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Determination of boron concentration in uranium fuel samples by ICP-OES following a separation step by cation exchange resin

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Abstract The boron content of uranium fuel samples with boron concentrations in the range of 0.05–10 μg/g was determined using inductively coupled plasma optical emission spectrometry (ICP-OES) after the uranium was separated by cation exchange. The samples were dissolved in 3 M HNO_3 on a hot plate at 150 °C and evaporated to near dryness. The residues were redissolved in 0.2 M HNO_3 and passed through a column loaded with Dowex 50WX8-400 resin. Uranium was adsorbed on the resin, while boron was easily eluted with 0.2 M HNO_3. The boron content of the effluent was determined using ICP-OES. Several strategies were employed to improve the reliability of the experimentally determined boron content. The addition of mannitol and proper control of the evaporation process were shown to be effective in preventing boron loss during sample dissolution and evaporation. The memory effect was eliminated by flushing the system with 1.5% ammonia for 30 s between successive sample runs, and the matrix match method was used to eliminate the matrix effect arising from mannitol during the ICP-OES analysis. The accuracy of the results of the analysis was determined by addition recovery tests and by comparison with the results of three Chinese certified reference materials (GBW04242, GBW04243, and GBW04232). Using the method we developed, the limit of detection for boron was as low as 0.05 μg/g in uranium fuel samples, and the relative standard deviations for 0.1–0.5 g uranium samples with 0.05–2 μg/g of boron were within 9%.

Keywords Boron determination · ICP-OES · Boron evaporation · Memory effect · Matrix match method · Resin separation

1 Introduction

Boron plays an important role in the nuclear power industry because of its high neutron absorption cross section, which can cause structural damage to reactor materials and affect the neutron economy if the boron content exceeds established values. Therefore, boron is one of the most concerning impurities in all nuclear materials [1]. In nuclear technology, the specifications for boron are very stringent as a part of quality assurance, and its determination at μg/g levels is of great importance. Several methods have been reported for the determination of boron content. These methods include spectrophotometry [2, 3], atomic absorption spectrometry [4–6], inductively coupled plasma atomic emission spectroscopy (ICP-OES) [7–9], mass spectrometry [10–12], and nondestructive methods such as the neutron activation analysis method [13, 14]. Of these, the most commonly employed technique is ICP-OES. Before ICP-OES analysis, nuclear fuels must be dissolved in HNO_3 or a mixture of HNO_3 and HF, followed by removal of the matrix components.

The challenges in the identification of boron in uranium fuels arise from the three steps involved: sample dissolution and acid volatilization, separation of boron from the uranium matrix, and ICP-OES analysis. Throughout the entire process, potential contamination in the laboratory complicates the evaluation of boron content [15]. Additionally, it is possible for boron to be highly volatilized above 70 °C in acid solutions [16]. To suppress boron loss,

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mannitol has been added to form less volatile mannitol–boron acid complexes [17, 18]. However, acid treatment can affect mannitol or its boron-complexing action in such a manner as to cause an enhancement of the intensity of the boron line, as reported by Wenzel and co-workers [19]. Furthermore, other authors reported that mannitol did not completely suppress boron volatilization in some cases, particularly when evaporation was continued to dryness [20].

Determination of the boron content in uranium fuels is seriously affected by matrix-based interference, because uranium emits thousands of lines in an inductively coupled plasma source [21]. The sensitive line of B at 249.677 nm overlaps with a U line at 249.720 nm, while the less sensitive line of B at 249.772 nm overlaps with a U line at 249.883 nm. Geyer et al. [22] reported the strong influence of high uranium content on boron analysis using emission spectroscopy. Researchers reported that boron could be separated from a uranium-based matrix by a variety of methods, such as solvent extraction using 2-ethyl-1, 3-hexane diol (EHD) in chloroform [23], distillation as methyl borate [24], and cation exchange separation [19]. During instrumental analysis, the determination of boron content is significantly affected by the memory effect. Several washing agents, such as sodium fluoride solutions, mannitol, triton X-100 surfactant, and ammonia, have been tested to minimize this effect [25–29].

Sample dissolution and acid volatilization are important steps of sample preparation during which loss of boron can occur. Separation of boron from the uranium matrix and elimination of the memory effect during ICP-OES are also crucial for the determination of boron content. The aim of this work is to establish a new procedure that can improve the accuracy of boron content determination by minimizing effects such as boron loss, spectral interference, and the memory effect. In this report, we establish a new method based on well-developed techniques to improve boron content determination results in uranium fuel samples. This new method was validated using three Chinese certified reference materials (GBW04242, GBW04243, and GBW04232), which are standard uranium samples containing known amounts of boron. A detailed statistical assessment, including the relative standard deviation and detection limit, was also applied to demonstrate the accuracy of this method.

2 Experimental

2.1 Samples and reagents

The chemicals and reagents used in our experiments were of the highest purity available. Ultra-pure water obtained from a purification system (Millipore, USA) was used to prepare solutions. Standard solutions of boron and uranium were prepared by serial dilution of 100 mg/L standards (AccuStandard, New Haven, USA) with 0.2 M HNO_3. HNO_3 (65%, extra pure) was purchased from Merck Chemicals (Darmstadt, Germany). Certified reference samples of U_3O_8 (GBW04242, GBW04243, and GBW04232, Baotou, China) were used for quality control. DOWEX 50WX8-400 was purchased from Sigma (St. Louis, MO, USA), and the other chemicals (mannitol, ammonia, NaOH, and HCl) used were of analytical grade.

2.2 Instrumentation

A hot plate (LabTech EH45 C, China) was used for sample dissolution. The ICP-OES measurements were taken using a dual-view Optima 8000 inductively coupled plasma optical emission spectrometer (PerkinElmer, Norwalk, CT, USA). The axial viewing position of the torch was used for the measurement of boron. The sample was introduced into the ICP-OES using a cross-flow nebulizer with a HF-resistant spray chamber. The ICP-OES operating parameters are listed in Table 1.

2.3 Column preparation

A polyacrylic column (internal diameter: 9–9.5 mm; filling height of resin: 8 cm) with a Teflon stopcock was used in our experiments. The resin in H^+ form (Dowex 50WX8-400, 200–400 mesh) was immersed in deionized water for 48 h. The column was then packed with 5 mL (wet-settled volume) of resin to reach an 8 cm resin bed height. To remove any trace impurities, the loaded column was successively cleaned with 20 mL of 1 M HCl and 1 M NaOH. Between acid and alkaline cleanings, water was passed though the resin to neutralize the column. The column was equilibrated with 50 mL of 1 M HNO_3 and then 150 mL of 0.2 M HNO_3 immediately before sample introduction. In the boron and uranium separation

Table 1 ICP-OES parameters

Parameters	Values
RF generator power (W)	1300
Nebulizer gas flow rate (L/min)	0.55
Auxiliary gas flow rate (L/min)	0.2
Plasma gas flow rate (L/min)	15
Analytical wavelength (nm)	B 249.772 U 409.014
Sample uptake (mL/min)	1.5

experiments, the flow rate through the column was adjusted to approximately 0.3–0.5 mL/min.

2.4 Proposed sample preparation method

The precisely weighed uranium fuel samples (0.1–0.5 g) were transferred into PFA beakers. 5 mL of 3 M HNO_3 and 500 µL of mannitol solution (5 mg/mL) were added to each beaker. The samples dissolved quickly, and the solutions were then evaporated to near dryness on a hot plate at 150 °C. Heating was stopped when only one drop of solution remained. The residue was recovered with 2 mL of 0.2 M HNO_3. Each of the thus-obtained solutions was transferred to a Dowex 50WX8-400 column. The column was washed with 12 mL of 0.2 M HNO_3 when the level of the solution reached the top of the resin bed. The first 2 mL was discarded, and then the remaining 10 mL of eluent was collected and analyzed using ICP-OES. The same procedure was also carried out for a blank solution. Boron standard solutions with a volume of 10 mL and concentrations in the range of 5–100 ng/mL were prepared by diluting a 1 µg/mL stock solution. 25 mg of mannitol was added to each solution.

3 Results and discussion

3.1 Memory effect of boron

The memory effect most likely arises from the fact that the inside surface of the spray chamber of the sampling system is covered with boric acid volatilized from liquid sample droplets. Aqueous ammonia can apparently convert volatile boric acid to nonvolatile ammonium borate. In this experiment, 1.5% ammonia (*v*/*v*) was introduced between successive runs. The boron content of a solution containing 1 mg/L B in 0.2 M HNO_3 was determined 10 times in succession. Between two runs, a blank solution of 0.2 M HNO_3 was nebulized immediately after each successive run for 60 s and the B signal was recorded. For the other group, the system was first flushed with deionized water for 15 s, washed with 1.5% (*v*/*v*) ammonia for 30 s and with water again for 15 s, and then the B signal of the 1 mg/L solution was recorded. The variation of the intensity of the B signal between the two groups with and without the ammonia flush procedure is shown in Fig. 1. When the system was flushed with 0.2 M HNO_3 between successive runs, the intensity increased obviously between the first run and the tenth. For the other group, in which the system was flushed with 1.5% ammonia between each successive run, the variation in the intensity was smaller, with a relative error of only 0.8%. This experiment demonstrated that the memory effect could be eliminated through the use of an ammonia wash between successive runs. A precise result could be obtained due to the release of ammonia gas during the 30-s nebulization period. The ammonia reacted with the volatile boric acid on the inside surface of the spray chamber to form nonvolatile ammonium borate [29].

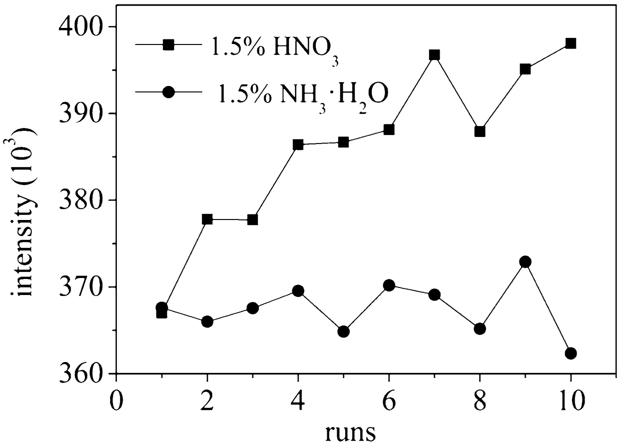

Fig. 1 Variation of boron intensity using flushing with $NH_3 \cdot H_2O$ or HNO_3 between successive runs (boron concentration: 1 mg/L)

3.2 Effect of mannitol addition on the volatilization of boron during the evaporation of the HNO_3 solution

It is well known that boron can easily evaporate at temperatures higher than 70 °C. Samples in the solid state must first be dissolved under heating prior to boron determination. Unfortunately, boron is easily volatilized during this process, especially when the sample is evaporated to near dryness, which limits the pretreatment of solid boron samples. The addition of mannitol suppresses boron volatilization due to the formation of the less volatile mannitol–boron acid complex. In addition, it has been reported that boron volatilization in solutions of water, hydrochloric acid, and nitric acid can also be effectively suppressed via the addition of mannitol during the evaporation process [8, 30].

In this experiment, a boric acid solution containing 20 ng/mL of B and 5 g/L of mannitol was prepared. A series of 5 mL aliquots of this solution containing 0.1 µg of boron (9.09 nmol) were transferred to 25-mL beakers made of PFA, followed by the addition of various amounts of mannitol solution to obtain mannitol-to-boron mole ratios ranging from 0 to 1000. Subsequently, 5 mL of 3 M HNO_3 was added to each sample, and the prepared solutions were then evaporated at 150 °C. All the samples were evaporated to near dryness within 1 h. To avoid extra heating, the heating time was fixed as 1 h. After this time, the residues were recovered by the addition of 5 mL of 0.2 M HNO_3 for boron determination. The minimum detectable concentration of boron using this procedure was as low as 5 ng.

The recovery yields of boron after evaporation of the 3 M HNO_3 solutions containing various amounts of mannitol were investigated. As can be seen in Table 2, the recovery ratio increased from 62.38% (no mannitol) to more than 90% (mannitol/boron mole ratio > 3). This is very similar to the results obtained by Ishikawa et al. [30], who reported an increase in the recovery yield from 9 to 90% for mannitol-to-boron mole ratios ranging from 0 to 1 in 3 M HF solution.

As can be seen in Fig. 2, the recovery yields remained constant for mannitol-to-boron mole ratios in the range of 4–100. In real samples, other elements, such as Ti, Ge, Sn, and Sb, might also complex with mannitol [31]. Therefore, an excess of mannitol should be added if possible. In our experiment, 25 mg of mannitol was added.

3.3 Influence of mannitol on ICP-OES measurements

The influence of the addition of mannitol on the results of the ICP-OES analysis was also investigated. A 20 ng/mL solution of boron in 0.2 M HNO_3 was prepared. Solutions (10 mL each) containing various amounts of mannitol in the range of 0–25 mg were tested. After the mannitol was dissolved and mixed into the solution, the solutions were sampled for ICP-OES analysis. The intensity of the boron signal was recorded for each sample. As can be seen in Table 3, the intensity of the boron signal depended on the amount of mannitol added, indicating that the boron emission intensity was enhanced by the addition of mannitol. The increase in signal intensity can be attributed to the addition of mannitol, which plays a role not only in forming boron–mannitol complexes, but also as a physical carrier. Similar results have been reported previously [32].

Table 2 Recovery yields of boron (9.09 nmol) from 3 M acid solutions during the evaporation process

Mannitol nmol	Mannitol:B mole ratio	nmol	%
0	0	5.67	62.38
4.55	0.5	7.65	84.16
9.09	1.0	7.88	86.69
13.64	1.5	8.50	93.51
18.18	2	8.10	89.11
27.27	3	8.62	94.83
36.36	4	9.42	103.6
45.45	5	9.38	103.2
90.9	10	9.25	101.8
181.8	20	9.14	100.5
454.5	50	9.36	103.0
909.0	100	9.22	101.4

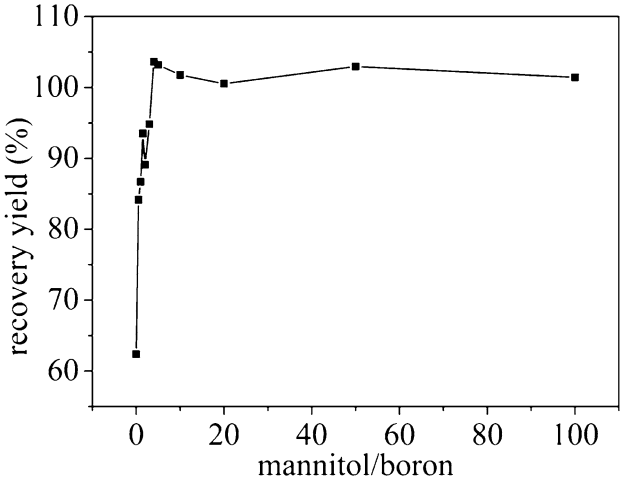

Fig. 2 Recovery yields of boron in 3 M HNO_3 for different mannitol-to-boron mole ratios (boron content: 9.09 nmol, heating temperature: 150 °C, and heating time: 1 h)

Table 3 Effect of mannitol on boron signal intensity during ICP-OES analysis at a boron concentration of 20 ng/mL

Mannitol (mg)	Intensity of boron signal
0	7659
5	8763
10	9168
15	9280
20	9457
25	9733

The matrix effect caused by mannitol can be eliminated using matrix-matched standards for quantification during ICP-OES analysis. Therefore, in this experiment, an equivalent amount of mannitol was added to the standards to compensate for the difference between samples and standards.

3.4 Resin adsorption capacity and elution curve of boron

In order to determine the boron content of uranium fuel using ICP-OES, the uranium must first be separated. In order to investigate the adsorption of uranium on resin, a dynamic column adsorption experiment was performed. 200 mL of a solution containing 8.5 g/L of uranium in 0.2 M HNO_3 was added to the column continuously. The solution was passed through the column loaded with 8 cm of Dowex 50WX8-400, and each 5 mL of solution was collected as a sample for analysis. The ratio of the uranium concentration between the effluent and the influent (C/Co) and the volume of the effluent solution were plotted for the breakthrough curves. Before measuring the uranium content using ICP-OES, the solutions were diluted appropriately. As can be seen in Fig. 3, no uranium was detected in the effluent solution at the beginning of the experiment. As more solution was added to the column, uranium was detected in the effluent, and the uranium concentration then quickly became as high as that of the influent. This is because the Dowex 50WX8-400 resin adsorbed uranium effectively, and after the binding sites were saturated, a decrease in uranium removal resulted [33]. The column did not become saturated until about 150 mL of uranium-ion-containing solution had passed through the column. Our results confirmed that the Dowex 50WX8-400 resin was suitable for uranium adsorption. In this experiment, uranium was not detected in the first 115 mL of effluent, but after this the concentration of uranium began to increase sharply. From the volume of effluent at which uranium breakthrough was observed, the maximum sorption attained for separation of boron and uranium was calculated to be 977.5 mg. Therefore, for this method, the weight of uranium should not exceed 977.5 mg.

Uranium adsorbed onto the resin, while boron was easily eluted using 0.2 M HNO_3. To determine the volume of effluent to be used, a boron elution curve was studied. The influent contained 0.2 ng of boron and 0.5 g of uranium in 2 mL of 0.2 M HNO_3. Figure 4 shows that boron was not detected in the first 2 mL of the effluent solution from the column. The boron concentration then increased sharply during the next two intervals and decreased to zero in the fifth interval. Uranium was not detected in the effluent. In order to elute boron completely, the first 2 mL was discarded, and then the subsequent 10 mL of effluent was collected for analysis.

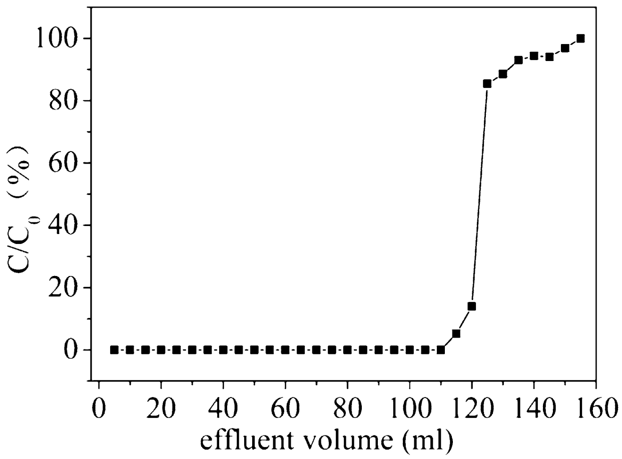

Fig. 3 Breakthrough curve for the adsorption of uranium (uranium concentration C_0: 8.5 g/L. Each 5 mL of effluent was collected as a sample for the analysis of the uranium concentration C)

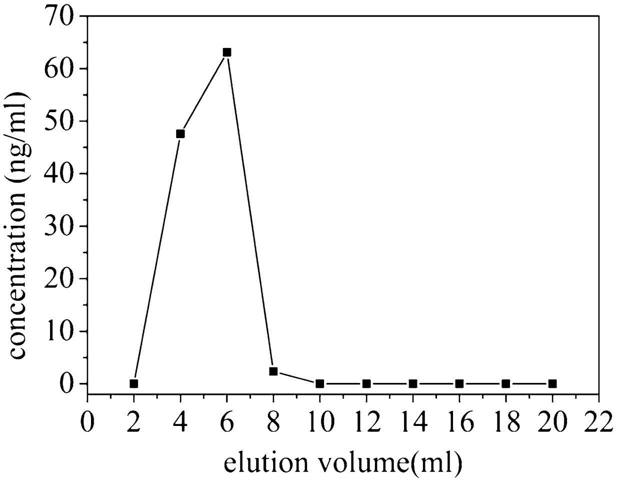

Fig. 4 Boron elution curve (boron concentration of influent: 100 ng/mL, influent volume: 2 mL, and eluent: 0.2 M HNO_3. Each 2 mL of effluent was collected as a sample for boron concentration analysis)

3.5 Analysis of the samples after separation of boron from the uranium fuel samples using cation exchange separation

In low-concentration nitric acid aqueous solutions, boron exists in the form of boric acid. When mannitol is added, it forms a series of mannitol–boric acid complexes, such as $[BL]^-$, $[BLB]^{2-}$, and the more polymerized $[BL]^{n-}$, where B and L are boron and mannitol, respectively [17]. Due to their negatively charged character, boron complexes can be eluted after passing through a cation exchange column, while the uranium matrix remained on the cation exchange resin DOWEX 50WX8-400 at low concentrations [34, 35]. Based on this principle, boron and uranium were separated from each other. In this experiment, various known amounts of boron were added to several uranium samples and pretreated according to the procedure outlined above. Uranium was adsorbed on the column, and boron was eluted with 0.2 M HNO_3 and collected for analysis by ICP-OES. The results were compared to the amount of boron added. The data for three samples with different boron concentrations are shown in Table 4. A relative standard deviation of less than 9% for the analysis of uranium samples with 0.1–1 μg boron was obtained using the method we established. The limit of detection (LOD) was calculated as 3 times the standard deviation of the blank and the limit of quantification (LOQ) as 10 times the standard deviation of the blank [36]. We found an LOD of 0.05 μg/g and an LOQ of 0.17 μg/g.

Table 4 Determination of boron added to uranium solutions ($n = 3$)

Boron added (μg)	Sample (g)	Boron found (μg)	Recovery yield (%)
0	0.3275	Not detected	–
0.1	0.3152	0.092 ± 0.003	92 ± 3.2
0.5	0.3152	0.47 ± 0.02	94 ± 4.2
1	0.3193	0.93 ± 0.02	93 ± 2.1

Table 5 Determination of B in different certified reference materials ($n = 3$)

Sample	ICP-OES (μg/g)	Certified value (μg/g)
GBW04232	1.24 ± 0.11	1.19 ± 0.08
GBW04242	0.22 ± 0.02	0.26
GBW04243	0.52 ± 0.02	0.54

The boron contents of three Chinese certified reference standards were determined using this procedure. As shown in Table 5, the data were in agreement with the certified values for all of the materials evaluated. The relative standard deviations ranged between 4 and 10%. Therefore, this determination method can be considered satisfactory for this element.

4 Conclusion

The proposed method, which is based on the prior separation of boron from uranium using a cation exchange technique, gives a precise, sensitive analytical procedure for the determination of boron content in uranium fuel using ICP-OES. From the experimental results, it can be concluded that: (1) the addition of mannitol and proper control of evaporation are effective in preventing boron loss during sample dissolution. (2) DOWEX 50WX8-400 resin is a good ion exchanger which allows the adsorption of uranium ions and the convenient elution of boron during the separation stage. (3) Ammonia flushing between successive runs can eliminate the memory effect, and matrix matching can be used to minimize the enhancement of the boron line intensity caused by mannitol during ICP-OES analysis.

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