ABSOLUTE DENDROCHRONOLOGICAL SCALE FOR PINE (*PINUS SYLVESTRIS* L.) FROM UJŚCIE (NW POLAND), DATED USING RAPID ATMOSPHERIC ^{14}C CHANGES

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ABSTRACT. Radiocarbon (^{14}C) analyses are commonly used to determine the absolute age of floating tree-ring chronologies. At best, with the wiggle-matching method, a precision of 10 years could be achieved. For the early Middle Ages, this situation has been markedly improved by the discovery of rapid changes in atmospheric ^{14}C concentrations in tree-rings dated to 774/775 and 993/994 AD. These high-resolution changes can be used to secure other floating tree-ring sequences to within 1-year accuracy. While a number of studies have used the 774 even to secure floating tree-ring sequences, the less abrupt 993 event has not been so well utilized. This study dates a floating pine chronology from Ujście in Wielkopolska (Greater Poland) (NW Poland), which covers the 10th century period and is critical for studies on the beginning of the Polish State to the calendar years 859–1085 AD using the changes in single year radiocarbon around 993/4 AD.

KEYWORDS: dendrochronology, Miyake effect, radiocarbon AMS dating, tree rings.

INTRODUCTION

In Poland, the dendrochronological method is often used for the precise dating of building structures, historic wooden monuments and artifacts. This is particularly important considering the period covering the beginnings of statehood in Poland, which was born in the region of today's Wielkopolska (Kara and Krąpiec 2005; Kara 2009). Since the 1990s, dating oak wood samples from Wielkopolska (Greater Poland) has been relatively easy because the long master chronology for the region has already been constructed, covering the period 449-1998 AD (Krąpiec, 1998). However, the possibility of dating pine wood looks, quite different. Combining the efforts of several years, a pine chronology for Wielkopolska has not been constructed due to the lack of suitable wood samples. This leaves a gap for this very important region between master pine chronologies in the neighboring areas which range 1091–2004 AD for Małopolska (Szychowska-Krąpiec 2010) and 1106–1994 AD for Kujawy and Gdańsk Pomerania (Zielski 1997).

This situation changed in 2008 due to extensive rescue archaeological excavations due to construction of a new sewage network required by the municipality in Ujście, covering the sub-stronghold zone (Rybacka Street) and the Piast stronghold castle (Old Market) (Figure 1).

Altogether, approximately 800 wood samples from a wide spectrum of species were gathered from all excavations, of which 300 fulfilled the requirements of the dendrochronological

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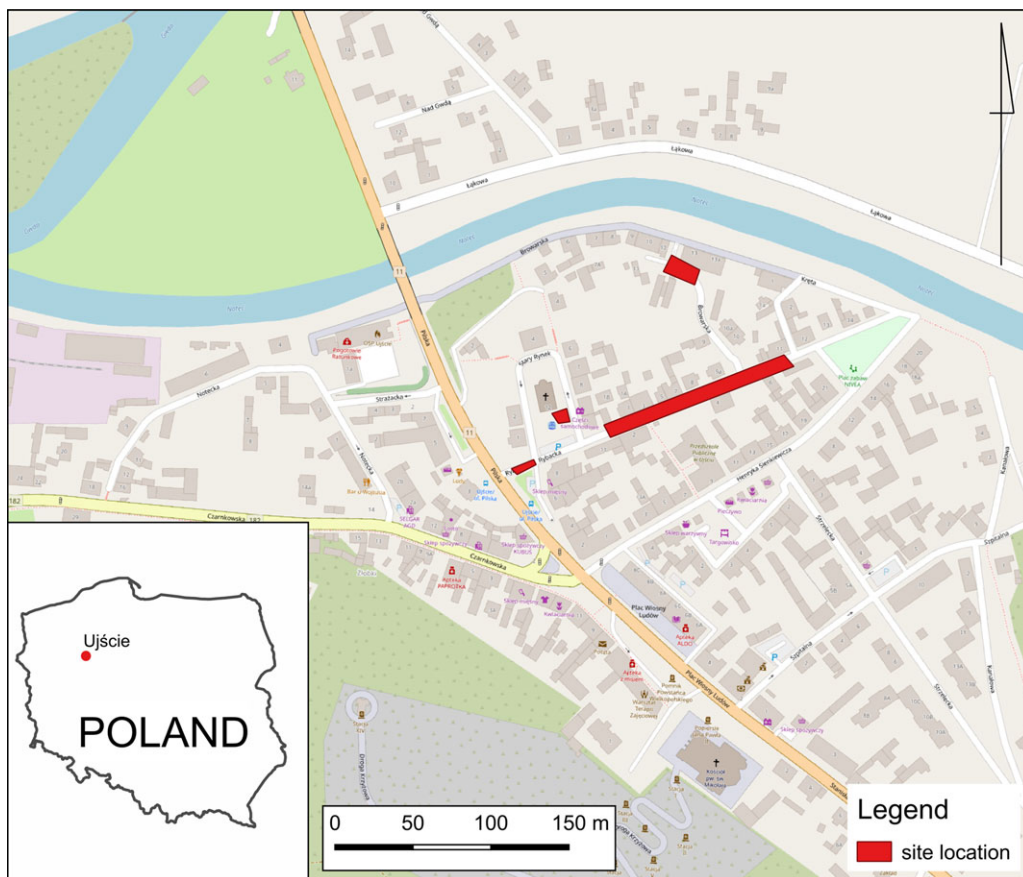*Corresponding author. Email: mkrapiec@agh.edu.pl.

Figure 1   Location of the site Ujście and archaeological trenches in which pine wood was encountered and sampled for analyses.

method, i.e. an adequate number of increments and a highly conserved wood structure. Among them, the elements made of pine wood predominated and should be considered as a special case, since generally, wooden structures from that time were made of oak wood. Presumably, the less durable pinewood was used in earthen structures because of a deficit of oak wood. The area around Ujście is characterized by poor sandy soils, predominated by pine forests and with the occasional mixed forests with oak.

Preliminary results, based on archaeological artefacts and dendrochronological dating of the oak wood, indicated that the pine wood found in the structures examined should allow to extend the existing pine master chronologies back in time.

Based on the most converging dendrochronological sequences, a floating pine chronology 2U_02A, covering 227 years, was compiled (Bolka and Krąpiec 2012). The comparison of the 2U_02A chronology with the master chronologies developed for pine from the neighbouring areas, i.e. the regional chronology from northern Poland covering the period 1106–1991 AD (Zielski 1997) and northern Germany 924–1995 AD (Heussner 1996), did not allow the identification of synchronous positions. In such a situation, the most suitable

dating method was the wiggle-matching method, with which the period represented by the curve2U_02 was determined for the years 860–1080 (±10) cal. AD (Bolka and Krąpiec 2012).

Precise dating, with a 1-year accuracy, of the chronology w 2U_02A, produced from almost 100 individual tree-ring sequences, would be of substantial importance for Polish regional archaeology. The chronology covers an important transition to state organization in the early Middle Ages and is urgently needed to refine a construction sequence from a series of defensive pine-constructions which could be dated to the year with a regional master pine chronology.

This purpose could be achieved by taking advantage of the sudden increase in the radiocarbon (^{14}C) concentration in annual tree rings, described by Miyake et al. (2012, 2013, 2014, 2017) and Park et al. (2017). Studies on the radiocarbon content in the annual growth of different tree species growing in both northern and southern hemispheres have demonstrated that the so-called "Miyake effects" dated to 661/660 BC, 774/775 AD and 993/994 AD are of a global extent (Usoskin et al. 2013; Jull et al. 2014; Güttler et al. 2015; Büntgen et al. 2016, 2018; Fogtmann-Schulz et al. 2017). Similar results have also been determined in annual increments of oaks growing around Krakow (S Poland) (Rakowski et al. 2015, 2018, 2019). The Miyake 775 AD peak was successfully used for dating of the period of building the Holy Cross chapel in Val Müstair in Switzerland (Wacker 2014) as well as the Baitoushan Volcano eruption in China (Hakozaki et al. 2018).

The sudden increase of 1.5% in atmospheric radiocarbon concentration between AD 774 and 775 is the strongest observed Miyake effect. The increase in radiocarbon concentration between AD 993 and 994 was less intense and amounted to 0.9% (Miyake et al. 2013, 2014). Both of the events have global character and show the same characteristic shape of increase in radiocarbon concentration, which made possible to use them for high-resolution wiggle-matching (HR wiggle-matching).

As the chronology 2U_02A does not cover the effect 774/775 AD, it was possible to date the sequence using the weaker increase of the ^{14}C contents in 993/994 AD ($\Delta^{14}C = 6.8‰$), which was detected in annual rings of subfossil oaks from Kujawy near Kraków (Rakowski et al. 2018).

MATERIAL AND METHODS

The wood sample selected for the analyses had the form of a slice, was labeled with the laboratory code 2UJC141 and obtained from a beam, which was a structural element of a timber trackway.

Measurements of annual growth rings were made with 0.01-mm accuracy using a DENDROLAB 1.0 apparatus, and subsequently, the ring-width sequences were processed using the software packages TREE-RINGS (Krawczyk and Krąpiec 1995), TSAP (Rinn 2005), and DPL (Holmes 1999). The sample contained 101 annual rings, the width of which allowed the separation of wood samples of the appropriate mass to obtain several mg of alpha cellulose. The dendrogram (annual increment width graph) of this sample has a clear resemblance to the 2U_2A chronology between 108 and 208 relative years, with t = 5.5, GL = 70%, where the t-value represents the significance of the correlation of two series in relations to their overlap and should not be below 3.5 (Baillie and Pilcher 1973). The GL (*Gleichläufigkeit*) was developed as a special tool for the cross-dating of tree ring series. The degree of similarity based on the positive or negative trend of each width is expressed as a percentage of the number of intervals (Eckstein and Bauch 1969), as shown in Figure 2.
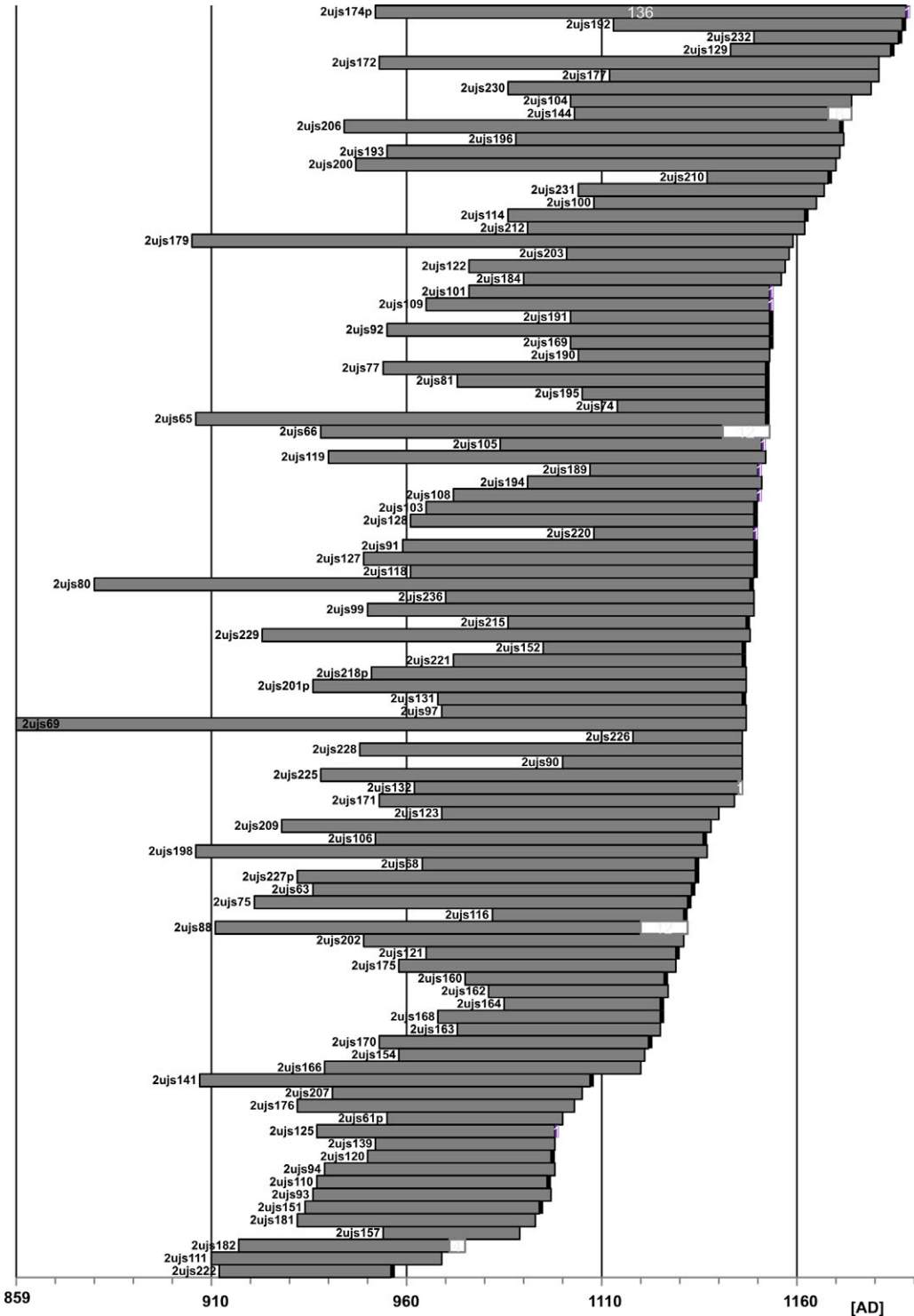
Figure 2   Diagram of the time extent of growth sequences forming the pine chronology 2U_02A from Ujście.

From the sample 2UC141, 36 annual rings were separated; their nos. 22–58 were equivalent to 125–165 relative years of the chronology. Of the selected rings, we extracted α-cellulose, a preferred component of wood for high-precision analysis of radiocarbon. Cellulose represents the most stable part of the wood, since carbon in the cellulose fraction does not cross ring boundaries, whereas other carbon fractions, such as lignin and hemicelluloses, may cross ring boundaries or be exchanged with the atmosphere after formation (Loader et al. 1995; Santos et al. 2001; Nemec et al. 2010).

For cleaning and α-cellulose extraction, we used a method described previously (Green 1963), with some modifications. To expedite the separation of single cellulose fibres from wood samples and thus to increase the penetration of reagents, an ultrasonic bath was used (Pazdur et al. 2005). Wood samples were dried, cut into shavings, and subsequently weighed and placed into glass tubes. Each sample was placed into a glass tube with deionized water, sodium chlorite, and 1% HCl. The tube was then placed in an ultrasonic bath at 70°C for 1 hr, and subsequently, sodium chlorite and 1% HCl were added to each sample. This procedure was repeated five times, and the samples were then rinsed with hot deionized water up to a neutral pH. This process removes most of the lignin, and the remaining sample consists mostly of holocellulose. For the next step, 10% NaOH solution was added to the samples, which were then placed into an ultrasonic bath at 70°C for 45 min. After the solution was removed, the sample was rinsed with cold deionized water. In the following step, 17% NaOH solution was added, and the samples were placed into an ultrasonic bath at room temperature for 45 min. Subsequently, deionized water was added; this step completely removes the remaining lignin and holocellulose. The samples were then rinsed with hot deionized water up to a neutral pH. Finally, 1% HCl was added to the samples, and the samples were again rinsed to a neutral pH. The obtained α-cellulose was dried overnight at 70°C (Michczyńska et al. 2018).

After the pretreatment, around 4 mg of α-cellulose, extracted from each sample, was combusted to CO_2 and subsequently reduced to graphite (Krąpiec et al. 2018). The resulting mixture of graphite and Fe powder was pressed into a target holder for AMS ^{14}C measurements. All prepared targets contained approximately 1 mg of carbon and were measured at the Center for Applied Isotope Studies at the University of Georgia, USA (Labcode UGAMS; Cherkinsky et al. 2010). The ^{14}C contents are reported as $\Delta^{14}C$ in per mil (‰) deviations from the standard sample, 0.7459 activity of NBS oxalic acid (SRM-4990C). Age correction and isotopic composition correction were calculated following formulas presented elsewhere (Stuiver and Polach 1977). The correction for isotopic composition was made based on $\delta^{13}C$, measured with an AMS system.

RESULTS AND DISCUSSION

In the first stage, the measurements were made for six α-cellulose samples, separated from the increments representing 33, 37, 42, 47, 52, and 57 relative years. Dating results indicated that the older increments selected contained the diagnostic signal from 994/5 AD. In the two following sessions, α-cellulose from rings 21–41 relative years was measured (Table 1).

To fix the floating chronology, we analysed the normalized sum of squares of offsets of the sequence 2U_2A from Ujście $D^{14}C$ and a combination of other available $D^{14}C$ data sets; we used data from the Kujawy tree ring sequence (Rakowski et al. 2018) and two sequences from Japan (Miyake et al. 2013, 2014).). Normalisation was obtained by dividing

Table 1 ^{14}C concentrations of measured tree rings from Ujście (northwest Poland). The calendar ages of the annual tree rings were obtained using comparison with the radiocarbon concentration during the Miyake effect around AD 993/994.

UGAMS no.	2UJS141 ring no.	F^{14}C	ΔF^{14}C	^{14}C age (BP)	Obtained age AD
41469	22	0.8771	0.0023	1084 ± 21	986
41470	23	0.8792	0.0023	1064 ± 21	987
41471	24	0.8706	0.0023	1146 ± 21	988
41472	25	0.8735	0.0023	1118 ± 21	989
41473	26	0.8771	0.0023	1084 ± 21	990
41474	27	0.8683	0.0023	1167 ± 21	991
36953	28	0.8736	0.0023	1117 ± 21	992
36954	29	0.8803	0.0023	1054 ± 21	993
36955	30	0.8842	0.0023	1018 ± 21	994
36956	31	0.8799	0.0023	1057 ± 21	995
36957	32	0.8769	0.0023	1086 ± 21	996
35620	33	0.8808	0.0022	1049 ± 21	997
36958	34	0.8751	0.0023	1103 ± 22	998
36959	35	0.8780	0.0023	1076 ± 21	999
36960	36	0.8836	0.0023	1023 ± 21	1000
35621	37	0.8801	0.0022	1055 ± 21	1001
41475	38	0.8704	0.0025	1147 ± 24	1002
41476	39	0.8721	0.0026	1131 ± 24	1003
41477	40	0.8713	0.0025	1139 ± 24	1004
41478	41	0.8703	0.0025	1149 ± 23	1005
35622	42	0.8747	0.0022	1107 ± 21	1006
35623	47	0.8751	0.0022	1103 ± 20	1011
35624	52	0.8754	0.0023	1100 ± 21	1016
35625	57	0.8792	0.0023	1064 ± 22	1021

sum of squares by number of offsets used for the calculation of the sum. We tried three different approaches. In the first approach, for each year, starting from 981 AD to 1013 AD, the average D^{14}C of the three sequences was calculated. When no data were available for a particular year, the final "average" sequence was calculated using two or one data points (Figure 3). The Ujście sequence starting point was then shifted year by year, and for each shift, the sum of squared offsets of Ujście and the "average" data points was calculated. The sum of squared offsets was then normalized by the number of offsets used in the calculation of the sum (Figure 4, x – symbols in the graph). In the second approach, we made similar calculations, but only the offsetsfor the Kujawy and Ujście data sets were analysed (Figure 4, stars). In the third approach, we separately calculated the offsets for each available data point for the Ujście data set and for any other data set. Subsequently, we calculated the squared sum of all the differences and normalized it (Figure 3, triangles). For each approach, the minimum of the normalized sum was obtained for the Ujście sequence starting in the year 986 AD (Figure 4). On this basis, it can be concluded that the pine chronology 2U-02A from Ujście was accurately dated to the period 859–1085 AD. Its value for absolute dating is difficult to overestimate; it serves as a tool for precise dating, but also as a potential record to be used in dendroclimatic studies both in traditional terms, using tree ring width variability as well as the contents of stable isotopes.
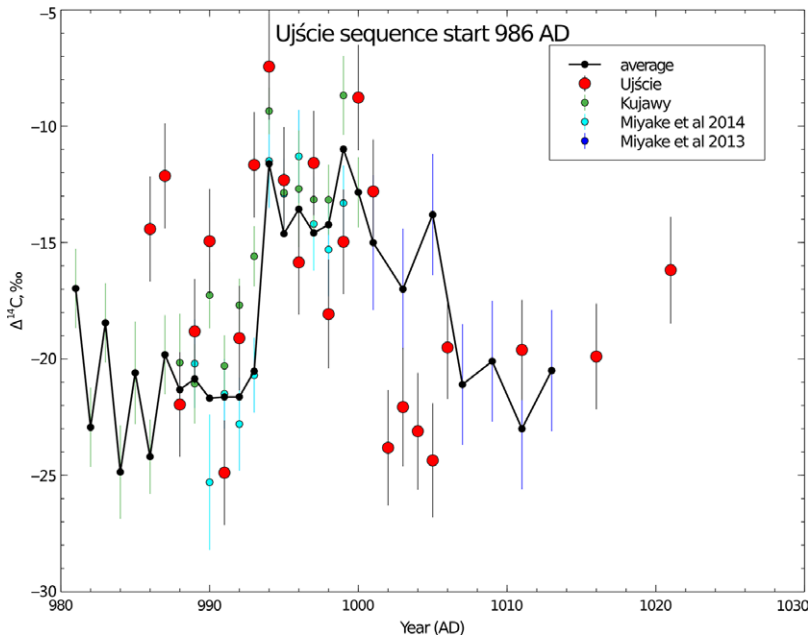
Figure 3 Δ^{14}C in Japan (cedar, Miyake et al. 2013, 2014) and Kujawy, Poland (*Quercus* sp., Rakowski et al. 2018) plus average values with results of measurements from this study (Pinus sylvestris, Ujście site).
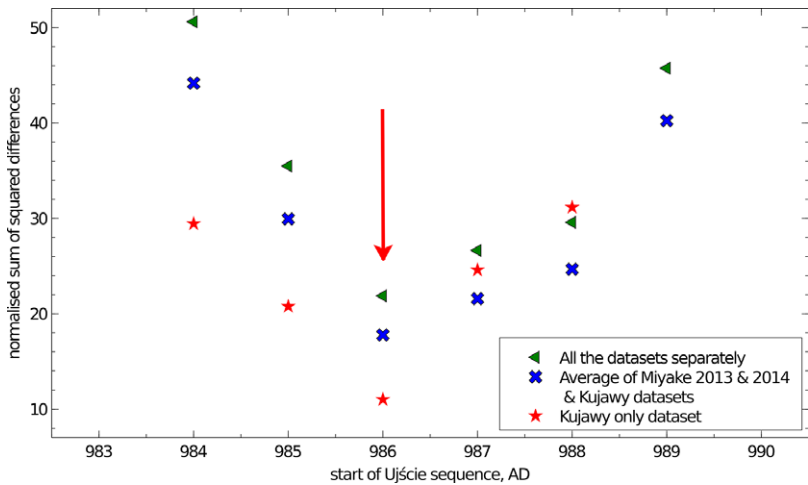

Figure 4   Normalized sum of squared distances between data sets for a given starting position. Kujawy data after Rakowski et al. (2018), Japan data sets after Miyake et al. (2013, 2014).

Dating using the Miyake effect (2013) allowed us to assess the quality of the earlier dating of the Ujście chronology with the wiggle-matching method, based on bulk wood samples containing several increments, with the LSC technique using the Hidex 3000 SL low-background spectrometer (Krąpiec and Walanus 2011). The result of the WM match fits

perfectly and deviates only by 1 year from the date obtained using the Miyake approach. Although such high convergence is probably an exceptional case, it still demonstrates that the LSC date of wood prepared with the AAA method does not deviate from the result obtained with the AMS technique and can be considered as a cheaper alternative to the pre-placement of sequences on the timescale.

We demonstrate that dating of a floating chronology containing annual rings from the period of abrupt changes in the radiocarbon content (so-called "Miyake effects") allows precise dating, but identification of the correct position requires the analysis of about a dozen annual increments. In cases where a hypothetical dendrological date was confirmed, the number of dated tree rings could be reduced to perhaps 5 to 10, as demonstrated by the study of Wacker et al. (2014). Due to the characteristic shape of the increase in radiocarbon concentration, and its global character (Büntgen et al. 2018), those events could be used for high-resolution wiggle-matching, resulting in 1-year resolution.

One has to keep in mind that the ^{14}C excursion event occurring in 993/994 AD has an amplitude of less than 10‰ (Miyake et al. 2013, 2014), which means that uncertainty of radiocarbon measurements for each sample in the analysed tree-ring sequence should be better than 3‰.

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