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# DETERMINATION OF $^{10}B/^{11}B$ ISOTOPIC RATIO AND CONCENTRATION OF BORON IN STAINLESS STEEL BY ICP MS

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The large thermal neutron absorption cross-section of the $^{10}B$ enables the use of boron as a neutron absorber for reactivity control in nuclear reactors. Precise information on both the isotopic composition and boron concentration in absorbing materials is crucial, as the degree of reactivity control depends directly on the $^{10}B$ content. In the work, a series of experiments were conducted to determine the boron concentration and isotopic ratio in samples of corrosion-resistant chromium-nickel stainless steel, which is used in the control and protection system rods of nuclear reactors. The study was performed using an inductively coupled plasma mass spectrometer on 5 stainless steel samples with a certified boron mass fraction. The external standard method was used to determine the $^{10}B/^{11}B$ isotopic ratio, using the ICP-MS-68A Standard (with a natural $^{10}B$:$^{11}B$ ratio of 19.9:80.1) as a reference. To determine the boron concentration in steel, the isotope dilution method (internal standard method) was used. A known amount of a spike with a specific isotopic ratio was added to samples of unknown boron content. Elemental amorphous boron powder with a $^{10}B$:$^{11}B$ isotopic ratio of 95.0:5.0 was used as the spike. The proposed methods allow determining the isotope ratio and boron concentration in a sample by measuring only the $^{10}B$ and $^{11}B$ isotopes. The results obtained were compared with the manufacturer's certified data. The values coincide within the measurement uncertainty, confirming the reliability of the proposed methods for steel analysis.

**Keywords:** *Boron isotopic ratio; Boronated steel; ICP MS; Isotope dilution*

**PACS:** 29.30.-h

## 1. INTRODUCTION

Due to the large cross-section of absorbing the thermal neutrons in the $^{10}B$ isotope, boron in various forms is used to control nuclear reactor reactivity by absorbing neutrons [1]. Since the degree of reactivity control depends on the amount of $^{10}B$, precise knowledge of both the isotopic composition and the concentration of boron in the neutron-absorbing material is highly important for the nuclear industry [2, 3]. In addition, information about the isotopic composition and the concentration of boron in different samples is important for other fields of science, such as archaeology, geology, and medicine [4-6].

Various methods are used to determine isotopic ratios, such as atomic absorption spectrometry [7], thermal ionization mass spectrometry (TIMS) [8], secondary ion mass spectrometry (SIMS) [9], as well as others. Each of these methods has its own specific advantages and disadvantages.Among them, TIMS is renowned for providing extremely high accuracy and precision in isotope ratio measurements. However, the high ionization potential of boron prevents the production of singly charged boron ions using TIMS. Attempts are made to bypass this problem by using different sample preparation procedures, but this complicates the sample preparation process and degrades the accuracy of isotope ratio measurement.

An alternative to TIMS for precise boron isotope ratio measurements is high-resolution inductively coupled plasma mass spectrometry (ICP MS) [10].

In this study, a methodology for determining both the boron isotope ratio and the boron concentration in samples of corrosion-resistant chromium-nickel stainless steel alloyed with boron using a single-collector inductively coupled plasma mass spectrometer will be presented. Boronated steel is one of the promising materials for use as a neutron absorber for control and safety rod systems in a nuclear reactor [11].

A feature of this study for determining the boron content in the sample is the use of the isotope dilution method [12,13], which is based on the internal standard principle. This method yields a faster result with fewer calculation steps, which reduces the error in determining the required data. Isotope dilution method is an analytical technique, the essence of which lies in introducing a known amount of a tracer (spike) with a different boron isotopic composition into a sample that has a defined boron isotopic composition but an unknown elemental mass content. After the addition of the spike, the boron isotopic ratio changes, and the boron content in the initial sample can be calculated from the magnitude of this change. As is known, boron has two stable isotopes ($^{10}B$ and $^{11}B$), which makes it an excellent candidate for using this method.

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## 2. EXPERIMENTAL TECHNIQUE

### 2.1. Instrumentation

Measurements were conducted using a single-collector inductively coupled plasma mass spectrometer, the ICP-SFMS ELEMENT 2, whose technical characteristics are described in [14, 15]. A substantial advantage of this mass spectrometer is its high resolution and high sensitivity (~ 10 cps per 1 ppb $^{115}In$), which allows for the analysis of extremely low element contents. The high-resolution mode (10000 imp. at 10 % peak height) was used in the work, and the signal stability was better than 1 % over 10 minutes.

Physicochemical sample preparation methods were used for sample investigation, involving the conversion of samples into liquid form using high-purity distilled water, acids, and organic solvents. Liquid solutions containing the analyzed sample were introduced into an argon torch in the form of an aerosol using a peristaltic pump. Sample injection occurred for a certain number of iterations N with a uniform time interval $\triangle t$. The analytical signal is the mass spectrum of the analyzed elements, whose peak areas were measured after subtracting the average background value, which was measured before the start of the sample investigation.

### 2.2. Materials

For the inductively coupled plasma mass spectrometric method of analysis, the required concentrations of the measured samples are displayed in the ppm and ppb ranges. To obtain such concentrations, we used small aliquots of the investigated material, namely 0.1 g. This allowed us, on the one hand, to accurately measure the sample mass, and on the other, to minimize the consumption of acids and other reagents used for sample preparation (sample dissolution). Chemical analytical glassware of Class 1 was used during sample preparation to reduce measurement error. To prepare the solution of the investigated boronated steel sample, steel chips with an increased boron content up to 2 % (limits specified in the manufacturer's certificate for boronated steel samples:1.6÷2.0 %) were collected and crushed. Sampling was performed in several places of the investigated sample to obtain a representative sample and a reliable analysis result.

For the external standard method, a solution of the multicomponent standard 48 Component ICP-MS-68A Standard at 10 µg/mL in 2% $HNO_3$ (High Purity Standards (USA)) [16] was used. The solution was prepared by diluting the standards in ultrapure concentrated nitric acid ($HNO_3$) to achieve an element concentration of 1 ppm.

Amorphous boron powder, enriched up to 95 % in the $^{10}B$ isotope (manufactured by the National High Technology Center of Georgia) [17], was used as the internal standard (spike). The reference material was not subjected to additional drying or homogenization. A portion of amorphous boron weighing 0.1001 ± 0.0001 g was dissolved in 10 ml of $HNO_3$ with the addition of a small amount of distilled water under slight heating and stirring. The boron concentration in the resulting solution (spike) was 10 ppm.

In parallel, a blank solution was prepared in the same way, but without dissolving the investigated material. All the procedures mentioned above were followed during its preparation, except for the addition of the aliquot. The blank solution was used in the studies to avoid the influence of impurities contained in the reagents used for dissolution on the sample measurement results.

### 2.3. Procedure

Table 1 presents the sequence (column 1) of the measurements performed (column 2).

**Table 1.** The measurement procedure for boron isotope ratios

| Step | Procedure |
|---|---|
| 0 | Rinsing of sample introduction tube, spray chamber and nebulizer by argon (600 s) |
| 1 | Measure signal of $^{10}B$ and $^{11}B$ of blank solution ($^{10}B_{blank}$, $^{11}B_{blank}$) |
| 2 | Rinsing of sample introduction tube, spray chamber and nebulizer by argon (600 s) |
| 3 | Measure $^{10}B/^{11}B$ of ICP-MS-68A Standard ($R_{standart}$) |
| 4 | Rinsing of sample introduction tube, spray chamber and nebulizer by argon (600 s) |
| 5 | Measure $^{10}B/^{11}B$ of sample solution ($R_{sample}{}^{meas}$) |
| 6 | Rinsing of sample introduction tube, spray chamber and nebulizer by argon (600 s) |
| 7 | Measure $^{10}B/^{11}B$ of spike solution ($R_{spike}{}^{meas}$) |
| 8 | Rinsing of sample introduction tube, spray chamber and nebulizer by argon (600 s) |
| 9 | Measure $^{10}B/^{11}B$ of mix solution ($R_{mix}{}^{meas}$) |

The signal intensity of $^{10}$B and $^{11}$B in the blank solution is represented as $^{10}B_{blank}$ and $^{11}B_{blank}$ (step 1).

The determination of the $^{10}$B/$^{11}$B isotopic ratio for the standard ($R_{standart}$), sample ($R_{sample}$), spike ($R_{spike}$) and mix ($R_{mix}$) solutions was performed using the formula:

$$R = \frac{I(^{10}B) -^{10} B_{blank}}{I(^{11}B) -^{11} B_{blank}}, \quad (1)$$

Where I($^{10}$B) and I($^{11}$B) are the signal intensities of $^{10}$B and $^{11}$B in the investigated solutions (steps 3, 5, 7, 9).

To reduce the influence of the background signal between measurements of different samples, the mass spectrometer was rinsing with argon for 10 minutes (steps 2, 4, 6, 8).

To determine the boron concentration in the steel sample, the enriched boron solution (mix) with a boron concentration of 0.01 ppm was added to the stainless steel solutions (sample). The measurement of the $^{10}$B/$^{11}$B isotopic ratio for the mixture (mix) was performed last (step 9).

## 3. DETERMINATION THE BORON ISOTOPE RATIOS

To determine the isotopic composition of the samples (sample, spike and mix), the mass spectrometer was calibrated using a standard of known elemental composition (ICP-MS-68A Standard). For the boron isotopes ($^{10}$B and $^{11}$B) the calibration characteristic — the relative sensitivity coefficient of the boron isotopes — was experimentally determined under specific conditions. This coefficient reflects the dependence of the analytical signal of the corresponding peaks on the content of these isotopes in the standard. In our case, this coefficient corresponds to the value of $R_{standart}$ (step 3 in Table 1). The stability of the $R_{standart}$ value was analyzed over a year in different measurement sessions, and Fig. 1 presents the dependence of $R_{standart}$ on the measurement session number.

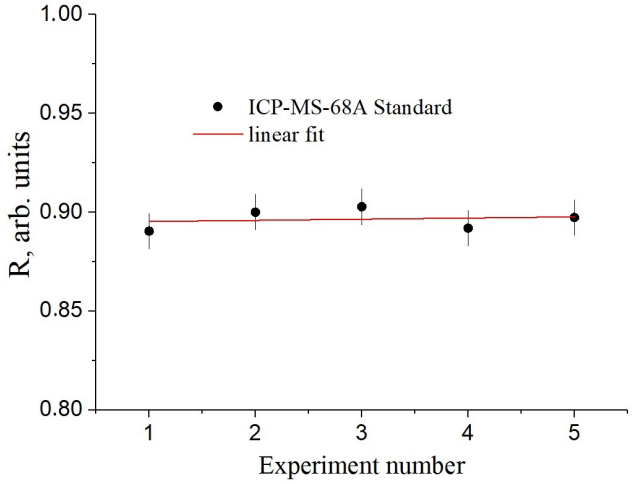


**Figure 1.** Dependence of $R_{standart}$ in different measurement sessions.

Fitting was performed using a linear function R = $a$+$b$·N, where $a$ = 0.895 ± 0.006 and $b$ = 0.001 ± 0.002. The slope ($b$) is insignificant. Also, it is visible from the figure that the $R_{standart}$ for different measurement sessions coincide within the error.

Considering the mass spectrometer calibration, the isotopic ratio of the samples was determined as $R^{corr}$=$R^{meas}$ / $R_{standart}$, where $R^{meas}$ corresponds to the measured value $R_{sample}{}^{meas}$, $R_{spike}{}^{meas}$ or $R_{mix}{}^{meas}$. The obtained results for the isotopic ratio values were compared with the certified data of the investigated samples: sample with a natural ratio of 19.9 % : 80.1 % (R = 0.248) and spike with a $^{10}$B-enriched ratio of 95.0 % : 5.0 % (R = 25.316).

Table 2 presents the values of $R_{sample}{}^{corr}$ and $R_{spike}{}^{corr}$ for the same 5 measurement sessions (Fig. 1), calculated using $R_{standart}$ according to the measurement session.

Within the errors, the determined $R_{sample}$ and $R_{spike}$ coincide with the certified values (0.248 and 25.316 respectively).

## 4. DETERMINATION OF BORON CONCENTRATION IN STAINLESS STEEL

The isotope dilution method, based on the internal standard principle [11, 12], was used to determine the boron content in the boronated steel. This method yields a quick result with fewer calculation steps, which reduces the error in determining the required data. Isotope dilution method is an analytical technique, the essence of which lies in introducing

**Table 2.** Determination the boron isotope ratios

| Session | $R_{sample}{}^{corr}$ | $R_{spike}{}^{corr}$ |
|---|---|---|
| 1 | 0.247±0.004 | 24.922±0.549 |
| 2 | 0.246±0.005 | 25.033±0.581 |
| 3 | 0.249±0.004 | 24.845±0.535 |
| 4 | 0.246±0.004 | 25.201±0.490 |
| 5 | 0.247±0.004 | 24.877±0.542 |

a known amount of a tracer ($M_{spike}$) with a different boron isotopic composition ($R_{spike}$) into a sample with a defined boron isotopic composition ($R_{sample}$) but an unknown elemental mass content ($M_{sample}$) (Fig. 2). After the addition of the spike, the boron isotopic ratio ($R_{mix}$) changes, and the boron content in the initial sample can be calculated from the magnitude of this change.

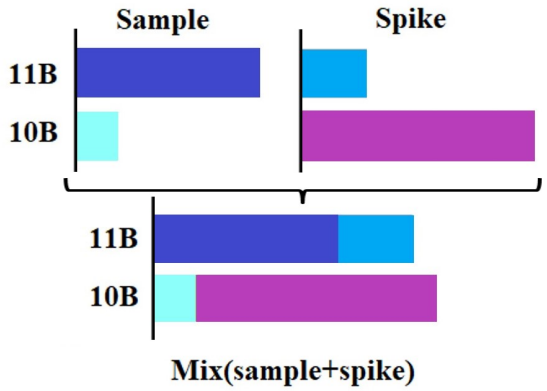


**Figure 2.** Schematic representation of the isotope dilution method.

For the resulting mixture (mix diagram at Fig.2), the mass content is $M_{mix} = M_{sample} + M_{spike}$. The introduction of the spike changes the isotopic ratio of the element, and the content of the element in the initial sample can be calculated from the magnitude of this change using the formula:

$$M_{sample} = M_{spike} \frac{1 + R_{sample}}{1 + R_{spike}} \frac{R_{spike} - R_{mix}}{R_{mix} - R_{sample}}. \tag{2}$$

Thus, if we add a known amount $M_{spike}$, the unknown mass content of boron in the sample, $M_{sample}$, can be determined from the measured R values using formula (2).

In our study, the boron content (concentration) in the boronated steel samples was measured for 5 different measurement sessions. An equal amount of the tracer (spike) was added to each sample, resulting in 5 mix samples. Thus, 5 combinations, each with 3 data sets (sample, spike and mix), were measured.

Fig. 3 shows the dependence of the peak intensity of the $^{10}B$ and $^{11}B$ isotopes as a function of measurement time for all components for one combination: sample (Fig. 3a), spike (Fig. 3b) and mix (Fig. 3c). The figure shows that the intensity dependence is linear without significant fluctuations.

The change in the relative contribution of each of the $^{10}B$ and $^{11}B$ isotopes in Fig. 3a, 3b and 3c is consistent with the logic of the isotope dilution method (Fig.2). The integrated value of the ratios $R(^{10}B/^{11}B)$ according to formula (1) was determined as the average over the entire measurement time.

In the next stage, the boron content value was calculated using formula (2). The calculation results are presented in the last column of the table. The uncertainties are statistical, taking into account the root mean square deviations for the distributions in Fig. 3.

From Table 3, it is evident that all values fall within the range specified in the manufacturer's certificate for boronated steel samples, 1.6÷2.0 %, which confirms the relevance of using this method for determining the boron content in the sample.

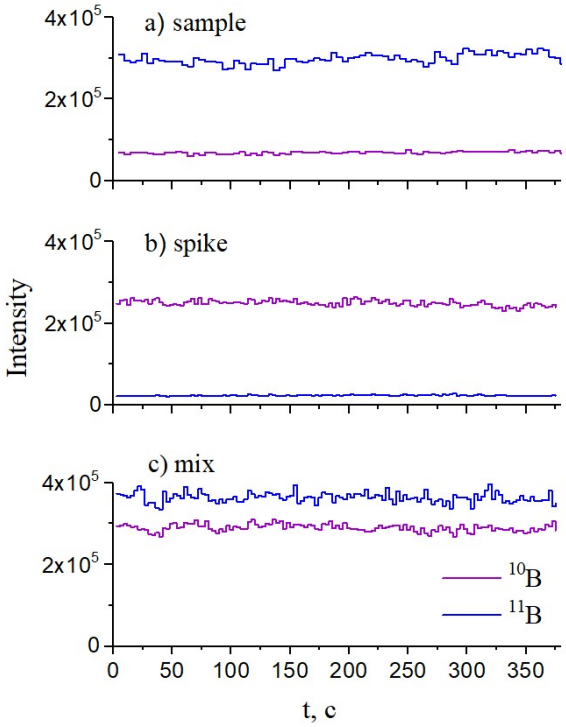


**Figure 3.** Values of the mass peak intensity for $^{10}$B and $^{11}$B isotopes as a function of measurement time for the three components: a) sample, b) spike, c) mix.

**Table 3.** Boron content in stainless steel.

| Session | Boron content, wt.% |
|---|---|
| 1 | 1.844±0.085 |
| 2 | 1.748±0.041 |
| 3 | 1.919±0.096 |
| 4 | 1.624±0.062 |
| 5 | 1.784±0.099 |

## 5. CONCLUSION

This work presents a methodology for measuring the isotopic ratio and concentration of boron in a multicomponent sample (stainless steel) based on the use of an inductively coupled plasma mass spectrometer (ICP-MS). A feature of the methodology is the use of external and internal calibration exclusively based on boron isotopes.

The multicomponent standard 48 Component ICP-MS-68A Standard at 10 µg/mL in 2% HNO3 (High Purity Standards (USA)) was used for external calibration, and amorphous boron powder, enriched up to 95 % in the $^{10}$B isotope (manufactured by the National High Technology Center of Georgia), was used for internal calibration.

The determination of the boron isotope ratio was performed using the external calibration method. Measurements were carried out during 5 time-separated sessions. The obtained results were compared with the certified values (19.9 % : 80.1 %, R = 0.248) for boronated steel and highly enriched boron powder (95.0 % : 5.0 %, R = 25.316). The measurement results agree with the certified data within the uncertain.

The isotope dilution method (internal calibration) was used to determine the concentration of boron in the steel sample, where a known amount of the element with a different isotopic composition is added to a sample with an unknown concentration of the element. By measuring the isotopic ratio in the resulting mixture, the unknown concentration of the element in the sample can be determined. An additional advantage of the method is its simplicity and speed, as it does not require measuring all elements of the steel and has fewer calculation steps, which reduces the error in determining the necessary data.

Measurements were performed for five different stainless steel samples in different measurement sessions and

compared with the certified data presented by the manufacturer. The data agree within the measurement uncertainty, which indicates the reliability of the method for determining the boron content in steel samples.

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## REFERENCES

[1] J.A. Evans, M.D. DeHart, K.D. Weaver, *et al.*, Nucl. Eng. and Des. **391**, 111726 (2022). https://doi.org/10.1016/j.nucengdes.2022.111726

[2] L. Desgranges, J.M. Escleine, P. Bienvenu, *et al.*, Nucl. Instr. and Meth. in Phys. Res. Section B: Beam Interactions with Materials and Atoms, **432**, 42 (2017). https://doi.org/10.1016/j.nimb.2018.07.011

[3] C. Subramanian, A.K. Suri, and T.S.R. Ch Murthy, BARC Newsletter, **313**, 42, (2010). https://inis.iaea.org/records/wb5s0-j1n27

[4] G.L. Foster, H.R. Marschall, and M.R. Palmer, *Boron Isotope Analysis of Geological Materials*, (Springer, 2018). https://doi.org/10.1007/978-3-319-64666-4_2

[5] L.B. Williams, and R.L. Hervig, Appl. Geochem. **19**, 1625 (2004). https://doi.org/10.1016/j.apgeochem.2004.02.007

[6] A.M. Hughes, and N. Hu, Cancers, **15**, 4091 (2023). https://doi.org/10.3390/cancers15164091

[7] H. Wiltsche, K. Prattes, M. Zischka, and G. Knapp, Spectrochim. A. Part B: Atomic Spectroscopy, **64**, 341, (2009). https://doi.org/10.1016/j.sab.2009.03.008

[8] T. Ishikawa, and K. Nagaishi, J. Anal. At. Spectrom. **26**, 359 (2011). https://doi.org/10.1039/C0JA00060D

[9] V. Karki, M. Singh, K.S. Bhushan, *et al.*, Intern. J. of Mass Spectrum, **460**, 116475, (2021). https://doi.org/10.1016/j.ijms.2020.116475

[10] D. Malinovsky, P.J.H. Dunn, and H. Goenaga-Infante, Nucl. Eng. and Des. **35**, 2723, (2020). https://doi.org/10.1039/D0JA00145G

[11] A. Saeed, Nucl. Eng. and Des. **413**, 112515 (2023). https://doi.org/10.1016/j.nucengdes.2023.112515

[12] P. Rodríguez-González, and J.I. García Alonso, J. Anal. At. Spectrom. **25**, 239, (2010). https://doi.org/10.1039/B924261A

[13] A. Quemet, A. Hubert, and A. Gourgiotis, *et al.*, J. Anal. At. Spectrom. **39**, 1665 (2024). https://doi.org/10.1039/D4JA00029C

[14] D.V. Kutnii, D.D. Burdeynyi, S.A. Vanzha, and N.V. Rud., East Europ. J. of Phys. (2), 104, (2020). https://doi.org/10.26565/2312-4334-2020-2-09

[15] D.V. Kutnii, S.A. Vanzha, D.D. Burdeynyi, *et al.*, East Europ. J. of Phys. (2), 75, (2022). https://doi.org/10.26565/2312-4334-2022-2-08

[16] ZeptoMetrix, ICP-MS Multi Element Solution Standard with 48 Components at 10 µg/mL, https://www.zeptometrix.com/us/en/icp-ms-standard-at-10-g-ml-solution-a-100-ml-ICP-MS-68A-A.

[17] National High Technology Center of Georgia, Amorphous boron powder with boron-10 isotope, http://geoisotopes.com/ka/bori-ge/b10-am-ge.

## ВИЗНАЧЕННЯ ІЗОТОПНОГО ВІДНОШЕННЯ $^{10}B/^{11}B$ ТА КОНЦЕНТРАЦІЇ БОРУ В НЕРЖАВІЮЧІЙ СТАЛІ ЗА ДОПОМОГОЮ ICP MS

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Великий переріз поглинання теплових нейтронів ізотопом $^{10}B$ дозволяє використовувати бор у якості поглинача нейтронів для контролю реактивності в ядерних реакторах. Інформація про точне значення як ізотопного складу, так і концентрації бору в нейтронпоглинаючому матеріалі є дуже важливою, адже ступінь регулювання реактивності залежить від кількості $^{10}B$. В роботі проведено серію експериментів з визначення концентрації бору та його ізотопного відношення в зразках корозійностійкої хромонікелевої неіржавіючої сталі, яка використовується як матеріал поглинаючих стрижнів системи управління та захисту ядерного реактора. Дослідження виконано з використанням мас-спектрометра з індуктивно-зв'язаною плазмою на 5 зразках неіржавіючої сталі з сертифікованим значенням масової частки бору. Для визначення ізотопного відношення $^{10}B/^{11}B$ використовувався метод зовнішнього стандарту. В якості зовнішнього стандарту використовувався спеціалізований багатокомпонентний калібрувальний розчин ICP-MS-68A Standard з природним ізотопним співвідношенням бору $^{10}B$:$^{11}B$=19.9:80.1. Для визначення концентрації бору в сталі використовувався метод ізотопного розбавлення (метод внутрішнього стандарту). До невідомого за вмістом бору зразка з природнім співвідношенням його ізотопів додавали відому кількість індикатора з іншим ізотопним відношенням. У якості індикатора використовували порошок елементарного аморфного бору зі співвідношенням ізотопів $^{10}B$:$^{11}B$=95.0:5.0. Запропоновані методи дозволяють визначити ізотопне відношення та концентрацію бору в зразку вимірюючи лише ізотопи $^{10}B$ та $^{11}B$. Отримані в роботі значення порівнювались з паспортними даними, представленими виробником. У межах невизначеності вимірювань дані співпадають, що може свідчити про надійність запропонованих методів для визначення ізотопного співвідношення та вмісту бору в зразках сталі.

**Ключові слова:** *ізотопне співвідношення бору; борована сталь; ICP MS; ізотопне розведення*