

# ELIMINATING ANALYTICAL UNCERTAINTY: ANALYZING GEOLOGICAL STANDARDS USING INDUCTIVELY COUPLED PLASMA MASS SPECTROSCOPY

Adelaide Clark<sup>1,3</sup> and Fang-Zhen Teng<sup>2,3</sup>, <sup>1</sup>Emory & Henry College, Department of Chemistry, <sup>2</sup>University of Arkansas, Department of Geosciences and <sup>3</sup>Arkansas Center for Space and Planetary Sciences.

**Introduction:** It is important to determine if the Earth has a chondritic composition because this determination will “shed light on the accretion of the Earth” [1]. This chondritic composition is a subject of much debate. It is possible this disagreement is due to the analytical techniques used, with analysis by ICP-MS being more precise than laser ablation [2].

Multicollector-inductively coupled plasma mass spectroscopy (MC-ICP-MS) measures isotope ratio and has the ability to analyze a broad range of elements, unlike other techniques such as Atomic Absorption. The MC-ICP-MS is described as a “hybrid” mass spectrometer, with the combination of an inductively coupled plasma source, an energy filter, a magnetic analyzer and multiple collectors. The ICP source “strips” off electrons, leaving a positive ion which is focused into a beam via slits in electrostatic plates. This focused beams of ions pass through magnetic fields which separate ions based on the mass to charge ratio [3]. MC-ICP-MS gives an order of magnitude more precise than LA-ICP-MS, however, ICP-MS does lead to instrument bias in elements of low atomic number [4].

Laser ablation inductively coupled plasma mass spectroscopy (LA-ICP-MS), on the other hand, uses a high energy ultraviolet laser ablation system. This laser creates a crater in the object, anywhere from 2 to 1200 microns. The material from the crater is taken up from the sample into the plasma of the ICP-MS, where it is ionized in a similar fashion of the MC-ICP-MS. Sample preparation for this technique involves have substances of a known matrix and calibration curve, similar to the limitations of AA analysis [5].

Magnesium has three stable isotopes (24, 25 and 26), each with a relative abundance of 78.99, 10 and 11.01 % respectively. They have a relatively large mass difference of 4 – 8%. These stable isotopes have fractionation potential, a characteristic that must be absent in order to study basalts from the upper mantle, proving whether it is homogeneous or heterogeneous [6].

In an attempt to solve the analytical uncertainty mentioned above, twelve homogeneous standards are distributed to different labs worldwide to be tested using that labs analytical procedure.

**Experimental:** The chemical procedures of these experiments were carried out in a clean lab environment.

Approximately 10 mg of homogeneous standards (except GBW07101, GBW07102, GBW07122, and GBW07123 which were 1 mg only) were dissolved in a ~3:1 concentrated HF-HNO<sub>3</sub> mixture in Savillex, screw-top beakers and then heated overnight at 160 – 180°C on a hot plate in a hood. The solutions were evaporated until completely dry the following day at 160°C. They were then dissolved again in a ~3:1 concentrated HCl-HNO<sub>3</sub> mixture overnight and then evaporated to dryness. The samples were dissolved again in only concentrated HNO<sub>3</sub> overnight and evaporated to dryness. Finally, 1N HNO<sub>3</sub> was added to each dried sample, amounts varying depending on how much magnesium oxide each contains by weight percent [6].

In order to separate the magnesium from the standards, they were run through cation exchange chromatography columns. After being washed with 10 mL of MQ H<sub>2</sub>O and 5mL of 1N HNO<sub>3</sub> and then loaded with BioRad 200-400 mesh AG50W-X8 resin, which had been precleaned with >20 times column value of 4N HCl and MQ. The resin was loaded first in water, then with 1N HNO<sub>3</sub> added to it. The Mg was collected in Savillex screw-top beakers and evaporated to dryness before adding 100 µL of 1 N HNO<sub>3</sub> to perform this column work again.

The Mg isotopic ratio was determined using a Nu Plasma ICP-MS. The pure Mg samples obtained from the column work were dried down and had 1 mL of 3% HNO<sub>3</sub> added before being run through the ICP-MS.

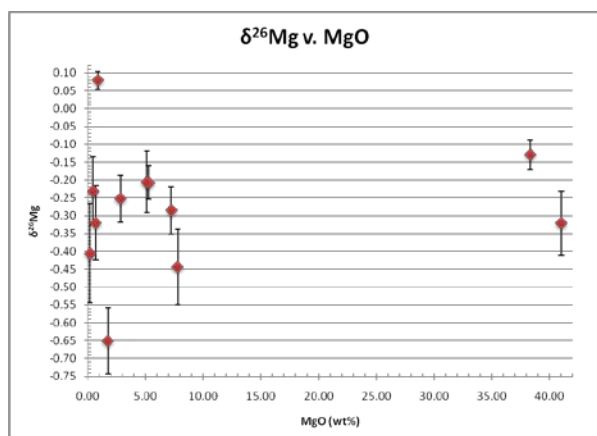
## Results:

Sample ID	$\delta^{26}\text{Mg} \pm 2\text{SD}$	MgO (wt%)
GBW07101	-0.32 ± 0.09	41.03
GBW07102	-0.13 ± 0.04	38.34
GBW07103	-0.23 ± 0.10	0.42
GBW07104	-0.65 ± 0.09	1.72
GBW07105	-0.44 ± 0.11	7.77
GBW07109	-0.32 ± 0.10	0.65
GBW07110	0.08 ± 0.02	0.84
GBW07111	-0.25 ± 0.07	2.81
GBW07112	-0.21 ± 0.05	5.25
GBW07113	-0.41 ± 0.14	0.16
GBW07122	-0.21 ± 0.09	5.08
GBW07123	-0.29 ± 0.07	7.20

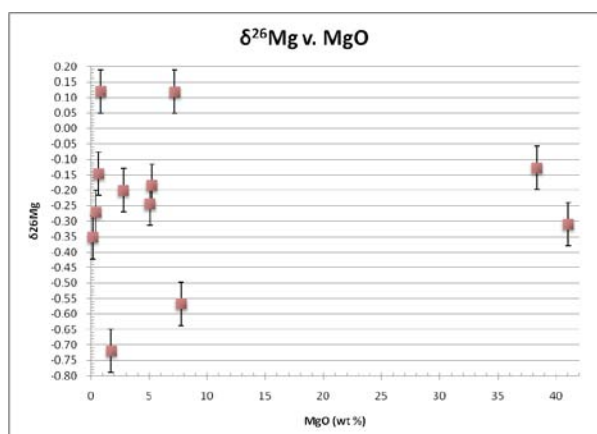
**Table 1:** Previous measurements and the MgO in each standard by weight percent.

Sample ID	Previous Value ( $\delta^{26}\text{Mg} \pm 2\text{SD}$ )	Measured Value ( $\delta^{26}\text{Mg} \pm 2\text{SD}$ )
GBW07101	$-0.32 \pm 0.09$	$-0.31 \pm 0.24$
GBW07102	$-0.13 \pm 0.04$	$-0.13 \pm 0.24$
GBW07103	$-0.23 \pm 0.10$	$-0.27 \pm 0.24$
GBW07104	$-0.65 \pm 0.09$	$-0.72 \pm 0.24$
GBW07105	$-0.44 \pm 0.11$	$-0.57 \pm 0.24$
GBW07109	$-0.32 \pm 0.10$	$-0.15 \pm 0.32$
GBW07110	$0.08 \pm 0.02$	$0.12 \pm 0.24$
GBW07111	$-0.25 \pm 0.07$	$-0.20 \pm 0.24$
GBW07112	$-0.21 \pm 0.05$	$-0.18 \pm 0.24$
GBW07113	$-0.41 \pm 0.14$	$-0.35 \pm 0.24$
GBW07122	$-0.21 \pm 0.09$	$-0.24 \pm 0.24$
GBW07123	$-0.29 \pm 0.07$	$0.12 \pm 0.32$

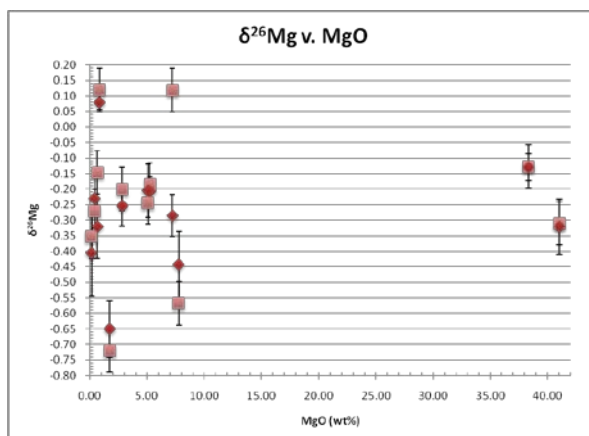
**Table 2:** The previous data compared to this experiment's data.



**Figure 1:** Previous standard measurements in our lab with error bars of two standard deviations.



**Figure 2:** Data for this experiment with error bars of two standard deviations.



**Figure 3:** Composite of Figure 1 and Figure 2 to illustrate overlapping data ranges.

**Conclusion:** As can be seen in Table 1 and Figure 3, all the data except for one point (GBW07123) at least overlap in the measurement of  $\delta^{26}\text{Mg}$  per sample as measured in the ICP-MS. This shows that our data previously measured holds true when repeating experimental procedure.

Reasons for the data points of GBW07123 not overlapping could be due to the fact that this measurement was taken in a separate batch processed through the ICP-MS along with GBW07109. Both of these measurements were slightly off from the previously determined values, but GBW07109 from this measurement still overlaps the previous measurement.

Based on the data presented here, we can conclude that our measurements for these geological standards are accurate within reasonable uncertainty. Further experiments need to be carried out, however, to confirm that this data is in fact accurate, especially with regard to geological standards GBW07109 and, most importantly, GBW07123. This data must then be compared to data from an LA-ICP-MS laboratory to determine if technique is the cause of the disagreement.

**References:** [1] Teng, F. -Z., et al. *Geochim. Cosmochim. Acta* (2010). [2] Yang et al. *Earth and Planetary Science Letters* 288 (2009). [3] Science Education Resource Center. *Geochemical Instrumentation and Analysis: Multicollector Inductively Coupled Plasma Mass Spectrometer*. Retrieved July 22, 2010 from <http://serc.carl-eton.edu/18402>. [4] Huang et al. *Chemical Geology* (2009). [5] Koenig, Alan. *USGS: Laser Ablation ICP-MS Laboratory*. Retrieved July 22, 2010 from [http://minerals.cr.usgs.gov/icpms/laser\\_ablation.html](http://minerals.cr.usgs.gov/icpms/laser_ablation.html). [6] Teng, F. -Z., et al. *Earth and Planetary Science Letters* 261(2007) 84 – 92.

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