

Interactive comment on “Inter-laboratory comparison of cryogenic water extraction systems for stable isotope analysis of soil water” by Natalie Orlowski et al.

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This inter-laboratory comparison study presents very significant findings regarding the performance of cryogenic water extraction (CWE) systems for soil water stable isotope analysis. The paper is well written with clear findings and illustrations. The lead and senior authors have developed and researched CWE system for several years and have previously published several papers on these systems (e.g. Orlowski et al. 2013, 2015, 2016). The present study represents a welcome initiative to help improve analytical techniques. The comparison study was well designed with the emphasis on documenting the difference between the known isotopic composition of a supplied wa-

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ter sample and the extracted isotopic composition of soil water derived from the known water by using it to wet dry soils. The key finding is the surprisingly large difference in performance of CWE systems in different laboratories (Fig. 3 and 4). A few laboratories performed relatively well but none were acceptable (as per the study criteria) for all soils, water contents and isotope systems. The fact that most laboratories performed very poorly for one or both soils using a system that has been regarded as the main stay of soil (and plant) water isotope analysis is a disturbing conclusion (the authors note their dismay). → What are the consequences for the reliability of the numerous previous studies relying on these and similar laboratories? Were studies conducted without the rigorous quality control (recovery of known water isotopic composition) carried out in the present study? These questions should be noted (if not answered) in the discussion. The interlaboratory comparison confirmed the influence of many factors that affect accuracy as documented in previous publications. However, the lack of systematic relationships between isotopic recovery of soil waters and CWE parameters prevented clear conclusions from being drawn regarding which future steps can be taken to improve performance. This suggest that a complex interaction of many factors including soil type, temperature, vacuum etc. influence CWE results. These may also include the specific design and operation of each CWE system. These findings are also highly significant in light of the Orlowski et al 2016 study (Hydrological Processes: Intercomparison of soil pore water extraction methods for stable isotope analysis. Natalie Orlowski, Dyan L. Pratt and Jeffrey J. McDonnell) which compared five different techniques for analysing soil water isotope composition. It appears that the differences in accuracy of these five techniques were no larger than the difference in accuracy between the sixteen CWE systems presented in the present study. → This raises the possibility that the success of any of these techniques may depend more on the specific understanding, design and settings/operation of each technique than an inherent superiority of one technique over another. This aspect should be added to the discussion. Specific comments: P5 L20: I understand that choices had to be made but it should be acknowledged that the drying and rewetting steps may have influenced the

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outcomes if not performed the same way - were instructions on these steps included? P5 L27-32: I can't locate the data from this reliability test, were all labs successful in this test? P6 L14: "alternating fashion" - it is unclear exactly how this step was carried out P6 L14: How much soil was loaded by each lab (both using their own method and the prescribed method)? The questionnaire asked this question, but the information doesn't seem to be presented. Is it possible that soil inhomogeneity was a factor if a lab used small amounts? P13 L27: Incomplete drying before wetting may also have led to >100% recovery during CWE P14 L5: Freezing of the wetted soils before loading in the CWE may reduce vapour loss during evaporation P17 L22-23: This sentence seems adrift here, but it is a valid point that should be expanded upon, possibly in the introduction/background. It is a valid question to ask whether it is actually relevant to extract all water from a soil sample - it will depend on the study context. P17 L29: Not all laser instruments (LAS) were of the OA-ICOS type (Los Gatos) in the WICO study (Wassenaar et al 2018) - several were CRDS instruments (Picarro). This section should also be modified with respect to organic interferences in LAS, the effects can be dramatically different between Los Gatos and Picarro instruments both in direction and magnitude. They are even different between different generations of Picarros (see e.g. Munksgaard et al. Rapid Commun. Mass Spectrom. 2014, 28, 2151–2161) P18 L28: Interferences can also be overcome by in-line high temperature oxidation prior to LAS measurement, although this will likely contribute small amounts of H₂O which may or may not be significant compared to the overall extraction amount. P20 L11: Does this mean that in effect each soil would have to be investigated (i.e. a standard addition technique) unless a series of samples have very similar contents and type of organics and clay? - a very tedious process. P21 L13: Can the authors expand on what these techniques could look like?

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