Reply to Reviewer 2

In the following, please find the corrections and comments to the referee's response.

This work provides a good initiative towards the standardization of a procedure that can be carried out in multiple ways. The inclusion of laboratories worldwide depicts this need as well as the large variety of extraction systems developed to carry out the CWE. The idea of involving multiple laboratories under the same approach is well done and supported with a good protocol (despite the possible sources of errors described by the authors). However, even if the authors do not mention a specific method or guidelines at the end of the paper; the information provided can lead to the best practice. On the Methods (page 5, lines: 27-32) and Discussion (page 14, lines: 15-22) sections, the authors mention the "performance test" carried out by the laboratories. The analysis of this data needs more attention on the Results section and it can be integrated with a Zscore graph as the one showed by Orlowski et al (2016). This type of plot will help to see the proportional laboratory efficacy to reach the labeled water. In addition, it will be important to add information about the performance test carried out by the laboratories, because the paper only mentions the data of two laboratories and leave on the dark the data from the other 14. The laboratory capacity to successfully extract soil water is essential for most of the projects relying on that information (the reason why the authors defined the experiment). This work shows that despite having identical soil and water samples, as well as the protocol extraction ("pre-defined extraction method"), no one of the laboratories was able to reach the water signature. The proposed rehydration process could be affected by small differences among the soil samples sent to each laboratory (nonhomogeneous composition between subsamples of the same soil). This brings the question if different subsamples of soil were analyzed to test the homogeneity among samples as the authors did with the water? In addition, did any of the laboratories send a sample of rehydrated soil for its physical and chemical analysis? Because this can help the authors to support their assumption (Discussion section, page 17; 13-15) Despite the authors sentence (Discussion section, page 20, lines: 1-2): "We found no clear tendency for which approach should be applied, thus at present, and much to our dismay, we cannot define any standard protocol for CWE"; the information contained in this paper can give important clues about the feasibility of applying one specific method. If the authors apply the Zscore graph (Orlowski et al, 2016) mentioned previously (second paragraph); they can determine which methods lead towards a more accurate extraction among all the setups evaluated considering the pre-defined protocol (analysis 1) and considering the laboratory protocol (analysis 2). In this way, the authors can provide as "take-home messages" the laboratory practices that lead towards better results. If the authors change the notation from 20. The amount of sample material used per laboratory is not reflected in table 1 and this information can help to understand the differences.

Response: We thank referee #2 for the very valuable and positive comments, which helped us to improve our manuscript.

Figures 3 and 4 provide mean differences to the reference DI water allowing assumptions on how good or bad individual labs performed with respect to the "reference". Simply put, a Z-score is a numerical measure of a value's relationship to the mean in a group of values. If a Z-score is 0, it represents the score as identical to the mean score. Figure 3 and 4 provide this information, not in dual isotope space but individually for each isotope. The figures further include the SD of the lab's

extraction results. It is this information that the paper intends to convey. We therefore would prefer, and we hope our argument above helps to demonstrate this, not to provide additional Z-score plots for the soil water extraction results. They would be redundant in some ways and would not convey the precise message we intend with Figures 3 and 4 in their current form.
With respect to the "performance test" results, we did not include these results in our manuscript since not every lab provided the full set of data. We therefore picked some examples as the data allowed. Again, a great suggestion but our data limited us in this regard.

We can rule out any inhomogeneity of the bulk soil sample's soil physico-chemical properties. The LUFA Speyer provides "standard soils" exactly for conducting lab studies (https://www.lufa-speyer.de/index.php/dienstleistungen/standardboeden/8-dienstleistungen/artikel/57-standard-soils). The LUFA already dried, homogenized and sieved (with a 2 mm screen) the bulk soil sample according to "Good Laboratory Practice". However, we repeated these steps before taking subsamples for the individual labs. Since every lab rehydrated the soil samples with the same deionized water (that we sent to them) and soils were only used once per extraction, we assume that potential changes in the soil properties due to the water additions are the same across labs. We did not see the necessity in determining the soil properties for a second time after water extraction, since samples were not extracted multiple times. Soil properties were determined according to German DIN-ISO norms on dried soil samples (e.g., DIN-ISO 11465, DIN-ISO 10390). We therefore did not see the need in analysing soil properties on a rehydrated soil sample.

With respect to the "take-home-message", we will further add the following: "Orlowski et al. (2018) recently explored the effect of CWE for tracing plant source water. The authors tested the ability to match plant water to its putative soil water source(s) by using different CWE conditions (30–240 min, 80–200 °C, 0.1 Pa) for a clayey loam (same as in this study) and a sand. They showed that with higher extraction temperatures and longer extraction times, gradually more enriched soil water was extracted, which surprisingly reflected the plants' source water…For most past studies, possible fractionation effects associated with CWE remain unknown and the applied extraction parameters or cryogenic system specifications are often not indicated. Orlowski et al. (2018) recently stated that observed isotopic fractionation effects potentially lead to errors when CWE isotope data is used for plant water source calculation. This miscalculation in plant's water source could be quite large and could lead to misinterpretations of the role different plant species play in hydrologic processes at the ecosystem or larger scales. Millar et al. (2018) used the most common water extraction methods (centrifugation, microwave extraction, direct vapor equilibration, high-pressure mechanical squeezing, and two different CWE systems) for their intercomparison study on spring wheat (Triticum aestivum L.). The authors showed that all methods yielded markedly different isotopic signatures. The various methods also produced differing concentrations of co-extracted organic compounds. Again, CWE was outperformed by other extraction methods...In the light of our experience with other soil water extraction techniques (Orlowski et al., 2016b), we argue that the success of any of these methods may depend more on the specific understanding and operation leading to internal reproducibility of each individual technique's results than an inherent superiority of one technique over another."

We will include the amount of sample material used per laboratory in Table 1. We will further include the following in the results section: “In relation to the amount of used sample material, most labs either introduced 10 or 20 g to their system no matter the extraction approach (I or II), soil type or WC. Only labs 11 and 13 chose different weights with respect to the WC, e.g., 10 g for the higher WC (20%) and 20 g for 8% WC for extraction approach I.”

References

Millar, C., Pratt, D., Schneider, D. J. and McDonnell, J. J.: A comparison of extraction systems for plant water stable isotope analysis, Rapid Commun. Mass Spectrom., 32(13), 1031–1044, doi:10.1002/rcm.8136, 2018.

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Orlowski, N., Winkler, A., McDonnell, J. J. and Breuer, L.: A simple greenhouse experiment to explore the effect of cryogenic water extraction for tracing plant source water, Ecohydrology, e1967, doi:10.1002/eco.1967, 2018.