

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 #1

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Analysis of oxygen isotopes of inorganic phosphate ($\delta^{18}\text{O}_p$) in freshwater: A detailed method description by Nisbeth et al.

A) General comments

The present manuscript submitted by Nisbeth et al. is supposed to be published as a technical note that provides a detailed description for the analysis of oxygen isotope ratios in inorganic phosphate obtained from freshwater samples. Such a detailed step-by-step guide is generally of great interest to the growing number of scientists using this approach to investigate sources and cycling of phosphorus in the environment. I have to admit this is the first time I review a technical note, but thanks to extensive lab

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experience, I have a good impression of what a sound technical note should look like.

What I expect from a technical note is a clear, straight-forward, user-friendly step-by-step description of the method and its respective steps. Having said this, we are already at the major issue of this manuscript. In its present form, the manuscript is a) too long (technical notes in HESS should be “a few pages only”), and b) it carries characteristics that are typical for other manuscript types. For example, it appears to be more of a review than a technical note at some points, which include citations and lengthy discussions. While this approach is fine for a classical review paper, it does not belong into a technical note. If, for example, your technical method is not suitable for a certain type of water sample, so just say it and do not attempt to come up with lengthy discussions of why and how it could work (for example, see lines 300-302 or 312-315). In sum, the two general questions the authors need to clarify are how should the manuscript look like as a sound technical note, and what needs to be done to get it there.

-Getting the manuscript into the right form also involves a substantial shortening of the present text. By removing text, there is also the question how to keep the aspect of novelty and not just repeat what has been previously published in other original papers (e.g., by Tamburini et al.) or reviews (e.g., by Davies et al. 2014), because this would be just a repetition of what has already been published. I therefore suggest to use your “own experience” additions (i.e., the only truly new information provided to the reader) in order to provide a clear optimized method description. Finally, the revised manuscript would be entitled something like “Analysis of oxygen isotopes of inorganic phosphate ($\delta^{18}\text{OPO}_4$) in freshwater: Detailed description of an optimized method” or something similar in this direction.

-What I also miss is a clear recommendation regarding quality control. How to assure your $\delta^{18}\text{PO}_4$ signal does not change during the numerous sample processing steps? As already laid out by the authors, there might be pronounced issues with high organic waters, bearing the risk that organic P becomes hydrolyzed to PO_4 , thereby altering

the original $\delta^{18}\text{OPO}_4$ signal.

-I further suggest to include a schematic overview of all the processes involved, which would also act as a sort of graphical guideline (see for example Davies et al. 2014, Figure 4 for such an example).

-To conclude, the manuscript in its present form requires substantial revision to meet the criteria of a technical report. Considering the value of such an optimized method description for the growing numbers of researchers working on aquatic P cycling, I would like to encourage the authors to submit a revised version of their manuscript. Given the general and specific comments (see below), I end up with the recommendation "reject with suggestion of resubmission"; however, it appears that HESS does not provide this recommendation option for reviewers, so I leave the decision to handling editor if it is going to be a "major revision" or "rejection".

B) Specific comments

-1.Introduction: We need to ask ourselves here the following question - do we really need a lengthy introduction regarding the application of $\delta^{18}\text{OPO}_4$ analysis in a technical note? I would strongly recommend to condense the entire 2-page long paragraph into a short paragraph of 3-5 sentences that refer to the common literature.

-In accordance with Coplen's 2011 "Guidelines and recommended terms for expression of stable isotope ratio and gas ratio measurement results" (Rap. Comm. Mass Spectr.) , I would recommend the consistent use of the term $\delta^{18}\text{OPO}_4$ throughout the text and avoid P_i and other non-conventional terms.

-Keep consistency regarding chemical concentrations; there are molar concentrations but also mg/L in the text, this should be consistent.

-Chapter 2.2, Step I: I have great doubt that the described procedure will be suitable to prevent co-precipitation of dissolved PO_4 by Fe-oxyhydroxides if you have high Fe^{2+} concentrations in your water samples. During pumping, subsequent storage and trans-

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portation, it is impossible to avoid diffusion of O₂ through pumping hose and plastic materials. This will in turn quickly react (within minutes) with the Fe²⁺ and form Fe-oxides, which in turn coprecipitate dissolved PO₄ from solution. Do the authors have prove for a successful application of their suggestion? If not I suggest to go for other ways to isolate the dissolved PO₄ from solution. This also brings me to the question if you really need such a lengthy sampling description in general; but this depends on where you want to focus your technical note.

-Line 188: This also applies to all previous steps, which means samples need to be processed immediately after sampling to avoid potential microbial alterations.

C) Purely technical corrections at the very end ("technical corrections": typing errors, etc.). line 248: There is no such section

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