

## Stable Cr isotope ratio measurements using a double-spike method and high-resolution MC-ICP-MS

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Reduction of Cr(VI) to Cr(III) in aqueous solutions is accompanied by a significant mass-dependent Cr isotope fractionation of more than -3‰ per atomic mass unit (Ellis et al., 2002). This makes stable Cr isotopes a sensitive tracer to detect and quantify redox changes in a variety of geochemical reservoirs.

We determine stable Cr isotope ratios on a ThermoFinnigan Neptune multicollector ICP-MS at high mass resolution mode. A  $^{50}\text{Cr}$ - $^{54}\text{Cr}$  double spike of well-known isotope composition is added to and homogenised with the sample chromium prior to chemical purification. The double-spike addition allows for accurate in-run determination of the instrumental mass bias, but also accounts for possible mass-dependent fractionation of Cr isotopes during chemical processing of the sample. Purification of chromium from the sample matrix is performed by a combination of anion and cation exchange chromatography adapted from the protocol of Ball and Bassett (2003).  $\delta^{53}\text{Cr}/^{52}\text{Cr}$  values of Cr standards that were passed through the ion exchange chromatography ( $n=2$ ) are equal to those that were not subject to chemical purification ( $n=15$ ). The overall reproducibility of the Cr standards was  $\pm 0.05$  ‰ (2 S.D.), however, the long-term reproducibility of the method remains to be assessed in more detail. An unspiked Cr(VI) standard was loaded onto the anion column followed by sequential release from the resin via reduction. Prior to isotope analyses, adequate portions of the double-spike were added to the eluted aliquots. Sequential release from the anion resin via reduction was accompanied by Cr isotope fractionation from -2.76‰ in the first to +5.19‰ in the last aliquot. Overall Cr recovery during chemical purification was determined to be 75-85%. Thus, the above-mentioned results demonstrate that the double-spike method is required to account for Cr isotope fractionation introduced during chemical purification. Chromites from both the Bushveld and Great Dyke layered intrusion do not show any variation in their Cr isotope composition with stratigraphic height and are indistinguishable from each other.

### References

- Ellis, A.S. et al., (2002), *Science*, 295, 2060-2062.  
Ball, J.W. and Bassett, R.L., (2000), *Chem. Geol.* 168, 123 ff.

## The Determination of atomic weights by new analytical techniques

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New analytical techniques, including single and multiple collector inductively coupled plasma-mass spectrometry (ICP-MS) and Secondary Ion Mass Spectrometry (SIMS), have recently provided atomic weights of chemical elements with unparalleled precision. For many elements (including, but not limited to, B, Si, Ca, Mo, Zn, and Cd) the comparison of results obtained by different laboratories using different analytical techniques is hampered by the lack of suitable reference materials of known (or at the very least agreed-upon) isotope composition. For example, ICP-MS analyses on boron reference materials distributed by the International Atomic Energy Agency have very low uncertainties in their atomic weights, but the values do not agree well with previous thermal ionization mass spectrometric (TIMS) measurements using both positive and negative ions. In fact, ICP-MS values from different laboratories do not overlap within 2s uncertainties. The reasons for the lack of agreement are not well understood. One of the main tasks of the International Union of Pure and Applied Chemistry (IUPAC) Commission of Isotope Abundances and Atomic Weights (CIAAW) is to evaluate newly published isotope abundance data to establish current best values of atomic weights that are both precise and accurate relative to a partitionable, highly homogeneous, and widely recognized reference material for each element. This presentation will highlight the challenges of making reliable isotope abundance measurements and the work that CIAAW is doing to ensure that atomic weight data are both precise and accurate.