



Normalization of O-isotope data from Ag₃PO₄ to VSMOW-VSLAP scale – one quest for a new (in-house) comparison material

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In order to be able to report comparable $\delta^{18}\text{O}$ values, ideally, laboratories should be able to use commonly available reference materials of known isotopic composition analysed according to the principle of identical treatment. In the case of Ag_3PO_4 , the typical analyte for the O-isotope analysis of PO_4 in CF-IRMS, this is not such an easy task to achieve due to the lack of PO_4 materials of known O-isotope composition. A widely used comparison material in these analyses is the NBS120C phosphate concentration standard, for which the $\delta^{18}\text{O}$ value of +21.7 ‰ has been widely accepted.

We started working on O-isotope analyses from skeletal phosphate by using organic reference materials for scale normalization and NBS120C as a control at the Laboratory of Chronology (Finnish Museum of Natural History). This approach has successfully been used in some laboratories. It came soon apparent that this normalization scheme did not perform well for us; the $\delta^{18}\text{O}$ value of NBS120c, scale normalized to organic reference materials, gave $\delta^{18}\text{O}$ values up to 2.3 ‰ higher compared to the consensus value of +21.7 ‰. The problem was confirmed when we were at a later stage able to include Ag_3PO_4 of known isotopic composition (AGPO-SCRI, Halas et al. 2011, Rapid Commun. Mass Spectrom., 25, 579-584) to our analysis runs. Therefore, we discarded the idea of using organic reference materials for scale normalizing O-isotope data from Ag_3PO_4 .

With these two materials at hand, NBS120C and AGPO-SCRI, we would be able to use 2-point normalization with matrix matched comparison materials (Ag_3PO_4). However, in some cases a comparison material with a lower $\delta^{18}\text{O}$ value is needed, so that the O-isotopic composition of the unknowns can be bracketed by these. In our search for suitable in-house comparison materials we came across an old O-isotope analyses from a magmatic apatite mineral, with a recorded $\delta^{18}\text{O}$ value of +5.2 ‰. Guided by this finding, we procured a large single crystal of apatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH},\text{F},\text{Cl})_2$) from the same source: Siilinjärvi alkali rock complex, located in eastern Finland. The crystal was homogenized and a batch of Ag_3PO_4 was produced by using conventional methods. The O isotope composition of the Ag_3PO_4 was determined at the University of Lausanne by laser fluorination and TC/EA-IRMS, and the results, a $\delta^{18}\text{O}$ value of +5.0 (laser fluorination) to $+4.9 \pm 0.3$ (TC/EA-IRMS), compared closely to the value of the old analysis. The single crystal pulverized in our lab is not a suitable source to serve the wider isotopic community. However, we would like bring attention to this type of material as a potential source of a O-isotope reference. A batch of apatite collected from this complex, or other similar complex could produce enough material to provide a large amount of reference material for O-isotope analysis, similar to NBS120C but excluding e.g. organic or other contaminants, and having a relatively low $\delta^{18}\text{O}$ value.