

RESEARCH ARTICLE

Influence of room temperature on magnesium isotope measurements by multi-collector inductively coupled plasma mass spectrometry

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Rationale: We observed that the accuracy and precision of magnesium (Mg) isotope analyses could be affected if the room temperature oscillated during measurements. To achieve high-quality Mg isotopic data, it is critical to evaluate how the unstable room temperature affects Mg isotope measurements by multi-collector inductively coupled plasma mass spectrometry (MC-ICP-MS).

Methods: We measured the Mg isotopes for the reference material DSM-3 using MC-ICP-MS under oscillating room temperatures in spring. For a comparison, we also measured the Mg isotopes under stable room temperatures, which were achieved by the installation of an improved temperature control system in the laboratory.

Results: The $\delta^{26}\text{Mg}$ values measured under oscillating room temperatures have a larger deviation ($\delta^{26}\text{Mg}$ from -0.09 to 0.08% , with average $\delta^{26}\text{Mg} = 0.00 \pm 0.08\%$) than those measured under a stable room temperature ($\delta^{26}\text{Mg}$ from -0.03 to 0.03% , with average $\delta^{26}\text{Mg} = 0.00 \pm 0.02\%$) using the same MC-ICP-MS system.

Conclusions: The room temperature variation can influence the stability of MC-ICP-MS. Therefore, it is critical to keep the room temperature stable to acquire high-precision and accurate isotopic data when using MC-ICP-MS, especially when using the sample-standard bracketing (SSB) correction method.

1 | INTRODUCTION

Multi-collector inductively coupled plasma mass spectrometry (MC-ICP-MS) has been widely used to measure metal stable isotopic compositions in recent decades (e.g.¹⁻¹²). Compared with gas source mass spectrometry and thermal-ionization mass spectrometry (TIMS), MC-ICP-MS has the considerable advantages of high ionization efficiency and high sensitivity (e.g.^{3,4,13}). However, we can obtain precise and accurate isotopic data when using MC-ICP-MS only if the instrumental mass bias (defined as the deviation of the measured isotope ratio from the true value) is accurately corrected for. Many parameters such as instrumental settings, matrix effects, concentration mismatch, and acidity mismatch can produce significant uncertainty for mass bias correction (e.g.¹⁴⁻²¹), resulting in poor precision and accuracy for isotopic data. In particular, it is important to achieve high precision (e.g., 2SD less than 0.05%) for metal stable

isotopes analyses in order to reveal small isotope fractionations in high-temperature geochemical processes. Thus, it is important to carefully investigate the effects of all the factors that can potentially influence the precision of isotope measurements.

When checking the temperature variation and Mg isotopic data measured in the last two years in our laboratory (USTC in Hefei, China), we found that the precision of the Mg isotopic data was poor when the room temperature was oscillating quickly (see Figures 1 and 2). These results infer that the variation in room temperature can potentially sabotage Mg isotope measurements. To acquire highly precise isotopic data, it is important to know the influence of oscillating room temperature on isotopic analysis.

Generally, based on our records, rapid room temperature oscillation mainly occurred during spring and autumn with large diurnal temperature variation. To test the effect of room temperature oscillation on isotopic analyses, we have measured Mg isotopes in

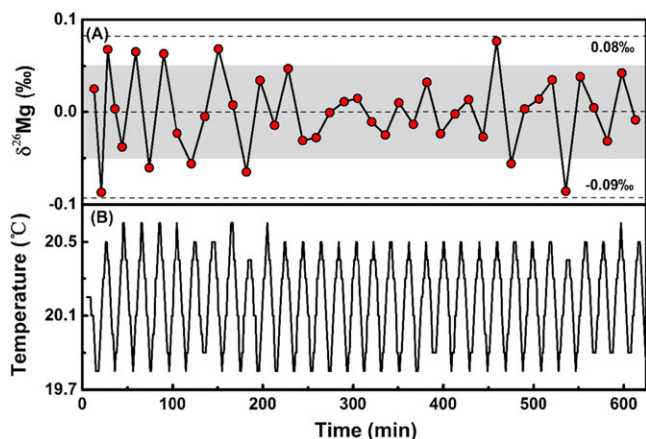


FIGURE 1 (A) Magnesium isotopic data under oscillating room temperatures. The red circles in (A) are $\delta^{26}\text{Mg}$ values of DSM-3, which vary from -0.09 to 0.08‰ with an average value of $0.00 \pm 0.08\text{‰}$ (2SD , $n = 42$). The gray area shows the long-term precision of Mg isotope measurements in our laboratory ($0.00 \pm 0.05\text{‰}$, 2SD). (B) The record of room temperature variation within 625 min; the room temperature could increase or decrease by 0.7°C within 10 min [Color figure can be viewed at wileyonlinelibrary.com]

spring, when the room temperature could quickly oscillate to a large extent when there were no auxiliary facilities to help further control the room temperature. We chose Mg isotopes for this test because Mg isotopes have been extensively used to study processes of cosmochemistry, deep carbon recycling, magma differentiation, and chemical weathering (e.g. $^{22-28}$). Some of these processes only slightly fractionate the Mg isotopes ($\sim 0.1\text{‰}$ for $\delta^{26}\text{Mg}$ values), and thus they require a very high precision of Mg isotope measurements ($< 0.05\text{‰}$, 2SD). Compared with heavy elements (e.g. strontium, cadmium and barium), there are larger relative mass differences between the Mg isotopes (^{24}Mg , ^{25}Mg and ^{26}Mg), and this may result in more significant mass discrimination during analyses. In particular, the sample-standard bracketing (SSB) correction method³ cannot effectively correct the instrumental mass bias

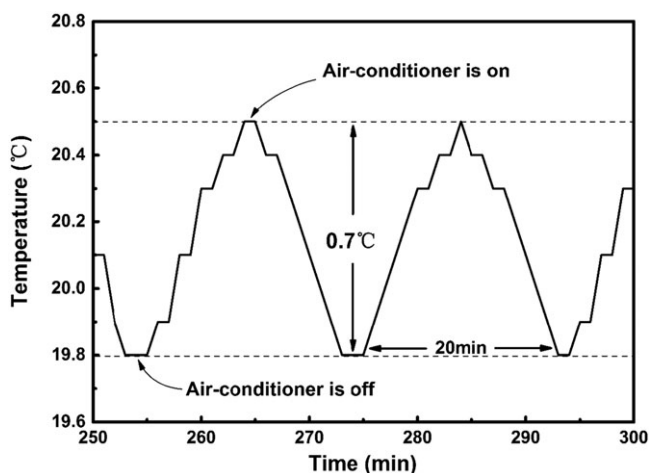


FIGURE 2 The pattern of room temperature oscillating in the Instrumental Room. In a period of 20 min, the room temperature increased from 19.8°C to 20.5°C after the central air-conditioner stopped working. The room temperature then decreased to 19.8°C after the air-conditioner restarted working. The whole process was repeated to produce the oscillating room temperature in the Instrumental Room

for Mg isotope measurements obtained with unstable room temperatures (Figures 1 and 2). Therefore, we improved our temperature control system (Figure 3), achieving a smooth variation in the temperature (e.g. less than $0.2^\circ\text{C}/\text{h}$). A comparison of Mg isotopic data revealed the critical role of stable room temperature in producing much better precision of Mg isotope analyses (Figure 4).

2 | MAGNESIUM ISOTOPE ANALYSES

The Mg isotopic data collected in this study were acquired using a Neptune Plus high-resolution multi-collector ICP mass spectrometer (Thermo Fisher Scientific, Bremen, Germany) at the CAS Key Laboratory of Crust-Mantle Materials and Environments at the University of Science and Technology of China (USTC) in Hefei, China. SK-L200TH II α temperature recording devices (SATO Keiryoki Mfg Co Ltd, Tokyo, Japan) were used to record the room temperatures during the analyses.

The Mg isotopes were measured following the procedure reported in An et al.²¹ The sample introduction system included a PFA MicroFlow nebulizer with uptake rate of $\sim 50\ \mu\text{L}\ \text{min}^{-1}$ and a quartz dual path cyclonic spray chamber (both from Elemental Scientific, Omaha, NE, USA), and a jet sampling and an H skimmer cone (Thermo Fisher Scientific). Analyses were carried out in low-resolution mode. ^{24}Mg , ^{25}Mg and ^{26}Mg were collected simultaneously in the L3, C and H3 Faraday cups, respectively. The sensitivity of ^{24}Mg is $\sim 30\ \text{V}\ \text{ppm}^{-1}$.

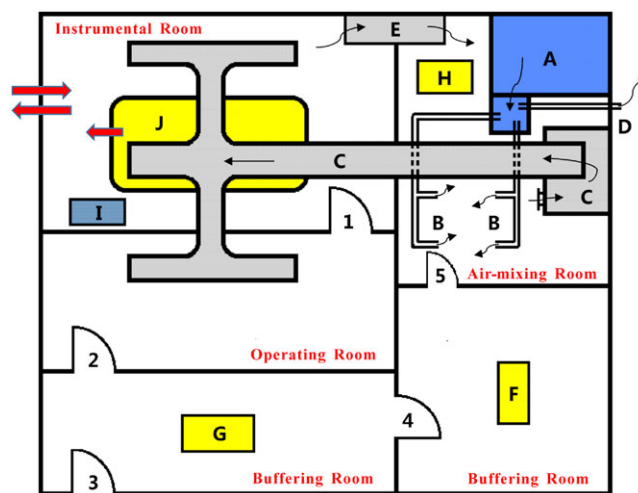


FIGURE 3 Sketch of the temperature control system of CAS Key Laboratory of Crust-Mantle Materials and Environments in USTC. Number 1 to 5 represent the doors in different rooms. A: the central air-conditioning unit, which is the main system producing cold air. B: air ducts, which can send the cold air produced by the central air-conditioning unit to the Air-mixing Room. C: air duct, which can send cold air from the Air-mixing Room to the Instrumental Room. D: air duct, which can exhaust cold air from the Air-mixing Room to outside the laboratory. E: a system, which can send the air from the Instrumental Room to the Air-mixing Room. F, G: two auxiliary air-conditioners. H: auxiliary electric heater. I: temperature recording device. J: the MC-ICP-MS instrument which is a stable source of heat during working. Black arrows are the air flow direction from the central air-conditioning unit to different rooms. The red arrows are the possible heat flux in the room [Color figure can be viewed at wileyonlinelibrary.com]

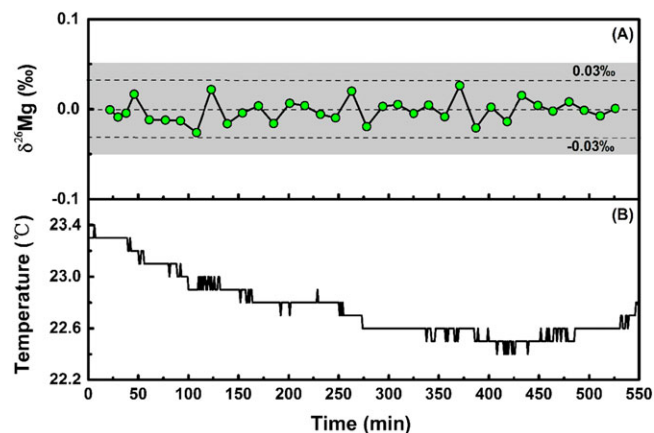


FIGURE 4 (A) Magnesium isotopic data measured under stable room temperature. The green circles in (A) are $\delta^{26}\text{Mg}$ values of DSM-3, which vary from -0.03 to 0.03‰ with an average value of $0.00 \pm 0.02\text{‰}$ (2SD, $n = 35$). The gray area shows the long-term precision of Mg isotope measurements in our laboratory ($0.00 \pm 0.05\text{‰}$, 2SD). (B) The record of room temperature within 550 min; the room temperature changes by less than $0.2^\circ\text{C}/\text{h}$ [Color figure can be viewed at wileyonlinelibrary.com]

The Mg isotope ratios were measured for one block with 60 cycles, with each cycle taking 2.097 s. The instrumental settings for data acquisition are summarized in Table 1.

The international reference material DSM-3 (supplied by Dr A. Galy, University of Cambridge, Cambridge, UK) was measured during all the analyses as both samples and bracketing standards. The details of DSM-3 are described in Galy et al.²⁹ Using this pure Mg solution can eliminate the influence of matrix effects on isotopic measurements. Using a single DSM-3 solution can also eliminate the influence of mismatch in concentration and acidity. The mass bias of the instrument was corrected by the SSB method, which assumes that the mass bias of the analyzed sample is the same as the average mass bias of the bracketing standards analyzed immediately before and after the

TABLE 1 Instrumental parameters for Mg isotope measurements

Instrumental parameters	Neptune plus (USTC)		
RF power	1200 W		
Cooling gas (Ar) flow rate	$\sim 16 \text{ L min}^{-1}$		
Auxiliary gas (Ar) flow rate	$\sim 0.8 \text{ L min}^{-1}$		
Sample gas (Ar) flow rate	$\sim 1.0 \text{ L min}^{-1}$		
Extraction voltage	-2000 V		
Vacuum	$4\text{--}8 \times 10^{-9} \text{ Pa}$		
Mass resolution	Low-resolution		
Typical ^{24}Mg sensitivity	30 V ppm^{-1}		
^{24}Mg background	$< 5 \text{ mV}$		
Cones	H skimmer cone, Jet sampler cone		
Desolvator	Quartz spray chamber		
Installed nebulizer	Teflon self-aspirating micro-nebulizer system		
Sample uptake	$\sim 50 \text{ uL min}^{-1}$		
Detector mode	Faraday cup static mode		
	L3-F	C-F	H3-F
Amplifier resistor	$10^{11}\Omega$	$10^{11}\Omega$	$10^{11}\Omega$
	^{24}Mg	^{25}Mg	^{26}Mg

sample.³ For the calculation, one DSM-3 measurement was treated as an unknown sample, and the average values of the 'before and after' measurements of DSM-3 were used as bracketing standards, expressed in the delta-notation:

$$\delta^{26}\text{Mg} = \left[\frac{2 \times \left(\frac{^{26}\text{Mg}/^{24}\text{Mg}}{\left(\frac{^{26}\text{Mg}/^{24}\text{Mg}}_{\text{former}} + \left(\frac{^{26}\text{Mg}/^{24}\text{Mg}}_{\text{latter}} \right) \right)_{\text{DSM-3}}}}{1} \right) - 1 \right]$$

If the mass bias does not change linearly during the entire sample-bracketing analysis batches as a result of environmental oscillation, the average of $\left(\frac{^{26}\text{Mg}/^{24}\text{Mg}}_{\text{former}} \right)$ and $\left(\frac{^{26}\text{Mg}/^{24}\text{Mg}}_{\text{latter}} \right)$ of the bracketing DSM-3 solution should not be equal to $\left(\frac{^{26}\text{Mg}/^{24}\text{Mg}}_{\text{DSM-3}} \right)$. Therefore, we would expect an apparent deviation of the calculated $\delta^{26}\text{Mg}$ value from zero.

3 | RESULTS

3.1 | Mg isotope measurements under unstable room temperature

Before we improved our temperature control system, we observed poor precision of Mg isotopic data under rapidly oscillating room temperatures (e.g. Figure 1). The $\delta^{26}\text{Mg}$ values of DSM-3 varied from -0.09 to 0.08‰ with average $\delta^{26}\text{Mg} = 0.00 \pm 0.08\text{‰}$ (2SD, $n = 42$) (Figure 1A) when the room temperature oscillated by 0.7°C within 20 min (Figures 1B and 2). Such precision is not good enough for the studies of many geochemical processes. The observation indicates that when the room temperature varied quickly, the instrumental stability could be sabotaged, which could affect the Mg isotopic data corrected by the SSB method.

The oscillating room temperature was produced by the working mechanism of the air-conditioning system in our laboratory. The wall of our lab is not well heat-insulated. Therefore, in summer, the temperature outside the lab was so high that the air-conditioner would keep working without stop, while the outside temperature in winter was so low that the room temperature could be below the setting range of the air-conditioner even without running it. In these two seasons, the room temperature does not oscillate on most days. However, in spring and autumn, the temperature outside the lab changes dramatically from the daytime to the night. In these two seasons, when the central air-conditioner starts to work, it usually provides excess refrigerating power and the room temperature decreases quickly to the lower limit (e.g. 19.5°C) of the setting range (e.g. $20.0 \pm 0.5^\circ\text{C}$). When the temperature sensor in the Instrumental Room sent a feedback to the air-conditioning unit, the compressor of the air-conditioning unit stopped working. Because the heat continuously produced from the MC-ICP-MS instrument was accumulated in the Instrumental Room, the room temperature would increase quickly. If the temperature of the Instrumental Room increased above the upper limit (e.g. 20.5°C) of the setting range, the air-conditioning unit subsequently restarted working to quickly cool down the room. The whole process would then be repeated to produce frequent oscillations in room temperature (Figures 1B and 2).

3.2 | Mg isotope measurements under stable room temperature

To effectively control the room temperature of the Instrumental Room (e.g. variation $<0.2^{\circ}\text{C}/\text{h}$), a temperature control system was constructed in our laboratory. A sketch of this system is presented in Figure 3, and the details of the instrumentation are listed in Table 2. The essential idea of this temperature control system is to maintain a balance between the heat flux from the MC-ICP-MS instrument, the air-conditioning system and the surrounding environment. In the Instrumental Room, the MC-ICP-MS instrument generally produces a stable amount of heat while it is working (J in Figure 3). As the building wall is not well insulated, the environment outside the building can strongly affect the room temperature in different seasons or even in the course of one day (Figure 3). In this case, the amount of cooling air from the air-conditioning system (A in Figure 3) into the Instrumental Room (Figure 3) should be accurately adjusted by the temperature control system to achieve a balance.

As shown in Figure 3, the cold air is transferred to different locations through a number of ducts (B, C and D in Figure 3). After being produced by the air-conditioner, the cold air is first transferred to the Air-mixing Room through duct B (Figure 3), and mixed with the air in the Air-mixing Room. If there is excess cold air, it can be exhausted to outside the laboratory through duct D (Figure 3). All the valves at the ends of air ducts B and D (Figure 3) can be rotated from 0° to 90° to control the flux of cooling air. Therefore, the amount of cold air transferred into different locations can be controlled by adjusting the valves. In the Air-mixing Room, an auxiliary electric heater (H in Figure 3) can help heat the mixing air when the temperature outside the building is too low in winter. In this way, the temperature in the Instrumental Room is always slightly higher than the lower limit of the setting range, and the central air-conditioner keeps working to provide a stable source of cold air (A in Figure 3). If the temperature in the Mixing Room is too high in summer, two auxiliary air-conditioners (F and G in Figure 3) can help cool down the mixed air before it is transferred into the Instrumental Room. The mixed air in the Air-mixing Room is then transferred to the Instrumental Room through duct C (Figure 3), and the air in the Instrumental Room is sent back to the Air-mixing Room through system E to form an internal circulation (Figure 3).

With this system, the variation rate of the room temperature can be controlled to less than $0.2^{\circ}\text{C}/\text{h}$. High-precision Mg isotope measurements can be achieved under this stable room temperature

TABLE 2 Equipment for the temperature-controlled system at the CAS Key Laboratory of Crust–Mantle Materials and Environments in USTC

Equipment	Model	Manufacturer
Central air-conditioner	PEX air-cool R22 unit	Emerson-Liebert (Shanghai, China)
Auxiliary air-conditioner	KFRd-71QW/620A	Haier (Qingdao, China)
Auxiliary electric heater	Electrical oil heater	Midea (Guangzhou, China)
Temperature recording device	SK-L200TH II α	SATO Keiryoki Mfg Co Ltd

(Figure 4). The $\delta^{26}\text{Mg}$ values of DSM-3 calculated by the SSB method vary from -0.03 to 0.03‰ with an average value of $0.00 \pm 0.02\text{‰}$ (2SD , $n = 35$).²¹

4 | DISCUSSION

In this study, two sets of Mg isotopic data measured under stable and unstable room temperatures were quantitatively investigated (Figures 1 and 4). Comparison of these observations suggests that the rapidly oscillating room temperature (increasing or decreasing 0.7°C within 10 min) could affect Mg isotope measurements by MC-ICP-MS, especially when using the SSB correction method.

The SSB method has been commonly applied to correct the instrumental mass bias during isotope measurements, and it is especially important for elements with less than four isotopes (e.g. Li, Mg, V and Cu).¹³ Using the SSB method requires that the instrumental mass bias is constant or changes linearly with time.³ This means that the accuracy and precision of the SSB method completely rely on the stability of instrumental mass bias, as clearly shown by the poor precision of Mg isotope measurements with rapidly oscillating room temperatures (Figure 1).

Previous studies proposed that mass bias in ICP-MS could originate in the sample introduction and transportation processes, and different processes occurring in the ICP.¹⁶ The space-charge effect is often considered as a dominant factor in causing instrumental mass bias because of the preferential transmission of heavier ions.^{30–33} A previous study also implied that any factors affecting plasma stability could cause variation in the instrumental mass bias.¹⁵ It is possible that the oscillating room temperature may influence the stability of the ICP torch or the ion transportation processes during isotopic measurements, and may further affect the stability of the instrumental mass bias. Under a rapidly oscillating room temperature, the instrumental mass bias cannot be stable or change linearly, which results in significant uncertainties for isotope analyses using the SSB method (Figure 1).

This effect of room temperature oscillation may also occur in other isotope measurements with the SSB correction method, such as lithium, iron, silicon, vanadium and copper. Therefore, a stable room temperature condition is necessary for achieving isotopic data with high precision and accuracy.

5 | CONCLUSIONS

We observed that oscillating room temperature could affect MC-ICP-MS instrumental stability. If the room temperature varies rapidly (e.g. varying by 0.7°C within 10 min), it is obviously harmful for metal stable isotope measurements. Under oscillating room temperature, the SSB correction method has poor precision for Mg isotope measurement. This may be due to inconsistent instrumental mass bias of samples with standards. High-precision Mg isotope measurements require a stable room temperature (e.g. varying less than $0.2^{\circ}\text{C}/\text{h}$) (Figure 4). Therefore, we recommend maintaining stable room temperature conditions (viz. varying by less than $0.2^{\circ}\text{C}/\text{h}$) for metal stable isotope measurements by MC-ICP-MS when using the SSB method. Such a condition can be achieved by balancing the heat flux of the Instrumental Room.

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