

## ***Interactive comment on “Analysis of oxygen isotopes of inorganic phosphate ( $\delta^{18}\text{O}_p$ ) in freshwater: A detailed method description” by Catharina Simone Nisbeth et al.***

### **Anonymous Referee #4**

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The manuscript by Nisbeth and colleagues describes a protocol to extract phosphate from freshwater samples for the analysis of phosphate oxygen isotope signatures ( $\delta^{18}\text{O}_p$ ). There are several protocols for this extraction to date, all having been developed for different sample matrices (seawater, freshwater, soil solution, waste water et cetera). One aim of the paper is to critically review these protocols for their adaptability to freshwater samples (which would be very welcome), and another is to provide the reader with a detailed method description on how to convert freshwater phosphate to IRMS-grade silver phosphate (which would be even more welcome).

However, the authors have decided to interweave both parts (the method review and

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the protocol), and therefore its usability as a concise method description is limited. It is also on the lengthy side for what I would expect from a technical note. Thus, it would strongly benefit from a better organisation; maybe a structure that separates the review part from the description of the (novel) method.

These formal issues aside, I am missing a robust, data-backed method validation. What have the authors undertaken to verify the integrity of the method? There is unfortunately no data on, for example, phosphate recovery in the individual steps,  $\text{Ag}_3\text{PO}_4$  yields,  $\delta^{18}\text{O}_\text{p}$  conservation. I consider this absolutely mandatory for a method paper (as a HESS technical note or elsewhere), and I strongly suggest that the authors include such evidence in a thorough revision before further consideration.

Other suggestions:

Title. A little misleading, because the actual measurement of  $\text{d}^{18}\text{O}_\text{P}$  is not part of the paper (Analysis of oxygen isotopes of inorganic phosphate ( $\delta^{18}\text{O}_\text{p}$ ) in freshwater: A detailed method description) – more apt would be something like "Purification of silver phosphate from freshwater samples for the determination of  $\delta^{18}\text{O}_\text{p}$ ".

Line 199 and elsewhere. Mg-brine is probably not a common term, why not using  $\text{MgCl}_2$ ? That said, I would avoid adding chloride anyway because one will have to get rid of it before  $\text{Ag}_3\text{PO}_4$  precipitation.

Quality of the figures. The photos appear rather blurry and the presentation of lab equipment/vials/glassware would benefit from better organization. Some photos may be redundant (e.g. Fig 10).

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