








RESEARCH ARTICLE

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Characterization and Correction of Evaporative Artifacts in Speleothem Fluid Inclusion Isotope Analyses as Applied to a Stalagmite From Borneo

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Key Points:

- Fluid inclusions water isotopes in speleothems do not always reflect the composition of precipitation
- Evaporation during analyses before water extraction affects the accuracy of measurements
- Artifacts can be corrected to produce reliable paleoclimate reconstructions

Supporting Information:

Supporting Information may be found in the online version of this article.

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Abstract Fluid inclusion water isotope measurements in speleothems have great potential for paleoclimate studies as they enable the reconstruction of precipitation dynamics and land temperatures. Several previous observations, however, suggest that inclusion waters do not always reflect the isotopic composition of surface precipitation. In such cases, dripwaters are thought to be modified by evaporation in the cave environment that results in more positive $\delta^2\text{H}$ and $\delta^{18}\text{O}$ values and shallow $\delta^2\text{H}/\delta^{18}\text{O}$ slopes. Although evaporation can occur in cave systems, water can also be lost to evaporation during analysis but before water extraction. Here, we examine the likelihood of this possibility with a stalagmite from Borneo. We demonstrate that many samples lose water, and that water loss is controlled by the type and size of inclusions. With multiple replicate measurements of coeval samples, we calculate an evaporative $\delta^2\text{H}/\delta^{18}\text{O}$ slope of 1.0 ± 0.6 (2SE). This value is consistent with model predictions of evaporative fractionation at high analytical temperature and low humidity. Finally, we propose a method to correct for this effect. We find that fluid–calcite $\delta^{18}\text{O}$ paleotemperatures calculated with corrected $\delta^{18}\text{O}$ data show excellent agreement with recent microthermometry temperature estimates for Borneo, supporting the validity of our approach and implying limited stalagmite $\delta^{18}\text{O}$ disequilibrium variations. Corrected fluid inclusion $\delta^{18}\text{O}$ and $\delta^2\text{H}$ values follow the expected hydroclimate response of Borneo to periods of reduced Atlantic Ocean meridional overturning circulation. Our results suggest that careful petrographic examination and multiple replicate measurements are necessary for reliable paleoclimate reconstructions with speleothem fluid inclusion water isotopes.

1. Introduction

The majority of stalagmite-derived paleoclimate reconstructions have traditionally been based on the oxygen isotope composition of carbonate (e.g., Fleitmann et al., 2003; Wang et al., 2001). More recently, thanks to recent technological improvements (e.g., Affolter et al., 2014; Dassié et al., 2018; de Graaf et al., 2020), research has increasingly focused on measurements of fluid inclusion water isotopes. Fluid inclusions in speleothems preserve a record of ancient dripwater. Measurements of $\delta^2\text{H}$ and $\delta^{18}\text{O}$ in fluid inclusion water offer unique advantages relative to carbonate $\delta^{18}\text{O}$ values alone. They provide a more direct constraint on rainwater $\delta^{18}\text{O}$ than carbonate $\delta^{18}\text{O}$. Thus, they can be used to reconstruct changes in precipitation dynamics such as rainfall amounts (e.g., Arienzo et al., 2015; Millo, 2017; van Breukelen et al., 2008) and in the sources of moisture (e.g., Matthews et al., 2021; Rogerson et al., 2019), especially when supported by suitable moisture transport information (e.g., Baker et al., 2015). They also allow disentangling the respective contributions of temperature and dripwater $\delta^{18}\text{O}$ variability on stalagmite carbonate $\delta^{18}\text{O}$ (e.g., Arienzo et al., 2015; Meckler et al., 2015, 2021; Wortham et al., 2022), yielding temperature estimates from the difference of $\delta^{18}\text{O}$ in inclusion water and the surrounding calcite. Additionally, in settings where temperature is the main control on rainfall isotope variability, fluid inclusion isotope measurements have also been used directly to estimate air temperature at the site of precipitation, assuming that present-day relationships hold true for the past (Affolter et al., 2019).

Fluid inclusions often yield meaningful isotope values, showing that the isotopic composition of the inclusion water is well preserved. Fluid inclusion isotope analyses thus contribute significantly to our understanding of speleothem paleoclimate records. However, observations from some stalagmites suggest that recovery of the original isotope signal of precipitation is not always possible. For instance, recently published fluid inclusion

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isotope data from five stalagmites show cases of large positive enrichments in $\delta^2\text{H}$ and $\delta^{18}\text{O}$ values that can plot far to the right of local meteoric water lines (LMWLs) (Affolter et al., 2019; Matthews et al., 2021; Nehme et al., 2020; Warken et al., 2022; Wortham et al., 2022). These data are usually obtained from samples with relatively low water yields, and follow typical evaporation lines in a $\delta^2\text{H}/\delta^{18}\text{O}$ diagram. Such observations suggest that fluid inclusion waters can be modified to such an extent that they no longer reflect the isotopic composition of surface precipitation. Modification of fluid inclusion water $\delta^{18}\text{O}$ through carbonate-water isotope exchange has been proposed (Demény et al., 2016) but fails to explain the enrichment observed in both $\delta^2\text{H}$ and $\delta^{18}\text{O}$ values. Additionally, such O isotope exchange has been shown not to readily occur in speleothems (Uemura et al., 2020).

Another common hypothesis to explain the mismatch between LMWLs and the fluid inclusion isotopes is that modifications are the result of evaporative fractionation in the soil, epikarst, or cave environment. Evaporation results in heavier oxygen and hydrogen isotope compositions relative to the initial liquid and in shallow $\delta^2\text{H}/\delta^{18}\text{O}$ slopes that intersect the steeper LMWLs (e.g., Craig H, 1961; Horita et al., 2008). While this hypothesis appears at first sight to explain the main observations outlined above, there are other possible explanations. Several studies have raised the possibility that evaporation could also occur prior to analysis when samples are placed in an analytical apparatus at high temperatures (typically 120°C; Matthews et al., 2021; Warken et al., 2022). However, additional data from experiments and measurements of natural samples are needed to determine which of the hypotheses explain the observations.

Here, we test the reliability of fluid inclusion isotope analyses with a stalagmite from Borneo that covers the last 26,000 years, including the last deglaciation. We specifically examine the extent to which fluid inclusion waters can evaporate during laboratory analyses before the crushing step that releases water into the analyzer. Borneo is the ideal location for this work for several reasons. Evaporation in Borneo caves is unlikely given the year-round 100% relative humidity of cave air at this tropical location (Meckler et al., 2015). If isotopic enrichments occur in fluid inclusion water isotopes, they are most likely the result of another process. Furthermore, the temperature and hydroclimate evolution of Borneo through the last deglaciation are relatively well understood. Changes in hydroclimate are constrained with carbonate $\delta^{18}\text{O}$ measurements, which reproduce well across four different stalagmites (Buckingham et al., 2022; Partin et al., 2007), suggesting minimal variations over time in isotopic disequilibrium during stalagmite formation. The interpretation of carbonate $\delta^{18}\text{O}$ is corroborated by calculated drip-water $\delta^{18}\text{O}$ values (Løland et al., 2022), and is consistent with isotope-enabled transient climate simulations (Buckingham et al., 2022; He et al., 2021). The regional temperature evolution is well-constrained with fluid inclusion microthermometry (Løland et al., 2022), a method that produces exceptionally precise paleotemperatures (Krüger et al., 2011). These available constraints allow us to compare fluid inclusion water isotope data to independently derived dripwater $\delta^{18}\text{O}$ and calculated fluid-calcite $\delta^{18}\text{O}$ temperatures with available microthermometry estimates.

2. Methods

2.1. Site and Sample Description

We measured water isotopes in fluid inclusions (also referred to as fluid inclusion isotopes here) from stalagmite SSC01 collected in Northern Borneo (Partin et al., 2007). Borneo is located in the tropical West Pacific, the region with the warmest modern ocean temperatures. The site remains year-round within the center of tropical convection as the Intertropical Convergence Zone (ITCZ) migrates north and south. The region receives an average of 5,000 mm annual rainfall with only weak seasonality. The stalagmite SSC01 was collected in 2003 from Snail Shell Cave (4°12'20.8"N, 114°56'26.9"E; see Figure S1 in Supporting Information S1 for a scan of the sample), which is located in Gunung Buda in the northwestern corner of Malaysian Borneo and has previously been dated and analyzed for carbonate $\delta^{18}\text{O}$ (Partin et al., 2007).

We compare our data to calculated dripwater isotope values and fluid inclusion microthermometry estimates from another stalagmite from an adjacent mountain, SC02 (Løland et al., 2022). Stalagmite SC02 was collected in 2006 from Secret Chamber in the Clearwater Cave system of Gunung Mulu National Park (Buckingham et al., 2022). Dripwater isotope values in Gunung Mulu caves have been shown to closely match rainfall $\delta^2\text{H}$ and $\delta^{18}\text{O}$ values with an average residence time in the karst of 3–18 months, and regional rainfall amount is thought to exert the dominant control on interannual rainfall isotope variability (Ellis et al., 2020; Moerman et al., 2014). The position of the LMWL is well constrained with quasi biweekly dripwater samples collected from 2006 to

2018 at three sites in two caves (see Text S2 in Supporting Information S1 for details). A linear regression drawn through these data results in the following equation: $\delta^2\text{H} = 8.06 (\pm 0.04) \times \delta^{18}\text{O} + 13.74 (\pm 0.3)$. Long multi-year measurements of cave temperatures exist for several caves in Gunung Mulu. These measurements show that cave temperatures remain close to 24°C with less than 0.2°C variations throughout the year and that temperatures are similar to the average annual temperature measured at the nearby Mulu airport (annual average: 24.3°C; seasonal range: 21.5–27.6°C) (Løland et al., 2022). Since long-term temperature measurements do not exist for Snail Shell Cave, we assume that the nearby Mulu temperature range is also representative for that site.

The SSC01 stalagmite was originally split in half along the central growth axis by Partin et al. (2007), who used one half for geochemical analyses, while the other half is used here. The stalagmite is approximately 44 cm in length with an average growth rate of 0.02 ± 0.002 mm/year. The stalagmite half was embedded in plaster, and a diamond wire saw (Well 6500) was used to cut slabs of 7 mm thickness lengthwise. Blocks of 20–40 mm length and 20 mm width were then cut from the slabs along the central axis. Each block incorporates approximately 1000–2000 years of growth. Before the blocks were sampled for fluid inclusion isotope analyses, thin sections were obtained for petrographic inspection. Samples for fluid inclusion analysis were then cut from the blocks with a smaller diamond wire saw (Well 3421). To this end, the calcite blocks were first glued to glass plates on both sides and fixed on the sample holder of the saw. The sample holder was attached to a translation stage, which enables stepwise lateral displacement of the sample with respect to the cutting direction resulting in a curvature of the cuts. The samples were cut along the growth bands closely following their three-dimensional orientation. This ensures that coeval samples are obtained. Two to five samples were obtained from each block and placed in acetone to remove the glued glass plates. These samples were then cut into three to four equally sized replicate samples of approximately 0.2–0.8 g.

2.2. Calcite Stable Isotope Measurements and Age Model

To place the results on the revised chronology of Partin (2008), calcite $\delta^{18}\text{O}$ was re-measured on the sample blocks and the results were compared to the published record (Figure S3 in Supporting Information S1). Samples were obtained by continuous milling at 1 mm increments with a Sherline 540 micromill. Measurements were performed in the Facility for Advanced Isotopic Research (FARLAB) at the Department of Earth Science at the University of Bergen using a MAT 253 isotope ratio mass spectrometer (IRMS-Thermo Fisher) coupled to a Kiel IV carbonate preparation device. A total of 347 calcite samples were measured. Measurements were normalized to the VPDB scale with in-house calcite standards, which were previously calibrated with NBS-19 and NBS-18 measurements. The average measurement error is less than 0.05‰ (1σ) for $\delta^{13}\text{C}$ and 0.10‰ (1σ) for $\delta^{18}\text{O}$.

2.3. Fluid Inclusion Isotope Measurements

Fluid inclusion water isotope measurements were conducted following the procedures described by de Graaf et al. (2020). The analytical line consists of a crusher device that is connected to a humidity generator and to a Picarro cavity ring-down spectrometer on opposite ends. The crusher device is constructed in stainless steel and follows the design of de Graaf et al. (2020) with some modifications. Samples were measured with all analytical components under constant humid conditions to avoid memory effects (Affolter et al., 2014; Dassié et al., 2018; de Graaf et al., 2020). The humidity generator is composed of a modified 300 ml stainless-steel cylinder that serves as an evaporation chamber and a microdrop dispenser head (MK-130, Microdrop GmbH, Germany) that ejects water droplets (<100 μm diameter) into the evaporation chamber by piezoelectric stimulation. Specific details on the design and performance of the humidity generator device are described in Sodemann et al. (2023). During analyses, the evaporation chamber of the humidity generator was maintained at a constant temperature of 60°C, a temperature high enough to allow for instant evaporation and within the tolerance of the microdrop dispenser. The crusher device and related stainless-steel lines were maintained at a temperature of 120°C. A distilled water standard of known isotopic composition (DI2; $\delta^{18}\text{O} = -7.63 \pm 0.06$ and $\delta^2\text{H} = -50.72 \pm 0.46$) was used to generate the humid background.

Depending on the expected water content, between 0.2 and 0.8 g of stalagmite sample were used for analyses, and whenever possible samples were measured a minimum of three times. A total of 119 replicate analyses from 40 samples were performed. Before analysis, samples were loaded into the pre-heated crusher device and approximately 15–25 min were needed for background stabilization. After stabilization, the sample was crushed, and the resulting water was measured by the analyzer. Raw $\delta^{18}\text{O}$ and $\delta^2\text{H}$ data for sample and background were corrected for the humidity dependency of the analyzer following the procedures described in Weng et al. (2020).

Sample $\delta^{18}\text{O}$ and $\delta^2\text{H}$ values were then calculated following the algorithm presented by Affolter et al. (2014). These were subsequently normalized to the VSMOW scale with a set of three in-house water standards, which had previously been calibrated against international standards. Standards were injected on a daily basis through a port that is installed immediately before the crusher device. The analytical precision (1σ) of replicate analyses for samples larger than 0.3 μL , as judged by injected calibration standards, are 0.3 and 1.3‰ for $\delta^{18}\text{O}$ and $\delta^2\text{H}$, respectively. For samples between 0.1 and 0.3 μL , precisions are 0.4 and 1.4‰ for $\delta^{18}\text{O}$ and $\delta^2\text{H}$. Final errors in fluid inclusion isotope data are presented as confidence intervals at 68% or 95%. These are calculated with the standard deviation of replicate analyses or the long-term reproducibility from water standards (reported above), whichever was greater.

The accuracy of water isotope measurements with the crushing line was assessed with standard waters sealed in borosilicate glass capillaries (following Weißbach, 2020), and with aliquots of a travertine sample that was previously measured in another laboratory (Semproniano travertine, de Graaf et al., 2020). The glass capillary samples were designed to mimic real inclusions since they receive the same treatment as calcite samples (i.e., water is released in the same location as calcite samples by turning the thread of the crusher device). The mean values obtained for both glass capillaries and the travertine are statistically indistinguishable from their “known” values. Stalagmite water amounts ($\mu\text{L/g}$) were calculated using a transfer function built with calculated water amounts (area under peaks) and water amounts from injections of standard waters (0.05–1 μL , $r^2 = 0.99$, RMSE = 0.02). The accuracy of water amount measurements was determined to be $\pm 5\%$ (1σ) based on weighted glass capillaries.

3. Results

3.1. Petrography—SSC01 Calcite Fabrics and Fluid Inclusions

Thick sections (200 μm) of stalagmite SSC01 were investigated using an optical microscope under plane polarized (PPL) and cross polarized light (XPL) to characterize the calcite fabrics as well as the occurrence and shapes of fluid inclusions. The calcite fabrics and occurrence of fluid inclusions can be classified into two general groups that are closely associated with the age of the sample. Holocene and late deglacial calcite fabrics are dominated by clear, transparent columnar calcite with straight crystal boundaries and up to centimeter-wide domains of uniform extinction under XPL (Figures 1a and 1b). The fabric is characterized by countless very small fluid inclusions that are dispersed in the matrix and range in length between 1 and 5 μm (Figure 1c). Based on microthermometry estimates, these lengths correspond to volumes of 1–10 μm^3 . Additionally, these samples contain some much larger up to 1 mm long inter-crystalline inclusions. Occasionally, the calcite fabric is interrupted by distinct layers with numerous inclusions that range in volumes between 10^2 and $10^3 \mu\text{m}^3$ (Figures 1a and 1b). These inclusion-rich layers possibly formed onto former growth hiatuses. In contrast, the older part of the stalagmite (early deglacial and glacial sections) consists of alternating layers of open columnar and microcrystalline calcite fabrics (Figures 1c and 1d). The latter are rich in organic matter, as confirmed by confocal fluorescence microscopy. Fluid inclusions are abundant in the columnar fabric and orders of magnitudes larger (10^3 and $10^5 \mu\text{m}^3$ or 25–100 μm in length) than the tiny inclusions that dominate the main part of the Holocene and late deglacial fabric of stalagmite SSC01 (Figure 1f). Large inter-crystalline inclusions are also present in the early deglacial and glacial parts of the stalagmite.

3.2. Relationship of Fluid Inclusion Isotope Data to the LMWL

A cross-plot of the stalagmite fluid inclusion water isotope data shows that the majority of samples (31 out of 40, i.e., 78%) have values that are outside 1σ (68% CI) analytical uncertainty of the LMWL (Figure 2a). Moreover, many data points are systematically shifted toward more positive $\delta^{18}\text{O}$ values with only small differences present in $\delta^2\text{H}$ values. This is most clearly seen in Figure 2b, where we present individual replicate analyses. We observe that replicate analyses do not fall randomly off the LMWL. They instead follow linear trends with shallow slopes that intersect the LMWL (Figure 3). To investigate the cause for the observed deviations from the LMWL, we calculated the slopes in $\delta^2\text{H}$ – $\delta^{18}\text{O}$ for samples with three or more replicate measurements, and where the standard deviation in $\delta^{18}\text{O}$ values was larger than 0.4‰ (i.e., the long-term analytical reproducibility; Figure 3a). This last step is necessary because many data points are within error of each other, and a larger spread in $\delta^{18}\text{O}$ values allows for a more accurate slope determination. Eleven samples met these criteria, and we obtained an average value of 1.0 ± 0.6 (2 SE) for the slopes among their replicate results (see supporting information 4 for details). Moreover, we observe a general relationship between $\delta^{18}\text{O}$ deviations from the LMWL ($\Delta\delta^{18}\text{O}$ LMWL) and the amount of water that was recovered during its analysis (i.e., less water was obtained from replicates that are

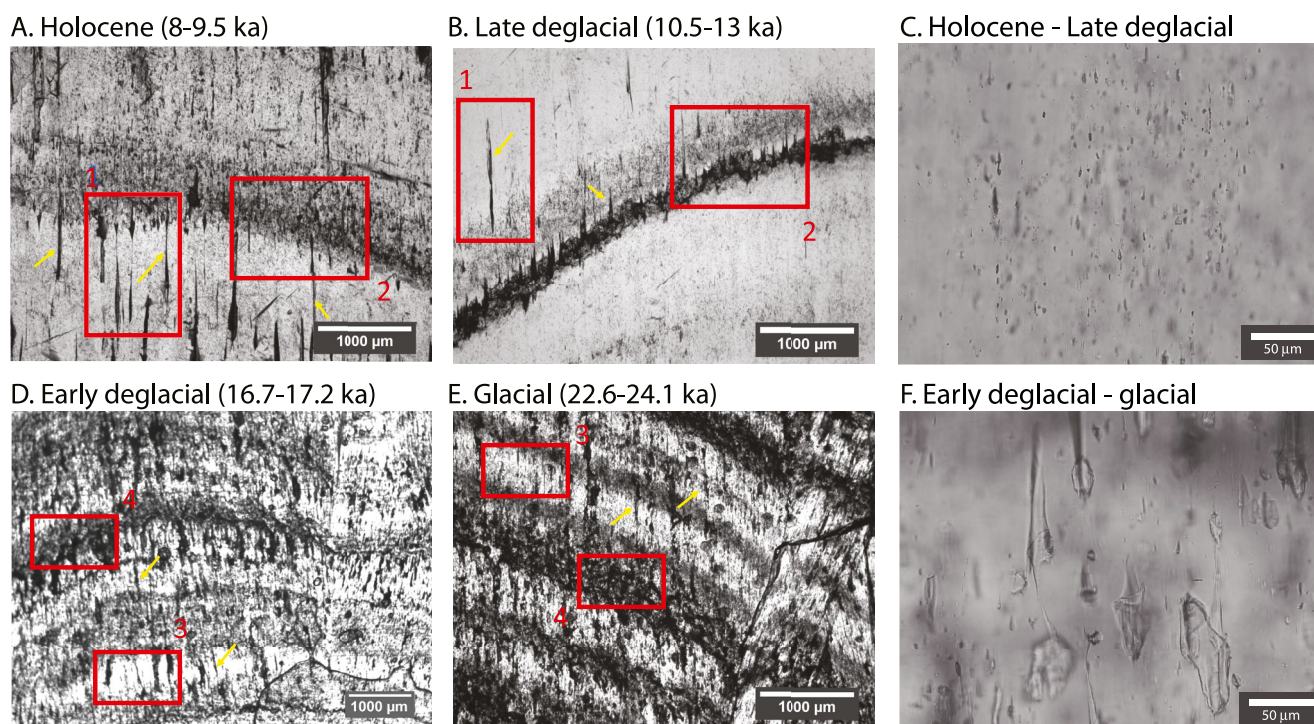


Figure 1. Optical microphotographs of polished thick sections from representative portions of the glacial, deglacial, and Holocene sections of SSC01. (a and b) Thin section images from Holocene and late deglacial sections taken under cross-polarized light. (c) Microphotograph at higher magnification showing the very small ($1\text{--}10\ \mu\text{m}^3$) inclusions present in the Holocene and late deglacial portions of the stalagmite. (d and e) Thin section images from the early deglacial and the glacial portions of the stalagmite taken under cross-polarized light. (f) Microphotograph at higher magnification showing the large ($10^3\text{--}10^5\ \mu\text{m}^3$) inclusions present in the early deglacial and glacial portions of the stalagmite. Yellow arrows indicate large elongated inter-crystalline fluid inclusions and red boxes highlight representative fabrics: (1) Columnar calcite with countless tiny fluid inclusions and some large inter-crystalline inclusions. (2) Dark inclusion-rich layers possibly overgrowing a former hiatus surface enriched in organic material. (3) Example of light, transparent open columnar layers with abundant large fluid inclusions. (4) Examples of dark-colored layers composed of organic-rich microcrystalline calcite.

further away from the LMWL). This is true for 9 out of the 11 samples that we considered, as can be seen by the negative slopes in water content ($\mu\text{L/g}$) versus $\Delta\delta^{18}\text{O}$ LMWL (‰) in Figure 3b.

3.3. Relationships Between Fluid Inclusions, Water Content, and $\delta^{18}\text{O}$ Deviations From the LMWL

There is a close association between water content, $\delta^{18}\text{O}$ deviations from the LMWL ($\Delta\delta^{18}\text{O}$ LMWL), and the occurrence of fluid inclusions in the stalagmite. Higher water contents are observed in samples from the early deglacial and glacial samples (Figure 4a); these samples have abundant large (i.e., 10^3 and $10^5\ \mu\text{m}^3$) fluid inclusions in the open columnar fabric. Much lower water amounts were observed in the Holocene and late deglacial samples (Figure 4a). These samples have abundant but small inclusions (i.e., 1 and $10\ \mu\text{m}^3$) and very few larger (i.e., up to $1\ \text{mm}$ long) inter-crystalline inclusions. The abrupt change in water content that we observe during the deglaciation is also mirrored in the proportions of samples and replicate analyses that fall off the LMWL (Figure 4b). Most of the early deglacial and last glacial maximum (LGM) samples fall close to the LMWL, that is, they have $\delta^{18}\text{O}$ values that are close to what is expected based on their $\delta^2\text{H}$ composition ($\Delta\delta^{18}\text{O}$ LMWL values are close to zero). The younger samples from the late deglacial and Holocene, however, have $\delta^{18}\text{O}$ values that, with few exceptions, fall off the LMWL and are farther away from the LMWL.

3.4. Comparison of Inclusion Isotope Data With Independent Temperature and Dripwater $\delta^{18}\text{O}$ Constraints

The time-dependence of observed offsets and their possible cause can be further investigated by comparing the fluid inclusion isotope results to available independent information on temperature (from fluid inclusion microthermometry; Løland et al., 2022) and dripwater $\delta^{18}\text{O}$ from stalagmite SC02 collected from a cave in an adjacent mountain (Figure 5). Dripwater $\delta^{18}\text{O}$ values for SC02 were calculated with microthermometry temperature estimates and the

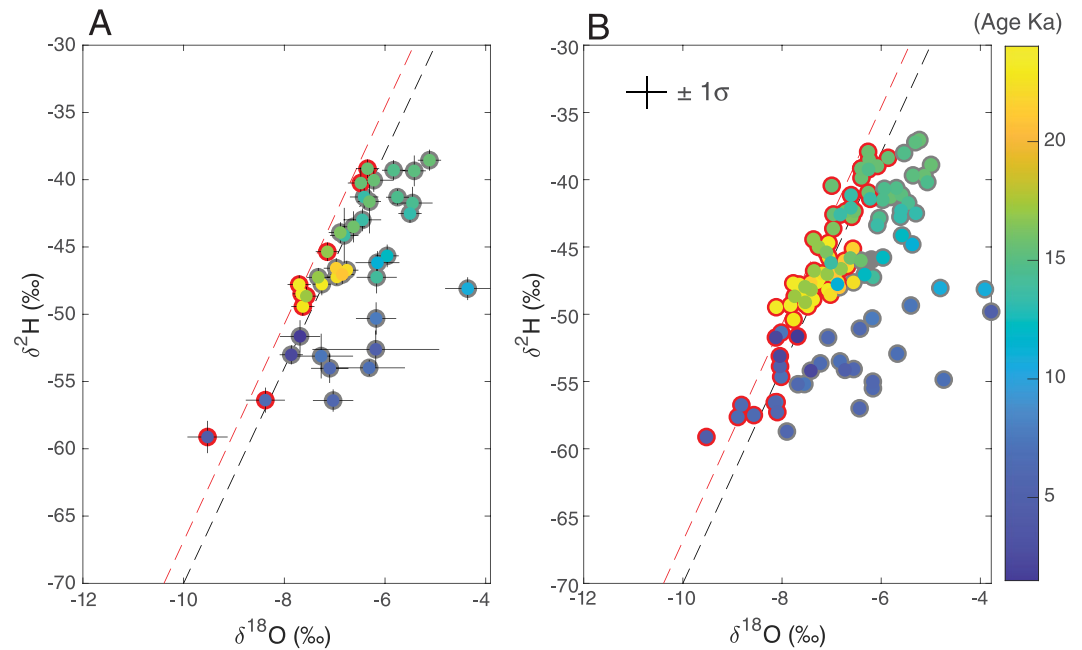


Figure 2. Fluid inclusion water isotope composition. (a) Crossplot of $\delta^2\text{H}$ versus $\delta^{18}\text{O}$ showing sample means. (b) Same as (a) but with replicate analyses. Data points with red outline are within $\pm 68\%$ (CI) of the local meteoric water line (LMWL). Data points are color coded by age as shown in the colorbar. The red dashed line is the LMWL and the black dashed line is the global meteoric water line. Error bars in (a) are 68% confidence intervals and the illustrative error bar in (b) is the long-term reproducibility. 1 ka in color bar corresponds to 1000 years.

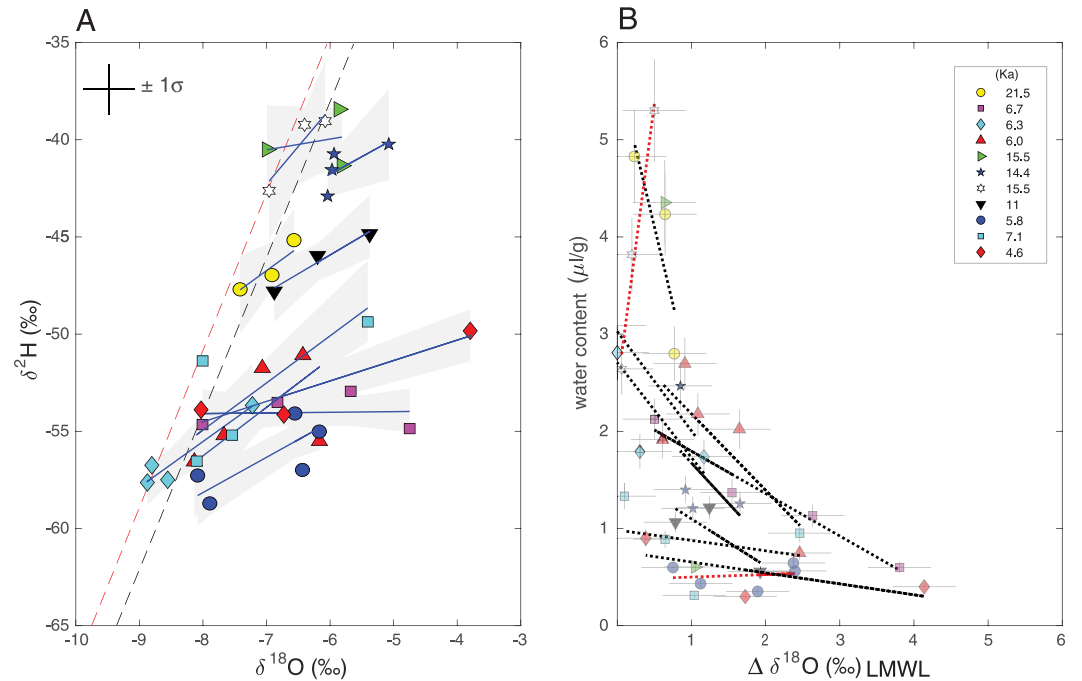


Figure 3. (a) Linear regressions through replicate water isotope measurements. Each color and symbol combination represents one sample (11 samples total). Only samples with three or more replicates and standard deviations in $\delta^{18}\text{O}$ greater than 0.4‰ are shown. Blue lines are individual linear fits calculated using the algorithm of York et al. (2004). Shaded gray regions are regression 68% confidence intervals obtained by Monte Carlo simulations considering errors in both $\delta^{18}\text{O}$ and $\delta^2\text{H}$ values. Black dashed line is the global meteoric water line and red dashed line is the local meteoric water line (LMWL). (b) Water content of individual replicate analyses versus $\Delta\delta^{18}\text{O}$ LMWL ($\delta^{18}\text{O}$ deviation from LMWL). Black dashed lines are linear regressions with negative slopes, and red dashed lines are linear regressions with positive slopes.

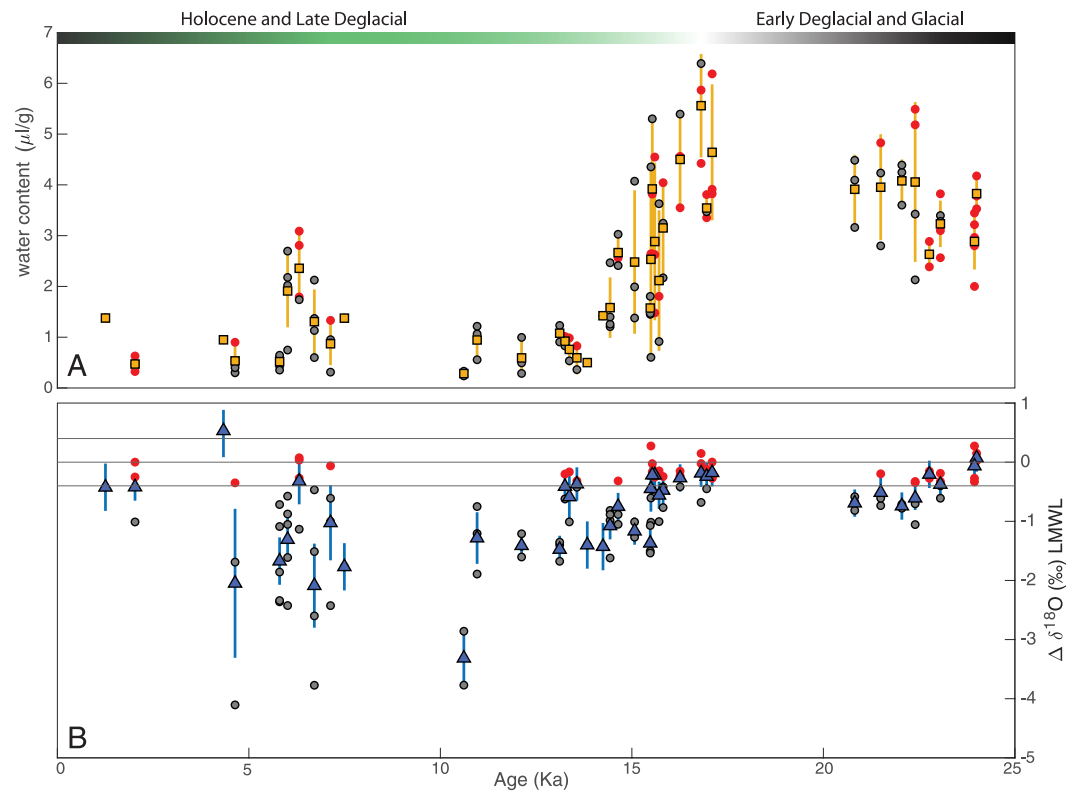


Figure 4. (a) Water content ($\mu\text{L/g}$) of SSC01 stalagmite plotted against age. Yellow squares are mean values of replicate analyses, and circles are individual analyses. Red circles are replicates with $\delta^{18}\text{O}$ and $\delta^2\text{H}$ values that fall within 68% CI of the local meteoric water line (LMWL). (b) $\delta^{18}\text{O}$ deviations from the LMWL ($\Delta\delta^{18}\text{O}$ LMWL). Circles are replicate analyses and blue triangles are sample means. Red data circles are replicates that have values that fall on the LMWL.

corresponding calcite $\delta^{18}\text{O}$ values using the empirical cave calibration for $\delta^{18}\text{O}$ of Tremaine et al. (2011) (see Løland et al., 2022). We observe that SSC01 fluid inclusion $\delta^{18}\text{O}$ values are generally more positive than calculated drip water isotope values from SC02. However, values are closer to those in SC02 in older parts of the SSC01 record than in younger parts. Moreover, we find closer agreement when only the SSC01 $\delta^{18}\text{O}$ values that fall on the LMWL are considered (Figure 5a). The agreement is even more apparent when only individual replicate analyses are considered; replicates that fall on the LMWL follow the same trends as in SC02 throughout the length of the record (Figure S5 in Supporting Information S1). In contrast to fluid inclusion $\delta^{18}\text{O}$, $\delta^2\text{H}$ values are much closer to the calculated SC02 drip water values, although still showing larger deviations in the younger part of the record (Figure 5b).

If we were to use our fluid inclusion water isotope results at face value, fluid-calcite $\delta^{18}\text{O}$ temperatures, calculated with the equation of Tremaine et al. (2011), yield mostly unrealistically high temperatures and are almost always warmer than microthermometry estimates (Figure 5c). A closer agreement is observed during the LGM and the early deglacial, where temperatures are on average 2.3°C warmer. In younger samples, on the other hand, temperatures are on average 7.3°C warmer than fluid inclusion microthermometry temperatures. In a similar manner to SSC01 fluid inclusion $\delta^{18}\text{O}$ values, we observe better agreement between both paleothermometers when only temperatures calculated with $\delta^{18}\text{O}$ values that fall on the LMWL are considered. This is true at the sample mean and the replicate level (Figure 5c and Figure S5 in Supporting Information S1). Overall, the comparison with independent constraints supports our interpretation that the offsets from the LMWL are likely artifacts and illustrates their impact on the interpretation of the results.

4. Discussion

4.1. Water Loss Through Evaporation During Analyses

We observe in the SSC01 stalagmite fluid inclusion $\delta^2\text{H}$ and $\delta^{18}\text{O}$ values that plot to the right of the LMWL, shallow $\delta^2\text{H}/\delta^{18}\text{O}$ slopes in replicate analyses, and poor agreement between fluid inclusion data and independent

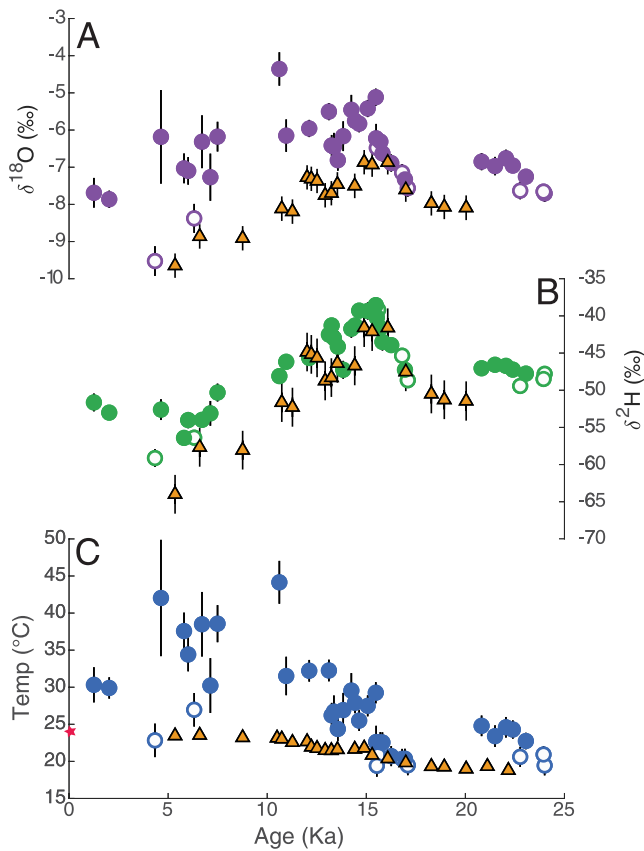


Figure 5. (a) Measured fluid inclusion $\delta^{18}\text{O}$ values of stalagmite SSC01 (circles) and calculated drip water $\delta^{18}\text{O}$ values of stalagmite SC02 from the same region (Løland et al., 2022). (b) Measured fluid inclusion $\delta^2\text{H}$ values of SSC01 (circles) and calculated drip water $\delta^2\text{H}$ values of SC02, using the local meteoric water line (LMWL) (triangles). (c) fluid-calcite $\delta^{18}\text{O}$ temperatures of SSC01 calculated with the temperature relationship of Tremaine et al. (2011) (circles) and fluid inclusion microthermometry temperatures of SC02 from Løland et al. (2022). Error bars are 68% confidence intervals. Open circles are samples that fall within 68% CI of the LMWL. The red star indicates the modern cave temperature in several caves of Northern Borneo (24°C).

paleoclimate proxies. These observations could be interpreted as an indication of evaporation in the cave. For the cave site investigated here, however, we expect that evaporation inside the cave is unlikely given year-round relative humidity in Borneo caves of nearly 100% (Meckler et al., 2015). For this reason, we now explore the alternative hypothesis that evaporation occurred in the crusher device at 120°C, immediately before the crushing step and analysis. The physical reasoning behind this hypothesis is the increase of fluid pressure with increasing temperature in an isochoric system. At a certain threshold the fluid overpressure inevitably induces irreversible volume changes of the inclusions, most likely due to the formation of cracks in the confining calcite host, preferentially along the cleavage planes and, in case of inter-crystalline inclusions, along crystal boundaries. The formation of cracks results in a volume increase of the inclusions, and thus, in a relaxation of the fluid overpressure. Upon further heating, the cracks propagate and waters can then leak along crystal boundaries, internal porosity and primary fractures in the stalagmite and evaporate. Partial evaporation of water from fluid inclusions would lead to isotopic fractionation, causing enrichments in $\delta^2\text{H}$ and $\delta^{18}\text{O}$ values and thus affecting the isotope signal that is measured after crushing the sample. Inclusions that lose all their water in the course of the 15–25 min of background stabilization, on the other hand, have no effect on the measured isotope signal.

Ideally, water loss can be observed as samples are heated and the evolving water is detected by the analyzer. However, this is not possible with our analytical set-up. When the crusher device is opened to load a sample, the water background measured by the analyzer quickly decreases to match the humidity of the laboratory air. Once the crusher is closed, the humidity slowly rises over the next 15–20 min to the level set by the humidity generator. This makes it impossible to observe any water loss from the sample that may be occurring during this time. For that reason, we designed an experiment to manometrically determine if water is lost as a sample is heated in a vacuum line (see supporting information 6 for details). Briefly, a sample was loaded into a glass tube, connected to a vacuum line and evacuated. The sample was then heated to approximately 120°C with a heat gun, and temperature was monitored with a hand-held thermocouple held in contact with the sample vial. Any released water was collected in a cold trap bathed in liquid N_2 . After 20 min, the liquid N_2 was removed, and the water pressure was measured in a manometer. The manometer pressure was calibrated for water amounts by adding known amounts of water to the same volume. The experiment was

performed with two different samples from the Holocene portion of the stalagmite; we estimate that for both samples approximately 10% of the water content was lost (see Text S6 in Supporting Information S1 for details).

4.2. Evaporation Model

We have demonstrated that in our samples, water loss can occur at temperatures of 120°C and have argued that sample specific evaporative trends are suggestive of analytical artifacts. In the next section, we use a Rayleigh model to simulate the isotopic composition of water during fluid inclusion analysis, in comparison to those that would be expected for evaporation in cave environments. These predictions are then used to determine if the $\delta^2\text{H}/\delta^{18}\text{O}$ slopes and the magnitudes of isotopic fractionations observed in the fluid inclusion data are characteristic of the temperature and humidity conditions of the crusher device.

Isotope ratios during evaporation can be calculated using the Rayleigh equation:

$$R_l = R_{l-0} \times f^{(\alpha_{\text{eff}}-1)} \quad (1)$$

where R_l is the isotope ratio (^{18}R or ^2R) of the liquid at each step of an evaporative process, R_{l-0} is the isotope ratio of the initial liquid, f is the fraction of liquid remaining at each step, and α_{eff} is the effective vapor-liquid isotope

fractionation factor during evaporation ($R_v/R_l < 1$). α_{eff} was calculated with the formulation of the Craig-Gordon model (Craig & Gordon, 1965) for evaporation into atmosphere of fixed humidity and isotopic composition:

$$\alpha_{\text{eff}} = \alpha_{\text{diff}} \left[\frac{\alpha_{\text{eq}} - h(R_a/R_l)}{1 - h} \right] \quad (2)$$

where α_{eq} is the temperature-dependent equilibrium vapor-liquid isotope fractionation factor, h is the relative humidity, R_a is the isotope ratio in air, R_l is the isotope ratio in the liquid, and α_{diff} is the isotope fractionation during diffusion in air. α_{diff} can be expressed as the ratio of the molecular diffusivities of the heavy (HDO and H_2^{18}O) and light (H_2O and H_2^{16}O) water isotopologues:

$$\alpha_{\text{diff}} = \left(\frac{D_H}{D_L} \right)^n \quad (3)$$

D_H and D_L are the heavy and light isotopologues, respectively. The exponent n varies from zero in flow under completely turbulent conditions to one in purely diffusional regimes (Gonfiantini et al., 2018; Horita & Wesolowski, 1994).

For evaporation in the crusher device, we used equilibrium fractionation factors at 120°C calculated using the relationships presented by Horita and Wesolowski (1994), and a value for relative humidity that corresponds to the range of humid backgrounds set by the humidity generator ($h \approx 1$ –1.5% for 10,000 to 20,000 ppmv of H_2O at 120°C). The diffusion fractionation factor was calculated using the molecular diffusivities of Merlivat (1978), and a range of n values reflective of evaporation dominated by molecular diffusion ($n = 1$ to 0.8), as expected for evaporation from porous materials such as soil or leaf waters (Gonfiantini et al., 2018; Horita et al., 2008). Isotope ratios of the water vapor in air and the initial liquid (^{18}R and ^2R) were calculated with the isotopic composition of the water background using a laboratory standard (DI2; $\delta^{18}\text{O} = -7.63 \pm 0.06$ and $\delta^2\text{H} = -50.72 \pm 0.5$) and with the average isotopic composition of the fluid inclusion data ($\delta^{18}\text{O} = -7.7$ and $\delta^2\text{H} = -48.1$), respectively.

For evaporation in the cave system, we used a range of temperatures from 19 to 25°C (approximate glacial-interglacial temperature change in Borneo) and the respective equilibrium fractionation factors from Horita and Wesolowski (1994). The isotope ratios of the initial liquid were approximated with the average isotopic composition of the fluid inclusion data, and the isotope ratios of the cave air vapor were assumed to be equal to the isotope ratios of vapor in isotopic equilibrium with the initial liquid. Even though measured cave humidity in Borneo has always been close to 100%, we used a range in relative humidity of 60%–90% to include the possibility of less humid conditions. Appropriate values of the n parameter for typical cave conditions are difficult to assign as they are not known. In laboratory experiments, however, high n values (close to 1) are obtained when evaporation rates are slow under quiet atmospheres (Gonfiantini et al., 2018). These conditions are likely similar to those experienced inside caves with poor ventilation. Similarly, n values close to 1 are expected if evaporation occurs in the epikarst where water evaporates through fractures and/or internal porosity. On the other hand, lower n values (close to 0.5) are observed in experiments when evaporation proceeds under open air conditions (Gonfiantini et al., 2018). These values may be more appropriate for caves with strong ventilation. Given these uncertainties, we use a range of n values of 0.5–1, which encompasses the full range of possible conditions that may occur in a cave system.

For evaporation in the crusher, the Rayleigh model predicts slopes that range from 1.36 to 1.46 (or 1.41 ± 0.06 ; 2σ) (Figure 6a, blue line). The range in values for the slope is primarily controlled by the range of n values and only slightly affected by errors in equilibrium fractionation factors or molecular diffusivities (Figure S7 in Supporting Information S1). The modeled slopes are statistically indistinguishable to the mean evaporative slope calculated with the replicate data set (1.0 ± 0.6 2SE). For evaporation in the cave system, on the other hand, the model predicts slopes that are significantly larger than the empirical estimate, ranging between 2.7 and 4.01 (Figure 6a, green area), and where magnitudes are primarily controlled by the n parameter (see Figure S7 in Supporting Information S1). These observations add additional support to our interpretation that isotopic enrichments are the result of partial evaporation prior to analysis.

The results obtained from the cave model indicate that evaporative fractionation likely did not occur in Borneo. However, this is not true for other cave systems, where the model results are generally in line with the isotopic enrichments that have been observed. Values of evaporative slopes are not always reported, but two estimates are available. Warken et al. (2022) report a slope of 2.4 for fluid inclusion data from a stalagmite from Puerto Rico that grew during the last deglaciation and interpret it as the result of evaporation on the top of the stalagmite. For

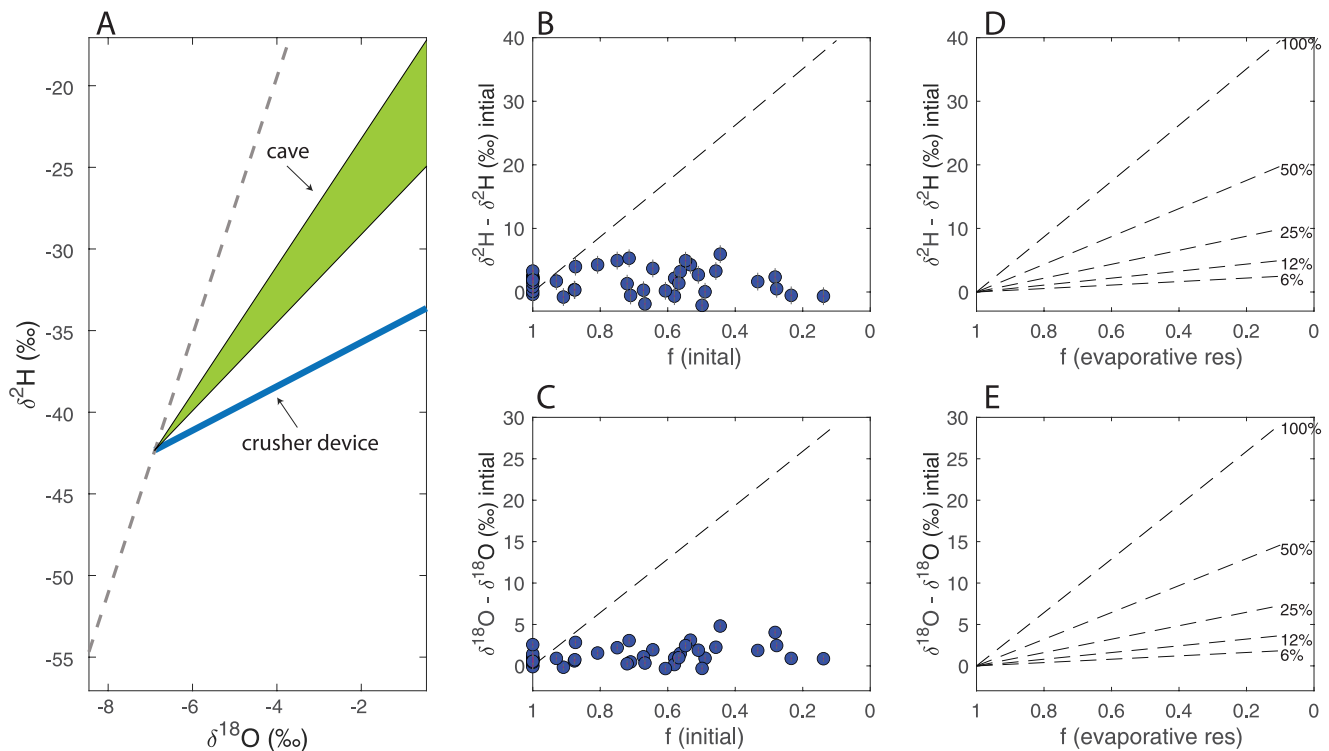


Figure 6. Results of the Rayleigh models for an evaporation process. (a) Slopes in $\delta^{18}\text{O}$ versus $\delta^2\text{H}$ space for evaporation in the crusher device (slope 1.4—blue line) and range of slopes for evaporation in a cave (slopes 3.9 to 2.7—green filled area). Dashed line is the local meteoric water line (LMWL). (b and c) Change in fluid inclusion $\delta^{18}\text{O}$ and $\delta^2\text{H}$ values, respectively, versus total water lost to evaporation (f = fraction of initial water remaining). Blue and green circles are $\delta^{18}\text{O}$ and $\delta^2\text{H}$ replicate analyses, respectively. f (initial) was calculated assuming that the initial water content for each sample is equal to that observed in the replicate analyses that plot closer to the LMWL. (d and e) Change in $\delta^{18}\text{O}$ and $\delta^2\text{H}$ values versus water lost in an evaporative reservoir. Dashed lines indicate the trends predicted given the different amounts of inclusions that experience evaporation (i.e., 100% means that all inclusions experience evaporation, and 25% means that only 1/4 of total inclusions loose water).

this cave, the model predicts slopes that range between 2.7 and 4.2 (Figure S7 in Supporting Information S1). For a stalagmite from a cave located in the Sierra Nevada, USA, Wortham et al. (2022) reported a slope of 3.9, which they interpreted as the result of evaporation in the soil, epikarst, or inside the cave. For this cave, the model predicts slopes that range between 2.8 and 4.7 (Figure S7 in Supporting Information S1). Thus, at face value, both evaporative slopes are consistent with the results of the model for these cave environments. Moreover, and in contrast to our Borneo stalagmite, evaporation is actually possible in both caves, as they experience seasonal changes in cave ventilation and humidity lower than 100%. However, we caution that evaporative slopes were calculated from samples that are not coeval. Evaporative slopes may therefore be uncertain if the primary isotopic composition of the unevaporated water was variable. Moreover, evaporative loss through analyses cannot be ruled out, so results may incorporate modifications by both processes.

The results of both model scenarios predict larger fractionations—and thus larger distances from the LMWL—in samples that experienced more evaporation (Figures 6b and 6c). Our observations from the replicate measurements are mainly consistent with these predictions. For example, in the replicate data set, there is a general relationship between lower water contents and larger $\delta^{18}\text{O}$ offsets from the LMWL (Figure 3b). Note that this is only true in 9 out of the 11 samples that we tested. Two samples show either no trend or a trend in the opposite direction (red dashed lines in Figure 3b), which cannot be explained by evaporative fractionation. However, we interpret these discrepancies as the result of analytical errors in $\delta^{18}\text{O}$, variable water contents, difficulties in measuring strictly coeval samples, and variability in the number of inclusions that experience partial versus total leakage. These are further discussed below (Section 4.3).

Although the model for the crusher device explains well the slopes of the evaporation trends, and the general relationship observed between distance from the LMWL and water loss, it does not correctly predict the magnitudes of the fractionations that we observe. For example, $\delta^{18}\text{O}$ and $\delta^2\text{H}$ enrichments in the model are more than 30‰

larger than our observations (Figures 6b and 6c). Smaller observed fractionations, though, can be explained by mixtures of water from pristine and partly leaked inclusions. While all inclusions become stretched upon heating to 120 °C, only some of them start leaking, that is, connect to the sample surface via cracks, crystal boundaries and interconnected pores and lose all or part of their water through evaporation. Inclusions that do not leak preserve the original drip water isotopic composition. When these are crushed, the pristine water mixes with waters from partially evaporated reservoirs, and the magnitude of total observed fractionation is reduced. These mixing effects can be easily incorporated into the evaporation model, but direct comparison between predictions and our data are not straightforward. The main reason is that we do not know the relative proportion of inclusions in our samples that are amenable to leakage. Nevertheless, we can show that the range of observed fractionations are consistent with mixtures of 95% to 75% pristine inclusions and 5%–25% evaporated reservoirs (Figures 6d and 6e). It is important to note that this does not necessarily mean that only a small percentage of inclusions lost water; on the contrary, in some cases the majority of water was probably lost. For instance, we observe a range of more than 80% in the water content of some samples (Figures 6b and 6c). Part of that range likely results from variable initial water contents, but a considerable part of it is probably due to leakage and complete loss of water from inclusions through evaporation. Since completely evaporated reservoirs do not affect the measured isotopic composition, they can explain large variabilities in recovered water with only small concomitant changes in delta values.

4.3. Fluid Inclusions, Water Contents, and Isotopic Modifications

The majority of early deglacial and glacial samples yield $\delta^{18}\text{O}$ values that fall close to the LMWL. The opposite is true for Holocene and late deglacial samples; most of them yield $\delta^{18}\text{O}$ values that plot to the right of the LMWL. We have demonstrated that isotopic enrichments can be explained by evaporative fractionation during analysis. Since we observe a close correlation between the extent of fractionation and the age of the samples, an open question is what features make some samples more amenable to isotopic fractionation in the crusher device? The most striking differences between the younger and older portions of the stalagmite are the size distributions of the fluid inclusions. While very large and elongated inter-crystalline inclusions are present throughout the stalagmite, the older parts have abundant and relatively large fluid inclusions that are orders of magnitude larger than the very small fluid inclusions observed in the Holocene and late deglacial portions of the stalagmite (see Section 3.1). These observations explain why more water was recovered from older samples, but the water content of the samples alone does not explain why the younger samples tend to show larger isotopic modifications.

We propose that the very large and elongated inter-crystalline fluid inclusions (present throughout the stalagmite) are more likely to leak and lose water than other inclusions in the calcite fabric. A possible reason is the length of these elongated inclusions and their position between crystal boundaries, which gives them a better opportunity to be connected to the surfaces of the samples via cracks and interconnected pores. These inclusions, thus, likely form a partially evaporated reservoir that mixes with a more pristine reservoir from the other inclusions when the samples are crushed. Younger samples show larger isotopic modifications because the very small pristine inclusions likely contribute only small amounts of water, and a relatively large fraction of water that was measured in these samples was provided by large, partially evaporated, inter-crystalline fluid inclusions. In the older samples, in contrast, where isotopic modifications are significantly smaller or absent, the inclusions that contribute to the pristine reservoir are orders of magnitude larger than those in the Holocene and late deglacial samples and likely provide most of the water. Although evaporation probably also occurs in these samples, the size of the partly evaporated reservoir relative to the pristine reservoir is much smaller. Given these observations, we suggest avoiding samples with low water contents and a large contrast of inclusion sizes. That being said, Figure 4b illustrates that isotope compositions close to the LMWL are also possible with samples that contain only little water. We further caution that additional observations from other stalagmites are necessary to draw broad conclusions on the susceptibility of different fluid inclusions to leakage and on sampling strategies for fluid inclusion analyses.

4.4. Correction of Evaporative Trends

In this section, we describe an approach to correct fluid inclusion water isotope measurements for evaporative fractionation during analysis and discuss the reliability of the proposed correction algorithm. The most straightforward approach to correct the data is to use the $\delta^2\text{H}/\delta^{18}\text{O}$ slope of the evaporative trends. This is possible

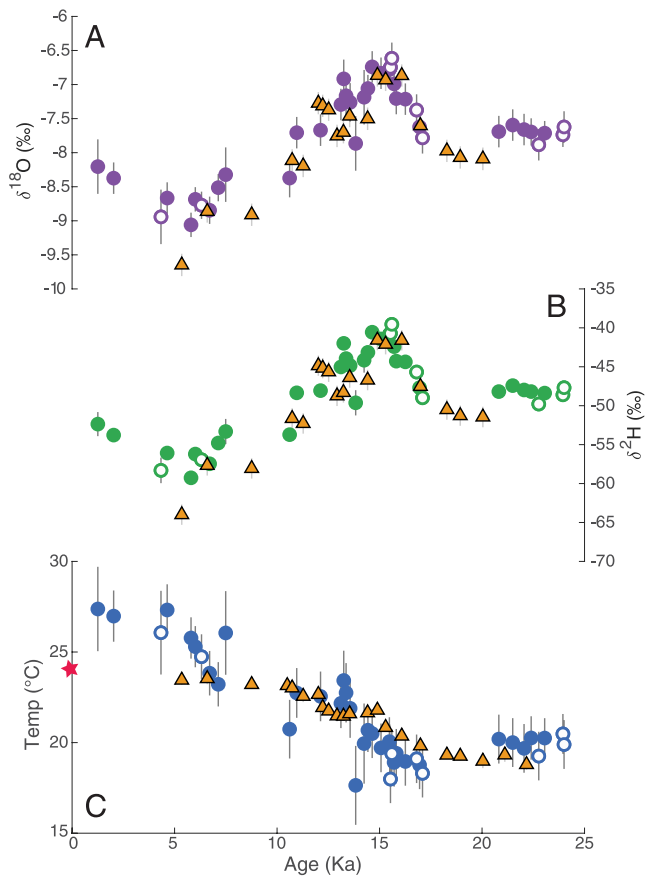


Figure 7. (a) Corrected fluid inclusion $\delta^{18}\text{O}$ values of the SSC01 stalagmite (circles) and calculated drip water $\delta^{18}\text{O}$ values of the SC02 stalagmite (triangles). (b) Corrected fluid inclusion $\delta^2\text{H}$ values of the SSC01 stalagmite (circles) and calculated drip water $\delta^2\text{H}$ values of the SC02 stalagmite with the local meteoric water line (LMWL) (triangles). (c) Fluid-calcite $\delta^{18}\text{O}$ temperatures of SSC01 from corrected $\delta^{18}\text{O}$ values (circles) and microthermometry temperatures of SC02 from Løland et al. (2022) (triangles). Fluid-calcite $\delta^{18}\text{O}$ temperatures were calculated using the Tremaine et al. (2011) relationship. Error bars are 68% confidence intervals. Open circles are samples that fell within 68% CI of the LMWL before correction. Red star is the approximate modern cave temperature (24°C).

because all samples should share the same slope, as they were subjected to the same conditions during analyses (e.g., temperature, relative humidity, flow regimes). To correct the data, we constructed linear models for each replicate measurement using the evaporative slope that was estimated with the Rayleigh distillation model ($\delta^2\text{H}/\delta^{18}\text{O} = 1.41 \pm 0.06 2\sigma$; Figure 6a). The original (pristine) isotope compositions were then determined by the points of intersection between the linear models and the LMWL. The uncertainty of each corrected replicate data point was estimated by Monte Carlo simulations ($N = 1,000$) propagating long-term analytical uncertainty of the isotope measurements, the uncertainty of the evaporative slope and the uncertainty of the slope and intercept of the LMWL. More details on error propagation are given in the supplemental information (supporting information 8 for details). Additionally, in the supplemental material we also provide estimates for evaporative slopes at other analytical temperatures, which may be useful for laboratories that perform analyses at lower temperatures (Figures S7a–S7f in Supporting Information S1). Corrected data are presented in Figure 7.

Unlike the raw fluid inclusion data (Figure 5), the corrected data are in very close agreement to the calculated SC02 dripwater isotope values (Figures 7a and 7b). For example, calculated and measured $\delta^{18}\text{O}$ and $\delta^2\text{H}$ values show the same trends throughout the length of the record, and differences between corrected SSC01 and calculated SC02 $\delta^{18}\text{O}$ and $\delta^2\text{H}$ values at coeval or interpolated locations are small and within our long-term reproducibilities in fluid inclusion water isotope measurements (see Figure S9 in Supporting Information S1). As expected, given the shallow slopes of the evaporative trends, the correction has a larger relative effect on $\delta^{18}\text{O}$ values than on $\delta^2\text{H}$ values. Furthermore, when the uncorrected data are close to the LMWL, such as for samples with ages before approximately 16 ka, our correction procedure only minimally affects them. Much closer agreement is also found between the fluid-calcite $\delta^{18}\text{O}$ temperatures and SC02 microthermometry estimates when the former are calculated using the corrected $\delta^{18}\text{O}$ values (Figure 7c). Differences between fluid-calcite $\delta^{18}\text{O}$ temperatures and microthermometry estimates are also within our long term reproducibility in fluid-calcite $\delta^{18}\text{O}$ temperatures (see Figure S9 in Supporting Information S1).

We have shown that the correction procedure removes the disagreement between calculated and measured dripwater isotope values, and between fluid-calcite $\delta^{18}\text{O}$ temperatures and microthermometry estimates. However, our correction routine relies on independent knowledge of the LMWL. We used the present-day LMWL to correct data for analytical artifacts; however,

in some settings, the LMWL may not be precisely known or it may have been different in the past. For example, the LMWL can vary because it depends on the deuterium excess of local precipitation, which is primarily controlled by the moisture conditions of the source regions (e.g., temperature, relative humidity, and isotope composition; Pfahl & Sodemann, 2014), and the presence/absence of non-equilibrium processes during precipitation (e.g., re-moistening and rain drop evaporation; Lee & Fung, 2008; Martinelli et al., 1996). This is the main disadvantage of our correction routine, and it can be an important source of bias in paleoclimate reconstructions.

To examine the sensitivity of corrected values to possible changes in the LMWL, we looked at results from the iTRACE experiment (He et al., 2021), which is a transient (20–11 ka) water isotope enabled simulation of global climate using the Community Earth System Model version 1.3. In the closest grid cell to the Borneo site, the model predicts variations in LMWL parameters of up to 3 ‰ in the intercept and 0.3 in the slope. The magnitude of these changes, however, is insufficient to impact corrected fluid inclusion data outside analytical error (i.e., less than 0.3‰ for $\delta^{18}\text{O}$ and $\delta^2\text{H}$ values; see Text S10 in Supporting Information S1). As an additional robustness check, we examined LMWLs from three Global Network of Isotopes in Precipitation sites in the Indo-Pacific region. These sites are located at latitudes where seasonal migrations of the ITCZ cause large differences in seasonal precipitation amounts. We use changes in LMWLs at these sites as proxies for hydrological conditions

in Borneo during the deglaciation, where regional arid conditions are thought to be caused by a southward shift in the ITCZ over the Pacific (Buckingham et al., 2022; Carolin et al., 2013; Partin et al., 2007). We find that seasonal ITCZ migration at these sites causes summer-winter variations in LMWL parameters of up to 4‰ in the intercept and 0.6 in the slope. However, we again find that changes of these magnitudes do not have an impact on data correction outside analytical errors (see supporting information 10). These observations suggest that the correction is not very sensitive to changes in the LMWL parameters within a realistic range.

4.5. Agreement Between Borneo Stalagmite Paleothermometers

Our corrected fluid inclusion data present an opportunity to test the performance of the empirical fluid-calcite $\delta^{18}\text{O}$ cave calibration of Tremaine et al. (2011). Empirical calibrations such as the one from Tremaine et al. (2011) are needed because speleothem calcite rarely forms in oxygen isotope equilibrium with dripwater (Guo & Zhou, 2019; Mickler et al., 2004). As previously discussed, fluid-calcite $\delta^{18}\text{O}$ temperatures determined with this cave calibration are statistically indistinguishable from independent microthermometry estimates over the length of both records (Figure 7c). Thus, the cave calibration of Tremaine et al. (2011) appears to perform very well in Borneo, possibly because disequilibrium remained approximately constant through time. This is surprising given the evidence for changes in temperature and hydroclimate in Borneo through the deglaciation, which were likely accompanied by changes in the environmental variables that drive disequilibrium (e.g., prior calcite precipitation, cave air $p\text{CO}_2$, drip rate, water film thickness, and growth rates; e.g., Carlson et al., 2020; Deininger et al., 2021; Johnston et al., 2013). While counterintuitive at first sight, our observation can be explained if disequilibrium—at the timescales captured by our samples—is not very sensitive to changes in these variables. There is some evidence from theoretical models to support this idea. For example, Guo and Zhou (2019) recently published results of a reaction diffusion model that tracks the concentrations of isotopologues in the $\text{CaCO}_3\text{-DIC-H}_2\text{O}$ system during speleothem formation. They found that their model is able to reproduce the temperature dependence of ^{18}O fractionations observed by Tremaine et al. (2011). Moreover, when environmental variables (PCP, cave air $p\text{CO}_2$, and water film thickness) are randomly varied by 25% in their model, the $\delta^{18}\text{O}$ value of calcite varies only by $\pm 0.23\text{‰}$ (2σ). This corresponds to only $\pm 1.2^\circ\text{C}$, a magnitude that cannot be detected given typical errors in fluid-calcite $\delta^{18}\text{O}$ temperatures. Thus, we hypothesize that disequilibrium in Borneo, despite changes in environmental variables that drive it, remained close to the average magnitudes observed in modern speleothems and within our error bars.

4.6. Deglacial Hydroclimate Evolution in Borneo

Corrected fluid inclusion data provide the first directly measured Borneo dripwater $\delta^{18}\text{O}$ and $\delta^2\text{H}$ values during the last deglaciation. Our data agree well with previous estimates of hydrological conditions inferred from calcite $\delta^{18}\text{O}$ and with proxies for the strength of the Atlantic Ocean meridional overturning circulation (AMOC) (Figure 8). For instance, we observe a shift to the most positive dripwater $\delta^{18}\text{O}$ values during Heinrich event 1 (H-1), a time interpreted as a period of weakened regional convection based on stalagmite $\delta^{18}\text{O}$ measurements (Buckingham et al., 2022; Partin et al., 2007) and calculated dripwater $\delta^{18}\text{O}$ (Løland et al., 2022). Both the measured and calculated dripwater $\delta^{18}\text{O}$ records show a gradual decrease in dripwater $\delta^{18}\text{O}$ values during H-1, a small decrease during the Bølling-Allerød (BA), and a return to LGM values during the Early Holocene (Figures 8c and 8d). Especially for H-1, our data add new details to the existing dripwater $\delta^{18}\text{O}$ record, showing a late onset of the hydroclimate response when compared to the AMOC record, and even suggesting a two-step evolution. These features are worth further investigation.

5. Conclusions and Recommendations

We measured the isotopic composition of fluid inclusion waters in a stalagmite from Borneo that grew during the last deglaciation. We find that many samples partially lost water due to evaporation during warm-up before crushing and analysis, and propose that water loss and the magnitude of isotopic modifications are controlled by the type and size distribution of fluid inclusions. Partial evaporation results in $\delta^{18}\text{O}$ and $\delta^2\text{H}$ enrichments that follow typical evaporation lines with shallow $\delta^{18}\text{O}/\delta^2\text{H}$ slopes. Through multiple replicate analyses of coeval samples, we constrain the mean value of the evaporative $\delta^{18}\text{O}/\delta^2\text{H}$ slope at 1 ± 0.6 (2SE). This value is indistinguishable from the predictions of a Rayleigh distillation model that incorporates kinetic ^2H and ^{18}O fractionations during evaporation in the hot crushing apparatus.

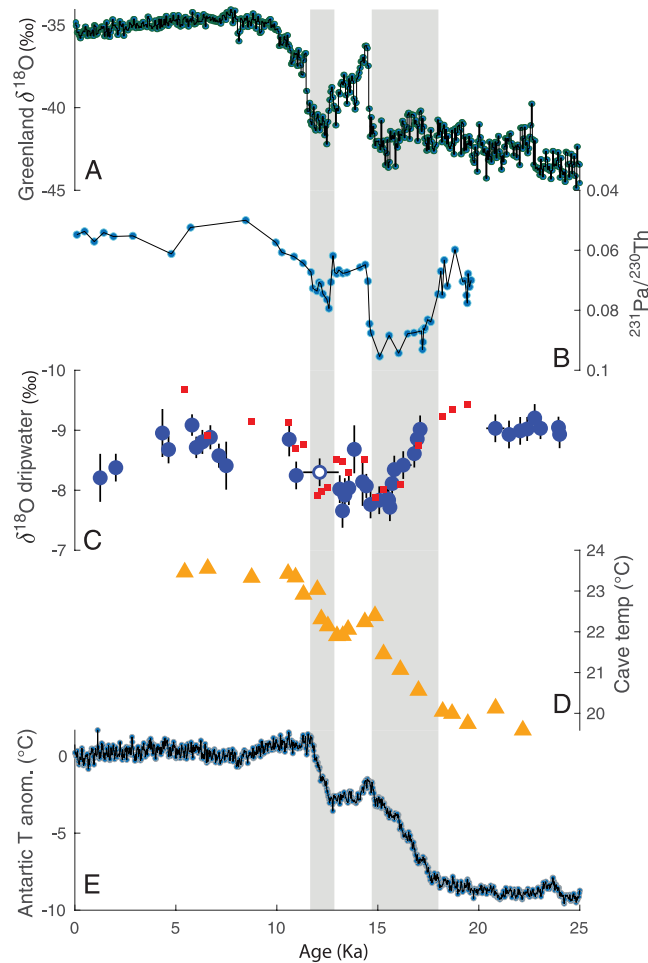


Figure 8. Comparison of Borneo dripwater $\delta^{18}\text{O}$ values with cave temperature and other paleoclimate records. (a) Greenland ice core (NGRIP) $\delta^{18}\text{O}$, a proxy for Greenland temperature (North Greenland Ice Core Project members, 2004). (b) Changes in North Atlantic Ocean meridional overturning circulation based on the $^{231}\text{Pa}/^{230}\text{Th}$ of N. Atlantic sediment core OCE326-GGC5 (McManus et al., 2004). (c) Measured SSC01 fluid inclusion $\delta^{18}\text{O}$ values after correction for analytical artifacts (blue circles, errors bars are $\pm 69\%$ ci), and calculated SC02 dripwater $\delta^{18}\text{O}$ values from Løland et al. (2022) (red dots). Both data series were corrected for ice volume related changes the $\delta^{18}\text{O}$ value of the global ocean (see Løland et al. (2022) for details). The open blue symbol shows a sample that was obtained across a hiatus in SSC01 stalagmite, implying uncertainty in its age. (d) SC02 cave temperatures from fluid inclusion microthermometry corrected for sea-level-induced changes in cave altitude (Løland et al., 2022). (e) Antarctic temperature anomaly (Parrenin et al., 2013).

We provide a robust and physically based method to correct for analytical modifications and demonstrate that it can be used to reliably correct data to pristine dripwater isotopic compositions. We find that corrected fluid-calcite $\delta^{18}\text{O}$ and resulting paleotemperatures show excellent agreement with independent dripwater $\delta^{18}\text{O}$ and temperature estimates, which implies only small variations in speleothem ^{18}O disequilibrium. The corrected data provide the first directly measured dripwater $\delta^{18}\text{O}$ and $\delta^2\text{H}$ estimates for Borneo across the last deglaciation and support the previously inferred hydroclimate evolution.

Our observations suggest that water loss during fluid inclusion water isotope analysis readily occurs and that the magnitude of isotopic modifications can vary from sample to sample. Future improvements to the analytical method should seek to remove these artifacts. In lieu of such improvements, we suggest that multiple replicate measurements of coeval samples and careful petrographic inspections be used to identify suitable samples, calcite fabrics, and the type of fluid inclusions that are prone to evaporative artifacts. Special attention should be given to samples with low water yields as these have been identified in this work and by others as samples that more likely show isotopic modifications. Caution is warranted when interpreting speleothem fluid inclusion isotope data that are not checked for or corrected for evaporation. This is especially true for $\delta^{18}\text{O}$ data as these are affected

by non-equilibrium fractionation to a much greater extent than $\delta^2\text{H}$ relative to expected respective variations in precipitation.

Data Availability Statement

Data are archived at Fernandez et al. (2023).

Acknowledgments

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