

# Constructing deposition chronologies for peat deposits using radiocarbon dating

N. Piotrowska<sup>1</sup>, M. Blaauw<sup>2</sup>, D. Mauquoy<sup>3</sup> and F.M. Chambers<sup>4</sup>

<sup>1</sup>GADAM Centre, Institute of Physics, Silesian University of Technology, Gliwice, Poland

<sup>2</sup>School of Geography, Archaeology and Palaeoecology, Queen's University Belfast, UK

<sup>3</sup>School of Geosciences, University of Aberdeen, UK

<sup>4</sup>Centre for Environmental Change and Quaternary Research, University of Gloucestershire, UK

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## SUMMARY

Radiocarbon dating is one of the main methods used to establish peat chronologies. This article reviews the basis of the method and its application to dating of peat deposits. Important steps in the radiocarbon dating procedure are described, including selection and extraction of material (and fractions) for dating, chemical and physical preparation of media suitable for measurements, measurements of <sup>14</sup>C activity or concentration, calculations, calibration of results and age-depth modelling.

**KEY WORDS:** accelerator mass spectrometry (AMS), age-depth model, <sup>14</sup>C activity, gas proportional counting (GPC), liquid scintillation counting (LSC).

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## 1. FUNDAMENTALS OF THE METHOD

Radiocarbon dating is commonly used to construct timescales for Late Pleistocene and Holocene records spanning the last *ca* 50 ka. The <sup>14</sup>C isotope was identified as a product of nuclear reactions between thermal neutrons and nitrogen nuclei (reaction <sup>14</sup>N(n,p)<sup>14</sup>C) at the University of California, Berkeley in the 1930s (see Libby 1967). On the basis of the estimated neutron flux in the upper atmosphere and using simple equilibrium assumptions and information about the total carbon content in upper reservoirs (atmosphere, biosphere, hydrosphere and lithosphere), Libby (1946) estimated the relative activity of <sup>14</sup>C in atmospheric CO<sub>2</sub> and modern vegetation to  $\pm 0.1$  disintegrations per minute in 1 gram of carbon (dpm g<sup>-1</sup> C). Libby's estimate is close to the  $A_0 = 13.56$  dpm g<sup>-1</sup> C value which was calculated later on the basis of precise measurements (Karlén *et al.* 1966). To verify his hypothesis regarding the natural origin and relative activity of <sup>14</sup>C, Libby used methane produced in the decomposition of modern plants. The last step towards creating the radiocarbon chronometry was to show that <sup>14</sup>C occurs uniformly in the Earth's biosphere (Libby *et al.* 1949).

In order to determine the age of a sample, the <sup>14</sup>C  $\beta$  decay half-life ( $T_{1/2}$ ) has to be known. The current best value of the half-life is  $5720 \pm 30$  years (Godwin 1962). However, the value of  $T_{1/2}$  that was established in the early days of the radiocarbon method during the 1950s was 5568 years (see Libby 1967), a 2.7 % difference. This value is still used in calculating radiocarbon dates and forms the basis for

the conventional definition of the radiocarbon time scale.

At present, the natural rate of radiocarbon production in the upper atmosphere is estimated to be around 2 atoms cm<sup>-2</sup> sec<sup>-1</sup> summed over the Earth's surface (Damon & Sternberg 1989). This means that the amount of <sup>14</sup>C in the Earth's atmosphere, as <sup>14</sup>CO<sub>2</sub>, is around 60 kg. This value increased fourfold during the period of nuclear weapon tests, in which radiocarbon was artificially produced. On a longer timescale, the <sup>14</sup>C concentration in atmospheric CO<sub>2</sub> has been decreasing due to ocean uptake and, in recent centuries, regional radiocarbon-free CO<sub>2</sub> emissions due to burning of fossil fuels (the Suess effect; Pazdur *et al.* 2007, Levin *et al.* 2008, Rakowski *et al.* 2010).

A fundamental limitation on the range and accuracy of radiocarbon dating is imposed by the low natural concentration of <sup>14</sup>C (<sup>14</sup>C/<sup>12</sup>C =  $10^{-12}$ , or one <sup>14</sup>C atom for every trillion <sup>12</sup>C atoms, in modern material). This concentration decreases with time according to the half-life, from the moment of death of living organisms that assimilated atmospheric CO<sub>2</sub> (Figure 1) or from the time of sedimentation of mineral deposits e.g. crystallisation of calcite.

The basis for standardised radiocarbon age calculations was provided by Stuiver & Polach (1977). The conventional radiocarbon age of a sample is usually reported as years BP, yr BP, conv. BP, or BP. Some archaeological publications use lower-case "bp" (also lower-case "ad", "bc") to distinguish uncalibrated (i.e. conventional) radiocarbon ages, but this practice has been largely

discontinued. Conventional ages are calculated as the time that has elapsed since the moment when exchange of carbon between the environment and the matter from which the sample was taken stopped until the ‘present’, which is set at AD 1950. The radiocarbon age ( $T$ ) is calculated from the established constant  $^{14}\text{C}$  concentration in the biosphere ( $A_0$ ) and the radiocarbon concentration in the sample at present ( $A$ ), according to the following formula:

$$T = -\frac{T_{1/2}}{\ln 2} \ln \frac{A}{A_0} = -8033 \ln \frac{A}{A_0} \quad [1]$$

The value of the conventional radiocarbon age  $T$ , as defined above, is calculated using half-life  $T_{1/2}$  equal to 5568 years. Practically, the  $A_0$  is determined as 95 % of the  $^{14}\text{C}$  activity of samples of standard material NBS Oxalic Acid I, referred to AD 1950 (Stuiver & Polach 1977).

Carbon isotope fractionation occurs through processes of carbon assimilation by living organisms, in the geochemical cycle of carbon in its different reservoirs, and during inter-reservoir exchange. Because of this, it is necessary to normalise the measured  $^{14}\text{C}$  concentration  $A_m$  by adding a  $\delta^{13}\text{C}$  correction. By convention, the radiocarbon concentration including isotope fractionation correction is calculated according to the following formula (Stuiver & Polach 1977):

$$A = A_m \left[ 1 - \frac{2(\delta^{13}\text{C} + 25)}{1000} \right] \quad [2]$$

Mass spectrometry is used to determine  $\delta^{13}\text{C}$ , and its value ranges from about +2 ‰ for carbonate sediments, down to around -40 ‰ in some samples of plants assimilating carbon using the C3 photosynthesis cycle. On average, the  $\delta^{13}\text{C}$  of atmospheric  $\text{CO}_2$  is about -8 ‰, while *Sphagnum* is characterised by a  $\delta^{13}\text{C}$  of around -25 ‰.

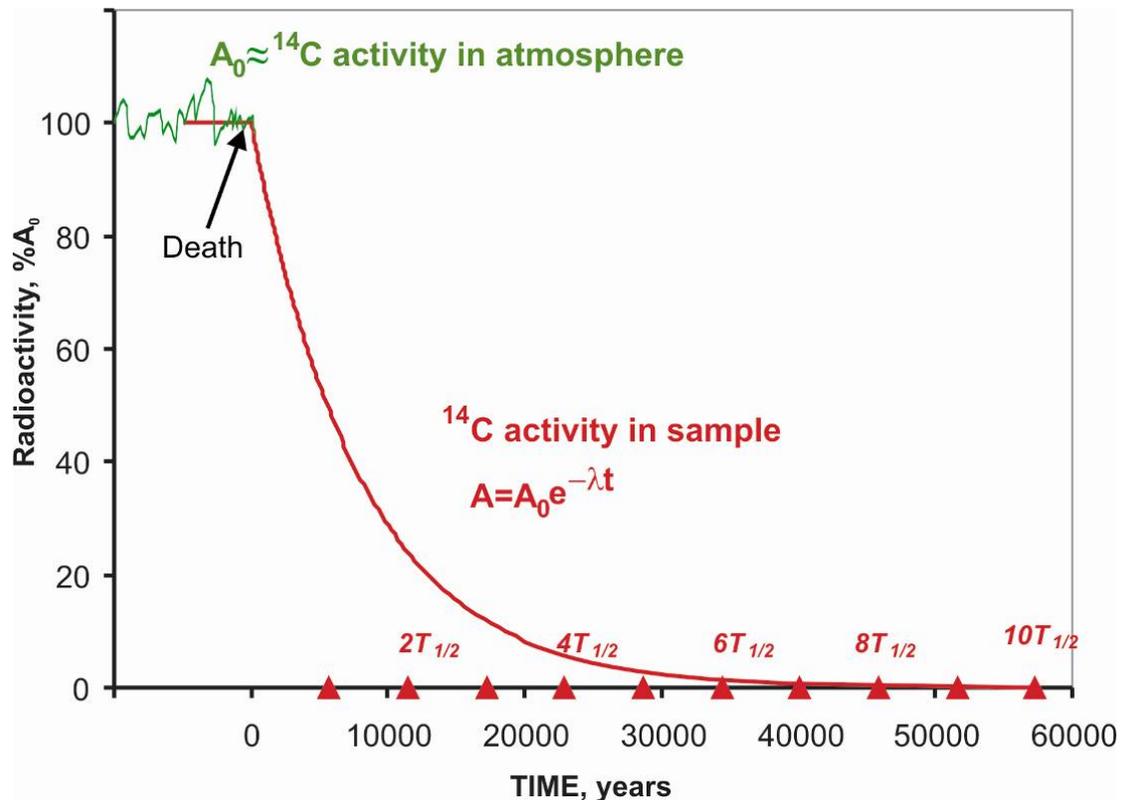


Figure 1. Changes of radiocarbon activity in organisms initially assimilating carbon from the atmosphere, then dying at TIME=0 and subsequently being incorporated into a peat sample. The initial radiocarbon activity of the sample would be equal to atmospheric  $^{14}\text{C}$  activity ( $A_0$ ) provided that no fractionation occurred during assimilation of  $\text{CO}_2$ . After death, the  $^{14}\text{C}$  content/activity ( $A$ ) decreases with time according to the law of radioactive decay ( $\lambda$ : decay constant;  $t$ : time;  $T_{1/2}$ : half-life).

## 2. SELECTION AND EXTRACTION OF MATERIAL FOR DATING

In order to ensure accurate dating of peat bog deposits, the botanical composition of the profile must be determined. The age assigned to a peat sample should reflect the time that the plants forming it died, and the most important issue is to identify the fraction which would provide a reliable result if present in sufficient quantity. The best material for ensuring accurate dates are above-ground remains of plants that assimilated atmospheric CO<sub>2</sub>, e.g. short-lived plant macrofossils and pollen, whose <sup>14</sup>C age is consequently not affected by a reservoir effect (explained later in this Section). *Sphagnum* macrofossils provide excellent material for <sup>14</sup>C sample selection, since these can be easily cleaned and selected using watch-maker's forceps under low-power microscopy. Suitable materials for sample selection are *Sphagnum* mosses (branches, stems and leaves) (see e.g. Nilsson *et al.* 2001). In well preserved peat samples, whole *Sphagnum* mosses with stems, branches and leaves still attached are sometimes present, and this makes it very easy to select a sample for dating. If *Sphagnum* mosses are not present in the sample, above-ground leaves and stems of dwarf shrubs (e.g. *Calluna vulgaris*, *Erica tetralix*, *Andromeda polifolia* and *Empetrum nigrum*) can be used. The composition of the sample that is submitted should always be checked and described, preferably by a botanist/palaeoecologist. Before final submission to the <sup>14</sup>C laboratory, contaminants such as dwarf shrub rootlets, fungal remains or foreign material that may have entered the sample during extraction or preparation (e.g. dust, hair or textile) should be meticulously removed. This cleaning can be performed by repeated washing in sterilised water, followed by manual removal (using watch-maker's forceps) of any remaining contamination that is visible under a low-power microscope.

For highly humified peat, from which it is difficult to select specific macrofossils, it is possible to date either the whole peat sample (termed a 'bulk' sample), or specific fractions of the peat which may be separated physically or chemically. For example, the 'humic acid' fraction (extracted by washing in alkali) can be dated separately from the 'fine particulate' fraction (< 250 µm) extracted by washing the disaggregated peat sample through a sieve.

The difference in dating results between various peat fractions was first investigated by Dresser (1970). For highly humified blanket peats he found that the fine particulate fraction (which includes pollen, testate amoebae and various other small

components of the peat) gave the best approximation of the true radiocarbon age of a peat sample. This was because the humic acid (and fulvic acid) fractions tended to be too young, owing to their downward mobility through the peat profile, whereas the bulk peat sample potentially contained younger rootlets. Arguably, an extracted charcoal fraction, if present, should also provide a reliable date if it consists of charred former surface plant material.

The fine particulate fraction does not always yield the best results. For example, dating of recent blanket peat in upland South Wales by Chambers *et al.* (1979) revealed extreme differences in <sup>14</sup>C ages between fine particulate and humic acid fractions, the fine particulate fraction being some thousands of years older than the humic acid. In this instance the humic acid fraction provided the more reliable age, because the fine particulate fraction was apparently contaminated by 'old' carbon in the form of soot from combustion of fossil fuel in the valleys of South Wales during the industrial revolution.

Another example of significant age differences between humic acids and corresponding humin fractions was given by Shore *et al.* (1995). The most extreme differences reported were the humin fraction 630 years older and 1200 years younger than the humic acid, without any consistency or correlations with δ<sup>13</sup>C, degree of humification or pollen concentration. Similarly, Nilsson *et al.* (2001) performed AMS <sup>14</sup>C dating on different fragments or fractions of peat samples, and reported age differences of 360–1000 years. Such differences can be explained by the properties of individual sites and by the vegetation composition of the dated samples. The main site-specific or even sample-specific problems include mobility of various fractions, penetration of older peat by younger rootlets and deposition of re-worked material.

Kilian *et al.* (1995) noted that, in their study, samples of raised bog peat containing fine *Calluna vulgaris* rootlets yielded ages that were 100–150 years too old. They attributed this to a "reservoir effect", perhaps due to re-use of 'old' carbon, notably methane, which might be fixed by mycorrhizal fungi in the Ericaceae rooting zone or merely used by fungi and incorporated into bulk peat samples. CO<sub>2</sub> emitted from decomposing layers of the bog can also become incorporated, with similar effect. A detailed study by Jungner *et al.* (1995) on two ombrotrophic sites in Finland indicated that the effect was irrelevant to the apparent age of the peat because the CO<sub>2</sub> was released from shallow depths of almost the same age as the dated samples, and amounted to around 20% of the CO<sub>2</sub> uptake by *Sphagnum*. Therefore, the

effect was significant only for the period of maximum-intensity nuclear weapons tests. Similarly, Blaauw *et al.* (2004) did not find significant  $^{14}\text{C}$  age differences between bulk peat (often containing heather rootlets) and pure *Sphagnum* samples; and the results of radiometric dating of bulk ombrotrophic bog peat samples from southern Poland did not show any age alteration, and a well-constrained chronology was constructed and reported by Fialkiewicz-Koziel (2009). Nonetheless, it is recommended that the use of bulk samples of raised bog peat containing large amounts of *Calluna* rootlets should be avoided.

### 3. PREPARATION OF SAMPLES

The purpose of chemical pretreatment of samples before radiocarbon measurement is to extract material that contains *in situ* carbon in sufficient quantity to measure the  $^{14}\text{C}$  concentration. The goal is to remove contaminating substances, which often have different ages, and produce the substance that is appropriate to the measurement technique, i.e. benzene for LSC (Liquid Scintillation Counting),  $\text{CO}_2$  for GPC (Gas Proportional Counting), and graphite for AMS (Accelerator Mass Spectrometry). This and subsequent stages of the dating process are performed at the radiocarbon dating laboratory.

The commonly accepted technique for chemical pre-treatment is an acid-alkali-acid washing sequence which removes any carbonates, bacterial  $\text{CO}_2$  and humic/fulvic acids. Afterwards, the material is subjected to preparation processes that depend upon the measurement technique, e.g. combustion and purification of the  $\text{CO}_2$  thus obtained, synthesis of benzene, or graphitisation of  $\text{CO}_2$ . Then radiocarbon activity or concentration is measured using suitable equipment. The procedures and equipment are based on some common techniques, but they are individually developed and depend on the laboratory. A more detailed description of an example procedure for graphite target preparation is given in Table 1.

### 4. MEASUREMENTS

The most important limitations on the techniques used for determining concentrations of radiocarbon arise from its long half-life and low maximum  $\beta$  radiation energy, which is 156 keV. These properties of the  $\beta$  particles originating from radiocarbon decay mean that a measurement device with low background and high sensitivity is required, along with the highest possible purity of

samples. The ‘conventional’ radioactivity measurement techniques applied in  $^{14}\text{C}$  dating include gas proportional counting (GPC) and liquid scintillation counting (LSC). The most commonly used GPC instruments are carbon dioxide-filled counters, whereas benzene is the working substance for LSC. In these techniques the number of electrons released in  $^{14}\text{C}$  isotope decay, estimated from the number of electrical impulses registered by a detector (counter), is proportional to the concentration of the isotope in the sample. As the precision of results depends on the number of particles detected, the measurement needs to be performed over a period of 2–7 days for each sample, depending on its age and size. Although both techniques produce similar results in terms of precision and measuring abilities (Pazdur *et al.* 2000, 2003, Tudyka & Pazdur 2010), commercially available liquid scintillation spectrometers (for LSC) have now superseded gas proportional counters (for GPC), which had to be custom-made and required considerable maintenance.

For low radioactivity measurements using radiometric techniques, reduction of the background count is extremely important, and this requires the construction of a large multi-layered shield. The counter is typically surrounded by a 2–5 cm thick passive shield of metal, usually lead. Because this material should not contain a significant amount of radioactive  $^{210}\text{Pb}$ , it must be at least 100 years old; lead from old water pipes has been used. The next layer is the active anticoincidence shield consisting of several Geiger-Müller counters. Then a further shield of lead, iron or steel is applied, in the latter case using pre-bomb steel from e.g. old gasometers (large storage reservoirs used by gas utility companies) or scrap metal. Some laboratories install the counters underground, which considerably reduces the background from cosmic radiation.

In the most modern technique, called accelerator mass spectrometry (AMS), the radiocarbon concentration in a sample is determined by counting the  $^{14}\text{C}$  atoms in an ion beam produced from the sample. This idea was first realised at Berkeley by Müller, who attempted to accelerate  $^{14}\text{C}$  ions with a cyclotron (Müller *et al.* 1977). A significant advance towards elimination of the influence of other ions with the same mass was introduced by Purser *et al.* (1977), who converted ions with charge -1 to multiple charged positive ions in a so-called tandem accelerator. The first AMS radiocarbon dating of wood samples previously dated by conventional techniques was reported by Nelson *et al.* (1977). The results were encouraging and proved the usefulness of AMS for dating small samples containing only milligrams of carbon. AMS counts

Table 1. Preparation of samples for AMS radiocarbon dating at the Gliwice Radiocarbon Laboratory (GADAM Centre), based on Goslar & Czernik (2000). For more details including photographs see <http://www.carbon14.pl/c14lab/>.

The aim of sample preparation for AMS dating is to produce a graphite target from carbon originating from the sample. Because the samples are often very small (milligrams), much effort is put into avoiding contamination with either modern or inactive carbon during sample preparation. The vacuum lines used for preparation require a high and stable vacuum of at least  $10^{-4}$  mbar. The glassware used for preparation is carefully washed and heated at 400 °C, quartz tubes at 900 °C; the metal tools are cleaned in distilled water in an ultrasonic bath.

#### *Combustion*

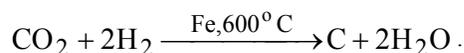
Carbon from sample extracts is released as carbon dioxide during the combustion process. This is carried out in a quartz tube with copper(II) oxide (the source of oxygen for combustion) and silver wool (for removal of gaseous sulphur and chlorine compounds). The tube with CuO (around 150 mg) and Ag (around 150 mg) is purified before further steps by sealing under vacuum after overnight pumping and heating at 900 °C for not less than three hours. The tube is then broken open, and a weighed quantity of sample material corresponding to approximately 1 mg of carbon sample introduced. The tube is connected to the vacuum line again, pumped to a high vacuum, sealed and placed in a muffle oven at 900 °C for about four hours.

#### *Purification of CO<sub>2</sub>*

The tube with CO<sub>2</sub> released from the sample is scratched and placed into an arm of vacuum line with a ball joint. After overnight pumping to high vacuum the arm is sealed off and the tube is cracked to release gases. Water vapour is sublimated in a trap cooled to around -70 °C with a mixture of dry ice and alcohol, and CO<sub>2</sub> is collected in a glass vial cooled with liquid nitrogen to about -180 °C. The amount of CO<sub>2</sub> collected is measured and the mass of carbon calculated, enabling estimation of the carbon concentration of the sample.

#### *Graphitisation*

CO<sub>2</sub> is reduced to graphite during the reaction with hydrogen at a temperature of 600–630 °C (depending on reduction rate for the specific sample) in the presence of iron as a catalyst. The summary reaction can be written as:



Graphite is deposited on the iron powder introduced into a small quartz tube. The ratio of mass of iron to mass of carbon (Fe/C) equals 2. The reactor and iron powder are first heated to 90 °C during continuous overnight pumping in order to remove contamination and residues of the sample graphitised previously. Immediately before graphitisation, the iron is oxidised for 15 minutes and reduced for 30 minutes at 400 °C in order to activate its catalytic properties. Then CO<sub>2</sub> and H<sub>2</sub> are introduced in stoichiometric amounts; the amount of CO<sub>2</sub> is equivalent to 1 mg carbon and approximately 20 % excess of H<sub>2</sub> is added to ensure complete reduction. The surplus CO<sub>2</sub> is stored in a separate vial and transferred to a special glass tube for the measurement of stable isotopes of carbon.

H<sub>2</sub>O produced during the reduction is instantly removed by freezing in a trap cooled with a mixture of dry ice and alcohol. The progress of the reaction is monitored by measuring the gas pressure. The reaction is usually complete after 3–4 hours.

#### *Pressing graphite targets*

The Fe-C powder is pressed into an aluminium holder to form a tablet, which is used as a target in the ion source of the AMS system. The targets are stored sealed in an argon atmosphere until the measurements are carried out.

#### *Measurement*

Prepared graphite targets are transported for AMS spectrometer measurements. Each batch is accompanied by at least three modern standards (prepared from Oxalic Acid II standard; Mann 1983) and three background samples (prepared from hard coal or marble containing negligible radioactive carbon), processed in the same way and at the same time as the dated samples.

Graphite targets produced from the samples are placed in the sputter ion source of the AMS system, which generates a current of negatively charged carbon ions. The ions with different masses are then sequentially injected into the channel of the electrostatic accelerator. They acquire high energy being accelerated by the electric field and change their charge during the stripping reaction with argon atoms. Positive ions leaving the accelerator are analysed in magnetic and electric fields in order to select the appropriate mass and energy. The currents of <sup>12</sup>C and <sup>13</sup>C ions are measured in Faraday cups and <sup>14</sup>C ions are detected and counted. Conventional radiocarbon ages are calculated from the carbon isotope concentrations measured in the test, modern standard and background samples.

are related to the total number of  $^{14}\text{C}$  atoms, and radioactive counts to the much smaller number of  $^{14}\text{C}$  atoms that disintegrate during the measurement. Another important advantage of the AMS method is the high efficiency of accelerator laboratories, in terms of the number of samples that can be measured. Although varying between laboratories, a counting time of tens of minutes per sample is typically needed to determine the  $^{14}\text{C}$  concentrations of contemporary samples with 1 % uncertainty, which in radiocarbon age measurement is equivalent to a standard error of about  $\pm 30$  years. Theoretically, more than 10,000 analyses per year should be possible. However, the capacity of laboratories capable of measuring AMS  $^{14}\text{C}$  is limited by the preparation throughput, as well as by high equipment and operating costs, which can reach millions of euros. At present, approximately 40 AMS spectrometers in different scientific institutions around the world are used for radiocarbon dating (the list can be found at <http://www.radiocarbon.org/Info/lablist.html>), and more institutes have preparation laboratories for AMS measurements. International intercomparison programmes with participation of the majority of laboratories have shown that results from AMS and conventional techniques are similar (see e.g. Scott 2003, Scott *et al.* 2010).

## 5. CALIBRATION OF RESULTS AND AGE-DEPTH MODELLING

To determine the time that has passed since the organisms in a peat sample died, it is necessary to compare the  $^{14}\text{C}$  isotope concentration in the sample with that in the organisms at the time of death. In radiocarbon dating a 'standard of modern biosphere' sample is used to define the  $^{14}\text{C}$  concentration at the time of death, and this provides the value of  $A_0$  in Equation 1. However, radiocarbon ages differ from 'real' (calendar or absolute) ages, mainly because the radiocarbon content of atmospheric  $\text{CO}_2$  fluctuates for various reasons such as changes in the Earth's magnetic field, changes in the carbon cycle and the influence of the solar wind on cosmic ray bombardment, as well as due to human influence. Thus, it is necessary to use a calibration procedure which takes these factors into consideration.

Discrepancies between radiocarbon ages and calendar ages in dated samples were first noted in the late 1950s. From that time, the issue became crucial to radiocarbon chronometry, and research is continuously undertaken. Calibration of the radiocarbon timescale involves determining the relationship between radiocarbon and calendar ages.

Knowledge of this relationship, expressed in the form of radiocarbon calibration curves, permits calibration of the radiocarbon age of any sample such that the calibrated age range is the best possible approximation to the sample's calendar age.

The construction of calibration curves relies on  $^{14}\text{C}$  dating of known-age archive samples such as dendrochronologically dated wood, corals dated by the U/Th method and annually laminated marine or lake sediments. The updated calibration curve IntCal09 is described by Reimer *et al.* (2009). This curve (Figure 2) shows clearly that the difference between radiocarbon age and calendar age generally increases with sample age, for example up to more than 1,000 years for samples from the early Holocene. A characteristic feature of the calibration curves is the presence of periods where single  $^{14}\text{C}$  ages correspond to multiple calendar ages, known as plateaux (periods of several hundred calendar years during which radiocarbon age is nearly constant) and wiggles (periods where radiocarbon age fluctuates rapidly). Radiocarbon dating of a single sample from a period of time that coincides with a plateau may mean that the calibrated calendar age will correspond to a time interval greater than a couple of centuries, with a corresponding large uncertainty. During a period with wiggles a single radiocarbon date may correspond to more than one age, each rather exact; but in such periods several radiocarbon dates may allow an unusually exact chronology to be made.

Calibration of a single radiocarbon age is performed using statistical methods. The probability density distribution of the calendar age of a sample is obtained (see example at Figure 3) and the ranges of 68.2 % and 95.4 % probability are calculated. Owing to the complex shape of the calibration curve, this distribution is asymmetric and may have several maxima. The calibration of a set of radiocarbon ages is usually performed with help of dedicated statistical programs such as OxCal (Bronk Ramsey 2008, 2009), BCal (Buck *et al.* 1999) or CALIB (Stuiver & Reimer 1993), and can be accompanied by age-depth model construction (e.g. Blaauw 2010, Blaauw *et al.* 2010, Bronk Ramsey 2008, 2009) in order to estimate the ages of undated depths in a peat deposit.

It is often assumed that peat deposits accumulate at rates which are relatively constant with few and gradual changes, so any age-depth model would tend to be rather 'smooth' apart from occasional hiatuses caused by local fires or bog bursts (Blaauw & Christen 2005). Such prior knowledge should ideally be incorporated when producing an age-depth model. However, use of one of the most

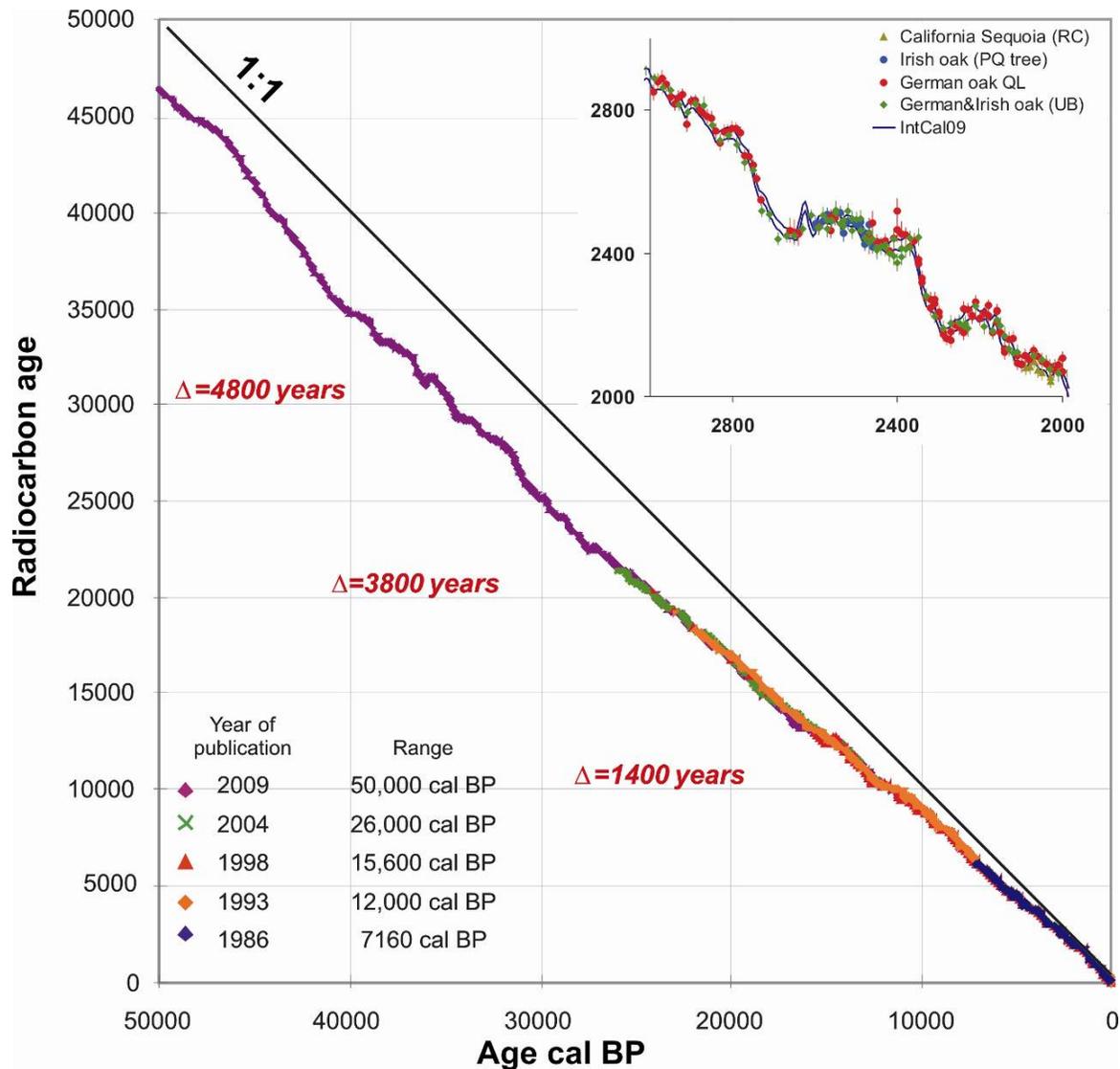


Figure 2. Historical comparison of radiocarbon calibration curves since 1986. The straight line would apply if radiocarbon and calendar ages were equal, and the  $\Delta$  values indicate approximate deviations of calendar ages from radiocarbon ages. The inset in the upper right corner shows a selection of the datasets used for construction of the radiocarbon calibration curve between 2 and 3 ka BP and the IntCal09 calibration curve. Datasets: IntCal09 (Reimer *et al.* 2009), IntCal04 (Reimer *et al.* 2004), IntCal98 (Stuiver *et al.* 1998), IntCal 93 (Stuiver *et al.* 1993), IntCal86 (Stuiver & Kra 1986). Datasets for the inset: German Oak QL, Irish oak (PQ tree) and California Sequoia (Stuiver & Braziunas 1993, Stuiver *et al.* 1998); German and Irish oak UB (Pearson *et al.* 1986).

popular types of age-depth model, namely linear interpolation between neighbouring dated depths, will result in abrupt and thus unrealistic accumulation rate changes between each pair of dated levels (Bennett 1994). An alternative is to apply a linear or higher polynomial regression to the dated depths, which will result in smoother age-depth curves that may not always pass through each individual dated level (e.g. Blaauw 2010). Age-

depth modelling often involves a trade-off between fitting a curve which passes through most if not all dated levels, and obtaining a smooth curve representing a 'likely' approximation of the true (but unknown) accumulation history of the peat deposit.

Age-depth models should become more realistic if they are constructed using underlying reasoning regarding expected shapes. Some theory suggests that bog peat accumulation slows down as the bog

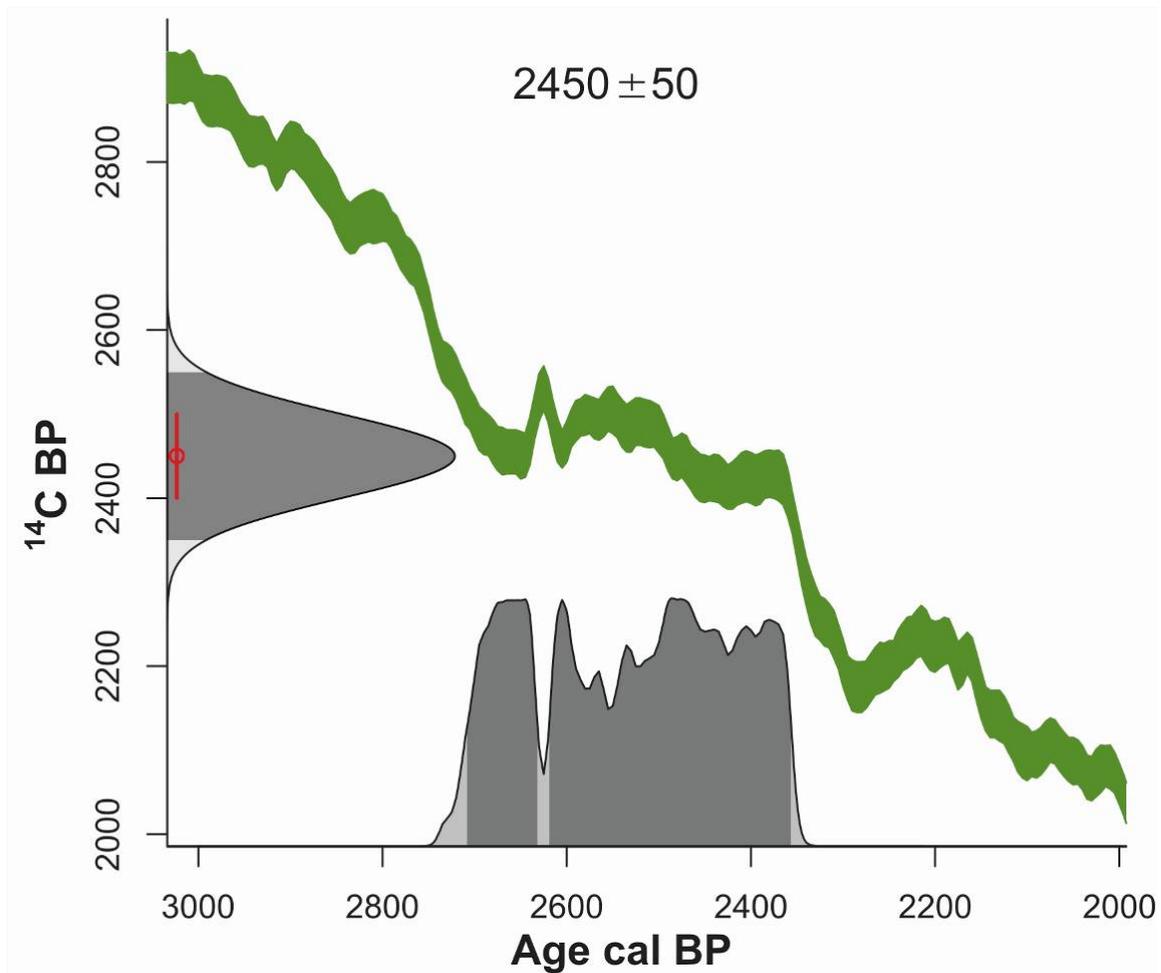


Figure 3. Example of the INTCAL09 calibration for a sample of radiocarbon age  $2450 \pm 50$  BP, shown (in red) as an open circle with 1 sigma range within the full probability distribution plotted on the vertical axis. The calibration curve is shown (in green), and the probability distribution of the sample's calendar age is plotted on the horizontal axis. Probabilities are plotted at arbitrary scale, and the dark grey areas represent 95.4 % ranges of age. This shows the wide possible calendar age range delivered for a sample with a radiocarbon age on a plateau in the radiocarbon calibration curve.

approaches its maximum height, giving a convex age-depth curve (Belyea & Baird 2006). On the other hand, concave age-depth models could also be conceived for bogs because autocompaction and continuous decomposition of organic matter would reduce apparent accumulation rates at depth (Yu *et al.* 2001). Linear accumulation, at least over limited time intervals, seems to be another credible assumption (Belyea & Clymo 2001). When applied to high-density  $^{14}\text{C}$  dated sequences, this can result in high-precision “wiggle-match” chronologies (e.g. van Geel & Mook 1989, Blaauw *et al.* 2003), where the  $^{14}\text{C}$  dates in a sequence are matched to the wiggles in the  $^{14}\text{C}$  calibration curve. Other more flexible types of age-depth model exist such as the

Poisson sequence in OxCal (Bronk Ramsey 2008), which simulates the accumulation of deposits through random small increments; or ‘Bacon’, which performs a comparable procedure while taking into account limitations on accumulation rate and its variability (Blaauw & Christen, accepted). It can be worthwhile to apply a range of age-depth models to a peat core in order to test different underlying assumptions (Yeloff *et al.* 2006, Blockley *et al.* 2007).

When interpreting fossil peat archives, it should be recognised that all dates and all models are uncertain (Telford *et al.* 2004). Often, calibrated dates possess multiple local maxima, are asymmetric, and correspond to longer time intervals

on the calendar scale than on the  $^{14}\text{C}$  scale. The choice of age-depth model type can greatly influence the ages assigned to specific depths, especially in peat cores which have been dated at low resolution. Numerical methods that aim to take into account these uncertainties are available; for example, proxies can be plotted as grey-scales that reflect their chronological uncertainty (Blaauw *et al.* 2007) and hypotheses regarding synchronicity, leads or lags between archives can be tested numerically rather than merely 'by eye' (e.g. Blaauw *et al.* 2007 and 2010, Parnell *et al.* 2008, Charman *et al.* 2009).

## 6. CASE STUDY

Situated 10 km from the Baltic Sea in the central part of the Polish coast, Słowińskie Błota is one of the country's best-preserved Baltic raised bogs. It covers an elliptical area of 1.2 km<sup>2</sup>. The contemporary vegetation is composed of several species of *Sphagnum* (*S. fallax* and *S. magellanicum*) with *Eriophorum vaginatum*, *Trichophorum cespitosum* and rare dwarf *Pinus sylvestris* (Herbichowa 1998). Near the outer edges, *Vaccinium uliginosum*, *Calluna vulgaris* and *Betula pubescens* are also present. A study involving collection of 59 cores revealed that the average thickness of the peat is 2.8 m and its stratigraphy is consistent over most of the bog. The base of the mire is dated at 3700–3960 BC. Raised bog vegetation (mainly *Sphagnum* and *E. vaginatum*) appeared around AD 50–360 (now 2 m below the surface), and *Sphagnum fuscum* peat started to develop between *ca* AD 470 and AD 1400 (Herbichowa 1998).

In 2006, two one-metre cores, spanning the present down to *ca.* AD 700 were taken from the centre of the bog for a high-resolution multiproxy investigation of past environmental conditions. Plant macrofossil, pollen, testate amoebae (Lamentowicz *et al.* 2009), stable carbon isotope, elemental geochemistry and lead isotope analyses were undertaken (De Vleeschouwer *et al.* 2009a, 2009b). An age-depth model for the profile was generated using the combined results of  $^{210}\text{Pb}$  dating (down to 34.5 cm depth) and  $^{14}\text{C}$  measurements for the deeper part of the core. A detailed description is included in De Vleeschouwer *et al.* (2009b), while Piotrowska *et al.* (2010) compared the results obtained for the uppermost 40 cm using the two methods.

The macrofossils for radiocarbon dating were selected carefully from one-centimetre-thick peat slices under a binocular microscope. Where insufficient *Sphagnum* macrofossils were available, other above-ground plants or large charcoal

fragments were chosen. Great care was taken to remove roots and rootlets in order to avoid contamination with carbon from overlying layers. Samples were pre-treated using an acid-alkali-acid washing sequence to remove any carbonate, bacterial CO<sub>2</sub> and humic/fulvic acids. The graphite targets were produced according to a protocol used in the Gliwice Radiocarbon Laboratory (see Box 1).  $^{14}\text{C}$  measurements were performed at Poznan Radiocarbon Laboratory (Poland; Goslar *et al.* 2004).

Calibration of radiocarbon dates was undertaken using Oxcal 4.0 software (Bronk Ramsey 2009). As *a priori* information, the  $^{210}\text{Pb}$ -derived ages were used in a Poisson sequence model (Bronk Ramsey 2008).

Calibration of radiocarbon dates was undertaken using the northern hemisphere terrestrial IntCal09 calibration curve (Reimer *et al.* 2009). All the  $^{210}\text{Pb}$  and  $^{14}\text{C}$  data were introduced into the 'Bacon' software (Blaauw & Christen, accepted). Using a Markov Chain Monte Carlo process, several thousands of independent likely age-depth models were constructed, each of them assigning calendar ages to all depths (dated or non-dated). From these models, calendar age distributions were constructed for all depths at a resolution of one centimetre. The distributions were illustrated as grey-scales, which depict more likely calendar ages as darker shades of grey (Figure 4). Sections of the age-depth model with lower chronological precision are indicated by lighter grey shades spread over larger areas. The resulting age-depth relationship can be written as a table, which provides the age of maximum probability and a range of 95.4% probability for each 1-cm slice of peat.

## 7. RECOMMENDATIONS

On the basis of this review, we can offer some practical recommendations for peat researchers, which are similar to those provided by Givélet *et al.* (2004) and Turetsky *et al.* (2004).

First of all, the material destined for dating must be carefully selected and identified, and further preparation steps should be discussed with radiocarbon laboratory staff. The results will always provide the ages of the selected fractions, and the question of how well the ages represent the dated levels needs to be addressed. This analysis is usually performed after collecting all radiocarbon dates and results from any other dating methods and other investigated proxies.

It is also advisable to perform reconnaissance dating at the beginning of the study, just after

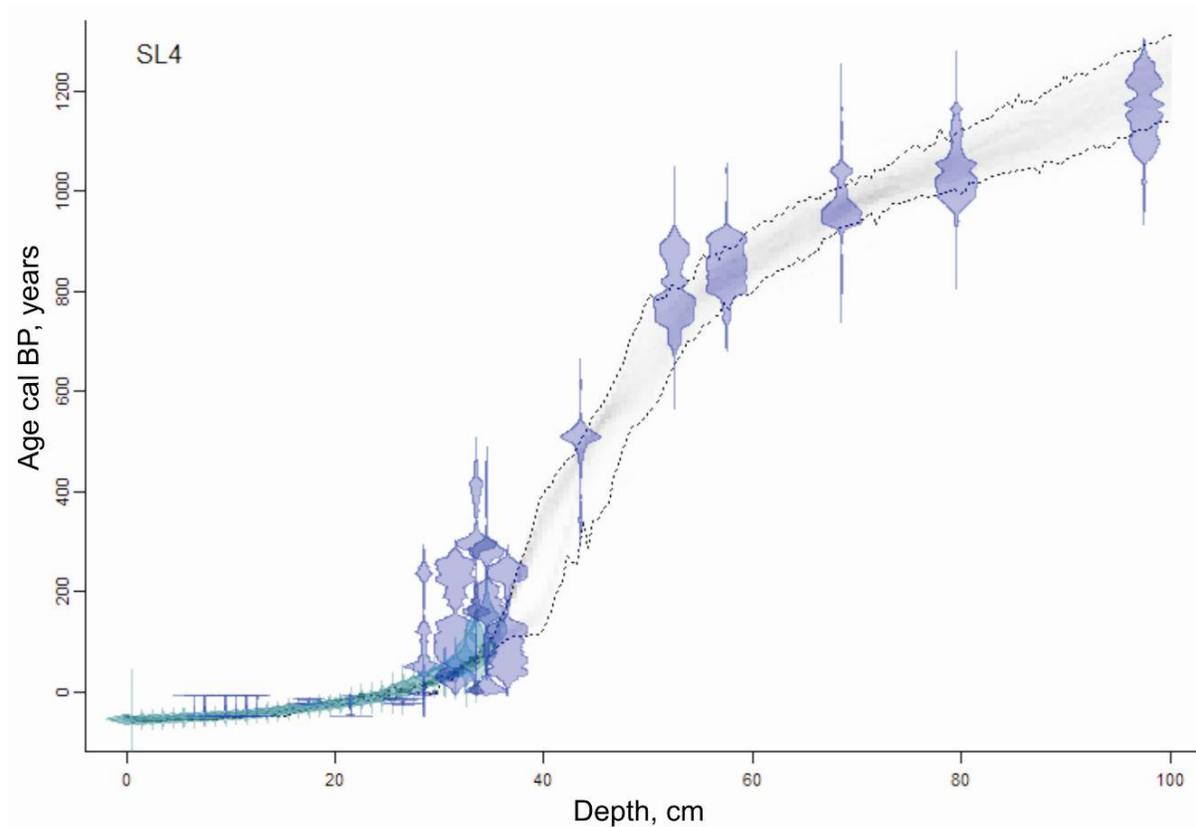


Figure 4. Results of radiocarbon and  $^{210}\text{Pb}$  dating, and age-depth model, for the Słowińskie Błota peat bog. See Section 6 for more details.

collecting the core. Usually, after the first dates become available, other preliminary results also emerge, and the levels of the core to be dated can be chosen subsequently. Such multiple-stage dating increases the possibilities for obtaining more precise age-depth models for the more interesting time intervals, e.g. those encompassing relevant changes in environmental or human activity indicators.

The next question is the choice of measurement technique to be applied, which is determined primarily by the amount of sample and the mass of selected C fraction available for analysis. For conventional techniques (GPC or LSC) a considerable amount (at least 1 g) of pure carbon is required, and even higher masses are desirable to get counting statistics which will provide a precision similar to AMS results within a reasonable time. Although small samples can be dated using radiometric techniques, the results will unavoidably have larger uncertainties (e.g. approximately 100 years for a contemporary sample of 0.3 g C). On the other hand, radiometric dating has some advantages. It usually provides an age for a sample which has been perfectly homogenised by combustion, which is especially desirable in the case of bulk peat

dating; and the cost of radiometric dating is around half that of AMS dating. However, the most commonly used modern coring tools (standard Russian or Wardenaar corers, De Vleeschouwer *et al.* 2010) would not secure a sufficient quantity of material for radiometric dating to be carried out in combination with multiproxy research. Thus, the main practical advantage of the AMS technique is the reduction of required sample mass to around 1 mg of pure carbon. Furthermore, AMS dating can be performed in a shorter time than radiometric measurements, especially when more than 5–10 samples are to be considered.

A subsequent step should inevitably be calibration of the radiocarbon dates. Although the uncalibrated results should always be reported in publications, only calibrated ones provide a robust basis for any interpretations in relation to the calendar age scale. If the interpretation or age-modelling tool requires point estimates of calibrated ages, their choice should be clearly described. Information about the calibration curve and programme used (with references), as well as any age-modelling technique applied to the results, should also be provided (Blaauw 2010). The age-

modelling technique should be carefully chosen, with understanding of underlying assumptions and accompanying uncertainties.

Modern investigations of past climate and human activity are often performed with an emphasis on obtaining the highest possible resolution for a given archive. However, it seems that the importance of robust and precise age models is still underestimated (Blaauw 2010) and, while a considerable amount of effort is devoted to multiproxy and interdisciplinary investigations, high-resolution dating is still a rarity, mostly because it is expensive. As shown by Hajdas & Michczyński (2010) using an example of laminated lake sediments, the uncertainty of an age-depth model based solely on radiocarbon dating can be similar to the uncertainties connected with annual layer counting; but exercises like this require dozens of dates per millennium. Although such resolution is unattainable for most users and sites, there is still a strong relationship between the number of dates and the resulting precision and accuracy of age models.

The robustness of age models can be significantly improved, and the uncertainties reduced, by using multiple dating methods on a single core (see Section 6 and Turetsky *et al.* 2004). Multiple dating allows for corroboration between various methods which would support one another in providing accurate chronologies (Marshall *et al.* 2007, De Vleeschouwer *et al.* 2009b). Most commonly, the uppermost layer can be dated using atmospheric fall-out radionuclides (e.g.  $^{210}\text{Pb}$ ; see Le Roux & Marshall 2011), and tephrostratigraphy can potentially be applied throughout the core (Swindles *et al.* 2010). With suitable statistical treatment, all results can be combined into one reliable chronology which provides the backbone for interpretations of palaeoclimatic and palaeoenvironmental change data.

## 8. ACKNOWLEDGEMENTS

The initial idea for this paper stemmed from the workshop *Peatland Archives of Holocene Climate Variability* held in Estonia in May 2009, which was sponsored by the National Science Foundation (USA), the Past Global Changes (PAGES) International Project Office, the International Union for Quaternary Research, the Quaternary Research Association (UK) and the University of Tartu (Estonia). The material presented was also a subject of the Paleochronology Building Workshop, San Miguel de Allende, Mexico (August 2010), sponsored by INQUA and PAGES. We thank François De Vleeschouwer for motivating us to start and complete this work; and William Marshall, R.S.

Clymo and an anonymous reviewer for help in improving this article.

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Submitted 22 Jun 2010, final revision 16 May 2011

Editor: Olivia Bragg

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Author for correspondence:

Dr Natalia Piotrowska, Department of Radioisotopes, GADAM Centre of Excellence, Silesian University of Technology, Institute of Physics, Krzywoustego 2, 44-100 Gliwice, Poland

Tel: +48 32 237 2663; Fax: +48 32 237 2254; E-mail: natalia.piotrowska@polsl.pl