



## **Combined chemical separation of Mg, Ca and Fe from geological reference samples for stable isotope determination by MC-ICP-MS and double-spike TIMS**

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Near quantitative and matrix free separation of analyte elements is required to obtain precise and accurate stable isotope data by MC-ICP-MS. Here, we report a suitable procedure for the combined chemical separation of Mg, Ca and Fe and results for geological reference samples (water samples, carbonates, igneous & sedimentary rocks and one chondrite).

The chemical separation of Mg, Ca and Fe is carried out using 1 ml Biorad AG50W-X8 (200-400 mesh) cation exchanger. For Mg separation from silicate rocks and seawater, the samples are loaded in 0.4M HCl and many matrix elements are rinsed off with further 0.4M HCl. If needed Ti (+ Be & Al) and Mn (+ Fe & V) are separated from Mg using 0.15M HF and 0.95% Acetone-0.5M HCl, respectively. Subsequently Mg was recovered in 1M HCl. For Mg purification from carbonate samples and/or for additional Ca and Fe separation, the samples are first loaded in 10M HCl onto the ion exchange resin and Mg together with most matrix elements is eluted in the same media. Calcium is then eluted in either 6.6M HBr or 8M HCl, followed by Fe elution using 4M HCl.

Magnesium isotope analyses were accomplished using the Axiom MC-ICP-MS in Kiel. The following results have been obtained for  $\delta^{26}\text{Mg}$  relative to the DSM3 standard (Galy et al., *JAAS* 2003) in per mil. Uncertainties refer to the external long term reproducibility (2 SD):

IAPSO seawater  $-0.79 \pm 0.10$  (n=20); NASS-5 seawater  $-0.84 \pm 0.16$  (n=8); SLRS-4

river water  $-0.97 \pm 0.05$  (n=3); Cal-S limestone  $-4.38 \pm 0.31$  (n=14); JDo-1 dolomite  $-2.38 \pm 0.18$  (n=12); JcP-1 coral  $-2.01 \pm 0.22$  (n=37); NIST 1486 bone meal  $-1.45 \pm 0.14$  (n=2); MAG-1 marine mud  $-0.23 \pm 0.09$  (n=5); SGR-1 shale  $-0.98 \pm 0.12$  (n=6); BIR-1 basalt  $-0.23 \pm 0.23$  (n=9); BCR-2 basalt  $-0.14 \pm 0.11$  (n=4); BE-N basalt  $-0.41 \pm 0.19$  (n=3); DTS-1 dunite  $-0.25 \pm 0.08$  (n=3); UBN serpentinite  $-0.12 \pm 0.08$  (n=6); Murchison CM2 chondrite  $-0.26 \pm 0.09$  (n=17). A total range of  $\sim 4$  per mil was observed for  $\delta^{26}\text{Mg}$ , whereby the largest variation occurred in the carbonate samples. Data for seawater, BIR-1, BCR-2 and Murchison are consistent with published results (Young & Galy, *Rev. Min. Geochem.* 2004; Baker et al., *Nature* 2005).

Natural variations in  $^{44}\text{Ca}/^{40}\text{Ca}$  for the above reference materials (except NIST 1486; DTS-1 and IAPSO) were measured using the Triton TIMS in Kiel in conjunction with a  $^{43}\text{Ca}$ - $^{48}\text{Ca}$  double spike (Heuser et al., *Int. J. Mass Spectrom.* 2002). The external reproducibility was  $\pm 0.3$  per mil (2 SD) on average. A total variation of 1.4 per mil for  $^{44}\text{Ca}/^{40}\text{Ca}$  was observed.

First Fe isotope data on basalt reference samples using the Neptune MC-ICP-MS in Frankfurt are consistent with previous analyses (e.g. Weyer et al., *EPSL* 2005).