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New high-precision U–Th dates for speleothems from the Assynt caves and their significance for past environmental change

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Abstract: The Late Quaternary climatic history of substantial parts of Scotland remains poorly understood because much of the sedimentological record that might have provided evidence has been erased by glacial and post-glacial erosion. Here we report 19 U/Th dates obtained from archived speleothem samples from Assynt, NW Scotland. We used state-of-the-art MC-ICP-MS U-series dating and find that speleothem deposition in Assynt was restricted to warm periods when the surface was ice-free and continuous permafrost absent. Our dataset reveals significant speleothem deposition during the Ipswichian (Marine Isotope Stage 5e) and the Holocene (MIS 1), and the formation of thin flowstone sheets during Greenland Interstadials 14 and 12 during the last glacial period. These dates highlight the potential for the use of speleothems to constrain the timings of regional glacial and periglacial conditions, and for the reconstruction of interglacial and interstadial environmental dynamics in Scotland.

Keywords: Assynt karst; U-series dating; Ipswichian interglacial; Holocene; last glacial period.

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Introduction

The whole of Scotland has repeatedly been subject to Quaternary glaciation, which left deep geomorphic marks in the landscape and removed most traces of pre-glacial surface sediments (Hall *et al.*, 2019, Merritt *et al.*, 2019, Bradwell and Ballantyne, 2021). This intense erosion has left us very few sedimentary archives, such as pre-glacial peat and lake deposits, which testify to the nature of environmental and climatic conditions prior to the formation of the last British–Irish ice sheet. However, sand and clay deposits in caves – and even more importantly speleothems – are to a large degree protected from the vagaries of surface dynamics. With their wide range of environmental proxies, speleothems can inform us about past variability in surface conditions, such as moisture supply, soil microbial activity, vegetation cover, or air temperature. In areas where caves are developed, they have potential for reconstructing a detailed picture of Scotland's Quaternary history. This potential might be obstructed by past presence of surface ice or permafrost.

The Assynt district, 15km northnortheast of Ullapool and 50km southsouthwest of Durness on the north coast, is Scotland's premier caving area (Fig.1; Lawson and Dowswell, 2022). Here, the rocks are mostly dolostones of the lowest two formations of the (Cambrian to Ordovician) Durness Group, stratigraphically some 200m thick but repeatedly further piled-up in a duplex structure caused by tectonic movement within the Moine Thrust Belt (Searle, 2023). Many of Assynt's caves contain broken, relict, and actively growing speleothems, which offer opportunities to provide a chronological framework of the region's glacial–interglacial history, and to reconstruct past climate and surface environmental conditions. Speleothems can be dated by U-series disequilibrium methods, and thus can provide an excellent chronological framework within which to locate specific palaeoenvironmental conditions and changes.

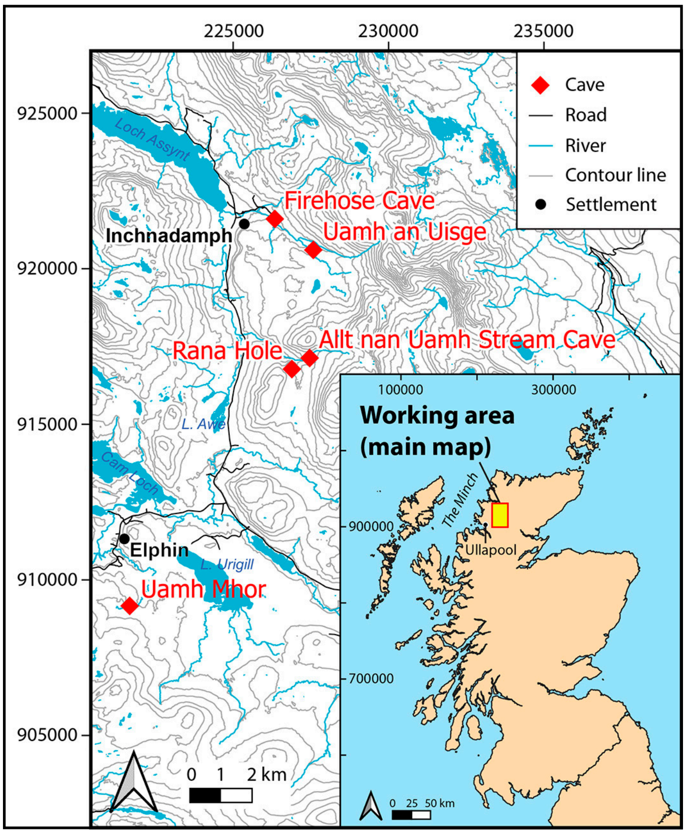

Figure 1: Location of the caves referred to in this study. The inset map shows the location of the Assynt area in northern Scotland. Coordinates are those of the British National Grid.

The presence of speleothems in northwestern Scotland did not escape the attention of early karst geomorphologists and palaeoclimatologists, and many were sampled decades ago with the aim of reconstructing details of past climatic and regional environmental changes, landscape evolution and glacial dynamics (Lawson 1981, Atkinson *et al*., 1986; Baker *et al*., 1993; Lawson and Atkinson, 1995; Hebdon *et al*., 1997; Baker *et al*., 2002; Proctor *et al*., 2002). The importance of the cave deposits in northern Scotland cannot be overstated, because this northwesternmost corner of Britain is today very exposed to the highly oceanic climate of the North Atlantic, and in Last Glacial times lay within the gathering grounds of the Minch Ice Stream, a feature of the Scottish Ice Sheet that extended and carried ice to the outer edge of the continental shelf (Bradwell *et al*., 2008). This makes the caves, and speleothems forming in them, potentially highly sensitive to changes in atmospheric circulation pattern at this latitude, including the North Atlantic Oscillation (NAO) (Hurrell, 1995; Wilby *et al*., 1997; Trouet *et al*., 2009). This far-reaching atmospheric circulation system influences the entire Northern Hemisphere, and especially the British Isles and maritime western Europe. Speleothems from Assynt caves have the potential to record detailed information on past hydroclimate, and are suitable for the reconstruction of seasonal to multidecadal-scale climate dynamics (Baker *et al*., 1993; Proctor *et al*., 2000, 2002; Trouet *et al*., 2009). Well-dated speleothems also provide information about the temporal distribution of full glacial conditions and continuous permafrost (Atkinson *et al*., 1978; Vaks *et al*., 2020; Panitz *et al*., 2024). The key to understanding this rich library of past climate and environment is developing a robust radiometric chronology, because without a well-defined '*when*' we have little chance to understand the '*why*' and '*how*' of past changes in temperature, precipitation, vegetation or ecotone composition.

Assynt speleothems collected in the 1980s and 1990s were originally dated by counting radioactivity of uranium and its thorium daughter isotope, using alpha spectrometry (AS) (Atkinson *et al*., 1986, 1995; Lawson and Atkinson, 1995; Hebdon *et al*., 1997). While this was the only technique available at the time, AS dating came with unavoidably large dating uncertainties. Nevertheless, the wide temporal resolution of early alpha spectrometry dates did suggest that speleothem growth occurred even within the most recent glacial period (the Devensian, c. 115–11.7 ka BP); examples include dates on speleothems SU12-80 from Uamh an Tartair and SU1-80 from Firehose Cave (Atkinson *et al*., 1986; Lawson *et al*., 2023).

Vadose speleothems are prevented from forming beneath ice sheets by sub-zero cave temperatures (if the ice sheet is locally frozen at its base) or by inundation with water (where the ice sheet has liquid water at its base) or (occasionally) sediment. Hence, the early dates from Assynt were interpreted in terms of temporal constraints on the periods when ice sheets might have covered the area (Lawson, 1981; Atkinson *et al*., 1986; Lawson and Atkinson, 1995; Hebdon *et al*., 1997).

In recent years a renewed interest in the local and regional dynamics of the last British–Irish Ice Sheet (BIIS) has developed, particularly for marine and coastal areas like the Minch at the Atlantic seaboard of Scotland (Gandy *et al*., 2018). Marine ice sheet instability is an increasingly relevant research topic in the face of accelerating global warming and sea-level rise (Garbe *et al*., 2020); Assynt with its karst offers many opportunities for developing a geochronological framework of Scotland's glacial and non-glacial history. In addition, well-dated speleothems from warm inter- and postglacial periods like the Ipswichian (MIS 5e, c. 130–119 ka BP) and the Holocene (the last c. 11,000 years) offer unique opportunities to reconstruct past surface conditions, including vegetation and wildfire dynamics, hydroclimate, and temperature changes (Wong and Breecker, 2015). In this context, a more precise set of dates for the Assynt speleothems could form the chronometric basis for significant progress in understanding environmental variability in both glacial and interglacial periods.

Advances in radiometric dating technique since the 1990s have improved precision, accuracy and maximum age limits dramatically. Modern mass-spectrometry measurements of U and Th isotopes now permit age determinations on sample weights of much less than one gramme, allowing age determinations with uncertainties as low as 0.5% or less. Here we present new, state-of-the-art dates on archived samples, that seemed critical for constraining the expansion and decay of the BIIS over Assynt, some with original AS dates, and other previously undated samples collected by two of the authors of the early studies (TJL and TCA). We use the re-dated and newly dated samples to discuss the relevance of these speleothems for understanding environmental change in Assynt over the last c. 120,000 years and the potential for palaeo-environmental studies.

Study Sites and Samples

Speleothem specimens collected from five different caves were dated in this study: Firehose Cave and Uamh an Uisge in the Traligill valley, Rana Hole and Allt nan Uamh Stream Cave (ANUSC) in the Allt nan Uamh valley, and Uamh Mhor in the Knockan basin. Descriptions of these caves together with adjoining sites and details of drainage basin hydrology are given in relevant sections of Lawson and Dowswell (2022). Cave locations are shown in Figure 1 and are considered here from north to south. In total, 19 dating samples from 10 different speleothems were analysed: 7 from Rana Hole, 5 from Uamh Mhor, 5 from Firehose Cave, one from Uamh an Uisge, and one from ANUSC (Fig.2).

Firehose Cave (Traligill valley)

The resurgence flow from this cave debouches from the valley side into a pool beneath a small waterfall. Access is tricky; one has to ascend a steep ramp (along a minor thrust plane) down which pours fast-flowing water. A series of interconnecting pools at the top of the ramp lead to a short duck into a well-decorated chamber before the cave sumps. Sample SU1-80, a stalactite attached to wall flowstone, was collected by P L Smart in 1980 and dated at the Scottish Universities Research and Reactor Centre (SURRC), East Kilbride, by alpha spectrometry (AS). Two AS dates suggested speleothem growth between 38 ± 6 ka and 26 ± 3 ka BP. An adjoining sample (UEA810529-5), exhibiting a similar internal structure to SU1-80 (Fig.2), was collected in 1981. Both were dated at Oxford University as part of the current study.

Uamh an Uisge (Traligill Valley)

One of the three entrances to the Cnoc nan Uamh cave system, Uamh an Uisge (also known as 'The Waterslide'), sees a voluminous flow of water cascade down a thrust plane associated with the Traligill Main Thrust, exposed at the surface through unroofing. The latter is assumed to have resulted from erosion under the last ice sheet. About halfway down on the far (i.e. left) side of the stream is a small grotto from which stalagmite TJL020817-3 was obtained in 2002; this sample was dated for the first time as part of the current study.

Allt nan Uamh Stream Cave (Allt nan Uamh valley)

This multilevel cave system – also referred to as 'ANUSC' – is accessed from above the normally dry stream bed via a chiselled hole leading into a solution tube. Sample UEA950905-6 is part of a collection of thin flowstone pieces deposited on the ceiling and walls of the entrance tube amongst scalloping and well-developed spongework. An AS analysis at the University of East Anglia (UEA) in the mid-1990s revealed the presence of detrital Th impurities at the time the specimen was formed, so the calculated date of 19.7 ± 2.4 ka BP should be regarded as an upper age limit rather than the 'true' age. A second aliquot from this material has now been re-dated.

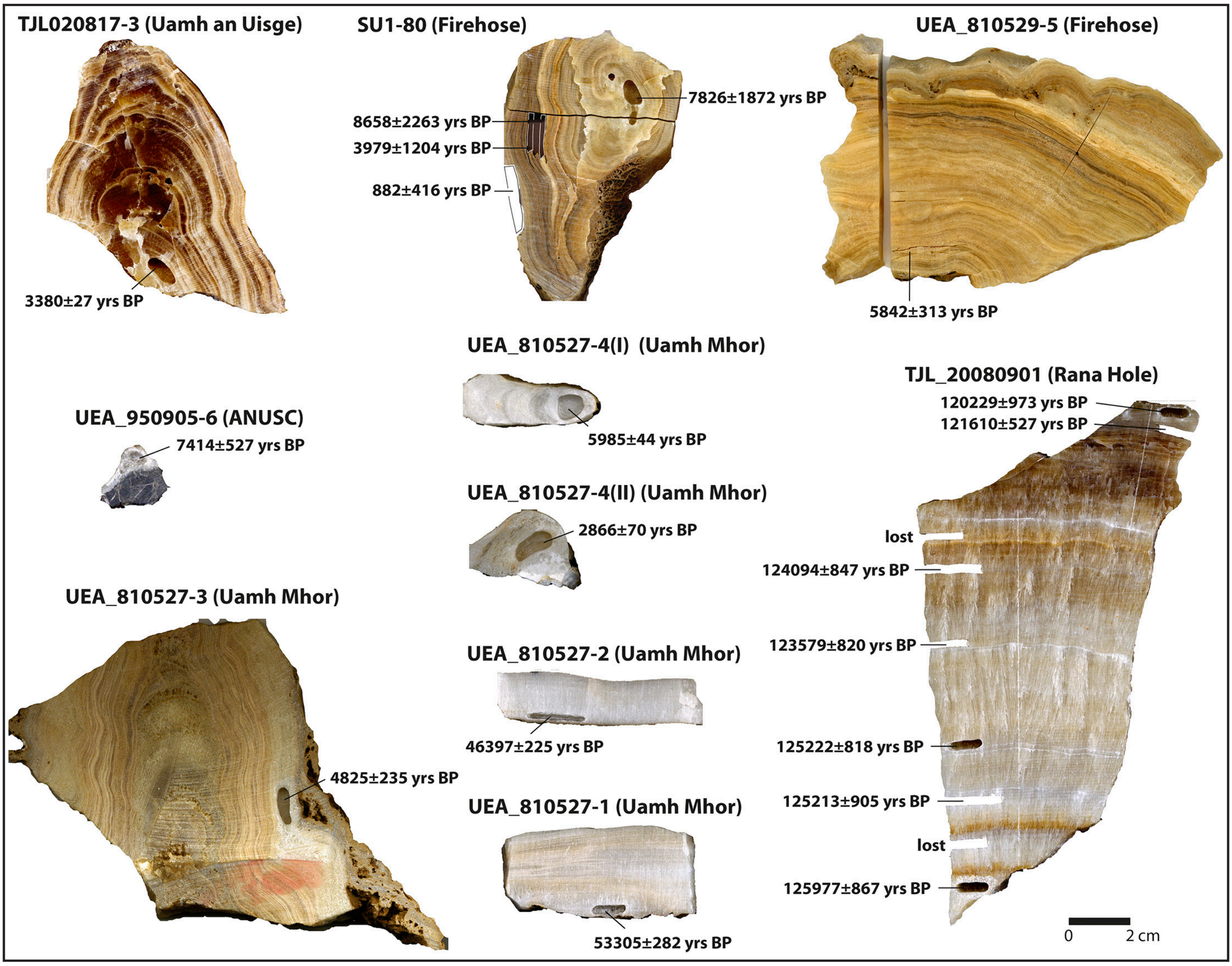

Figure 2: *Speleothem samples and their ages. The cave names are shown in brackets. All samples are presented at the same scale.*

Rana Hole (Allt nan Uamh valley)

Rana Hole is accessed via a 30m shaft excavated over 12 years (1995–2007), which leads down to a multilevel cave system in the upper part of the Creag nan Uamh, eventually connecting with Uamh an Claonaite, Scotland's longest cave system, via a 6m pitch at Black Cuillin Chamber. Sample TJL20080901 is a broken section of flowstone, some 18cm thick, deposited at the bottom of Black Rift on a slope of breakdown. The original location of the *in situ* flowstone floor from which this sample is derived has not been identified thus far, although it is likely to be within a few metres above the current position of breakdown.

Uamh Mhor (Knockan basin)

The Abhainn a' Chnocain stream sinks at Uamh an Tartair (Knockan). Crawling along the subterranean stream gives access to the bottom of a large open pothole (6m in diameter and 20m deep) known as Uamh Mhor, representing a former large cave chamber that was presumably unroofed by erosion under the last ice sheet. Sample UEA810527-3 is a c. 50cm-tall dripstone column whose structure indicates initial growth of a stalagmite joined from above by a thick stalactite, all covered in a later layer of dirty, vuggy calcite. It was collected from a large muddy aven on the northeast side of the cave. Sample UEA810529-4 consisted of two small broken white stalactites lying on the sloping surface of peaty sediment forming the floor of the highest part of the aven. From a small, sloping solution tube in the western wall of the former chamber, a clean, c. 2cm-thick flowstone of white calcite was sampled (sample UEA810527-1), separated by a thin (1–2mm-thick) layer of silty mud from another layer of white calcite (specimen UEA810527-2) deposited on top, in turn overlain by an indurated layer of sandy material containing quartzite pebbles and gravel clasts. Samples taken from each of these specimens were newly dated as part of the present study.

Methods and Results

All speleothems were carefully inspected for recrystallization, clay or other dirt inclusions, and cleaned by ultrasonication in distilled water for 2–3 minutes before subsamples were either milled or cut with a wire saw for U/Th dating. Powder samples were taken from speleothems UEA950905-6, UEA810527-1, UEA810527-2, UEA810527-3, UEA810529-5, TJL020817-3, and TJL20080901 (Fig.2) using a Sherline micromill at Northumbria University. All other dating samples were cut from the speleothems using a wire saw with a 0.3mm diameter diamond-studded steel wire at Northumbria University. These 19 dating samples were analysed at the radiometric dating laboratory at the University of Oxford using a multi-collector inductively-coupled plasma mass spectrometer (MC-ICP-MS) following the method outlined in Vaks *et al*. (2013). Some of the results have already been presented in Lawson *et al*. (2023) but are included here to provide a comprehensive overview of newly available MC-ICP-MS dates from Assynt caves. Results are presented in Table 1 and in Figure 3. The samples are characterized by generally relatively low U concentrations (0.06–1.3 ppm). Relative age uncertainties range from 0.5% to 47% of the absolute age, depending upon age and on the concentrations of uranium and detrital thorium (Table 1).

Cave name	Sample name	previously dated?	Chem name	Sample mass (g)	^{232}Th (ppb)	^{238}U (ppb)	232/230 (atomic) *	uncorr. Age (yrs)	95% Error (yrs)	% Error	corr. age (yrs before analysis in 2022)	corr. years BP (1950 CE)	95% Error (yr)	% Error
Rana Hole	TJL 20080901_U3	N	hoca4	0.20974	2.0	270	441	121851	767	0.6	121682	121610	781	0.6
Rana Hole	TJL 20080901_U4	N	hoca5	0.13633	1.4	512	173	123717	823	0.7	123651	123579	820	0.7
Rana Hole	TJL20080901_U5	N	hoc_a11	0.2557	2.2	502	265	124270	840	0.7	124166	124094	847	0.7
Rana Hole	TJL20080901_U6	N	hoc_a12	0.2788	1.5	453	200	125362	901	0.7	125285	125213	905	0.7
Rana Hole	TJL20080901_U7	N	hoc_a13	0.2442	0.4	244	98	120338	975	0.8	120301	120229	973	0.8
Rana Hole	TJL20080901_U8	N	hoc_a14	0.2258	8.5	547	908	126398	784	0.6	126049	125977	867	0.7
Rana Hole	TJL20080901_U9	N	hoc_a15	0.2495	1.9	479	235	125386	809	0.6	125294	125222	818	0.7
Firehose Cave	SU-1-80_U1	Y **	hoca2	0.38309	2.2	135	60156	1430	43	3.0	954	882	416	43.7
Firehose Cave	SU-1-80_U2	Y **	hoca3	0.20481	5.5	114	46598	5440	81	1.5	4051	3979	1204	29.7
Firehose Cave	SU-1-8P0_U3	Y **	hoc_a16	0.3073	12.3	137	42847	11323	126	1.1	8730	8658	2263	25.9
Firehose Cave	SU-1-80_U4	Y **	hoc_a17	0.2467	11.4	163	39879	10038	124	1.2	7898	7826	1872	23.7
Firehose Cave	UEA 810529-5_U-B	N	hoca1	0.25927	1.5	133	10068	6255	78	1.3	5914	5842	313	5.3
Uamh Mhor	UEA_810527-4(I)_U1	N	hoc_a18	0.2941	0.4	583	599	6077	39	0.6	6057	5985	44	0.7
Uamh Mhor	UEA-810527-4(II)_U1	N	hoc_a19	0.3123	1.7	707	4448	3013	23	0.8	2938	2866	70	2.4
Uamh Mhor	UEA_810527-1_U1	N	hoc_a21	0.1775	1.7	1053	225	53430	278	0.5	53377	53305	282	0.5
Uamh Mhor	UEA_810527-2_U1	N	hoc_a23	0.2226	2.9	1288	366	46546	211	0.5	46469	46397	225	0.5
Uamh Mhor	UEA_810527-3_U1	N	hoc_a22	0.2990	2.1	249	9220	5156	53	1.0	4897	4825	235	4.8
Uamh an Uisge	TJL_020817-3_U1	N	hoc_a25	0.2344	0.9	1145	912	3469	21	0.6	3452	3380	27	0.8
ANUSC	UEA950905-6_U1	Y **	hoc_a24	0.1825	1.0	60	11598	7985	251	3.1	7486	7414	527	7.0

***Table 1**: U-series dates, obtained using MC-ICP mass spectrometry, from Assynt speleothems.*
* ^{230}Th/^{232}Th atomic ratio of 5.38E^{-6} (+5.38E^{-6}, −4.38E^{-6}) used to correct samples for initial thorium.
** see Table 1 in Lawson *et al*. (2023) for details.
ANUSC = Allt nan Uamh Stream Cave.

The ages range from 126 ka BP to 0.88 ka BP, placing most samples into the Holocene (MIS 1), two into the middle part of the last glacial (MIS 3), and one flowstone into the last interglacial (Ipswichian, MIS 5e) (Fig.3). Interestingly, the flowstone samples from Uamh Mhor formed in two interstadial periods during the last glacial (Table 1). The speleothems from Firehose Cave and ANUSC that had previously been analysed using AS and were thought to date from the last glacial (Lawson and Atkinson, 1995) are now re-dated to the Holocene (see also Lawson *et al*., 2023).

Discussion

In mid- to high-latitude and high-altitude regions, speleothems can generally form only during non-glacial and/or non-continuous permafrost periods (Atkinson *et al*., 1978; Vaks *et al*., 2020), i.e. at times when water is sufficiently aggressive to dissolve carbonate and can actively infiltrate the epikarst and reach the caves. By dating specific speleothem carbonate layers, we can establish the times when continuous permafrost or surface ice were absent and sufficient CO_2 was available to form carbonic acid above the caves wherein these speleothems formed.

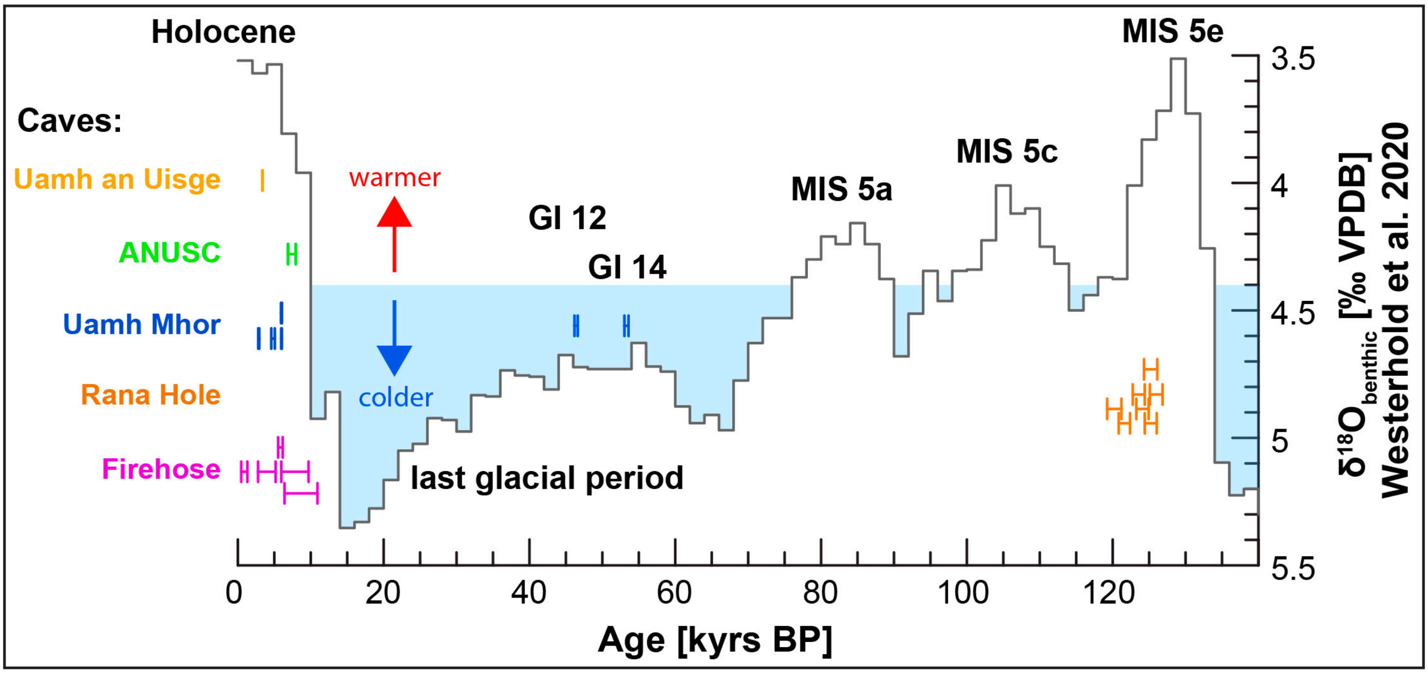

***Figure 3:** Temporal distribution of the new dates obtained on speleothems from Assynt caves in comparison to the benthic $\delta^{18}O$ CENOGRID stack record (Westerhold* et al.*, 2020). The interglacial period (MIS 5e) began c. 126 ka BP and lasted until c. 119 ka BP.*

The new dates from the five caves thus begin to give us fresh insights into the environmental past of this region. Firstly, speleothem deposition in Assynt was certainly more prevalent in the warmer and wetter times of the Ipswichian (the last interglacial) and the Holocene (MIS 1) (Fig.3). The oldest sample is TJL20080901, from Rana Hole, which started to form sometime before 126 ka BP. It should be noted that this flowstone had begun to grow even earlier, because this sample was found broken and does not contain the base of this deposit. While carbonate deposition in Rana Hole continued until c. 120 ka BP, petrographical observations on this sample (crystal appearance, growth rate, and colour) suggest that environmental conditions changed towards the late interglacial period MIS 5e (which lasted until c. 119 ka BP, Rasmussen *et al*., 2014).

Secondly, we find the next speleothem growth periods in our sample-set within the last glacial period. The two flowstone samples from Uamh Mhor were deposited c. 53.3 ka BP and c. 46.4 ka BP. In each case, the depositional period was probably only a few hundred years, although this needs to be confirmed by additional dating. These growth periods align with Greenland interstadials GI 14 (54.2–49.6 ka BP) and GI 12 (46.9–44.4 ka BP) (Rasmussen *et al*., 2014; Lawson *et al*., 2023) and indicate:

i) the absence of an ice sheet above Uamh Mhor;
ii) absence of continuous permafrost around Uamh Mhor, because otherwise water would not be available for the delivery of dissolved inorganic carbon required for the deposition of speleothems, and
iii) the presence of a cave ceiling above this now-unroofed chamber. In addition, the exposure at the original sampling site indicates that these flowstones covered a thin layer of fine-grained (clay-silt) cave sediment, which was likely deposited in a relatively low-energy environment of ponded water in the cave, conceivably due to flooding of the cave passage beneath a glacier.

The new dates place all other speleothems from our sample set firmly in the Holocene. This contrasts with the AS dating of several of these samples, which placed them in the last glacial, and highlights the importance of reassessment of archive material with state-of-the-art methods. However, even with the latest methods, several of our dates are characterized by large uncertainties due to high concentrations of detrital thorium. This issue of contamination is highlighted in stalactite SU1-80 (Fig.2), whose two layers were originally dated to 38 ± 6 and 26 ± 3 ka BP, i.e. during the last glacial (Lawson *et al*., 2023). Although the new MC-ICP-MS dates now place this sample in the Holocene, the age uncertainties caused by the presence of detrital thorium remain too large to allow high-resolution palaeoclimatic proxy work on this sample.

Conclusions

We obtained 19 new U-series dates from ten different speleothem samples. Our results indicate speleothem growth occurred not only during the Holocene, but also during two interstadials in the Devensian glacial and in the Ipswichian interglacial. The distribution and quality of the U-series dates indicate excellent potential for these and other archived speleothems to refine the regional dynamics of the northwestern sector of the British–Irish Ice Sheet and to reconstruct interglacial and interstadial climate conditions. Extending this approach to other karst areas, and utilizing archived specimens where possible, will be essential for understanding the history of ice advance and retreat in Scotland. Explicit temporal constraints on the presence of ice sheets in Assynt, and in Scotland as a whole, can emerge only once a larger set of precise and accurate dates has been assembled.

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