Cryogenic vacuum distillation (CVD) methods have been widely used to extract water from unsaturated soil materials. However, recent studies have reported that extraction conditions (e.g., extraction time) and soil type can influence the stable isotope ratios of soil water extracted by CVD. We examined (i) the effects of extraction time on the stable isotope ratios of soil water extracted by CVD, and (ii) how the stable isotope ratios differed from those of soil water extracted by other methods. We first examined extraction times of 1–6 h, and observed no significant differences in the δH or δ18O values of extracted soil water for extraction times >2 h. However, extraction for 1 h collected only 48% of soil water, and the δH and δ18O values were significantly lower than those for extraction times >2 h. We then compared the stable isotope ratios of soil water extracted by CVD and centrifugation. Although the stable isotope ratios for both extraction methods were within the range of that of rainwater, the stable isotope ratios for water extracted by CVD were lower than those for water extracted by centrifugation. Our results highlight questions surrounding the use of CVD for stable isotope analysis of soil water.

KEYWORDS centrifugation; cryogenic vacuum distillation; isotope fractionation; soil water extraction; stable isotopes

INTRODUCTION

Cryogenic vacuum distillation (CVD) is the most commonly used laboratory-based technique for water extraction from unsaturated soil and plant xylem (Orlowski et al., 2018). In this method, soil or plant materials are heated within a tube under a defined vacuum, and the resulting vapor is frozen in a cryogenic trap. The advantage of this method is that sufficient amount of water may be collected for stable isotope analysis from small samples (< 10 g) (Orlowski et al., 2018). CVD procedures are dependent on laboratories (Table 1 of Orlowski et al., 2018), and a standard cryogenic vacuum distillation procedure has yet to be established (Orlowski et al., 2016a).

Recent studies have shown that extraction conditions (extraction time, temperature, and pressure) and soil type considerably affect the stable isotope ratios of water extracted by CVD (Orlowski et al., 2013; Meißner et al., 2014; Newberry et al., 2017a), suggesting that stable isotope data may contain errors associated with CVD. One factor influencing the stable isotope ratios of water extracted by CVD is extraction time (West et al., 2006; Jia et al., 2012; Orlowski et al., 2016a). In CVD, incomplete extraction leads to isotopic fractionation of water between liquid water and evaporating vapor due to Rayleigh distillation (Orlowski et al., 2013). Because this method is time-consuming and labor-intensive, recent improvements in CVD have optimized extraction times for soil and plants, allowing high sample throughput (West et al., 2006; Koeniger et al., 2011; Jia et al., 2012). Previous studies have examined the minimum extraction times required to obtain unfractionated soil water samples in their extraction systems, e.g., 30 and 40 min for sandy and clay soils, respectively (West et al., 2006), 15 min for sandy soil (Koeniger et al., 2011), and 35 and 40–45 min for sandy and loam soils, respectively (Jia et al., 2012). However, extraction time depends strongly on soil type and water content (Orlowski et al., 2016a). For example, Geris et al. (2015) set extraction times to >120 min due to relatively high water content soil (Histosols and Podzols) in their extraction system. Several studies have also set extraction times exceeding these minimum extraction times, e.g., >420 min (Vargas et al., 2017), 360 min (Mora and Jahren, 2003), and 120 min (Newberry et al., 2017b) (see also Table I). These studies suggested that extraction time should be specified for target soil types in each extraction system.

Another critical issue for the CVD method is that the stable isotope ratios differ between soil water extracted by CVD and that extracted by other lab-based methods such as centrifugation (Orlowski et al., 2016b) and microwave extraction (Munksgaard et al., 2014). Orlowski et al. (2016b) demonstrated that the centrifugation method showed the highest proficiency for obtaining δH and δ18O values of labelled water, whereas δH and δ18O values...
declined during CVD and microwave extractions. These studies suggested that water extraction affects isotope results. However, although there is more extensive application of CVD in hydrological studies compared to microwave extraction, a comprehensive inter-comparison of soil water extraction methods and their impacts on the resulting stable isotope ratios has not yet been conducted (McDonnell, 2014; Sprenger et al., 2015).

In this study, we aimed to elucidate (i) the effects of extraction time on the δ2H and δ18O values of soil water and (ii) how the δ2H and δ18O values differed between soil water extracted by CVD and centrifugation, as a commonly used lab-based extraction method. We first determined the extraction time of our extraction system to obtain unfractonated δ2H and δ18O values for the target soil type. We then evaluated differences in δ2H and δ18O values among soil water obtained by the two extraction methods, focusing on water collected at a research site and rainwater (i.e. the local meteoric water line).

**METHODS**

**Sample preparation**

Soil samples were collected in the Kiryu Experimental Watershed (KEW) in Shiga Prefecture, Japan. The watershed is underlain by granitic bedrock. The soil is classified as brown forest soil and the particle size distribution of the surface soil is approximately 30, >50, and 10% for particles >2.0, 0.1–2.0, and <0.1 mm in diameter, respectively (Ohte and Suzuki, 1990), which is indicative of sandy soil.

Soil samples were collected at a depth of approximately 15 cm on September 26th, 2014, as wet soil (gravimetric soil water content: 42%) and January 29th, 2015, as dry soil (gravimetric soil water content: 24%) at the KEW and transferred to the laboratory. Soil samples were preserved in 30-mL glass vials.

To investigate variations in the δ2H and δ18O values of rainfall and soil water, we collected rainfall and soil water at the KEW. Rainfall was collected in a bottle with a funnel in an open field at the KEW, and sampled once per month in 2014. These sampling dates were different from the sampling dates for wet and dry soil. To prevent isotope fractionation from evaporation, an oil film was poured into the rainfall collector. Soil water was collected at depths of 10 and 20 cm once per month in 2014 using tension lysimeters, which were vacuumed at a constant pressure of approximately –13 kPa using a vacuum pump (Hobara et al., 2001; Kawasaki et al., 2002). A sampling point for soil water using tension lysimeters was ~30 m apart from a sampling point for wet and dry soil.

**Soil water extraction by cryogenic vacuum distillation**

Soil water was extracted using a CVD system installed at the Laboratory of Forest Hydrology, Kyoto University. The vacuum was generated with an oil-sealed rotary vacuum pump (GLD-136C; ULVAC, Inc., Chigasaki, Japan) and monitored using a Pirani gauge (GP-2ARY and GP-1G;...
ULVAC, Inc.). The entire system was decompressed to a pressure of 10–15 Pa. After decompressing the lines including frozen soil samples, the samples were isolated from the vacuum pump by closing valves and then extraction procedures started. During the entire extraction procedure, soil samples were heated to 70°C until water sublimation. The evolved vapor was frozen in a cryogenic (liquid nitrogen) trap (Ingraham and Shadel, 1992). After defrosting the trapped water, its stable isotope ratios were analyzed.

**Experiment 1. Effects of extraction time on stable isotope ratios**

We extracted water from wet soil samples (45.3 ± 1.7 g soil by dry weight; \( n = 4 \)) at extraction times of 1–6 h using the CVD method. To evaluate whether soil water was completely extracted, we gravimetrically analyzed soil water content after extraction. Soil samples were oven-dried at 105°C for 24 h after extraction, and the extraction rate was calculated as extracted water by CVD divided by the total amount of water in the soil sample.

**Experiment 2. Effects of extraction method on the stable isotope ratios**

We first extracted soil water from wet soil samples (\( n = 4 \)) and dry soil samples (\( n = 3 \)) using a centrifuge (Model-7780; Kubota Corporation, Tokyo, Japan) with a rotor for soil (100 mL × 4; RH-1004, Kubota Corporation). Centrifugation was conducted at 8,400 rpm for 1 h. This condition is equivalent to the permanent wilting point of \( p_F = 4.2 \), which is approximately equal to 1.5 MPa (Jones, 2014). The soil water extracted by centrifugation could be considered as plant-available water. After centrifugation, half of the soil sample (50 mL) was preserved in a 50-mL glass vial. We then extracted the remaining water (\( p_F > 4.2 \)) by CVD. We also conducted CVD for wet soil samples from the start without centrifugation. This extraction was conducted over 6 h.

**Isotope analysis**

A cavity ring-down spectrometer (L2120-i; Picarro, CA, USA) was used to analyze the isotopic composition of soil water samples. We determined \( \delta^2\text{H} \) and \( \delta^{18}\text{O} \) values with respect to the Vienna Standard Mean Ocean Water. The precision levels of laboratory quality control standards for the \( \delta^2\text{H} \) and \( \delta^{18}\text{O} \) values in water were 0.11‰ and 0.06‰ or less, respectively (Katsuyama, 2014). We calculated the \( \delta^2\text{H} \) and \( \delta^{18}\text{O} \) of total soil water extracted using both centrifugation and CVD (\( p_F > 4.2 \)) in Experiment 2 based on the mass balance of the two soil waters. To evaluate the evaporative effects on stable isotope ratios of extracted soil water, we determined the deuterium excess value \( d \) (Dansgaard, 1964) as follows:

\[
d = \delta^2\text{H} - 8\delta^{18}\text{O}.
\]  

**Statistical analyses**

We conducted analysis of variance with Tukey-Kramer tests for both Experiments 1 and 2. Differences were considered statistically significant at \( p < 0.05 \). All statistical analyses were performed with the R v. 3.4.0 software (R Core Team, 2017).

**RESULTS**

**Differences in the stable isotope ratios among extraction times**

The extraction rate for 1-h extraction was only 50% (Figure 1a). Following 1-h extraction, the extraction rate was >98.8%. The pressure within the system stopped increasing after 2–3 h, and then began to decline at 3 h (Figure 1b). Slight increases in pressure were observed from 4 to 6 h. Both the \( \delta^2\text{H} \) and \( \delta^{18}\text{O} \) values of water extracted for 1 h were much lower than those extracted for 2–6 h (Figure 1c, d). These differences were up to −59.3‰ for \( \delta^2\text{H} \) and −11.1‰ for \( \delta^{18}\text{O} \). The differences in both \( \delta^2\text{H} \) and \( \delta^{18}\text{O} \) values among extraction times of 2–6 h were not significant (\( p > 0.05 \)). We also found significant difference in \( d \) between

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Figure 1. (a) Extraction rate, (b) pressure within the extraction system, (c) \( \delta^2\text{H} \), (d) \( \delta^{18}\text{O} \), and (e) \( d \)-excess of soil water extracted using cryogenic vacuum distillation at extraction times of 1–6 h. The error bars in (b) show the standard deviation.
1 h and 2–6 h (p < 0.05; Figure 1e). At extraction times of 4–6 h, δ¹⁸O increased slightly and d decreased slightly.

**Differences in the stable isotope ratios between extraction methods**

The δ²H and δ¹⁸O values of soil water collected at the KEW using tension lysimeters were within the range of the local meteoric water line, and those of soil water extracted by centrifugation and CVD were also within the range of soil water collected at the KEW, as shown by the dual isotope plot (Figure 2). The differences in δ²H and δ¹⁸O derived from the centrifugation and CVD methods for dry soils appeared to be larger than those for wet soils. Differences in δ²H and δ¹⁸O values between tension lysimeters and centrifugation/CVD could be partly explained by the differences in sampling points and dates.

The soil water content for pF > 4.2 (extracted by CVD) was lower than that for pF < 4.2 (extracted by centrifugation) in both wet and dry soils (Figure 3). The proportion of pF > 4.2 soil water content to total water content was larger in dry soils (39–47%) than in wet soils (32–37%). In wet soils, the δ²H values of water extracted by CVD (pF > 4.2 and total water extraction) were lower than those of centrifugation-extracted water (p < 0.05; Figure 3a). The differences in the δ¹⁸O and d values for soil water between the two methods were not significant (p > 0.05).

The soil water content for pF > 4.2 (extracted by CVD) were lower than those of water extracted by centrifugation (p < 0.05; Figure 3b). The differences in the δ¹⁸O and d values of soil water between the two methods were not significant (p > 0.05). The differences in δ²H and δ¹⁸O values between the two methods were larger for dry soils (–17.5 and –1.8‰) than for wet soils (–12.0 and –0.2‰).

**DISCUSSION**

**Effects of extraction time on the stable isotope ratios**

At extraction times of >2 h, the extraction rate was >98.8% and unfractionated δ²H and δ¹⁸O values were observed (Figure 1). For sandy soil, unfractionated water can be obtained by collecting more than 98% of total water (Araguás-Araguás et al., 1995). In contrast, 1-h extraction collected only 48% of total soil water, and both the δ²H and δ¹⁸O values for 1-h extraction were significantly lower than those for >2-h extraction (Figure 1). Although previous studies have reported minimum extraction times of approximately 1 h (Table I), 1-h extraction resulted in reduced δ²H and δ¹⁸O values in the sandy soil samples used in this study.

We observed slight increases in pressure and δ¹⁸O and slight decreases in d at extraction times of 4–6 h (Figure 1b,
d, e), indicating that a small amount of sublimated heavy water was trapped during the last extraction. However, the isotopic effect of the heavy water on total water can be relatively small due to its small amount.

Effects of extraction method on the stable isotope ratios

Although both the δH and δ18O values of water extracted by CVD and centrifugation were within the range of the local meteoric water line and soil water collected using lysimeters (Figure 2), these values were lower for water extracted by CVD than for that extracted by centrifugation in both wet and dry soils (Figure 2, 3), especially δH (Figure 3). This decline in δH and δ18O may have been caused by the presence of organic matter in soil (Iizuka et al., 2004; Orlowski et al., 2016a). Orlowski et al. (2016a) reported a decline in δH with increasing organic carbon content when using the cryogenic vacuum distillation method. Different exchangeable hydrogen fractions exist in environmental organic matter (i.e. oxygen-bonded, nitrogen-bonded, and sulfur-bonded or aromatic hydrogen); these can easily interact with ambient water (Ruppenthal et al., 2010). Exchangeable hydrogen fractions are dependent on the concentrations of organic matter in soil samples (Ruppenthal et al., 2010). For example, 30% of the hydrogen atoms in cellulose are exchangeable when bonded to oxygen (Filot et al., 2006). Thus, the organic carbon content of soil samples may affect the isotopic composition of extracted water. In this study, the δH value of water extracted by CVD (pF > 4.2) in wet soil was not significantly different from that for CVD from the start (Figure 3a), indicating that the δH value of extracted water by CVD was not dependent on pF values. This may be because exchangeable hydrogen fractions of soil organic matter would equilibrate isotopically with soil water.

Another possible factor affecting the isotopic composition of extracted water is clay minerals of soil samples (Meißner et al., 2014; Gaj et al., 2017a, b; Orlowski et al., 2016a). Meißner et al. (2014) and Orlowski et al. (2016a) reported decreases in δH and δ18O of soil water extracted by CVD with increasing clay minerals. This phenomenon may be caused by the removal of clay interlayer water (Munksgaard et al., 2014; Gaj et al., 2017a, b), which is bound to the mineral surface. All water including clay interlayer water cannot be released from mineral soils at temperatures lower than approximately 205°C (Gaj et al., 2017a), which is much higher than the extraction temperatures of CVD in previous studies (Table 1). Thus, the δH and δ18O values of water extracted by CVD from soils with high clay minerals may decrease. In this study, the isotopic effects of clay minerals on the isotopic composition of extracted water may be small because we used sandy soils for two experiments, i.e. small clay minerals in the soil materials (see Methods). Previous studies observed more pronounced decreases in the δ18O value of water extracted by CVD from soils with high clay minerals (Orlowski et al., 2016a; Newberry et al., 2017a). However, we observed relatively small decreases in the δ18O value of water extracted by CVD (Figure 3). The δH value without dependence on a pF level (Figure 3) also suggests that the isotopic effects of clay minerals on the δH value of extracted water would be small in this study. If the depletion of the δH value for CVD was caused by the release of clay interlayer water, it should depend on a pF level because the relative fraction of clay minerals would be higher in CVD (pF > 4.2) than CVD from the start. Our results suggest that organic matter could be the main cause of decreases in the δH values of water extracted by CVD. Further research is needed to examine the effects of clay minerals (i.e. interlayer water) and soil organic matter (i.e. exchangeable bonded hydrogen) on isotopic compositions of soil water extracted by CVD. We recommend further experiments including water extraction from soil materials with various clay contents and detecting the type and amounts of soil organic matter in soil materials.

CONCLUSION

We observed no significant differences in the δH and δ18O values of extracted soil water for extraction times >2 h, whereas the δH and δ18O values for 1-h extraction were significantly lower than those for >2-h extraction. The δH and δ18O values for soil water extracted by CVD were smaller than those for soil water extracted by centrifugation, even though CVD was conducted for >2 h. CVD could cause an extraction method-induced bias in the δH and δ18O values of extracted soil water.

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