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Oxygen Isotope Dendrochronology of Oak Timbers

Neil J Loader, Darren Davies, Danny McCarroll, Daniel Miles, Cathy Tyers, and Giles HF Young

Discovery, Innovation and Science in the Historic Environment

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SUMMARY
As part of an initiative to investigate the practical extent of the south-central England chronology for isotopic dating, two timbers that had been securely dated by ring-width dendrochronology from Dacre Hall were sampled for oxygen isotope analysis. Sixty-five measurements were obtained on latewood from single growth-rings of core LCP-A17 from main floor beam 1 in the first-floor frame (rings 2–66 of the measured ring-width series which spans AD 1369–1491) and 62 measurements were obtained on latewood from single growth-rings of core LCP-A25 from joist 7, bay 4, in the first-floor frame (rings 0–61 of the measured ring-width series which spans AD 1364–1481).

The two isotopic series cross-match when offset by 10 years. This is consistent with the ring-width cross-matching. The 72-year isotopic mean cross-dates tentatively (t =4.69, 1/p=174, IF=20) with the south-central England oxygen isotope master chronology, at a position that is compatible with that provided by ring-width dendrochronology. The two isotopic series independently do not date.

The location of Dacre Hall, well to the north of the south-central England master chronology is currently challenging for oxygen isotope dendrochronology. This initial study suggests that, at present, secure dating may not be routinely obtained from short series of isotopic measurements on single timbers in this region. An oxygen isotope master chronology for northern England is likely required to enhance the potential for applying oxygen isotope dendrochronology in this region.

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CONTENTS

INTRODUCTION

In 2018 Historic England and Swansea University established a collaborative research initiative to explore the applicability of oxygen isotope dendrochronology in England, with a view to transferring this method into professional practice. This project investigates, firstly, the geographic limits and practical boundaries of the present reference chronology (Loader *et al* 2019) which relates closely to the region covered by the England and Wales Precipitation record (Young *et al* 2015). A second objective was to explore whether shorter ring sequences that commonly remain undated by traditional dendrochronology can be dated by the isotopic approach.

Six buildings were selected for study, each located in areas well beyond the periphery of the south-central England chronology (Cumbria, Northumbria, the Vale of York, Cornwall, Kent, and East Anglia). These buildings were chosen specifically for this study because the site master chronologies obtained through ring-width cross-dating exhibited strong correlations with local ring-width reference chronologies rather than with ring-width reference chronologies across a broader region. To provide a degree of replication, bearing in mind the locations of the selected sites, two ring-width dated samples were selected from each building. This also provided the opportunity to explore the cross-matching of isotope series. The other five buildings included in this study are individually reported in the Historic England Research Report Series (Loader *et al* 2021a–e).

Selection of individual core samples from each building was guided by strong ring-width intra-site cross-matching combined with the aim of obtaining a mean isotope record of a minimum of *c* 80 rings. The selected core samples were typical of those routinely retrieved through dendrochronological sampling in the selected locations with some providing practical challenges in relation to the presence of potential contaminants (glue, ink, charring) and narrow growth rings with little or no latewood. All timbers were provided to the isotope laboratory “blind” without information on the site location or calendar age of the samples.

Dacre Hall

Dacre Hall, which is Grade I listed (List Entry Number 1087500), is the principal above-ground remnants of the late twelfth-century cloistral buildings of the Augustinian Priory at Lanercost, Cumbria (Fig 1).

Ring-width dendrochronology has been undertaken on 56 timbers from five different areas of the Hall (Arnold *et al* 2004a). Cross-matching between 52 of these ring series produced five site chronologies, two of which could be dated. LCPASQ01, which includes 29 samples and is 155-rings in length, spans AD 1350–1504, and LCPASQ02, which includes six samples and is 280-rings in length, spans AD 977–1256.

STABLE OXYGEN ISOTOPE DENDROCHRONOLOGY

Sample selection

Two core samples from LCPASQ01 (Table 1; Fig 2) were selected for inclusion in this study (LCP-A17 and LCP-A25) because the individual ring-width series that are combined to form this chronology strongly cross-match by ring-width dendrochronology (see Arnold *et al* 2004a), and the chronology itself has the highest level of similarity with other sites across northern England (Table 2). These samples, therefore, have typical growth characteristics of timber used in historic buildings in this region. Although these samples had a sufficient number of rings for this study, both showed a decline in ring width (generally <1mm) in the outermost sections as the parent trees matured (Fig 2). This meant that insufficient latewood was available for measurement from the outer parts of these samples.

Method

Oxygen isotope dendrochronology relies upon the same fundamental principles, limitations and assumptions as conventional (ring-width-based) dendrochronology. However, rather than using ring-width measurements it uses the ratio of heavy to light oxygen (McCarroll and Loader 2004) in the late-wood cellulose ($\delta^{18}O$). The isotopes can have a higher signal to noise ratio than ring-width measurements and strong signals do not require the trees to be growing under any environmental stress (Young *et al* 2015).

The method relies on a regional master chronology (Loader *et al* 2019) constructed using ring-width dendrochronologically-dated oak timbers sourced from across a *c* 45,200km^2 (20,000 mile^2) region, centred on Oxfordshire, in south-central England. The chronology was developed as part of a Leverhulme Trust funded project (RPG-2014-327) and currently covers a period from AD 1200–2000 with replication (sample depth) of 10 trees throughout the chronology.

Ring sampling

A thin slice (4mm) is removed from the base of the cores selected for isotopic analysis. This initial sub-sampling ensures that the original measured surface from which the reported ring-width measurements were derived is physically preserved and archived for future scientific analysis, as is the case for all samples obtained during Historic England funded investigations on historic buildings.

Several physiological studies of oak trees have shown that the earlywood is partially formed from carbohydrates fixed in previous years (Richardson *et al* 2013; McCarroll *et al* 2017). To avoid this chemical carry-over effect in oak, only the latewood of each tree-ring is prepared for chemical analysis and dating. Each annual latewood increment is carefully removed as thin slivers (*c* 40μm thick) using a scalpel and dissecting microscope. Where tree rings are indistinct, physically degraded, contaminated, or comprise only earlywood then these rings are not sampled for isotopic analysis. Consequently, the isotope sequence used for dating may not provide an isotope measurement for each ring that forms part of the measured ring-width series.

Laboratory methods

Latewood samples are converted to α-cellulose using an acidified sodium chlorite solution with removal of hemicelluloses by sodium hydroxide (after Loader *et al* 1997). Samples are homogenised using an ultrasonic probe and vacuum-dried at −50˚C for 48 hours. 0.30–0.35mg of dry α-cellulose is weighed into individual silver foil capsules for pyrolysis to carbon monoxide (CO) at 1400ºC (Woodley *et al* 2012). The resulting carbon monoxide is analysed using a Delta V isotope-ratio mass spectrometer. Data are expressed as per mille (‰) deviations relative to the Vienna Standard Mean Ocean Water (VSMOW) international standard. Analytical precision is typically 0.30‰ (σ_{n-1}, n=10) (Loader *et al* 2015). The master chronology was prepared as two independent pools of five trees to ensure quality control and the resulting data combined to form the ten-tree master chronology. Individual samples for dating are prepared and analysed separately, using identical preparation protocols. The resulting stable isotopic data are presented as chronologies (time series).

Statistical analysis and dating

Tree-ring oxygen isotope data have statistical properties that are quite different from ring-widths, requiring different pre-treatment. The Baillie-Pilcher filter that works well for ring width dating (Baillie and Pilcher 1973) is not appropriate for isotope data and would result in unrealistically high *t*-values (Loader *et al* 2019). Thus the isotope data are filtered using a simple nine-year rectangular filter, with indices derived by subtraction. Degrees of freedom are corrected for autocorrelation and filtering resulting in *t*-values that conform to a Student's *t*-distribution and can be used to calculate one-tail probabilities of error. The probabilities are corrected for multiple testing by division by the number of possible matches against the master chronology (a 'Bonferroni' correction) (Dunn 1959; 1961). The ratio of probabilities for the first and second highest *t*-values provides an 'isolation factor'. All *t*-values pertaining to isotope data in this report are Student's *t*-values, not 'Baillie-Pilcher'*t*-values.

Loader *et al* (2019) have suggested that potential stable isotope dates should only be considered for acceptance when the corrected probability of error (1/p) is less than one in a hundred and the probability for the best match is more than an order of magnitude less likely to be in error than the next best match (i.e. the isolation factor > 10). The level for these thresholds necessary for isotopic dating to have equivalent security to traditional ring-width dendrochronology is currently uncertain. As the aim of this study is to explore the spatial extent of the south-central England chronology using dendrochronologically-dated samples of known age, dating results are reported irrespective of whether or not they pass these thresholds to enable an assessment of dating performance across the study region.

Cross-matching between individual isotope series is achieved using the same approach, with the number of possible matches determined by setting a minimum size of overlap. Student's *t*-values, corrected one-tail probabilities, and the isolation factor are reported as well as the highest correlation coefficient, offset in ring number from the most recent ring measured for oxygen isotope analysis, and the number of overlapping isotopic measurements. When cross-dating individual series, a Student's *t* value of 3.5 is currently used as a working indication of match position to inform chronology development, although it is again currently unclear what this threshold should be to be of equivalent security to the Baillie-Pilcher *t*-value threshold of 3.5 commonly used for cross-matching oak series in ring-width dendrochronology in England.

In isotope dendrochronology it is not always necessary or possible to measure isotopically each tree-ring, in which case the last ring measured isotopically must be placed within the context of the entire sequence of tree-rings present in the sample. This may require addition of years present in the sample, but not measured isotopically. Once a date for the last ring has been calculated, a felling date, or felling date range based on an appropriate sapwood estimate, may be assigned using identical methods to those in ring-width dendrochronology (English Heritage 1998).

Results

Sample LCP-A17 comprises 123 ring-width and 65 isotope measurements (rings 2–66 of the ring-width series; Table 1) and LCP-A25 comprises 118 ring-width and 62 isotope measurements (rings 0–61 of the ring-width series; Table 1). In both instances the isotope measurements were only carried out on the wider growth rings forming the inner sections of the cores, as the outermost rings were too narrow and friable to precisely excise sufficient latewood for isotopic analysis (Fig 2). The innermost ring of the ring-width series for LCP-A17 (ring 1) was excluded from analysis as its ring boundary was indistinct in this sample, being close to the pith. In sample LCP-A25 the innermost ring provided sufficient latewood for it to be included for isotope measurement (ring 0), but as a partial ring it was not included in the ring-width measurement series. The oxygen isotope data from both cores are provided in the Appendix.

The isotopic series from LCP-A25 cross-matches with a relative offset of -10 years against the last (most recent) isotopic measurement from LCP-A17 (Table 3; Fig 3), which is consistent with the relative position of these samples produced by the ring-width dendrochronology (Arnold *et al* 2004a, fig 9). The Student's *t*-value (4.79) between the isotopic series is much higher than the Baillie-Pilcher *t*-value (1.93) between the ring-width series for these samples.

A 72-year mean isotope series was compiled at this offset of -10 years between LCP-A25 and LCP-A17which, when compared against the south-central England oxygen isotope master chronology, produced the strongest cross-matching where the last ring of the mean isotopic series dates to AD 1434 (Table 4; Fig 4). This suggests that the last ring on which isotopic measurements could be obtained from LCP-A17 (ring 66) dates to AD 1434, and that the last ring on which isotopic measurements could be obtained from LCP-A25 (ring 61) dates to AD 1424. This is compatible with the cross-dating for these timbers suggested by ring-width dendrochronology (Table 2; Arnold *et al* 2004a), which indicates dates of AD 1369-1491 for the 123-year ring-width series from LCP-A17 and AD 1364–1481 for the 118-year ring-width series from LCP-A25.

The cross-dating of the mean isotopic series from Dacre Hall passes the thresholds for consideration as dated suggested by Loader *et al* (2019), but only marginally (Student's t=4.69, df=61, 1/p=174, IF=20). In this test case, there is independent ring-width dendrochronology available which supports the date suggested by the oxygen isotope dendrochronology. Individually the timbers did not produce cross-dating statistics that pass these thresholds for consideration.

Although dates were obtained that were in agreement with those attained using ring-width dendrochronology, the relative weakness of the dating statistics may indicate that the oxygen isotope signals recorded in these trees from the north-west of England share less in common with the south-central England signal than contemporaneous trees growing within the region from which the reference chronology originates (Loader *et al* 2019). This would suggest that development of an isotopic reference chronology developed from trees growing closer to this study site may provide a more locally-representative dating signal.

CONCLUSIONS

The two oxygen isotopic series obtained from Dacre Hall cross-match with each other (Student's t-value of 4.79) with an offset consistent with the ring-width analyses performed previously (Arnold *et al* 2004a, table 1 and figure 9). Combination of these two series into a 72-year mean record dates against the south-central England oxygen isotope master chronology, marginally passing thresholds for consideration (Loader *et al* 2019) and returning a date of AD 1434 for the last (most recent) ring of the measured isotope series (Table 4).

This date is consistent with that obtained for the same rings using conventional ring-width dendrochronology. Dacre Hall is located well beyond the south-central England region where the master chronology was constructed. The dominant control on the latewood oxygen isotope composition of tree-rings across the UK is summer precipitation, it is therefore likely that the weaker cross-dating against the master chronology observed from the Cumbrian samples reflects, at least in part, differences in the precipitation regimes between the sample and chronology regions. This study suggests that, at present, dates are unlikely to be obtained from short series of isotopic measurements on single timbers in this region using the extant south-central England oxygen isotope master chronology (Loader *et al* 2019). An oxygen isotope master chronology for northern England and the Borders, well to the north of the England-Wales precipitation region, is likely required to enhance the potential for applying oxygen isotope dendrochronology in the north-west of England.

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TABLES

Table 1: Sample description: timber type and position, material analysed, number of complete tree rings (N), number (N_i) and range of rings for which $\delta^{18}O$ measurements were undertaken, and laboratory code. The presence of a zero ring indicates that latewood only was preserved at the pith-end of the sample, this was measured isotopically but not included in the ring-width analyses

Sample	Timber and Position	Species	N	N_i	$\delta^{18}O$ (Measured rings)	Code
LCP-A17	Main floor beam number 1 (7)	Latewood α-cellulose *Quercus* spp	123	65	2 – 66	SWAN-32a
LCP-A25	Joist 7, bay 4 (h/s)	Latewood α-cellulose *Quercus* spp	118	62	0 – 61	SWAN-32b

Key: h/s=heartwood/sapwood boundary; (7) = number of sapwood rings preserved

Table 2: Results of the cross-dating of 155-year ring-width site chronology LCPASQ01, which includes samples LCP-A17 and LCP-A25 against a selection of independent ring-width reference chronologies when the first-ring date is AD 1350 and the last-ring date is AD 1504 against the enhanced network of oak site reference chronologies now available

Reference chronology	Span of chronology	*t*-value	Reference
Unthank Hall, Stanhope, County Durham	AD 1386–1592	7.8	Howard *et al* 2001
Askerton Castle, Kirkhambeck, Cumbria	AD 1324–1493	7.8	Esling *et al* 1990
Hitchins Onset, Scaleby, Cumbria	AD 1364–1491	7.6	Howard *et al* 1997
Girlington Hall, Ovington, Co Durham	AD 1268–1504	7.6	Arnold *et al* 2019
Whalley Abbey, Whalley, Lancashire	AD 1362–1559	7.4	Arnold and Howard 2015
Moot Hall, Market Place, Hexham, Northumberland	AD 1341–1539	7.4	Arnold *et al* 2004b
Aydon Castle, Corbridge, Northumberland	AD 1406–1545	7.3	Arnold *et al* 2002
Dandra Garth, Garsdale, Cumbria	AD 1373–1635	7.2	Arnold and Howard 2014
Tunstall Hall Farm, Hartlepool, Cleveland	AD 1316–1484	7.1	Howard *et al* 2002
Headlands Hall, Liversedge, West Yorkshire	AD 1388–1487	7.1	Tyers 2001

Table 3: Cross-dating matrix for samples LCP-A25 and LCP-A17 identifying number of rings [N_i] for which $\delta^{18}O$ measurements have been undertaken. Upper right: significant Student's t*-value and position (offset; the LCP-A25 isotopic series ends 10 years before that of LCP-A17). Lower left (shaded cell): Pearson's correlation coefficient and degrees of freedom for position of best match (series compared column versus row)*

	LCP-A17 [65]	LCP-A25 [62]
LCP-A17 [65]	-	4.79 -10
LCP-A25 [62]	0.577 46	-

Table 4: Stable oxygen isotope dating of the composite and individual samples from Dacre Hall against the south-central England master chronology (Loader et al *2019) over the period AD 1200–AD 2000. Number of whole rings present in core sample (N), number of rings on which $\delta^{18}O$ measurements were undertaken (N_i), Pearson's correlation coefficient (r), degrees of freedom (adjusted for autocorrelation and multiple sampling), Student's* t*-value, probability (1/p), isolation factor (IF), and date.*

Sample	Description	N	N_i	r	df	*t*	1/p	IF	Date
LCP-x	Mean of LCP-17 & LCP-25	123	72	0.515	61	4.69	174	20	AD 1434
LCP-A17	Main floor beam number 1 (7)	123	65	0.479	55	4.05	17	2	FAIL
LCP-A25	Joist 7, bay 4 (h/s)	118	62	0.455	51	3.65	4	6	FAIL

FIGURES

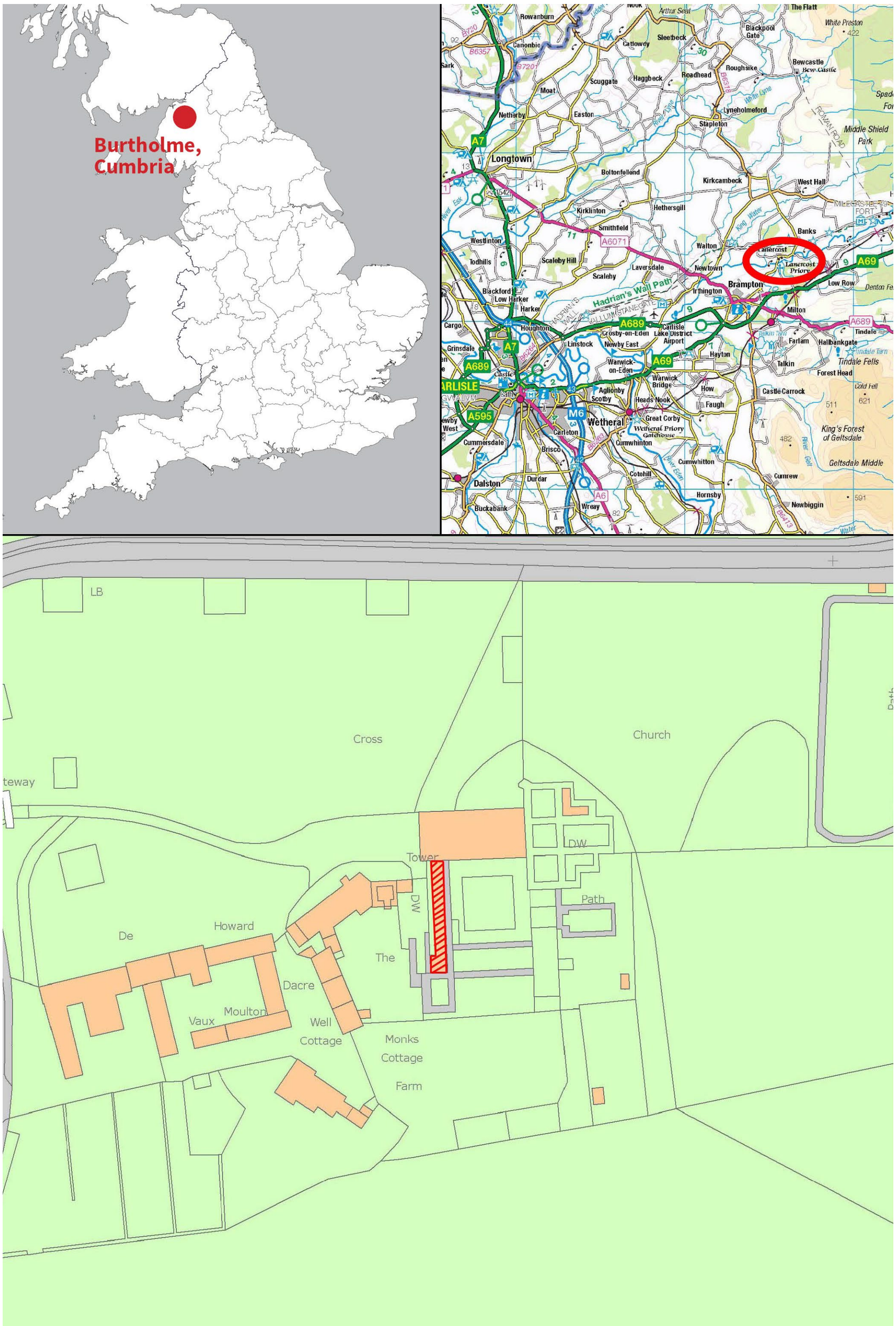


Figure 1: Maps to show the location of Dacre Hall, Burtholme in Cumbria, marked in red. Scale: top right 1:300000; bottom 1:1500. © Crown Copyright and database right 2020. All rights reserved. Ordnance Survey Licence number 100024900. © British Crown and SeaZone Solutions Ltd 2020. All rights reserved. Licence number 102006.006. © Historic England

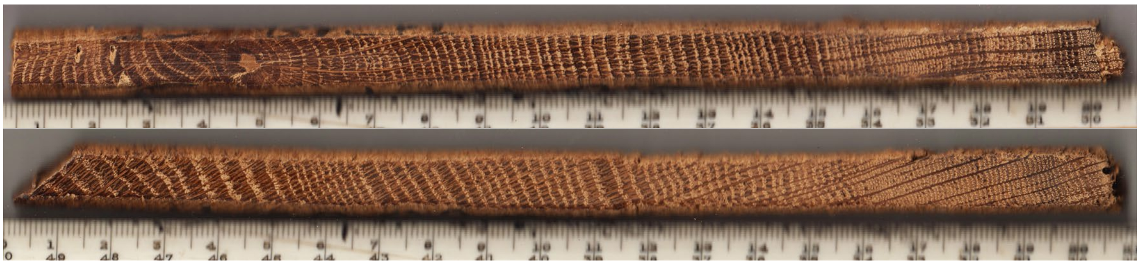


Figure 2: Samples LCP-A17 (top) and LCP-A25 (bottom) showing the reduction in growth rate in the latter sections of both of these cores as the parent trees matured

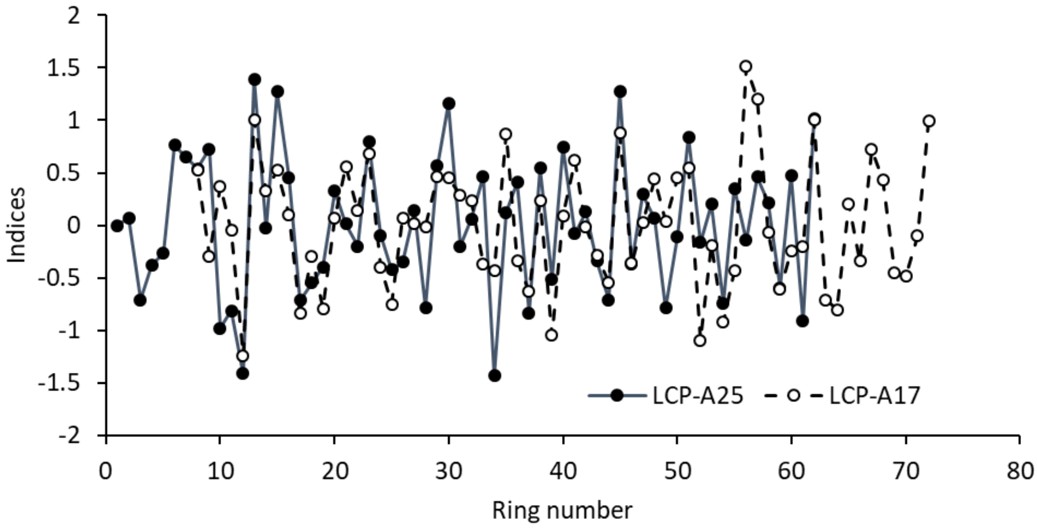


Figure 3: Time series of the filtered and indexed $\delta^{18}O$ values from the two samples plotted at the position of strongest match (Student's t-value of 4.79)

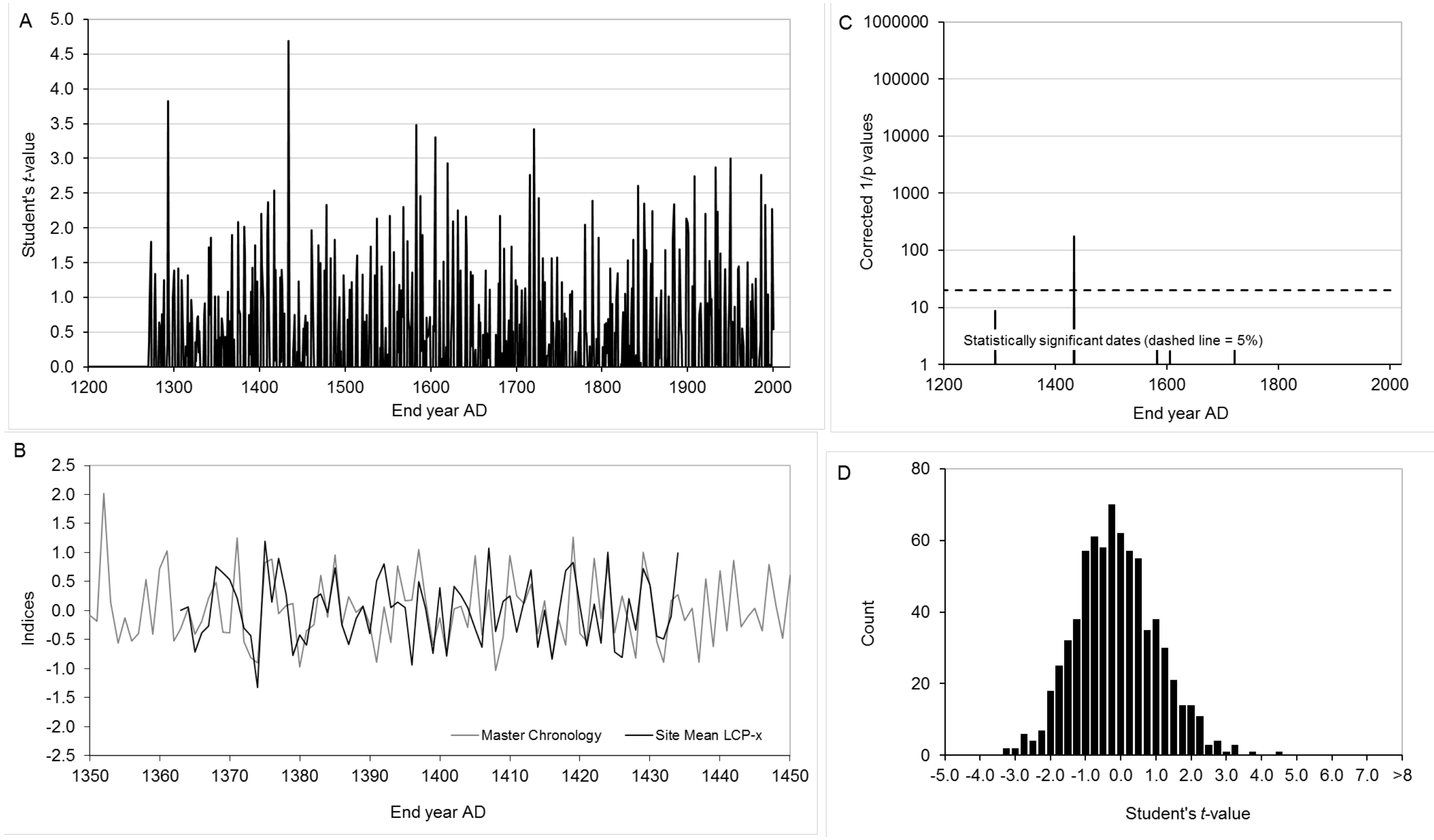


Figure 4: Dating results for the 72-year mean isotope chronology (LCP-A17 and LCP-A25). A: Student's t*-values for all possible end dates with full overlap against the master chronology. B: Time series of the site isotopic mean plotted against the master chronology. C: End dates with corrected probabilities (1/p) of more than one. Those below the dashed line (1/p = 20) are not statistically significant. D: Distribution of Student's* t*-values for all possible matches*

APPENDIX

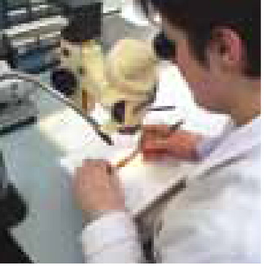
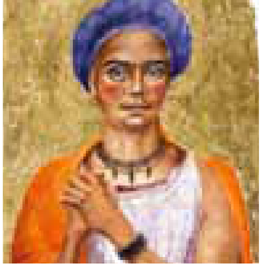
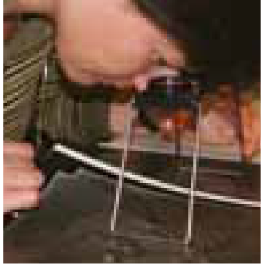
Oxygen isotope ratios ($\delta^{18}O$) for the measured tree ring series
Data are reported as per mille (‰) deviations relative to the VSMOW standard (Coplen 1995).

Sample LCP-A17

Ring	$\delta^{18}O$	Ring	$\delta^{18}O$	Ring	$\delta^{18}O$
1		31	26.86	61	28.27
2	28.33	32	27.72	62	28.18
3	27.75	33	26.44	63	27.37
4	28.43	34	27.46	64	27.26
5	27.99	35	28.17	65	27.65
6	26.80	36	27.64	66	28.73
7	28.89	37	27.42		
8	28.18	38	27.40		
9	28.21	39	28.92		
10	27.76	40	27.71		
11	26.98	41	28.21		
12	27.41	42	28.59		
13	26.96	43	28.27		
14	27.76	44	28.51		
15	28.20	45	28.62		
16	27.92	46	27.13		
17	28.55	47	28.13		
18	27.62	48	27.39		
19	27.37	49	27.77		
20	28.24	50	29.63		
21	28.26	51	29.39		
22	28.21	52	28.18		
23	28.69	53	27.60		
24	28.68	54	27.87		
25	28.55	55	27.72		
26	28.37	56	28.71		
27	27.62	57	27.00		
28	27.45	58	26.97		
29	28.50	59	27.92		
30	27.18	60	27.33		

Sample LCP-A25

Ring	δ18O	Ring	δ18O	Ring	δ18O
0	27.57				
1	27.75	31	27.98	61	28.73
2	27.10	32	28.34		
3	27.59	33	26.40		
4	27.85	34	27.76		
5	28.84	35	28.13		
6	28.71	36	26.87		
7	28.58	37	28.22		
8	28.99	38	27.30		
9	27.31	39	28.50		
10	27.55	40	27.80		
11	26.95	41	28.08		
12	29.65	42	27.62		
13	28.07	43	27.34		
14	29.43	44	29.19		
15	28.71	45	27.54		
16	27.65	46	28.29		
17	27.60	47	28.07		
18	27.83	48	27.30		
19	28.38	49	27.77		
20	27.93	50	28.80		
21	27.73	51	27.73		
22	28.79	52	28.12		
23	27.83	53	27.26		
24	27.53	54	28.29		
25	27.73	55	27.74		
26	28.22	56	28.24		
27	27.21	57	28.07		
28	28.61	58	27.20		
29	29.08	59	28.27		
30	27.73	60	26.83		



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