

HESS Technical Note:

Simple, exact and reliable way to extract soil water for stable isotope analysis

Jiří Kocum et al.

AUTHORS' RESPONSE

REVIEWER 1

Reviewer's Comments:

This paper introduces yet another method to dozens of highly operational approaches over the decades to soil porewater extraction of water for stable isotope analysis. Each method proponent claims a superior and more reliable approach (not true). This new method involves a low cost means of recirculating air through a heated (105 °C) closed-system soil sample vessel coupled with an inline 8 °C condensation coil to collect the evaporated soil water until completion (e.g. > 99.5 % water recovery). The author's aim is to demonstrate reliable isotopic data (recovery) and propose its use in field studies, though it may be less suited for high-throughput applications at only 4 samples per day. Nevertheless, the pilot results appear to be promising, and further testing and replication by others is warranted to find out all of the pros and cons of this approach.

The authors should greatly temper their enthusiastic language about top performance because they only tested a few relatively easy porous materials with a high water content (20 %) – there is no performance information on different and low porewater content (<<5-10 %), nor on high organic matter content materials, or on low conductivity clays, etc. Stick to a basic description of pilot performance of the circulating air experiment, recognizing that you have not tested all possibilities. Moreover, until your system has been tested identically and independently in another laboratory, its should remain as a pilot proposal.

Authors' Response:

First and foremost, we would like to thank the reviewer for the high quality and inspiring review, which helped us to improve our work in many ways. Based on your suggestions, we will edit the final manuscript to make it easier to understand.

Thank you for the comment on the enthusiastic language; the revised version will be written in neutral tone. We are aware of the limitations imposed by the type of soil samples used to test the functionality of the extractor in spite of test reproducibility indicating the correct functioning of the apparatus. We agree that samples with low porewater content and more clayey composition would represent a more robust basis for evaluating the proposed extraction method. To explain the soil types selection, they represent most common types in the Central Europe (and also in our experimental areas) for which the extraction method was primarily designed. This was the reason, why we preferred these types of soil. However, in order to support our claims concerning efficiency of the proposed method, consequent experiments were carried out with silt loam and clay soils (with 10 % gravimetric water content). The

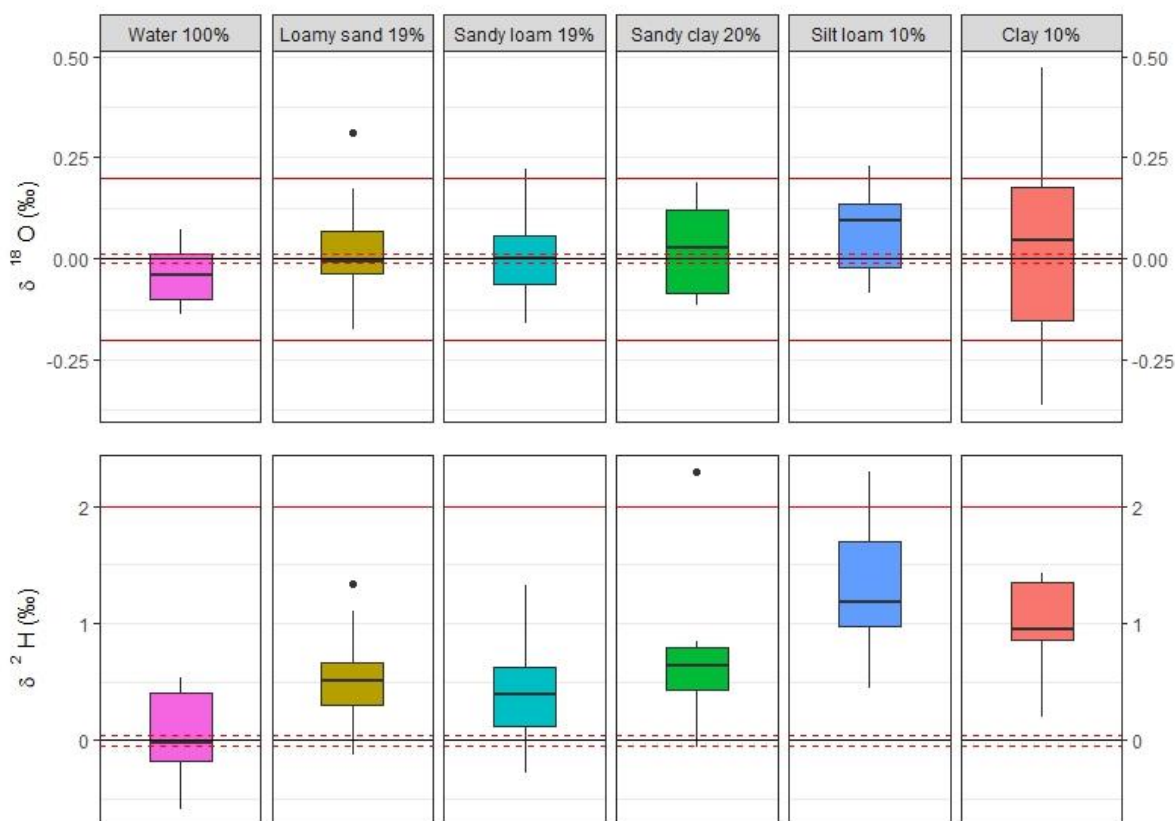
obtained results are within the limits of acceptability (0.2 ‰ for $\delta^{18}\text{O}$ and 2 ‰ for $\delta^2\text{H}$) and support efficiency of the method and apparatus also in this range of soil texture, water content and extreme chemical composition (in case of the Ethiopian soil sample). The results will be included in the revised manuscript.

Added soils:

	Origin	Clay (%)	Silt (%)	Sand (%)	Soil (g)	Water (ml)	W (%)	θ (%)
Silt loam	Czechia	24	60	16	150	15	10	9
Clay	Ethiopia	44	28	28	150	15	10	9

Where W is gravimetric water content and θ is volumetric water content.

The results of newly added tests:



RC:

Title should be tempered to describe the method as giving pilot results – avoid adding qualitative judgement (simple, exact, reliable).

AR:

We understand the reviewer's objection and we propose the following title:

A new laboratory approach to extract soil water for stable isotope analysis from large soil samples

RC:

The authors incorrectly use the terms accuracy and precision in the manuscript, which is highly confusing. In terms of accuracy, it would be more appropriate to use the term “bias” which must be clearly defined as the delta change relative to H and O isotopic offset from the experimental starting water isotopic composition prior to the extraction and analysis. All methods have some form of bias (show stats), and most soil extraction methods show a positive bias due to lack of 100 % recovery or other factors.

AR:

We agree that the correct nomenclature is shift and bias. Our choice of terminology was based on standard terms used in other studies on the same topic (Revesz & Woods (1990), Koeniger et al. (2011), Ignatev et al. (2013), Sprenger et al. (2015), Gaj et al. (2017)). The terminology will be changed to shift and bias. At their first appearance a piece of information will be added that these terms are also referred to as accuracy and precision in some studies.

RC:

Regarding precision – this is mess – they are reporting precision values that are extraordinarily low (for a laser or IRMS – ± 0.06 permil system precision for $\delta^{18}\text{O}$ is frankly, impossible!) and are completely unrealistic from an over systems point of view. The authors should propagate all the sources of system uncertainty, including uncertainty of the primary reference waters (VSMOW, SLAP), laboratory water standards using on the Picarro, replicates of the experimental waters used, and replicated soil porewater extractions. A more realistic reporting of precision in this case is more likely to be inline with other methods. Avoid using hyperbolic terms like “better than” or “more accurate” than other methods – simply show comparative results factually.

AR:

We have added a table with all measured values at the end of this response. Long-term standard deviation of our lab standards used for these purposes – snow water from our experimental area, distilled water and SMOW – are on average 0.05 and 0.36 for $\delta^{18}\text{O}$ and $\delta^2\text{H}$, respectively. However, in the individual runs (the number used for recalculation to the “real values”) the values are similar to the standard deviations of the labelled water reported in our study. The precision reported in our study is lower than that guaranteed by the Picarro, showing the impact of the extraction method and sample handling on the results. Also, in our opinion, it is important that within the Picarro run, the extracted values come out the same as the labelled water used.

RC:

They should also be clear that low Bias (their Accuracy) is only achievable on experimental and manipulated test samples – there is no guarantee of that “low bias” will be obtained on any unknown field samples given the wide range of porosities, grain size and organic matter contents within a single core or soil samples. Urge caution making such sweeping statements based on a few material types.

AR:

We addressed this point in one of the previous responses. We will clarify it in the manuscript.

The real samples can exhibit the larger error. We compare our results with studies performing similar tests of accuracy of their methods with different soil textures. Analogously to e.g.: Revesz and Wood

(1990), West et al. (2006), Koeniger et al. (2011), Munskgaard et al. (2014), Jiang et al. (2021), we used just three materials with different grain compositions.

RC:

Why was 105 °C used? In the literature the T range is wider and higher. What would be the benefits or concerns with other T. Once one citation was chosen.

AR:

We will add more information concerning the selected procedure to the manuscript's methods.

The extraction temperature of 105 °C was chosen based on the standard Czech methodology (ISO 11 465, 1998 - information will be included in the revised version), which is consistent with standard methodologies used in UK (BSI 1377: 105±5 °C) and US (ASTM D2216: 110±5 °C).

According to the standard Czech methodology (ISO 11 465, 1998), it is recommended to dry at 105 °C to a constant weight. For a sample volume of 100 cm³, a drying time of 24 hours is sufficient as well as a drying time of 6 hours for a weight of up to 10-20 g. In our case the soil sample is disturbed, hence, soil water is more exposed to evaporation in the chamber.

O'Kelly (2004) presents that pore water remains in the soil when temperatures below 100 °C are used. At 105 °C – potential oxidation or loss of water of crystallization – difference in weight compared to drying at 60 °C is 0.4 – 0.7 % of dry weight. In case of drying at 60 °C, a disproportionately long time is needed. For example, 40 g of soil required 36 hours drying at 60° before the weight stabilised. It was also shown that the greatest increase in water released from minerals is between 80-90 °C (O'Kelly 2005). Therefore, drying to as low as 80 °C could be considered, but this would be reflected in the extraction time and, as introduced above, would not guarantee that all the water is extracted from the pores. Due to the amount of water extracted, the water released from the minerals does not have much effect and therefore we stayed at 105 °C ± 5 °C

Here are some state standards and recommendations for reference:

American norm – ASTM D2216 – 110 ± 5 °C

STM D2216. Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass; American Society for Testing and Materials: Philadelphia, 1998.

<https://www.astm.org/d2216-19.html>

British norm – BSI 1377 – 105 ± 5 °C

BS1377-2. Methods of Test for Soils for Civil Engineering Purposes, Classification Tests; British Standards Institution: London, 1990.

<https://knowledge.bsigroup.com/products/methods-of-test-for-soils-for-civil-engineering-purposes-classification-tests-and-determination-of-geotechnical-properties?version=standard>

Czech norm – ČSN ISO 11465, 1998 – 105 °C

https://csnonlinefirmy.agentura-cas.cz/html_nahledy/83/51736/51736_nahled.htm

RC:

What happens (during the baking process) if the soil sample, especially those of clay or high fine or organic content compact/matte and seals the inner exposure to drying?

AR:

Since our heating process is comparable to the heating mechanisms used for CVD, we do not see a major issue with this. However, if so it might only affect non-disturbed samples e.g., from soil rings where the soil structure is kept intact and might affect the drying process. Disturbed samples have a larger surface area where the applied temperature might be able to speed up the drying process. Despite this fact, we were still concerned about this phenomenon, which was the reason why we always used a new set of dried samples for the sandy-clay samples, as opposed to the other tests, since we were worried that they could not be re-saturated. At the end of the extraction, we did not observe any significant compaction or sealing of the sample. Within the test samples, there was no significant drop in recovery rate between soil types.

The statement on line 143-144: „In this case, a new sample was prepared for each extraction run as the clay samples could not be re-hydrated after extraction.“ is incorrectly formulated since it was only our assumption.

RC:

How do you know the extraction is complete for unknown samples? Is a secondary gravimetric water content test conducted?

AR:

We supposed that extraction is completed if there is no noticeable moisture in the cooling system apart from the collection vessel. The extraction results were tested by weighing the dry weight of the samples from the extractor which was always lighter or at most the same as the dry weight of the sample from a conventional Memmert dryer, where the sample was also dried at 105 °C for 24 h. This procedure is copied from CVD extraction efficiency determinations and is therefore not unique to our method.

We will make this clearer in the revised manuscript version.

RC:

How do you prevent surface evaporation of the sample during handling?

AR:

Again, this potential issue is not unique to our approach. Thus, we assume that we did not exceed the handling time used by other similar methods (e.g. sampling of soil for the CVD method). Soil samples are handled briskly to prevent both the possible absorption of moisture from the surroundings and evaporation from the soil surface. The laboratory is kept at a constant temperature of 20 °C and humidity around 40 %. Saturation experiments are carried out directly in the evaporation chamber to suppress these effects as much as possible.

RC:

Silicone tubing is highly H₂O gas permeable (look it up) – why was PTFE tubing not used? There could be water loss or gain through the silicone tubing.

AR:

Thank you for the valuable comment. The choice of construction materials is a compromise between handling and operating the extractor and material resistance/neutrality with respect to the extracted water. It is true that PTFE tubing (even better, stainless steel tubing) would limit the vapor diffusion through the pipe walls. However, this would come at the price of too rigid pipe structures unsuitable for manipulation. On the other hand, the engineering estimates of humidity gains and losses during the extraction procedure are less than 0.5 % of the total sample mass, regardless of the amount of water extracted and the extraction time not exceeding 24 h. The estimates are based on the water-silicone solubility and permeability (Barrie & Machin (1969)), 50 % relative humidity in the room outside the extractor, and 8 °C cooling water. At these conditions, the absolute air humidity inside the extractor is higher (during the proceeding extraction) or equal to the ambient air humidity, allowing for minor sample losses (<0.5 %) via vapor permeation when the extraction proceeds, and no losses once the sample is almost or completely dry.

Assumptions for the calculation:

- Constant air properties in the hose and room throughout the extraction (temperature humidity)
 - we have controlled conditions in the laboratory
- Validity of the ideal gas equation of state
- Hose temperature is the same as the gas temperature in the hose (insulation + oven heating)

RC:

What does a system “blank” look like (recirculate for 5h or overnight with no sample) – any moisture collected? – you claim there is moist air at the start – so it cannot be zero. What would be its isotopic composition if condensed from ambient air? Does the system gain over a long blank time?

AR:

If the apparatus is running empty, fine fogging occurs in the cooling system. It has been calculated that based on the characteristics of the apparatus (length of the circuit, air flow rate, temperature used), up to 50 mg of water vapor could remain in the apparatus. The second source of moisture is the silicone tubing itself – which, as mentioned above – can be responsible for sample loss due to its permeability and can also absorb water vapor from the air. The water absorbed in the silicone hoses is released back into the circuit when heated (by calculation estimated to approximately 50 µg). To eliminate the residual moisture, a membrane pump is installed as shown in Figure 1a. If the circuit is purged with a large amount of air for 5 minutes before the extraction starts (assuming the oven is already switched on and the apparatus is purged with hot air), the residual moisture is removed and outflowed from the apparatus by the air pressure at the open end. When the circuit is closed again, the



apparatus is no longer visually fogged (even after 8 hours of operation).

This procedure is not described in the manuscript, since all the results obtained in the work are obtained with these residual humidities. Analysing the results, the amount of this residual water is sufficiently small that it only affects the final extracted sample negligibly.

We are not able to evaluate isotopically this small amount of residual water.

Given that we process samples from one campaign at a time, the possible memory effect is also negligible. However, if we know that we will extract water with very different isotopic compositions, execution of an initial purge between extractions would be appropriate. We will add this information to the revised manuscript version.

RC:

Cooling system – why 8 °C and not a more effective cryo-coolant (< 0 °C)

AR:

Using the tap water for cooling is motivated by its availability, cooling temperature close to the ambient air dew temperature (preventing the ambient air condensation on the cooling loops and possible sample contamination), and prevention of formation of frosting inside the apparatus, which in our experience could increase the risk of blocking the inlet pipes, damaging the glass parts and increase the difficulty of extracted sample handling (frost on the cooler and collecting vessel walls would have to be melted first, prior the sample handling). On top of that, with respect to the vapor pressure at the extraction temperature (105 °C: 121 kPa), there is no such a difference in the extraction rates between using the cooling circuit operated at 8 °C (1 kPa) and the one operating at, for example, -10 °C (0.3 kPa).

RC:

From the photo in Figure 1 – the sample boxes with clip on lids do not look very airtight to me – how was airtight ensured and demonstrated?

AR:

These are stainless steel containers containing an airtight silicon seal on the underside of the lid. A leak test was performed by sucking the air out of the box. The box maintained the low pressure. During extraction, there are no apparent pressure changes inside the boxes that could push the present moisture out or suck in the surrounding air.

RC:

Extraction time – this will depend on many factors like material and porosity etc.

AR:

We will emphasize in the text that the reported extraction times are valid for the soils we used and that the time may vary depending on soil properties, size and amount of water in the sample. We will also emphasize the recommendation to insert disturbed soil samples into the extractor.

RC:

It would be helpful to know what a reasonable uncertainty target for this type of work is. For example, for most hydrological studies (and historically), 2 permil for 2H has been perfectly acceptable, as is 0.2

permit for 18O. Would this not be a more objective bar to compare with? This is never going to be in paleo-climate ice core territory.

AR:

We have already mentioned these values (2 ‰ for $\delta^2\text{H}$ and 0.2 ‰ for $\delta^{18}\text{O}$) in the manuscript (below Figure 5). For objectivity, we compare our results also with other already published methods, as we think it is useful for potential future users to know how the method compares to other approaches.

RC:

The stated Picarro performance precision is absurd – its even lower than IRMS or what the manufacturer reports. Be realistic. No mention of well-known corrections for memory or drift are given (all adding to over uncertainly budget with or without).

AR:

Three standards were always used for all stable isotope analyses, specifically V-SMOW, local distilled water and snow from our experimental station. The standard deviations of these standards during all measurements ranged from 0.018 to 0.03 for $\delta^{18}\text{O}$ and 0.057 to 0.179 for $\delta^2\text{H}$. From these values, a mean was calculated that corresponds to our reported values of measurement error (0.03 for oxygen and 0.15 for hydrogen). The precision reported in our study is lower than that guaranteed by the Picarro for the L2140-i isotopic analyzer which was used in this study (please see Table and the link below).

Picarro precision	$\delta^{18}\text{O}$ ‰	$\delta^2\text{H}$ ‰
What we have stated	0.03	0.15
Picarro Guaranteed performance	0.025	0.1
Picarro Typical performance Standard mode	0.010	0.05
Picarro Typical performance Express mode	0.015	0.05

Reported Picarro performance by Picarro for L2140-i isotopic water analyzer:

https://www.picarro.com/sites/default/files/product_documents/Picarro_L2140-i%20Analyzer%20Datasheet.pdf

RC:

Never use the term “signature” for delta “values”. A signature is a representation of something else. Not the case here.

AR:

Our excuse, the error will be fixed in the text.

RC:

In 3.1 please add the statistical test results and p-values for significance. If you say the results were depleted – is this a mean value observation or a statistically defensible statement, rather odd when the

bias is considerably less than the SD. Variance increased – statistically defensible? Strongly recommend to add fully propagated uncertainty to all reported values in the Tables and Figure 4.

AR:

Kolmogorov-Smirnov test at 5% significance level was performed for all sets of the experimental data to determine their normality. The measured data for all six tests exhibit a normal distribution. Furthermore, one sample t-test was performed at 5% significance level to determine whether the extracted values are significantly different from the standard used in the given test. We will add this to the text.

For the first set of results (extraction test with water only), the mean of the data is not statistically different from the standard used. In the remaining extraction tests, using soil, the mean is always statistically identical to the standard used only in the case of $\delta^{18}\text{O}$. In the case of $\delta^2\text{H}$ values, the null hypothesis was always rejected.

Bootstrapp analysis will be added to the revised manuscript version to show the 95 % confidence interval of the presence of the mean values for individual tests.

Statistical test results						
		Variance	KS p-values	H ₀	t-test p-values	H ₀
1st test	$\delta^{18}\text{O}$	0.004	0.870	TRUE	0.052	TRUE
	$\delta^2\text{H}$	0.134	0.837	TRUE	0.553	TRUE
2nd test	$\delta^{18}\text{O}$	0.007	0.766	TRUE	0.284	TRUE
	$\delta^2\text{H}$	0.126	0.976	TRUE	0.004	FALSE
3rd test	$\delta^{18}\text{O}$	0.018	0.985	TRUE	0.337	TRUE
	$\delta^2\text{H}$	0.270	0.983	TRUE	0.002	FALSE
4th test	$\delta^{18}\text{O}$	0.012	0.786	TRUE	0.440	TRUE
	$\delta^2\text{H}$	0.375	0.228	TRUE	0.004	FALSE
5th test	$\delta^{18}\text{O}$	0.014	0.933	TRUE	0.121	TRUE
	$\delta^2\text{H}$	0.349	0.978	TRUE	$4 \cdot 10^{-4}$	FALSE
6th test	$\delta^{18}\text{O}$	0.068	0.850	TRUE	0.909	TRUE
	$\delta^2\text{H}$	0.162	0.761	TRUE	$4 \cdot 10^{-6}$	FALSE

Kolmogorov-Smirnov H₀: Data set has normal distribution.

t-test H₀: The sample mean is equal to the reference value.

TRUE means accepting the null hypothesis, FALSE means rejecting it.

The values were rounded to three valid decimal figures respecting uncertainty of the experimental errors.

RC:

Regarding the claim of moisture left in the system – was this determined gravimetrically or just visual inspection of droplets?

AR:

The residual moisture mentioned is the residual air humidity (non-condensable), left in the extractor circuit. Based on the estimated gas volume of 4L, the ideal-gas law and equilibrium conditions at 8 °C, the amount of water left in the circuit is approximately 50 mg.

RC:

Define IRMS and IRIS

AR:

In the revised manuscript, both terms will be defined – IRMS as Isotope Ratio Mass Spectrometry and IRIS as Isotope Ratio Infrared Spectroscopy.

RC:

Section 4.3 is not relevant and should be deleted.

AR:

Section 4.3 will be rewritten; however, we feel, it is important for the potential future reader to know how the method compares relatively to other approaches in order to consider its possible use. We assume that after reading our work the reader would go and look for the following data anyway.

RC:

Stick to reporting the experimental pilot results – its premature to compare this to other methods when you have not even compared this method and approach in another laboratory.

AR:

It is a good point, which we considered thoroughly, but we kept the original version. This is totally valid and is common practice in the literature (e.g., see Revesz and Woods 1990; West et al., 2006; Wasenaar et al., 2008; Koeniger et al., 2011; Ignatev et al., 2014; Munskgaard et al., 2014; Jiang et al., 2021). These authors also did not use comparisons from multiple laboratories when presenting their results. However, we would like to trigger the interest of the readers in our method (by comparison with other methods) and we will be happy to conduct this kind of comparison in the future.

RC:

Shift or bias in Table 3?

AR:

Our excuse, we will fix it (the average shifts from the labelled water used \pm its standard deviation).

RC:

N.O. is missing from the Author Contributions.

AR:

Natalie Orłowski is not singled out, but her contribution is included in: „all authors reviewed and edited the manuscript“. We will rewrite this sentence and add abbreviations for all authors.

We would like to express our gratitude to the reviewer for his/her very encouraging and stimulated remarks contributing – we hope – to the substantial improvement of the manuscript. Thank you really very much.

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Table of all measured data:

N	1st test (water)				2nd test (loamy sand)				3rd test (sandy loam)				4th test (sandy clay)			
	Extracted water		Labelled water		Extracted water		Labelled water		Extracted water		Labelled water		Extracted water		Labelled water	
	$\delta^{18}\text{O}$	$\delta^2\text{H}$	$\delta^{18}\text{O}$	$\delta^2\text{H}$	$\delta^{18}\text{O}$	$\delta^2\text{H}$	$\delta^{18}\text{O}$	$\delta^2\text{H}$	$\delta^{18}\text{O}$	$\delta^2\text{H}$	$\delta^{18}\text{O}$	$\delta^2\text{H}$	$\delta^{18}\text{O}$	$\delta^2\text{H}$	$\delta^{18}\text{O}$	$\delta^2\text{H}$
1	-9.74	-66.10	-9.60	-66.25	-9.30	-64.26	-9.23	-64.62	-9.06	-63.36	-9.36	-64.66	-9.64	-75.42	-9.53	-75.85
2	-9.68	-66.49	-9.60	-66.38	-9.36	-64.25	-9.22	-64.52	-9.20	-63.74	-9.37	-64.67	-9.64	-75.98	-9.53	-75.95
3	-9.65	-66.93	-9.62	-66.33	-9.26	-64.16	-9.21	-64.54	-9.15	-63.53	-9.38	-64.77	-9.49	-73.63	-9.53	-75.96
4	-9.73	-66.71	-9.61	-66.39	-9.40	-64.68			-9.44	-64.44			-9.65	-75.55		
5	-9.59	-66.36			-9.22	-64.47			-9.35	-64.08			-9.45	-75.27		
6	-9.60	-66.52			-9.26	-64.62			-9.47	-64.29			-9.39	-75.28		
7	-9.71	-66.41			-9.23	-63.89			-9.34	-63.38			-9.62	-75.20		
8	-9.57	-65.83			-9.15	-63.92			-9.28	-64.31			-9.52	-75.89		
9	-9.59	-65.92			-9.1	-63.46			-9.43	-64.70			-9.39	-75.31		
10	-9.54	-65.94			-9.22	-64.04			-9.21	-64.01			-9.35	-75.07		
11	-9.71	-66.04			-9.25	-64.04			-9.5	-64.97			-9.51	-75.07		
12	-9.64	-65.81							-9.37	-64.58						
13	-9.64	-66.09							-9.53	-64.97						
14									-9.31	-64.12						
15									-9.41	-64.32						
Average	-9.65	-66.28	-9.605	-66.337	-9.25	-64.16	-9.22	-64.56	-9.34	-61.19	-9.37	-64.70	-9.51	-75.24	-9.54	-75.92
SD	0.06	0.35	0.01	0.05	0.08	0.34	0.01	0.04	0.13	0.5	0.01	0.05	0.11	0.58	0.01	0.05

5th test (silt loam)				6th test (clay)			
Extracted water		Labelled water		Extracted water		Labelled water	
$\delta^{18}\text{O}$	$\delta^2\text{H}$	$\delta^{18}\text{O}$	$\delta^2\text{H}$	$\delta^{18}\text{O}$	$\delta^2\text{H}$	$\delta^{18}\text{O}$	$\delta^2\text{H}$
-9.43	-64.22	-9.34	-66.12	-9.17	-65.28	-9.34	-66.12
-9.25	-63.77	-9.33	-66.02	-9.11	-65.18	-9.33	-66.02
-9.12	-64.41	-9.38	-66.05	-8.88	-65.16	-9.38	-66.05
-9.13	-64.78			-9.31	-65.86		
-9.24	-64.98			-9.30	-64.64		
-9.25	-65.17			-9.70	-65.76		
-9.36	-65.06			-9.43	-64.72		
-9.41	-65.62			-9.17	-64.65		
				-9.71	-65.11		
				-9.37	-65.12		
				-9.70	-65.09		
				-9.23	-64.70		
-9.27	-64.75	-9.35	-66.06	-9.34	-65.11	-9.35	-66.06
0.11	0.55	0.02	0.05	0.25	0.39	0.02	0.05