Realization of SI traceability for Mg isotope amount ratios & delta values

BAM
Bundesanstalt für Materialforschung und -prüfung

Jochen Vogl [1], Olaf Rienitz [2], Martin Rosner [3], Björn Brandt [1], Simone Kasemann [4], Rebecca Kraft [5], Dmitriy Malinovskiy [6], Anette Meixner [4], Janine Noordmann [2], Savelas Rabb [5], Jan A. Schuessler [7], Robert D. Vocke [5]
contact: jochen.vogl@bam.de

Problem

Due to heterogeneity issues with NIST SRM 980 a new scale defining standard, DSM3, was introduced into canonical use in 2003. This standard, however, is not SI-traceable, not calibrated against other standards and not commonly accessible.

Solution

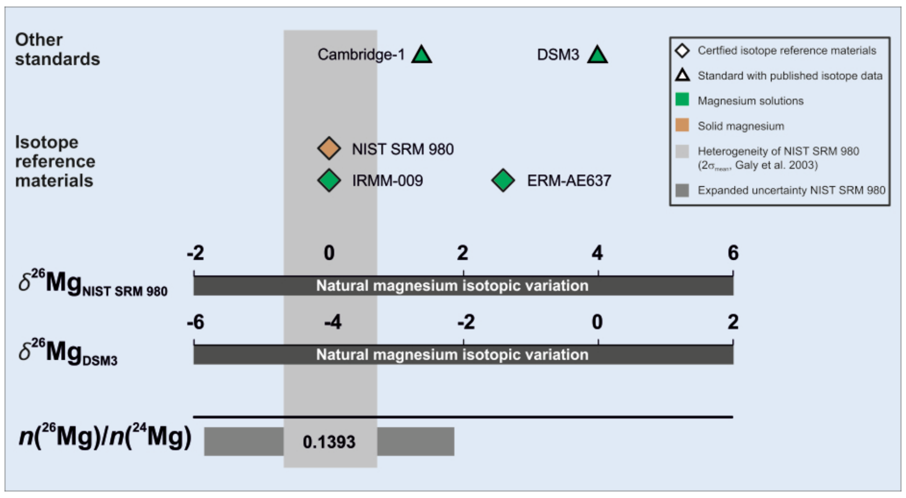
A new set of Mg isotope reference materials (iRM) that are fully traceable to the SI was certified. All other standards were then calibrated against them such, that they can be used equally well. The new iRMs are commercially available.

Previous situation

General

Accurate measurements of stable isotope ratio variations are often reported using artifact based δ-scales, which rely on suitable isotope reference materials (iRM) for their realization.

Variations in Mg isotope ratios in natural systems are typically reported as $\delta^{26/24}$Mg and $\delta^{25/24}$Mg values that represent the relative difference between the ^{26}Mg/^{24}Mg or ^{25}Mg/^{24}Mg ratio measured in a sample relative to its measurement in a δ-scale defining iRM.



Heterogeneity and traceability problem

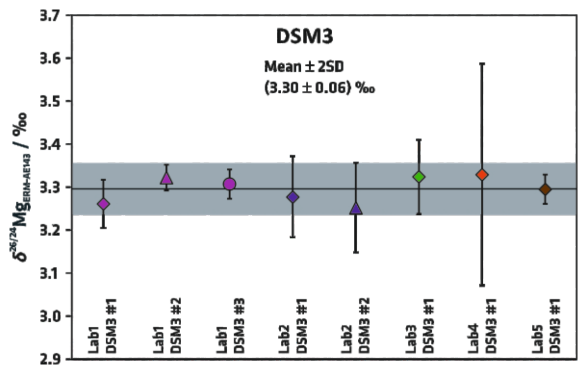
In the past, $\delta^{26/24}$Mg and $\delta^{25/24}$Mg measurements were referenced to NIST SRM 980, the initial zero of the Mg δ-scale. With the development of MC-ICP-MS, small but measurable isotopic differences in different chips of NIST SRM 980 became apparent [1].

The δ-scale defining iRM was then replaced by DSM3 [2], which unfortunately provides no $n(^{26}\text{Mg})/n(^{24}\text{Mg})$ and $n(^{25}\text{Mg})/n(^{24}\text{Mg})$ isotope amount ratios and thus is not traceable to the SI.

Applied approach & results

Absolute isotope ratios

Applying the isotope mixture approach for the calibration of MC-ICP-MS, three iRMs were certified for their Mg isotope amount ratios in an interlaboratory comparison [3,4]. For the first time, measurement uncertainties could be achieved which are close to the typical precision of δ-scale measurements (0.1 ‰, 2SD).



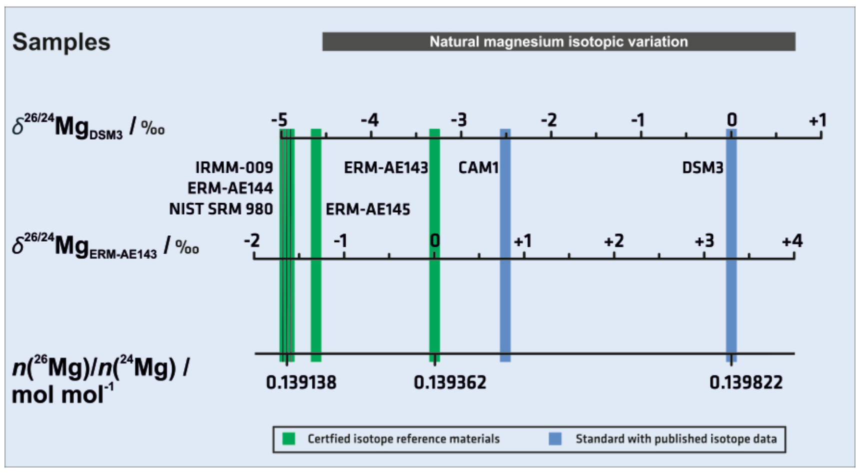
Calibration of Mg δ-scales

In a 2nd interlaboratory comparison, the δ-values of all currently used Mg iRMs (NIST SRM 980, IRMM-009) and internationally accepted standards (CAM1, DSM3) were determined against ERM-AE143. Thus for all materials, SI-traceable isotope amount ratios can be calculated and all Mg δ-scales can be interconverted into one another.

New situation

Primary isotope reference materials ERM-AE143, ERM-AE144 & ERM-AE145

- Certified isotope amount ratios $n(^{25}\text{Mg})/n(^{24}\text{Mg})$ & $n(^{26}\text{Mg})/n(^{24}\text{Mg})$
- Accompanied by an uncertainty budget with $u_{c,rel} \leq 0.015$ %
- **SI-traceability established**
- No homogeneity issues, because iRMs are Mg solutions (no solid material)
- Spread between ERM-AE143 and ERM-AE144 approximately 1.65 ‰ in $\delta^{26/24}$Mg



Mg δ-scales

- All δ-standards and thus all δ-scales are calibrated against each other
- Analysts are free to use either scale, the DSM3 or the ERM-AE143 scale
- **For continuity, however, it is recommended to keep the DSM3-scale and anchor it by ERM-AE143 at +3.30 ‰**
- Users are encouraged to apply ERM-AE143 for calibrating their own in-house standard to guarantee a long-lasting stock of the primary iRM

References

[1] Galy et al., *J Anal At Spectrom* 18 (2003) 1352-1356
[2] Brand et al., *Pure Appl Chem* 86 (2014) 425-467
[3] Brandt et al., *J Anal At Spectrom* 31 (2016) 179-196
[4] Vogl et al., *J Anal At Spectrom* 31 (2016) 1440-1458

Partner

1) BAM 2) PTB 3) IsoAnalysis UG 4) marum Universität Bremen 5) NIST 6) LGC 7) GFZ Helmholtz Centre Potsdam