Geophysical Research Abstracts, Vol. 9, 06046, 2007
SRef-ID: 1607-7962/gra/EGU2007-A-06046

REFERENCE MATERIALS FOR OSMIUM ISOTOPIC ANALYSIS

T. Meisel and LOsST Collaborative Trial

General and Analytical Chemistry, University of Leoben, Austria
(thomas.meisel@mu-leoben.at)

Certified reference materials (CRM) are a prerequisite for validating analytical procedures such as the determination of the osmium isotopic composition ($^{187}Os/^{188}Os$ and $^{186}Os/^{188}Os$). CRM can be used to establish metrological traceability of a measurement result to a common reference (ideally SI units). Traceability makes the results of different laboratories comparable. Measurement uncertainties need to be associated to the measurement and are estimates of the range of values, that can be reasonably attributed to the isotope ratio. CRM can be used to estimate measurement uncertainty and to control accuracy within the uncertainty. In reality no CRM exists for osmium isotopic analysis. Many labs use quality control materials ("in-house standards") to asses the repeatability of a measurement result but this is not sufficient to reach the goals of traceability and to estimate measurement uncertainty.

To demonstrate the need of commonly used CRM a round robin test was launched a couple of years ago with a "liquid osmium standard" (LOsST) distributed by the University of Leoben. 17 labs participated applying different techniques (ICP-QMS, ICP-SFMS, MC-ICP-MS and N-TIMS) for the estimation of as many osmium isotope ratios as possible. Another purpose of the experiment was to provide measurement uncertainty for the results.

Based on this round robin test a certification is not possible, since international protocols for reference material certification (e.g. IAG protocol) do not allow to eliminate obvious outliers. In addition the uncertainty of the certified isotope ratio needs to be significantly smaller (ca. $1/5^{th}$ to $1/10^{th}$) than routine measurements that are fit for the purpose. The results of the LOsST trial demonstrates that significant inter-laboratory bias in isotope ratio determination exists. In particular, it is clear that a very large range

of values are possible for the $^{186}Os/^{188}Os$ ratio, for which a measurement uncertainty of better than 50 ppm is essential. Hence, measurement uncertainties have been underestimated and several influence quantities (e.g. effects during measurement and/or normalization) were not under control. In order for accurate, high precision analyses of Os isotope ratios to become widespread, there is an obvious need to establish a CRM and to establish some common measurement parameters.

Although the isotopic composition of this solution is neither certified for $^{187}Os/^{188}Os$ nor for $^{186}Os/^{188}Os$, the use of this solution is highly recommended, since it is the best determined RM and since no common reference solution is currently used, although isotope ratios have been determined precisely for more than sixteen years. A new solution (5 L with 5000 ppm) was acquired by Durham University and is now ready for a proper certification process following the certification protocol of the IAG. Participating laboratories will be selected based on the results of the LOsST results and based on their demonstrated role as expert labs. Once certified, any CRM solution should not be used on a daily basis, since they are too precious for such a task due to their tedious certification efforts and their limited supply. Quality control materials (QCM) which are solutions currently used for estimating repeatability should be used instead. The IAG has identified the needs of the geoanalytical community and has initiated a Certification Committee that will intensify the production and certification of RM, such as RM for microanalytical techniques, for PGE and Os isotopic analysis.