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CAN WE CERTIFY ISOTOPE REFERENCE MATERIALS?

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The production and certification of certified reference materials is always and always will be behind the advances of cutting edge science. If this was not the case, sciences expanding into new frontiers will not happen or only at a much lower pace as what is currently happening in the field of fractionation of non-traditional isotopes. The advances in this previously almost unknown field of isotope geosciences was triggered by the now established analytical method of MC-ICP-MS.

Producing and publishing numbers on mass fractionations determined with mass spectrometers, only make sense if these number are not only repeatable (i.e. during a short period of time with the same instrument and person) but also reproducible (i.e. in other labs with other instruments). In addition, if difference between samples are detected than these differences need to be statistically significant. Although these concepts are well established and most of the tools known that need to be applied to achieve these goals, a large discrepancy between proper use metrological concepts and the urge to produce new data has occurred.

As we have to abandon the idea of determining the true isotope ratio, reference materials were introduced to make numbers comparable between lab. These RM are used as “delta zero” or “primary standards” and the numbers on unknown samples are published as relative deviation form these RM. Applying this route, certified RM (“secondary standards”) can be used to validate an analytical procedure and to identify systematic deviations between labs.

Several workshops have been initiated in order to achieve the goals of the basic con-

cepts of isotope ratio measurement, in particular for fractionation of non traditional isotopes. Reasons for these workshops are the need for the agreement on which RM should be used as a common reference and on how to produce certified RM. Since the variance of some isotopic systems is so little, great care has to be taken on the choice, the homogeneity, the proper use, the distribution, availability and sustainability of the RM. If the goal of using a common reference has been achieved the next goal would be the production of certified RM.

There are several attempts to achieving this goal by NIST or the IAG (for Os). But the question is, if it possible at all the reach this goal? What are the requirement for producing a useful RM? The most important feature for a certified reference material is the fitness-for-purpose criteria. It does not help to have an uncertainty of certified isotope ratio of a reference material that is larger or even similar to the expected uncertainty of measurement of an unknown sample. The stated uncertainty of the RM needs to have an uncertainty of at least 1/3 or 1/10 of the routine measurement uncertainty in order to detect a significant difference between the two values. This is a prerequisite which barely can be achieved for pure and homogenous solutions. As soon as several laboratories participate in a single round certification, the variance from the average of the labs will be larger than quoted routine measurement uncertainties (see collaborative trial for a Os isotope solution LOsST). Using real samples as reference materials will add additional uncertainty components to the uncertainty budget. As sampling and sample inhomogeneity are the dominating uncertainty components, the uncertainty of a certified value will inflate to unacceptable levels. In order to overcome this problem, we either improve our method to be unbiased and capable of producing precisions much better than for routine analysis, or we accept the fact that at the current state-of-the-art it is not possible to distinguish differences between two data points produced by different labs that are smaller than 3x the uncertainty associated with the certified value. The latter will be difficult to accept by authors of many papers.