New offset δ^{11}B isotope reference materials for geochemical and environmental boron isotope studies

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The isotopic composition of boron is a well established tool in various areas of science and industries. Boron isotope compositions are typically reported as δ^{11}B values which indicate the isotopic difference of a sample relative to the internationally accepted isotope reference material NIST SRM 951. A significant drawback of all of the available boron isotope reference materials is that non covers a natural boron isotope composition apart from NIST SRM 951.

To fill this gap of required offset δ^{11}B reference materials we produced three new solution boric acid reference materials that cover two-thirds of the natural boron isotope variation (-20 to 40 ‰ δ^{11}B) of about 100 ‰. The new reference materials are certified for their δ^{11}B values. The certified δ^{11}B values of -20.2 ‰ for ERM-AE120, +19.9 ‰ for ERM-AE121 and 39.7 ‰ for ERM-AE122 have been established by three independent analytical methods (gravimetric, TIMS $Na_2BO_2^+$ and $Cs_2BO_2^+$). The δ^{11}B reference materials are commercially available through European Reference Materials (http://www. erm-crm. org) or the webshop of the BAM Federal Institute of Materials Research and Testing (http://www. webshop. bam. de).

The newly produced and certified boron isotope reference materials will allow straight forward method validation and quality control of boron isotope data.

Micro-XRD and ICP-MS analysis of sub-milligram sized mineral samples

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We present a study that combined micro-analytical X-ray diffraction (XRD), emplying the image plate - diffraction image integration software (IP-DIIS) method, with trace element analysis on the very same grain fragment via ultra-low blank solution inductively coupled plasma – mass spectrometry (ICP-MS). The experimental procedure involves three steps. First, trace element homogeneity in the mineral is studied via laser ablation ICP-MS by analysis of multiple spots. Subsequently, a very small fragment of material (typical edge length ~15-25μm) is removed adjacent to the laser ablation pit. This is then mounted in a 114.6 mm Gandolfi camera. An IP is used to quantitatively record the diffraction inter-planar spacing (d-spacing) and intersity (I). The latent image on the IP is digitized through a propietary IP scanner, after which the image plate can be erased and reused. The scanned image is then converted to a digital diffractogram using appropriate not-for-profit software. We demonstrate that simple XRD technique yields a fully quantitative digital diffractogram amenable to Rietveld structural refinement. The accurate and percise crystallo-graphic parameters are thus known for the mineral fragment from the structural refinement. The final analytical step involves ultra-low blank miniaturized mineral acid digestion of the mineral fragments that typically weigh between 0.1 and 1 milligrams. The digests are subsequently analyzed for the trace element suite of interest by solution ICP-MS.

When crystallographic parameters and chemical information are combined in this fashion, it is possible to empirically test the control excerted by the crystal lattice on apparent trace element partition coefficients. In particular, this method allows a test of the theoretically derived and experimentally confirmed crystal lattice strain model (CLSM) [1, 2]) for naturally occuring mineral phases. Namely, the fundamental premise of the CLSM is that isovalent trace element distribution (D) in a mineral is controlled by the size (Ro) and elastic modulus (E) of the substituent crystallographic site. The XRD data can be used to compute Ro while the trace element data can be used to calculate an independent value for Ro from the strain-free admittance of a fictitious atom of radius Ro.

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