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THE BEST POSSIBLE TIME RESOLUTION: HOW PRECISE COULD A RADIOCARBON DATING METHOD BE?

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ABSTRACT. Today, accelerator mass spectrometry (AMS) technology enables us to carry out very precise measurements of radiocarbon (¹⁴C). Unfortunately, due to fluctuations in the ¹⁴C calibration curve, the resulting calibrated time intervals vary from decades up to centuries in calibrated age. Within a time scale of several decades, we can find several time intervals on the ¹⁴C calibration curve which correspond with periods of rapid increases in atmospheric ¹⁴CO₂ activity. Some of these “high slope” parts of the calibration curve could be used for fine time resolution for radiocarbon dating of individual samples. Nevertheless, there are certain limitations owing to the properties of the samples measured. We have prepared a time-resolution curve for the ¹⁴C dating method, applying calibration curve IntCal13 and assuming an uncertainty of ¹⁴C analyses ±15 yr BP (for recent samples). Our curve of the time resolution covers the last 50 ka. We found several time intervals with time resolution below 50 yr BP for the last 3 ka. Several time intervals which can enable substantially better time resolution compared to neighboring parts of the calibration curve were also found for periods older than 3 ka.

KEYWORDS: curve of time resolution, IntCal13, radiocarbon dating, tree rings.

INTRODUCTION

Radiocarbon (¹⁴C) dating is used in a wide range of research disciplines, including archaeology, geology, paleobotany, sedimentology, karst studies and paleoclimatology. The number of samples processed for ¹⁴C dating is still increasing every year. Obviously, users request as high precision as possible. Better time resolution can help to resolve some unanswered research issues, which can sometimes be resolved with this increased resolution.

The most precise dating results use the dendrochronological method for the dating of wooden artifacts. This method is limited by minimum number of tree rings (Prokop et al. 2017). It is suggested that tree-ring width series should not be shorter than 40 (Cook and Kairiūkštis 1990) or even 50 years (Haneca et al. 2009; Rybniček et al. 2015, 2018) for precise dating. In addition, wood species identification, provenance of samples and existence of a tree-ring width chronology are essential inputs for successful dendrochronological dating. Also, excellent time resolution can be reached by using the radiocarbon wiggle-matching method and such results can approach dendrochronological precision, if sufficiently long sequences of tree rings are available (van der Plicht and Mook 1989; Bleicher 2014; Jacobsson et al. 2018; Krapiec et al. 2018; McDonald et al. 2018). Unfortunately, the majority of samples for

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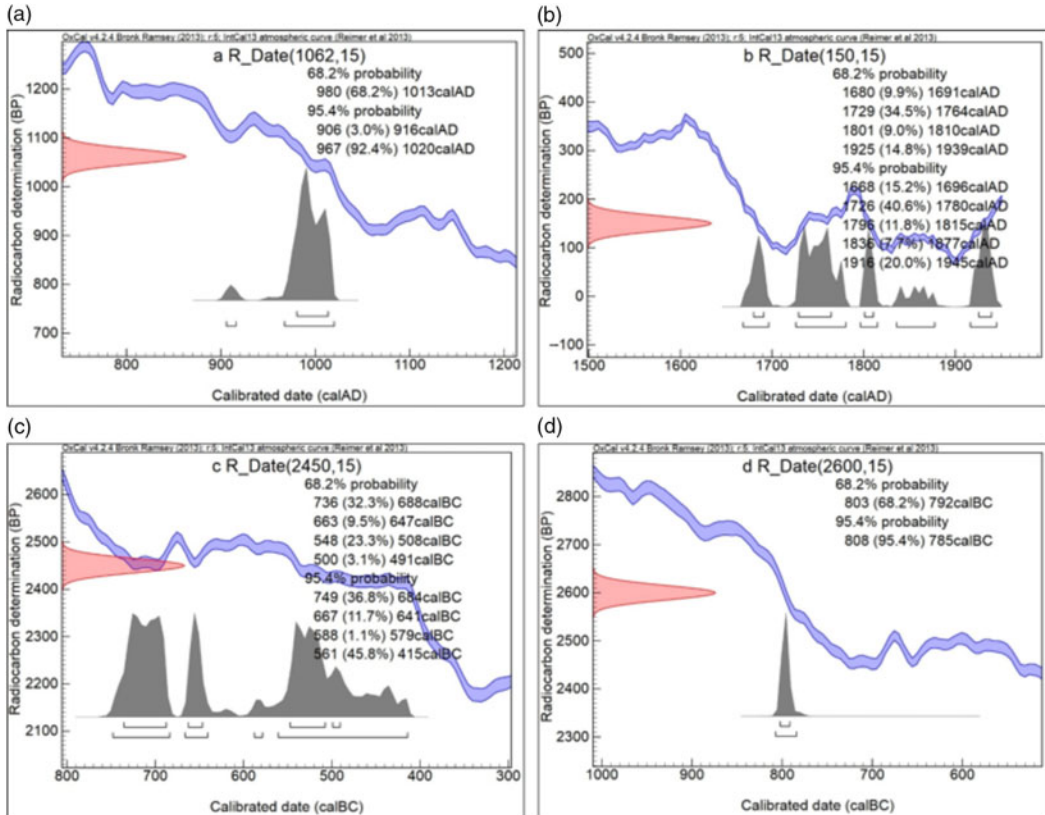


Figure 1 (a)–(d): Examples of characteristic parts of the calibration curve IntCal13 (for better visual comparison is the horizontal scale, about 500 years for each diagram).

radiocarbon dating (archaeological, geological, etc.) are isolated samples, without the possibility of grouping them with other samples with a known relative time gap.

Using high-precision accelerator mass spectrometry (AMS) measurements, we can determine ^{14}C activity with uncertainties of about 15 years of conventional radiocarbon age in samples several thousand years old. The current internationally agreed calibration curves for dating of the terrestrial samples are IntCal13 for the Northern Hemisphere (Reimer et al. 2013) and SHCal13 for the Southern Hemisphere (Hogg et al. 2013). For both hemispheres, these curves cover the past 50,000 years.

Over a time scale of several decades, parts of the radiocarbon calibration curves (reported in the years of conventional radiocarbon age) can be differentiated into four main types: (a) relatively monotonic parts of the curve, resulting in one main time interval; (b) fluctuating parts of the curve, with several resulting separate time intervals with similar probabilities, e.g. 1640–1950 AD, which is rather difficult for radiocarbon dating; (c) plateaus in the curve where refining of a radiocarbon measurement uncertainty is not reflected in a narrower time interval, thus, corresponding approximately to a width of the plateau; and (d) relatively steep parts of the curve which enable definite and very narrow time intervals. These various results are shown as examples in Figure 1.

The steep parts are often seen close to the edges of a plateau. Thus, under favorable circumstances for ^{14}C dating, it is possible to reach a very narrow resulting time interval. The steep parts of the curve could then be possibly used as a “time magnifier” or an exact “time marker”. Time periods which correspond to the steep parts of the curve have relatively short duration. Therefore, the likelihood of results which would also correspond to these time periods on the steep parts of the curve can be also low. Likewise, dating using steep parts of the calibration curve could be connected with some discrepancies implying small (but significant) differences in ^{14}C activity in the calibration curve and in real samples. This could be caused by slight geographical differences of ^{14}C activity course, by different periods of a wood mass growth caused by small microclimatic differences in examined sites, or by a species of the tree with a different period of wood growth (we can assume that small annual seasonal changes of ^{14}C activity occurred also in the distant past). These regional effects should be amplified in steep section of the curve.

For ^{14}C laboratories and the users of ^{14}C dating, it is very important to provide realistic results with the time intervals which are as precise as reasonably possible. On the other hand, the results should not offer an uncritically high, yet distorted time resolution, which is more likely a result of random influences caused by improper sample selection, insufficient sample treatment or underestimation of related uncertainties, e.g. systematic ones.

The purpose of this study is to provide a basic survey of the time resolution available for radiocarbon dating and validate application of the steep parts of the calibration curve IntCal13 for the purpose of precise radiocarbon dating of solitary samples. It can be assumed that the slope parts of calibration curves can provide a possibility to validate actual curves and their uncertainties. We also recommend some relevant types of samples, which can be proper for precise dating. Relevant samples with known origin from the periods corresponding to the rapid slope sections of the calibration curve can also provide a particularly sensitive “natural” material (due to rapid changes of ^{14}C activity at this period) for testing and comparing sample treatment routines, which are focused on the removal of interfering carbon chemical forms.

METHODS

Following the considerations discussed above, we carried out the first step of the experiment using tree-ring samples. Together with dendrochronologists, we evaluated the ^{14}C activity in tree rings corresponding to the selected steep parts of the actual calibration curve IntCal13.

a) Processing of the IntCal13 curve

The expected resolution of ^{14}C dating of a sample corresponding to a specific calendar age was calculated as the weighted average length of all dating intervals at 95% probability level which contain this date after calibration using the IntCal13 curve. The dating intervals were weighed by the probability distribution of the ^{14}C date after calibration,

$$r_x = \frac{\sum_{i=1}^n r_i p_{xi}}{\sum_{i=1}^n p_{xi}} \quad (1)$$

where r_x is the weighted average dating interval length (resolution) at a specific calibrated age x , r_i is the length of the interval obtained by calibrating ^{14}C date i (at 95% probability level), p_{xi} is the value from the probability distribution of the date i after calibration at

Table 1 A list of utilized sequences of oak tree-rings.

Sampling place	Region	Length of tree-ring series (years)	Start year (AD)
Dětmárovice	Moravian-Silesian	198	467
Strážnice	South Moravia	194	901
Bělčice u Ostředka	Central Bohemia	125	1187

calendar age. x Since we used the uncalibrated ^{14}C dates from the IntCal13 dataset as input data, n is the number of records in this dataset. To obtain probability distributions in calendar years for each date, we calibrated them using the same IntCal13 curve while assuming an uncertainty of measurement proportional to their radiocarbon age.

We assumed that the uncertainty of ^{14}C analyses of samples with activity (reported as Conventional Radiocarbon Age) about 0 BP is just 15 yr BP (Stuiver and Polach 1977). Uncertainties of the samples with lower activities were calculated using the formula:

$$u_i = u_1 \cdot e^{\frac{+A_i}{2.8033}} \quad (2)$$

where u_i is the expected uncertainty of ^{14}C analysis of the sample at a specific radiocarbon age, u_1 is the expected uncertainty of analysis of 15 yr BP (for a sample with activity 0 yr BP), A_i is the activity (reported as ^{14}C yr BP) of the sample.

b) Selection of relevant tree rings

All samples were taken from archaeological or subfossil findings from the eastern part of the Czech Republic, as shown in Table 1. After careful surface preparation to maximize the visibility of tree-ring borders (Gärtner and Nievergelt 2010), tree-ring widths were measured with the VIAS TimeTable system with accuracy of 0.01 mm. The resulting annual ring width values were visually synchronized and cross-dated using PAST4 (Knibbe 2004), as well as statistically controlled via COFECHA (Grissino-Mayer 2001). The final tree-ring width series were dated according to the latest version of the Czech oak tree-ring width chronology (Kolář et al. 2012; Prokop et al. 2017; Dobrovolný et al. 2018). Then, each precisely dated annual oak increment was separated and finely cut (separately for early wood and late wood) with a scalpel or razor blade under a stereomicroscope into small pieces.

c) Description of a sample treatment (at CRL Lab, Prague, Czech Republic) and AMS measurements (at ICER Atomki Lab, Debrecen, Hungary)

The samples were washed repeatedly with 4% HCl followed by 4% NaOH and finally 4% HCl again. Before and after the alkaline extraction, the samples were rinsed with distilled water in order to adjust the pH of the extract to 6–8 (Gupta and Polach 1985). Subsequently, the samples were dried at 60°C to reach constant weight. After the pretreatment, the dry samples together with a small amount of CuO were torch sealed under a dynamic vacuum into a quartz glass tubes and combusted at 900°C for at least 8 hr. The resulting carbon dioxide was then purified and transferred into the graphitization reactor. The batch method of graphitization with pure Zn as a sole reduction agent was derived from similar routines described by Rinyu et al. (2015) and by Orsovski and Rinyu (2015). Following the graphitization step, the samples were sealed in vacuum and sent for AMS measurements to the ICER laboratory in Debrecen with international code “DeA-“ (Molnár et al. 2013a, 2013b; Handlos et al. 2018).

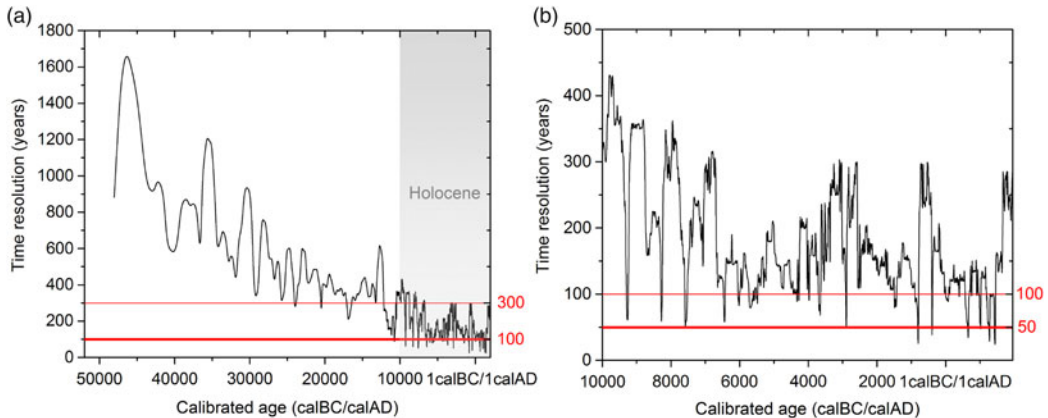


Figure 2 Dependence of the time resolution of the radiocarbon dating method (for 2σ interval of ^{14}C analyses) on the calibrated age of the sample (reported in the years calBC/calAD¹); two or more resulting time intervals are integrated into one “covering” interval (the absolute probability of such integrated intervals is slightly exceeding the declared 95% for two sigma): (a) The whole scale covered by IntCal13 with highlighted time resolutions of 100 years (bold red line) and 300 years (thin red line); (b) Holocene epoch in detail with highlighted time resolutions of 50 years (bold red line) and 100 years (thin red line). (Please see electronic version for color figures.).

The measurements were carried out using the EnvironMICADAS compact tandem accelerator (Kromer et al. 2013; Molnár et al. 2013b). Graphitized samples of oxalic acid NIST HOX II SRM 4990-C were used for calibration (Schneider et al. 1995). Graphitized samples of anthracite (preheated under dynamic vacuum at 800°C for at least 15 min) were used for correction of background contributions. The resulting ^{14}C activity and its combined uncertainty are given in yr BP (Before Present) as the conventional radiocarbon age according to Stuiver and Polach (1977), including $\delta^{13}\text{C}$ correction measured by the MICADAS (Curie 1955).

The calibration software OxCal was used for determination of the sample age; with respect to the available data, the radiocarbon calibration curve IntCal13 was chosen (Bronk and Lee 2013; Reimer et al. 2013). After assignment of uncertainties given by the radiocarbon calibration curve, the conventional radiocarbon age and its combined uncertainty were converted to calibrated age intervals (Table 3).

RESULTS

Using the routine described above, we obtained a diagram of the dependence of the possible time resolution for a sample calibrated age (Figure 2).

The intervals of the improved time resolution found for Holocene samples using IntCal13 (for time interval widths below 50 years of the calibrated age) are reported in Table 2, including minimal width for given interval of the sample origin.

Several samples of tree rings, late wood (lw) from the given year, excluding early wood sample 18_121B (ew), dated by dendrochronology were analyzed with uncertainties above 20 years. A summary of the results obtained is reported in Table 3.

¹Following OxCal style of calibrated date reporting.

Table 2 Time intervals with the time resolution below 50 years and the minimal possible width in a given interval.

Intervals (2σ)	Minimal width (yr)
1418–1446 AD	24
1257–1281 AD	29
1016–1018 AD	48
642–661 AD	34
400–395 BC	39
809–794 BC	26

Table 3 Results of ^{14}C analyses of tree rings dated by dendrochronology.

CRL lab code	AMS ^{14}C lab code	Dendrochron. age, years AD	^{14}C age \pm (1σ), yr BP	Calibrated age (2σ), years AD	Width of interval, years
CRL18_120A	DeA-15959	648, lw	1470 ± 25	552–642	90
CRL18_120B	DeA-15960	653, lw	1426 ± 23	592–657	65
CRL18_120C	DeA-15961	657, lw	1415 ± 25	597–661	64
CRL18_121A	DeA-15962	1013, lw	1065 ± 26	946–1021	75*
<i>CRL18_121B</i>	<i>DeA-15963</i>	<i>1015, ew</i>	<i>1132 ± 24</i>	899–924 846–986 827–841 778–790	50*
CRL18_121C	DeA-15964	1018, lw	1062 ± 24	949–1022 901–922	73*
CRL18_122A	DeA-15971	1266, lw	747 ± 22	1244–1286 1226–1232	42*
CRL18_122B	DeA-15972	1273, lw	767 ± 21	1223–1279	56
CRL18_122C	DeA-15973	1280, lw	724 ± 22	1260–1294	34

*The main interval.

A relation between the results of radiocarbon dating and the year of sample origin, as determined by dendrochronology is shown in Figure 3. As evident from the diagram, there is a visible systematical shift of radiocarbon results toward older periods for both sample sequences 18_120 and 18_121. We have also shown all the results of the radiocarbon dating visualized on the calibration curve in Figure 4.

DISCUSSION

Using radiocarbon calibration curve IntCal13, six intervals providing time resolution of the sample origin below 50 years were found, with an uncertainty in ^{14}C analyses of ± 15 years (^{14}C years) and two sigma interval (± 15 years corresponding to analyses of recent samples) for the period covering the last three thousand years (see Table 2). In the case of older periods, 13 time intervals enabling higher time resolution (below 100 years) during the rest of the Holocene (Figure 2a) and 4 intervals below 300 years from 10 ka BC to 24 ka BC (including a wide interval from 10,473 to 12,175 BC) with a subinterval 10,744 to 10,775 BC

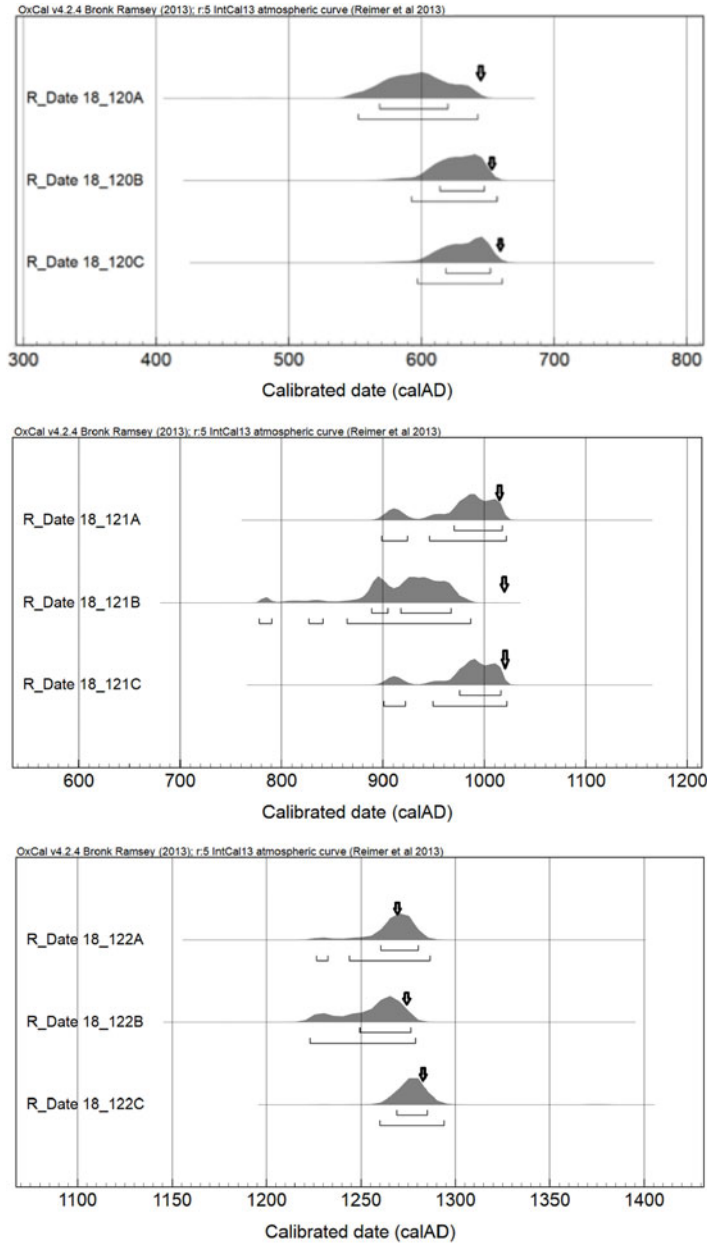


Figure 3 Comparison of radiocarbon dating results (probability density curves) of tree rings with dendrochronology determination (bold arrow) of the year of a given sample origin.

with possible resolution below 100 years (Figure 2b) were found. On the residual part of IntCal13 calibration curve, we found two local minima with the best time resolution of <400 years.

Using nine dendrochronologically dated tree-ring samples, with uncertainties of ^{14}C analyses using AMS in the range 21–26 yr BP, we obtained eight results which are in agreement with

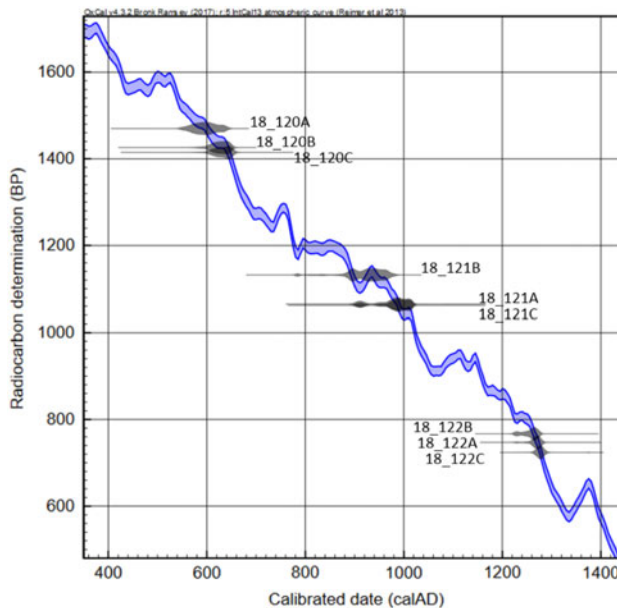


Figure 4 Visualization of the radiocarbon dating results on steep parts of the calibration curve.

dendrochronology determination (although the resulting time interval of the sample 18_120A is three years below the year found by dendrochronology). All these samples in good agreement were prepared from late wood of the investigated tree rings. In the case of the sample prepared from early wood, a significant difference exceeding 29 years between dendrochronology and “older” radiocarbon dating was found. Despite the fact that uncertainties of ^{14}C analyses substantially exceeded 15 yr BP, the narrowest resulting time interval was 34 years for the sample 18_122C, compared to 29 years obtained for the best possible resolution for this time period.

As evident from Figure 3, there is a visible systematical shift of the radiocarbon dating resulting intervals compared to dendrochronology results. This shift is caused by the systematically smaller ^{14}C activities of the samples analyzed (although on the border of the statistical significance). Such a shift can be probably given by the reduced routine of samples pretreatment, where only acid-alkali-acid leaching was used, without extraction of tree resins and bleaching routines. The reduced routine was applied due to limited amounts of material. Previously, it was shown that for most wood samples the results obtained with this simple method are comparable with more complicated treatments, which have the disadvantage of low yields (Hajdas et al. 2017). In the case of neighboring periods, when available time resolution is several times worse, systematical shifts of the resulting time interval will be several times smaller. Likewise, such cases can be judged against the possibility of pretreated sample contamination from the applied reagents, glassware or from laboratory surroundings, which increases with the number of sample preparation steps. In the case of the expected sample origin in the period with a possible fine time resolution, several different routines of the sample pretreatment should be carried out to get comparable results. If there are no significant differences between the results, all these

routines can be sufficient for a given sample type, both most “robust” and most “vulnerable”. In the opposite case, several different more “robust” routines should be compared, if the sample quantity would be sufficient. Samples with the origin from “fine time resolution period” seem to be the most sensitive for the comparison of the sample processing routines.

Early wood in oaks at the Czech Republic territory grow from April until the beginning of June. The late wood starts about the first half of June and is usually finished in August. During a cold period of a given year (including spring), decreasing activities of the atmospheric ¹⁴CO₂ have been observed since the beginning of monitoring of this parameter in the 20th century (Levin et al. 1995; Levin and Kromer 1997, 2004). It can be presumed that annual seasonal changes of ¹⁴CO₂ activity occurred also in the past (McDonald et al. 2018). The second influence, causing decreased ¹⁴C activities of early wood can be connected with elevated amount of allochthonous carbon forms (especially humic and fulvic acids) due to the increased porosity of such samples (Vavřík et al. 2008; Shchupakivskyy et al. 2014). Hence, in this case it would be interesting to carry out a sample pretreatment using extended routines (bleaching and extraction of resins). Nevertheless, to obtain more conclusive data, a greater collection of early wood samples from this period would be needed.

In general, for the purpose of accurate radiocarbon dating, samples with a well-defined and short period of sample formation can be used, e.g. late wood from tree rings, fine tree branches (representing only several years of growth), plant seeds, plant macrofossils, animal/human teeth, fatty acids, or fine sediment layers with distinctive summer/winter lamination.

In contrast, there are samples with a longer period of growth (several decades) and with unclear speed of sample formation/accumulation such as charcoal powder, bone collagen, pollen concentrates, carbon from historical steel, peat and karst sinters. In this case, the uncertainty of ¹⁴C analyses should be expanded in the relation with the estimated period of the sample formation. Results of radiocarbon dating then should be reported as intervals corresponding to time mean of the sample formation, supposing processed and measured sample fraction to be homogenous.

When a sufficient sequence of (tree-ring) samples with known time gap is available, the wiggle-matching method (WM) provides results with a time resolution of several years for the Holocene period (Hogg et al. 2017). Although the dendrochronology dating results in an even better time resolution of a single year, the wiggle-matching method provides absolute dating independently of local calibration scales (in the general). Demands on quality of wood material are relatively low in the case of WM method, but compared to dendrochronology dating, it usually requires slightly shorter sequences. Comparing these methods, the radiocarbon dating can provide the best time resolution of about 25 years for the last three thousand years. In the older periods the time resolution is substantially worse. The periods of rapid changes in atmospheric ¹⁴C are of relatively short duration (Figure 2d) and correspond only to short parts of the calibration curve. Therefore, finding samples from such periods is, in general, rare. However, if such a sample is found, and it is suitable material, the steep part of the calibration curve applies and this can result in excellent resolution, several times better than the usual results of radiocarbon dating.

The steep parts of the calibration curve also provide a way to select possible significant geographical differences of calibration curves (Guilderson et al. 2005). In this way, local

curves can be also tested in the vicinity of greater long-term carbon sinks as peat bogs, greater freshwater bodies, karst systems, permafrost, volcanic areas, etc., where past releases of CO₂ with depleted amount of ¹⁴C could be expected. As discussed in the introduction, due to the relatively rapid changes of ¹⁴C activity (i.e. steep parts of the curve), corresponding samples also provide a pretreatment testing material, where the possible presence of persistent interfering older or younger carbon forms is several times better observable than in the samples from neighboring periods. This applies especially to the samples of late wood from oak trunks.

Future attention should be paid to the methodology of expanding uncertainties of analyses in the case of dating sample types with longer periods of growth (especially if the time of sample growth is not so clear, e.g. pollen concentrates, fine charcoal particles, organic compounds from paleosoils, etc.) or relevance of sample types, including various tissues of trees, herbs or animals species. It could be possible to report a mean time of sample growth, if the growing time is regular.

SUMMARY

The IntCal13 calibration curve was employed to check the possible time resolution which is available for the radiocarbon dating method, assuming a resulting uncertainty of ¹⁴C analysis about ± 15 ¹⁴C years for recent samples. Several time periods were found corresponding to the steep parts of the calibration curve, which enables a fine time resolution below 50 years (for samples with origin from the last three thousand years). Corresponding samples can provide a good tool for a selective validation of the future calibration curves in view of significance of geographical differences. Likewise, such samples seem to be relevant perceptible material also for testing of sample pretreatment routines in laboratories. In connection with fine time resolution, we proposed a basic list of the proper samples with a short and well-defined period of growth. Dating interpretation of different types of samples should be a subject for future discussion about rules/methodology for expanding uncertainties, better understanding of results (which moment was dated), or time characterization of aggregated samples (concentrates, sediments, paleosoils, etc.).

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