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# Carbon-13 in α-cellulose of oak latewood (Jędrzejów, Southern Poland) during the Maunder Minimum

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We have studied the $\delta^{13}C$ signature in latewood -cellulose of an ancient oak *Quercus robur* L.) from a Cisterian Abbey belfry in Jędrzejów (Southern Poland). The time scale for the δ C record during 1631–1765 AD was built on the basis of detailed dendrochronology studies. Techniques available for extraction of α-cellulose from small samples have been used, the mean value of α-cellulose extraction efficiency being *ca*. 35%. In the $\delta^{13}C$ record of α-cellulose the cooling between 1650–1700 AD is clearly visible as a decrease of about 1.5‰. This period is consistent with the interval of the lowest solar activity between 1645 and 1715 AD, which is known as the Maunder Minimum. Anti-correlation between $\delta^{13}C$ and $\Delta^{14}C$ during the Maunder Minimum was observed but $\Delta^{14}C$ record is delayed by about 30 years with respect to the Maunder Minimum.

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## INTRODUCTION

Reconstructing palaeoclimate using trees offers distinct advantages over ice cores, laminated sediments or speleothems. Annual tree rings represent valuable archives of carbon in the atmosphere, as well as reflecting the hydrogen and oxygen isotopic composition of water during the time of tree growth (Epstein *et al*., 1976). In Central Europe the best wood material for palaeoclimatic studies are oak and pine as they have well defined annual rings. Information on climatic conditions from tree rings can be obtained via stable isotope composition measurements (Schleser *et al*., 1999).

Stable isotope analyses of oak and pine tree rings, combined with traditional dendrochronological research, give possibilities for providing information about temperature and precipitation in the past with annual time resolution (Krąpiec *et al.*, 1998). Such investigations have been performed in south-west Germany to reconstruct a temperature record for the last millennium (Lipp *et al*., 1991) in Eastern England and in southern-west Finland (Robertson *et al*., 1997*a*, *b*). The stable isotopes of hydrogen, carbon and oxygen are used in such environmental studies, because their relative abundance is high and their natural cycle is well known (Griffiths, 1998).

The dendrochronological pine time scale for Northern Europe extends to 7000 years BP and the oak time scale for Ireland, England and Central Europe ranges back to 8000 years BP. These can be used as time scales for proxy-data in palaeoclimate studies (McCarroll and Loader, 2003).

Studies of temperature reconstruction in the past are carried out by using, for example, α-cellulose in pine and oak tree rings from the temperate climate zone. The relative concentrations of heavy stable isotopes are measured in this wood component by mass spectrometry techniques as deviations in per mil of the isotope concentrations from international standards: PDB for carbon ($\delta^{13}C$) and SMOW for hydrogen and oxygen ($\delta^{2}H$ and $\delta^{18}O$).

The dependence between $\delta^{13}C$ measured in α-cellulose from oak tree rings and air temperature and humidity has been documented for eastern England by Robertson *et al*. (1997*b*).

Comparison of the $\delta^{13}C$ temperature record with meteorological data indicates a high correlation between $\delta^{13}C$ and temperature for the period 1945–1994. A similar positive correlation between $\delta^{13}C$ in pine tree rings α-cellulose and mean July–August temperature was found by Pawełczyk *et al*. (2004) for north-east Poland over the 1899–1968 period. For a more general relationship between air temperature, relative humidity, $CO_2$ concentration in the air and $\delta^{13}C$ in tree material see Farquhar *et al.* (1982).

The source of hydrogen and oxygen isotopes for cellulose production is meteoric water. Transpiration and biological fractionation processes change the isotopic composition of meteoric water, and the relation between deuterium and $^{18}O$ isotopes in local precipitation and cellulose has been investigated by various authors (Yapp and Epstein, 1982; Switsur and Waterhouse, 1998). In these studies the temporal record of $\delta^{2}H$ in 1894–1994 shows a positive correlation with the temperature record indicated by $\delta^{13}C$ changes.

For palaeoclimatic isotopic studies both living trees and ancient wood from historical buildings can be used. In this study the α-cellulose from an ancient oak (*Quercus robur* L.) from the Cisterian Abbey belfry at Jędrzejów (Southern Poland) was used for $\delta^{13}C$ determination. Dendrochronology studies and $\delta^{13}C$ measurements were carried out as preliminary studies of:
— α-cellulose efficiency in our laboratory extraction method,
— changes of $\delta^{13}C$ in tree rings of oak late wood induced by climatic conditions in a very well known and defined time period, namely the Maunder Minimum. The techniques available for extraction of small samples have been described by Loader *et al*. (1997) and applied in Gliwice Radiocarbon Laboratory.

## SITE

Jędrzejów (20°18'E, 50°39'N) is located in south-east Poland on the Jędrzejów tableland (Fig. 1). This town is situated 37 km from Kielce and 78 km from Kraków and the Jędrzejów commune covering 228 km$^2$, and is one of the largest in the Świętokrzyski province. The most important monument in Jędrzejów is the Cistercian Abbey, founded in 1149.


**Fig. 1. Locality of the Jędrzejów site**

**Fig. 2. The Cistercian Abbey and the tree material investigated from the Abbey belfry**

The wood sample used for research, shown in Figure 2, comes from the bell-supports in the free-standing Baroque belfry which is situated near the Abbey. At the beginning of the 1990s, during renovation, this oak structure was replaced with a metallic one. From the oak beams preserved, five slices were taken for dendrochronological analysis.

The wood used to build the belfry probably comes from trees (*Quercus robur* L.), which grew in the neighbouring forests.

## DENDROCHRONOLOGY

Ancient wood samples from Jędrzejów, after suitable preparation consisting of cutting the outermost wood layer and revealing the tree-ring anatomy details in cross-section, were subjected to measurements of the annual ring widths with 0.01 mm accuracy in the Tree-Ring Laboratory of AGH–University of Science and Technology in Kraków. The sequences of annual growths displayed high similarity with the dendrochronological curves obtained (*t* value of *ca*. 6) indicating that they came from trees growing at the same time. The *t* value is calculated on the basis of a statistical method called the Student method (Baillie and Pilcher, 1973; Zielski and Krąpiec, 2004) and usually this value is higher than 4 for highly correlated sequences of annual growth. The local chronology produced from these curves was 146 years long. It was absolutely dated

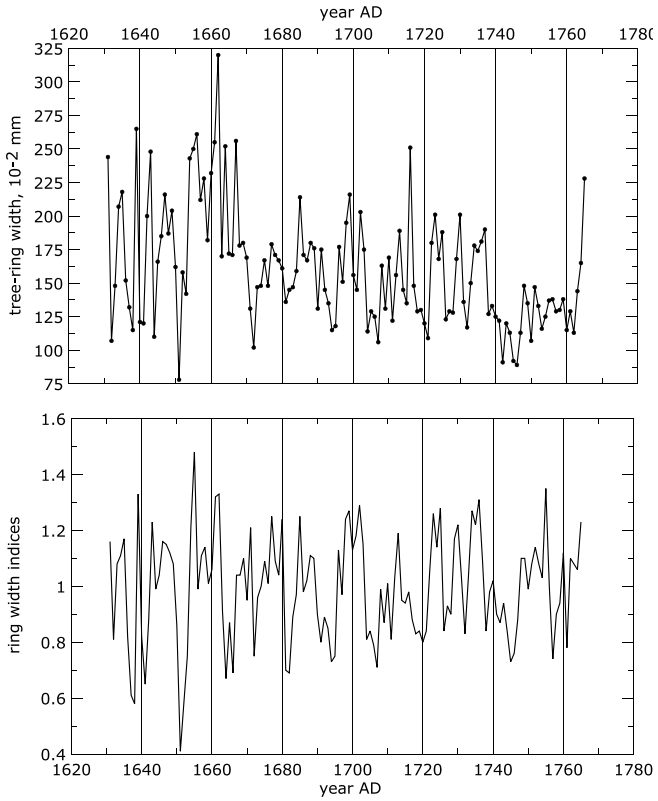


**Fig. 3. Oak tree-ring widths and ring width indices over the time period investigated, 1631–1765 AD**

to 1622–1767 AD against the Małopolska regional chronology covering the period 910–1997 AD (Krąpiec, 1998). The fact that the youngest ring of the chronology is entirely developed and situated directly beneath the bark indicates that the trees were cut down in winter of 1767 AD. Therefore, the belfry was most probably constructed in the next year, *i.e.* 1768 AD because the cut trees then were left for one or two years to dry before being used for construction. Among five dendrochronologically analyzed samples, the slice labeled as JDJ3, comprising the wood increments from 1631–1765 AD, was selected for isotope analyses. This sample showed a good state of wood preservation and regular annual increments, without anatomical anomalies. Tree ring widths and ring width indices of sample JDJ3 in the period investigated, 1631–1765 AD, are shown in Figure 3.

## METHODS

**α-cellulose extraction.** Dendrochronologically dated wood was cut to one-year late wood growth samples, packed and labeled. The extraction of α-cellulose was made on the basis of a modification of Green (1963) method with use of ultrasonic baths to accelerate the separation of single cellulose fibers from wood samples and to increase the penetration of reagents (Loader *et al.*, 1997). The scheme of -cellulose extraction used in Gliwice Radiocarbon Laboratory is shown in Figure 4.

The dried and weighted wood samples were cut into small pieces and placed into the glass tubes. 175 ml of distilled water, 2.5 g of sodium chlorite and 1.7 ml of acetic acid was added per 1g of each sample. The glass tubes were put into an ultrasonic bath kept at 70°C for one hour. 2.5 g of sodium chlorite and 1.7 ml of acetic acid was then added per 1 g of sample. This process was repeated five times. In the next step the solution was removed by decanting and samples were rinsed with hot distilled water and then with cold distilled water up to neutral pH. The result of this part of extraction is holocellulose. This treatment removes most of the lignin by oxidative degradation. In each tube (75 ml) of 10% NaOH solution was added to the holocellulose, put into an ultrasonic bath and kept at 70°C for 45 minutes. Afterwards the solution was removed and samples were rinsed with (50 ml) of cold distilled water. Then (67 ml) of 17% NaOH solution was added and samples were put into ultrasonic bath at room temperature for 45 minutes. Then the solution was removed from the tubes and samples were bathed with 50 ml of distilled water. This treatment removes hemicelluloses and the remaining lignin. At the end of the extraction process the samples were treated with *ca.* (20 ml) of 1% HCl solution and rinsed up to neutral pH. Finally the obtained α-cellulose was dried on a hot plate at 70°C for 4 hours.

**α-cellulose extraction efficiency**. The "α-cellulose efficiency" in extraction procedure is defined as the ratio of the

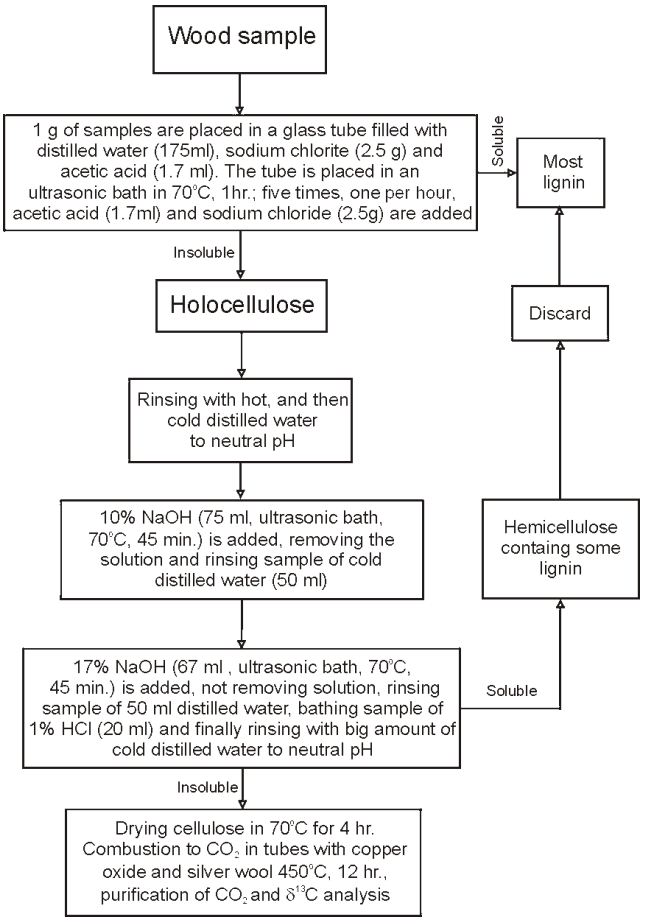


**Fig. 4. The α-cellulose extraction procedure used in Gliwice Radiocarbon Laboratory**

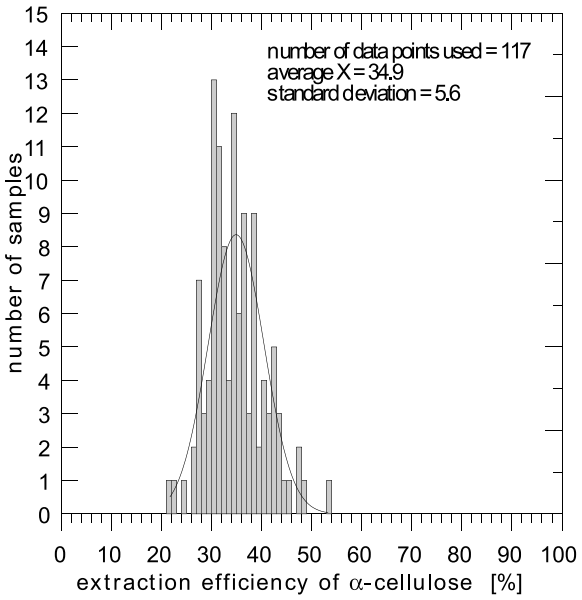


**Fig. 5. Histogram of -cellulose extraction efficiency from oak latewood**

The Gaussian distribution is given by the solid line

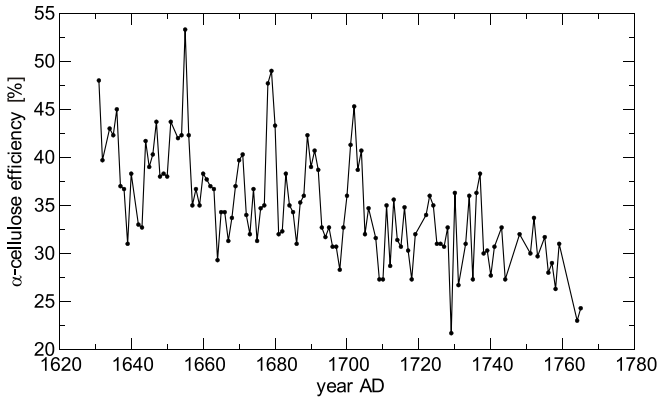


**Fig. 6. The -cellulose extraction efficiency for oak latewood over the time period investigated, 1631–1765 AD**

mass of α-cellulose obtained to the mass of wood sample taken for extraction. The distribution of experimental values of α-cellulose efficiency is shown in Figure 5. The efficiency varies from *ca*. 20 to 50%, with a mean value of 34.9

which is consistent with the value obtained in experimental procedures and predicted on the basis of tree physiology (Robertson and Waterhouse, 1998). Comparison of experimental and normal distribution shown by a Gaussian curve indicates some difference between experimental and predicted values. For 24 samples of 117 the efficiency is equal to 30–32%. Deviation can be expected because of differences in cellulose contents in individual tree rings. The variations of efficiency for single oak late wood rings are given in Figure 6. Fluctuation of the efficiency value with a relatively high amplitude on a trend that decreases with the growing age of the rings can be observed. The highest and lowest values of efficiency can be caused by laboratory errors in

Table 1

**Correlation coefficients for three relationships of investigated wood sample**

| Relationship | Number of measurement points n | Correlation coefficient *R* |
|---|---|---|
| -cellulose efficiency [%] (tree rings width, mm) | 117 | 0.23 |
| $^{13}$C in α-cellulose [‰] (tree rings width, mm) | 103 | 0.10 |
| $^{13}$C in α-cellulose [‰] (tree rings width indices, mm) | 103 | 0.07 |

The coefficients were obtained on the basis of linear regression; names in brackets indicate variables and names without brackets are values — f(x)

the extraction procedure: higher values can be observed for α-cellulose insufficiently dried after extraction, and lower values by loss in the extraction procedure.

A weak positive correlation of -cellulose efficiency and tree ring width is shown in Table 1. For 117 experimental points the correlation coefficient is 0.23.

**α-cellulose combustion for $\delta^{13}$C measurements**. 10 mg of the α-cellulose obtained was put into a glass tube with copper oxide, high purity copper and silver wool and evacuated in a vacuum line to a pressure of *ca*. $10^{-3}$hPa. The copper oxide oxidizes α-cellulose to $CO_2$. The silver wool is used for purification of $CO_2$ mainly from sulfur compounds. After evacuation the tubes were sealed off by a torch and then put into laboratory oven at 450°C for 12 hours. After that the tubes were kept in an oven at 100°C prior to further processing in a vacuum line. During the next step the released $CO_2$ was purified from water in traps cooled by a dry ice and alcohol mixture at a temperature of –70°C and finally $CO_2$ was frozen in new glass tubes. These tubes were sent to the Mass Spectrometry Laboratory in Lublin for $\delta^{13}$C analysis. Figure 7 shows the vacuum line used in the Gliwice Radiocarbon Laboratory for $CO_2$ purification and preparation for mass spectrometry measurements.

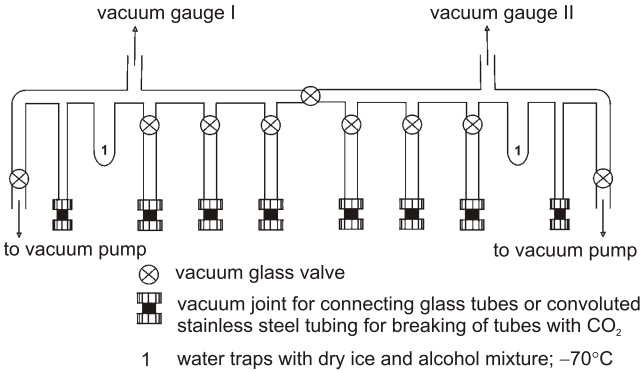


**Fig. 7. The vacuum line for $CO_2$ purification and preparation of samples for $\delta^{13}$C measurements in Gliwice Radiocarbon Laboratory**

**$\delta^{13}C$ measurements**. The measurements were made on a dual inlet and triple collector mass spectrometer (significantly modified MI-1305 type), which gave fast and reproducible $\delta^{13}C$ determinations. The standard deviation (1σ) of $\delta^{13}C$ is 0.07‰. $\delta^{13}C$ was calculated from the measured 45 and 46 mass signals values using the appropriate formula (Craig, 1957; Santrock *et al*., 1985). The standard gas is calibrated to V-PDB by using NBS-19 and NBS-22 standards. The samples, measured to the standard gas, are thus finally expressed on the V-PDB scale. In addition a homogeneous cellulose standard (cellulose powder from spruce; Standard ISONET) was run every 10 samples. This standard is very convenient in analysis of tree-ring cellulose, because its $\delta^{13}C$ is –24.5‰, which is close to those measured for our samples.

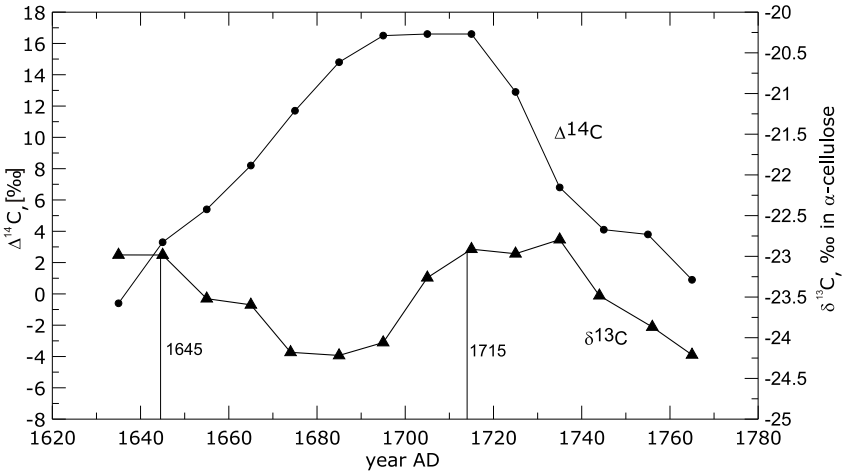


**Fig. 9. Comparison of 10-year averages of $\delta^{13}C$ in -cellulose and $\Delta^{14}C$ in tree rings, over the 1631–1765 AD period**

The $\Delta^{14}C$ time record as a fragment of the calibration curve was taken (Intcal 98 curve, Stuiver *et al*., 1998); the Maunder Minimum (1645–1715 AD) is indicated by vertical lines

## $^{13}C$ MEASUREMENT RESULTS AND PALAEOCLIMATIC IMPLICATIONS

**$^{13}C$ time record and palaeoclimate.** The $^{13}C$ time record in oak latewood α-cellulose with annual resolution over the 1631–1765 AD interval is shown in Figure 8. The smoothed curve presents 5-year running average time record. Within the period investigated a rapid cooling climatic event is well known and has been correlated with extremely low sunspot numbers. The period of low solar activity between 1645 and 1715 is commonly known as the Maunder Minimum (Fagan, 2000; Wei-Hock Soon and Yaskell, 2004). During this period sunspots were infrequent and, as a result, it was assumed that solar activity and the solar wind was either weak or switched off.

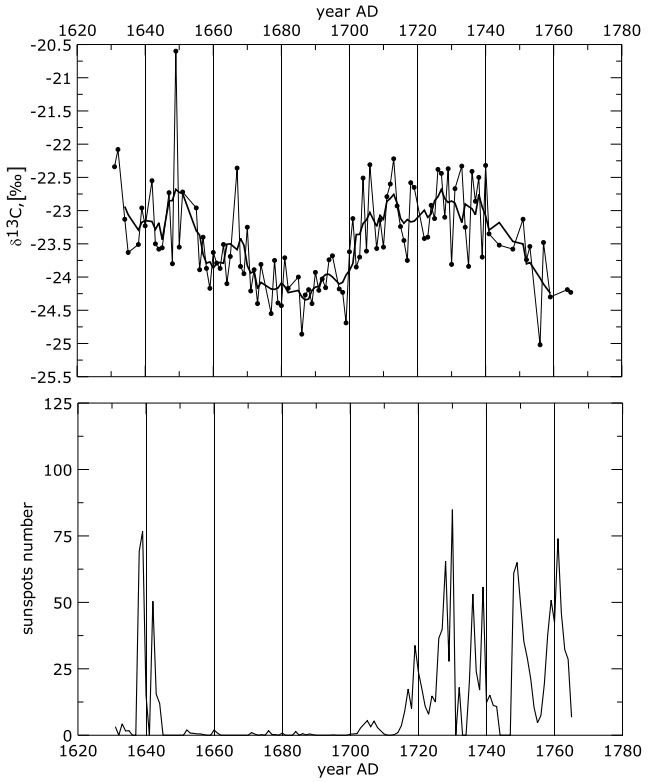


**Fig. 8. Comparison of the $\delta^{13}C$ time record in -cellulose from oak latewood and sunspot numbers (Hoyt and Schatten, 1998) over the 1631–1765 AD period**

In the upper figure the 5-year running average is shown as a thicher line

In the $\delta^{13}C$ record of the α-cellulose time record the cooling between *ca*. 1650–1700 AD is clearly visible. The lower values of $\delta^{13}C$ in this period correlate very well with extremely low sunspot numbers (when sunspots almost disappeared). During the Little Ice Age (1300–1850) the Earth's temperature was lower by 1 to 2°C with respect to modern temperature (Fagan, 2000). Local temperature changes during the Maunder Minimum, recorded in $^{13}C$ carbon content in oak tree rings, can be higher or lower of course. This temperature decrease is visible as changes of average $\delta^{13}C$ values from –22.7 to –24.3‰, i.e. by about 1.5‰. The value of the temperature gradient of $\delta^{13}C$ for NE Poland (Augustów Wilderness) on the basis of pine tree rings (*Pinus sylvestris* L.) investigations was estimated as changing from 0.36 to 0.51‰/°C in different time periods (Pawełczyk *et al*., 2004). Using these temperature gradient values, the decrease in temperature during the Maunder Minimum can be estimated as ranging from 3 to 4°C for Southern Poland.

The correlation coefficients (Table 1) between $^{13}C$ and ring width and indices are indeed low; therefore there is no significant correlation between them. However, that does not necessarily mean that the correlation between ring widths and temperatures are as low.

**$^{13}C$ and $^{14}C$ isotopes content in tree rings and the Maunder Minimum**. Solar activity can be reconstructed using the $^{14}C$ isotope (Stuiver and Braziunas, 1993). Radiocarbon as a cosmogenic nuclide is produced by galactic cosmic rays, mainly protons. The stream of protons is modulated by the in-

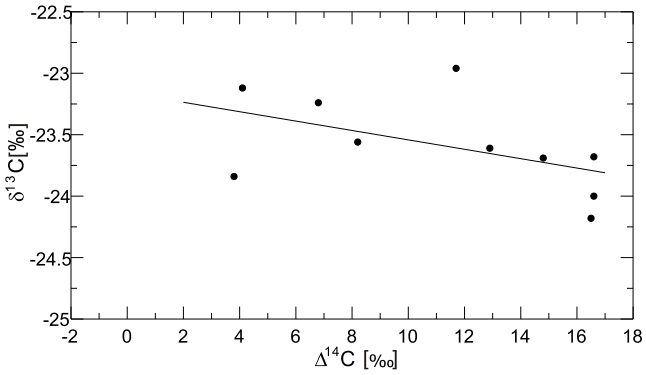


**Fig. 10. Correlation between $\delta^{13}C$ in latewood -cellulose and $\Delta^{14}C$**

The correlation coefficient was calculated after displacement of $\Delta^{14}C$ values of 30 years towards lower values and the removal of four points; number of points n = 10, negative correlation is shown and the correlation coefficient $R = -0.51$.

terplanetary magnetic field and the solar wind. In the periods with lower sunspots (lower Sun activity) the production rate of $^{14}C$ is higher and the relative concentration ($\Delta^{14}C$) of this isotope in atmospheric $CO_2$ and after that in tree rings increases. Such events are observed in the $\Delta^{14}C$ time record used for construction of a calibration curve (Stuiver *et al.*, 1998). Variation of the radiocarbon content in 711-yr-old cedar tree rings growing in Japan, during the Spoerer Minimum (1415–1537 AD) was observed recently by Miyahara *et al*. (2004).

Figures 9 and 10 shows the $\delta^{13}C$ values of -cellulose taken from Figures 8 and 9 with points representing 10-year averages. Also shown are the 10-year averaged $\Delta^{14}C$ values taken from calibration data (Stuiver *et al.,* 1998). It can be seen that the maximum in the $\Delta^{14}C$ record falls 30–40 years after the minimum in the $\delta^{13}C$ record. The highest anti-correlation between these records is obtained with a correlation coefficient of $R = -0.51$ after the $\Delta^{14}C$ values were shifted *ca*. 30 years towards lower ages, see Figure 10. The reason of this shift may be a finite "response time" due to carbon cycling throughout geochemical reservoirs.

## CONCLUDING REMARKS

Dendrochronologically dated wood, ancient latewood of *Quercus robour* L. over the interval 1631–1767 AD, from the Cistercian Abbey at Jędrzejów, Southern Poland, was used for $^{13}C$ carbon isotope analysis. The extraction of α-cellulose made on the basis of modification of Green's method, with the use of ultrasonic baths to accelerate separation of individual cellulose fibers from wood samples and increase the penetration of reagents, provided very suitable material for isotope palaeoclimatic studies. The α-cellulose extraction efficiency in this study was high. Relatively small changes of $\delta^{13}C$ (decrease of *ca*. 1.5‰) in the Maunder Minimum correlate with cooling at this time and can be compared with the carbon $^{14}C$ content in wood using a fragment of the calibration curve. Anti-correlation of both records is clearly visible.

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