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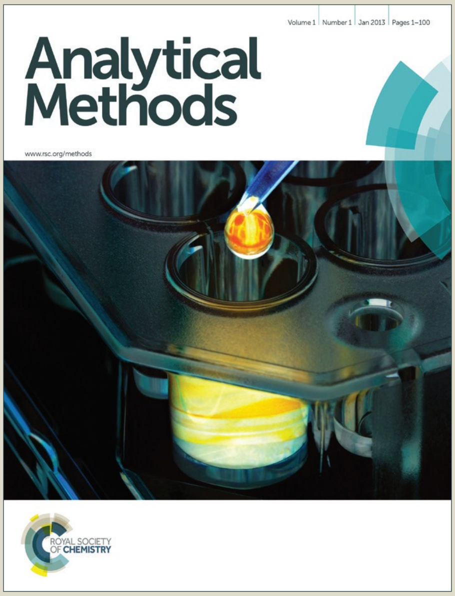

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Efficient separation of boron using solid-phase extraction for boron isotope analysis by MC-ICP-MS

Maoyong He[1, 2, 3*], Zhangdong Jin[1], Hai Lu[2], Tongxiang Ren[2]
[1] State Key Laboratory of Loess and Quaternary Geology, Institute of Earth Environment, Chinese Academy of Sciences, Xi’an 710061, China
[2] Division of Chemical Metrology & Analytical Science, National Institute of Metrology, Beijing 10013, China
[3] Shaanxi Provincial Key Laboratory of Accelerator Mass Spectrometry Technology and Application, Xi’an 710061, China

Abstract In nature, boron presents a large range of isotopic compositions (up to 150‰), making it as one of powerful geochemical tracers. How to improve the separation and purification procedures of boron in complex matrices and low trace boron contents is a challenge to obtain high-precision and accurate boron isotope ratios. A method of ion exchange using solid-phase extraction (SPE) for efficient separation of boron from samples with low boron contents was developed for determining boron isotopes by MC-ICP-MS. The results showed that the total amount of residual boron in solution after the ion exchange decreased as the vacuum pressure decreased, from 60 µg at a vacuum pressure of 0.09 MPa to 0 at a vacuum pressure of 0.04 MPa. When vacuum pressure was between 0.008 to 0.04 MPa, boron was not detected in the solution. The flow rate of solution increased as the vacuum pressure increased, from 50 µL/min at a vacuum pressure of 0.008 MPa to 1358 µl/min at a vacuum pressure of 0.09 MPa, and the volume of eluent required increased from 300 µl at 0.008 MPa to 600 µl at 0.04 MPa. Then the vacuum pressure was recommend below 0.04 MPa based on these results. The $\delta^{11}B$ values of NBS 951 after chemically treatment using the SPE were between -0.014 ‰ and 0.016 ‰. Using this procedure, boron in river water, rainwater and seawater samples was separated and analyzed their B isotope compositions by MC-ICP-MS. The accuracy and precision of our method was assessed by comparing measured these samples $\delta^{11}B$ values to published results using positive ion thermal ionization mass spectrometry. The improved efficient

*Correspondence authors.
E-mail address: hemy@ieecas.cn (M. -Y. He).

separation of boron with a low boron content using SPE is applicable to a wide range of boron isotopic separation applications.

Keywords: separation, boron, ion exchange, solid phase extraction, MC-ICP-MS

1. Introduction

Boron has two stable isotopes, ^{10}B (19.9 %) and ^{11}B (80.1%) [1]. The large relative mass difference between the two isotopes results in significant boron isotopic fractionation in nature, ranging from -70‰ to +75‰ [2]. In addition, boron has only a single valence and no redox reactions in nature. These characteristics make B isotopes useful for sensitive tracer applications, including the interactions between fluids and minerals from the surface to the mantle [3-9].

With the development and improvement of boron isotopes theory and isotope analysis technology, the separation and purification of boron isotope with complex matrices and low trace boron contents continue to improve, satisfying the requirements for high-precision and accurate measurement of boron isotope ratios.

Before each boron isotope analysis by the mass spectrometry, the samples were chemically purified to avoid a matrix effects. The extraction of boron from aqueous samples is commonly accomplished using the ion exchange resin Amberlite IRA 743 [10-15]. A typical volume of 1 ml of resin is used for the extraction of a few micrograms of boron. With the development of boron isotope testing technology, the amount of boron required becomes increasingly smaller, along with corresponding reductions in the size of the ion exchange column and the amount of resin. The amount of resin now required is approximately 50 μl. In this case, the solution separated was unlikely to pass through the ion exchange resin by autogenous gravity. Thus, the use of a peristaltic pump coupled with an ion exchange micro-column for extraction of boron was developed [16-19]. The emergence of this method significantly expanded the scope of application of boron isotopes. However, with the experiment, it was found that the PVC tubing on the peristaltic pump could be resulting in isotopic shifts derived from pre-treatments between different samples, although the nitric acid and pure water were used to wash the tubes.

The Solid-phase extraction (SPE) procedures is based upon liquid - solid phase chromatography theory. It is a method to enrichment, separation and purification sample using selective adsorption and selective elution. It is a physical extraction including liquid and solid phase process [20-21].

The main objective of this study is to improve the efficient separation of boron from rainwater with a low boron content using SPE techniques and isotope analysis by MC-ICP-MS. The factors that may affect the separation efficiency and thereby affect the accuracy of isotope ratio include the pump pressure (affecting the proportion of recovered boron), velocity of flow, elution volume, and the δ^{11}B values of the NBS 951 after chemically treatment by SPE, and these factors were investigated to obtain optimum conditions. The present method is applicable to a wide range of boron isotopic separation applications with low boron content.

2. Instruments and reagents

2.1 Instrumentation

Boron contents were measured on a PE ICP-MS (Perkin Elmer-Nexion 300D, PerkinElmer Corporation, USA) at the Division of Chemical Metrology & Analytical Science, National Institute of Metrology and the State Key Laboratory of Loess and Quaternary Geology, Institute of Earth Environment in Xi'an, Chinese Academy of Sciences (IEECAS). The machine was fitted with a PFA concentric nebulizer and a quartz double-pass spray chamber. Nickel sampler and skimmer cones were used.

Isotopic measurements were performed on a NEPTUNE Plus MC-ICP-MS (Thermo Fisher Scientific, Germany) at the State Key Laboratory of Loess and Quaternary Geology, Institute of Earth Environment in Xi'an, Chinese Academy of Sciences (IEECAS). This instrument is a double-focusing magnetic sector instrument with variable dispersion (17%) ion optics. Nine Faraday cups are installed on the instrument, with eight of them on two sides of the center cup.

A designed SPE system was used to separate boron isotope (Fig. 1). The equipment included three primary sections: a SPE device, an ion exchange column, and auxiliary equipment. The SPE device consists of four components: a lid cover (Fig. 1A-1), an air chamber (Fig. 1A-2), a pressure gauge (Fig. 1A-3), and a vacuum port with gauge that is used to control the vacuum applied to the chamber (Fig. 1A-4). The ion exchange column was a PFA mini-column (Savillex, I.D. = 2.4 mm) (Fig. 1A-a). Auxiliary equipment included a waste tank (Fig. 1A-b), a sample collection tube (Fig. 1B-c), and the bracket for the sample tube (Fig. 1B-d). The height of the apparatus was 15 cm, and the diameter of it was 20 cm. The SPE device was made using transparent poly (methyl methacrylate) (PMMA). PTFE Thread Seal Tape was used for sealing of columns.

2.2 Reagents

Nitric acid (HNO_3, guaranteed reagent (GR)), acetic acid, sodium acetate, NH_3•H_2O and ultrapure water (18.2 M Ω) were utilized in the study.

The standard material for the B isotope analysis, NBS951, was obtained from the National Institute of Standard and Technology (NIST, 100 Bureau Dr., Gaithersburg, MD 20899, USA). The H_2O used in the experiment was purified with a Milli-Q system at a resistance of 18.2 MΩ. The HNO_3 and NH_3•H_2O used in the experiment was purified with a DST-1000 sub-boiling distillation device (Savillex Co., 10321 West 70th Street Eden Prairie, MN 55344-3446, USA). All experiments were performed in an ultra-clean chemical laboratory.

3. Experimental methods

3.1 Separation of B with an ion-exchange process

B in samples was separated from other cations using SPE. The Amberlite IRA-743 B-selective resin (Sigma Co.) was crushed to 100-200 mesh [18, 22, 23]. A resin volume of 50μL was packed in a PFA mini-column (Savillex, I.D. = 2.4 mm) and the flow rate was controlled at approximately 50 μl min^{-1} by a vacuum pumping system. The detailed methods of the experiment are listed Table 1 [24].

3.2 The factors affect the separation efficiency

In this paper, the pump pressure (affecting the separation efficiency of boron), elution volume and the $\delta^{11}B$ values of the NBS 951 after chemically treatment by SPE experiments were carried out.

3.2.1 The effect of pump pressure on the separation efficiency of boron

To study the influence of pressure on the separation efficiency of boron using Amberlite IRA-743 B-selective resin, a series of boric acid solutions, containing the same boron concentration (500 μg), was passed through the column at different pressures (from 0.008 to 0.09 Mpa). During the experiment, the load sample was sampled at an interval of 50 μl. The solution passed from the resin which selectively adsorbed B was held in a container, and the boron concentration in solution was analyzed by ICP-MS. The boron in solution was calculated as the proportion of recovered boron.

3.2.2 The effect of pump pressure on the elution volume

Ion exchange efficiency was controlled by the vacuum pressure. The volume of elution solution was also susceptible to the vacuum pressure. To study the impact of pressure on elution volume, a series of boric acid solutions, containing the same boron concentration was adsorbed by Amberlite IRA-743 B-selective resin. Then, the boron in the resin was eluted with 2% HNO_3 at a different pressure (from 0.008 to 0.04 Mpa). During the experiment, the eluted solution was sampled at an interval of 50 μl. The boron concentration was analyzed by ICP-MS.

3.3 Measurements of the B concentrations and isotope ratios

The elemental analysis of B was performed using a PerkinElmer NexIon 300D quadrupole ICP-MS equipped with a SC Fast peristaltic pump. The system was operated in standard mode, monitoring isotopes m/z 10 and 11 for B with a dwell time of 50 ms and integration time of 1 s. The quantification was performed by external calibration with boron standards for ICP (Sigma-Aldrich). The reported values were the average result of 5 measurements.

All B isotope measurements were performed on a NEPTUNE Plus MC-ICP-MS (Thermo Fisher Scientific, Germany), which enables a static measurement of m/z 10 and m/z 11 on Faraday cups[17-19, 25, 26]. The operating parameters are given in Table 2. The total procedural blanks from the ion-exchange procedure is in the range of 0.38 ± 0.04ng (2RSD) as determined by ID-MS. The average signal intensity of ^{11}B was 0.7 V.

4. Results and discussion

4.1 The effect of pump pressure on the separation efficiency of boron

To study the influence of pressure on the separation efficiency of boron, the boron in solution passed from the resin was analyzed by ICP-MS. The total amount of residual boron in solution after the ion exchange was described in Fig. 2. The results show that the total amount of residual boron in solution decreased as the vacuum pressure decreased, from 60μg at a vacuum pressure of 0.09 MPa to 0 at a vacuum pressure of 0.04 MPa. When vacuum pressure was between 0.008 to 0.04 MPa, boron was not detected in the solution. This result was indicated that the boron in the loaded samples was completely adsorbed by resin at this time. The residence time of the sample loaded was associated with vacuum pressure, if the vacuum pressure was higher, the residence time of the loaded sample was shorter, and the B in the solution was not adsorbed by the resin and it was taken away. Figure 2 also shows that the flow rate of solution was correlated with vacuum pressure. The flow rate of solution increased as the vacuum pressure increased, from 50 μl /min at a vacuum pressure of 0.008 MPa to 1358μl/min at a vacuum pressure of 0.09 MPa. Therefore, from what has been discussed above, the pump pressure should be less than ≤ 0.04 MPa, where the B was completely adsorbed by the B-selective resin in our experiment.

4.2 The effect of pump pressure on elution volume

Pump pressure not only affected the separation efficiency, also affected the elution efficiency. From Figure. 3, it can be observed that the volume of elution was larger when the vacuum pressure was larger. The volume of elutent was increased as required from 300 μl at 0.008 MPa to 600 μl at 0.04 MPa.

$B(OH)_4^-$ was adsorbed by Amberlite IRA-743 resin, and it was difficult to eluted by alkali. However, boron could be eluted by any acid solution from the resin due to the $B(OH)_4^-$ would be converted to $B(OH)_3$ which did not adsorbed by resin. When the vacuum pressure was higher, HNO_3 was outflowed quickly through the resin column and did not have enough time to exchange boron. At this circumstances, the boron was completely eluted need more HNO_3. In contrast, HNO_3 had adequate time to exchanged boron adsorbed on resin at the small pressure pump.

4.3 The $\delta^{11}B$ values of the NBS 951 and seawater after chemical treatment using ion exchange SPE

Boron isotope fractionation was detected when B was not completely recovered in the process of separation and purification. During the elution process, ^{11}B preferentially leached out, and ^{10}B tended to be retained in the resin. Therefore, the elution process will yield a higher ^{11}B / ^{10}B ratio than the actual value when B is not completely recovered. To test whether the process of ion exchange SPE produces isotopic fractionation, the isotope standard reference material of NBS951 and ten10-μl and ten 20-μl aliquots of South China Sea surface water seawater was used in ion exchange SPE processing. The pump pressure was fixed at 0.04 MPa, and the elution volume was 1000 μl. The $\delta^{11}B$ of NBS 951 and seawater that was chemically treated using ion exchange SPE was determined by MC-ICP-MS.

Fig. 4 illustrated that the results of the $\delta^{11}B$ of NBS 951 chemically treated using ion exchange SPE. The $\delta^{11}B$ values were between -0.014 ‰ - and 0.016 ‰, indicating that the process did not produce significant isotope fractionation.

Boron concentrations and δ^{11}B value of seawater was uniform in the open ocean, with values of 4.5 ppm and 39.6 ‰, respectively. Therefore, seawater was commonly used as an interlaboratory standard. The average (n = 5) δ^{11}B values of 10-μl and 20-μl samples were 39.42 ± 0.13‰ and 39.67 ± 0.10‰, respectively. The accuracy and precision of our method was assessed by comparing measured seawater δ^{11}B values to other published results. [23]

4.4 Boron isotope measurements of river water and rainwater separation using ion exchange SPE

The boron concentrations of BuHa river water and QingHai rainwater was roughly 100 μg/ml and 5 μg/ml, respectively. Then the 1500 μl river water and 20000 μl rainwater samples was separated using ion exchange SPE, and the B isotope compositions were analyzed by MC-ICP-MS. The measured δ^{11}B values of these samples were given in table 3.

Positive ion thermal ionization mass spectrometry (PTIMS) is the most widely used mass spectrometric technique for determining boron isotope ratios due to its inherent capability of providing high-precision isotope ratios. As is clearly shown in the table that the accuracy and precision of our method was assessed by comparing measured these samples δ^{11}B values to published results using PTIMS. It is indicated that our new procedure can be to obtain precise and accurate B isotope compositions for low boron waters.

5. Conclusions

Herein, we present here a method to improve separation of boron from samples using SPE technique and isotope analysis by MC-ICP-MS. It can be efficiently separate more than 10,000 μl of solution with a low boron content. The present technique will extend the application of boron isotope analysis to boron-poor natural samples, including rivers, rainwater and other natural samples.

Acknowledgements

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Table 1. Columns for B Separations using Amberlite IRA 743 resin.

Stage	Column Procedure	Reagent	illustrate
1	Pretreat Sample		Fig. 1A
2	Condition Column	2% HNO_3 (100 μL × 4)	Fig. 1A
		Milli-Q water (100 μL × 4)	Fig. 1A
		Acetic buffer (100 μL × 4)	Fig. 1A
		Milli-Q water (100 μL × 4)	Fig. 1A
3	Load Sample	50 μL × 10	Fig. 1A
4	Wash Column	Milli-Q water (50 μL × 4)	Fig. 1A
		Milli-Q water (100 μL × 4)	Fig. 1A
5	elute B	2% HNO_3 (50 μL × 6)	Fig. 1B
		2% HNO_3 (100 μL ×4)	Fig. 1B

Table 2. Typical operating parameters for B isotopes measurement on Neptune MC-ICP-MS

Parameter	Value
RF forward power	1280 W
Ar cooling gas	16 L min^{-1}
Ar auxiliary gas	0.8 L min^{-1}
Ar sample gas	1.0 L min^{-1}
Extraction voltage	2000 V
Acceleration voltage	10 kV
Detection system	L3, H3 Faraday cups
Nebulizer	Low-flow PFA microcentric (50μl min^{-1})
Spray chamber	47 mm PFA spray chamber
Skimmer cone	High performance skimmer cone
Uptake time	90 s
Number of cycles	20
Integration time	4.194 s
Measurement time	4 min
Rinse time	20min

Table 3. the $\delta^{11}B$ (‰) of samples measured by MC-ICP-MS (2SE，n=4)

Sample	MC-ICP-MS	TIMS
River water-1	15.17 ± 0.18	15.38 ± 0.02 [27]
River water-2	11.21 ± 0. 21	11.57 ± 0.03 [27]
River water-3	8.47 ± 0.19	8.70 ± 0.04 [27]
Rain water-1	13.11 ± 0.13	13.41 ± 0.05 [27]
Rain water-2	9.58 ± 0.17	9.72 ± 0.07 [27]

Figure captions

Figure 1. The experimental apparatus of SPE.

Figure 2. The relationship between total amount of residual boron in solution after the ion exchange, velocity of flow and pump pressure.

Figure 3. The relationship between pump pressure and elution volume.

Figure 4. The $\delta^{11}B$ of NBS 951 with chemically treated using ion exchange SPE

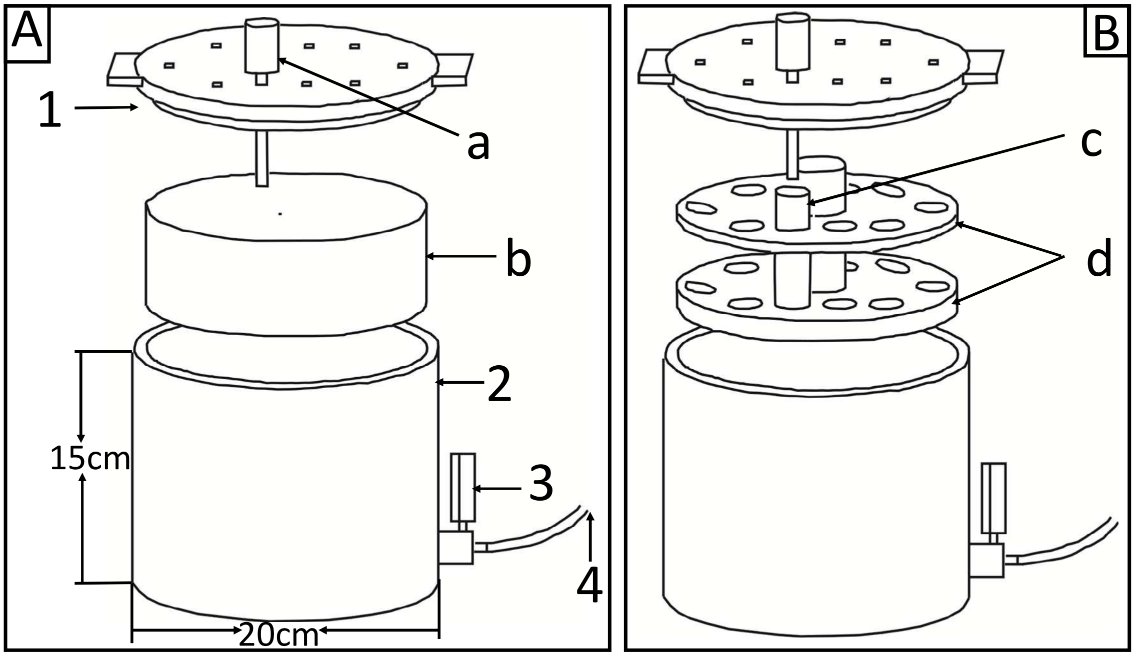

Figure. 1

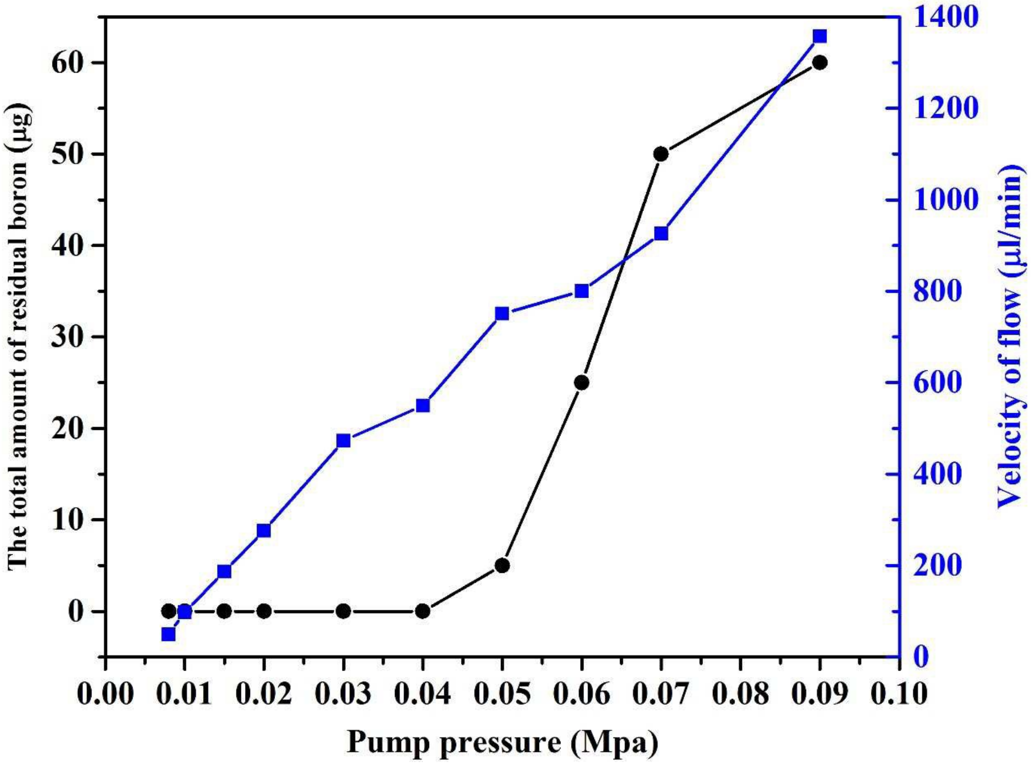
The total amount of residual boron (μg)
0
10
20
30
40
50
60
Velocity of flow (μl/min)
0
200
400
600
800
1000
1200
1400
Pump pressure (Mpa)
0.00
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.10

Figure. 2

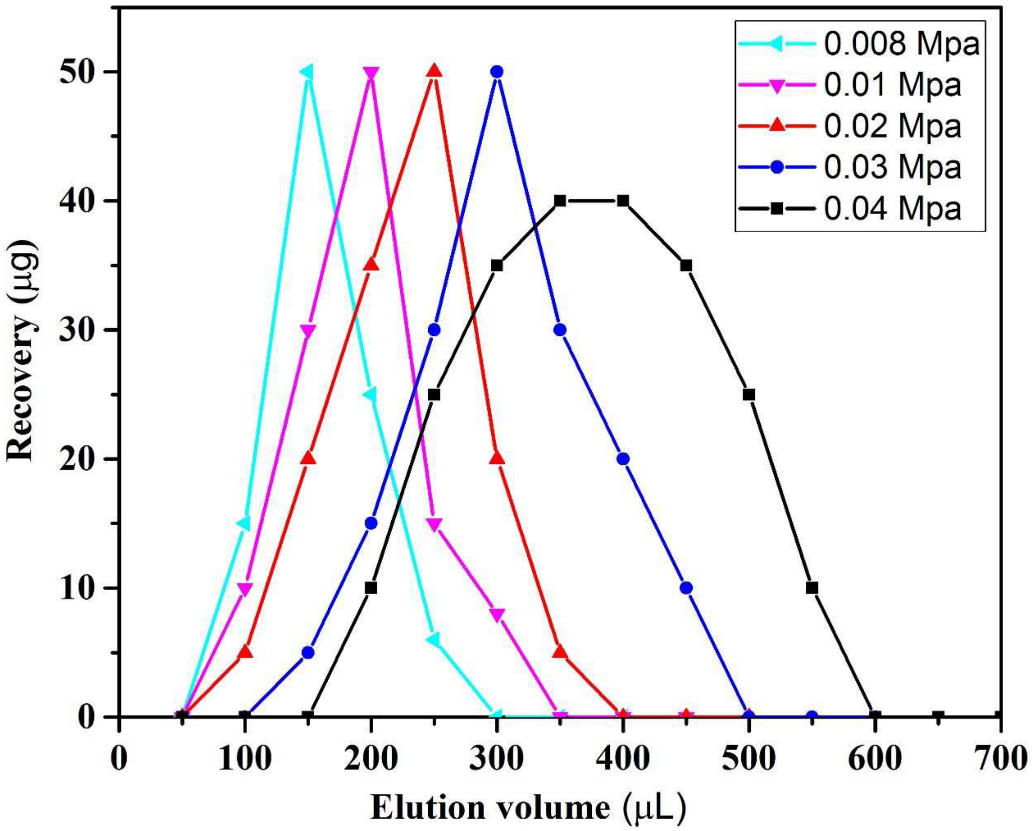
0.008 Mpa
0.01 Mpa
0.02 Mpa
0.03 Mpa
0.04 Mpa
Recovery (μg)
0
10
20
30
40
50
Elution volume (μL)
0
100
200
300
400
500
600
700

Figure. 3

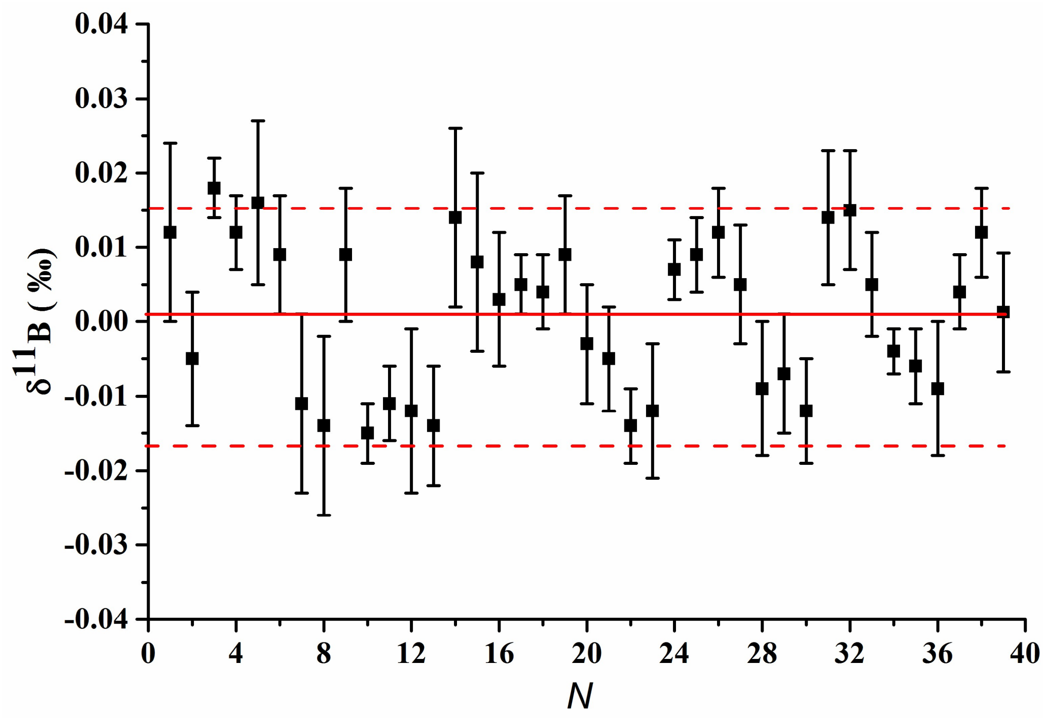
0.04
0.03
0.02
0.01
0.00
-0.01
-0.02
-0.03
-0.04
$\delta^{11}B$ (‰)
0
4
8
12
16
20
24
28
32
36
40
N

Figure. 4