Redox and pressure controls on iron isotope variations in MORBS determined by NRIXS spectroscopy

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Iron isotopic variations are commonly reported in materials produced in high temperature environments such as planetary mantles. However, a reliable database of equilibrium fractionation factors between melts and igneous minerals is still lacking to interpret the rock record. In the case of iron, these factors can be directly derived from Nuclear Resonant Inelastic X-ray Scattering (NRIXS) experiments at a synchrotron facility [1, 2] through the determination of mean force constants. This method is insensitive to kinetic effects during mineral and glass synthesis, contrasting with conventional experimental approaches [3].

Because iron is a multivalent element, its isotopes could be good tracers of redox conditions during melting [4, 5]. To establish Fe isotope systematics as a redox proxy, we used NRIXS to measure mean force constants of synthetic ^{57}Fe-labelled olivine and silicate glasses synthesized at different oxygen fugacities in a gas mixing furnace and in piston-cylinders at different pressures with and without dissolved water.

At a given Fe^{3+}/Fe^{2+} ratio, the force constants of tholeitic to andesitic glasses are almost identical. For all samples, the force constant increases linearly with Fe^{3+}/Fe_{tot}. The effect of pressure and water content is moderate. Thus, for mafic melts, there is little structural control; redox effects seem to dominate. Our data demonstrate that even at high temperature (1100 °C), the equilibrium δ^{56}Fe fractionation between the two oxidation states of iron in MORBS is large (+0.25 ‰). The iron isotopic fractionation factors derived from NRIXS data may explain the heavy iron isotopic compositions measured in MORBS.

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Approaching real uncertainty estimates for δ^{11}B data

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The measurement uncertainty is an indispensable quality criterion of isotope ratio data and sets important limits for the general use and geochemical interpretation of such data. Despite traceability to the International System of Units (SI) or an accepted standard an uncertainty statement allows the direct comparison of isotope data which have been obtained in different laboratories and/or using different procedures. To realise traceability and comparability of isotope data a realistic uncertainty statements should include uncertainty contributions from all influence quantities. In isotope geochemistry, however, often the term uncertainty is used for terms describing the statistical dispersion of measurement results such as precision, repeatability or reproducibility. Because an measurement uncertainty must include all influence quantities even the so-called external precision or reproducibility will underestimate the overall uncertainty. Without uncertainty statement traceability and comparability of measurement results cannot be established. In cases where no uncertainty for isotope ratio data is presented it is highly recommended that at least the measurement results of a quality control sample with known isotope composition are presented which can be used to evaluate the accuracy and roughly estimate uncertainty.

Here we present an approach to calculate uncertainty budgets for δ^{11}B data which can easily be used for other δ-values. Our approach considers uncertainty components from the mass spectrometric isotope ratio determination (repeatability and reproducibility), the chemical preparation (contamination, isotope fractionation) and the isotope reference material (or δ-zero material). Having quantified the single uncertainty components the identification of the most important contributors to the overall uncertainty is possible and strategies to improve the measurement uncertainty can be investigated. The here presented expanded uncertainties for δ^{11}B values between 1.2 and 0.4 ‰ of matrix standards (IAEA B1-3) and processed isotope reference materials (NIST SRM 951, ERM-AE101) show that typically intense sample preparation is the biggest uncertainty contributor. Uncertainties derived from unprocessed reference materials or standards will therefore significantly underestimate the uncertainty of real samples.

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