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ISOTOPE DILUTION METHOD FOR THE DETERMINATION OF BORON CONTENT IN STAINLESS STEEL BY INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY

To determine the boron content in borated samples of corrosion-resistant chromium-nickel stainless steel, the isotope dilution method is proposed, which is an internal standard method. The study was performed on 10 boron-alloyed stainless-steel samples with a natural isotope ratio using an inductively coupled plasma mass spectrometer. As an internal standard, elemental amorphous boron powder with a ^{10}B isotope ratio up to 95.0% is used. For comparison, mass spectrometric measurements of the boron content in the same samples are additionally performed using the calibration solution method, which is an external standard method. In this case, the boron content is determined as the ratio of the boron intensity to the total intensity of all elements in the sample. It is found that the results of both internal and external standard methods are qualitatively identical. However, the isotope dilution method turned more accurate for determining elemental concentrations, and its results are not affected by non-spectral interferences associated with the drift of instrument sensitivity over time and the matrix effects, since the isotope ratio rather than the absolute isotope concentrations is measured.

Keywords: isotope dilution analysis, mass spectrometer, borated stainless steel, boron isotopes.

1. Introduction

Neutron absorbers [1, 2] are the main component of the absorber rods in the control and protection systems. They perform a number of important functions in the operation of the reactor. In particular, they directly regulate the reactor power, ensure a uniform distribution of neutrons in the core, manage the reactivity reserves in the installation, and – most importantly – maintain the safety of the reactor operation. One of the promising materials for application as a neutron absorber is boron-containing austenitic stainless steel [3–5], which is associated with the ability of boron, especially the isotope ^{10}B, to absorb neutrons [6]. An additional effect of introducing boron into steel is the improvement of the final mechanical

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steel properties: borated steel retains its mechanical strength and wear resistance. It is thermally stable, corrosion-resistant, and easy to be processed. Borated steel is quite resistant to radiation exposure, which extends its service life. Such properties allow borated steel to be used as a neutron absorber in various structural components of nuclear reactors: in control and emergency rods or/and as a radiation protector [7].

Accurate information about the amount of boron in neutron-absorbing materials is extremely important, because its inadequate amount can lead to uncontrolled reactions in the reactor: the safety of its operation depends on the amount of boron in the control rods. In this regard, there is a need to control both the percentage of boron and its isotopic ratio in steel.

Most modern studies of the substance composition are based on mass spectrometric data [8,9]. Inductively coupled plasma mass spectrometry (ICP-MS) occupies a leading position in both elemental and isotopic analyses due to its high sensitivity, multicomponent capability, versatility, and high analysis speed. Modern mass spectrometry allows the simultaneous measurements of low and ultralow levels of several dozen elements to be carried out with a sensitivity of up to 10^{-13}% [10]. To obtain quantitative data, rigorous and appropriate calibration and correction methods are required. One of them is the simple external standard method, which requires preliminary measurements of the intensities of reference materials containing the analyzed elements with known concentrations (the calibration standard method). This method is the simplest calibration method, but it can only be performed, if the samples and the reference materials are matrix-matched, and the instrument operating conditions are accurately reproduced [11]. To make a more accurate quantitative analysis of samples, the simple external standard method should be combined with other methods, which increases the number of required measurements, calculation steps, and, in general, the research time.

In this work, we propose the isotope dilution analysis [12–15], which is based on the internal standard principle, to determine the boron content in borated steel. This method gives a faster result with fewer calculation steps, which reduces the error in determining the required data. The isotope dilution analysis is an analytical method. Its essence consists in introducing a known amount of an indicator with a different boron isotope composition into a sample with a known boron isotopic composition but an unknown elemental mass contribution. After the addition of the additive, the boron isotopic ratio changes, and the magnitude of this change can be used to calculate the boron content in the initial sample. As is known, boron has two stable isotopes, ^{10}B and ^{11}B, which makes it an excellent candidate for testing the specified method for determining the boron content in borated stainless steel.

The elemental content of stainless steel was determined using an inductively coupled plasma mass spectrometer Element 2 (Thermo Fisher Scientific GmbH, Germany). Besides using the isotope dilution analysis (the internal standard method), the boron content was also determined using the calibration standard method (the external standard method). A comparison of the results obtained by two methods made it possible to conclude whether the isotope dilution analysis is suitable for determining the mass content of boron in stainless steel.

2. Experimental Technique. ICP-MS

In our experiment, the measurements were carried out using a single-collector inductively coupled plasma mass spectrometer ICP-MS ELEMENT 2; its technical parameters are described in work [10]. The advantage of this mass spectrometer is its high resolution and high sensitivity ($\sim$10 cps per 1 ppb ^{115}In), which allows it to be used to analyze extremely low element contents. For all measurements in this work, the high-resolution mode was used (10000 pulses at 10% of peak height). The signal stability was better than 1% for 10 min.

2.1. Preparation of research samples

Ten samples of corrosion-resistant chromium-nickel stainless steel with an increased boron content (1.6÷2.0%), and a natural ratio of boron isotopes were studied in this work.

To study the sample using ICP MS, physicochemical sample preparation methods were used, which convert the samples into a liquid form using high-purity distilled water, acids, and organic solvents. By means of a peristaltic pump, the indicated liquid solutions were sent in the aerosol form into an argon burner. The injection of samples was made for a certain number of iterations, N, with a constant time

interval δt. The aerosol generated by the atomizer entered the base of an inductively coupled plasma torch, where ionization took place, through an injector with the help of a transporting argon flow.

As the analytical signal, the mass spectrum of the peaks of the analyzed elements was used. The peaks' areas were measured by subtracting the average background value; the latter was measured before the sample study was started. To reduce the influence of the background signal, the mass spectrometer was flushed with argon for 30 min between the study cycles of different samples.

2.1.1. Stainless steel samples

To prepare the solution of a studied borated steel sample, steel shavings with a high boron content (up to 2%) were ground. Sampling was carried out at several places of the examined sample to provide a representative sample and a reliable analysis result. A weighted sample portion of 0.1 ± 10^{-4} g was taken from the selected and prepared sample.

The sample portion was dissolved in a 100-ml heat-resistant glass. High-purity concentrated sulfuric acid was used as a solvent, to which 50% hydrogen peroxide solution was added dropwise until its complete dissolution. Then the solution was heated under constant stirring until the portion completely dissolved and the solution acquired an emerald-green color. A white precipitate formed during the dissolution process was dissolved by adding distilled water and further heating.

The solution thus obtained was poured into a 100-ml volumetric flask, the glass was washed twice with a small amount of distilled water, the washings were poured into the flask, and the solution volume in the flask was brought to 100 ml with distilled water. The solution was thoroughly mixed to obtain an equilibrium concentration.

In parallel, a reference solution (Blank) was prepared in the same way, but without dissolving the test material. When preparing it, all the procedures specified above were followed, except for adding a sample. The reference solution was applied in the studies to avoid the influence of impurities contained in the reagents used in the dissolution on the results of sample measurements.

For quantitative mass spectrometric measurements of the concentrations of the given elements in the borated stainless steel sample, the obtained initial solution of the test sample was diluted using a 2% nitric acid solution as a solvent. The initial solutions (the steel sample solution and the reference solution) were diluted 400 times.

2.1.2. Calibration standard solutions

For calibration using the external standard method, solutions of multicomponent standards were applied: the 48-component ICP-MS-68A Standard at 10 μg/mL in 2% HNO_3 (High Purity Standards, USA) and the 11-component ICP-MS-D-M Standard at 10 μg/mL in 2% HNO_3+Tr HF (High Purity Standards, USA). The need to use two solutions simultaneously arises due to the elemental composition of the stainless steel studied in this work. Among all studied elements, only boron was present in both solutions, which was taken into consideration for further calculations. The calibration solutions were prepared by diluting the standard solutions in ultrapure HNO_3 to achieve an element concentration of 1 ppm.

2.1.3. Enriched boron samples

The isotope dilution analysis requires measuring the isotopic composition of enriched boron. As a sample, we used an elemental amorphous boron powder with the boron isotope ratio $^{10}B : {}^{11}B = 95.0 : 5.0$. A 0.1 ± 10^{-4} g portion of amorphous boron was dissolved in 10 ml of concentrated nitric acid with the addition of a small amount of distilled water under slight heating and stirring. During the dissolution, the release of brown gas NO_2 was observed. The transparent solution was poured into a 100-ml volumetric flask and brought to 100 ml with distilled water. Then the mixture was stirred until the equilibrium concentration was established. The resulting solution had a boron concentration of 1000 ppm.

To prepare a solution for the mass spectrometric analysis, 0.1 ml of the indicated solution was added to 10 ml of a 2% nitric acid solution, and they were mixed well. The boron concentration in the resulting solution was 10 ppm.

2.1.4. Mixture of stainless steel and enriched boron

The enriched boron solution with a concentration of 10 ppm was added to the stainless steel solutions

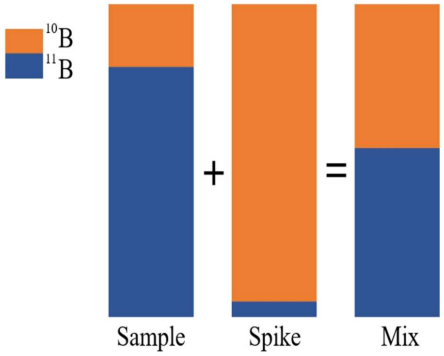

Fig. 1. Scheme of the isotope dilution analysis

prepared according to the method described in section 2 2.1 in order to obtain solutions with boron concentrations of 0.01, 0.05, and 0.1 ppm. The dosage was carried out during the measurements, with regard for the volume of the solution under study.

2.2. Isotope dilution analysis (internal standard)

The isotope dilution analysis (IDA) is an analytical method used to determine the amount of certain chemical elements in a sample [16, 17]. The application of IDA in mass spectrometric measurements (IDA-MS) has increased substantially because of the widespread use of ICP MS and the availability of standard samples from various research institutes [18]. For example, this method is most widely used for the determination of the amount of uranium and plutonium at interlaboratory comparisons [19].

The basic idea of IDA (Fig. 1) consists in introducing a known amount M_{spike} of an indicator with a known isotopic composition β_{spike} (the diagram Spike) into a sample containing an unknown amount M_{sample} of an element with a known isotopic composition β_{sample} (the diagram Sample); the isotopic compositions of the indicator and the sample are different, $\beta_{\text{spike}} \neq \beta_{\text{sample}}$. The mass content of the resulting mixture (the diagram Mix) $M_{\text{mix}} = M_{\text{sample}} + M_{\text{spike}}$. After the addition of the additive, the isotopic ratio of the element changes to β_{mix}, and the value of this change can be used to calculate the boron content in the initial sample [14, 20].

For boron, which has two stable isotopes, ^{10}B and ^{11}B, the isotopic composition is defined as follows [21, 22]:

$$\beta_{10\text{B}} = \frac{S_{10\text{B}}}{S_{10\text{B}} + S_{11\text{B}}}, \quad \beta_{11\text{B}} = \frac{S_{11\text{B}}}{S_{10\text{B}} + S_{11\text{B}}}, \tag{1}$$

where S is the area of the peak corresponding to the signal of a particular boron isotope in mass spectrometric measurements. From Fig. 1, we can write the relationship

$$\beta_{\text{sample}} M_{\text{sample}} + \beta_{\text{spike}} M_{\text{spike}} = \beta_{\text{mix}} M_{\text{mix}}. \tag{2}$$

The calculations can be performed for any isotope, since $\beta_{10\text{B}} + \beta_{11\text{B}} = 1$.

If we use $M_{\text{mix}} = M_{\text{sample}} + M_{\text{spike}}$, then, from Eq. (2), we can obtain

$$M_{\text{sample}} = M_{\text{spike}} \frac{\beta_{\text{spike}} - \beta_{\text{mix}}}{\beta_{\text{mix}} - \beta_{\text{sample}}}. \tag{3}$$

If we use the ratio of isotopic composition values $R = S_{10\text{B}}/S_{11\text{B}}$ [17], then we can obtain the relationship between β and R,

$$R = \frac{\beta}{1 - \beta}.$$

Now, relationship (3) can be rewritten in the form

$$M_{\text{sample}} = M_{\text{spike}} \frac{1 + R_{\text{sample}}}{1 + R_{\text{spike}}} \frac{R_{\text{spike}} - R_{\text{mix}}}{R_{\text{mix}} - R_{\text{sample}}}. \tag{4}$$

Thus, if we add a known amount M_{spike}, then, by measuring only one variable, β_{mix} or R_{mix}, we can determine the unknown boron content in the sample, M_{sample}, from Eq. (3) or (4).

As the analyzed material (Sample), we used stainless steel with an increased boron content (1.6÷2.0% according to the sample passport) and the natural boron isotope ratio $^{10}\text{B} : {}^{11}\text{B} = 19.9 : 80.1$ ($\beta_{\text{mix}} \approx \approx 0.2$). As an indicator (Spike), we used elemental amorphous boron powder with the boron isotope ratio: $^{10}\text{B} : {}^{11}\text{B} = 95.0 : 5.0$ ($\beta_{\text{spike}} \approx 0.95$). The procedure for preparing samples for the study is described in detail in section 2.1.

2.3. Calibration standard method (external standard)

To quantitatively determine the boron content in a sample, the calibration standard method was used, with normalization by the sum of all alloy elements.

At the first stage, the mass spectrometer was calibrated using standard samples with a known elemental composition (High Purity Standards, USA). The elemental composition of the examined borated stainless steel included B, Al, Si, Ti, Cr, Mn, Fe, Ni, and Cu. Under specific conditions, the relative sensitivity was experimentally determined for each element of the standard. As a result, the dependences of the analytical signals of the corresponding peaks on the elemental content in the standard sample were obtained.

In Fig. 2, the values of the relative sensitivity coefficients are shown for all elements of the standard. For convenience, the maximum value (for Fe) was chosen as the reference, and all other values were normalized by it.

The analytical signals of the peaks of the elements in the analyzed sample (Sample) were determined under the same conditions as when measuring the calibration characteristic. The method of preparing samples for the study is described in detail in Section 2.1. Before starting the study of calibration solutions and samples, background values (Blank) were measured for selected elements.

Figure 3 demonstrates the intensity dependences of the isotopes ^{10}B (the orange curve) and ^{11}B (the blue curve) on the measurement time. The data for Blank are presented in Fig. 3, *a*, and for Sample in Fig. 3, *b*. Every histogram bin corresponds to the area under the peak for the selected isotope in the mass spectrum, the number of bins is equal to the number of iterations N, and the bin width is the interval δt for constructing the mass spectrum.

From Fig. 3, one can see that the ratio ^{10}B/^{11}B is the same and close to the natural one for both Blank and Sample. It can also be noted that the value for Blank was approximately 150 times smaller than for Sample. This fact testifies that the background signal of boron in Blank did not substantially affect the measurement results.

Similar distributions as in Fig. 3 were obtained for all sample elements. Those distributions were used to determine the arithmetic mean value for each element. In further calculations, the Blank values were subtracted from the corresponding Sample values. After in view of the tabular data on the distribution of elements and the relative sensitivity coefficient for a given measurement session (Fig. 2), the final value of the peak area S was obtained, and the content of each element in the sample was calculated.

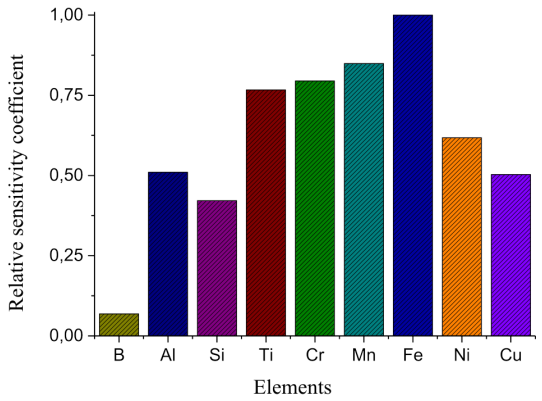

Fig. 2. Relative sensitivity coefficients of elements for the calibration standard solution

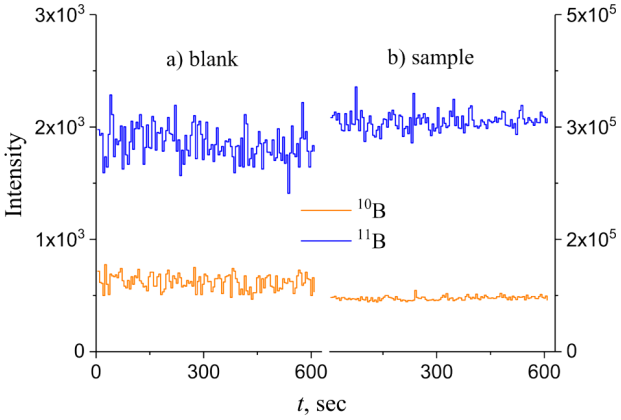

Fig. 3. Dependences of the intensities of the isotopes ^{10}B and ^{11}B on the measurement time t for Blank and Sample

For example, the value of the boron content C_{B} was determined using the expression

$$C_{\mathrm{B}} = \frac{S_{\mathrm{B}}}{S^{\mathrm{sum}}}, \tag{5}$$

where

$$S^{\mathrm{sum}} = S_{\mathrm{B}} + S_{\mathrm{Al}} + S_{\mathrm{Si}} + S_{\mathrm{Ti}} + S_{\mathrm{Cr}} + S_{\mathrm{Mn}} + S_{\mathrm{Fe}} + S_{\mathrm{Ni}} + S_{\mathrm{Cu}}$$

is the sum of the peak areas of all elements in the mass spectrum.

3. Experimental Results

3.1. Calculation of boron content using the isotope dilution analysis

In our study, the boron content was measured in 10 samples of borated steel. The same amount of

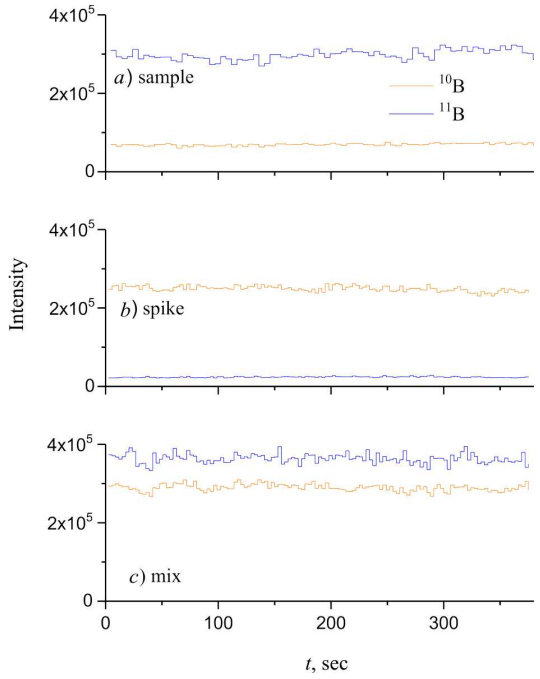

Fig. 4. Dependences of the intensities of the isotopes ^{10}B and ^{11}B on the measurement time t for the components Sample (a), Spike (b), and Mix (c) of the isotope dilution analysis

Table 1. **β-values for Sample, Spike, and Mix and boron content in examined steel samples**

Sample	β_{sample}	β_{spike}	β_{mix}	C_{B}, %
01	0.190	0.913	0.444	1.844 ± 0.085
02	0.191	0.913	0.454	1.748 ± 0.041
03	0.189	0.913	0.465	1.615 ± 0.062
04	0.188	0.913	0.464	1.624 ± 0.062
05	0.189	0.913	0.449	1.784 ± 0.099
06	0.191	0.913	0.443	1.882 ± 0.051
07	0.191	0.913	0.438	1.919 ± 0.096
08	0.190	0.913	0.461	1.675 ± 0.061
09	0.189	0.913	0.439	1.907 ± 0.041
10	0.190	0.913	0.451	1.773 ± 0.071

Spike was added to each sample (Sample), so there were 10 Mix samples. Thus, 10 combinations with three data sets for each sample (Sample, Spike, and Mix) were measured.

Figure 4 illustrates the intensity dependences of the peaks of isotopes ^{10}B (the solid histograms) and ^{11}B (the dotted histograms) on the measurement time for all IDA components (see Fig. 1) – Sample (Fig. 4, a), Spike (Fig. 4, b), and Mix (Fig. 4, c) – for one of the combinations. From the figure, one can see that the intensity dependences are linear, without any substantial fluctuations. The changes in the relative contribution of each isotope, ^{10}B or ^{11}B, in panels a, b, and c correspond to the logic of IDA (Fig. 1). The integral intensity value for each isotope was determined as the average over the entire measurement time.

The values of β_{sample}, β_{spike}, and β_{mix} were determined using formula (1). Although β_{sample} and β_{spike} have referenced values (0.199 and 0.95, respectively, they were also determined from experimental data in order to control the ratio of boron isotopes in the samples. The values of β_{sample}, β_{spike}, and β_{mix} determined for the isotope ^{10}B in 10 steel samples are quoted in Table 1 (columns 2 to 4, respectively).

It should be noted that the values experimentally obtained for β_{sample} and β_{spike} ($\sim$0.19 and $\sim$0.91, respectively) differ from the corresponding referenced values (0.199 and 0.95, respectively). This is a result of the fact that when determining the absolute value of β, it is necessary to take into account the relative sensitivity coefficient for the corresponding isotope, as was described in section 2.3. However, when determining the boron content C_{B} in the alloy using the IDA method, i.e., according to formula (3), this coefficient disappears, which makes it possible to neglect it in calculations. This is one of the advantages of the IDA method, which does not require the use of calibration standards.

At the next stage, formula (3) was applied to calculate the value of the boron content C_{B}. The calculation results are presented in the last column of Table 1. The errors are statistical, taking into account the mean square deviations for the distributions in Fig. 4. As one can see from Table 1, the boron content values fall within the limits specified in the manufacturer's passport for borated steel samples (1.6÷2.0%).

3.2. Calculation of boron content using the calibration standard method

The boron content C_{B} was also measured in the same 10 samples of borated stainless steel, but using the calibration standard method. The intensities were measured for all elements in the alloy, blank, and calibration solutions. The boron content was calculated using formula (5). The content of all elements included in the alloy could be additionally calculated using the same procedure. For example, for a cer-

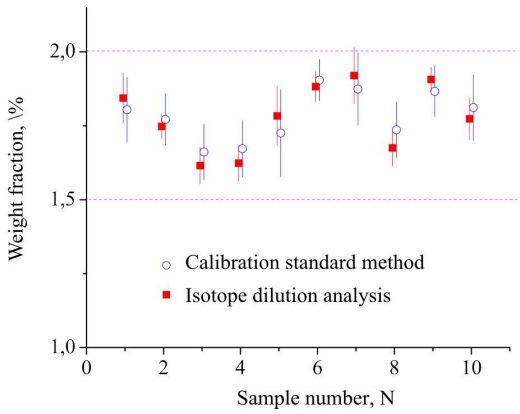

Fig. 5. Boron content in 10 steel samples

Table 2. **Boron content in steel samples obtained using the calibration standard method**

Sample	C_{B}, %
01	1.805 ± 0.109
02	1.772 ± 0.088
03	1.661 ± 0.095
04	1.673 ± 0.096
05	1.725 ± 0.147
06	1.905 ± 0.071
07	1.874 ± 0.123
08	1.736 ± 0.094
09	1.867 ± 0.087
10	1.812 ± 0.112

tain steel sample, the following values were obtained: 1.87% for B, 0.22% for Al, 0.31% for Si, 0.10% for Ti, 21.74% for Cr, 0.22% for Mn, 58.36% for Fe, 17.16% for Ni, and 0.02% for Cu.

Table 2 demonstrates the boron content values for all 10 steel samples obtained by this method. The errors are statistical. As can be seen, the boron content values also lie within the limits indicated in the manufacturer's passport for samples of borated stainless steel (1.6÷2.0%).

3.3. Comparison of the results obtained for the boron content in steel samples using two measurement methods

In Fig. 5, a comparison is made between the boron content values in the examined boron-containing stainless steel samples, which were obtained by the isotope dilution analysis (internal standard) and calculated on the basis of measurements using the calibration standards method (external standard). As was previously indicated, 10 samples of boron-containing austenitic stainless steel were studied in this work. The squares in the figure correspond to the results of the isotope dilution analysis, and the circles to the results obtained using the calibration solution method. The figure demonstrates that the data of both measurements have the same behavior and coincide within the error limits. A larger uncertainty in the measurements using the calibration standard method has to be noted. This fact is associated with a larger number of stages required to obtain experimental data by this method.

Thus, the data obtained using two methods are qualitatively and quantitatively consistent, which confirms the possibility of applying the isotope dilution analysis, making use of enriched boron to determine the boron content in borated stainless steel.

4. Conclusions

In this work, a method for measuring the boron content in stainless steel is presented, which is based on the application of an inductively coupled plasma mass spectrometer (ICP-MS) that allows internal calibration by means of the isotope dilution analysis. The analyzed material was stainless steel with an increased boron content and the natural boron isotope ratio $^{10}\mathrm{B}:{}^{11}\mathrm{B} = 19.9:80.1$. Elemental amorphous boron powder with the boron isotope ratio $^{10}\mathrm{B}:{}^{11}\mathrm{B} = 95.0:5.0$ was used as an indicator. The boron content was also determined by the calibration standard method (external standard).

The results of the boron content measurements using two methods (internal and external standards) were compared, and their qualitative coincidence was obtained for the entire series of samples. This fact evidences the efficiency of the isotope dilution analysis for determining the boron content in steel. An additional advantage of the method is its simplicity and speed as compared with the calibration standard method, since it does not require measurements for all alloy elements and has a smaller number of calculation stages, which reduces the error in determining the necessary data.

1. J.A. Evans, M.D. DeHart, K.D. Weaver, D.D Keiser. Burnable absorbers in nuclear reactors – A review. *Nucl. Eng. Design* **391**, 11176 (2022).
2. L. Desgranges, J.M. Escleine, P. Bienvenu *et al.* A new methodology for studying neutron absorber materials first

results with boron carbide. *Nucl. Instrum. Methods B* **432**, 42 (2017).
3. Aly Saeed. Developed borated austenitic stainless steel alloys as nuclear reactor control rods. *Nucl. Eng. Design* **413**, 112515 (2023).
4. S. Hafez, R.M. Elshazly, M.M. Eissa, S.U. El-Kameesy. High borated stainless-steel alloys for nuclear reactor domains. *Arab J. Nucl. Sci. Appl.* **54**, 97 (2021).
5. C. Kurta, L. Dorta, F. Mittermayr, K. Prattes *et al.* Rapid screening of boron isotope ratios in nuclear shielding materials by LA-ICPMS – a comparison of two different instrumental setups. *J. Anal. At. Spectrom.* **39**, 185 (2014).
6. J. Wang, Y. Cui, J. Bai *et al.* Effect of B addition on the microstructure and corrosion resistance of S31254 super austenitic stainless steels after solid solution treatment. *Mater. Lett.* **252**, 60 (2019).
7. A.A. Babenko, R.R. Shartdinov, V.A. Salina *et al.* The effect of boron on the properties and structure of austenitic stainless steels. *Metallurgist* **68**, 1032 (2024).
8. K.P. Jochum, B. Stoll, K. Herwig, M. Willbold. Validation of LA-ICP-MS trace element analysis of geological glasses using a new solid-state 193 nm Nd:YAG laser and matrix-matched calibration. *J. Anal. At. Spectrom.* **22**, 112 (2007).
9. N. Jakubowski. Analytical plasma ion sources for elemental mass spectrometry: where are we coming from—where are we going to? *Anal. At. Spectrom* **23**, 673 (2008).
10. D.V. Kutnii, D.D. Burdeynyi, S.A. Vanzha, N.V. Rud. Determination of ^{234}U/^{238}U, ^{235}U/^{238}U, ^{236}U/^{238}U isotope ratios in uranium oxide by sector-field ICP-MS. *East Eur. J. Phys.* **2**, 104 (2020).
11. J. Lin, Y. Liu, Y. Yang, Z. Hu. Calibration and correction of LA-ICP-MS and LA-MC-ICP-MS analyses for element contents and isotopic ratios. *Solid Earth Sci.* **1**, 5 (2016).
12. S.S. Kasana, O.P. Pandey. Effect of heat treatment on microstructure and mechanical properties of boron containing Ti-stabilized AISI-321 steel for nuclear power plant application. *Mater. Today Commun.* **26**, 101959 (2020).
13. P. De Bievre, H. Peiser. Basic equations and uncertainties in isotope-dilution mass spectrometry for traceability to SI of values obtained by this primary method. *Fresenius J. Anal. Chem.* **359**, 523 (1997).
14. P. Rodriguez-Gonzalez, J.M. Marchante-Gayon, J.I. Garcia-Alonso, A. Sanz-Medel. Isotope dilution analysis for elemental speciation: a tutorial review. *Spectrochim. Acta B* **60**, 151 (2005).
15. P. Rodriguez-Gonzslez, J. Ignacio Garcia Alonso. Recent advances in isotope dilution analysis for elemental speciation. *J. Anal. At. Spectrom* **25**, 239 (2010).
16. R. Hoelzl, C. Hoelzl, L. Kotz *et al.* The optimal amount of isotopic spike solution for ultratrace analysis by isotope dilution mass spectrometry. *Accred Qual Assur.* **3**, 185 (1998).
17. A. Quemet, A. Hubert, A. Gourgiotis *et al.* An isotope dilution mass spectrometry overview: tips and applications for the measurement of radionuclides. *J. Anal. At. Spectrom.* **39**, 1665 (2024).
18. J. Vogl. Characterization of reference materials by isotope dilution mass spectrometry. *J. Anal. At. Spectrom.* **22**, 475 (2007).
19. A.M. Gaffney. Guideline on isotope dilution mass spectrometry. *Lawrence Livermore National Laboratory, LLNL-TR-731685* (2017).
20. J. Vogl, W. Pritzkow. Isotope dilution mass spectrometry – A primary method of measurement and its role for RM certification. *Mapan – Journal of Metrology Society of India* **25**, 135 (2010).
21. P.P. Coetzee, L. Greeff, F. Vanhaecke. ICP-MS measurement of $^{11B}/^{10}$B isotope ratios in grapevine leaves and the investigation of possible boron isotope fractionation in grapevine plants. *S. Afr. J. Enol. Vitic.* **32**, 28 (2011).
22. J.K. Aggarwal, K. Mezger, E. Pernicka, A. Meixner. The effect of instrumental mass bias δ^{11}B measurements: A comparison between thermal ionization mass spectrometry and multiple collector ICP-MS . *Intern. J. Mass Spectr.* **232**, 259 (2004).

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МЕТОД ІЗОТОПНОГО РОЗБАВЛЕННЯ ДЛЯ ВИЗНАЧЕННЯ ВМІСТУ БОРУ В НЕІРЖАВНІЙ СТАЛІ З ВИКОРИСТАННЯМ ICP MS

Для визначення вмісту бору в зразках корозійностійкої хромонікелевої неіржавної сталі, легованої бором, запропоновано використання методу ізотопного розбавлення, який є методом внутрішнього стандарту. Дослідження виконано з використанням мас-спектрометра з індуктивно-зв'язаною плазмою на 10 зразках неіржавної сталі, легованої бором, з природним співвідношенням ізотопів бору. Як внутрішній стандарт використовували порошок елементарного аморфного бору зі збагаченням за ^{10}B до 95,0%. Для порівняння додатково проведено мас-спектрометричне вимірювання вмісту бору в цих самих зразках за допомогою методу калібрувальних розчинів (метод зовнішнього стандарту). В цьому випадку вміст бору визначався як відношення інтенсивності бору до сумарної інтенсивності всіх елементів зразка. Встановлено, що результати визначення вмісту бору якісно співпадають для обох методів визначення (внутрішній та зовнішній стандарти), але можна зробити висновок про те, що метод ізотопного розбавлення є більш точним методом визначення вмісту елементів. На результати такого аналізу не впливають неспектральні інтерференції, які пов'язані з дрейфом чутливості приладу з часом і матричними ефектами, оскільки вимірюються не абсолютні концентрації ізотопів, а їх співвідношення.

Ключові слова: метод ізотопного розбавлення, мас-спектрометр, борована неіржавна сталь, ізотопи бору.