Radiocarbon, Vol 63, Nr 5, 2021, p 1445–1452 DOI:10.1017/RDC.2021.75
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AN INTERCOMPARISON PROJECT ON ^{14}C FROM SINGLE-YEAR TREE RINGS

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ABSTRACT. A laboratory intercomparison project was carried out on 20 annually resolved late-wood samples from the Danish oak record. The project included the following three laboratories: (1) the University of Arizona AMS Laboratory, University of Arizona, USA (AA); (2) HEKAL AMS Laboratory, MTA Atomki, Hungary (DeA); and (3) Aarhus AMS Centre (AARAMS), Aarhus University, Denmark (AAR). The large majority of individual data points (96%) lie within ±2σ of the weighted mean. Further assessment of the accuracy associated with the individual laboratories showed good agreement, indicating that consistent and reliable ^{14}C measurements well in agreement with each other are produced at the three laboratories. However, the quoted analytical uncertainties appear to be underestimated when compared to the observed variance of differences from the geometric mean of the samples. This study provides a general quality check of the single-year tree-ring ^{14}C measurements that are included in the new calibration curve.

KEYWORDS: IntCal, intercomparison, radiocarbon dating, single-year measurement, tree ring.

INTRODUCTION

In the radiocarbon (^{14}C) community, there is a long tradition for proficiency testing of laboratory protocols and ^{14}C analysis in terms of accuracy and measurement variability (e.g. Gulliksen and Scott 1995; Naysmith et al. 2007; Scott et al. 2010). Because the international ^{14}C calibration curve is the backbone for almost all ^{14}C dating world-wide, it is of utmost importance to carefully test the accuracy and analytical precision of tree-ring samples submitted as datasets for an updated calibration curve (see Wacker et al. 2020). Two of the three labs in this study (Arizona and Aarhus) have taken part in the large intercomparison study organized by the Swiss AMS group (Wacker et al. 2020), but the present study aimed specifically at demonstrating laboratory performance on single-year wood from the Danish oak record. This could eventually help secure consistent ^{14}C measurements and also strengthen cooperation among laboratories internationally. This is crucial for comparison of data, and for achieving reliable datasets used for future calibration curves.

To validate recently produced ^{14}C measurements on single-year late-wood tree rings, which have been submitted to the new calibration curve, an intercomparison project including the three laboratories HEKAL AMS Laboratory, MTA Atomki, Hungary (DeA), the University of Arizona AMS Laboratory, University of Arizona, USA (AA), and Aarhus AMS Centre (AARAMS), Aarhus University, Denmark (AAR), was performed. The samples used for this intercomparison derive from the Danish oak record, which also provided samples for the updated radiocarbon calibration curve (Reimer et al. 2020). The

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https://doi.org/10.1017/RDC.2021.75 Published online by Cambridge University Press

samples were first analyzed at AARAMS and then distributed to HEKAL and the Arizona AMS Laboratory with no prior information on their calibrated or ^{14}C age. The ^{14}C data produced by AARAMS were published by Fogtmann-Schulz et al. (2017, 2019) and Kudsk et al. (2019). Several ^{14}C data produced at AA and HEKAL have also been accepted for the new calibration curve (e.g., Jull et al. 2018a; Manning et al. 2018; Pearson et al. 2018).

METHODS

Twenty late-wood samples from two pieces of Danish oak were isolated at AARAMS and sent to the University of Arizona Laboratory, Tucson (Arizona), and HEKAL, Debrecen. The two pieces of wood originate from Ravning Enge and Haderslev Fjord, Denmark, and span the periods 693–702 and 1011–1020 CE, respectively. Oak trees grow by producing annual tree rings composed of an early- (EW) and a late-wood (LW) component. In the beginning of the growing season (spring and early summer), EW is produced and succeeded by LW, which is produced in the late summer (Speer 2010). The EW fraction is partially produced from a reserve stored in the tree the previous growing season and may thus contain ^{14}C atoms from the previous year (Pilcher 1995; Speer 2010). In this intercomparison project, only the LW fraction is used as this is the most reliable wood-component for ^{14}C analysis (Kudsk et al. 2018). Details on wood cutting, EW and LW separation, and dendrochronology can be found in Fogtmann-Schulz et al. (2019) and Kudsk et al. (2019). The samples were not homogenized. The possible carry-over effect from the previous growing season is expected to be undetectable in the measured data since the data derive from a time period where the calibration curve is relatively flat, and because the samples solely consist of LW. At all three laboratories, cellulose was extracted from the LW samples prior to ^{14}C analysis, using each laboratory's own standard extraction method.

Arizona AMS Laboratory (AA)

α-cellulose extraction was performed using the method of Lange et al. (2019). Wood samples were first subjected to a standard acid-base-acid extraction: 3N hydrochloric acid (HCl) at 70°C overnight, type-1 water washes, 1.2N sodium hydroxide (NaOH) at 70°C overnight with additional changes of the NaOH solution and incubation until the wash solution remained clear, type-1 water washes, followed by 3N HCl at 70°C overnight, followed by water washes. The wood was then placed in acidified sodium chlorite (0.3N $NaClO_2$, in $60{\cdot}10^{-3}$ mN HCl), and incubated at 75°C with solution changes until the wood was white in color. This was followed by another round of type-1 water washes. Extraction continued with 17.5% NaOH at room temperature for 2 hr, rinsing to a neutral pH with type-1 water, a final acid wash with 3N HCl for 1 hr, and a finishing rinsing to a neutral pH with type-1 water. Holo-cellulose purified from Brown Coal (Tertiary Age, from S. Levitt, Laboratory for Tree-Ring Research, University of Arizona) was used as process blank. Combustion, graphitization, and AMS measurement on a 3MV NEC 9SDH Pelletron AMS running at 2.5MV were carried out as described by Jull et al. (2006).

Hertelendi Laboratory of Environmental Studies, HEKAL (DeA)

Holo-cellulose was extracted using a base-acid-base-acid-bleaching procedure (BABAB) (method fully described in Molnár et al. 2013a). Samples were alternately suspended in 4% (1N) NaOH and 4% HCl two times, and lastly treated with 5% $NaClO_2$ acidified with 4% HCl. All steps were performed at 75°C.

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HEKAL has run IAEA-C9 (fossil wood) samples together with the tree-ring samples to monitor the process blank level and other exogenous contamination. The cellulose was combusted in sealed glass tubes containing MnO_2, and then converted to CO_2, cryogenically purified, and graphitized by a sealed-tube graphitization method (Rinyu et al. 2013; Janovics et al. 2018). The graphite targets were analyzed in the EnvironMICADAS AMS system at HEKAL AMS Laboratory, ATOMKI, Debrecen, Hungary (Molnár et al. 2013b).

Aarhus AMS Centre, AARAMS (AAR)

α-cellulose was extracted from wood samples using a combination of the methods proposed by Loader et al. (1997) and Southon and Magana (2010), summarized in Fogtmann-Schultz et al (2020). The wood samples were first bleached at 70°C with 1N $NaClO_2$ acidified with 1N HCl, then treated at room temperature with 17% NaOH to dissolve hemicelluloses. Lastly, samples were treated with 1N HCl at room temperature to remove any contamination from atmospheric CO_2. Between each part of the pretreatment, the samples were rinsed with Milli-Q water to neutral pH. Kauri wood from Marine Isotope Stage 7 provided by P. Reimer at Queen's University Belfast was pretreated along with the wood samples, and used as a process blank. Subsequently, the α-cellulose was sealed in quartz tubes containing copper oxide (CuO) and combusted to CO_2 at 900°C, cryogenically purified, and then reduced to graphite using hydrogen and iron as catalyst. All samples were analyzed using the 1MV High Voltage Engineering AMS system at the Aarhus AMS Centre, Aarhus University, Denmark (Olsen et al. 2017).

All laboratories use international secondary standards, such as IAEA-C3, IAEA-C7 and IAEA-C8, as well as wood-blank materials, including charcoal, oak (see e.g., Jull et al 2018b; Fogtmann-Schultz et al. 2019; Kudsk et al. 2019) and Brown Coal. At AARAMS, ^{14}C ages are calculated using an in-house graphical normalization program in Matlab, whereas Arizona uses the in-house software and methods described in Donahue et al. (1990) and Burr et al. (2007). HEKAL uses the standard BATS software for sample calculation and data reduction (Wacker et al. 2010). Assessment of secondary standards and blanks for the AARAMS analysis can be found in Fogtmann-Schultz et al. (2019) and Kudsk et al. (2019), while for Arizona, Brown Coal holocellulose process blanks were measured with a $F^{14}C$ value of 0.0017 ± 0.0004 (n=8). The geometric mean and scatter of the processed IAEA-C9 samples ($F^{14}C = 0.0016 \pm 0.0003$, n=3) and IAEA-C3 samples ($F^{14}C = 1.2929 \pm 0.0019$, n=3) from HEKAL were in good agreement with their consensus values. Other intercomparisons of HEKAL wood preparation and AMS ^{14}C analyses were recently reported by Jull et al. (2018b). For all three laboratories, the uncertainty of the primary standard (OX-II) and blank samples is included in the analytical result using propagation of errors.

RESULTS AND DISCUSSION

The average cellulose extraction yield of the three laboratories is very similar, amounting to 39.5%, 39.7%, and 38.5% for the Arizona AMS Laboratory, HEKAL, and AARAMS, respectively. For each year, we calculated the weighted average ^{14}C age, and a reduced χ^2 test is used to assess the validity of the weighted average values. The deviations from the weighted average in units of standard deviations (z-scores) are calculated for each laboratory and plotted in Figure 2, along with the deviation mean for each individual

https://doi.org/10.1017/RDC.2021.75 Published online by Cambridge University Press

laboratory. One sample, AAR-27731, differs more than 6σ from the weighted average values and is regarded as an outlier, and it will therefore not be discussed further. All data are presented in Table 1 and in Figure 1.

Overall, the ^{14}C results from the three laboratories agree within statistical error (Figure 1). Three samples have z-scores equal to or higher/lower than ±2σ, corresponding to 4.4% of the total samples (Table 1). A total of 5% of the samples may be expected to be outside the ±2σ range if normally distributed. The reduced χ^2 values range from 0 to 3.3, and three samples year (697 CE; χ^2: 3.3≤3.0, 698 CE; χ^2: 3.2≤3.0 and CE 700; χ^2: 2.9≤2.6) fail the reduced χ^2 test. This may indicate that the ^{14}C age variability of these three samples is too large, and thus that the individual measurements are not statistically agreeing (Table 1). However, at the 95% significance level, 5% of the samples are expected to fail the χ^2 test (a total of 5.0% failed). If tested at the 97% significance level, all samples pass the χ^2 test. Thus, no obvious outliers can be observed in the dataset apart from AAR-27731 (Figure 2). The average value of the reduced χ^2 values is 1.6 with a standard deviation of 1.0. Normally distributed samples would display a reduced χ^2 value around 1 with a χ^2 standard deviation of 1.0. In the present dataset, 40% of an expected 70% of the samples show reduced χ^2 values lower than or equal to 1.2, suggesting that even though they pass the reduced χ^2 test the sample variance for individual years is higher than expected in 30% of the cases. This may indicate that the analytical errors most likely are underestimated.

The average analytical error for samples from Arizona (AA) is ±28 years (±3.4‰), while HEKAL and AARAMS both yielded ±16 ^{14}C years (±2.0‰) (Table 1). To evaluate the mean measurement uncertainty, we calculate the geometric mean (μ) of the results, and subsequently the mean and standard deviation of the difference (Δμ) from the geometric mean (Table 1). For the Arizona AMS Laboratory, the standard deviation of the differences from the geometric mean is ±23 ^{14}C years, which is lower than the quoted analytical error. For HEKAL and AARAMS, the standard deviation of the differences from the geometric mean is ±18 and ±20 ^{14}C years, respectively (Table 1). This may suggest that HEKAL and AARAMS underestimate the analytical errors with 2 and 4 ^{14}C years, respectively. However, if the analysis only considers HEKAL and AARAMS, then the differences from the geometric mean would be ±16 and ±17 ^{14}C years, respectively, and thus in better agreement with the quoted analytical uncertainties for both laboratories. Due to the limited number of measurements of each sample, it is difficult to assess the specific cause behind the missing error, and if the high variability is associated with one or more laboratories. The missing error could originate from the data reduction calculations, but it could also relate to an unknown variability in sample preparation or graphitization protocols.

It is clear, however, that the average reduced χ^2 value of 1.6 suggests that the errors generally are underestimated. If we use the average reduced χ^2 value to revise the common error of the complete dataset, then errors should increase by a factor of $\sqrt{1.6}$, or 1.26. This would correspond to mean errors of 35, 20, and 20 ^{14}C years for Arizona, HEKAL, and AARAMS, respectively. The mean values of the difference from the geometric mean of 6, –3, and –2 ^{14}C years, along with mean z-score values of 0.2, –0.1, and 0.1, indicate that all three laboratories accurately measure the ^{14}C age with no systematic errors (Table 1). The standard deviation of all z-scores from each laboratory is around ±1, further indicating that the quoted mean values are normally distributed within errors (Figure 2).

https://doi.org/10.1017/RDC.2021.75 Published online by Cambridge University Press

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Table 1 First column shows the sample calendar year in CE. Columns with Lab ID denote ID of the particular measurement and laboratory code. ^{14}C age (BP) denote the measured carbon ^{14}C age in years before present (BP). Z-score is the deviation in units of standard deviations from the calculated weighted average of the particular year, which is given in the column weighted mean (μ_w). Reduced χ^2 statistics are provided as the χ^2 value of the sample, which is to be compared with the limiting χ^2 value calculated at 95% confidence interval. The geometric mean (μ) and the individual sample difference from the geometric mean ($\Delta\mu$) are furthermore shown. AAR data are previously published in Kudsk et al. (2019). Outliers are marked with *.

Age (CE)	Lab ID	Yield %	Size mgC	^{14}C age (BP)	z-score	$\Delta\mu$	Lab ID	Wood mg	Yield %	^{14}C age (BP)	z-score	$\Delta\mu$	Lab ID	Yield %	Size mgC	^{14}C age (BP)	z-score	$\Delta\mu$	μ_w ^{14}C age (BP)	χ^2 test	μ ^{14}C age (BP)
693	AA-111878	40	1.23	1308 ±36	0.3	8	DeA-20586	15.5	40.1	1307 ±14	0.7	7	AAR-27736	43.3	1.0	1284 ±15	–0.9	–16	1297 ±10	0.7≤3.0	1300
694	AA-111879	39	1.42	1302 ±31	0.7	18	DeA-20587	14.8	32.9	1290 ±15	0.6	6	AAR-27735	35.4	1.1	1257 ±16	–1.5	–27	1281 ±9	1.1≤2.6	1284
																1288 ±16	0.4	4			
695	AA-111880	40	1.35	1289 ±32	0.0	–3	DeA-20588	16.3	37.0	1268 ±15	–1.5	–24	AAR-27734	42.6	1.0	1319 ±17	1.7	27	1290 ±11	2.5≤3.0	1292
696	AA-111881	41	1.43	1265 ±24	0.5	14	DeA-20589	15.8	36.1	1273 ±15	1.4	22	AAR-27733	37.3	1.1	1246 ±15	–0.4	–5	1252 ±9	1.7≤2.6	1251
																1221 ±19	–1.6	–30			
697	AA-111882	38	1.66	1323 ±28	1.3	26	DeA-20590	16.1	40.9	1261 ±14	–1.8	–36	AAR-27732	41.1	1.0	1306 ±16	1.3	9	1286 ±10	3.3≤3.0	1297
698	AA-111883	39	1.35	1249 ±21	–2.0	–32	DeA-20593	18.9	36.1	1283 ±20	–0.4	2	AAR-27731	37.8	1.1	1312 ±14	1.6	31	1290 ±10	3.2≤3.0	1281
																1185 ±16*					
699	AA-111884	39	1.27	1272 ±26	–0.4	–12	DeA-20594	15.5	41.3	1317 ±20	1.8	33	AAR-27730	42.2	1.1	1264 ±16	–1.1	–20	1282 ±11	2.2≤3.0	1284
700	AA-111885	41	1.31	1252 ±21	–0.6	–10	DeA-20595	14.4	39.1	1244 ±20	–1.1	–18	AAR-27729	38.5	1.1	1304 ±16	2.4	42	1265 ±9	2.9≤2.6	1262
																1247 ±16	–1.1	–15			
701	AA-111886	40	1.31	1299 ±24	0.2	3	DeA-20596	15.7	41.6	1304 ±20	0.5	8	AAR-27728	41.3	0.9	1286 ±15	–0.5	–10	1294 ±11	0.3≤3.0	1296
702	AA-111887	38	1.10	1247 ±24	–1.9	–42	DeA-20597	18.2	40.6	1321 ±20	1.4	32	AAR-27727	41.2	1.0	1310 ±16	1.1	21	1293 ±9	2.6≤2.6	1289
																1278 ±16	–0.9	–11			
1011	AA-111868	39	1.56	1035 ±23	–0.6	–11	DeA-20576	18.8	42.4	1038 ±14	–0.7	–8	AAR-27821	38.1	1.0	1065 ±15	1.1	19	1048 ±9	1.1≤3.0	1046
1012	AA-111869	43	1.66	1045 ±21	0.0	0	DeA-20577	19.2	40.8	1042 ±14	–0.1	–3	AAR-27822	36.8	1.0	1047 ±15	0.2	2	1044 ±9	0.0≤3.0	1045
1013	AA-111870	36	1.44	1068 ±23	1.7	34	DeA-20578	18.7	45.6	1029 ±15	–0.1	–5	AAR-27823	35.9	1.0	1006 ±19	–1.3	–28	1030 ±10	2.2≤3.0	1034
1014	AA-111871	40	1.54	1056 ±26	1.1	23	DeA-20579	19.3	34.6	1034 ±15	0.4	1	AAR-27824	37.2	1.0	1009 ±17	–1.1	–24	1028 ±10	1.3≤3.0	1033
1015	AA-111872	40	1.45	1068 ±45	0.7	23	DeA-20580	18.6	42.2	1035 ±14	0.0	–10	AAR-27825	34.8	1.1	1031 ±16	–0.3	–14	1035 ±10	0.3≤3.0	1045
1016	AA-111873	39	1.18	1062 ±23	–0.2	–4	DeA-20581	18.0	43.0	1066 ±15	0.0	0	AAR-27826	36.7	0.8	1064 ±16	–0.1	–2	1066 ±9	0.1≤2.6	1066
																1072 ±18	0.3	6			
1017	AA-111874	38	1.34	1091 ±40	1.6	48	DeA-20582	17.9	42.0	1021 ±15	–0.5	–22	AAR-27827	39.2	0.9	1028 ±16	–0.1	–15	1029 ±9	0.9≤2.6	1043
																1031 ±18	0.1	–12			
1018	AA-111875	40	1.28	1042 ±24	0.6	10	DeA-20583	18.2	35.3	1009 ±14	–1.3	–23	AAR-27828	37.9	1.0	1044 ±16	1.1	12	1027 ±10	1.6≤3.0	1032
1019	AA-111876	40	1.39	1096 ±38	2.0	59	DeA-20584	19.3	42.1	1018 ±14	–0.1	–19	AAR-27829	36.9	1.0	1000 ±15	–1.3	–37	1020 ±9	2.2≤2.6	1037
																1035 ±17	0.9	–2			
1020	AA-111877	39	1.15	1046 ±23	–0.8	–15	DeA-20585	18.6	40.7	1053 ±15	–0.8	–8	AAR-27830	35.7	1.1	1085 ±15	1.3	24	1065 ±10	1.6≤3.0	1061
Mean		39.5		28	0.2	7			39.7	16	–0.1	–3		38.5		16	0.0	–3			
Std		1.4		±7	±1.0	±24			±3.3	±2	±0.9	±18		±2.5		±1	±1.1	±20			
Min		36.0		21	–2.0	–42			32.9	14	–1.8	–36		34.8		14	–1.6	–37			
Max		43.0		45	2.0	59			45.6	20	1.8	33		43.3		19	2.4	42			

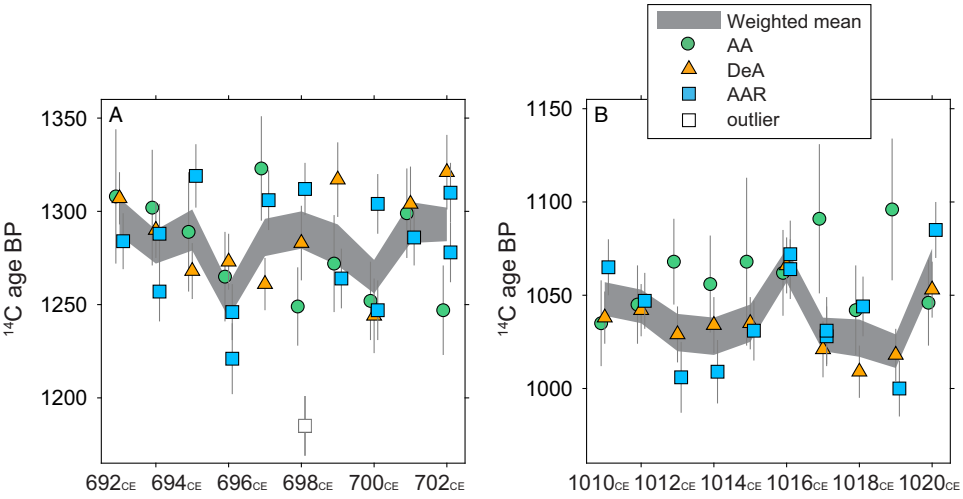

Figure 1 Measured ^{14}C ages from each laboratory. Figure A shows results of the wood piece from Ravning Enge (693–702 CE) and Figure B of the piece from Haderslev Fjord (1011–1020 CE).

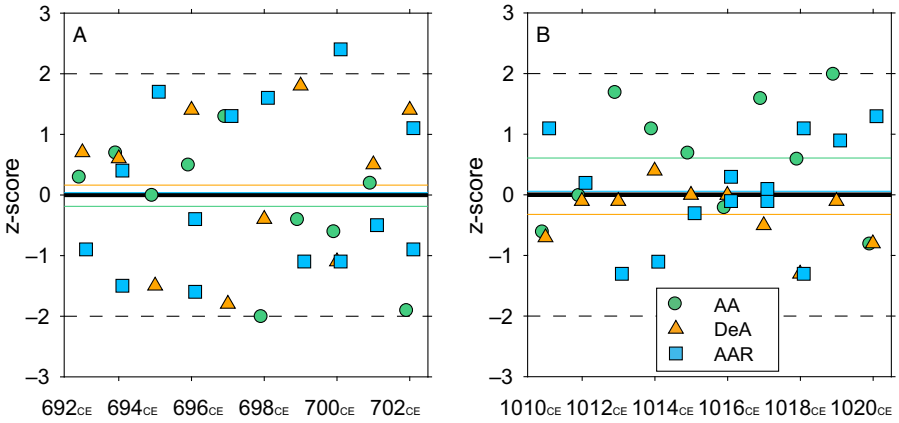

Figure 2 z-scores of the measurements from the three laboratories. Horizontal colored lines indicate the mean of the z-scores for each laboratory. Figure A shows z-scores for the wood piece from Ravning Enge (693-702 CE) and Figure B of the piece from Haderslev Fjord (1011–1020 CE). The outlier AAR-27731 has been omitted.

This study shows that the three laboratories, using different chemical pretreatments, instruments, and data-handling procedures, are able to produce consistent and reliable ^{14}C measurements that are in good agreement with each other. Further, the statistical outcome from the high-precision datasets from HEKAL and AARAMS, obtained using different instruments, are very similar (Table 1). Although the laboratories have used different cellulose extraction methods, there appears to be no systematic influence on the ^{14}C measurements or the cellulose yield, which is in agreement with previous work (e.g., Němec et al. 2010). This small intercomparison study was conducted to evaluate the laboratory procedures for producing single-year ^{14}C data for the international calibration curve. The IntCal working group assesses the quality (analytical errors and agreement among different

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datasets) of all datasets that are considered for inclusion in a forthcoming calibration curve. The discussion presented here therefore highlights a need for further reducing the scatter and improving estimates of the associated uncertainties.

CONCLUSION

An intercomparison project involving the three laboratories: (1) University of Arizona AMS Laboratory, Arizona; (2) HEKAL, Debrecen; and (3) AARAMS, Aarhus, has been carried out on two pieces of Danish oak covering a total of 20 years. Using reduced χ^2 statistics and individual sample differences from the geometric mean, it can be concluded that the three laboratories are able to produce accurate and reliable ^{14}C measurements. The quoted analytical uncertainties appear to be underestimated when compared to the observed variance of differences from the geometric mean. Furthermore, no systematic effects related to the sample pretreatment, type of instrument, modes of operation, and data handling could be observed.

ACKNOWLEDGMENTS

We are grateful for the support of the technical staff at all three laboratories. Sabrina Gjødvad Kaiser Kudsk, Mads Faurschou Knudsen and Christoffer Karoff sincerely acknowledge the financial support from the Villum Foundation. This work was partly supported by the European Union and the State of Hungary, co-financed by the European Regional Development Fund in the project of GINOP-2.3.2-15-2016-00009 "ICER".

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