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# Church of Saint Mabena
# St Mabyn
# Cornwall

## Oxygen Isotope Dendrochronology of Oak Timbers

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

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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 the church of St Mabena, St Mabyn, Cornwall were sampled for oxygen isotope analysis. Sixty-three measurements were obtained on latewood from single growth-rings of core SMC-04 from the north brace of truss 9 (rings 1–63 of the measured ring-width series which spans AD 1421–1494) in the north aisle and 82 measurements were obtained on latewood from single growth-rings of core SMC-14 from the north rafter of truss 40 (rings 0–82 of the measured ring-width series which spans AD 1413–1495) in the north aisle.

The two isotopic series cross-match when offset by 11 years. This is consistent with the ring-width cross-matching. The 82-year isotopic mean cross-dates ($t$ =7.40, 1/p > 1 million, 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 two isotopic series cross-date independently at positions consistent with the ring-width dendrochronology.

The location of the church of St Mabena, well to the south-west of the south-central England master chronology is in an area, Cornwall, that can be challenging for ring-width dendrochronology. This initial study suggests that, at present, secure dating may be obtained from series of isotopic measurements on single or multiple timbers in this region. An oxygen isotope master chronology for south-western England may further 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* 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.

## The Church of St Mabena, St Mabyn

The church of St Mabena lies in the historic core of the settlement of St Mabyn, Cornwall (Fig 1). The existing church is listed at Grade I (List Entry Number 1161735), and the surviving fabric is thought to date mainly from the later fifteenth century. The church has wagon roofs in the nave, chancel, north and south aisles, and porch with carved wall plates and carved bosses (Pevsner and Beacham 2014, 575–6).

Ring-width dendrochronology was undertaken on core samples from 23 timbers from the north aisle, nave, and porch roofs in association with repair works in the 2000s (Tyers 2008). Cross-matching between 15 of these ring-width series produced two site chronologies: StMabyn1, which included six samples, four from the nave roof and two from the porch (Tyers 2008, table 3), and StMabyn2, which included nine samples from the roof of the north aisle (Tyers 2008, table 2). Both chronologies could be dated by ring-width dendrochronology: StMabyn1, which is

108-rings in length, spans AD 1370–1477, and StMabyn2, which is 95-rings in length, spans AD 1409–1503 (Tyers 2008, table 4).

## STABLE OXYGEN ISOTOPE DENDROCHRONOLOGY

### Sample selection

Two core samples from StMabyn2 were selected for inclusion in this study (SMC-04 and SMC-14; Table 1) because the individual ring-width series that are combined to form this chronology strongly cross-match by ring-width dendrochronology (Tyers 2008, table 2), and the chronology itself has the highest level of similarity with other sites across south-western 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 < 1 mm), or degradation, in the outermost sections as the parent trees matured (Fig 2). Nevertheless, it was possible to identify sufficient latewood for most of both of these ring series.

### 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* 45,200 km$^2$ (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‰ ($\sigma_{n-1}$, 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 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 (ie the isolation factor >10). The level for these thresholds necessary for isotopic dating to have equivalent confidence 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 SMC-04 comprises 74 ring-width and 63 isotope measurements (rings 1–63 of the ring-width series; Table 1) and SMC-14 comprises 83 ring-width and 82 isotope measurements (rings 0–82 of the ring-width series; Table 1). Sufficient latewood was available to enable both cores to be sub-sampled for isotopic analysis, although the outermost rings of SMC-04 (ring-width series rings 64 to 74) were too narrow or friable to precisely excise sufficient latewood for isotopic analysis, as were ring-width series rings 81 and 83 of SMC-14 (Fig 2). The innermost ring of sample SMC-14 provided sufficient latewood for an 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 SMC-04 cross-matches with a relative offset of −11 years against the last (most recent) isotopic measurement from SMC-14 (Table 3; Fig 3), which is consistent with the relative position of these samples produced by the ring-width dendrochronology (Tyers 2008, fig 4). The Student's *t*-value (14.09) between the isotopic series compares favourably with the Baillie-Pilcher *t*-value (7.75) between the ring-width series for these samples.

An 82-year mean isotope series was compiled at the offset of −11 years between SMC-04 and SMC-14 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 1494 (Table 4; Fig 4). This suggests that the last ring on which isotopic measurements could be obtained from SMC-04 (ring 63) dates to AD 1483, and that the last ring on which isotopic measurements could be obtained from SMC-14 (ring 82) dates to AD 1494. This is compatible with the cross-dating for these timbers indicated by ring-width dendrochronology (Table 2; Tyers 2008) which indicates dates of AD 1421–1494 for the 74-year ring-width series from SMC-04 and AD 1413–1495 for the 83-year ring-width series from SMC-14.

The cross-dating of the mean isotopic series from the church of St Mabena passes the thresholds for consideration as dated suggested by Loader *et al* (2019), (Student's $t$=7.40, df=68, 1/p > 1 million, IF > 1000). In this test case, there is independent ring-width dendrochronology available which supports the oxygen isotope date. Individually both the timbers produce cross-dating statistics that pass these thresholds for consideration (SMC-04: Student's $t$=6.70, df=52, 1/p=181,726, IF > 1000; SMC-14: Student's $t$=7.02, df=68, 1/p > 1 million, 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 indicating that the oxygen isotope signals recorded in these trees from the south-west of England share much in common with the south-central England signal as contemporaneous trees growing within the region from which the reference chronology originates (Loader *et al* 2019). This would suggest 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 oxygen isotope series obtained from the church of St Mabena cross-match with each other (Student's $t$-value of 14.09) with an offset consistent with the ring-width analyses performed previously (Tyers 2008, tables 1 and 2). Combination of these two series into an 82-year mean record dates strongly (Student's $t$-value of 7.40) against the south-central England oxygen isotope master chronology (Loader *et al* 2019), returning a date of AD 1494 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. The church of St Mabena, St Mabyn, Cornwall 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 isotopic composition of precipitation in south-western England shares many of the changes in precipitation regime recorded in the south-central England master chronology. This study suggests that dates are likely to be obtained from series of isotopic measurements on timbers in this region, although further investigation of other timbers in this region is clearly appropriate. The development of a more area-specific chronology may provide further potential for stronger, more localised dating, particularly with respect to short ring series that prove problematic for conventional ring-width dendrochronology.

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# TABLES

*Table 1: Sample description: timber type and position, material analysed, number of complete tree rings (N), number (Ni) 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 | Material | N | $N_i$ | $\delta^{18}O$ (Measured rings) | Laboratory code |
|---|---|---|---|---|---|---|
| SMC-04 | North aisle: north brace, truss 9 (h/s) | Latewood α-cellulose *Quercus* spp | 74 | 63 | 1–63 | SWAN-34a |
| SMC-14 | North aisle: north rafter, truss 40 (h/s) | Latewood α-cellulose *Quercus* spp | 83 | 82 | 0–82 (81 missing) | SWAN-34b |

Key: h/s=heartwood/sapwood boundary

*Table 2: Results of the cross-dating of 95-year ring-width site chronology StMabyn2, which includes samples SMC-04 and SMC-14, against a selection of independent ring-width reference chronologies when the first-ring date is AD 1409 and the last-ring date is AD 1503 against the enhanced network of oak site reference chronologies now available*

| Reference chronology | Span of chronology | *t*-value | Reference |
|---|---|---|---|
| St Martin's church, East Looe, Cornwall | AD 1363–1518 | 8.58 | Arnold *et al* 2006a |
| Holy Cross church, Crediton, Devon | AD 1317–1536 | 7.28 | Tyers 2004a |
| St Andrew's church, Alwington, Devon | AD 1342–1490 | 7.17 | Arnold and Howard 2009 |
| Pendennis Castle, nr Falmouth, Cornwall | AD 1358–1541 | 7.03 | Tyers 2004b |
| Church of St Idlierna, Lansallos, Cornwall | AD 1355–1514 | 7.00 | Arnold and Howard 2006 |
| Dublin region | AD 1357–1556 | 6.79 | Baillie 1977 |
| Castle of Park, Dumfries and Galloway, Scotland | AD 1350–1551 | 6.74 | Queens University Belfast unpubl |
| Warleigh House, Tamerton Foliot, Devon | AD 1367–1539 | 6.70 | Arnold *et al* 2006b |
| The Gildhouse, Poundstock, Cornwall | AD 1405–1543 | 6.42 | Arnold and Howard 2007 |
| Church of St Barnabas, Brampton Bryan, Herefordshire | AD 1233–1622 | 6.32 | Arnold *et al* 2021 |

*Table 3: Cross-dating matrix for samples SMC-04 and SMC-14 identifying number of rings [Ni] for which δ¹⁸O measurements have been undertaken. Upper right: significant Student's t-value and position (offset; the SMC-04 isotopic series ends 11 years before that of SMC-14). Lower left (shaded cell): Pearson's correlation coefficient and degrees of freedom for position of best match (series compared column versus row)*

| | SMC-04 [63] | SMC-14 [82] |
|---|---|---|
| SMC-04 [63] | - | 14.09 −11 |
| SMC-14 [82] | 0.890 50 | - |

*Table 4: Stable oxygen isotope dating of the composite and individual samples from the church of St Mabena, St Mabyn, Cornwall, 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 (Ni), 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 |
|---|---|---|---|---|---|---|---|---|---|
| SMC-x | Mean of SMC-04 & SMC-14 | 83 | 82 | 0.668 | 68 | 7.40 | > 1 million | >1000 | AD 1494 |
| SMC-04 | Aisle T9 N brace | 71 | 63 | 0.681 | 52 | 6.70 | 181,726 | >1000 | AD 1483 |
| SMC-14 | Aisle T40 N rafter | 83 | 82 | 0.648 | 68 | 7.02 | > 1 million | >1000 | AD 1494 |

FIGURES

*Figure 1: Maps to show the location of the church of St Mabena, St Mabyn, Cornwall, circled. Scale: top right 1:30000; bottom 1:1500. © Crown Copyright and database right 2022. All rights reserved. Ordnance Survey Licence number 100024900. © British Crown and SeaZone Solutions Ltd 2022. All rights reserved. Licence number 102006.006. © Historic England.*

*Figure 2: Samples SMC-04 (top) and SMC-14 (bottom) showing the reduction in growth rate in the latter sections of both of these cores as the parent trees matured. Note: core diameter is 12mm.*

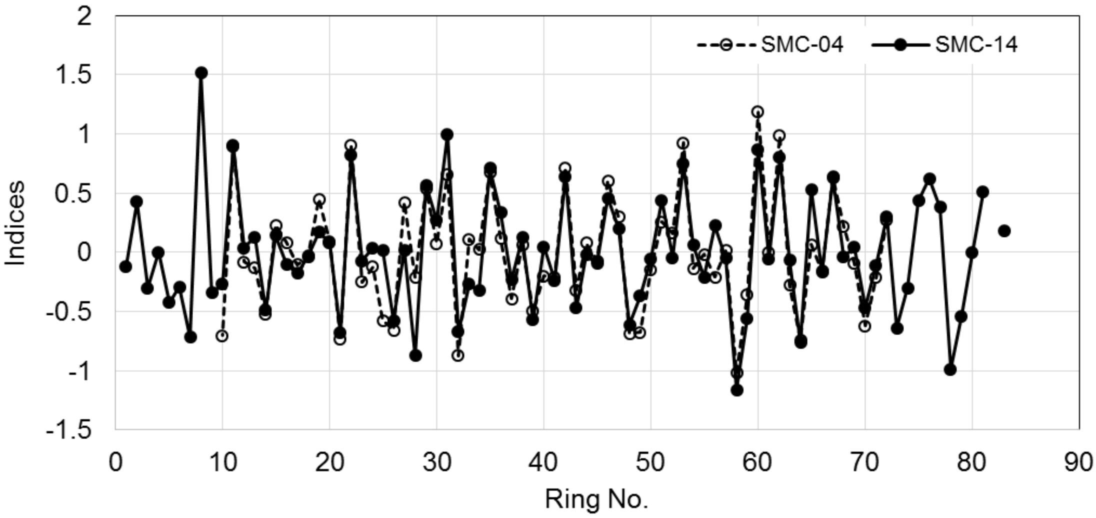



*Figure 3: Time series of the filtered and indexed $\delta^{18}O$ values from the two samples plotted at the position of strongest match (Student's t-value of 14.09). Note: It was not possible to obtain a suitable latewood sample for SMC-14 rings 81 and 83.*

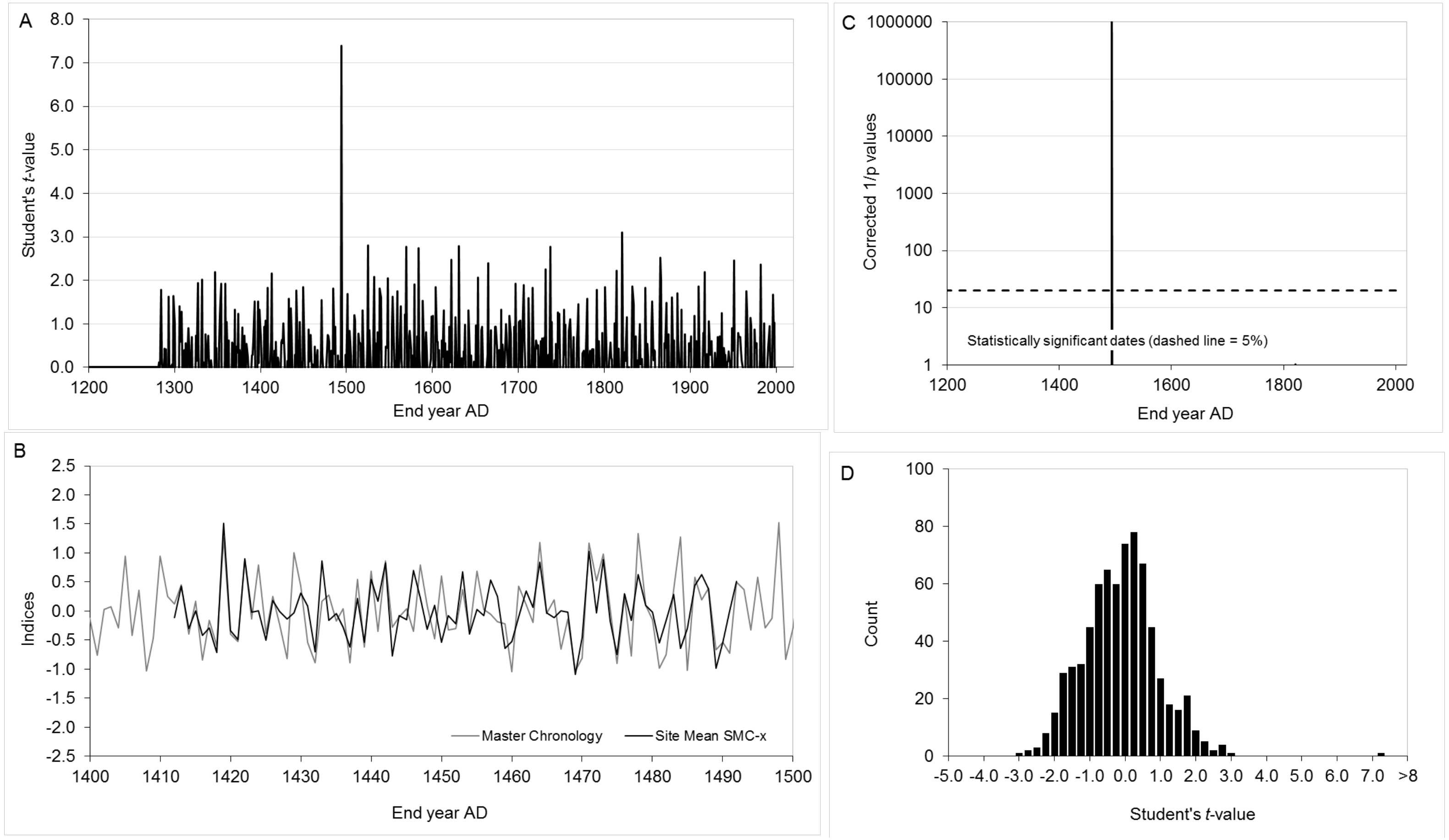



*Figure 4: Dating results for the 82-year mean isotope chronology (SMC-04 and SMC-14). A: Student's t-values for all possible end dates with full overlap against the master chronology. B: Time series of the site isotopic mean plotted against the master chronology. C: End dates with corrected probabilities (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 (δ18O) for the measured tree ring series

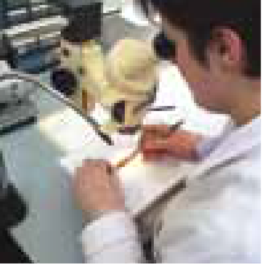
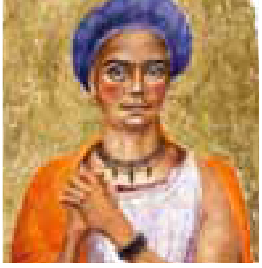
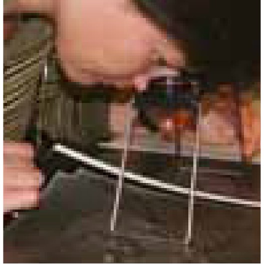
Data are reported as per mille (‰) deviations relative to the VSMOW standard (Coplen 1995).

Sample SMC-04

| Ring | δ18O | Ring | δ18O | Ring | δ18O |
|---|---|---|---|---|---|
| 1 | 29.03 | 31 | 29.00 | 61 | 28.07 |
| 2 | 30.73 | 32 | 28.97 | 62 | 28.46 |
| 3 | 29.76 | 33 | 30.00 | 63 | 28.89 |
| 4 | 29.66 | 34 | 28.99 | | |
| 5 | 29.13 | 35 | 29.37 | | |
| 6 | 29.99 | 36 | 29.19 | | |
| 7 | 29.71 | 37 | 29.90 | | |
| 8 | 29.39 | 38 | 29.56 | | |
| 9 | 29.51 | 39 | 28.63 | | |
| 10 | 29.97 | 40 | 28.75 | | |
| 11 | 29.51 | 41 | 29.29 | | |
| 12 | 28.56 | 42 | 29.63 | | |
| 13 | 30.10 | 43 | 29.51 | | |
| 14 | 28.97 | 44 | 30.37 | | |
| 15 | 28.99 | 45 | 29.28 | | |
| 16 | 28.57 | 46 | 29.37 | | |
| 17 | 28.60 | 47 | 29.28 | | |
| 18 | 29.69 | 48 | 29.50 | | |
| 19 | 29.02 | 49 | 28.46 | | |
| 20 | 29.84 | 50 | 29.11 | | |
| 21 | 29.48 | 51 | 30.58 | | |
| 22 | 30.23 | 52 | 29.40 | | |
| 23 | 28.67 | 53 | 30.31 | | |
| 24 | 29.63 | 54 | 29.17 | | |
| 25 | 29.50 | 55 | 28.71 | | |
| 26 | 30.08 | 56 | 29.30 | | |
| 27 | 29.38 | 57 | 28.93 | | |
| 28 | 28.90 | 58 | 29.52 | | |
| 29 | 29.40 | 59 | 29.08 | | |
| 30 | 28.78 | 60 | 28.73 | | |

Sample SMC-14

| Ring | δ18O | Ring | δ18O | Ring | δ18O |
|---|---|---|---|---|---|
| 0 | 28.78 | - | - | - | - |
| 1 | 29.34 | 31 | 28.50 | 61 | 29.74 |
| 2 | 28.55 | 32 | 28.98 | 62 | 29.06 |
| 3 | 29.10 | 33 | 28.89 | 63 | 28.40 |
| 4 | 28.64 | 34 | 29.84 | 64 | 29.58 |
| 5 | 28.81 | 35 | 29.33 | 65 | 28.80 |
| 6 | 28.50 | 36 | 28.78 | 66 | 29.45 |
| 7 | 30.84 | 37 | 29.21 | 67 | 28.76 |
| 8 | 29.03 | 38 | 28.46 | 68 | 28.77 |
| 9 | 29.12 | 39 | 28.98 | 69 | 28.10 |
| 10 | 30.36 | 40 | 28.64 | 70 | 28.45 |
| 11 | 29.56 | 41 | 29.59 | 71 | 28.81 |
| 12 | 29.44 | 42 | 28.47 | 72 | 27.86 |
| 13 | 28.82 | 43 | 28.91 | 73 | 28.05 |
| 14 | 29.47 | 44 | 28.81 | 74 | 28.77 |
| 15 | 29.08 | 45 | 29.42 | 75 | 28.96 |
| 16 | 28.87 | 46 | 29.15 | 76 | 28.73 |
| 17 | 29.05 | 47 | 28.40 | 77 | 27.43 |
| 18 | 29.26 | 48 | 28.76 | 78 | 27.95 |
| 19 | 29.10 | 49 | 29.11 | 79 | 28.45 |
| 20 | 28.31 | 50 | 29.52 | 80 | 28.96 |
| 21 | 29.76 | 51 | 29.05 | 81 | no value |
| 22 | 28.87 | 52 | 29.90 | 82 | 28.67 |
| 23 | 28.84 | 53 | 29.10 | | |
| 24 | 28.88 | 54 | 28.75 | | |
| 25 | 28.39 | 55 | 29.22 | | |
| 26 | 29.02 | 56 | 28.93 | | |
| 27 | 28.09 | 57 | 27.80 | | |
| 28 | 29.55 | 58 | 28.40 | | |
| 29 | 29.25 | 59 | 29.78 | | |
| 30 | 30.14 | 60 | 28.90 | | |



# Historic England Research and the Historic Environment

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A good understanding of the historic environment is fundamental to ensuring people appreciate and enjoy their heritage and provides the essential first step towards its effective protection.

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