length and 2 mm in width, is cut from a 100 μm tantalum plate. This rectangle is then shaped into the desired form using a specially designed stamp, as illustrated in **Figure 1**. The cylindrical evaporator is attached to the holder using a dot clamp.

A cylindrical sample is placed in the evaporator and secured to the lower cathode holders. A current is supplied to the evaporator, allowing potassium tetrafluoroborate to vaporize. For analysis, boron trifluoride modified with isotopes, boric acid, boron anhydride, boron carbide, basic boron, and ferroboron are transferred to potassium tetrafluoroborate. Since these compounds vaporize at high temperatures, heat can damage electronic optics, compromise alignment quality, and impair the tool's ability to separate components. Additionally, the evaporation process is inherently unstable.

3. Resects and Discussion

To determine the atomic fraction of boron isotopes, the compounds must first be converted to potassium tetrafluoroborate. The following processes occur during mass spectrometric analysis of potassium tetrafluoroborate. There exists a low-energy bond between the potassium and boron groups, which is easily broken. The electronic charge is primarily localized on the boron trifluoride group. In the resulting spectrum, the peaks with m/z = 48; 49 are the most prominent. Additionally, there are intense peaks at m/z = 29; 30, and m/z = 67; 68.

It is important to note that fluorine is a mono isotopic element, meaning there is no isotopic overlap on the relevant mass lines, which helps reduce errors caused by discrimination. The atomic proportions of boron isotopes can be determined by analyzing the intensities of the BF^+ ions (m/z = 67; 68), as well as the intensities of the BF_2^+ (m/z = 48; 49) and DF_3^+ (m/z = 67; 68) ions. However, in the spectrum of potassium tetrafluoroborate, the mass lines corresponding to BF_2^+ ions with m/z = 48; 49 are the most intense. Therefore, it is preferable to use these mass lines for isotope analysis.

Isotope ratio:

$$y = {}^{10}\mathrm{B}/{}^{11}\mathrm{B} \tag{1}$$

It is calculated as follows:

$$y = I_{48}/I_{49} \tag{2}$$

where I_{48}, I_{49} refer to the intensities of the mass lines at m/z = 48; 49, respectively.

The atomic fraction of the isotopes of ^{10}B is calculated using the following formula:

$$X_{{}^{10}\mathrm{B}}\% = y/y+1*100\% \tag{3}$$

And atomic composition of the isotope ^{11}B can be expressed using the following formula:

$$X_{^{11}B}\% = 1/y + 1 * 100\% \quad (4)$$

The atomic fractions of boron isotopes can be calculated based on the intensities of ions with mass-to-charge ratios of m/z = 28; 29 and m/z = 67; 68. This approach also allows for verification of the results if needed, making the method self-verifiable. This characteristic enhances the reliability of the method.

Isotopic analysis of different compounds with the same atomic share of boron isotopes by converting them into potassium tetrafluoroborate was performed on three different МИ-1201 type mass spectrometers by different operators.

The measurement results of the atomic fraction of the boron-10 isotope are presented in Table 1:

Table 1. Analysis results of the Boron-10 isotope.

Measurement №	^{10}B %	^{10}B %	^{10}B %
1	20.2	60.4	86.7
2	20.0	60.2	86.7
3	20.3	60.2	86.7
4	20.0	60.1	86.6
5	20.1	60.1	86.7
6	20.1	59.9	86.6
7	20.3	60.2	86.8
8	20.2	60.0	86.8
9	20.2	60.0	86.7
10	20.0	60.0	86.7

Results of measuring the maximum amount of the boron-11 isotope are presented in Table 2:

Table 2. Analysis results of the Boron-11 isotope.

Measurement №	^{11}B %
1	99.9969
2	99.9969
3	99.9969
4	99.9971
5	99.9969
6	99.9970
7	99.9971
8	99.9969
9	99.9969
10	99.9972

To eliminate the "memory effect", the source is boiled in distilled water after each measurement. This process effectively eliminates the error caused by this effect.

4. Conclusions

By adopting this approach, standard methods were developed to determine the atomic fraction of boron isotopes in various materials, including boron trifluoride, boron carbide, basic boron, ferroboron, boron anhydride, and boric acid. These methods were successfully implemented in both the boron isotope separation process and in the quality control and certification of the final product.

Based on the research conducted, the following conclusions can be drawn:

The conversion process of boron trifluoride, boron carbide, boron anhydride, ferroboron and boric acid to Potassium tetrafluoroborate prevents changes in isotopic content.

The method eliminates influence of the "memory effect" on isotopic analysis results.

The method is characterized by its high reliability and accuracy, and it can be tested independently.

The method enables the determination of the atomic fractions of boron-10 and boron-11 isotopes, ranging from 0.003% to 99.997%.

Isotopic analysis is conducted using the absolute measurement method and does not require instrument calibration or any additional actions.

This method does not require any additional steps.

Conflicts of Interest

The authors declare no conflict of interest regarding the publication of this paper.

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