

General High-Pressure Closed Acidic Decomposition Method of Rock Samples for Trace Element Determination Using Inductively Coupled Plasma Mass Spectrometry

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Received April 16, 2018; revised April 17, 2020; accepted April 17, 2020

Abstract—Complete decomposition is an essential prerequisite for accurate trace element quantification in geological samples. This work presents a general high-pressure closed digestion approach for rock samples from basic to acidic rocks by inductively coupled plasma mass spectrometry. Using HNO₃–HF mixed system, different geological reference materials have been investigated for trace element determination, with acid ratio, decomposition time, digestion temperature, sample mass and reagent amount discussed in detail. In brief, 2.0 mL of HNO₃–HF with the ratio of 1 : 1 and a digestion time of 12 h at 185°C in high-pressure sealed bomb are optimal for 50 mg rock sample decomposition. With relative errors under 10%, the analytical results of W-2a, BCR-2, GSP-2, AGV-2 and GSR-1 agree well with certified values. This proposed high-pressure sealed digestion method is characterized with less acid consumption, complete digestion and less damage for digestion process, well meeting the requirements for large sample throughput in geological laboratory.

Keywords: high-pressure closed decomposition, acidic digestion, geological samples, ICP–MS

DOI: 10.1134/S1061934820100147

Trace elements in geological samples provide important information on understanding the rock formation, mantle and crustal evolution and magmatism of planetary bodies [1–3]. Undoubtedly, the quality of analytical data is an essential index in the quantification of geochemical processes. To accurately determine trace elements in geological samples, complete sample decomposition is definitely a critical and fundamental stage [4–6].

There are many digestion technologies developed for the decomposition of geological materials, including alkali fusion [7, 8], open vessel acid digestions [9, 10], high-pressure closed acid digestions [11–13] and microwave dissolution [14, 15]. Due to the complex composition of geological samples, complete digestion of all sample types remains a problem in routine laboratory analysis. This is especially true for rocks containing resistant minerals such as zircon, garnet and spinel [16–18]. Owing to the relatively high backgrounds from the flux and high levels of total dissolved solids, it is known that alkali fusion method is subject to matrix-induced instability in inductively coupled plasma mass spectrometry (ICP–MS) analysis [19]. Despite being an amazing choice for decomposing rocks with acid-resistant accessory minerals, alkali fusion method is not recommended for trace element

quantification in geological samples. Open-vessel acid digestion and microwave digestion have also been successfully applied to decompose a range of geological samples. However, the drawbacks of digestion efficiency and the loss of volatile elements limit the usage of open-vessel acid system [20]. Also, for the regular microwave digestion procedure, the potential safety problem and frequently undissolved residues caused by short digestion time make it unfavorable in geological sample decomposition [21, 22]. High-pressure closed acid digestion method which shows great advantages in terms of high efficiency for insoluble minerals, zero loss of volatile elements and less reagent consumption [23] has become the most popular sample digestion technique in routine geological analysis.

Numerous scientific works focused on high-pressure closed acidic digestion method in geological research. With this high-pressure sealed technique, Liu et al. [24] tested the accuracy of the certified values in 56 Chinese National Standard Reference Materials, and He et al. [25] assessed the possibility of simultaneous determination of 47 elements in geological samples. Gao et al. [26] successfully applied high-pressure closed acid digestion method using concentrated HNO₃, HF and HCl to study the major, minor and trace element compositions in marine geological sam-

ples. However, the solution chemistry related in high-pressure closed dissolution procedures remains unclear. Hence, sample decomposition is still a popular research theme in the scientific field of analytical geochemistry. Sun and He [27] discussed the influence of particle size of geological samples during sealed digestion process. Wang et al. [28] developed a new type of anticorrosive digestion vessel with a double inner arc seal design to improve the digestion efficiency for deep-sea marine sediments. Zhang et al. [29] studied the decomposition abilities for felsic rocks among different acid digestion patterns using high-pressure closed method. Zhu et al. [30] showed that the acid mixture of HF–HNO₃–HClO₄ had optimum volume ratio of 10 : 5 : 1 in decomposing Chinese National Standard Reference Materials. Hu et al. [31] investigated the capability of NH₄F–HNO₃ mixture in granite digestion using screw top PTFE-lined steel bomb at 190°C. Regarding fluoride complexes inevitably formed during the high-pressure digestion process, Chen et al. [32] added another 2 h high-pressure step at 190°C for 50 mg sample fortified with 20% HNO₃ solution (v/v). According to the literature, there is still a lack of reports on general high-pressure closed acidic decomposition method of rock samples ranging from basic to acidic materials for routine trace element determination.

In this current study, we systematically described a general high-pressure closed digestion approach using HNO₃–HF mixture as the decomposition agent for rock samples, with trace elements quantified by ICP–MS. Herein, the digestion parameters, including acid mixture ratio, decomposition time, digestion temperature, sample mass and reagent amount, were investigated in detail. Under the optimum experimental conditions, the digestion capability of the present approach was evaluated by determining the trace elements in a range of geological standard materials from basic to acidic rocks. Results show that the trace elements of W-2a, BCR-2, GSP-2, AGV-2 and GSR-1 are in great agreement with certified values, giving relative errors (REs) under 10% and relative standard deviations (RSDs) less than 5% ($n = 5$). The proposed high-pressure closed digestion method exerts conspicuous characteristics of complete digestion, less acid consumption and less damage for digestion process, which well meets the requirements for large sample analysis throughput in geological laboratory.

EXPERIMENTAL

Reagents and standard solutions. High purity acids and ultra-pure water were used throughout sample assay. All the commercially available acids including HNO₃ (68% v/v, AR grade) and HF (40% v/v, AR grade) were heated by sub-boiling distillation in Teflon stills (Savillex DST-1000-PFA, USA) to remove metallic or cationic impurities prior to usage. Deion-

ized water was passed through a Milli-Q water purification system (Millipore, Bedford, MA, USA) to produce high-purity water with the resistivity of 18.2 MΩ cm.

All the standard solutions stored in a refrigerator at 4°C were prepared using ultra-pure water and kept in polytetrafluoroethylene (PFA) bottles. The PFA bottles and pipet tips were cleaned in 50% (v/v) HNO₃ for 12 h and carefully rinsed three times with Milli-Q water before use. Four solutions (5, 10, 20, 50 ng/mL for all the elements) in 2% HNO₃ (v/v) used as the external calibrators were prepared by gravimetric dilution from 10 µg/mL Multi-element Calibration Standard solutions (Agilent Technologies, Tokyo, Japan). Multi mono-element solutions were prepared from 1.0 mg/mL single element standard solutions purchased from the National Institute of standards and technology, China. Herein, all the calibration standard solutions were progressively diluted from the above stock standard solution using 2% HNO₃ (v/v).

Instrumental apparatus. The instrument utilized in this work was a Thermo Fisher Scientific X series ICP–MS instrument (Waltham, MA, USA) in the Laboratory of Mineralization and Dynamics, College of Earth Sciences and Land Resources, Chang'an University. This ICP–MS apparatus was equipped with a concentric nebulizer for samples injection, a cyclonic spray chamber, a standard quartz torch and a quadrupole mass analyzer.

Before element determination, the instrument was first optimized to obtain stable and relative maximum intensities for ⁷Li, ⁵⁹Co, ¹¹⁵In and ²³⁸U using a 10 ng/mL tuning solution containing Li, Co, In, Ce, U, etc. At the same time, the ratios for oxide formation (CeO⁺/Ce⁺) and doubly charged species (Ce²⁺/Ce⁺) were well controlled under 3.0%. Thereafter, a rock solution was flushed for at least 30 min prior to the instrument tuning to minimize the drift. With rhodium as the internal standard element, drift corrections were done by repeated analysis of a standard rock sample as a drift monitor (quality control rock solutions) between every 5 unknown samples without internal standards. All the detailed operating conditions and instrumental parameters were summarized in Table 1.

Geological materials. Among all kinds of geological samples, it is well known that felsic rocks are very difficult to be decomposed due to the presence of refractory minerals such as zircon [16–18]. Therefore, the granodiorite reference material USGS GSP-2, the most typical refractory geological sample with Zr content of 550 µg/g, was selected to evaluate the decomposition capacity of HF–HNO₃ mixed acid in this work. To further evaluate the general application of the proposed method, a series of rock reference materials covering the compositional spectrum of igneous rocks was also analyzed. Herein, the studied geological reference materials ranged from basic (basalts BCR-2

Table 1. Operating parameters for Thermo Fisher X series ICP-MS

Instrument parameter	Operating condition
Spray chamber	Cone chamber with impact bead at 2°C
Dwell time, ms	10
Scan type	Peak jumping
Sample/skimmer cone, mm	Nickle, 1.1/0.75
Channel spacing, ms	0.02
Output power, W	1250
Coolant Ar, L/min	14.0
Auxiliary Ar, L/min	0.75
Nebulizer Ar, L/min	0.71
Sampling depth, 0.1 mm	100
Readings/replicate	5

and diabase W-2a), intermediate (andesite AGV-2 and granodiorite GSP-2) to acidic (granites GSR-1).

Sample decomposition procedure. All labware was immersed in aqua regia HNO₃-HCl (3 : 1, v/v) at 120°C for 12 h, followed by treating in Milli-Q water for another 12 h at 120°C. Prior to usage, the labware was carefully rinsed three times with Milli-Q water. The rock reference materials with known amounts of elements were weighed in 15 mL Teflon bombs, then decomposed according to the following procedures

(Table 2). (1) 1.0 mL of HF and 0.5 mL of HNO₃ (methods A, B, C, D, E and F), 1.0 mL of HF (method G) or 1.0 mL of HNO₃ (method H) were added into the samples. (2) Then, the bombs were placed on the hotplate with sample evaporated to incipient dry at 140°C. (3) Thereafter, 1.0 mL of HF and 1.0 mL of HNO₃ (methods A, B, C, D, G and H) or 1.0 mL of HF and 1.5 mL of HNO₃ (method E) or 1.0 mL of HF and 2.0 mL of HNO₃ (method F) were inserted and sealed in a high-pressure metal jacket before placing in an oven at 185°C for 12 h. (4) After cooling, the Teflon bombs were opened and put on the hotplate at 140°C. (5) When becoming incipiently dry, the samples were fortified with 1.0 mL of HNO₃ and again evaporated to incipient dry. (6) With 2.5 mL of 40% HNO₃ (v/v) added, the samples were diluted to around 50 mg by 2% HNO₃ (v/v) after cooling (method D) or re-dissolved at 135°C for 4 h with bombs inserted in the high-pressure metal jacket (methods A, B, C, E, F, G and H). (7) The final solutions from methods A, B, C, E, F, G and H were transferred to polyethylene bottles after cooling and then gravimetrically diluted to approximately 50 mg using 2% HNO₃ (v/v). (8) Finally, the trace element contents in the studied samples were detected by ICP-MS directly.

RESULTS AND DISCUSSION

Effect of the ratio of HNO₃-HF acid mixture. The acid combination of HNO₃-HF-HCl-HClO₄ is the conventional choice for geological sample digestion. This acid combination becomes unpopular nowadays owing to the obvious spectrum interferences from Cl

Table 2. Brief decomposition procedures for rock materials detected by inductively coupled plasma mass spectrometry

Procedure		Method							
		A	B	C	D	E	F	G	H
Hotplate pressure relief at 140°C	HNO ₃ , mL	0.5	0.5	0.5	0.5	0.5	0.5	—	1
	HF, mL	1	1	1	1	1	1	1	—
High-pressure closed digestion at 185°C	HNO ₃ , mL	1	1	1	1	1.5	2	1	1
	HF, mL	1	1	1	1	1	1	1	1
	Time, h	12	8	48	12	12	12	12	12
Evaporation at 140°C	HNO ₃ , mL	1	1	1	1	1	1	1	1
Re-dissolution at 135°C	HNO ₃ , mL	1	1	1	—	1	1	1	1
	H ₂ O, mL	1.5	1.5	1.5	—	1.5	1.5	1.5	1.5
	Time, h	6	6	6	—	6	6	6	6

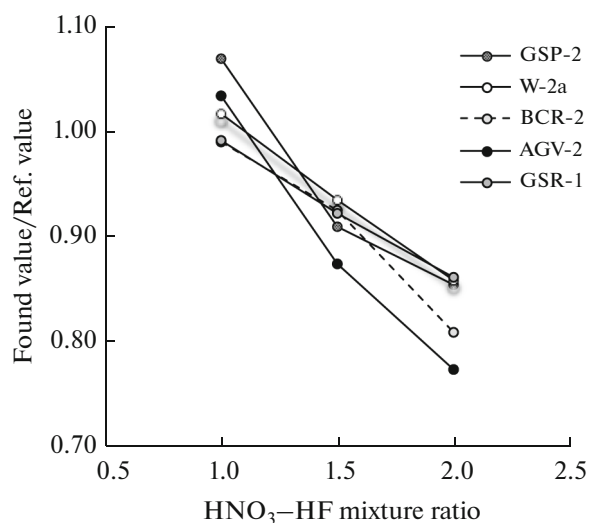


Fig. 1. Relationships of recovery vs. HNO₃-HF mixture ratio. The utilized quantity of HF is 1.0 mL and the studied HNO₃ volumes are 1, 1.5 and 2 mL, respectively.

[33, 34] and lower recoveries of Nb and Ta for ClO₄⁻ addition [29]. There has been lots of reports investigating the ability of HNO₃-HF mixture with different ratios on geological sample decomposition [29, 32]. According to the literature, we mainly studied three combinations of HNO₃-HF, with ratios of 1 : 1, 1 : 1.5, and 1 : 2. As shown in Fig. 1, it is clear that the recovery of Zr obviously declined with increasing HNO₃-HF ratio under the constant 1.0 mL of HF indicating that the addition of HNO₃ greatly influenced the digestion capability of HF for refractory minerals, which agreed with the previous report [29]. Hence, the HNO₃-HF ratio of 1 : 1 was chosen as the optimum digestion acid mixture.

To strengthen the safety index and decrease the reaction pressure mainly from silicon tetrafluoride formation during high-pressure closed digestion procedure, a pressure releasing step was added before placing the samples into the oven. A series of acid combinations including 1.0 mL of HF and 0.5 mL of HNO₃ (method A), 1.0 mL of HF (method G) or 1.0 mL of HNO₃ (method H) was first introduced into the samples, and the samples were then put on the hotplate at 140°C until incipient dryness. By taking both element digestion efficiency and reaction pressure into consideration, the combination of 1.0 mL of HF and 0.5 mL of HNO₃ was selected in this work.

Effect of digestion time and temperature. Decomposition temperature and time belonging to fundamental parameters in high-pressure closed digestion amazingly affect digestion efficiency. Despite less digestion time needed corresponding to a higher temperature, we applied a digestion temperature of 185°C

in routine sample analysis due to the limitations of the physical and chemical properties of the Teflon bomb.

Under the temperature of 185°C, the effect of digestion time on geological samples was also studied. With Li, Rb, La and Zr selected as the representative elements, the recovery results defined as the ratio of the quantified value to certified value are given in Fig. 2. As clearly seen in Fig. 2, the recoveries of Li, Rb and La exhibit negligent changes for decomposition time from 8, 12 to 48 h, whereas the recoveries of Zr achieve their maximum at 12 h and then remain nearly constant. Hence, the optimum decomposition time of rock samples is 12 h in this study.

Effect of insoluble fluorides and the test sample mass. Many previous works showed that HF can enhance the stability of Nb and Ta by forming fluoro-complexes in solution [35]. However, for regular high-pressure digestion closed method using HF or HF-HNO₃ insoluble fluorides of AlF₃ will be formed and AlF₃ precipitation can incorporate trace elements into their lattices causing erroneous results [36, 37]. Takei et al. [38] and Tanaka et al. [39] solved this problem by “Mg-addition” and “Al-addition” method, and Chen et al. [32] reported that the insoluble fluorides can be re-dissolved by adding another 2 h high-pressure step at 190°C after high-pressure digestion step. In the current work, it was found that a clear solution could be observed by naked eyes via heating at 135°C on the hotplate for 6 h with bombs inserted in the high-pressure metal jacket. Results showed that there were excellent agreements between the determined and reference values demonstrating the effect of possible insoluble fluorides could be neglected.

Although a positive correlation exists between the sample size and digestion efficiency, there is no definite standard on the optimum sample mass ensuring representative sampling. In fact, the mineralogy of rocks, particle size, detection limit and insoluble residues formation during decomposition procedures affect the sample amount [40, 41]. Considering the high sensitivity of modern ICP-MS analytical technique, the relationship between sample mass of 200 mesh (25, 50, 80 and 100 mg) and digestion efficiency was also discussed in this work. With the REs controlled under 10%, a mass of 50 mg for all geological standard materials is sufficient to provide a representative sample. However, the sample mass of 25 mg shows significant REs larger than 15% for REEs with content less than 1.0 µg/g. Hence, the optimal sample mass is set as 50 mg, which is also the routine laboratory sampling amount for all real geological samples.

Results for reference materials. Under the experimental conditions in method A digestion pattern which is also the routine decomposition approach in our Lab, the geological reference materials from the basic (basalts BCR-2 and diabase W-2a), intermediate (andesite AGV-2 and granodiorite GSP-2) to acidic (granites GSR-1) were analyzed to test the accuracy of

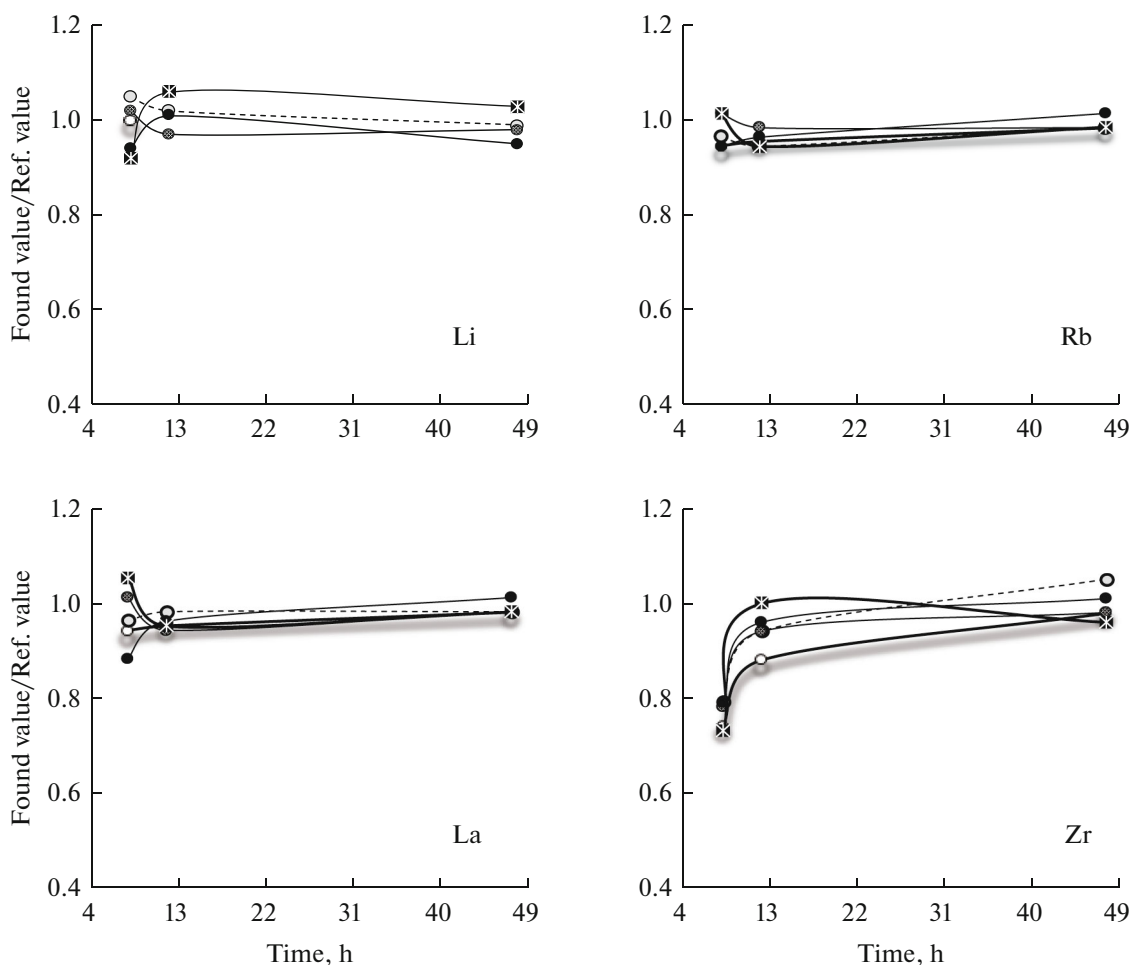


Fig. 2. Relationships of recovery vs. digestion time for Li, Rb, La, Zr. Lines with white ball, gray ball, dotted ball, black ball and square are results for W-2a, BCR-2, GSP-2, AGV-2 and GSR-1, respectively.

this proposed method. With rhodium as the online internal standard element to compensate for matrix effects and instrumental signals drifting, the final sample solutions were analyzed using ICP-MS and the results involving relative errors for 35 trace elements were summarized in Tables 3 and 4. As can be seen in these tables, all the trace elements are in good agreement with certified values ($RSDs < 5.0\%$, $n = 5$) and the corresponding REs are less than 10% (Fig. 3) indicating the good capability of this proposed approach for geological sample decomposition.

CONCLUSIONS

In this present paper, a high-pressure closed digestion approach for rock samples from basic to acidic rocks by ICP-MS has been proposed. Results showed that the Method A decomposition process was optimal in routine rock sample assay. In short, by using 2.0 mL of HNO_3 -HF with acid ratio of 1 : 1, a rock sample of 50 mg was decomposed at 185°C for 12 h in a high-pressure sealed Teflon bomb following the pressure

relieving step. After re-dissolution at 135°C for 6 h in 2.5 mL of 40% HNO_3 (v/v) with bombs inserted in the high-pressure metal jacket, the sample was 1000-fold diluted using 2% HNO_3 (v/v) after cooling and quantified by ICP-MS. With the REs less than 10%, the analytical results of W-2a, BCR-2, GSP-2, AGV-2, and GSR-1 well agree with certified values. The proposed high-pressure closed digestion method shows advantages of complete digestion, less acid consumption and higher safety for digestion process, promising practical value for large sample throughput in geological laboratory.

FUNDING

The authors gratefully acknowledge the financial support from National Natural Science Foundation of China (no. 41503035), the Fundamental Research Funds for the Central Universities of Chang'an University, China (nos. 310827161008 and 300102278201) and the Natural Science Foundation of Shaanxi Province, China (no. 2019JQ-681). The corresponding author also thanks the China Scholar-

Table 3. Analytical results of 22 trace elements for Geological Standard Materials^{a, b}

Element	W-2a				BCR-2				GSP-2				AGV-2				GSR-1			
	found value	RSD, %	RE, %	ref. value	found value	RSD, %	RE, %	ref. value	found value	RSD, %	RE, %	ref. value	found value	RSD, %	RE, %	ref. value	found value	RSD, %	RE, %	ref. value
Li	9.22	3.2	-0.9	9.30	8.97	2.8	-0.3	9.00	35.7	2.1	-0.8	36.0	10.7	1.4	-2.5	11.0	129	2.6	-1.4	131
Rb	20.6	3.0	8.9	18.9	47.9	1.9	-0.2	48.0	221	2.0	-9.6	245	68.3	2.6	-0.4	68.6	487	3.0	4.5	466
Sr	201	1.2	2.7	196	351	2.2	1.5	346	236	1.4	-1.6	240	692	2.8	5.1	658	106	2.5	-0.1	106
Y	21.5	2.5	-2.5	22.0	36.8	1.4	-0.5	37.0	26.1	2.0	-6.8	28.0	20.1	1.4	0.5	20.0	65.2	1.9	5.2	62.0
Zr	93.6	2.4	1.8	92.0	186	2.0	-1.0	188	588	1.0	6.9	550	238	2.2	3.5	230	166	1.2	-0.8	167
Nb	7.52	3.8	0.3	7.50	12.5	2.6	-0.7	12.6	27.0	1.9	-0.2	27.0	31.4	2.0	4.6	30.0	41.2	3.1	2.9	40.0
La	11.1	2.9	2.7	10.8	26.6	1.8	6.6	25.0	195	1.6	8.5	180	40.9	2.1	7.6	38.0	58.5	1.9	8.3	54.0
Ce	24.8	2.6	6.1	23.4	57.7	1.2	8.8	53.0	421	3.2	2.6	410	70.3	1.0	3.3	68.0	114	2.0	5.9	108
Pr	3.24	3.3	7.9	3.00	7.01	1.4	3.1	6.800	51.7	0.8	1.4	51.0	8.33	2.4	0.3	8.30	13.4	1.4	5.9	12.7
Nd	13.8	2.6	6.4	13.0	30.0	1.1	7.2	28.0	213	1.5	6.6	200	30.5	3.6	1.5	30.0	50.3	2.6	7.0	47.0
Sm	3.33	3.4	0.9	3.30	7.08	2.5	5.7	6.70	28.9	1.0	7.0	27.0	5.60	2.8	-1.7	5.70	10.3	1.1	6.3	9.70
Eu	1.13	2.7	4.2	1.08	2.13	1.0	6.3	2.00	2.41	2.2	4.8	2.30	1.56	1.2	1.1	1.54	0.89	2.9	4.8	0.85
Ga	3.69	3.0	0.8	3.66	7.26	1.1	6.8	6.80	12.7	2.8	6.1	12.0	5.00	1.8	6.6	4.69	9.11	2.7	-2.1	9.30
Tb	0.62	3.8	-0.7	0.62	1.13	1.6	5.1	1.07	1.36	2.0	0.2	1.36	0.69	1.1	7.2	0.64	1.73	1.6	5.0	1.65
Dy	4.09	2.5	8.0	3.79	6.34	2.4	-1.0	6.40	6.60	1.4	8.3	6.10	3.86	3.0	7.3	3.60	11.0	1.3	7.6	10.2
Ho	0.79	3.9	-0.1	0.79	1.43	1.3	7.7	1.33	1.08	0.6	7.5	1.00	0.72	1.3	2.0	0.71	2.19	1.8	6.7	2.05
Er	2.28	3.3	2.8	2.22	3.86	2.1	5.5	3.66	2.30	1.4	4.4	2.20	1.67	1.7	-6.5	1.79	7.02	4.0	8.0	6.50
Tm	0.35	4.1	5.3	0.33	0.56	1.1	3.5	0.54	0.30	2.5	4.8	0.29	0.28	2.9	8.8	0.26	1.03	1.9	-2.5	1.06
Yb	2.21	3.0	7.9	2.05	3.68	1.8	5.1	3.50	1.67	2.6	4.2	1.60	1.71	1.4	6.5	1.60	7.45	2.5	0.7	7.40
Lu	0.33	3.2	7.5	0.31	0.56	0.9	8.9	0.51	0.23	1.8	1.0	0.23	0.24	2.0	-2.1	0.25	1.25	2.2	9.0	1.15
Hf	2.52	1.6	3.0	2.45	5.08	1.3	5.8	4.80	15.1	3.7	8.0	14.0	5.24	1.9	3.1	5.08	6.14	3.0	-2.5	6.30
Ta	0.51	1.4	7.7	0.47	0.80	2.4	8.5	0.74	0.95	2.2	5.3	0.90	0.91	2.8	2.2	0.89	6.95	1.7	-3.5	7.20

^a Unit: $\mu\text{g/g}$; ^b Ref. value: reference value.

Table 4. Analytical results of other 13 trace elements for Geological Standard Materials ^{a, b}

Element	W-2a				BCR-2				GSP-2				AGV-2				GSR-1			
	found value	RSD, %	RE, %	ref. value	found value	RSD, %	RE, %	ref. value	found value	RSD, %	RE, %	ref. value	found value	RSD, %	RE, %	ref. value	found value	RSD, %	RE, %	ref. value
Be	0.65	4.1	-8.0	0.71	1.88	3.0	-9.5	2.08	1.46	1.9	-2.9	1.50	2.10	2.1	-8.7	2.3	12.7	1.4	2.1	12.4
Sc	36.4	1.9	1.4	35.9	33.1	2.1	0.3	33.0	5.87	2.0	-6.8	6.30	13.4	1.9	2.7	13.0	6.15	1.8	0.9	6.10
V	271	1.2	1.1	268	411	1.2	-1.2	416	52.7	1.4	1.3	52.0	125	3.3	4.3	120	24.4	2.5	1.7	24.0
Co	48.3	2.2	7.2	45.0	36.9	1.9	-0.2	37.0	7.29	1.9	-0.2	7.30	17.4	2.8	8.5	16.0	3.27	2.7	-3.9	3.40
Ni	76.0	2.1	5.6	72.0	12.1	2.2	-1.4	12.3	17.1	1.5	0.6	17.0	19.9	2.1	4.6	19.0	2.43	0.8	5.8	2.30
Cu	107	0.8	2.4	105	18.7	2.5	-1.8	19.0	44.4	2.2	3.2	43.0	52.7	1.9	-0.6	53.0	3.18	1.7	-0.8	3.20
Zn	80.1	2.3	4.0	77.0	129	1.3	1.6	127	111	2.6	-7.5	120	90.2	3.7	4.9	86.0	25.9	2.6	-7.7	28.0
Ga	17.5	3.2	-2.7	18.0	22.4	1.5	-2.7	23.0	22.2	2.7	0.8	22.0	21.4	1.4	7.1	20.0	19.6	2.4	3.1	19.0
Cs	0.93	4.0	0.7	0.92	1.15	2.4	4.5	1.10	1.19	1.7	-0.9	1.20	1.18	1.8	2.0	1.16	38.4	3.4	0.1	38.4
Ba	168	1.7	-2.1	172	704	3.6	3.1	683	1413	2.6	5.5	1340	1179	2.6	3.4	1140	332	2.3	-3.1	343
Pb	8.10	1.9	5.2	7.70	10.8	1.9	-1.4	11.0	42.8	1.9	2.0	42.0	13.3	1.6	2.6	13.0	31.0	1.5	-2.6	31.0
Th	2.31	2.1	6.5	2.17	6.33	3.4	2.1	6.20	116	3.6	7.6	108	6.23	2.7	2.2	6.10	52.7	2.4	-2.3	54.0
U	0.51	0.8	-0.1	0.51	1.84	1.1	9.0	1.69	2.47	3.0	2.9	2.40	1.99	2.0	5.8	1.88	20.4	2.7	8.4	18.8

^aUnit: µg/g; ^bref. value: reference value.

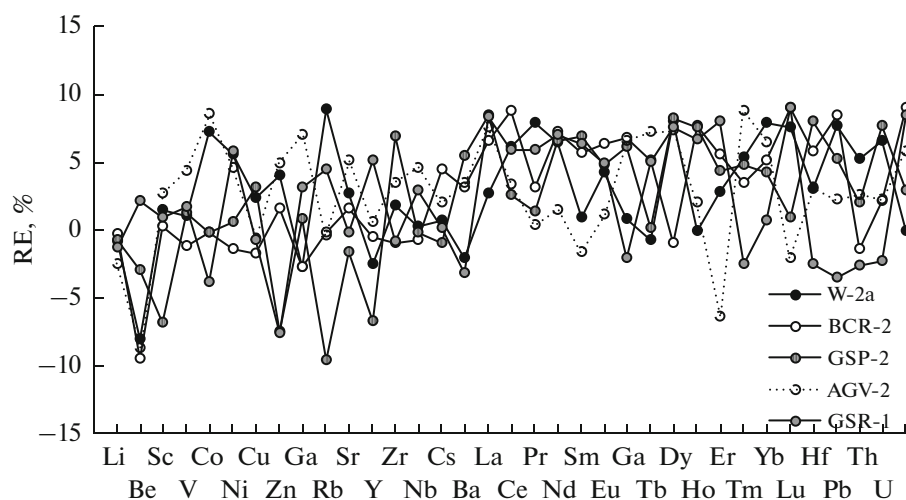


Fig. 3. Relative errors for trace element determination for Geological Standard Materials by ICP-MS. The analyzed sample mass of Geological Standard Material is 50 mg.

ship Council (CSC) for financially supporting during her study in Switzerland.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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