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Review on *in situ* Isotopic Analysis by LA-MC-ICP-MS

Jie Lin[1], **Ao Yang**[1], **Ran Lin**[1], **Ji Mao**[1], **Zhaochu Hu**[1, 2], **Yongsheng Liu***[1]
1. *State Key Laboratory of Geological Processes and Mineral Resources*, *China University of Geosciences*, *Wuhan* 430074, *China*
2. *Faculty of Materials Science and Chemistry*, *China University of Geosciences*, *Wuhan* 430078, *China*
Jie Lin: https://orcid.org/0000-0002-6780-6223; Yongsheng Liu: https://orcid.org/0000-0002-9340-9205

ABSTRACT: The method of LA-MC-ICP-MS has become one choice for the analysis of many isotopic systems due to its relatively low cost, high analysis speed, high spatial resolution, and low matrix effect. However, there are still many challenges in the accuracy, precision, and spatial resolution of *in situ* isotopic composition determination by LA-MC-ICP-MS, which mainly include: (1) how to improve instrument sensitivity, further improve the spatial resolution, and achieve simultaneous determination of isotopes, multiple isotopes, or isotopes with trace elements in low-content samples? (2) how to deduct and correct interference to improve the accuracy of isotopic determination? (3) how to correct isotope fractionation? (4) how to reduce the matrix effect of isotopes? how to develop matrix-matched reference materials? and (5) how to achieve non-matrix-matched correction of isotopes? The high-sensitivity cone combinations, gas mixture, shield torch, and reasonable detector array can be applied to improve the elemental sensitivity. The interferences include the background interference and the interferences from the matrix elements (the isobaric interference, the polyatomic interference, and doubly charged ions interference). To reduce or even eliminate the interference, commonly used methods including interference elimination, background correction, and interference correction by evaluating with an interference-free isotope. Isotopic fractionation correction mainly involves external standard calibration and internal and pseudo-internal standard normalization. For non-matrix matched calibration, many methods can be applied, such as the femtosecond laser, line scan, low laser energy, wet plasma condition, optical setup below the sample surface, and matching the intensity of the sample and standard. In this review, we systematically summarized the above challenges and solutions to promote the study and application of LA-MC-ICP-MS in isotopic determination.

KEY WORDS: LA-MC-ICP-MS, isotopic determination, interference, isotopic fractionation, matrix effect, reference materials, isotopes, geochemistry.

0 INTRODUCTION

In 1984, the first generation of commercial inductively coupled plasma mass spectrometers (ICP-MS) appeared. Gray (1985) pioneered the connection of a laser system with ICP-MS and applied it to the determination of Pb isotopes and elemental compositions in granites. The technique was subsequently applied to trace elemental composition determination in geological samples (Jackson et al., 1992), partition coefficient studies (Jenner et al., 1993), and *in situ* U-Pb dating of uranium-rich minerals (Fryer et al., 1993). Since 1984, quadrupole mass spectrometry (Q-ICP-MS), magnetic mass spectrometry (SF-ICP-MS), and time-of-flight mass spectrometry (TOF-ICP-MS) have been introduced, all of which have greatly improved the accuracy and precision of elemental content determination (Liu et al., 2008). Multi-collector inductively coupled plasma mass spectrometry (MC-ICP-MS) has greatly contributed to the accuracy and precision of isotope determination by its ability to obtain signals of isotopes simultaneously. For example, Misra and Froelich (2009) used Q-ICP-MS for Li isotope determination with a precision of >1‰ (2 SD), which is much lower than that of MC-ICP-MS (0.2‰ – 0.25‰, Lin et al., 2016a; Nishio et al., 2015). Walder et al. (1993) first combined the laser ablation system (LA) with MC-ICP-MS to determine the Pb isotopic composition in NIST 610 and noted the potential of LA-MC-ICP-MS for *in situ* microanalysis of isotopic composition. Subsequently, the LA-MC-ICP-MS was successfully used to determine the Hf isotopes in zircon and Sr isotopes in plagioclase by Thirlwall and Walder (1995) and Christensen et al. (1995), respectively. Currently, LA-MC-ICP-MS is becoming increasingly more mature for the radioisotopic analysis, e.g., Sr (Dauphas et al., 2022; Yin et al., 2022; Zhu et al., 2020; Zhang et al., 2018), Nd (Guéguen et al., 2015; Xu et al., 2015), Pb (Yu et al., 2022; Mitchell et al., 2011) and Hf (Yang M et al., 2023; Hu et al., 2012), which has been widely developed in earth sciences, life sciences, environmental sciences, medicine, and archaeology. With the improvement of accuracy in isotope analysis by LA-MC-ICP-MS, the application of LA-MC-ICP-MS in non-traditional stable isotope analysis has also attracted the attention of an increasing number of ana-

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*Corresponding author: yshliu@cug.edu.cn

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lytical geoscientists. Horn et al. (2006) first applied fs-LA-MC-ICP-MS for the non-matrix matched analysis of Fe isotopes. Subsequently, LA-MC-ICP-MS was gradually applied to non-traditional isotopic analysis of Li (Lin et al., 2017; Kimura et al., 2016; Martin et al., 2015; Le Roux, 2010), B (Lloyd et al., 2018; Lin et al., 2014), Mg (Oeser et al., 2014; Janney et al., 2011; Xie et al., 2011; Young et al., 2002), Si (Frick et al., 2016; Schuessler and von Blanckenburg, 2014; Steinhoefel et al., 2009b; Chmeleff et al., 2008) and Cu (Lazarov and Horn, 2015; Ikehata and Hirata, 2013; Ikehata et al., 2008). LA-MC-ICP-MS has become one of the most essential techniques for *in situ* isotope analysis due to its high sensitivity, low detection limit, wide dynamic linear range, low yield of double charge, low yield of multiatomic ionization, and low sample consumption (Fig. 1). LA-MC-ICP-MS *in situ* microanalysis provides high spatial resolution (>5 μm profile analysis and $n \times 10$ to $n \times 100$ nm depth analysis) and high efficiency (single point analysis <3 min) (Günther et al., 1998; Perkins et al., 1993). In addition, LA-MC-ICP-MS can avoid a series of problems in the sample digestion process of SN-MC-ICP-MS (e.g., incomplete digestion of minerals; Zhang W et al., 2016b, 2012; Cotta and Enzweiler, 2012; Hu et al., 2010), poor stability of sample solution (Tong C L et al., 2009; Liu Y S et al., 2003) and severe memory effects (Hu et al., 2008b; Münker, 1998), the severe oxide interference caused by water (Oeser et al., 2014; Košler et al., 2005) and hydride interference (Czas et al., 2012; Regnery et al., 2010). Therefore, LA-MC-ICP-MS has become the method of choice for *in situ* microanalysis of both radio- and non-traditional stable isotopes. To date, 23 kinds of isotope/isotopic systems (Li, B, C, Mg, Si, S, Cl, Ca, Ti, V, Fe, Ni, Cu, Zn, Sr, Zr, Sn, Ba, Hf, Os, Pb, Nd, U-Pb; Bao et al., 2022; Huang et al., 2022; Lin et al., 2022, 2017; Lu et al., 2022b; Lü et al., 2022a; Luo and Hu, 2022; Xu et al., 2022b; Zhang S H et al., 2022; Zhu et al., 2022; Liu et al., 2021; Xiong et al., 2021; Hogmalm et al., 2019; Zhang W et al., 2019a, b, 2018; Yuan et al., 2018; Schuth et al., 2017; Weyrauch et al., 2017; Frick et al., 2016; Hu et al., 2012; Mitchell et al., 2011; Kasemann et al., 2009; Fietzke et al., 2008a) have been determined by LA-MC-ICP-MS. Despite these advantages of LA-MC-ICP-MS, there are still many problems with this technique in the isotopic analysis.

(1) Improve the instrumental sensitivity to further improve spatial resolution and achieve simultaneous determination of isotopes, multiple isotopes (Yuan et al., 2018, 2008), or isotopes with trace elements for low-content samples (Steinmann et al., 2019; Zhu et al., 2019). The accuracy of isotopic composition determination is closely related to the signal intensity, and the accuracy of the result decreases rapidly as the signal intensity falls below a threshold value (Lin et al., 2017; Hu et al., 2012). LA-MC-ICP-MS is constrained by the tiny spatial scale (the level of μm), which is too small to be analyzed (0.25–2 μg; Günther et al., 1999), and many of the elements to be analyzed are mainly present in geological samples as trace elements (e.g., Li in the mantle is only 1.5 $\mu g \cdot g^{-1}$; Magna et al., 2006). Therefore, when determining the isotopic composition of a sample with low element content, the obtained signal intensity is low. Whereas the isotopic fractionation of geological samples is small (e.g., Ca isotopic composition of natural rocks varies <2‰, DePaolo, 2011), lower signal intensities and smaller isotopic fractionation have greater demands of the analytical precision and accuracy of the instrument. Therefore, improving the signal intensity of the element is one of the main solutions to obtain the high precision and accuracy of isotopic composition determined by LA-MC-ICP-MS.

(2) Interferences (Konter and Storm, 2014; Tanner et al., 2002; Moens et al., 2001; Jiang et al., 1988) and matrix effect (Günther and Koch, 2008; Horn, 2008) caused by matrix elements, can be avoided by the application of ion exchange resins in the isotopic analysis by SN-MC-ICP-MS. However, a large number of matrix elements in the sample would enter the mass spectrum with the elements to be measured in the isotopic analysis by LA-MC-ICP-MS, thus, the isotopic composition of the elements to be measured will be interfered with by matrix elements (polyatomic interference, double and multiply charged interference) (Agatemor and Beauchemin, 2011; Fontaine et al., 2009; Sylvester, 2008; Wieser and Schwieters, 2005). To reduce the influence of the matrix effect, the commonly used method is to develop matrix-matched standard reference materials (Lü et al., 2022b; Yang et al., 2022; Zhang and Hu, 2019). In addition, the study of the non-matrix-matched correction method is also helpful to the accurate *in situ* isotopic microanalysis by LA-MC-ICP-MS (Lin et al., 2022; Li Z et al., 2016).

(3) Isotopic fractionation effect and matrix effect (Horn and von Blanckenburg, 2007; Kuhn et al., 2007; Jackson and Günther, 2003). Isotope fractionation refers to the obtained isotope ratio being different from the actual isotope ratio. When LA-MC-ICP-MS is used to determine the isotopic composition, the ideal condition is that the uniform size of aerosol is generated in the process of laser ablation, there is no isotopic fractionation between aerosol particles, and there is no loss of aerosol during the transmission, and aerosol particles are completely ionized in ICP, and the isotopic composition of the aerosol particles obtained is consistent with that of the initial material. However, in practice, the aerosol particles produced by laser ablation have different sizes, the aerosol particle size distributions produced by different substances are inconsistent (d'Abzac et al., 2012), and particles with different sizes have different isotopic compositions (Hergenröder, 2006). In addition, small-sized particles will be absorbed into the transmission pipeline during the transmission process (Horn and von Blanckenburg, 2007), while large-size particles are easy to precipitate (Jackson and Günther, 2003), thus, the aerosol particles cannot be completely transmitted. Furthermore, large aerosol particles cannot be completely ionized during the ionization process in ICP (Horn et al., 2001). In addition, a large number of substances are introduced into the ICP, which results in the loading effect (Kroslakova and Günther, 2007), and a lot of different chemical components are introduced into the ICP resulting in a matrix effect. Thus, the obtained isotopic composition is different from the actual isotopic composition of the sample, i.e., the isotopic fractionation was introduced.

In conclusion, there has been great progress and development for the *in situ* isotopic microanalysis by LA-MC-ICP-MS, but it is still faced with a series of factors affecting the accuracy, precision, and spatial resolution of the isotopic analy-

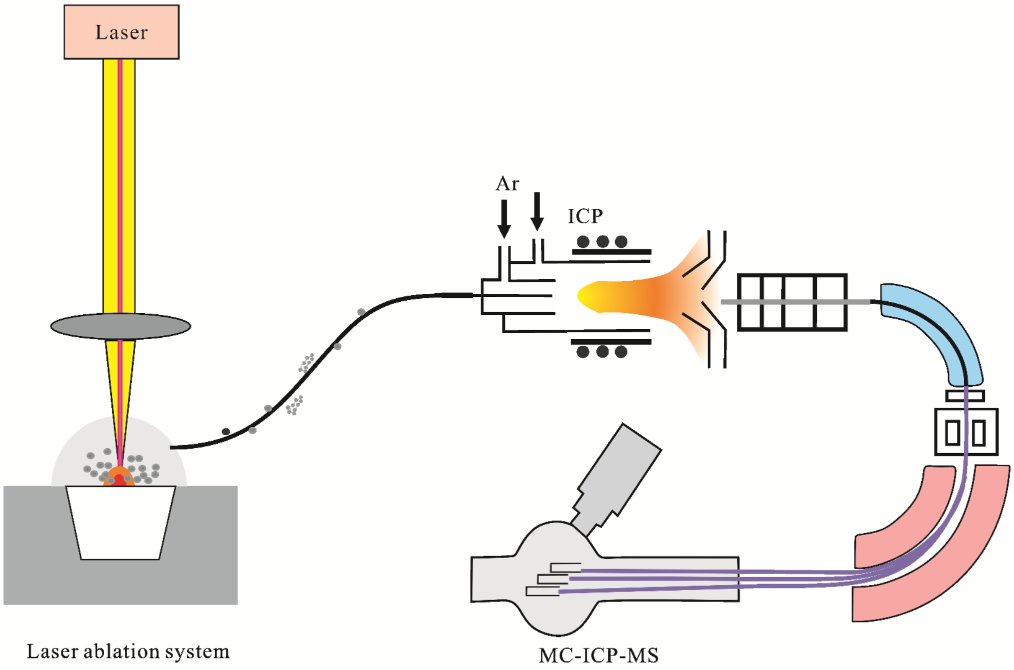

Figure 1. The schematic drawing of LA-MC-ICP-MS.

sis. In this study, the problems existing in the LA-MC-ICP-MS technique for isotope analysis and the corresponding solutions are described to further promote the research and application of the technique.

1 ACCURATE DETERMINATION OF ISOTOPES IN SAMPLES WITH LOW SIGNAL INTENSITY

Whether it is to improve spatial resolution, achieve isotope determination of low-content samples, or achieve simultaneous determination of multiple isotopes, it is necessary to improve the signal intensity. For laser ablation, the larger laser spot, higher laser frequency or higher laser energy can improve the amount of the ablated aerosol to improve the signal intensity (Iizuka and Hirata, 2005; Woodhead et al., 2004). Increasing the ablated sample amount to increase signal intensity will not only reduce the spatial resolution but also increase isotopic fractionation, which will affect the accuracy and precision of isotopic composition (Hu et al., 2012). In addition to improving the parameters of laser ablation, the following conditions for MC-ICP-MS can also be considered to improve the signal intensity.

1.1 Applying High-Sensitivity Cone Combination

The geometry of the sample cone and skimmer cone used in MC-ICP-MS will affect the instrument sensitivity. Hu et al. (2012) used LA-MC-ICP-MS to analyze the Hf isotopes and discussed the relationship of Hf signal intensity with the std + H, std + X, and Jet + X cone combination, respectively. The experimental results showed that the signal intensity of Hf, Yb, and Lu can be improved by the factor of 1.4 and 2.5 when std + X and Jet + X cones were applied relative to std + H cones. Lin et al. (2014) and Xu et al. (2015) also discussed the signal intensity by applying three combinations of cones for the determination of B and Nd isotopes, and the results showed that compared with std + H cones, Jet + X cones could increase the signal intensity of B and Nd by the factor of 3.8 and 3, respectively. The reason why Jet + X cones improved the signals can be summarized as the large aperture and larger length of the Jet cone. The hole diameter of the Jet cone (1.2 mm) is larger than that of standard sample cones (1.0 mm), which can improve the extraction efficiency of ions (Flamigni et al., 2014). In addition, the larger length of the Jet cone can make its hole closer to the torch tube (Hu et al., 2012), which is more conducive to ion extraction. However, not all elements can get the highest signal intensity by applying the Jet + X cone, and the choice of cone combination is related to the element types. For example, Newman (2012) found that the signal intensity of Sr, Nd, Pb, and Hf was higher when the Jet + X cones were used. While with the Jet + X cones, the signal intensity of Li and Mg was lower. Lin et al. (2017) used std + X and Jet + X cones to determine Li isotope, the results indicated that the signal intensity of Li with std + X cones would increase by 46% compared with Jet + X cones.

1.2 Improving the Behavior of Plasma and Laser Ablation with the Gas Mixture

1.2.1 Improving laser ablation behavior with He as the carrier gas

The carrier gas, as an important component in the aerosol transport system of LA-MC-ICP-MS, is used to transport the aerosol particles produced by laser ablation into the plasma. Besides, the chemical composition of the carrier gas can significantly affect the laser ablation behavior and the aerosol transport efficiency (Horn and Günther, 2003). For example, there would be an induced plasma above the ablated crater during the laser ablation, and the laser ablation ability would be reduced with the induced plasma (Shaheen et al., 2012). When He is used as the carrier gas, the laser-induced plasma is weaker and the aerosol particles leave the sample surface vertically in the form of a mushroom cloud. Thus, the transport efficiency of the sample aerosol particles can be increased, and the deposition of aerosol particles around the ablation crater can be reduced (Horn and Günther, 2003). However, when Ar is used as the carrier gas, the stronger laser-induced plasma can be generated above the ablation crater, and aerosol particles leave the sample surface in a direction parallel to the sample surface, easily leading to the deposition of aerosol particles around the ablation crater (Koch et al., 2007). Furthermore, He can quickly dissipate the heat from the laser-induced plasma, reducing the chance of re-condensation between aerosol particles, allowing finer aerosol particles to be produced in the He atmosphere, increasing the ionization degree of aerosol particles in ICP, and thus reducing the isotopic fractionation caused by incomplete ionization of large particles in ICP (Guillong and Günther, 2002) and increases the signal intensity by a factor of 2 – 5 (Günther and Heinrich, 1999; Eggins et al., 1998). Table 1 compared the kinetic principle of aerosol particles as Ar and He used as the carrier gas.

1.2.2 Improving plasma behavior with the gas mixture

The elemental signal intensity could also greatly increase with the introduction of reactive substrates into the plasma, e. g., oxygen (Hutton, 1986), nitrogen (Scheffler and Pozebon, 2014; Louie and Soo, 1992), hydrogen (Louie and Soo, 1992), methane (Fliegel et al., 2011) and organic reagents or water (Liu et al., 2014). For example, Guillong and Heinrich (2007) showed that the elemental signal intensity was significantly increased when nitrogen, methane, hydrogen, and helium were added. And the sensitivities of 47 kinds of elements can increase by 2–4 times as 4–9 mL min^{-1} of hydrogen was added. Hu et al. (2008a) indicated that the addition of 5–10 mL min^{-1} N_2 to the central channel gas increased the sensitivity of 65 kinds of trace elements by the factor of 2–3, while oxide interference (ThO^+/Th) and hydride interference (ArH^+/Ar^+) were reduced by the factor of 1 – 3, respectively. The main reason for the improved signal intensity by the addition of N_2 is that the physicochemical conditions, shape, temperature, and ion density distribution of the Ar plasma were changed (Montaser et al., 1987). Hu et al. (2008a) and Witte and Houk (2012) indicated that the thermal conductivity of N_2 is 23 times higher than that of Ar (Hu et al., 2008a) and that N_2 can act as an “energy trap” during the heat transfer, transferring energy rapidly from the periphery of the plasma to the central channel and increasing the temperature of the central channel (Holliday and Beauchemin, 2004; Ishii et al., 1988). The increase in the central channel temperature increases the evaporation efficiency of the analytes (especially the high field-strength elements Zr, Hf; Hu et al., 2012), Yb and Lu; Iizuka and Hirata, 2005, etc.). However, the addition of N_2 does not increase the signal intensity of all elements, especially those with lower ionization energies. For example, Lin et al. (2014) noted that the signal intensity of B can be reduced with the addition of N_2 in the B isotopic analysis by LA-MC-IC-MS. Because B has the first ionization energy of 8.3 eV, which is a readily ionizable element compared to the first ionization energy of Ar plasma (15.8 eV), and an increase in thermal conductivity has little effect on the ionization degree of B, thus B signal intensity cannot be increased. In addition, the signal intensity of S (Fu et al., 2016), Sr (Zhang et al., 2018; Tong et al., 2016), and Li (Lin et al., 2021) was also found not to be increased with the addition of N_2. Fortunately, the stability of the isotope ratios can be improved, contributing to the accuracy and precision of the isotopic determination.

In addition, the introduction of small amounts of organic reagents into the dry aerosol produced by laser ablation can also help to improve signal intensity. Liu et al. (2014) showed that the introduction of small amounts of ethanol into the dry aerosol produced by a 193 nm laser improved the sensitivity of 60 kinds of elements by a factor of 1.5 to 3.0. The reasons for the improvement of signal intensity with the introduction of organic reagents are controlled by many factors. For example, Kralj and Veber (2003) pointed out that the increased degree of elemental sensitivity was related to the volatility of the organic reagent, with the more volatile reagents having a greater sensitizing effect. Llorente et al. (1997) pointed out that polyols, mono alcohols, and organic acids have different levels of elemental sensitization, suggesting that the sensitization of organic reagents may be related to the content of functional groups. Hu et al. (2004) pointed out that the increased degree of elemental sensitivity was related to the volatility and concentration of the organic reagents, the mass and the first ionization energy of the analyzed element, as well as the operating conditions of the plasma. And the increased sensitivity was independent of the functional groups of the organic reagents. Although the mixture of gas as well as the organic reagent will greatly increase the signal intensity of elements. However, there are also some related disadvantages. For example, the addition of a mixture of gas can increase polyatomic ion interference for some elements (Scheffler and Pozebon, 2014). In addition, the introduction of organic reagents might also result in carbon deposits blocking the cone holes (Liu et al., 2014), reducing the instrumental sensitivity and affecting the accuracy and precision of isotopic analysis.

Table 1 Comparison of the properties of Ar, He, and the kinetic properties of aerosol particles when Ar and He are used as carrier gas

Characteristic	Ar	He	References
Density	High	Low	Horn and Günther (2003)
Viscosity	High	Low	Horn and Günther (2003)
Thermal conductivity	Low	High	Horn and Günther (2003)
Plasma shielding	Big	Small	Koch et al. (2007)
Particle diameter	Big	Small	Horn and Günther (2003)
Movement direction of aerosol	Horizontal	Vertical	Koch et al. (2007)
Particle deposition amount	High	Low	Horn and Günther (2003)
Mass fractionation	Big	Small	Jackson and Günther (2003)

1.3 Applying the Shield Torch

The shield torch is a grounded shield between the load coil and the torch tube to reduce the potential difference. The main function of the shield torch is to eliminate the secondary discharges and reduce background noise and polyatomic ion interference (Zheng et al., 2004; Gray, 1986). In addition, the shield torch reduces the plasma bias potential and the secondary discharge effect, the use of a shield torch can also reduce the kinetic energy distribution of ions and improve the ion transport efficiency by a factor of 20 (Appelblad et al., 2000). The signal intensity of 39 kinds of elements was determined with/without the shield torch by Chen et al. (2015), and the experimental results showed that the use of a shielded torch would increase the sensitivity of the elements by a factor of 17–58, except for Li, Be and Na. In addition, Xu et al. (2015) noted that the signal intensity of Nd can be increased by a factor of 1.6 with the addition of nitrogen by using the shield torch. However, the signal can be decreased with the addition of N_2 when the shield torch was not used. The main reason was the addition of N_2 expanded the kinetic energy distribution of ions in the plasma, while the use of a shield torch decreased the kinetic energy distribution of ions, and the combination of the two factors did not decrease the ion transport efficiency and increases the signal intensity of Nd.

1.4 Reasonable Configuration of Detectors

At present, the most commonly used detectors of MC-ICP-MS are the Faraday cup, secondary electrons multiplier (SEM), and Daly detector (Koppenaal et al., 2005).

Faraday cup is the most commonly used ion detector for direct detection of ion current intensity in the mass spectrometer, which is suitable for measuring relatively large ion current intensity. The amplifiers currently fitted to Faraday cups typically have resistance values of 10^{11}, 10^{12}, and 10^{13} Ω. Faraday cup has good stability for isotopic composition determination. However, Faraday cup detectors are less sensitive and have higher electronic noise than SEM. The noise of the Faraday cup can seriously affect the determination of isotopic ratios in samples with low elemental content (Koornneef et al., 2013; Wieser and Schwieters, 2005). The electron noise can be described as the following equation.

$$\Delta V = \sqrt{\frac{4\kappa_B RT}{t_m}}$$

where ΔV is the electronic noise (V), κ_B is the Boltzmann constant (1.38×10^{-23} J/K), R is the amplifier resistance value (Ω), T is the Kelvin temperature (K), and t_m is the integration time (s). The electronic noise of a Faraday cup is related to the amplifier resistance value, the temperature, and the integration time. When the amplifier resistance is expanded by a factor of 10 or 100, the signal intensity is expanded by a factor of 10 or 100, and the electronic noise of a Faraday cup is proportional to the square root of the amplifier resistance. Therefore, theoretically with 10^{12} and 10^{13} Ω, the signal-to-noise ratio relative to the 10^{11} Ω resistors, the signal-to-noise ratio will be increased by the factor of 10 and 100, respectively. To extend the capability of Faraday cups to determine low signal intensity, 10^{12} and 10^{13} Ω have been exploited (Koornneef et al., 2015, 2014, 2013). For example, Lloyd et al. (2018) used a 10^{13} Ω Faraday cup amplifier to achieve high-precision determination of B isotopes in samples with ^{11}B signal intensity of less than 4.2 mV (2SD = 0.3‰).

The sensitivity of SEM is several orders of magnitude higher than that of the Faraday cup, so it is widely used in various mass spectrometer. Relative to the Faraday cup, the sensitivity of SEM can be improved by a factor of 10^5–10^8, and the SEM has a faster response speed. However, SEM has a series of shortcomings in determining isotope composition, including mass discrimination effect, poor peak shape, and dead time of the detector. In addition, because of the low counting statistics of SEM, the detection accuracy of isotopes is lower than that of the Faraday cup (Becker, 2008). The SEM can be used to determine the radioisotope composition of low-content samples (Zhu et al., 2019), and the spatial resolution of U-Pb dating can be improved (Xie et al., 2017). For example, Zhu et al. (2019) used SEM as a detector for the determination of the Os isotope, which could accurately determine the Os isotopic composition when the signal intensity of Os was below 10 mV. Xie et al. (2017) used a full SEM as a detector to carry out *in situ* U-Pb dating of zircons with a spatial resolution of 5.8–7.4 μm (beam spot) × 3 μm (depth) with an accuracy of <1%.

Daley detector is another kind of detector. Compared with SEM, its outstanding characteristics are high sensitivity, low background noise, linear range of 8 orders of magnitude, long service life, and no dead time correction and linear range correction. Although the Daley detector is considered a substitute for the Faraday cup, the signal intensity of the Daley detector is 1–2 orders of magnitude lower than that of the Faraday cup (Becker, 2008).

For the elements with large differences in isotope abundance, the isotopic composition can also be determined using Faraday cups with mixed amplifier resistance and a mixture of Faraday cups and SEM. For example, for the Li isotope, the abundances of ^{7}Li and ^{6}Li are 92.5% and 7.5%, respectively, with the former being 12.3 times more abundant than the latter. Steinmann et al. (2019) used Faraday cups configured with 10^{11} and 10^{13} Ω for the collection of ^{7}Li and ^{6}Li, respectively, for samples with Li content greater than 10 μg·g^{-1}. For samples with Li content less than 10 μg·g^{-1}, Steinmann et al. (2019) used Faraday cups configured with 10^{13} Ω and an SEM to collect ^{7}Li and ^{6}Li, respectively. It is worth noting that a τ correction is needed when 10^{13} Ω Faraday cups are used. In the case of the SEM, a dead time correction, as well as a dynamic linear range correction of the signal is required (Richter et al., 2016; Nelms et al., 2001).

2 ELIMINATION/REDUCTION AND CORRECTION OF MASS SPECTRAL INTERFERENCE

The first consideration for mass spectral interference is to use appropriate measures to eliminate or reduce them. However, in cases where mass spectral interference cannot be eliminated, an interference correction method should be considered. Interference correction can be divided into blank correction and interference correction.

2.1 Elimination/Reduction of Mass Spectral Interference

The elimination/reduction of mass spectral interference is mainly achieved by three methods: optimizing plasma ionization conditions (cold plasma, shielding torch, sampling depth, active gas, carrier gas flow rate, etc.), physical separation (change resolution by slit control) and chemical separation (collision cell or reaction cell).

At first, the mass spectral interference can be reduced or even eliminated by optimizing plasma ionization conditions. For example, the interference of ArH, ArN, ArO, and ArC can be reduced by the application of cold plasmas (lower RF power or higher sample gas flow rates), thus enabling the determination of the isotopic composition of elements such as low levels of K, Ca, and Fe (Chernonozhkin et al., 2017; Murphy et al., 2002). However, the application of cold plasma can reduce the ionization capacity of the plasma, and use of the low RF power can significantly reduce the ionization efficiency of difficult-to-ionize elements, resulting in a significant reduction in signal intensity. In addition, the lower temperature of the central plasma channel under cold plasma conditions results in incomplete matrix decomposition and thus poor matrix tolerance in isotope determination (Chernonozhkin et al., 2017). In addition to the use of cold plasmas, the application of appropriate instrumental components and reasonable parameters can also reduce interference to a large extent. For example, the use of a shield torch can reduce the yield of doubly charged ions (Chen et al., 2015; Gray, 1986). Additionally, the doubly charged ion and polyatomic ion yields can also be reduced by optimizing instrumental parameters such as plasma gas flow rate, sampling depth, and RF power (Chen et al., 2015). Another straightforward way to reduce interference is to improve the instrumental resolution. For example, for *in situ* determination of Fe isotopes by LA-MC-ICP-MS, the use of high resolution can reduce the interference of $^{40}Ar^{14}N$, $^{40}Ar^{16}O$, $^{40}Ar^{16}O^{1}H$ and $^{40}Ar^{18}O$ to ^{54}Fe, ^{56}Fe, ^{57}Fe, and ^{58}Fe (Oeser et al., 2014; Steinhoefel et al., 2009a), but the sensitivity would be greatly reduced with the high resolution.

Another method to eliminate/reduce mass spectrometry interference is to use chemical separation, using a collision reaction cell to eliminate interfering elements based on their properties. For example, the interference of ^{87}Rb on ^{87}Sr in the Rb-Sr isotope system can be eliminated using a collisional reaction cell, where Sr is formed by reacting with the gas CH_3F to form SrF^+ and Rb does not react with the gas, thus eliminating the interference of ^{87}Rb on ^{87}Sr and obtaining accurate Rb-Sr dating based on the difference in properties of the elements (Bevan et al., 2021. Moens et al., 2001). For the interference of Lu and Yb on Hf in the Lu-Hf isotope system, Wu et al. (2023) used NH_3 as the reaction gas in the collision reaction cell to achieve accurate Lu-Hf isotope dating of xenotime, apatite, and garnet. At present, the collisional reaction cell technique has also been successfully applied to the determination of isotopes of K, Ca, and Fe (Gao et al., 2022; Wang et al., 2022; Zheng et al., 2022; Chen et al., 2021; Li W Q et al., 2016). In addition to chemical reactions, mass spectral interference can be eliminated and reduced by physical adsorption and gas exchange. For the interference of Hg on Pb isotope determination, Hirata et al. (2005) and Yuan et al. (2015) used charcoal filters or activated carbon to reduce most of the Hg in the Ar gas; Hu et al. (2015) and Zhang et al. (2016a) used Au-plated de-mercury homogenizers and gas exchange devices to rapidly reduce Hg from samples and carrier gases, respectively. Additionally, recent research indicated that the background interference can be reduced by the wet plasma and the addition of N_2. For example, the main interferences ($^{12}C^{14}N^+$ and $^{48}Ca^{2+}$) in Mg isotopic analysis by LA-MC-ICP-MS can be significantly suppressed, making it applicable for the application of low mass resolution. Zhang et al. (2018) pointed out that the interference of Kr on Sr would be greatly reduced when 12 $mL\cdot min^{-1}$ N_2 was introduced into the central gas for *in situ* Sr isotopic determination using LA-MC-ICP-MS.

2.2 Correction of Mass Spectral Interference

2.2.1 Correction of blank interference

For the isotopic determination by LA-MC-ICP-MS, the main component of the plasma is the small number of other gases in addition to Ar, e.g., typically 99.999% Ar will contain about 1 $\mu g\cdot g^{-1}$ of O_2, 2 $\mu g\cdot g^{-1}$ of H_2O and a small number of other gases (CO_2, N_2). When blank interferences are related to the working gases, e.g., $^{40}ArO^{16+}$ interferes with $^{56}Fe^+$, $^{40}Ar^{4+}$ interferes with B^{10+}, the blank interferences can be corrected using blank deduction. The blank value was obtained as all the flow rates and instrument parameters are kept the same as the sample being analyzed (Lin et al., 2017). However, this blank deduction method is proved to be not reasonable because, during the measurement of the actual sample, sample aerosol particles are introduced into the whole system, i.e., a certain mass loading (matrix effect) is generated, and the presence of the mass loading affects the signal intensity; whereas no aerosol particles are introduced during the determination of the blank signal intensity. Therefore, to deduct the true blank signal value, a sample without the element to be measured (i.e., a blank sample) needs to be stripped (Kimura et al., 2013b; Ramos et al., 2004; Christensen et al., 1995). The blank signal intensity is more related to the instrumental contamination by the measurement process and the memory effect of the element (Lin et al., 2016a; McGinnis et al., 1997). For elements with a strong memory effect, the background signal intensity increases with increasing the analysis time. To reduce the effect of memory effect on the blank deduction, the silicate glasses that do not contain the analyzed element can be ablated and reduce the deposition of the aerosol in the transport process (Lin et al., 2017; Gilbert et al., 2014a). In addition, for such elements with strong memory effects, the blank deduction can also be performed by performing a linear fit based on the relationship between the blank value and the analysis time, calculating the real-time blank signal value during the measurement, and then making an accurate deduction for the blank value (Kimura et al., 2016; Tang et al., 2007).

2.2.2 Correction of interference from the sample matrix

For the *in situ* microanalysis of isotopes, not only the analyzed elements but also the sample matrix will enter the ICP simultaneously. Therefore, the sample matrix may interfere with the determination of the isotopic composition of the elements to be measured. The sample matrix-related interference includes isobaric interference ($^{54}Cr^+$ vs. $^{54}Fe^+$), polyatomic ion interference ($^{40}Ca^{16}O^+$ vs. $^{56}Fe^+$) (Xu et al., 2021; Zheng et al., 2018), and doubly charged interference ($^{88}Sr^{2+}$ and $^{86}Sr^{2+}$ vs. $^{44}Ca^{+}$ and $^{43}Ca^{+}$) (Zhang L et al., 2020). For interference correction from the sample matrix, the interference can be corrected by measuring the signal intensity of the non-interfering isotope of the element and then combining this with a mass fractionation correction. For example, for *in situ* Ca isotope determination by LA-MC-ICP-MS, $^{86}Sr^{2+}$ and $^{88}Sr^{2+}$ can lead to doubly charged ion interference to $^{43}Ca^{+}$ and $^{44}Ca^{+}$. Accurate determination of the signal intensity of the non-interfering isotope $^{87}Sr^{2+}$, i.e., mass number 43.5, combined with the mass fractionation factor f_{Sr} allows accurate deduction of the signal intensity of $^{86}Sr^{2+}$ and $^{88}Sr^{2+}$ (Zhang et al., 2019a).

$$I_{^{42}\mathrm{Ca}} = I_{42} - \left(\frac{I_{^{87}\mathrm{Sr}^{++}} \times r_{\left(^{84}\mathrm{Sr}/^{86}\mathrm{Sr}\right)} \times \left(M_{84}/M_{86}\right)^{f_{\mathrm{Sr}}}}{r_{\left(^{87}\mathrm{Sr}/^{86}\mathrm{Sr}\right)} \times \left(M_{87}/M_{86}\right)^{f_{\mathrm{Sr}}}} \right)$$

$$I_{^{43}\mathrm{Ca}} = I_{43} - \left(\frac{I_{^{87}\mathrm{Sr}^{++}}}{r_{\left(^{87}\mathrm{Sr}/^{86}\mathrm{Sr}\right)} \times \left(M_{87}/M_{86}\right)^{f_{\mathrm{Sr}}}} \right)$$

$$I_{^{44}\mathrm{Ca}} = I_{44} - \left(\frac{I_{^{87}\mathrm{Sr}^{++}} \times r_{\left(^{88}\mathrm{Sr}/^{86}\mathrm{Sr}\right)} \times \left(M_{88}/M_{86}\right)^{f_{\mathrm{Sr}}}}{r_{\left(^{87}\mathrm{Sr}/^{86}\mathrm{Sr}\right)} \times \left(M_{87}/M_{86}\right)^{f_{\mathrm{Sr}}}} \right)$$

where *I* means the signal intensity obtained on LA-MC-ICP-MS; *r* means the isotope ratio; *M/M* means the isotope mass ratio.

3 CORRECTION OF ISOTOPIC MASS FRACTIONATION

During the laser ablation process, the aerosol ionization process, and the ion extraction-transport process, the conversion or transport efficiency of the material is not consistent. Therefore, the final isotopic ratio deviates from the initial ratio, which refers to isotopic fractionation (Zhang and Hu, 2020; Yang L et al., 2018). Thus, isotope fractionation occurs in the process of particle formation, aerosol transport, and ICP ionization (Garcia et al., 2009). Due to the isotopic mass fractionation, the determined isotopic composition cannot be directly used and need to be corrected for isotopic composition using the fractionation correction method. Currently, isotope fractionation correction methods commonly used in LA-MC-ICP-MS include internal (pseudo-internal), external, and linear regression methods (Meija et al., 2012; Yang, 2009; Fietzke et al., 2008b; Albarède et al., 2004).

3.1 Mass Fractionation Correction Method

3.1.1 Internal and pseudo-internal standardization

Internal standard methods were commonly used for fractionation corrections of radioisotopic compositions, e. g., Sr (Müller and Anczkiewicz, 2016; Jochum et al., 2009; Fietzke et al., 2008b; Vroon et al., 2008; Woodhead et al., 2005), Hf (Fisher et al., 2014, 2011a; Hu et al., 2012; Woodhead et al., 2004; Griffin et al., 2002) and Os (Pearson et al., 2002), which used a stable isotope pair as an internal standard to calculate the mass fractionation factor, and then accurately corrected for the isotopic composition. For example, for the determination of Nd isotope, the mass fractionation factor was calculated using $^{146}Nd/^{144}Nd$ as the internal standard, which is used for the determination of $^{143}Nd/^{144}Nd$ (Fisher et al., 2011b; Iizuka et al., 2011; Yang et al., 2008; Foster and Vance, 2006). For the elements with only two isotopes (e.g., Cu) or elements with multiple isotopes but no two stable isotopes (e. g., Pb), accurate isotopic corrections cannot be made using the internal standard method. However, mass fractionation correction can be made using mass fractionation factors obtained for isotopes of similar mass to the measured isotope, i.e., the pseudo-internal standard method. For example, the mass fractionation of the Pb, Re, Lu, and Cu isotopes can be corrected using $^{205}Tl/^{203}Tl$ (Thirlwall, 2002), $^{193}Ir/^{191}Ir$ (Pearson et al., 2002); $^{173}Yb/^{171}Yb$ or $^{172}Yb/^{171}Yb$ (Fisher et al., 2014) and $^{66}Zn/^{64}Zn$ (Maréchal et al., 1999). For the case of *in situ* isotopic microanalysis by LA-MC-ICP-MS, the specific measures of introducing pseudo-internal standard elements can be implemented using a combination of nebulizer and chamber (Zhang W et al., 2022) or a membrane desolation device (Feng et al., 2018; Chen K Y et al., 2017). The specific isotopic correction using internal and pseudo-internal standardization can be referred to (Lin et al., 2016b).

The internal and pseudo-internal standardization methods can improve the accuracy and precision of isotopic determinations, and two points that need to be noted: 1. The atomic mass of the internal standard or pseudo-internal standard isotope pair should be as close as possible to that of the measured isotope (Vance and Thirlwall, 2002); 2. The internal scale must be a constant natural ratio. However, many scholars have pointed out that what was previously considered to be a natural ratio is not constant. For example, Ma et al. (2013) and Fietzke and Eisenhauer (2006) showed that the $^{88}Sr/^{86}Sr$ ratio is not constant, so the correction of $^{88}Sr/^{86}Sr$ may vary depending on the used $^{88}Sr/^{86}Sr$. However, it is still not possible to give an accurate assessment of whether the errors caused by differences in internal scale values affect the interpretation of geological problems (Lin et al., 2015). Similar problems also exist for the determination of Nd, Hf, and Os isotopic ratios (Yang, 2009).

3.1.2 External standard-sample bracketing (SSB) method

For isotope systems that lack a suitable internal standard or a pseudo-internal standard, mass fractionation correction using an external standard method is required. For example, for low-mass elements (e. g., Li, B, Mg, and Fe), isotope fractionation corrections cannot be made using internal or pseudo-internal standards, even if isotopic pairs of similar masses are selected. Because the difference in mass fractionation factors is large at the low mass end of the spectrum, and mass bias is more severe at the lower mass elements, an external calibration is required (Johnson et al., 2004). The external standard calibration method, also known as the standard-sample bracketing method (SSB), involves inserting two standards before and after the measured sample, and the average of the fractionation factors of these two standards is used as the fractionation factor of the sample. For isotopic composition determination by SN-MC-ICP-MS, the standard solution (e. g., L-SVEC solution for Li isotope; Qi et al., 1997) can be directly determined, whereas for isotope ratio determination by LA-MC-ICP-MS, the solution sample cannot be directly determined and a solid standard (e. g., the calibration standard) is usually ablated, and the measured isotopic composition can be calculated using the following equation (Steinmann et al., 2019; Kasemann et al., 2005).

$$\delta_{\mathrm{std}}^{\mathrm{sam}} = \left(r_{\mathrm{sam}}/r_{\mathrm{cali\text{ - }std}} - 1\right) \times 1\,000 + \delta_{\mathrm{std}}^{\mathrm{cali\text{ - }std}}$$

where, $\delta_{\mathrm{std}}^{\mathrm{sam}}$ refers to the isotopic composition of the measured sample relative to the reference standard solution, r_{sam} and $r_{\mathrm{cali\text{-}std}}$ refer to the isotopic composition of the measured sample and the calibration standard, and $\delta_{\mathrm{std}}^{\mathrm{cali\text{ - }std}}$ refers to the isotopic composition of the calibration standard relative to the reference solution. However, Lin et al. (2019) used the formula to calculate the isotopic composition of Li in tourmaline, noting that the calculation method introduced large errors when the isotopic compositions of the calibration standard and samples differ significantly and need to be corrected accordingly. This problem was subsequently sorted out by Zhang and Hu (2020), who pointed out that the above calculation method should only be used if the isotopic compositions of the sample and the calibration standard are similar or if the isotopic composition of the calibration standard is close to 0‰. The universal and accurate correction method can be used as the following equation.

$$\delta_{\mathrm{std}}^{\mathrm{sam}} = \left(r_{\mathrm{sam}}/r_{\mathrm{cali\text{ - }std}} - 1\right) \times 1\,000 + \delta_{\mathrm{std}}^{\mathrm{cali\text{ - }std}} \times \left(r_{\mathrm{sam}}/r_{\mathrm{cali\text{ - }std}}\right)$$

the key to isotopic composition correction by the external standard method is the selection of a standard that matches the actual sample matrix. This means that the fractionation factor of the standard can be approximated to that of the sample to ensure accurate isotopic fractionation correction. The commonly used USGS and MPI-DING glasses have been successfully used in the accurate calibration of a wide range of isotopes. For example, the determination of the isotopes of Li (Lin et al., 2017, 2016a; Le Roux, 2010), B (Kimura et al., 2016), Mg (Lin et al., 2022; Oeser et al., 2014), Si (Frick et al., 2016), Fe (Oeser et al., 2014). In contrast, the geological samples are the most complex and diverse, and the commonly used reference glass cannot meet the needs of isotopic analysis in geological samples. The new standards including natural mineral samples as well as synthetic standards have been developed. To provide a reference for future isotopic analysis in different geological samples, the existing isotope standards with different isotopes were compiled in Table 2.

Table 2 The commonly used reference materials for *in situ* isotopic determination

Isotopes	Mineral	Standard name	References
Li	Garnet glass	WHS01-06	Hoover et al. (2021)
	Andesite glass	ARM-1/2/3	Wu et al. (2021)
	Tourmaline	Elbaite #98144	Wiedenbeck et al. (2021)
		Schorl #112566	
		IAEA-B-4	
		IMR RB1	
	Clinopyroxene	SAE152	Tang et al. (2007)
		CpxBZ226/CG	Decitre et al. (2002)
		06JY06 /29 /31 cpx	Su et al. (2015)
		CPXA/B01	Yang A et al. (2023)
	Orthopyroxene	06JY06/31 /34 opx	Su et al. (2015)
		Opx BZ226	Decitre et al. (2002)
	Zircon	MNHN#146260	Ushikubo et al. (2008)
		M257	Li X H et al. (2011)
	Olivine	MW-sc	Tang et al. (2007)
		OlBZ29	Decitre et al. (2002)
		SC-1 Ol	Kobayashi et al. (2004)
		Ol334	Tang et al. (2014)
		09XDTC1-24	Su et al. (2015)
		06JY06/29/31/34 Ol	
B	Tourmaline	IAEA B4	Tonarini et al. (2003)
		IMR RB1	Hou et al. (2010)
		DD-01	Lin et al. (2014)
		Dravite #108796	Dyar et al. (2001)
		Elbaite #98144	
		Danburite #115089	
		Schorl #112566	
	Obsidian	IAEA B6	Gonfiantini et al. (2003)
	Andesite glass	ARM-1/2/3	Wu et al. (2021)
C	Calcite	SXD8	Chen W et al. (2017)

Table 2 Continued

Isotopes	Mineral	Standard name	References
	Dolomite	DOL-8	Lu et al. (2022b)
	Magnesite	MGS-1	
	Siderite	SD-5	
Mg	Olivine	San Carlos	Norman et al. (2004)
		ALM-1/2	Pearson et al. (2006)
		ALM 3	Beyer et al. (2006)
		Brenham	Norman et al. (2006); Young et al. (2009)
		Pallasite	Mittlefehldt et al. (1998); Wasson et al. (1999)
	Dolomite	DOL-8	Lu et al. (2022a)
	Siderite	SD-5	
	Magnesite	MGS-3	
	Carbonate	JCp-1	Inoue et al. (2004)
		JCt-1	
		eBlue	Sadekov et al. (2020)
		eYel	
Si	Quartz mineral	NBS 28	Ding et al. (2005)
		Qinghu-Qtz	Li et al. (2013)
	Si	IRMM 017	Frick et al. (2016)
	Quartz glass	Glass-Qtz	Liu et al. (2022)
	Zircon	Qinghu-Zir	Li et al. (2013)
	Diopside glass	Penglai-Zir	Li et al. (2010)
		JER	Reid et al. (2001)
S	Pyrite	PPP-1	Gilbert et al. (2014b)
		PY-4	Bao et al. (2017)
	Synthetic sulfide	MASS-1	Wilson et al. (2002)
		PS-1	Wilson et al. (2002)
	Synthetic Ag_2S	IAEA-S-1	Ding et al. (2001)
	Sphalerite	NBS123	Chen K Y et al. (2017)
		SPH-1	Lü et al. (2022b)
	Galena	NWU-GN	Lü et al. (2022b)
	Chalcopyrite	GC-1	Lü et al. (2022b)
		HTS4-6	Li et al. (2020)
		CPY-1	Li et al. (2020)
		TC1725	Bao et al. (2021)
	Ag_2S	IAEA-S-1/2/3	Ding et al. (2001)
		GBW04414/04415	Ding et al. (2001)
	S	IAEA-S-4	Qi and Coplen (2003)
	Barite	NBS127	Halas and Szaran (2001)
Cl	Apatite	Eppawala-AP	Li et al. (2020)
		Durango	McDowell et al. (2005)
Ca	Calcium phosphate	SRM 1486	Zhang L et al. (2020)
	Igneous apatite	Durango	Zhang L et al. (2020)
	Hydroxyapatite	SRM 1400	Tacail et al. (2016)
		HAPs	Tacail et al. (2016)
Ti	Ti	Alfa-Ti	Huang et al. (2022)
	Rutile	KNW	Huang et al. (2022)
		USNM 83191	Simon et al. (2017)
	Ilmenite	PZH12-15	Huang et al. (2022)
	Synthetic glass	CMAS	Williams et al. (2016)
V	V	Alfa Aesar V metal	Schuth et al. (2017)

Table 2 Continued

Isotopes	Mineral	Standard name	References
Fe	Fe	IRMM-014	Taylor et al. (1992)
		IRMM-524A	Xu et al. (2022b)
	Pyrite	JMC	Hirata and Kon (2008)
	Ilmenite	PZH12-15	Xu et al. (2022b)
		PZH12-18	Xu et al. (2022b)
Ni	Ni steel	D-184-1	Weyrauch et al. (2017)
		NIST 1226	Weyrauch et al. (2017)
	Ni	Ni-rod	Weyrauch et al. (2017)
Cu	Chalcopyrite	TC1725	Bao et al. (2021)
	Cu	NIST SRM 976	Ikehata and Hirata (2013)
		SSC-1, 3, 4	Yang et al. (2021)
		CUPD-1	Yang et al. (2021)
Zn	Zn	NIST SRM 683	Yang et al. (2018)
	Sphalerite	NBS 123	Bao et al. (2022)
Sr	Synthetic glass	CPX05G	Tong et al. (2016)
	Apatite	Durango	Zhang et al. (2018)
		AP1	Yang et al. (2014)
		Slyudyanka	
		SDG	
	Clinopyroxene	JJG1424	Zhao et al. (2020)
		YY09-47	
		YY09-04	
		YY09-24	
		YY12-01	
		HNB-8	Zhang et al. (2018)
	Potassium feldspar	Tuyk	Zhang et al. (2018)
	Carbonate	NanoSr	Weber et al. (2020)
		MNP	Liang et al. (2023)
	Scheelite	HTPW	Li et al. (2018)
		XJSW	Li et al. (2018)
	Plagioclase	AMNH-107160	Mulder et al. (2023)
		G29958	
		Hrappsey 14-2	
		BDL-1/2/3	Xu et al. (2022a)
		YG0440	Zhang et al. (2018)
		YG0383	Zhang et al. (2018)
		YG4301	Zhang et al. (2018)
	Epidote	PSV	Huang et al. (2023)
Zr	Zircon	GJ-1	Jackson et al. (2004)
		91500	Wiedenbeck et al. (1995)
		Plešovice	Sláma et al. (2008)
		Tanz	Hu et al. (2021)
		Paki	Zhang S H et al. (2022)
		Aus	
		Mala	
		Ban-1	Xie et al. (2023)
Sn	Sn	Sn rod	Schulze et al. (2017)
	Cassiterite	synthetic cassiterite	Zhang et al. (2023)
Ba	Barite	NBS127	Zhang S H et al. (2022)
		IAEA-SO-5,6	

Table 2 Continued

Isotopes	Mineral	Standard name	References
Nd	Titanite	SP-REN, SP-HUL	Foster and Vance (2006)
		BLR-1 OLT1 Ontario MKED1 T3	Ma et al. (2019)
	Allanite	DAI	McFarlane and McCulloch (2007)
	Synthetic glass	JNdi-1 LREE	Fisher et al. (2011b)
	Apatite	Durango	Kimura et al. (2013a)
		MAD	Yang et al. (2014)
	Monazite	Trebilcock	Xu et al. (2015)
Hf	Zircon	91500	Wiedenbeck et al. (1995)
		GJ-1	Jackson et al. (2004)
		Mud Tank	Black and Gulson (1978)
		Plesovice	Sláma et al. (2008)
		Temora	Black et al. (2003)
		QingHu	Li et al. (2013)
		M127	Klötzli et al. (2009)
		M257	Nasdala et al. (2008)
		CZ3	Kinny et al. (1991)
		CN92-1	Feng et al. (1993)
		SK10-2	Yuan et al. (2003)
		Tanz	Hu et al. (2021)
		Jilin	Luo et al. (2021)
		SLZA SLZB SLZC	Hu et al. (2023)
		SA01	Huang et al. (2020)
		SA02	Huang et al. (2021)
		BB	Santos et al. (2017)
		Ban-1	Xie et al. (2023)
		KV01	Wei et al. (2020)
	Rutile	RMJG	Zhang and Hu (2020)
		R10	Luvizotto et al. (2009)
		Sugluk-4 PCA-S207	Bracciali et al. (2013)
		JDX	Li Q L et al. (2011)
	Eudialite Titanzircon	LV01 ZrKA	Wu et al. (2010)
Os	Sulfide	CR-1 CO-1 COR-1 POR-1	Zhu et al. (2016)
U-Pb	Zircon	91500	Wiedenbeck et al. (1995)
		GJ-1	Jackson et al. (2004)
		Mud Tank	Black and Gulson (1978)
		Plesovice	Sláma et al. (2008)
		Temora	Black et al. (2003)

Table 2 Continued

Isotopes	Mineral	Standard name	References
		QingHu	Li et al. (2013)
		M127	Klötzli et al. (2009)
		M257	Nasdala et al. (2008)
		CZ3	Kinny et al. (1991)
		CN92-1	Feng et al. (1993)
		SK10-2	Yuan et al. (2003)
		Tanz	Hu et al. (2021)
		Jilin	Luo et al. (2021)
		SLZA	Hu et al. (2023)
		SLZB	
		SLZC	
		SA01	Huang et al. (2020)
		SA02	Huang et al. (2021)
		BB	Santos et al. (2017)
		Ban-1	Xie et al. (2023)
		KV01	Wei et al. (2020)
	Xenotime	MG-1	Fletcher et al. (2004)
		Z6413	Stern and Rayner (2003)
		XN01	Vasconcelos et al. (2018)
		XN02	
	Rutile	R10	Luvizotto et al. (2009)
		Sugluk-4	Bracciali et al. (2013)
		PCA-S207	
		RMJG	Zhang L et al. (2020)
		R632	Axelsson et al. (2018)
		JDX	Li Q L et al. (2011)
	Allanite	Tara	Gregory et al. (2007)
		AVC	Barth et al. (1994)
		Bona	von Blackenburg (1992)
		Daibosatsu	Hoshino et al. (2005)
		EM-1	Rubatto et al. (2011)
		LE40010	Smye et al. (2014)
		LE2808	
		Mucrone	Cenki-Tok et al. (2011)
		Swiss	von Blackenburg (1992)
		LE2808	Yang et al. (2022)
		LE40010	
		A007	
		A011	
		A012	
	Apatite	AP1	Yang et al. (2014)
		AP2	
		Durango	McDowell et al. (2005)
		MAD	Thomson et al. (2012)
		Otter Lake	Chew et al. (2011)
		NW-1	Zhou et al. (2013)
		Slyudyanka	Chew et al. (2011)
		UWA-1	Zhou et al. (2007)
		McClure Mountain	Chew et al. (2011)
		SDG	Zhou et al. (2012)

Table 2 Continued

Isotopes	Mineral	Standard name	References
	Bastnaesite	K-9	Yang et al. (2014)
	Eudialite	LV01	Wu et al. (2010)
	Cerium-niobium perovskite	LOP01	Mitchell et al. (2011)
	Titanzircon	ZrKA	Wu et al. (2010)
	Perovskite	Ice River	Heaman (2009)
		Tazheran	Kinny et al. (1997)
		AFK	Wu et al. (2013)
	Titanite	BLR-1	Aleinikoff et al. (2007)
		OLT-1	Kennedy et al. (2010)
		Khan	Heaman (2009)
		GST	Kennedy et al. (2010)
		Ontario	Sun et al. (2012)
		Fish Canyon Tuff	Schmitz and Bowring (2001)
		T3	Ma et al. (2019)
		Pakistan	
		MKED1	Spandler et al. (2016)
	Potassium feldspar	Albany K-feldspar	Liebmann et al. (2023)
	Garnet	Willsboro	Seman et al. (2017)
		Mali	
		PL57	Li et al. (2022)
		WS20	Yang et al. (2018)
		QC04	Deng et al. (2017)
		IUC-1	Aysal et al. (2023)
		IUC-2	

3.1.3 Regression model calibration method

The regression model calibration method is a newly used *in situ* isotopic microanalysis calibration strategy. The linear regression method takes all collected isotope signals (signal intensity + blank intensity) and analyses them as a whole and derives a linear regression line from all instantaneous isotope ratios, the slope of which is used as the final obtained isotope ratios (Feng et al., 2022a; Fietzke et al., 2008a). The advantages of the regression model calibration method are that (1) the artificial subjective influence of setting signal integration regions is avoided; (2) all the obtained data during the experiment are used for the analysis; (3) the contribution of each data point to linear regression line depends on its signal intensity and both blanks and signals are equally valid; and (4) deviations from the linear regression line due to transient signal fluctuations in isotopic analysis can be detected. Fietzke et al. (2008b) applied this method to LA-MC-ICP-MS for the Sr isotopic analysis of carbonates and showed that the method was able to improve the analytical precision by a factor of five, with an increase in accuracy. Feng et al. (2022a) coupled the regression model calibration method with a short-time, high-frequency laser ablation approach to the Sr and Hf isotope analysis in AMD, MACS-3, Plešovice, and GJ-1, and the obtained results were consistent with the recommended values within the certainty. In addition, the regression model calibration method has been widely used in other instruments, such as liquid chromatography coupled with MC-ICP-MS (Guéguen et al., 2015); gas chromatography coupled with mass spectrometry (Renpenning et al., 2015); and capillary electrophoresis coupled with MC-ICP-MS (Martelat et al., 2018).

3.2 Development of Standard Materials

The importance of standards is evident, not only for the SSB calibration method but also for monitoring data quality and instrumental drift correction. However, there are some problems with the use of standards for the analysis of different isotopes by LA-MC-ICP-MS, such as the depletion of existing standards during the determination (e.g., 91500; Hu et al., 2021), the limited variety of standards available and the low content of some elements. Therefore, it is imperative to obtain suitable standard materials (such as exploring the natural samples as standard samples or synthesizing standard materials using artificial synthesis techniques).

3.2.1 Exploring the natural sample

Exploring the homogeneous crystals or single minerals in nature as standards in LA-MC-ICP-MS analysis is the best way to satisfy the analysis of actual geological samples (Hu et al., 2021; Huang et al., 2020; Su et al., 2015). Natural samples are very close to the analyzed samples in terms of physical properties such as mineral color, transparency, hardness, and similar content of major trace elements, all of which are advantages that synthetic standards are difficult to get. For example, Su et al. (2015) collected five olivine samples, three plagioclase samples, and three monazite samples, and concluded that these samples could be used as Li isotopic standards for SIMS and LA-MC-ICP-MS with the homogeneity tests for elemental content and isotopic composition. Hu et al. (2021) obtained the largest international sample of natural zircon crystal (Tanz) for high-quality U-Pb dating and Zr-O-Hf isotopic analysis.

However, due to the complexity of the geological environment, the environmental requirements for mineral growth, and the relatively high cost of finding geological samples in the field, exploring homogeneous crystals or minerals in nature is rare. Therefore, artificially prepared synthetic standards are indispensable for the future development of LA-MC-ICP-MS standards for each isotope (Liu et al., 2013).

3.2.2 Powder pressing and high-temperature melting techniques

Common techniques for the preparation of synthetic standards include powder pressing techniques (Fu et al., 2016; Zhu et al., 2016) and high-temperature melting techniques (Wu et al., 2021).

The powder pressing technique refers to grinding sample particles to the micron-nanometer level, selectively mixing them with a binder, and mechanically polymerizing them together under conditions of applied pressure. For example, Zhu et al. (2016) used pyrite powder (GBW07267) and chalcopyrite (GBW07268) mixed with different concentrations of Ir, Re, and Os solutions and applied the powder pressing technique to develop four standards (CR-1, CO-1, COR-1, and POR-1) that can be used in the determination of the Re-Os isotopic composition of sulfides. Bao et al. (2017) mixed natural pyrite, chalcopyrite, and sphalerite with small amounts of galena respectively, and then ground the samples into ultrafine powders of several microns in diameter, and obtained homogeneous standards for sulfur and lead isotopes after pressing the tablets.

High-temperature melting technique refers to the melting of an initial powder sample at high temperatures, followed by rapid quenching to form a glass after homogenization of the isotopes in the melt. This method was divided into flux-melting techniques (Bayon et al., 2009) and flux-free melting techniques (Shimizu et al., 2011). With the development of high-temperature heat source equipment and methods to suppress the loss of highly volatile elements, the flux-free melting technique has become the dominant method for the preparation of LA-(MC)-ICP-MS standards. For example, Wu et al. (2021) used a flux-free high-temperature melting technique to synthesize three andesite standards with high homogeneity in the isotopic composition of Li-B-Si-O-Mg-Sr-Nd-Hf-Pb. The advantages of the high-temperature melting method are that the laser ablation behavior of the developed glass standards is close to that of natural minerals, the laser ablation signal is stable, easy to preserve and polish, and easy to use. The USGS series of silicate glasses (Jochum et al., 2005) and MPI-DING silicate glasses (Jochum et al., 2006) were successful examples of the high-temperature melting technique.

The use of the powder pressing technique for the development of reference materials was a simple process that allows the preparation of a large number of initial powders at the same time to meet practical needs, and in particular, the latest preparation method used ultrafine powders to achieve binder-free compacts, which have been widely promoted for the development of reference materials (Wu et al., 2018; Mukherjee et al., 2014). However, the internal porosity of the standard obtained from the powder pressing technique remains, and the mechanical strength of the standard was weaker than that of natural minerals or high-temperature melted glass, which would result in a higher ablation rate than that of natural samples, leading to a mismatch in signal intensity between the samples and causing severe fractionation effects of elements and isotopes as the ablation pits become deeper. In addition, the standards produced by the powder pressing technique were prone to oxidation and swelling and can be easily cracked and broken in the air, making them difficult to preserve. There are also problems with the high-temperature melting method of developing standards, for example, the sulfide glasses prepared by this method are often loose and fragile, and the sulfide glasses obtained by the high-temperature and high-pressure technique, although dense, are usually small in size, making it impossible to obtain a sufficient number of samples for distribution in the same batch. In addition, there is isotopic fractionation between different batches of melted samples during the development process (Zhang et al., 2017). Thus, the suitable producing process of the standard should be chosen based on the properties of the analyzed samples.

3.2.3 The method of spark plasma sintering

Spark plasma sintering technique (SPS) is an emerging technique for the preparation of solid standard samples, which involves the simultaneous application of a pulsed current and pressure to a mold containing an initial powder sample, resulting in a dense, grain growth-inhibited solid sample development method. The rapid and efficient heating by current applied to the sample at temperature of 1 200 °C and less than 100 MPa allows the sample grains to clump together without major phase change processes (Champion, 2013). This spark plasma sintering technique allows a significant reduction in the porosity of the sample, resulting in sintered materials that reach or exceed 98% of the theoretical maximum density. Tacail et al. (2016) used this method to prepare natural and synthetic hydroxyapatite as a standard for in situ Ca isotope testing and experimentally verified that the Ca isotope composition was homogeneous. Feng et al. (2022b) showed that the plasma spark sintered pyrite and chalcopyrite had a smoother surface, denser structure, and similar laser exfoliation behavior to natural sulfides and that the Fe and S isotopic compositions of pyrite and chalcopyrite were homogeneous. Based on the high- efficiency and practicality of the plasma spark method, the method has full potential for the preparation of isotopic standards by LA-MC-ICP-MS.

Furthermore, the 3D printing method (Gao et al., 2023) and sintering method (Onuk et al., 2017) can also be used as the preparation method of standards. For example, the elemental-homogeneity standards can be produced by Gao et al. (2023). Sphalerite standards can be made with the resulting powder mixture being pressed into pellets and sintered at 400 °C for 100 h using argon as an inert gas by Onuk et al. (2017).

3.3 Exploration of Non-matrix Matched Calibration Method

In addition to the development of matrix-matched isotopic standards for isotopic fractionation calibration, analytical geochemists have recently explored methods to achieve non-matrix-matched fractionation calibration, which can largely avoid the development of matrix-matched standards and greatly improve analytical efficiency. Commonly used main methods to reduce matrix effects to achieve non-matched calibration include the use of femtosecond lasers and the use of wet plasma.

3.3.1 Adopting the femtosecond laser

The pulse width (energy duration) of the femtosecond laser (fs, 10^{-15}s) is one hundred thousandth of the pulse width of a nanosecond laser (ns, 10^{-9}s). The short residence time of the femtosecond laser pulse on the sample surface greatly reduces the thermal effects during ablation and thus reduces fractionation at the ablation pits. In addition, the extremely high peak power of the femtosecond laser (10^{15} W/cm^2) allows for aerosol particle size in the nanometer range to be obtained from the ablation. The production of small aerosol particles improved the transport and ionization efficiency of the aerosol, thereby increasing the signal intensity of the ions, and thus improving the analytical accuracy and spatial resolution, as well as reducing the isotopic fractionation and matrix effects during laser ablation (Kimura et al., 2017; Poitrasson and d'Abzac, 2017; Shaheen et al., 2012; Hergenröder, 2006; Zeng et al., 2005). It showed that the femtosecond laser has significantly improved in terms of laser ablation capability, particle size distribution, sensitivity, fractionation effect, and spatial resolution. Currently, femtosecond laser ablation systems have been successfully applied for the *in situ* isotopic microanalysis of Li (Lin et al., 2019; Steinmann et al., 2019), Mg (Lin et al., 2022; Oeser et al., 2014), Fe (Xu et al., 2021; Zheng et al., 2018; Oeser et al., 2014; Steinhoefel et al., 2009a; Horn et al., 2006), Si (Schuessler and von Blanckenburg, 2014; Chmeleff et al., 2008), Cu (Lazarov and Horn, 2015; Ikehata et al., 2008), Ti (Huang et al., 2022), and Sr (Zhang et al., 2018). Although previous studies have shown that the use of femtosecond laser can effectively eliminate or reduce matrix effects in isotope analysis (Oeser et al., 2014; Chmeleff et al., 2008; Horn et al., 2006). However, matrix effects have also been reported during femtosecond laser ablation. Ikehata et al. (2008) used an infrared wavelength femtosecond laser to analyze the Cu isotopic composition of pure copper, hematite, pyroxene, and chalcopyrite and found that the use of pure copper as an external standard accurately corrected the instrumental mass fractionation of hematite. However, it resulted in systematic deviations in the copper isotopic determination of pyroxene and chalcopyrite (0.5‰ to 2.0‰). Similar matrix effects were also reported by Ikehata and Hirata (2013) and Zheng et al. (2018). Therefore, femtosecond laser-induced isotope fractionation and matrix effects need to be further investigated.

3.3.2 Adopting the Wet Plasma

Wet plasma mode is used, whereby a small amount of water is added before and after the ablation cell during the laser ablation process to form the wet plasma (Fig. 2). The use of wet plasma increases the tolerance of ICP to mass loading and is one of the main ways to achieve LA-MC-ICP-MS non-matrix-matched isotope calibration. Among others, O'Connor et al. (2006) showed that the use of wet plasma conditions improved the plasma tolerance, and both mass loading effects and matrix effects are greatly reduced. Oeser et al. (2014) and Schuessler and von Blanckenburg (2014) realized non-matrix-matched correction of Mg, Fe, and Si isotopes using wet plasma. Zheng et al. (2018) used ns- and fs-LA-MC-ICP-MS to determine the Fe isotopic composition of magnetic pyrite and magnetite using pure Fe as calibration standard, respectively, and showed that the matrix effect can be reduced under the

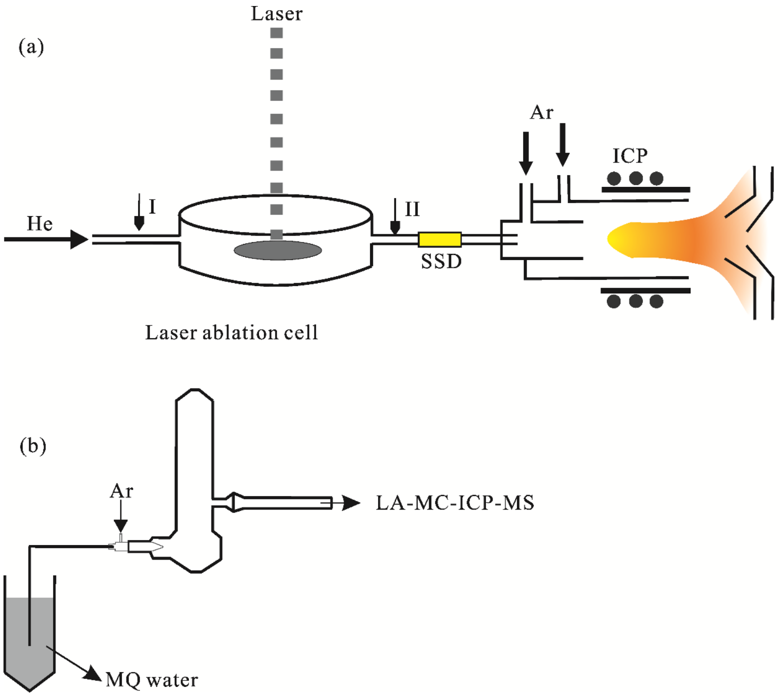

Figure 2. Diagrams showing the wet plasma. I. Water was added before the ablation cell using the ns-laser; II. water was added after the ablation cell using the fs-laser (modified from Lin et al., 2019).

condition of adding water after the ablation cell of the fs-LA-MC-ICP-MS. Luo et al. (2018) used 193 nm and 213 nm-LA-ICP-MS for U-Pb dating of single mineral and innovatively used the addition of water before the ablation cell to achieve NIST 610 glass corrections of U-Pb dating in zircon, titanite, and apatite. Lin et al. (2019) used ns-LA-MC-ICP-MS and fs-LA-MC-ICP-MS to achieve the NIST 610 glass correction of Li isotopes in tourmaline with the addition of water before and after the ablation cell with the non-matrix-matched calibration. Lü et al. (2022a) pointed out that non-matrix-matched calibration of Cu isotopes in Chalcopyrite by Cu metal sheet can be realized under the condition of wet plasma. In the determination of isotopic compositions by LA-MC-ICP-MS, the application of wet plasma not only reduces the matrix effects but also reduces polyatomic ion interference. For example, Lin et al. (2022), in the determination of Mg isotopic composition by fs-LA-MC-ICP-MS, noted that the use of a wet plasma can significantly reduce the $^{12}C^{14}N^+$ interference to ^{26}Mg, allowing accurate determination of Mg isotope even at low mass resolution.

In addition to the application of femtosecond laser and wet plasma for non-matrix-matched calibration, line ablation mode can be used to reduce the mass fractionation effect during laser ablation; low laser energy density (1–3 $J \cdot cm^{-2}$) can be used to ensure stable ablation (Lazarov and Horn, 2015); focusing the laser lower than the ablation position to avoid the ionization of He above the sample which consumes laser energy (Steinmann et al., 2019; Lazarov and Horn, 2015); adjusting the laser ablation conditions to ensure that the signal intensity of the standard and the sample are identical and that the same mass loading is applied can also reduce the matrix effect (Lin et al., 2017; Chmeleff et al., 2008). A variety of these measures can also significantly reduce the matrix effect, thus facilitating the implementation of non-matrix-matched calibration.

4 DATA REDUCTION SOFTWARE

With the isotopic mass fractionation and interference in the LA-MC-ICP-MS, suitable data reduction software is required to efficiently process laser ablation isotope data in a visual format. Currently, most data reduction software is only used in elemental content calculations (Mischel et al., 2017; Guillong et al., 2008; Jackson, 2008), U-Th-Pb dating (Petrus and Kamber, 2012; Andersen, 2008), and map scanning analysis (Petrus et al., 2017; Sforna and Lugli, 2017), but there are not much software available for LA-MC-ICP-MS data reduction (Table 2). ICPMSDataCal (Liu et al., 2008), Iolite v4 (Paton et al., 2011) and Iso-Compass (Zhang W et al., 2020) are three commonly used data reduction software.

ICPMSDataCal is compatible with calibration methods in the software for isotopic systems such as Li, Sr, Nd, Hf, Os, and Pb. And the software was written in the Visual Basic programming language and works on Microsoft Excel. This software integrates all the above calculation and correction methods for LA-MC-ICP-MS analysis of isotopic ratios. It gives users a unique analytical environment, based on the interactive selection of background and sample intervals from the time-resolved signals provided by (MC)-ICP-MS. It provides real-time and online data reduction for the LA-MC-ICP-MS analysis, and features linked graphics and analysis tables, greatly improving both productivity and the flexibility of analysis. At present, data analyzed by (MC)-ICPMS of Agilent, thermo X and Neptune, Elan, Varian, Nu Plasma, and AttoM can be directly used without additional adjustment. In addition, the software is upgraded and modified based on the different requirements

Iolite v4 is a successful commercial software based on iolite (Paton et al., 2011). It can import Almost all instrument data and run as independent software without the support of the operating platform. By arranging the data in time series, it is

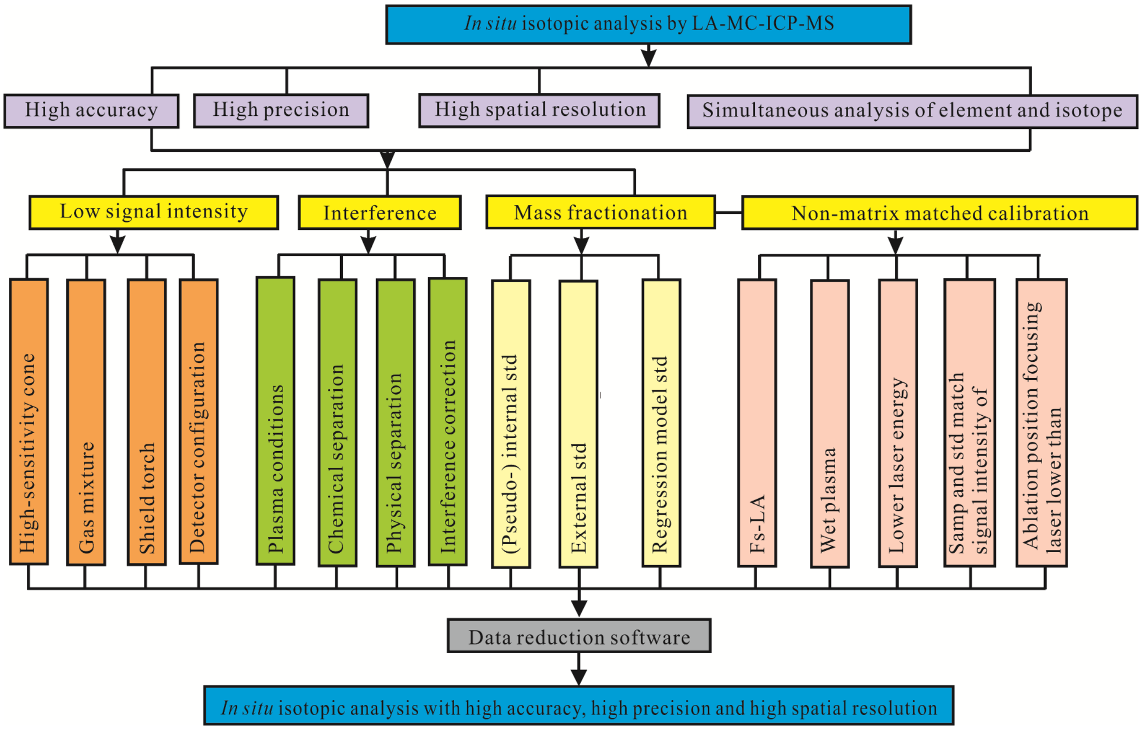

Figure 3. The problems and solutions of accurate isotope determination by LA-MC-ICP-MS.

easy to achieve batch processing of a large amount of data and the restoration of high spatial resolution information, supporting the rendering of surface scanned images of the mapping analysis. By optimizing drift correction and excellent deep fractionation correction (Paton et al., 2010), high-precision single-point isotope ratio measurement results can be obtained. To date, the Iolitev4 has been widely used in U-Th-Pb dating and Hf isotopic calculation. In addition, as a data processing process template, Iolitev4 supports secondary development using Python language, and in its latest version, it also supports database storage, greatly facilitating data storage and comparison.

Iso-compass is non-commercial software for isotope data reduction in LA-MC-ICP-MS, it is implemented using the programming language C#, which can be used independently on a Windows system without installing other commercial software. This software can provide highly practical functions, including a simple data input interface, a manually defined selection of laser and background signals, background correction, internal or external mass bias correction, interference correction, visual data presentation, and formatted data output. These functions can achieve an easier and more efficient reduction of LA-MC-ICP-MS data. The flexible formula editing mode enables Iso-Compass to be used in different isotope systems, thereby offering the potential to become a universal isotope data reduction platform for the geochemical community.

In summary, due to the differences in experimental processes and calibration methods, it is difficult to compare the results and errors obtained by the different software. For high-precision isotope ratio measurement, it is crucial to provide appropriate calibration methods and convenient data reduction software.

5 CONCLUSIONS AND OUTLOOK

LA-MC-ICP-MS is the method of choice for the *in situ* isotopic microanalysis. The isotopes of twenty-three kinds of elements have been accurately determined using LA-MC-ICP-MS. This review integrated the problems and solutions of accurate isotope determination for low signal-intensity samples, mass spectral interferences, isotope fractionation, and non-matrix-matched correction by LA-MC-ICP-MS (Fig. 3). This review aims to promote the further application of LA-MC-ICP-MS in analytical geochemistry, improve the accuracy and precision of the existing analyzable isotopes, and realize the accurate determination of more elements' isotopes.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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