GEOCHEMICAL ANALYSIS

Validation of Trace Element Analysis of Geological Materials by Single-Pulse Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS)

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ABSTRACT
Laser ablation–inductively coupled plasma – mass spectrometry (LA–ICP–MS) is a powerful tool for determining the elemental and isotopic compositions of various samples. The disadvantage of LA–ICP–MS is that it consumes more sample material than other types of in situ analytical methods. To extend the application of this method to trace element analysis, we conducted single-pulse LA–ICP–MS analysis of the Reference Materials of the National Institute of Standards and Technology (SRM 610, 612, and 614) and the United States Geological Survey (BCR-2G, BHVO-2G, and BIR-1G). The accuracy of the determined concentrations and the precision of the single-pulse LA–ICP–MS analysis were verified by comparison with recommended concentrations for these standards and those obtained by conventional analysis with the same system. Time-resolved profiles of the analyzed elements in the glass samples show that the background-corrected intensity reaches a peak immediately after the start of the laser pulse and then gradually decreases. During each analysis, the isotope ratios are stable until the background-corrected intensity of the elements reaches 5% of the peak values. The relative standard deviation of the single-pulse LA–ICP–MS analyses is significantly higher than for conventional LA–ICP–MS analysis. However, most of the determined concentrations obtained from the reference materials except for NIST SRM614, NIST SRM612, BCR-2G, and BHVO-2G mostly agreed with the recommended values within 20%. In addition, the geochemical features of basaltic reference glasses were accurately reproduced by the single-pulse LA–ICP–MS analyses. In conclusion, the single-pulse LA–ICP–MS method provides usable results for the determination of multiple trace elements in geological materials.

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**Introduction**

Laser ablation – inductively coupled plasma–mass spectrometry (LA–ICP–MS) is a widely accepted technique for determining the elemental and isotopic compositions of geological samples. It typically consumes considerably more material than secondary-ion mass spectrometry. However, improvements in quadrupole mass spectrometers have enhanced the signal sensitivity and stability, enabling quantitative analyses of smaller amounts of material. High-spatial-resolution laser sampling with a very small laser beam diameter has been successfully used to obtain reliable concentration data (e.g., Petrelli, Laeger, and Perugini 2016). In many cases, LA–ICP–MS analysis has been applied to relatively thick geological samples (e.g., Ito et al. 2009; Tamura et al. 2015) due to the long ablation time required to achieve the necessary precision. Moreover, the optimization of LA–ICP–MS analytical conditions for ablation of ordinary thin-sections (30 μm thick) is challenging (e.g., Morishita, Ishida, and Arai 2005a, Morishita et al. 2005b; Simonetti et al. 2006; Scarciglia et al. 2009; Yamasaki et al. 2015).

The ability to quantify trace elements in very small samples with a limited number of laser pulses would be advantageous for the analysis of various samples, such as biofilms and elements absorbed onto particulates in soils. In addition, such analyses are sometimes necessary when it is desirable to avoid chemical zoning, which refers to a texture developed in solid-solution minerals wherein there are changes in the chemical composition of the mineral from the core to the rim of the sample, and small inclusions. Geochemical depth profiling by LA–ICP–MS has yielded accurate trace element concentrations with a resolution of 0.1 μm perpendicular to the sample surface (Heinrich et al. 2003), suggesting the feasibility of single-pulse LA–ICP–MS analysis. Furthermore, single-pulse laser sampling, in combination with multi-collector ICP–MS, has been successfully used to obtain isotopic data from geological samples (Cottle, Horstwood, and Parrish 2009; Krachler et al. 2019).

In this study, the trace element compositions of the National Institute of Standards and Technology (NIST) and the United States Geological Survey (USGS) standard reference glasses were determined using single-pulse LA-quadrupole–ICP–MS. The obtained data were compared with recommended values for these standards and data obtained by conventional LA–ICP–MS methods using the same system to assess the accuracy of the single-pulse technique.

**Methods**

**Sample preparation**

Standard reference material (SRM) glasses (610, 612, and 614) of the National Institute of Standards and Technology (the United States Department of Commerce) (NIST) and microanalytical reference materials of the United States Geological Survey (BCR-2G, BHVO-2G, and BIR-1G) were embedded in epoxy resin disks and polished to a flat, scratch-free surface using 1 μm diamond paste. The NIST SRM glasses are synthetic silicate glasses with a SiO$_2$-rich composition (70–72 wt.%; Morishita et al. 2005b) and nominal trace element concentrations from 100 to 500 μg g$^{-1}$ (610), 10 to 80 μg g$^{-1}$ (612), and 0.5 to 50 μg g$^{-1}$ (614), whereas the USGS glasses have a basaltic composition.
After polishing, the sample surfaces were cleaned to the extent possible. However, contamination was sometimes detected during the surface analysis (e.g., for Pb); the reason for this is still unclear. Given that surface contamination is problematic in single-pulse LA–ICP–MS analysis, surfaces of the samples and reference materials were pre-ablated using a laser with a large spot size prior to elemental analysis. Although the methodology involved in cleaning the topmost surface of the analyzed material is key to "surface" analysis, it is out of the scope of this study. However, the analytical techniques presented in this paper are applicable to many materials and for many purposes, including situations where the topmost surface of the sample cannot be analyzed.

**Instrumentation and operating conditions**

The analyses were conducted at the Kanazawa University, Ishikawa, Japan, using a quadrupole ICP–MS with the S-lens option (Agilent 7850, Agilent Technologies, Tokyo, Japan) coupled to a commercially available 213 nm YAG LA system (UP-213 New Wave Research, UK) (Table 1). The diameter of the laser beam ranged from 4 to 100 μm (AI mode). To stabilize the laser fluence, the laser was warmed for 10 s prior to analysis and ablation was conducted by opening the shutter. The LA operating conditions were as follows: beam diameter of 100 μm and energy density of 7 J cm⁻² (attenuator ~55%). A small-volume cylindrical sample cell (5.5 cm in diameter) with a custom-built sample holder was used instead of the sample cell supplied with the UP-213 instrument. To reduce the space in the sample cell, a sample holder optimized for thin sections was prepared (Figure S1). The volume in the cell is approximately 1.5 mL with laser ablation.

A bottle-type stabilizer (20 mL) was installed between the sample cell and the Ar carrier gas line. Ablation was conducted in a He atmosphere (approximately 0.3 mL

| Table 1. Operating conditions for single-pulse LA-ICP-MS in this study. |
|-------------------------------|--------------------------|
| ICP-MS                        |                           |
| Model                         | Agilent 7850 (Agilent Technologies) |
| Forward power                 | 1200 W                   |
| Reflected power               | < 1 W                    |
| Carrier gas flow              | 1.10 L min⁻¹ (Ar)         |
|                               | 0.3 L min⁻¹ (He)          |
| Auxiliary gas flow            | 1.0 L min⁻¹               |
| Plasma gas flow               | 15 L min⁻¹                |
| Interface                     | Ni sample cone            |
|                               | Ni skimmer cone (S-lens option) |
| Isotopes                      | ²⁹Si, ⁴²Ca, ⁴³Ca, ⁴⁹Ti, ⁸⁵Rb, ⁸⁸Sr, ⁸⁹Y, ⁹⁰Zr, ¹³⁷Ba, ¹⁴⁰Ce, ¹⁴⁶Nd, ¹⁴⁷Sm, ¹⁵⁵Eu, ¹⁵⁷Gd, ¹⁶³Dy, ¹⁶⁶Er, ¹⁷²Yb, ²⁰⁸Pb, ²³²Th, ²³⁸U |
| Dwell time                    | 20 ms (except ²⁹Si and ⁴²Ca for 10 ms) |
| Sweep time                    | 0.419 s                  |
| Laser                         | UP-213 (New Wave Research) |
| Model                         |                            |
| Wavelength                    | 213 nm (Nd-YAG)           |
| Pulse length                  | 5 nanoseconds             |
| Ablation cell                 | cylindrical: 5 cm³ reduced from 16 cm³ by sample holder (See Supplementary Material for details) |
| Spot size                     | 100 μm                   |
| Energy density at target      | 7 J cm⁻² (Attenuator: 50-60%) |
| Warming up                    | 10 s                      |
and the ablation gas was subsequently combined with Ar carrier gas (approximately 1.1 mL min\(^{-1}\)). Nickel sampler and skimmer cones were used for all analyses. The sensitivity and operating conditions of the ICP–MS were optimized by monitoring the counts of \(^7\text{Li}, \, \, ^{89}\text{Y}, \, \, ^{232}\text{Th}\) obtained during LA of NIST SRM 612 glass. The \(^{248}\text{ThO} / ^{232}\text{Th}\) ratio was less than 0.5%, and other potentially interfering oxide species are assumed to be negligible.

The data were collected by peak hopping in time-resolved mode. Given that the mass of sample to be ablated in this study was limited, a shorter sweep time was required. Depending on the sensitivity and relative isotope abundance, the dwell times of the isotopes were set to 10 ms for \(^{29}\text{Si}\) and \(^{42}\text{Ca}\) and 20 ms for \(^{43}\text{Ca}, ^{49}\text{Ti}, ^{85}\text{Rb}, ^{88}\text{Sr}, ^{89}\text{Y}, ^{90}\text{Zr}, ^{137}\text{Ba}, ^{140}\text{Ce}, ^{146}\text{Nd}, ^{147}\text{Sm}, ^{153}\text{Eu}, \, ^{157}\text{Gd}, ^{163}\text{Dy}, ^{166}\text{Er}, ^{172}\text{Yb}, ^{208}\text{Pb}, ^{232}\text{Th}\), and \(^{238}\text{U}\).

The washout time for each analysis was 10–15 s. Five single laser pulses on the same spot of each glass sample at 20 s intervals were used to assess the accuracy and detection limits of the analytical technique (Figure 1). Although the long-term reproducibility of this method has not yet been fully evaluated, the same analysis presented here was performed multiple times. The precision and accuracy of this analytical method was the same for all sets of analyses. In this article, one of these analytical datasets will be described in detail.
The concentrations of 35 trace elements were also measured by conventional LA-ICP-MS, following the procedures of Morishita, Ishida, and Arai (2005a, Morishita et al. 2005b) and Tamura et al. (2015). Details of the analytical conditions and data quality for conventional LA-ICP-MS are described in the Supplementary Material and Tables S1 and S2.

**Results and data reduction**

Figure 1 shows time-resolved isotope profiles for five single pulses of selected elements during ablation of the NIST SRM 612 and 610 glasses. The peak intensity was reached just after the start of the laser pulse, indicating that a large quantity of material reached the plasma at the start of the analysis and then gradually decreased. Reproducibility of the five single-pulse signal profiles shows that particles from a single pulse were ionized and contributed equivalently to the signal profile.

All signal intensities were corrected for background by measuring the gas blank before starting the ablation. Figure 2 shows the time-resolved isotope ratios of a single-pulse analysis. We averaged the counts during the time period when uniform isotope ratios were obtained and calculated the average $^{29}\text{Si/element}$ ratio. The isotope ratios were uniform for 4-10 sweeps after the first sweep of data acquisition, following the values subsequently deviated from the average value.

Data reduction followed the protocol outlined by Longerich, Jackson, and Günther (1996). $^{29}\text{Si}$ was used as an internal standard to correct for variations in the absolute amount of material ablated and transported during analysis. The external standards were NIST SRM 612 and BCR-2G. The elemental concentrations of NIST SRM 612 and BCR-2G used for calibration were the Geological and Environmental Reference Materials (GeoReM, http://georem.mpch-mainz.gwdg.de) preferred values (Jochum and Nohl 2008; Supplementary Material). The measured concentrations for each analysis and mean values and standard deviations of five single-pulse analyses on the analytical glass are shown in Tables S3 and S4.

**Discussion**

*Evaluating the precision of single-pulse analysis*

The time required to obtain a precise isotope ratio depends on the concentration of the analyte. This was confirmed from the relationship between the background-corrected intensity of each element and the background-corrected intensity of the peak. The isotope ratios deviated markedly from the average when the counts per second (cps) decreased to $\leq 5\%$ after the start of ablation (Figure 2b) and the background intensity was $< 100\text{ cps or both}$. Given the low abundance of elements in the NIST SRM 614 and some elements in the BIR-1G (e.g., Rb), fewer than four sweeps met these conditions (Figure 3). In contrast, for the other glasses, eight sweeps may be used for most elements. The decline in signal is dependent on the instrument parameters, such as the volume of the sample cell, gas flow rate, and size and length of the tube connecting the sample cell to the inductively coupled plasma mass spectrometer. Under these analytical conditions, a peak of 2000 cps is required to obtain eight sweeps. Many of the elements
in the NIST SRM 612 and some of the elements in the BIR-1G had peak signal intensities of less than 2000 cps (Figure 4). Below we discuss the possibility for quantitative analysis when the number of sweeps available for data reduction is limited, such as for NIST SRM 614.b

**Figure 2.** (a) Typical LA-ICP-MS time-resolved measurements showing the background-corrected intensity (NET) for a single laser pulse analysis (pulse #3 of Fig. 1) of the isotopes $^{29}$Si, $^{42}$Ca, $^{88}$Sr, $^{89}$Y, $^{140}$Ce, and $^{172}$Yb in NIST SRM 612. (b) Regression of the background-corrected intensity in terms of the peak intensity (Peak NET) to the sweep (Sweep NET) ratio plotted versus time for the NIST SRM 612. (c) Ratios of $^{29}$Si to the other analyzed isotopes ($^{29}$Si NET (cps)/Isotope NET (cps)) for the NIST SRM 612. Each open square in (a) represents the average of eight sweeps (within the gray time interval) of each isotope. cps = counts per second.
As mentioned earlier, several sets of five single-pulse analyses were performed under the same analytical conditions. The data were evaluated based on a set of five single-pulse LA-ICP-MS analyses. First, the precision of the single-pulse LA-ICP-MS analysis was assessed by the relative deviation (RD) of the ratio of $^{29}$Si to other isotopes obtained during each single-pulse sampling from the average value of five analyses of the NIST SRM 612 and BCR-2G (Figure S2). The relative difference of each single-pulse analysis from the average value of five measurements is less than 10% for most elements in NIST SRM 612 and BCR-2G. This relative difference for elements at low concentrations, low cps (< 2000 cps), or both is larger, perhaps because the values are so low that accurate statistical estimates cannot be made. Next, the precision of the single-pulse LA-ICP-MS analysis was evaluated by the relative standard deviation (RSD) of the ratio of $^{29}$Si to other isotopes for each sweep of one single-pulse analysis (pulse #3) as compared with the average of five laser pulses during analysis of the NIST SRM 612 and BCR-2G (Figure S3). The relative difference for each sweep of single-pulse analysis from the average of five analyses is almost always less than 50%.

Figure 3. (a) Typical LA-ICP-MS time-resolved data (NET (cps)) for a single laser pulse analysis (pulse #3 of Fig. 1) of $^{29}$Si, $^{42}$Ca, $^{88}$Sr, $^{89}$Y, $^{140}$Ce, and $^{172}$Yb (cps = counts per second) for NIST SRM 614. (b) Regression of the background-corrected intensity in terms of the sweep to the peak ratio (Sweep NET (cps)/Peak NET (cps)) plotted versus time for the NIST SRM 614.
Comparison of single-pulse LA–ICP–MS analyses using the NIST SRM 612 and BCR-2G as standards with recommended values

We measured the elemental concentrations of NIST SRM 610, NIST SRM 612, BCR-2G, BHVO-2G, and BIR-1G by single-pulse LA–ICP–MS using NIST SRM 612 and BCR-2G as standards (Tables S3 and S4) and compared our data with the recommended values for these standards (GeoReM database; Jochum and Nohl 2008).

The NIST SRM 610, NIST SRM 612, BCR-2G, and BHVO-2G data obtained using NIST SRM 612 and BCR-2G as standards mostly agree to within 20% with the recommended values (Tables S3 and S4; Figures S4 and S5). The high relative difference of the average Pb concentration of the five single-pulse analyses of BHVO-2G (+51%) using NIST SRM 612 as the standard is due to the exceptionally high Pb concentration of one single-pulse analysis. Analysis showing high Pb concentrations may be due to unexpected Pb contamination or analytical noise. Excluding this one single-pulse analysis, the difference between the average of the four single-pulse analyses and the recommended value is 24%. The relatively high concentrations of Dy and Er obtained for NIST SRM 610 using BCR-2G as a standard may be due to the large relative standard deviation caused by the low abundance of these elements in BCR-2G. The measured values for BIR-1G agreed to within 30% of the reference values, and the large relative differences may be due to the poor counting statistics for low-abundance elements (Figure 4).

The trace element concentrations of the basaltic glasses calibrated against NIST SRM 612 and BCR-2G are in good agreement with the recommended values, despite the large difference in matrix compositions of the NIST SRM 612 and basaltic glasses. This indicates that the single-pulse LA–ICP–MS method yields similar data irrespective of whether silica-rich glass (NIST SRM series) or basaltic glass (BCR-2G) are used as the standard reference material.

Comparison of single-pulse LA–ICP–MS with conventional LA–ICP–MS using the same system

We compared the Si-normalized average trace element concentrations obtained from the five single-pulse LA–ICP–MS analyses with conventional LA–ICP–MS data. The
relative standard deviations of the five analyses obtained with the conventional method using laser beam diameters of 100 and 55 μm are less than 5% for most elements (for other elements it is typically < 10%). The relative standard deviation of the five single-pulse analyses (< 15%) is higher than these values. However, the relative difference between the recommended values and each single-pulse analysis is usually less than 10% and, in many cases, is almost equivalent to the results obtained by conventional analysis (Figure S6). Chondrite value (Sun and McDonough 1989)-normalized trace element patterns for the BHVO-2G and BIR-1G obtained by five single-pulse LA–ICP–MS analyses using the NIST SRM 612 and BCR-2G as standards and for the BCR-2G obtained using the NIST SRM 612 as a standard are shown in Figure S7. The patterns and their negative and/or positive anomalies are clearly reproduced by the single-pulse LA–ICP–MS analysis. The single-pulse LA–ICP–MS method used in this study yields reliable results for elements with concentrations > 1 μg g⁻¹.

**Evaluation of single-pulse LA–ICP–MS measurements with a small number of sweeps**

Materials with low concentrations of elements, such as the NIST SRM 614, only yield reasonable signal intensities for a small number of sweeps (Figure 3). We examined the data from five single-pulse analyses of the NIST SRM 614 with an average of four sweeps and compared these data with the recommended concentrations and also those obtained from conventional LA–ICP–MS analysis.

The relative standard deviation of five analyses using the conventional method with laser beams having a diameter of 100 and 55 μm were less than 10% for most elements (the others are typically less than 20%) (Figure S8). The relative standard deviation of the five single-pulse analyses is less than 20% for elements with peak counts exceeding 1000 cps (e.g., Sr, Y, Zr, Ce, and U), whereas it is higher for elements with peak counts greater than 1000 cps (up to 50%; Figure S8). The relative differences between each single-pulse analysis and the recommended values are variable but are within 30% for elements with low relative standard deviations. This highlights the potential of obtaining reliable low-level elemental concentration data, even with a small number of sweeps. In addition, by determining the only most important elements in each sample, more sweeps can be obtained even for elements with a low abundance, potentially resulting in more accurate measurements.

**Summary**

We performed single-pulse LA–ICP–MS analyses using geological glass reference materials. Although the time-resolved isotope profiles of the single pulse due to the ablation of the analytical glass show a gradual decrease after reaching the peak intensity, the time-resolved isotope ratios of the single-pulse analysis were uniform from the first sweep of data acquisition up to 4 to 10 sweeps. The accuracy values of the concentrations measured by single-pulse LA–ICP–MS analysis were validated by comparing them with the recommended concentrations for these standards and with concentrations obtained by conventional analysis with the same LA–ICP–MS system. The relative
difference of each single-pulse analysis from the average of five measurements is less than 10% for most elements in the NIST SRM 612 and BCR-2G. Most of the analytical concentrations obtained from the NIST SRM610, NIST SRM612, BCR-2G, and BHVO-2G as standard samples agreed with the recommended values within 20%, except for the concentration of Pb, which was probably due to surface contamination. The chondrite-normalized trace element patterns of analytical glasses by single-pulse LA-ICP-MS using the NIST SRM 612 and BCR-2G as standard samples accurately reproduced the patterns of negative and positive anomalies of selected elements found in the reference values. The single-pulse LA-ICP-MS method used in this study can provide reliable data for elements with concentrations greater than 1 μg g⁻¹.

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