

## ***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.***

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

Anonymous Referee #4

Received and published: 11 December 2019  
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

C1

(seawater, freshwater, soil solution, waste water et cetera). One aim of the paper is to critically review these protocols for their adapt-ability 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 inter-weave both parts (the method review and C1 the protocol), and therefore its usability as a concise method description is limited. Firstly I would like to thank the reviewer for the useful comments and suggestions. In regard to the point made that the usability of the technical note as a concise method description, there must be some appreciation that in order to give some level of context and background to the many problems encountered by researchers applying the method a level of description is required to give that detail. As another reviewer outlined, describing some pitfalls or mistakes is very useful as it can save the reader a lot of time, effort and money in the technique application. 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. Granted this is a valid point and we will work to be more succinct and concise in our descriptions in the corrected document. 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,  $\delta^{18}\text{O}_p$  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.

This is a valid point and we will make reference to method validation through phosphate recovery by using a reference report in the corrected manuscript. Colleagues will also send me the estimate losses with the Magic steps which were estimated to be around 10-15%, but we will be more precise in the corrected manuscript. We will add this info also in the revised version of the paper.

C2

Other suggestions: Title. A little misleading, because the actual measurement of  $\delta^{18}\text{O}_\text{P}$  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  $\delta^{18}\text{O}_\text{P}$ ".

We can reformulate the title of the manuscript in the corrected document to something similar to what is suggested here, focusing on the processing procedure. . However, as noted previously, the revised paper with added experimental data might call for yet another title – we will decide later.

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.

Mg-Brine is a term sometimes used but we can change this to concentrated  $\text{MgCl}_2$  as suggested in the corrected manuscript.

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

We can endeavour to improve the photos and we will organise them better. I would like to thank the reviewer for the useful comments.

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Interactive comment on Hydrol. Earth Syst. Sci. Discuss., <https://doi.org/10.5194/hess-2019-469>, 2019.