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DATING OF WOODEN HERITAGE OBJECTS IN THE GLIWICE ^{14}C AND MASS SPECTROMETRY LABORATORY

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ABSTRACT. We present case studies on three objects of high importance for cultural heritage in southern Poland, dated in years 2018–2022 at the Gliwice ^{14}C and Mass Spectrometry Laboratory with radiocarbon (^{14}C) and dendrochronology methods. The first was a richly ornamented wooden cane, discovered during excavations on the market in Bytom city. The cane can be associated with medieval court proceedings. The archaeological context indicates the 13th century AD, and the ^{14}C result corresponds perfectly with this time, confirming that it is the oldest object of this type in Poland. The second was a 4-m-tall oak column from St. Leonard Church in Lipnica Murowana, a UNESCO heritage site. The local story said it was previously devoted to Światowid, a pagan deity. Our analysis excluded the pre-Christian age, as the tree was felled no earlier than the late 15th century, which is in agreement with historical records. The third was a wooden Saint Lawrence Church in Bobrowniki. The presbytery was covered with up to five layers of polychromic paintings, some of high artistic value. We dated three samples from the original wooden board, and by wiggle-matching, the calibrated age interval was narrowed to 1731–1754 cal AD.

KEYWORDS: dendrochronology, heritage, radiocarbon dating, wood.

INTRODUCTION

The trees as annually resolved archives form an inexhaustible resource of datasets for many branches of Earth and environmental science. Dendrochronology, derived from wood biology, is the science whose primary task is the dating of wood (Ważny 2001). Radiocarbon (^{14}C) dating and dendrochronology have been linked since the first steps of the ^{14}C method, and the history of this symbiosis was recently reviewed by Pearson et al. (2022). From the radiocarbon perspective, tree rings investigations enabled the reconstruction of past ^{14}C content necessary for calibration of radiocarbon dates (Reimer et al. 2020; van der Plicht et al. 2020; Hua et al. 2022), provided the insight into the carbon cycle on the Earth (e.g., Levin and Hesshaimer 2000) and delivered data on changing astronomical variables such as cosmic ray intensity (Miyake et al. 2012). ^{14}C concentration in growth-rings were explored as records of environmental and climatic conditions (e.g., Pazdur et al. 2007) as well as anthropogenic influence on the environment (e.g., Ežerinskis et al. 2018; Sensuła et al. 2018; Kontuľ et al. 2022).

The age determination of wooden objects of heritage was one of the first reported applications of the radiocarbon method, forming the famous "curve of knowns" by Libby (1961). Radiocarbon dating of wooden archaeological samples was also performed in the ^{14}C and Mass Spectrometry Laboratory in Gliwice since its early days (Mościcki and Zastawny 1976).

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Many studies have demonstrated a successful approach in obtaining accurate ages of wooden objects, however, the results need to be treated with caution. The ^{14}C method provides the age of tree felling, and not the age of any work performed like sculpting, forming or using for construction. The development of accelerator mass spectrometry technique (AMS), which allowed the loss of material necessary for this destructive analysis to be minimized, significantly enhanced ^{14}C dating on precious cultural heritage objects. In particular, when ^{14}C supported by dendrochronology allowed application of wiggle-matching procedures, performed with use of the Bayesian analysis (Bronk Ramsey et al. 2001), the high precision and reliability was reached (e.g., Quarta et al. 2010; Fukuyo et al. 2019; Matskovsky et al. 2019). However, ethical issues arise with respect to the authentication of such objects, particularly those in private possession or destined for the market (Huysecom et al. 2017). Following UNESCO conventions on heritage objects, the ^{14}C and Mass Spectrometry Laboratory in Gliwice follows the recommendations for diligence protocol formulated by Hajdas et al. (2019). In all cases, the samples were collected following proper allowances and in the presence of persons representing interested parties: architects, historians, conservators, museum curators and representatives of the Catholic Church, whichever was applicable. They provided knowledge of the historical context and expertise in interpretation of results, and most of them coauthor this manuscript. Presented case study describes radiocarbon and dendrochronological dating of three wooden objects of significant cultural heritage. These objects are from the area of southern Poland and their analyses were conducted between AD 2018 and 2022 at the Gliwice ^{14}C and Mass Spectrometry Laboratory.

CONTEXT OF THE SAMPLES

Cane from Bytom Market

The richly ornamented wooden cane was discovered in AD 1998 during archaeological excavations on the main market in Bytom city (Upper Silesia, 50°20′54″N 18°54′56″E), founded in AD 1254 under German law. The cane was found in a Trench No. 1, measuring 9.5 by 38 m, in Layer No. XIII, Bedding No. IV, with a large accumulation of antiquities, including numerous wooden objects. The Bedding No. IV was placed on a clay floor and covered with a thin insulating layer made up of small twigs and wood chips. This layer was overlaid with chopped wood branches, forming a compact surface of the medieval market place. Three alternating layers of clay, sand, gravels, and paving stones were located above Bedding No. IV, up to the latest cobblestone surface. The archaeological context implies that Layer No. XIII corresponds to the 13th century AD, when small wooden stalls were in use. Accurate dating of these objects was possible due to previous dendrochronological studies of structural elements and analysis of collected movable relics, as summarized by Andrzejewska (2000).

The cane is 57.7 cm long and has a diameter of 2.9 cm by the ornament. Two distinct incisions, most probably intentionally made, caused a cavity in ca. 1/3 of the length (see Figure 1A). The cane is supposed to have been used in court proceedings in the Germanic law circle, and the cuts were aimed at weakening the cane for easier breakage. Historical sources document the judge's practice to break the cane (named *Rechtsstab, Gerchichtsstab*) over the head of the accused at the time of the sentence.

The cane is currently displayed for the public at the Museum of Upper Silesia, Department of Archaeology, Bytom. For sampling, it was brought to ^{14}C Laboratory, examined visually, photographed, and a piece of wood (1.68 g; GdA-6743) was cut with a knife from the area near

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Figure 1 Sampling details with sample position indicated by dotted ellipses and laboratory numbers; (A) wooden cane from Bytom; complete cane and sampling area; (B) oak column from Lipnica Murowana: sketch of the column, drilling point and increment core with indicated tree rings boundaries; (C) wooden board from Bobrowniki: front view with paint, fragment of back and view on the polished side (marked in light green) and previous LSC ^{14}C date.

the incisions (Figure 1A). Further interference in cane, for example, cutting, polishing, or drilling, was omitted to protect the object; thus the number of tree rings sampled remains unknown.

Column from Church of St. Leonard in Lipnica Murowana

Lipnica Murowana town (Lesser Poland region, 49°51′28″N 20°31′45″E) was founded in AD 1326 by king Władysław Łokietek. However, an ecclesiastical organization have been functioning in the area for much longer, and some sources suggest even half of the 12th century. Several precious cultural heritage sites located in Lipnica Murowana have survived to our time, including the timber Church of St. Leonard, which is a UNESCO World Heritage Site since AD 2003. The thorough renovation of the church was carried out in the 1950s and currently it is in an excellent state of preservation.

The detailed description and 3-D scans of the church are provided by Budziakowski et al. (2020). In brief, the church was constructed as a timber building with wooden log walls placed on a stone base, and a steep roof. The interior space was covered with a flat, timber decks with coffers to its sides. The decks were painted and feature rich ornamentation. The main altar

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originates from the beginning of the 16th century and was furnished with a triptych painting depicting the church's patron, St. Leonard of Limoges, along with St. Lawrence of Rome and St. Florian, and side panels illustrating scenes from the saint's life. The back side of the altar is supported by 4-m-tall oak trunk with a rounded top (Figure 1B). At the base the altar and column are affixed with metal braces. Several cuts on the column can be observed: a wedge-shaped cut near the top, carvings depicting rosettes. A socket in the upper part may be supposed to be used to affix a horizontal beam, which indicates the object had been a cross or an element of a larger structure previously. The column lacks other symbols or ornaments, particularly any that could be linked with a pre-Christian culture. Nevertheless, the local story associated with the church states that a column was reportedly an element from an older Slavic shrine and was to depict or symbolize the figure of a Slavic deity called Światowid.

The column was sampled on site with an increment borer in AD 2018, and the 5-mm-diameter core was extracted, measuring ca. 12 cm in length. The dendrological analyzes indicated that the core comprised 16 tree rings. In order to avoid potential contamination of the external layer, ^{14}C dating was performed using a fragment of the internal part of the core, which encompassed two incomplete rings (Figure 1B; GdA-5702). Unfortunately, for such a short sequence, the dendrochronological matching with a reference chronology was not possible. The results formed part of the Budziakowski et al. (2020) publication aimed at the architecture scientist, and we aim here to present this interesting case study to a wider radiocarbon community.

Board from Saint Lawrence Church in Bobrowniki

Bobrowniki village was founded in AD 1273 in the Upper Silesia (50°22′48″N 18°59′41″E). Saint Lawrence Church located there is a unique wooden church with two towers. The earliest notes about its existence come from AD 1619, and the inscription on the ceiling beam states, "Built in 1669, restored in 1857." The church has a complicated renovation history, part of which remains undiscovered, with most pronounce reconstructions made in 19th century, when the wood coming from 16th century was reused for wall building (Konieczny 2010), and the two towers were erected. The presbytery of the church is constructed by wooden boards covered by five layers of polychromic paintings, which are an object of ongoing conservation works (Poloczek-Imielińska 2021). The boards do not show any features indicating on being reused material—they are well fitted, the junctions are precise without any shifts or damage, and the first polychromic layer shows stylistic continuity, as far as it was discovered. The following second layer is poorly preserved and also shows some figures, which was covered by third painting. This one was made in AD 1923 by Otto Kowalewski, a renowned Silesian painter, as documented by the signature and date placed by one of the paintings and also was present on a photograph taken in AD 1930 (Poloczek-Imielińska 2021). This layer is also in a poor state of preservation and was covered in 20th century by two further paint layers without figures, and of relatively low artistic value.

This oldest layer is in the best state of preservation, and of the highest artistic value. The exposed fragments show baroque polychrome paintings, which may come from ca. 17th century, or else be baroque-styled 19th century works. The analyzes of pigments from the first layer were inconclusive, reported as "non-dating," meaning used from the antiquity. The first attempt to radiocarbon dating a fragment of wooden board in AD 2011 with LSC technique yielded a wide range of possible calibrated ages (see Table 1 and Figure 4), and the present study was aimed at narrowing this range.

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Table 1 Results of radiocarbon dating and calibrated age ranges for independent and modeled dates.

Lab ID	Sample name	Tree species/ dated fraction	^{14}C age BP	Calibration results AD	Calibration results AD modeled
Cane from Bytom Market					
GdA-6743	Cane Bytom	Yew (*Taxus baccata* L.) Holocellulose	720 ± 30	*68.3% probability* 1271 (68.3%) 1296 *95.4% probability* 1230 (2.5%) 1244 1257 (84.2%) 1305 1365 (8.7%) 1384	*n/a*
Column from Church of St. Leonard in Lipnica Murowana					
GdA-5702	Lipnica 1	Oak (*Quercus* spp.) Alpha cellulose	370 ± 25	*68.3% probability* 1460 (44.8%) 1510 1592 (23.5%) 1618 *95.4% probability* 1454 (54.6%) 1524 1558 (40.9%) 1632	*Date + N(26,7)* *68.3% probability* 1487 (43.4%) 1540 1614 (24.9%) 1647 *95.4% probability* 1475 (54.5%) 1555 1581 (40.9%) 1660
Board from Saint Lawrence Church in Bobrowniki					
GdA-6744	Board 1	Pine (*Pinus sylvestris* L.) Holocellulose	120 ± 30	*68.3% probability* 1690 (16.5%) 1728 1808 (42.6%) 1894 1903 (9.2%) 1921 *95.4% probability* 1679 (26.0%) 1741 1752 (2.4%) 1764 1800 (67.1%) 1940	*68.3% probability* 1707 (36.1%) 1730 1901 (32.2%) 1928 *95.4% probability* 1701 (53.5%) 1772 1830 (2.7%) 1854 1892 (39.3%) 1940 *Agreement 95.2%*

(Continued)

Table 1 (*Continued*)

Lab ID	Sample name	Tree species/ dated fraction	^{14}C age BP	Calibration results AD	Calibration results AD modeled
GdA-6745	Board 2	Pine (*Pinus sylvestris* L.) Holocellulose	125 ± 30	*68.3% probability* 1688 (11.4%) 1712 1717 (6.1%) 1730 1806 (8.0%) 1824 1830 (32.5%) 1894 1904 (10.3%) 1925 *95.4% probability* 1675 (26.6%) 1744 1750 (3.4%) 1765 1798 (65.4%) 1942	*68.3% probability* 1716 (36.1%) 1739 1910 (32.2%) 1936 *95.4% probability* 1710 (53.5%) 1780 1839 (2.7%) 1862 1902 (39.3%) 1948 *Agreement 87.7%*
GdA-6746	Board 3	Pine (*Pinus sylvestris* L.) Holocellulose	215 ± 30	*68.3% probability* 1648 (27.6%) 1676 1742 (5.8%) 1750 1764 (34.9%) 1799 *95.4% probability* 1641 (33.8%) 1688 1730 (52.3%) 1807 1925 (9.4%) ...	*68.3% probability* 1731 (36.1%) 1754 1925 (32.2%) 1952 *95.4% probability* 1725 (53.5%) 1796 1854 (2.7%) 1878 1916 (39.3%) 1964 *Agreement 61.5%*
GdA-6744 6745 6746			*D_Sequence* – calibration with dendrochronological information		*68.3% probability* 1731 (36.1%) 1754 1925 (32.2%) 1952 *95.4% probability* 1725 (53.5%) 1796 1854 (2.7%) 1878 1916 (39.3%) 1964 Agreement: n =3 A_{comb} = 68.1% (A_n = 40.8%)

Table 1 (*Continued*)

Lab ID	Sample name	Tree species/ dated fraction	^{14}C age BP	Calibration results AD	Calibration results AD modeled
GdS-1270	Bobrowniki board/LSC	ABA treated wood	120 ± 40	*68.3% probability* 1690 (17.1%) 1728 1808 (51.2%) 1922 *95.4% probability* 1674 (31.7%) 1766 1774 (0.5%) 1776 1798 (63.3%) 1942	*n/a*

An original wooden board from the presbytery was removed and brought to the ^{14}C Laboratory for subsampling. The cross section surface of the wood of the board was polished, revealing a tree-ring structure which allowed to the determination of their number and width. Three samples were taken for ^{14}C dating (Figure 1 C): Board 1 (GdA-6744) comprised 3 rings followed by a gap of 9 rings, Board 2 (GdA-6745) comprised 4 rings, followed by a gap of 15 rings, and Board 3 (GdA-6746) comprised 3 rings. The weights of the samples were respectively: 630.62 mg, 807.52 mg, 619.42 mg.

METHODS

The wood samples were subjected to chemical preparation to obtain pure cellulose, taking into account the individual sample conditions and in general following the procedures described by Michczyńska et al. (2018). Overall, the preservation state of all samples examined was very good, in particular, for the oak wood of the Lipnica column (GdA-5702).

All samples have been shredded with a scalpel to small pieces (1–2 mm in size). Contamination with preservatives and/or paint could not be excluded for cane (GdA-6743) and board (GdA-6744, 6745, 6746) samples, so Soxhlet extraction was applied using a sequence of toluene-ethanol (equal in volume), ethanol, and demineralized water washes. In case of oak from Lipnica column (GdA-5702), the Soxhlet treatment was omitted, as the danger of contamination was very low for a sample taken at a distance of 11 cm from the surface of this solid and hard trunk. Subsequently, NaOH mercerization (4%, 12 hr, 75ºC) was applied, followed by a regular ABA protocol (4% HCl, 1 hr, 75ºC; 4% NaOH, 1 hr, 75ºC; 4% HCl, 1 hr, 75°C) with demineralized water washes between steps. Bleaching was carried out in two steps: first with $NaClO_2$ + HCl at pH = 2 (2 hr, 75ºC) and the second in the same solution and time, but including 15 min in an ultrasonic bath. In this way, fine homogeneous holo-cellulose fibers were obtained, which were dried in an oven (75°C). In the case of Lipnica sample (GdA-5702) the preparation was extended by two additional NaOH treatments (10% NaOH, 45 min, 70ºC; 17% NaOH 45 min, room temperature, ultrasonic bath) to obtain alpha-cellulose.

For combustion, ca. 3.5 mg of each cellulose was weighted, packed in a tin boat, and introduced to the VarioMicro elemental analyzer (Elementar TM) coupled to the AGE-3 graphitization system (Wacker et al. 2010). Reference materials: Oxalic Acid II and background samples (coal and old wood beyond the ^{14}C range) were prepared in the same way, analyzed in the same batch, and used for calculations. All samples yielded amount of material sufficient to prepare standard cathodes with 1 mg of carbon. Radiocarbon concentration measurements were performed at the Poznań Radiocarbon Laboratory using the NEC 0.5MV Compact Carbon AMS (Goslar et al. 2004). Radiocarbon results were calibrated with use of OxCal v4 software (Bronk Ramsey 2009) and IntCal20 calibration curve (Reimer et al. 2020).

The samples were observed under the Zeiss Stemi 305 trino microscope (coupled with the Axiocam 208 color) to determine the taxonomic affiliation of the wood. For the wooden board from the Bobrowniki church, dendrochronological examinations were performed using LinTab™ equipment and TSAP-Win™ software (Rinn 2003). The obtained measurement sequences of growth-ring width were compared with available master chronologies.

RESULTS AND DISCUSSION

The result obtained for all samples are summarized in Table 1 and presented in Figures 2–4.

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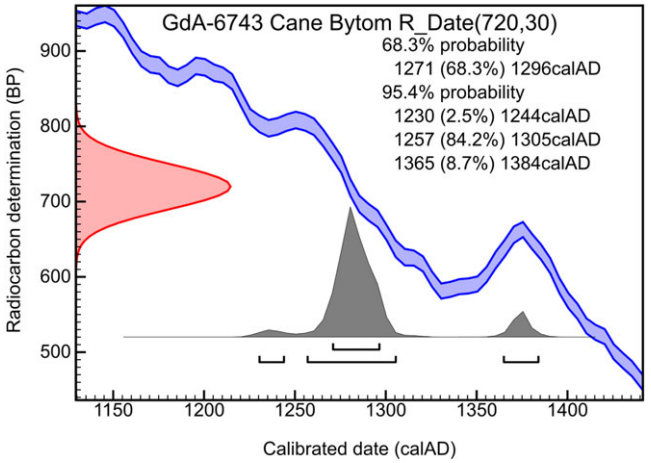

Figure 2 Calibration of ^{14}C date obtained for yew cane from Bytom.

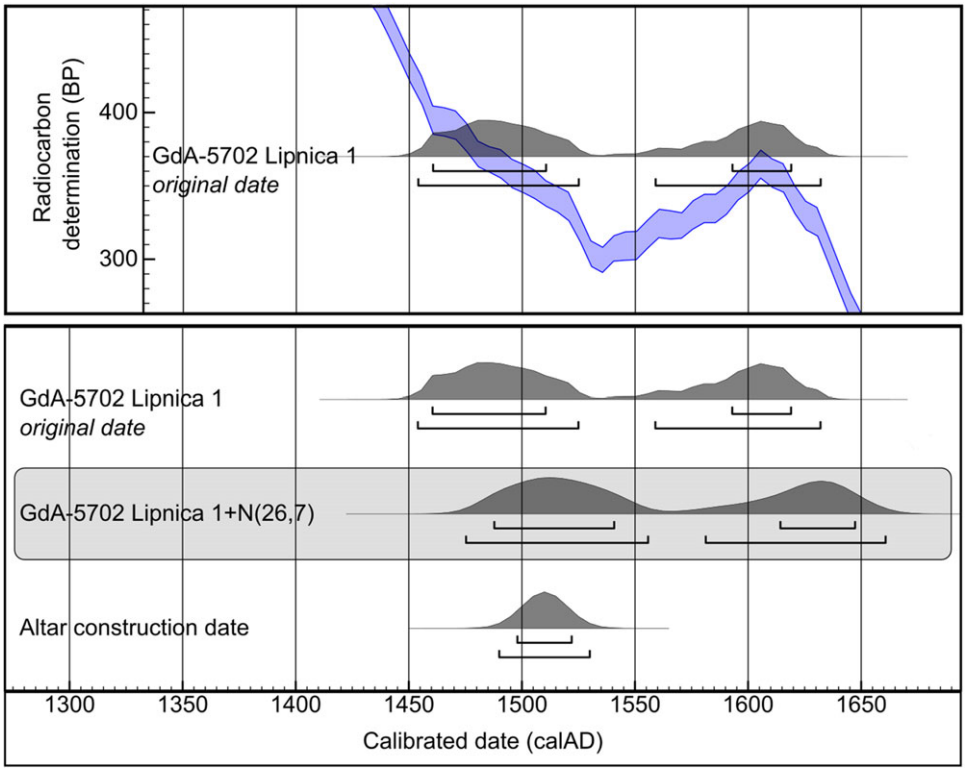

Figure 3 Calibration results for GdA-5702 Lipnica 1: probability distribution of the original date, date shifted by 26 ± 7 calendar years and the age of the altar construction, assumed as AD 1510 ± 10.

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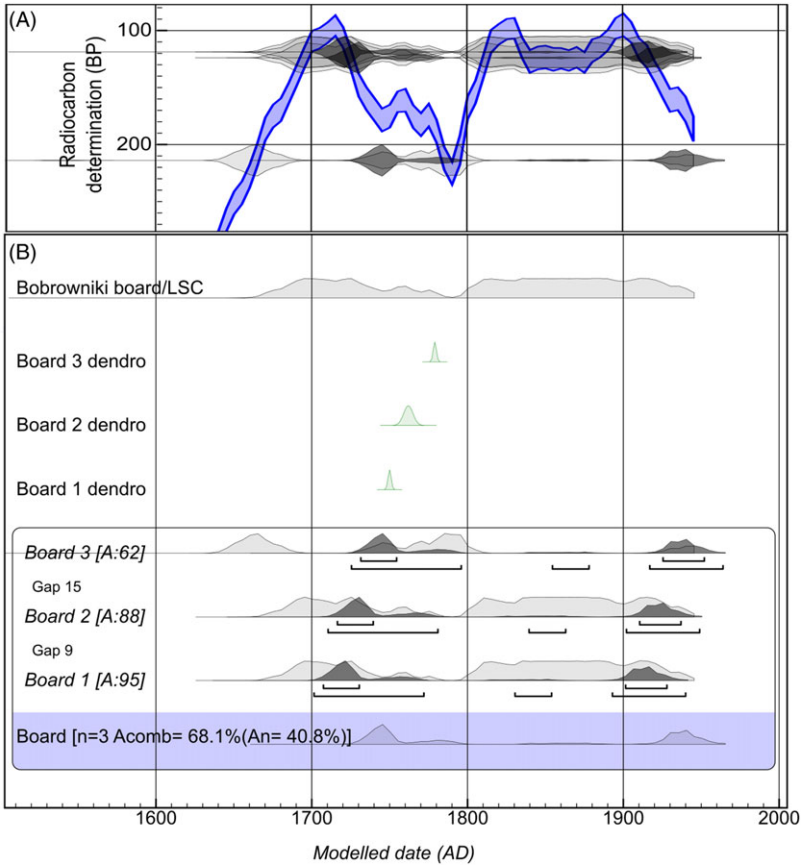

Figure 4 Probability distribution of calibrated ages for ^{14}C-dated samples from Bobrowniki before modeling with D_Sequence (light gray) and after (dark gray). (A) Plotted on the background of calibration curve. (B) Details and results of D_Sequence analyzes compared with calendar ages determined by dendrochronology.

The Cane from Bytom

Dendrological analysis allowed identification of the species as yew wood (*Taxus baccata* L.), which is strong and flexible, yet durable and resistant to moisture. The use of this species was common, and the choice may have been intentional with a symbolic meaning.

Radiocarbon dating result for GdA-6743 was 720 ± 30 BP, and the calibrated age range at 1σ level was 1271–1296 cal AD (Figure 2). Previous dendrochronological analysis of 12 fir trunks was performed by M. Krąpiec (as reported by Andrzejewska 2000). These trunks were used in construction of the medieval drainage ditch and years the trees were felled were determined from AD 1272 to AD 1294. Therefore, the age 1271–1296 cal AD is in a perfect agreement with archeological context of the Layer No. XIII.

The Column from Lipnica Murowana

The radiocarbon date of 370 ± 25 BP was determined for the GdA-5702 sample. However, the dated material was collected from the internal part of the trunk, and the age obtained did not

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correspond to the age the tree was felled, which was at least 14 years later than indicated by the measurement results. Furthermore, the external part of the trunk was cut during carpentry work. The unknown number of rings that were removed must have included a sapwood layer, which in the case of oak trees from Lesser Poland covers 12 +7/–6 rings (Zielski and Krąpiec 2004). In summary, the age of the tree felling must have been later by at least 26 ± 7 years relative to the date result. This correction was added during calibration as a normal distribution, and the results are presented in Figure 3.

For comparison, the date of the altar's construction is plotted, assumed to be AD 1510 with an uncertainty of 10 years. Due to the wiggle in the calibration curve, the probability distribution of the calendar age of the date of the tree fall has two distinct ranges, the first indicating the late 15th to half of the 16th century AD and the second the first half of the 17th century AD. The earlier age range is more probable in light of historical records. As such, the analysis excluded the legendary pre-Christian provenience of the column and its relation to the Światowid deity —if this were to be true, the oak must have been a few hundred years older.

The Saint Lawrence Church in Bobrowniki

The dendrochronological analysis of the board confirmed that it was made of pine wood. The whole sequence covered 71 tree rings (see Figure 1 C). The sequence was matched to three different pine master chronologies, and all attempts gave the same results, i.e., AD 1746–1816 for the whole sequence with acceptable correlation coefficient (TV=5.4, TVBP = 4.1, TVH = 3.6). The sample Board 1 (GdA-6744) composed of 3 rings was dated to AD 1749–1751, Board 2 (GdA-6745) composed of 4 rings dated to AD 1760–1763, and Board 3 (GdA-6746) composed of 3 rings dated to AD 1778–1780.

As expected from the previous dating and the historical context, all the ^{14}C ages fell into a period of pronounced wiggles in the calibration curve, resulting in multiple calibrated age ranges, extending in total to over 200 years (Table 1; Figure 4A). The additional information from dendrochronological data on the number of tree rings between dated samples was added using *D_Sequence* function in OxCal (Bronk Ramsey et al. 2001). Statistical indicators confirm that the model assumptions are fulfilled by the analysis results to an acceptable degree, as all individual agreement indices exceed the threshold of 60% and the combination agreement index A_{comb} = 68.1% exceeds the acceptance threshold of 40.8% (Table 1; Figure 4B).

At the same time, the results reveal two relatively narrow but clearly separated calendar age ranges, which for 68.3% probability are: 1731–1754 cal AD (36.1%), and 1925–1952 cal AD (32.2%). For 95.4% probability the ranges are wider but cover roughly the same periods: second half of 18th century or first half of 20th century. In addition, with a minor probability (2.7%) the age may be from 1854–1878 cal AD. However, the third layer of polychrome painting was undoubtedly documented to have been created by Otto Kowalewski in AD 1923, which implies the 20th century age range should be excluded.

CONCLUSIONS

In all presented cases, the radiocarbon and dendrochronological analyses showed a synergy leading to enhanced knowledge about the dated objects. For the cane from Bytom, the dendrochronological analysis was not possible; however, the species identification as a yew tree provided useful historical information, which will be further explored. In case of the column from Lipnica Murowana, the scientific results excluded a legendary story of its provenance,

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although it cannot be expected to erase already existing regional beliefs. The most spectacular success was achieved for ^{14}C wiggle-matching procedure applied to the board from Bobrowniki church, where by adding the information about dendrochronological sequence and historical records the calibrated age interval was spectacularly narrowed from 1650–1950 cal AD to 1731–1754 cal AD. While the range borders are provided with single-year precision, it should not be overestimated, being a result of Bayesian modeling. Moreover, this interval is shaped by the calibration curve, which may be adjusted in the future. Even though, the middle 18th century should remain the most accurate age of the wooden board.

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