



Lodge Farmhouse, Middle Road, Denton,  
Norfolk

## Oxygen Isotope Dendrochronology of Oak Timbers

Neil J Loader, Darren Davis, Danny McCarroll, Daniel Miles,  
Cathy Tyers, and Giles H F 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 Lodge Farmhouse, Denton were sampled for oxygen isotope analysis. Fifty measurements were obtained on latewood from single growth-rings of core LFD-04 from a section of a west wall-plate (rings 0–49 of the measured ring-width series which spans AD 1252–1335) and 96 measurements were obtained on latewood from single growth-rings of core LFD-12 from the northernmost east principal post (rings 1–96 of the measured ring-width series which spans AD 1225–1320).

The two isotopic series cross-match when offset by 20 years. This is consistent with the ring-width cross-matching. The 96-year isotopic mean cross-dates ( $t = 6.15$ ,  $1/p = 105264$ ,  $IF > 1000$ ) with the south-central England oxygen isotope master chronology, at a position that is compatible with that provided by ring-width dendrochronology. The isotopic series LFD-12 dates independently ( $t = 6.15$ ,  $1/p = 103843$ ,  $IF > 1000$ ), but the shorter LFD-04 does not date independently.

The location of Lodge Farmhouse in East Anglia is in an area that can be challenging for ring-width dendrochronology. It is also located well to the east of the south-central England master oxygen isotope chronology. This initial study suggests that, at present, secure dating using oxygen isotope measurements may be possible in this region, but may not routinely be obtained from short series of isotopic measurements on single timbers. An oxygen isotope master chronology for East Anglia may be required to enhance the potential for applying oxygen isotope dendrochronology in this region.

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# CONTENTS

Introduction .....	1
Lodge Farmhouse.....	1
Stable Oxygen Isotope Dendrochronology .....	2
Sample selection.....	2
Method.....	2
Ring sampling .....	2
Laboratory methods.....	3
Statistical analysis and dating .....	3
Results .....	4
Conclusions .....	5
References .....	6
Tables.....	9
Figures .....	11
Appendix .....	14
Oxygen isotope ratios ( $\delta^{18}\text{O}$ ) for the measured tree ring series. ....	14

## 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* 2020; 2021a–d).

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 age of the samples.

### Lodge Farmhouse

Lodge Farmhouse ([List Entry Number 1373629](#)), now called East Hall (Norfolk Heritage Explorer), is a mid fourteenth-century raised aisled hall (which has been part demolished), reverting to an aisled hall at one end hidden beneath a brick skin. It is located in Denton, Norfolk (Fig 1). Ring-width dendrochronology has been undertaken on 10 of the 17 sampled timbers from the ground and first floors of the structure (Groves and Hillam 1993). Cross-matching between seven of these ring series produced a site chronology which could be dated. The Lodge Farm site master chronology, which includes seven samples and is 121-rings in length, spans AD 1215–1335 (Groves and Hillam 1993).

# STABLE OXYGEN ISOTOPE DENDROCHRONOLOGY

## Sample selection

Two core samples from the Lodge Farmhouse site master chronology (Table 1) were selected for inclusion in this study (LFD-04 and LFD-12) because the individual ring-width series that are combined to form this chronology strongly cross-match by ring-width dendrochronology (Groves and Hillam 1993, table 2), and the chronology itself has the highest level of similarity with other sites across East Anglia (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, sample LFD-04 exhibited a large crack and a decline in ring width in the outermost section as the parent trees matured (Fig 2). This meant that it was not possible to prepare sufficient latewood for measurement from the outer parts of this core.

## 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 ( $\delta^{18}\text{O}$ ). 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 45,200 km<sup>2</sup> (20,000 mile<sup>2</sup>) 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  $\alpha$ -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  $-50^{\circ}\text{C}$  for 48 hours. 0.30–0.35mg of dry  $\alpha$ -cellulose is weighed into individual silver foil capsules for pyrolysis to carbon monoxide (CO) at  $1400^{\circ}\text{C}$  (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‰ ( $\sigma_{n-1}$ ,  $n=10$ ) (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, 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 (ie 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 LFD-04 comprises 84 ring-width and 50 isotope measurements (rings 0–49 of the ring-width series; Table 1) and LFD-12 comprises 96 ring-width and 96 isotope measurements (rings 1–96 of the ring-width series; Table 1). For LFD-04 the isotope measurements were only carried out on the wider growth rings forming the inner section of the core, as the outermost rings separated by a crack were too narrow and friable to precisely excise sufficient latewood for isotopic analysis (Fig 2). In sample LFD-04 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 LFD-04 cross-matches with a relative offset of –20 years against the last (most recent) isotopic measurement from LFD-12 (Table 3; Fig 3), which is consistent with the relative position of these samples produced by the ring-width dendrochronology (Groves and Hillam 1993, fig 2). The Student's  $t$ -value (7.64) between the isotopic series compares favourably with the Baillie-Pilcher  $t$ -value (5.60) between the ring-width series for these samples.

A 96-year mean isotope series was compiled at this offset which, 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 1320 (Table 4; Fig 4). This suggests that the last ring on which isotopic measurements could be obtained from LFD-04 (ring 49) dates to AD 1300, and that the last ring on which isotopic measurements could be obtained from LFD-12 (ring 96) dates to AD 1320. This is compatible with the cross-dating for these timbers indicated by ring-width dendrochronology (Table 2), which indicates dates of AD 1252–1335 for the 84 year ring-width series from LFD-04 and AD 1225–1320 for the 96 ring-width series from LFD-12.

The cross-dating of the mean isotopic series from Lodge Farmhouse passes the thresholds for consideration as dated suggested by Loader *et al* (2019) (Student's

$t=6.15$ ,  $df=82$ ,  $1/p=105264$ ,  $IF>1000$ ). In this test case, there is independent ring-width dendrochronology available which supports the date suggested by the oxygen isotope dendrochronology. Individually, timber LFD-04 did not produce cross-dating statistics that pass these thresholds for consideration, although its best match was consistent with the ring-width dating (Student's  $t=3.85$ ,  $df=42$ ,  $1/p=7$ ,  $IF=3$ ). By contrast, cross-dating of timber LFD-12 independently passes the thresholds for consideration as dated suggested by Loader *et al* (2019) at a date consistent with the ring-width dendrochronology (Student's  $t=6.15$ ,  $df=82$ ,  $1/p=103843$ ,  $IF>1000$ ).

The dates obtained by oxygen isotope dendrochronology were in agreement with those attained using ring-width dendrochronology, with the strength of the dating statistics suggesting that the oxygen isotope signals recorded in these trees from East Anglia have something in common with those from contemporaneous trees included in the south-central England reference chronology (Loader *et al* 2019). This suggests that isotopic dendrochronology using the south-central England chronology may be considered for dating timbers from this region, although the 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 isotopic series obtained from Lodge Farmhouse cross-match with each other (Student's  $t$ -value of 7.64) with an offset consistent with the ring-width analyses performed previously (Groves and Hillam 1993, table 2 and fig 2; Table 2). Combination of these two series into a 96-year mean record dates against the south central England oxygen isotope master chronology, passing thresholds for consideration as dated (Loader *et al* 2019) and returning a date of AD 1320 for the last (most recent) ring of the measured oxygen isotope series.

This date is consistent with that obtained for the same rings using conventional ring-width dendrochronology. Lodge Farmhouse is located to the east of the south-central England region where the master oxygen isotope 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 variability in dating success observed may reflect, at least in part, differences in the precipitation regimes between the samples and chronology regions. This study suggests that dates may be obtained from series of isotopic measurements in this region. An oxygen isotope master chronology for East Anglia, however, would probably enhance the potential for applying oxygen isotope dendrochronology across this region.

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## TABLES

*Table 1: Sample description: timber type and position, material analysed, number of complete tree rings (N), number (N<sub>i</sub>) and range of rings for which  $\delta^{18}\text{O}$  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 <sub>i</sub>	$\delta^{18}\text{O}$ (Measured rings)	Code
LFD-04	West wall-plate	Latewood $\alpha$ -cellulose <i>Quercus</i> spp	84	50	0–49	SWAN-36a
LFD-12	East principal post	Latewood $\alpha$ -cellulose <i>Quercus</i> spp	96	96	1–96	SWAN-36b

*Table 2: Results of the cross-dating of 120-year ring-width site chronology for Lodge Farmhouse against a selection of independent ring-width reference chronologies when the first-ring date is AD 1215 and the last-ring date is AD 1335 (against the enhanced network of oak site reference chronologies available since the original analysis (Groves and Hillam 1993, table 4))*

Reference chronology	Span of chronology	<i>t</i> -value	Reference
Oxburgh Hall, Norfolk	AD 1221–1427	10.86	Tyers 2004a
Castle Acre Priory, Norfolk	AD 1237–1356	7.75	Tyers 2000
Hales Hall, Loddon, Norfolk	AD 1236–1494	6.74	Arnold and Howard 2014
Peterborough Cathedral presbytery roof, Cambridgeshire	AD 1208–1500	6.55	Tyers 2004b
Flores House, Oakham, Rutland	AD 1173–1392	6.36	Arnold <i>et al</i> 2008
Old Vicarage, New Buckenham, Norfolk	AD 1271–1451	6.31	Tyers 2004c
Horn Farm, Salcott, Essex	AD 1230–1339	6.24	Tyers and Groves 2001
Grange Farm Barn, Ingham, Norfolk	AD 1243–1377	6.15	Bridge 2002
7 West Street, Coggeshall, Essex	AD 1222–1393	5.89	Tyers 2011
Church of St George, Toddington, Bedfordshire	AD 1226–1392	5.75	Bridge 2001

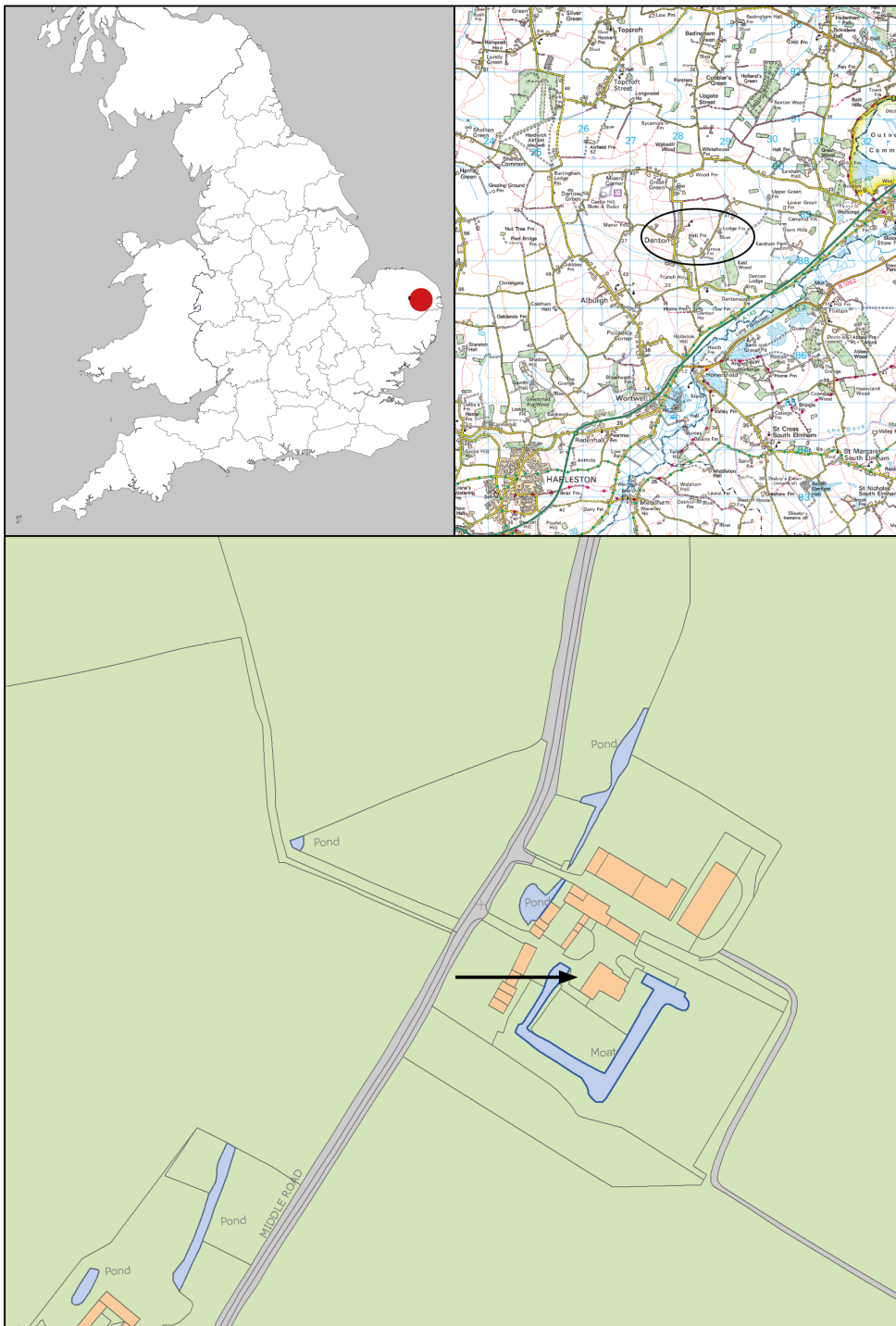
*Table 3: Cross-dating matrix for samples LFD-04 and LFD-12 identifying number of rings [ $N_i$ ] for which  $\delta^{18}O$  measurements have been undertaken. Upper right: significant Student's  $t$ -value and position (offset; the LFD-04 isotopic series ends 20 years before that of LFD-12). Lower left (shaded cell): Pearson's correlation coefficient and degrees of freedom for position of best match (series compared column versus row)*

	LFD-04 [50]	LFD-12 [96]
LFD-04 [50]	-	7.64 20
LFD-12 [96]	0.76 42	-

*Table 4: Stable oxygen isotope dating of the composite and individual samples from Lodge Farm 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 of last ring measured isotopically.*

Sample	Description	$N$	$N_i$	$r$	df	$t$	$1/p$	$IF$	Date
LFD-x	Mean of LFD-04 & LFD-12	111	96	0.562	82	6.15	105264	>1000	AD 1320
LFD-04	West wall-plate (-)	84	50	0.511	42	3.85	7	3	FAIL
LFD-12	East principal post (-)	96	96	0.562	82	6.15	103843	>1000	AD 1320

## FIGURES



*Figure 1: Maps to show the location of Denton, Norfolk (red dot), Denton (circled) and Lodge Farmhouse (arrow). Scale: top right 1:10000; bottom 1:1250. © Crown Copyright and database right 2021. All rights reserved. Ordnance Survey Licence number 100024900. © British Crown and SeaZone Solutions Ltd 2021. All rights reserved. Licence number 102006.006. © Historic England*





Figure 2: Samples LFD-04 (top) and LFD-12 (bottom) showing the reduction in growth rate in the latter sections of both of these cores

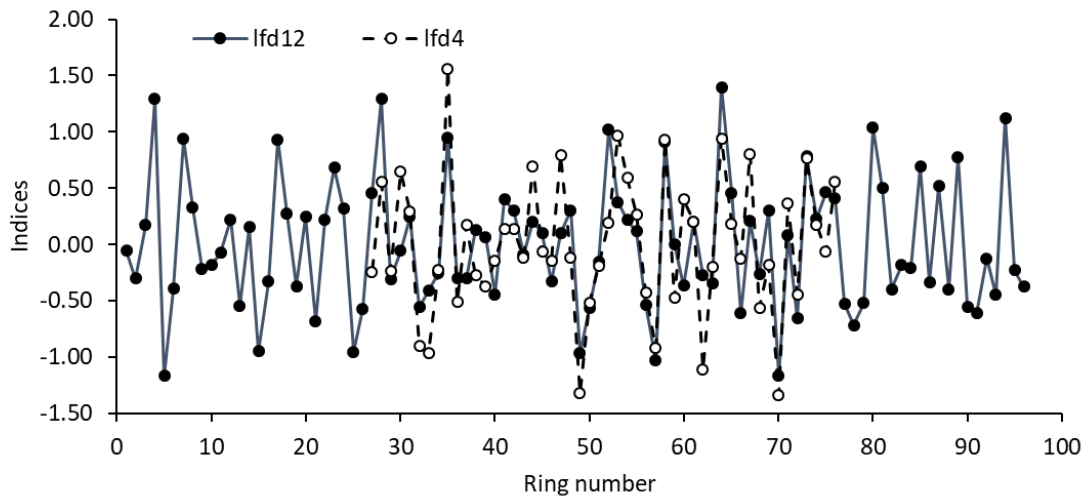


Figure 3: Time series of the filtered and indexed  $\delta^{18}O$  values from the two samples plotted at the position of strongest match (Student's t-value of 7.64)

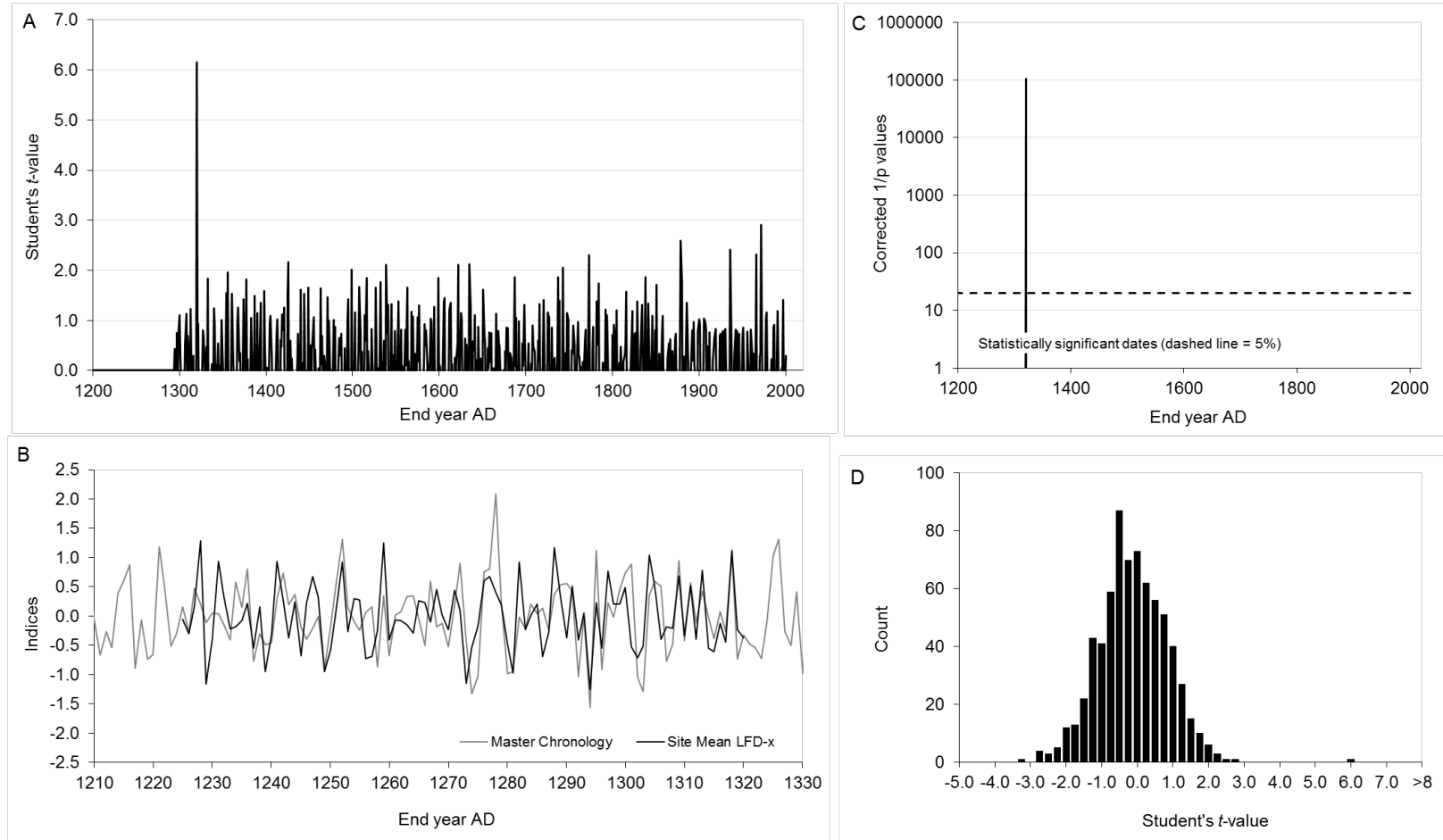


Figure 4: Dating results for the 96-year mean isotope chronology (LFD-04 and LFD-12). A: Student's  $t$ -values for all possible end dates with full overlap against the master chronology. B: Time series of the site isotopic mean plotted against the master chronology. C: End dates with corrected probabilities ( $1/p$ ) 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 ( $\delta^{18}\text{O}$ ) for the measured tree ring series.

Data are reported as per mille (‰) deviations relative to the VSMOW standard (Coplen 1995).

#### Sample LFD-04

Ring	$\delta^{18}\text{O}$	Ring	$\delta^{18}\text{O}$
0	29.03		
1	29.69	31	30.17
2	28.70	32	28.67
3	29.56	33	29.61
4	29.27	34	29.45
5	27.97	35	28.17
6	27.82	36	29.06
7	28.54	37	30.19
8	30.21	38	29.35
9	28.08	39	28.86
10	28.89	40	29.95
11	28.58	41	28.56
12	28.53	42	28.93
13	28.71	43	27.81
14	29.08	44	29.60
15	29.07	45	28.81
16	28.93	46	30.15
17	29.78	47	29.63
18	28.90	48	29.65
19	28.76	49	30.18
20	29.69		
21	28.82		
22	27.64		
23	28.56		
24	28.98		
25	29.29		
26	30.04		
27	29.94		
28	29.63		
29	29.00		
30	28.52		

Sample LFD-12

Ring	$\delta^{18}\text{O}$	Ring	$\delta^{18}\text{O}$	Ring	$\delta^{18}\text{O}$	Ring	$\delta^{18}\text{O}$
1	28.71	31	28.95	61	29.25	91	27.85
2	28.54	32	28.02	62	28.84	92	28.20
3	28.96	33	27.97	63	28.71	93	27.86
4	30.06	34	28.18	64	30.43	94	29.45
5	27.54	35	29.40	65	29.56	95	28.08
6	28.24	36	28.05	66	28.30	96	27.97
7	29.52	37	28.16	67	29.13		
8	28.81	38	28.68	68	28.60		
9	27.97	39	28.64	69	29.07		
10	28.07	40	28.06	70	27.55		
11	28.04	41	28.96	71	28.91		
12	28.13	42	28.84	72	28.20		
13	27.37	43	28.41	73	29.65		
14	28.12	44	28.71	74	29.01		
15	26.99	45	28.54	75	29.35		
16	27.68	46	27.99	76	29.42		
17	28.90	47	28.37	77	28.62		
18	28.37	48	28.70	78	28.31		
19	27.84	49	27.44	79	28.49		
20	28.67	50	27.87	80	29.99		
21	27.74	51	28.38	81	29.48		
22	28.56	52	29.54	82	28.57		
23	29.11	53	28.79	83	28.88		
24	28.98	54	28.88	84	28.83		
25	27.66	55	28.88	85	29.65		
26	28.12	56	28.24	86	28.45		
27	29.19	57	27.71	87	29.23		
28	29.91	58	29.66	88	28.24		
29	28.20	59	28.73	89	29.30		
30	28.50	60	28.54	90	27.95		



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