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Interactive comment on "Fast-freezing with liquid nitrogen preserves bulk dissolved organic matter concentrations, but not its composition" *by* Lisa Thieme et al.

Anonymous Referee #3

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Freezing dissolved organic matter was a common technique and recently has come under increased scrutiny due to potential impacts to the solution chemistry and chemical character of compounds in solution. There is a growing body of literature on the influence of freezing on streamwater, but apparently not any experiments to my knowledge that treat this problem in soil solution. The paper by Thieme et al. appears to be the first attempt to investigate the issue of freezing soil solution and therefore, is a compelling topic that could benefit the research community working in soil and stream dissolved organic matter. Overall, the manuscript has a solid foundation, but there are several areas where the authors could improve the manuscript. I feel there is also a major shortcoming that may not be egregious enough to prevent publication, but

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represents a major confounding issue in the interpretation of the results. The major shortcoming in my view is the method of compositing both grassland and forest soils together for analysis. These soil systems are very different and one would expect them to behave quite differently in terms of the dissolved organic matter produced within the soil matrix as well as exported from the site. Investigating the results of the freezing on these samples separately is a missed opportunity and I suggest that it should be a priority to do a follow-up to see if the results are similar or some important patterns in the results have been masked by the increased level of variability due to the composite samples of these two soil types. General comments: Introduction: The introduction focuses a great deal on fluorescence, while not mentioning the experimental approach of freezing until much later in the discussion. The extensive literature review on fluorescence isn't necessary given the common nature of the technique and the focus of the paper. I suggest reducing the discussion of fluorescence and spending more time summarizing current research on freezing and identifying knowledge gaps in this area. Specifically, I think it would be important to see if any experiments have been conducted on freezing soil solution. Highlighting the novelty of the approach is critical for the impact of this study. In addition, keep the information of freezing organic matter in general. Some time could also be dedicated to discussing what might be different between stream samples and soil samples after freezing. Finally, a clear justification and rationale for the study needs to be part of the introduction. Sampling and sample preparation: The approach for sampling, replication, and defining the subject for the analysis needs to be clarified. The existing description is hard to follow. It might help to provide a diagram for where the samples originate and their fate, with a clear identification of what is composited and analyzed. This will clearly highlight the mixing of the grassland and forest samples. The freezing procedures are somewhat tedious. Is this operational? What happens if a large quantity of water is stored? Is there a potential difference given the small amounts used as test subjects in the study? Results: The overall average change of 6% (1.6 mg L-1) seems small given the high DOC concentrations in the samples. Is the lower average a result of the composite? L30: This doesn't

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make sense. SUVA values increase, so aromatic compounds or aromaticity increase. But, humification index decreases? Conclusion: There needs to be some discussion of the results related to very high DOC concentrations in the sample. What are the implications for changes in the DOM character with freezing? Also, is freezing with N2 practical? Figure 1: Is cDOC an accepted convention? A label of DOC with the units usually implies a concentration. I suggest adding 'in' for Change in DOC concentration, Or DOC change.

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