

Precise determination of magnesium isotopes

Introduction

Magnesium (Mg) isotopes are critical to research into questions as diverse as crustal contamination within volcanic arcs and heart disease in humans. We present the results of Mg isotope measurements performed on the Plasma 3 HR-MC-ICP-MS (Figure 1), illustrating the ability of the instrument to determine precise Mg isotope ratios.

Instrumentation

Two experiments were undertaken. In the first, a single 450 ppb Spex Certiprep Mg solution was analysed repeatedly over approx. 2.5 hours. In the second, a 385 ppb Spex Certiprep Mg solution was analysed as a sample against a 350ppb Spex Certiprep Mg solution bracketing standard. In both experiments the solutions were introduced into the mass spectrometer under 'dry plasma' conditions using an Aridus 3 system with a 0.1 mL/min C-Flow nebuliser and Nu Instruments standard ES cones.

Data was collected on Faraday detectors fitted with $10^{11} \Omega$ amplifiers using static analysis. Each analysis consisted of one block of 50 integrations of 5 second on-peak measurement on Mg, totalling approx. 4 minutes. For the single concentration experiment, an on-peak zero measurement was performed using a 2% HNO_3 blank solution at the beginning of the sequence. Each analysis during the variable concentration experiment was preceded by an on-peak zero measurement in a 2% HNO_3 blank solution and was followed by a 3 minute wash in 2% HNO_3 . No tuning of the instrument or sample introduction system was made once the experiments had started. Measurements were carried out on the interference-free shoulders of the ^{24}Mg , ^{25}Mg and ^{26}Mg peaks in Pseudo-high resolution mode (40 μm source defining slit) with a resolving power of approx. 6700 at 15% transmission (Figure 2). Isotopes were analysed according to the cup configuration in Table 1.



Figure 1: Nu Plasma 3

Collector	HMD	H10	X	X	H9	X	H8	H7	H6	H5	H4	H3	H2	H1	Ax	L1	L2	L3	L4	L5	D0	D1	D2	X	D3	X	D4
Position	-14	-13	-12	-11	-10	-9	-8	-7	-6	-5	-4	-3	-2	-1	0	1	2	3	4	5	6	7	8	9	10	11	12
Mg									26					25						24							

Table 1: Plasma 3 collector configuration for the static measurement of Mg isotopes. H/Ax/L = Standard Faraday detectors. X = Dummy bucket. D = Daly.

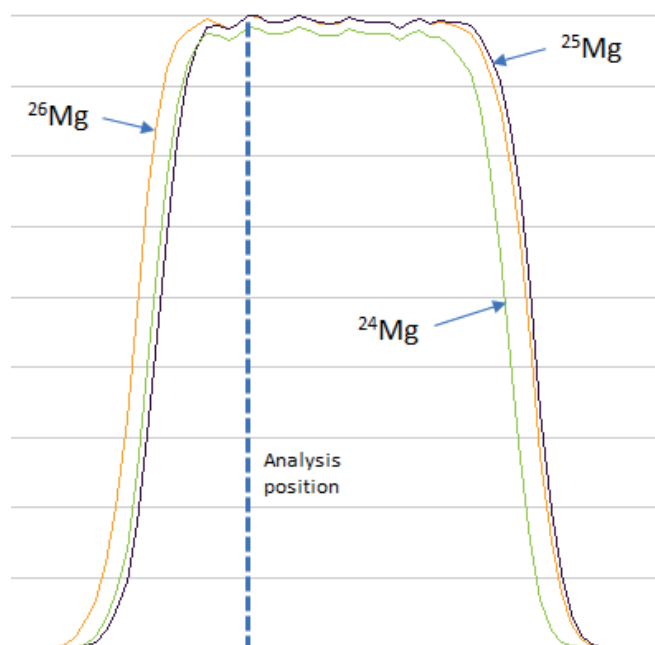


Figure 2: Measurement position on the interference-free shoulders of the three Mg peaks.

Discussion

Bracketing results for the single-concentration experiment are shown in Table 2, where δ indicates the per mil deviation of the measured 'sample' analysis ratio relative to the average of the two 'standard' bracketing analysis ratios measured immediately before and after.

	$^{25}\text{Mg}/^{24}\text{Mg}$ (meas)	$^{26}\text{Mg}/^{24}\text{Mg}$ (meas)
Average δ (‰)	0.00	-0.01
SD (‰)	0.01	0.01

Table 2: Results of 33 repeated analyses of 450ppb Spex Certiprep Mg solution, bracketed against itself.

The total Mg sensitivity achieved in Pseudo-high resolution mode was approx. 85 V/ppm (15% transmission; equivalent to 565 V/ppm at 100% transmission). Ratios were internally normalised to a $^{25}\text{Mg}/^{24}\text{Mg}$ ratio of 0.126633, and the corrected $^{26}\text{Mg}/^{24}\text{Mg}$ ratios are plotted in Figure 2. As can be observed from Figure 3, these ratios display a linear correlation ($R^2 = 0.9959$) on a log-log plot whose slope is within uncertainty of the theoretical value (0.5), consistent with mass-dependent fractionation.

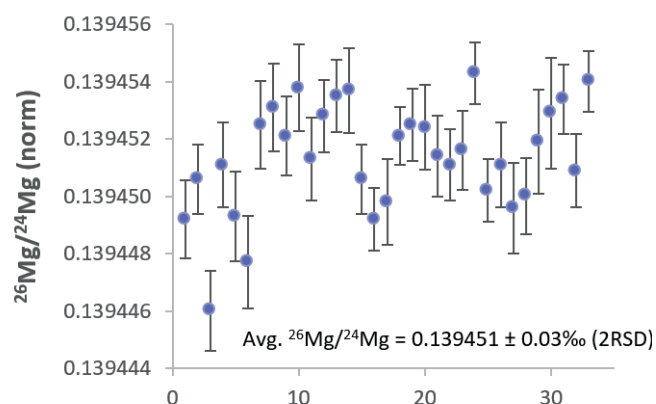


Figure 2: Internally-normalised $^{26}\text{Mg}/^{24}\text{Mg}$ ratios for the single concentration experiment.

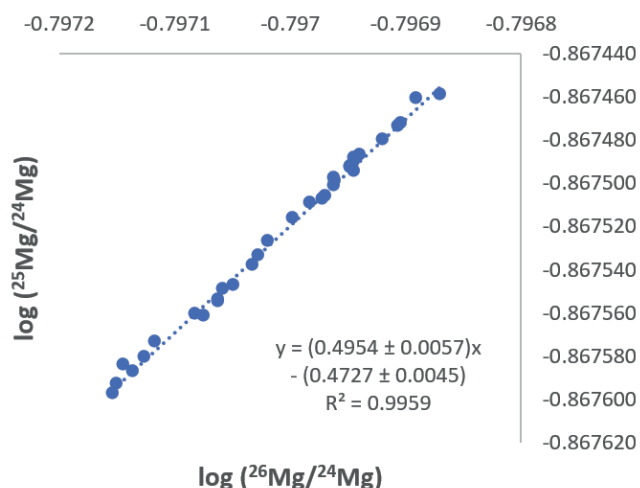


Figure 3: Log-log plot of the unnormalised Mg ratios measured in the single concentration experiment.

Bracketing results for the variable-concentration experiment are shown in Table 3, using the same notation as before. This time, 6 analyses of a 385 ppb Mg solution were analysed by standard-sample bracketing against a 350 ppb solution (Figure 4). It is well known that a concentration mismatch between standard and sample can induce an additional matrix effect, resulting in elevated delta values⁽¹⁾. However, these results demonstrate that standard-sample bracketing of Mg on the Plasma 3 can yield accurate results even with a concentration mismatch of 10%, as the measured ratios track each other through time.

	$^{25}\text{Mg}/^{24}\text{Mg}$ (meas)	$^{26}\text{Mg}/^{24}\text{Mg}$ (meas)
Average δ (‰)	0.01	0.03
SD (‰)	0.02	0.02

Table 3: Results of analysing a 385 ppb Mg solution, bracketed against a 350 ppb solution.

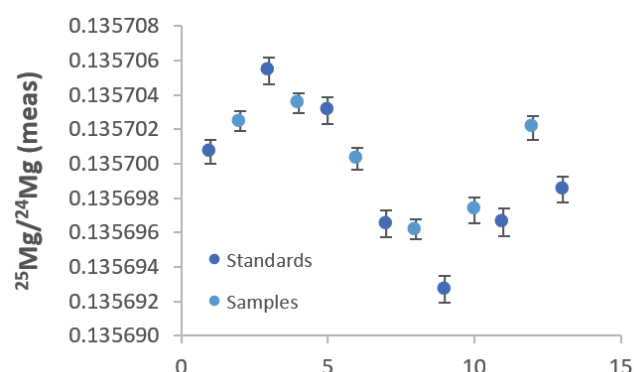


Figure 4: Measured $^{25}\text{Mg}/^{24}\text{Mg}$ ratios for the standard (350 ppb) and sample (385 ppb) Mg solutions.

Conclusions

This study demonstrates the capability of the Plasma 3 for precise Mg isotope measurements, even when there is a 10% mismatch between the standard and sample solution concentrations.

References

[1] Teng, F.-Z. and W. Wang (2014). Comparison of factors affecting the accuracy of high-precision magnesium isotope analysis by multi-collector inductively coupled plasma mass spectrometry. Rapid Comms. Mass Spec. 28(1), 19-24.