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

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15 **Abstract.** Water stable isotope analysis in ecohydrological studies often requires soil water extraction. Here, we present a new soil water extraction method based on the principle of complete evaporation and condensation of the soil water in a close circuit. We have developed an apparatus that has four extraction slots and can be used multiple times a day. Thanks to its simple design, there is no need for any chemicals, gases, high pressure or high-temperature regimes. A set of system functionality tests confirmed that the extraction method has high accuracy and high precision and does not cause 20 any isotope fractionation effects leading to erroneous results. When extracting pure water samples, the accuracy is 0.04 ‰ and 0.06 ‰ for $\delta^{18}O$ and $\delta^{2}H$, respectively, with a precision of $\pm$ 0.06 ‰ and $\pm$ 0.35 ‰ respectively. Soil water extraction tests were conducted with three soil types (loamy sand, sandy loam and sandy clay) using 50-80 grams of soil and water content of 20 %. The accuracy for the extraction of oven-dried and rehydrated soils ranged between -0.04 and 0.03 ‰ for $\delta^{18}O$ and 0.06 and 0.68 ‰ for $\delta^{2}H$ with precision of $\pm$ 0.06 to 0.13 ‰ and $\pm$ 0.34 to 0.58 ‰ for $\delta^{18}O$ and $\delta^{2}H$, 25 respectively in individual tests. These results are more accurate than results achieved by cryogenic vacuum extraction, which is the most widely used extraction method for soils. So far, our method was only tested for soil water extractions.

1 Introduction

Measurements of soil water isotopic composition (^{2}H and ^{18}O) are useful tool to describe soil water movement and mixing processes in the vadose zone (Stumpp et al., 2018). In some cases, different trends or just proof that two soil water samples 30 are different without knowing the absolutely exact isotopic composition (tracer experiments to prove interconnection) may be sufficient. For inter-laboratory comparison, characterizing the transport processes and residence time, accurate proof of sample origin or soil water dynamics modelling, the exact value of the isotopic composition is required. For all these

purposes, soil water extraction is necessary. Unlike liquid water samples of precipitation, snow cover, stream or groundwater, where the isotopic composition is easily accessible, the extraction of matrix-bound soil water or tightly bound

35 soil water is challenging when it comes to exact values of isotopic composition. It has been shown that the storage and sample preparation for extraction, soil texture, soil water content as well as organic matter and carbonate content have a major impact on the final results (West et al., 2006; Wassenaar et al., 2008; Koeniger et al., 2011; Meißner et al., 2014; Hendry et al., 2015; Orlowski et al., 2016a; Newberry et al., 2017). Also, the specifics of extraction methods e.g. the different pore spaces that may or may not be extracted via the different approaches (Orlowski et al., 2019; Kübert et al.,

40 2020) and the modification of the procedures themselves (Orlowski et., 2018) can affect the isotope results.

There are many extraction methods to choose from and several studies that compare them (Zhu et al., 2014; Sprenger et al., 2015; Orlowski et al., 2016b, 2018). For a brief overview, there are methods using a) various chemical compounds or elements like toluene for azeotropic distillation (Revesz and Woods, 1990; Thorburn et al., 1993), dichloromethane for accelerated solvent extraction techniques (Zhu et al., 2014), zinc or uranium for microdistillation

45 (Kendall and Coplen, 1985; Brumsack et al., 1992); b) microwave water extraction (Munksgard et al., 2014); c) force in terms of mechanical squeezing (Wershaw et al., 1966; White et al., 1985; Böttcher et al., 1997) or centrifugation (Mubarak and Olsen, 1976; Batley and Giles, 1979; Barrow and Whelan, 1980; Peters and Yakir, 2008); d) equilibration methods such as in situ equilibration (Garvelmann et al., 2012; Rothfuss et al., 2013, 2015; Volkmann and Weiler, 2014; Gaj et al., 2016), CO_2- and H_2-equilibration (Jusserand, 1980; Scrimgeour, 1995; Hsieh et al., 1998; McConville et al., 1999; Koehler et al.,

50 2000; Kelln et al., 2001) and the direct liquid-vapour equilibrium laser spectroscopy (DVE-LS) method (Wassenaar et al., 2008; Hendry et al., 2015) and e) cryogenic vacuum extraction (CVE) (Dalton, 1988; West et al., 2006; Koeniger et al., 2011; Goebel and Lascano, 2012; Orlowski et al., 2013, 2016; Gaj et al., 2017), modified CVE – He-purging method (Ignatev et al., 2013) and automatic cryogenic vacuum distillation (ACVD) system LI-2100 (Lica United Technology Limited Inc.). In addition, many laboratories use modifications of these methods (Walker et al., 1994; Munksgaard et al.,

55 2014; Orlowski et al., 2018). For a more detailed description of the methods, we refer to Sprenger et al. (2015) and Ceperley et al. (2024).

The most commonly used methods for soil water extraction today are DVE-LS and CVE. Both provide very accurate results, but only under certain conditions. For DVE-LS, the different equilibrium times, low water content as well as the selection of bags play a crucial role (Hendry et al. 2015; Grahler et al., 2016). Also, it has been shown, that soil samples

60 with high content of fine particles, thus high soil tension can cause isotope fractionation in closed systems (Gaj and McDonell, 2019). Soils containing clay minerals are also a major limitation of CVE. It has been shown that higher temperature is needed for such soils (up to 300 °C). This can affect the results by releasing water by oxidation of organics and dihydroxylation of hydroxide-containing minerals such as goethite (Gaj et al., 2017). Another disadvantage of CVE is that obtained results are not incomparable between different laboratories due to apparatus modifications and different

65 workflows (Orlowski et al., 2018). Laboratories' differences in their setup are: the extraction containers (form, size, volume, and material), the heating module and its application temperature (heating tapes or lamps, water baths or hot plates), the type

of fittings and connections (glass, stainless steel), and the vacuum-producing units. In addition, different temperatures, pressures, extraction times and sample sizes are used by different laboratories. However, if the certain setup of all these parameters for the given situation is chosen, very accurate results can be achieved for certain soil types and water contents.

70   In this study, we present a new extraction method. It is an undemanding and relatively inexpensive method that can handle soil samples of different sizes, moisture contents and textures. It is based on the simple principle of complete evaporation and condensation in a closed circuit and does not require working with hazardous substances (acids, toluene, liquid nitrogen), high temperatures or pressures. In the following, we (1) introduce the new extraction principle, (2) present the results of soil extraction efficiency testing, and (3) compare the results with other state-of-the-art approaches. The

75   advantage of this method over others is its excellent accuracy, even with clay samples, which are known to cause inaccurate results for other extraction methods (Ceperley et al., 2024). However, this accuracy is at the expense of lower throughput. Therefore, rather than for large-scale studies, this method is suitable for studies where quality is more important than quantity.

2 Methodology

80 ### 2.1 Principle of extraction

Circulating air extraction method (CAEM) is based on the principle of complete evaporation and subsequent condensation of soil water in a closed circuit, with air as the circulating medium. The soil sample is heated to 105 °C inside the evaporation chamber, and the evaporated soil water is carried by air circulation to a cooling unit. There, the air moisture is condensed and the liquid water is collected. Dried cool air is then circulated back into the evaporation chamber. The process continues until

85   there is no visible air moisture condensation.

2.2 Description of the apparatus

The newly designed apparatus (Fig. 1a) consists of three main units – the heating system, the cooling system and the air circulation system (Fig. 2). The apparatus consists of four separate circuits allowing for simultaneous extraction of water from four soil samples. The heating system comprises a standard kitchen oven (model VT 332 CX; MORA MORAVIA s. r.

90   o., Czechia) housing four evaporation chambers – stainless steel boxes equipped with an airtight insulation. Each box has two openings, one for dry air inlet and second for moist air outlet. The soil sample inside the box is placed on a stainless-steel wire-mesh bed allowing for good contact between the sample and air, enhancing the water evaporation rate (Fig. 1b). The dry air is led to the evaporation chamber through a silicone rubber tube coiled inside the oven; its length (~ 2 m) is sufficient to preheat the air close to the oven temperature (Fig. 1c). The hot and moist air from the evaporation chamber is

95   led through the insulated silicone tube to the cooling system; the length of the outlet tubes is as short as possible to minimize the heat losses and prevent undesired water condensation. To monitor the extraction process, a temperature sensor is installed inside each box close to the air outlet. The cooling system consists of three glass components – spiral cooler,
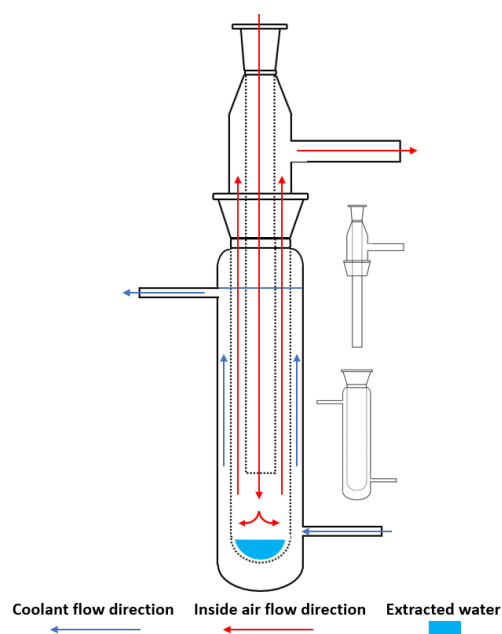
custom-made connecting part and jacketed collecting vessel (Fig. 3). Both, the spiral cooler and collecting vessel are cooled using a tap water ($\sim$ 8 °C); two separate cooling water circuits are used for the spiral coolers and for the collecting vessels

100 (Fig. 2). The cooled and dried air from the cooling system is fed back to the evaporation chamber by means the air circulation system comprising two regulated high-speed fans per circuit ensuring the air flow rate of $\sim$ 10 L/min. The temperature sensors and fan speed for each circuit are monitored by the control unit running on the Arduino platform.

105 **Figure 1: Photo of the newly designed apparatus (a); detail of the inside of the heating chamber with wire-mesh bed and aluminium fabric bedding (b); internal arrangement of heating chambers and coiled supply hoses (c).**
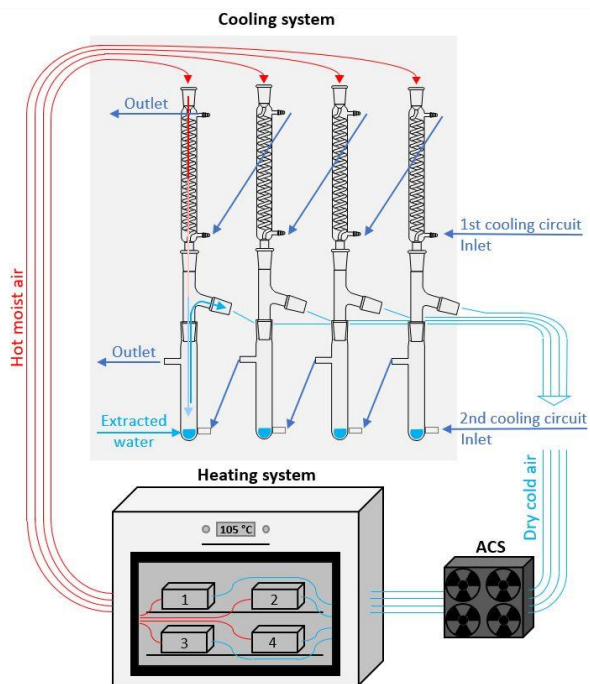
Figure 2: Simplified diagram of the three main components of the apparatus (heating system, cooling system and air circulation system (ACS)). The apparatus consists of four separate drying circuits and two cooling circuits.

Figure 3: Lower part of the cooling system – custom-made connecting part and jacketed collecting vessel. The arrows indicate the direction of flow within the assembly. Thumbnails show individual parts before assembly.

2.3 Extraction procedure

Soil samples are inserted into the evaporation chambers on the wire bed. A standard temperature for drying soil samples of 105°C (Suchara, 2007) is reached approximately 15 minutes after the start-up. From that time, the first intensive part of the drying process, during which both cooling circuits operate and most of the water is extracted, is in process. The upper cooling circuit (Fig.2) is disconnected once the spiral cooler starts to dry out. The extraction continues with the bottom cooling circuit only. During this time, residual moisture in the apparatus is collected. Once there are no visible signs of moisture on the walls of the cooling apparatus, the extraction is complete. Depending on the sample type, water content, and size, the extraction times range from 3 to 5 hours per sample. Please note that not all water from the circuit is collected as some of it remains in the form of residual air humidity; based on the circuit volume ($\sim$ 5 L) and cooling water temperature ($\sim$ 8 °C), the residual water content of $\sim$ 50 mg is estimated. Between each extraction, the circuit is disassembled to retrieve the extracted water and exchange soil samples. The collection vessel must be dried between individual extractions to avoid contamination with water from previous extractions.

2.4 Functional tests

In total, four functional tests were performed with twelve repetitions for each. All the tests aimed to recover the same amount of water that was used for the test without any changes in its isotopic composition. The first test served to verify the principle of the extraction and the waterproofing and airtightness of the apparatus. The second, third and fourth tests verified the accuracy of the extraction with soil samples via spike experiments (Orlowski et al., 2018). In total, three sets of spike experiments with different soil textures were performed because it is well-known, that soil texture plays a crucial role during soil water extraction (Orlowski et al., 2016a).

For the first test, only water of known isotopic composition and quantity (15 ml) was inserted into the heating chambers. For the second test, disturbed soil samples (80 g each) of loamy sand texture were first oven-dried at 105 °C for 24 hours and then spiked with 15 ml of water of known isotopic composition. The soil samples were reused and re-hydrated in total 4 times in this experiment. In the third test, the procedure was the same, with the use of sandy loam soil samples (Tab. 1).

For the fourth test, 50 g of samples were prepared in the laboratory by mixing sand with clay in proportions of 60 % sand and 40 % clay. They were oven-dried at 105 °C for 24 hours and then spiked with 10 ml of water of known isotopic composition. A smaller sample size and amount of water were used to reduce the extraction time. In this case, a new sample was prepared for each extraction run as the clay samples could not be re-hydrated after extraction.

Table 1: Parameters of samples used to verify the functionality of the apparatus.

Test	Water (ml)	Soil (g)	Water content (%)	Soil texture	% sand	% silt	% clay
1st	15	-	100	-	-	-	-
2nd	15	80	18.75	loamy sand	85.5	5.5	9
3rd	15	80	18.75	sandy loam	56.5	34.8	8.7
4th	10	50	20	sandy clay	60	-	40

Labelled water with a slightly different stable isotope composition was used for each test (Tab. 2). Isotopic signatures were analysed at the Institute of Hydrodynamics (Czech Academy of Sciences) on a L2140-i isotope analyser (Picarro Inc., US). Standard mode (precision of $\pm$ 0.03 and $\pm$ 0.15 ‰ for $\delta^{18}O$ and δ^2H, respectively) was used with 6 injections per sample with first 3 injections discarded. The isotope ratios are reported in per mil (‰) relative to Vienna Standard Mean Ocean Water (VSMOW) (δ^2H or $\delta^{18}O = (R_{sample}/R_{standard}-1) \times 1000$ ‰), where R is the isotope ratio of the sample and the known reference (i.e., VSMOW) (Craig, 1961).

3 Results

3.1 Waterproof and airtightness test

To test the extraction method and the water- and airtightness of the apparatus (1st test), 15 ml of water of known isotopic composition was used. Extraction of this amount of water took on average 5 hours. The resulting sample quantity after the extraction process averaged 99.7 % of the volume of the used labelled water. The missing fractions of grams of water were mainly due to the residual thin moisture film that remained on the walls inside the collection vessel. The stable isotope signature of labelled water used for this test was -9.61 $\pm$ 0.01 ‰ for $\delta^{18}O$ and -66.34 $\pm$ 0.05 ‰ for δ^2H with a d-excess of 10.5 ‰ (N=3) (Tab. 2). The resulting average of mean stable isotope composition of extracted water (N=12) was depleted by 0.04 $\pm$ 0.06 ‰ in $\delta^{18}O$ and enriched by 0.06 $\pm$ 0.35 ‰ in δ^2H which is within measurement inaccuracy of the isotope analyser. The d-excess increased to 10.9 ‰ (Fig. 4).

3.2 Spike experiments

The other three tests – spike experiments – to verify the functionality of the extraction took on average 3 hours for the second (loamy sand), 4 hours for the third (sandy loam) tests and 5 hours for the fourth test (sandy clay). The resulting recovery rate after the extraction process averaged 99.2 % of the volume of the used labelled water. The missing fractions of grams of water were mainly due to the residual thin moisture film that remained on the walls inside the collection vessel. For the second test, the stable isotope signature of labelled water was -9.22 $\pm$ 0.01 ‰ for $\delta^{18}O$ and -64.56 $\pm$ 0.04 ‰ for δ^2H with d-excess of 9.2 ‰ (N=3) (Tab. 2). The average obtained isotopic signature was depleted by 0.03 $\pm$ 0.08 ‰ in $\delta^{18}O$ and

enriched by 0.4 ± 0.34 ‰ in δ^2H (N=12). As in the first test, the δ^{18}O values were slightly depleted but almost matched the labelled water. However, the δ^2H values were relatively enriched and thus the d-excess increased to 9.8 ‰ (Fig. 4).

175   For the third test, the stable isotope signature of labelled water was -9.37 ± 0.01 ‰ for δ^{18}O and -64.70 ± 0.05 ‰ for δ^2H with d-excess of 10.3 ‰ (N=3). The mean isotope composition of extracted water was enriched for both isotopes. For δ^{18}O by 0.03 ± 0.13 ‰ and for δ^2H by 0.51 ± 0.5 ‰ and the d-excess increased to 10.5 ‰ (N=12). Compared to the second tests, the variance of the values has increased.

     For the fourth test, the stable isotope signature of labelled water was -9.54 ± 0.01 ‰ for δ^{18}O and -75.92 ± 0.05 ‰ 180 for δ^2H with d-excess of 0.4 ‰ (N=3). The mean isotope composition of extracted water was enriched in both isotopes. For δ^{18}O by 0.03 ± 0.11 ‰ and for δ^2H by 0.68 ± 0.58 ‰. The d-excess increased to 0.9 ‰ (N=12).

     While the average δ^{18}O values were almost identical to the original values or have a minimal deviation in all four tests (the resulting values are within measurement inaccuracy of the isotope analyser), the δ^2H values were in all cases slightly enriched and therefore the d-excess increased. We observed a higher deviation of the values from the spike water 185 with a higher amount of fine particles (silt, clay), but the average remained satisfactory. The complete set of results is summarized in Table 2 and Figure 4.

Table 2: Summary of individual test results.

	1st test		2nd test		3rd test		4th test	
	LW	EW	LW	EW	LW	EW	LW	EW
δ^{18}O	-9.61	-9.65	-9.22	-9.25	-9.37	-9.34	-9.54	-9.51
SD	± 0.01	± 0.06	± 0.01	± 0.08	± 0.01	± 0.13	± 0.01	± 0.11
δ^2H	-66.34	-66.28	-64.56	-64.16	-64.70	-64.19	-75.92	-75.24
SD	± 0.05	± 0.35	± 0.04	±0.34	± 0.05	± 0.50	± 0.05	± 0.58
D-ex	10.5	10.9	9.2	9.8	10.3	10.5	0.4	0.9
SD	± 0.2	± 0.5	± 0.1	± 0.5	± 0.04	± 0.7	± 0.1	± 0.9
N	3	12	3	12	3	12	3	12
Sample type	Water		Loamy sand		Sandy loam		Sandy Clay	
Extraction time (h)	5		3		4		5	

LW indicates the labelled water used in the test, EW indicates the extracted water. The isotope ratios (δ^{18}O, δ^2H) and their 190   *standard deviations (SD) are reported in per mil (‰) relative to Vienna Standard Mean Ocean Water (VSMOW). D-ex stands for Deuterium excess and N stands for the number of samples.*
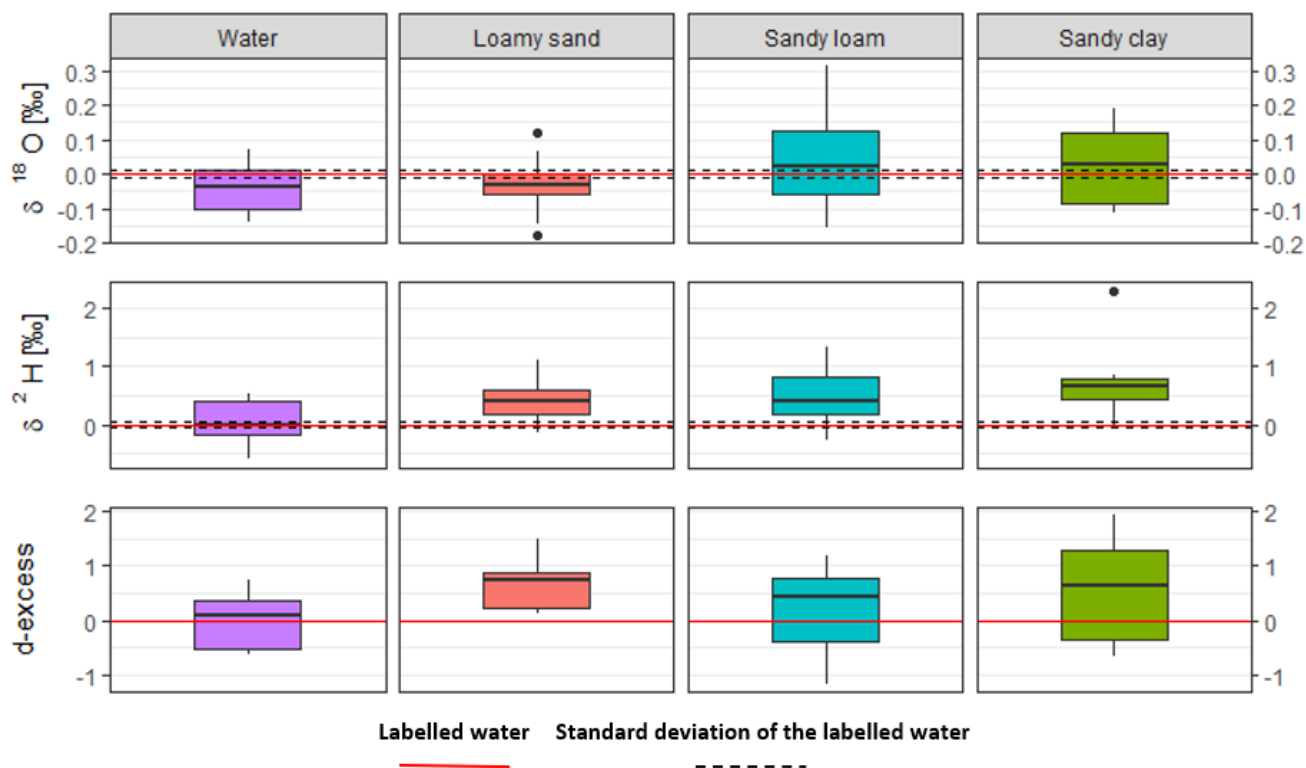
Figure 4: Relative deviation of the isotopic ratio of extracted water compared to the labelled water (red line) and its standard deviation (black dashed line). For better clarity, all results are recalculated as if the used labelled water had a VSMOW composition.

4 Discussion

4.1 Residual moisture in the apparatus

The apparatus is designed to handle an entire standard soil core (100 cm³). The sample size is limited only by the size of the heating chamber (roughly 400 cm³ of usable space) and the size of the collection vessel (25 ml). An advantage of extracting a bigger soil sample, is that it represents a larger area in the soil in comparison to smaller subsamples (e.g.< 10g), which might not be as representative. For this reason, the extraction time and total throughput are lower compared to other methods (e.g. CVE, DVE-LS). The large amount of obtained extracted water with our extraction apparatus might overprint a potential inaccuracy due to lower sampling amounts in other extraction methods. Additionally, it offers the advantage of running the same extracted water sample on an IRMS or IRIS machine. We are aware that even with this apparatus not all water ends up in the collection vessel, but a negligible fraction of molecules (approx. 50 mg) remain somewhere inside the circuit. However, this amount is marginal compared to the amount of water extracted, so it does not have a major effect on the results (as demonstrated).

In addition to the residual moisture impact on the extracted water, some sample contamination is possible also from

210   the initial air humidity prior to the extraction. However, similarly to the residual moisture, this initial air-water content is estimated as marginal, accounting for less than 50 mg of the water sample.

Both these potential error sources can be suppressed by using larger sample sizes. However, for even more accurate results, it might help to completely seal the apparatus during idle time, pre-drying the empty apparatus or purging the apparatus with dry air, or nitrogen (as inert gases). However, the extraction procedure would be more complicated and the

215   nuances that this would resolve are negligible in comparison to other factors (e. g. the amount of clay in the sample, the accuracy of measurements of the stable isotope composition itself) which will affect the final composition much more.

The missing fractions of grams in the recovery rate are not due to residual moisture in the apparatus, which will contribute only a very small fraction to this error. The incomplete recovery rate is mainly due to the water adhering to the walls of the collecting vessel, whereby the residual amount always remains there while pouring the sample into the vials.

220   Thorough mixing of the sample before pouring and catching all droplets from the walls will ensure homogeneity of the sample. Thus, the residual film in the glass will not affect the isotopic composition of the sample after that.

4.2 Extraction time

For many methods, extraction time often plays a significant role in the resulting isotopic composition of the sample (Revesz & Woods, 1990; West et al., 2006; Zhu et al., 2014; Hendry et al., 2015; Orlowski et al., 2018; Orlowski & Bauer, 2020). In

225   our case, we did not observe any significant differences between ending the extraction at the time when the circuit is visibly dry or prolonging the extraction by an hour or more, because the same dry, cold air is still flowing when the extraction is completed. Once the extraction is complete, the apparatus reaches an equilibrium state at which the amount and composition of water sample does not change. Our method is among the slower ones when compared to other extraction methods. The extraction time varies between 3 to 5 hours depending on the soil texture; the larger surface area and porosity of the sample

230   reduces the extraction time significantly. The extraction time using CVE ranges from 15 minutes (Orlowski et al., 2018) to 6 hours (Mora & Jahren, 2003). However, it should be added that for CVE, sample sizes of 10-20 g are used, whereas for our extraction the sample size were 50 and 80 g and obtained liquid water amounts up to 15 ml.

In large-scale studies, high sample throughput is an important factor. For these purposes, apparatuses with high throughput that can handle 30 or more samples in an 8-hour working day are used (Goebel & Lascano, 2012; Orlowski et al.,

235   2013; Yang et al, 2023). Our apparatus currently has only four circuits, so four soil samples can be processed at the same time. Depending on the soil type and water content a maximum of two runs per day can be processed. Rather than for large-scale studies and obtaining trends instead of exact values, the apparatus is suitable for studies where high precision is needed to unambiguously separate the different water pools (McDonell, 2014). Reducing the sample size would increase the throughput by reducing the extraction time, but it could affect the accuracy of the results. To apply this method in large-scale

240   studies, it would be necessary to either use more of these apparatuses, change the heating source and use a larger oven to fit more heating chambers or increase the circulation speed in the apparatus.

4.3 Comparison of soil water extraction approaches

In order to compare our method of soil water extraction with other approaches, we gathered precision and accuracy values presented in scientific papers. The results showed (Tab. 3 and Fig. 5) that the presented method has high accuracy and high

245   precision compared to others. With clay rich soil sample we achieve better results than DVE-LS (Wassenaar et al., 2008), which has a high precision (± 0.02 ‰ and ± 0.5 ‰ for $\delta^{18}O$ and $\delta^{2}H$, respectively) but low accuracy (+ 1 ‰ and + 2 ‰ for $\delta^{18}O$ and $\delta^{2}H$, respectively). A direct comparison with the most commonly used method, CVE, is difficult, given the huge range of values presented by different laboratories (Orlowski et al., 2016b, 2018). In this study, we used the reported values of Yang et al. (2023), Newberry et al. (2017) and Koeniger et al. (2011) as a reference. The reported accuracy was between -

250   0.16 to -0.59 ‰ and -2.6 to 2 ‰ for $\delta^{18}O$ and $\delta^{2}H$, respectively and the precision between ± 0.14 to 0.4 ‰ and ± 1.3 to 3 ‰ for $\delta^{18}O$ and $\delta^{2}H$, respectively, with the worst accuracy for clayey soils. Compared to all these cases, our method performed better by more than one order. These reported values are depleted in both isotopes which is the opposite of most of the values reported in our study. Orlowski et al. (2016b) showed, that in the case of extraction from sandy samples, the extracted water by CVE is almost identical to the applied label water. However, as the proportion of clay particles in the sample

255   increases, the accuracy decreases greatly and the difference to the labelled water for clay samples is more than 1.5 and 12 ‰ for $\delta^{18}O$ and $\delta^{2}H$, respectively. In our case, only a gradual decrease in accuracy is visible with increasing amount of clay in the sample. For both isotopes, there is a higher enrichment of heavy isotopes in the sample and the dispersion of the values increases. However, the results of all tests are safely below the limits of ± 0.2 ‰ for $\delta^{18}O$ and ± 2 ‰ for $\delta^{2}H$, which is considered reasonable for hydrologic studies (Wassenaar et al., 2012).

260   Many laboratories also have considerable problems with the extraction of water alone (Orlowski et al., 2018). The best reported accuracy and precision of extracted water in the interlaboratory study by Orlowski et al. (2018) were 0.1 ± 0.1 ‰ for $\delta^{18}O$ and -0.8 ± 0.4‰ for $\delta^{2}H$, which was again almost an order of magnitude worse than in our case. But only 2 of the 16 laboratories in the CVD interlaboratory comparison study (Orlowski et al., 2018) were able to obtain such results, with the others being much worse. This proves that the problem with accuracy is not the method itself (CVE can give very

265   accurate results), but it depends on the settings of the apparatus. Also, slight differences may occur due to the measurement of the isotopic composition itself, depending on the instrument and method used (Penna et al., 2010).

The only method that produced comparable results to our study is a modification of CVE by Ignatev et al. (2013), which used He as carrier gas instead of water vapour diffusion only. Mass transfer coupled with gas flow has proven to be a more effective process compared to diffusive mass transfer (Ishimaru et al., 1992) and because of that, much more accurate

270   results can be achieved. Accuracy and precision of this method were 0.03 ± 0.08 ‰ and 0.7 ± 0.7 ‰ for $\delta^{18}O$ and $\delta^{2}H$, respectively. In comparison with our method, $\delta^{18}O$ results were better for the He-purging method and $\delta^{2}H$ on the contrary for our method. However, it should be noted that these differences of hundredths ($\delta^{18}O$) to units of tenths ($\delta^{2}H$) are mostly within measurement inaccuracy of an isotope analyser. Compared to other methods (extraction with accelerated solvent,

centrifugation, and azeotropic distillation), the accuracy difference in our case is within an order of magnitude higher (Zhu et

275 al., 2014; Leaney et al., 1993; Revesz & Woods, 1990).

Table 3: Comparison of accuracy and precision of selected soil water extraction methods in different studies.

Method	Study	Sample type	Average δ^{18}O shift ± SD (‰)	Average δ^2H shift ± SD (‰)
Extraction with accelerated solvent	Zhu et al. (2014)	unknown soil	0.36 ± 0.37	3.6 ± 0.89
Azeotropic distillation	Revesz & Woods (1990)	Sandy soil	0.35–0.77 ± 0.2	2–3.2 ± 2
Ultrasonic centrifugation	Zhue et al. (2014)	unknown soil	0.49	1
Centrifugation	Leaney et al. (1993)	Clayey soil	0–3	-
Direct equilibrium	Scrimgeour (1995)	unknown soil	-1.5– -0.11 ± 0.4	– ± 2
Direct equilibrium	Mcconville et al. (1999)	Sandy soil	3 ± 0.12	-
Direct equilibrium	Wasenaar et al. (2008)	Clay-rich soil	1 ± 0.02	2 ± 0.5
ACVD	Yang et al. (2023)	Clay loam	-0.16 ± 0.14	-2.6 ± 1.3
CVE	Koeniger et al. (2011)	Sandy soil	– ± 0.4	– ± 3
CVE	Newberry et al. (2017)	Sandy soil	-0.59	-
CVE	Orlowski et al. (2018)	Water	0.1 ± 0.1	-0.8 ± 0.4
He-purging	Ignatev et al. (2013)	Clay & silt	0.03 ± 0.08	0.7 ± 0.7
CAEM (our study)	1st test	Water	-0.04 ± 0.06	0.06 ± 0.35
	2nd test	Loamy sand	-0.03 ± 0.08	0.40 ± 0.34
	3rd test	Sandy loam	0.03 ± 0.13	0.51 ± 0.50
	4th test	Sandy clay	0.03 ± 0.11	0.68 ± 0.58

ACVD stands for automatic cryogenic vacuum distillation, CVE stands for cryogenic vacuum extraction and CAEM stands

for Circulating air extraction method. The CVE results from the study by Orlowski et al. (2018) show only the best results

280 *achieved in the comparison of CVEs made in that study. Average $\delta^{18}O$ and δ^2H shifts represent deviation from the mean of*

used labelled waters (accuracy). SD stands for standard deviation (precision).
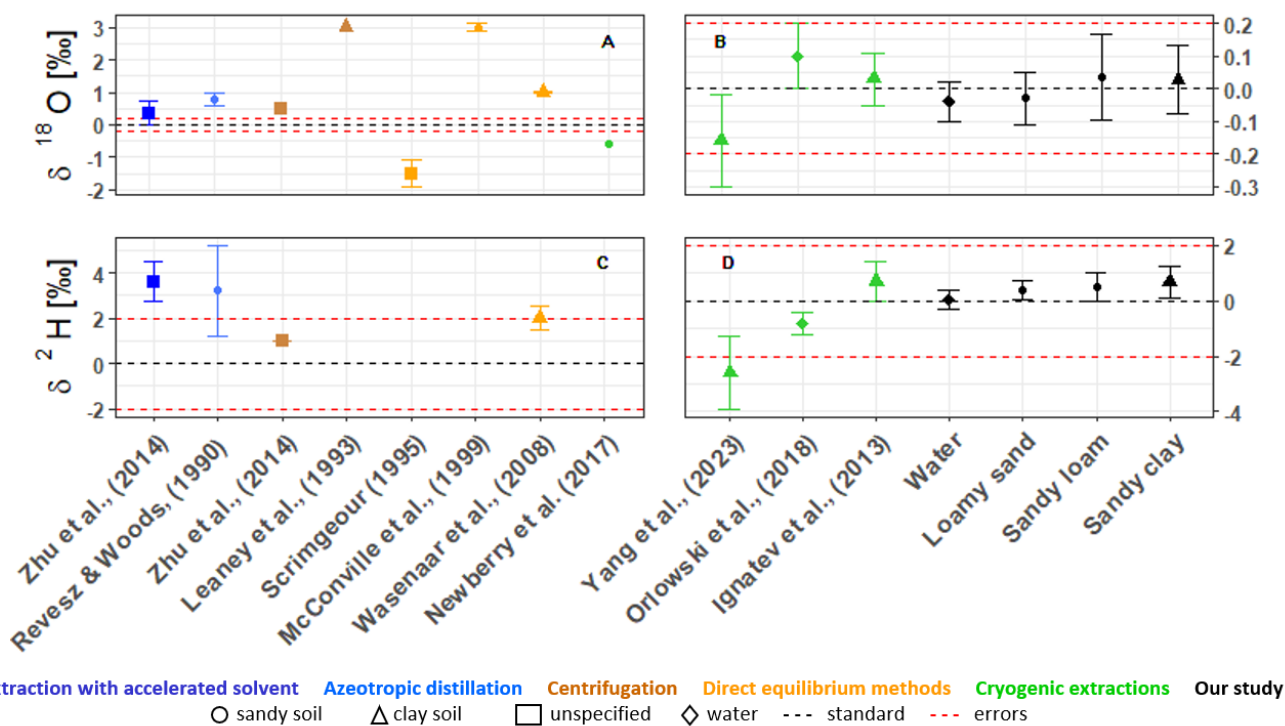
Figure 5: Graphical comparison of our results with other methods (A, B for $\delta^{18}O$; C, D for δ^2H). Different markings indicate
285 different sample types. Dashed black line represent the standards used in those tests. Dashed red lines represent errors of ± 0.2 ‰
for $\delta^{18}O$ and ± 2 ‰ for δ^2H, which is considered reasonable for hydrologic studies (Wassenaar et al. 2012). The right side of
oxygen graph (B) with more accurate methods has a zoomed y-axis.

5 Conclusion

In this study, we presented a new method for soil water extraction – Circulating air extraction method (CAEM) – that works
290 on the principle of complete evaporation and condensation in a closed circuit and the apparatus developed for this purpose.
We successfully extracted the soil water from dried and rehydrated soil samples (soil types: loamy sand, sandy loam and
sandy clay) with an accuracy between -0.04 and 0.03 ‰ for $\delta^{18}O$ and 0.06 and 0.68 ‰ for δ^2H and a precision of ± 0.06 to
0.13 ‰ and ± 0.34 to 0.58 ‰ for $\delta^{18}O$ and δ^2H, respectively in individual tests depending on the soil texture. The differences
between extracted and used label water were often within measurement inaccuracy of the isotope analyser. It provides a
295 better accuracy of results than most other soil water extraction/equilibration methods such as CVE and DVE-LS and up to an

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order of magnitude better results than other methods (extraction with accelerated solvent, centrifugation, azeotropic distillation). The developed apparatus for this method has a medium throughput with a maximum of eight samples a day, high accuracy and high precision. The method has proven to handle various soil types with different soil texture and is suitable for experiments and studies where high precision is required to distinguish individual water pools and where mere

300   trend detection is not sufficient.

Appendix

Table A1: List of used components

Components	Type	Supplier	Reference	Quantity	Price [€]
Oven	VT 332 CX	MORA MORAVIA, s.r.o	https://www.alza.cz/mora-vt-332-cx-d6977919.htm?o=1	1	218.76
Aluminium profile	40x40 - 104040	ALUTEC KK, s.r.o.	https://katalog.aluteckk.cz/produkt/profil-40x40-104040/	13 m	448.61
Silicon tube	R973851; R098081	P-LAB	https://www.p-lab.cz/hadicka-silikonova-silnostenna?search=hadice	25 m	509.56
Spiral cooler	Dimroth 14/23	VERKON, s.r.o.	https://www.verkon.cz/chladic-spiralovy-dle-dimrotha/?keyword=dimrotha	4	549.34
Customized glass	Figure 3	Institute of Chemical Technology in Prague		8	238.69
Glass elbow	14/23	VERKON, s.r.o.	https://www.verkon.cz/koleno-s-nz/?keyword=koleno	4	45.03
Stainless steel bowl	1400 ml	GoEco	https://www.dedra.cz/sk/da30751-dozivotni-celonerezova-doza	4	95.48
Temperature sensors	(TP-01) K	HOTAIR	https://www.hotair.cz/detail/merici-pristroje/teplomery-a-sondy/termoclankova-sonda-typu-k-tp-01-s-kevlarovou-izolaci-295cm.html	4	39.78
Technical stainless steel fabric	2/0.56/1000 mm	Euro Sitex, s. r. o.	https://eshop.eurositex.cz/produkt/281/technicka-tkanina-nerezova-2-0-56-1000-mm/	1	51.48
Technical stainless steel fabric	0.05/0.035/1000 mm	Euro Sitex, s. r. o.	https://eshop.eurositex.cz/produkt/257/technicka-tkanina-nerezova-0-05-0-035-1000-mm/	1	73.08
Fan	PF40281B1-000U-A99	SUNON	https://www.gme.cz/v/1500620/sunon-pf40281b1-000u-a99-dc-ventilator	8	132.07
Hose couplings	R034351	P-LAB	https://www.p-lab.cz/spojka-hadicova-system-	15	96.19

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			keck?v=R035451_V_7406		
Hose couplings	R034351	P-LAB	https://www.p-lab.cz/spojka-hadicova-system-keck?v=R035451_V_7406	10	84.38
3D printing material	PETG	Prusa Research a. s.	https://www.prusa3d.com/cs/produkt/prusament-petg-jet-black-2kg/	4kg	95.40
Control unit + accesories	Arduino	Arduino	https://store.arduino.cc/?gad_source=1&gclid=CjwKCAjwkJm0BhBxEiwAwT1AXIKf44cTbvuNm3HGYdzOgppb_OPpGEhaKcywffRo7OP_m2G709MI9RoCE-EQAvD_BwE	-	79.56
Rubber hose insulation	KAIFLEX EF	HORNBACH BAUMARKT CS, s r. o.	https://www.hornbach.cz/p/potrubni-izolace-kaiflex-ef-tube-ef-o-22-mm-sirka-vrstvy-13-mm-delka-1-m/5852909/	8	11.78
Other components	Fittings; hose holders, reducers; bolts and nuts	-	-	-	238.69
				Total:	3,007.88

Author contribution

JH, OG created the concept; JK, JH and OG set the methodology; JH, OG wrote the software; JK carried the investigation; JK, KF, VS, MS and LV performed the validation; JK created visualizations; JK and KF wrote the manuscript draft; all authors reviewed and edited the manuscript; LV was supervising the work.

Competing interests

At least one of the (co-)authors is a member of the editorial board of Hydrology and Earth System Sciences.

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