

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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Received and published: 17 January 2020

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

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(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 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, Ag_3PO_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.

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.

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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 MgCl_2 ? That said, I would avoid adding chloride anyway because one will have to get rid of it before Ag_3PO_4 precipitation.

Mg-Brine is a term sometimes used but we can change this to concentrated 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.

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

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