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Pitfalls in the AMS radiocarbon-dating of terrestrial macrofossils

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ABSTRACT: The AMS ^{14}C technique has the advantage that small samples of Late Quaternary age can be dated with high accuracy, and that errors due to reservoir effects can be avoided if specifically determined terrestrial micro- and macrofossils are measured. However, to obtain such high-accuracy measurements, it is important how small samples are handled prior to treatment in the radiocarbon laboratories. Here we present a set of 51 AMS ^{14}C measurements, of which 31 dates gave expected ages and 20 dates resulted in anomalously young ages, despite the fact that all samples consisted of clearly identified Late Weichselian terrestrial plant macrofossils. To evaluate possible sources of error, we compared these samples in respect to preparation methods, sample storage and sample weight. Our results show that the long-term storage of wet macrofossil samples appears to have a significant effect on the radiocarbon age obtained, even when the samples are kept cool. Fungi or micro-organisms may easily be incorporated into a sample during preparation and identification, and can easily contribute to the contamination of a sample, if stored cool and wet for several months. © 1998 John Wiley & Sons, Ltd.

KEYWORDS: AMS radiocarbon dates; terrestrial plant material; preparation methods; anomalously young ages.

Introduction

'If a ^{14}C date supports our theories, we put it in the main text. If it does not entirely contradict them, we put it in a foot-note. And if it is completely "out of date", we just drop it' (Säve-Söderbergh & Olsson, 1970, p. 35, citing the American archeologist Brew).

The last decade has seen an increasing application of the accelerator mass spectrometry (AMS) technique for radiocarbon dating Late Quaternary sediments. Compared with a conventional radiocarbon measurement, the AMS method has the advantage that small samples can be dated with high accuracy and, if only well-determined terrestrial macro- or microfossils are measured, errors due to reservoir effects (Olsson, 1986) can be avoided. Although terrestrial plant fragments, such as leaves, seeds and fruits are ideal for AMS ^{14}C measurements, problems may be encountered in that the samples may give unexpectedly old or young ages. A measurement older than expected can be explained through the contamination of the sample by older, reworked and more resistant plant material. Dates that are much younger than expected are, however, more difficult to interpret, especially when contamination by recent or subrecent plants can be excluded.

Within a research project that originally was designed to AMS ^{14}C date Swedish Late Weichselian varved clays (Wohlfarth *et al.*, 1993), we have measured a total of 71 terrestrial macrofossil samples during the last few years. Out of these, 36 samples resulted in 'correct' radiocarbon ages, 32 samples were several hundred to several thousand years younger than expected, and only 3 samples were considered as too old. The unexpectedly large number of samples that gave radiocarbon ages that were too young led us to examine closely the likely contamination sources. The errors obtained for 12 out of 32 anomalously young dates may be explained by the fact that fragments of subrecent Gramineae, undetermined leaves, wood, *Betula pubescens, Pinus* or insects were included in the subsamples. The remaining 20 samples with unexpectedly young ages consisted of leaves, seeds, fruits or flower stems of typical Late Weichselian plants (Liedberg-Jönsson, 1988) (e.g. *Betula nana, Dryas octopetala* and *Salix* species). However, these plants did not grow in southern Sweden during the last 9000 ^{14}C years BP. Contamination by Holocene or subrecent plant material can thus be excluded and other factors, such as sample storage, sample size and/or sample preparation have to be considered as likely contamination sources.

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To evaluate the contribution of these different factors in our dating series, we compare here a selected set of 51 dates, which consist entirely of clearly identified Late Weichselian terrestrial plant macrofossils. All other samples, which included, for example, unidentified plant material, wood fragments or insects are excluded. Of these 51 samples, we regard 31 dates as acceptable and 20 dates as too young, and we compare these to each other with respect to preparation procedures and carbon content.

Study area and methods

The AMS ^{14}C dates presented here were obtained on terrestrial plant macrofossils from Late Weichselian glacial varved clays. These clays were deposited as annually laminated sediments in the Baltic Ice Lake during the recession of the Fennoscandian inland ice and can now, due to isostatic land uplift, be easily accessed in land areas along the present shore of the Baltic (Fig. 1). The glacial varved clays have the advantage that (i) deposition occurred in the same sedimentary basin and (ii) that varve diagrams from different coring sites can easily be correlated with each other within nearby areas. The matching of successively overlapping varve-thickness diagrams from south to north, in the direction of the ice recession, has led to the establishment of the so-called Swedish Time Scale (see summary in Wohlfarth *et al.*, 1993), which is a varve chronology more than 13 000 yr long.

Our investigations concentrated on the oldest varved clays in southeastern Sweden, where varve chronologies had been established in the provinces of Blekinge (Ringberg, 1991), Småland, Östergötland (Kristiansson, 1986) and Västergötland (Strömberg, 1994) (Fig. 1). According to pollen-stratigraphic investigations, glacial varves were deposited during the later part of the Bølling and during the Older Dryas pollen zone in Blekinge and during the early part of the Allerød in Småland (Björck and Möller, 1987; Wohlfarth *et al.*, 1994; J. Ising, in preparation). The varved clays in the southern part of Östergötland can be attributed to the later part of the Allerød and to the beginning of the Younger Dryas pollen zone (J. Björck, in preparation). In Västergötland, varved clays were deposited during the Younger Dryas pollen zone (Björck and Digerfeldt, 1989).

Multiple corings for varved clays were carried out at different localities in Blekinge, Småland, Östergötland and Västergötland, in order to obtain sufficient plant material for AMS ^{14}C measurements (Table 1a–c). For each locality we established varve-thickness diagrams, which were, within the respective area, correlated to each other and to the local varve chronology (Wohlfarth *et al.*, 1994; Wohlfarth *et al.*, in preparation). Based on this correlation, the same varve-year intervals were subsampled (25–100 yr sample^{-1}) at each locality. In this way, all samples that were obtained from, e.g. Blekinge, range within the same time interval of ca. 300 varve years. Irrespective of the depth of the samples or the

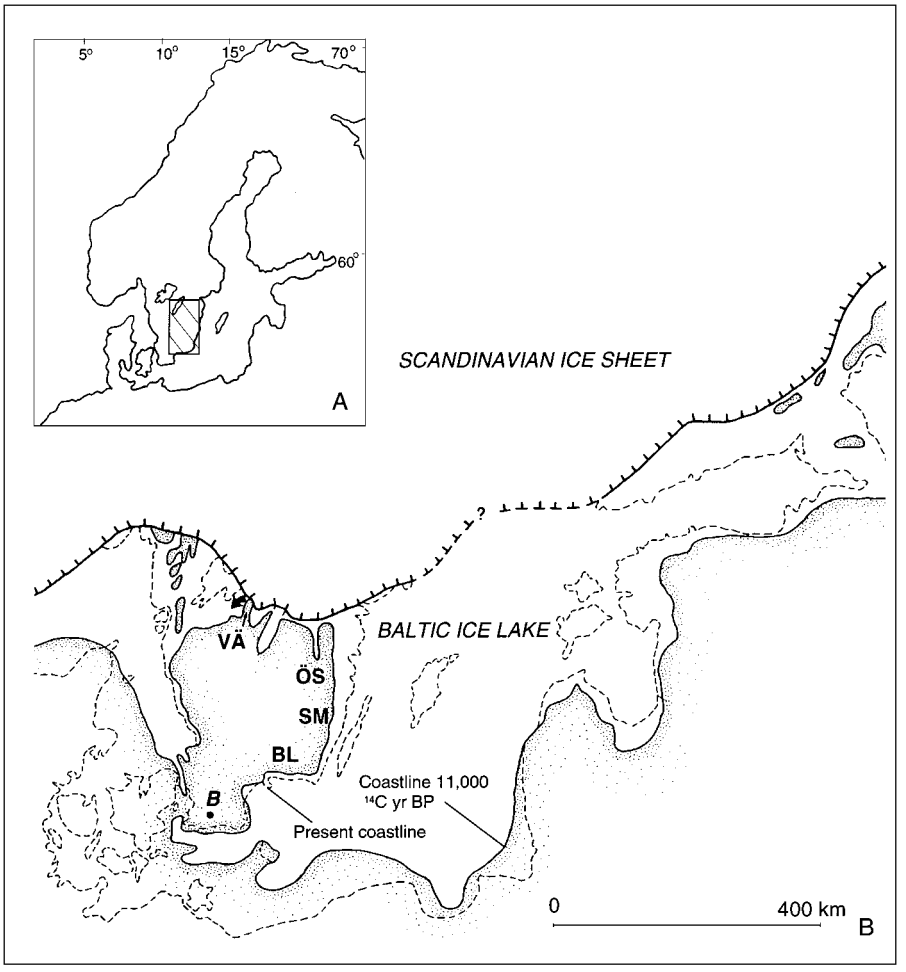

Figure 1 (A) Map of Scandinavia showing the study area and (B) the location of the investigated varved-clay sites in relation to the Baltic Ice Lake and to the present coast line (after Björck, 1995). BL = Blekinge, SM = Småland, ÖS = Östergötland, VÄ = Västergötland. The peat bog Bjärsjölagård, where test samples were obtained, is marked with 'B'.

Table 1 (a) Sample series 1, submitted in slightly acid distilled water, no pre-treatment carried out: Lf = leaves and leaf fragments, F = flower, B = budscales

Laboratory number Ua-	Locality	Macrofossils submitted	Dry weight (mg)	Carbon content (mg)	Preparation time[a,b] (months)	AMS radiocarbon date (yr BP)	$\delta^{13}C$ (‰) PDB
Blekinge group							
2468	Farslycke	*Salix reticulata* (Lf), *Salix* cf. *polaris* (Lf)	1.31	0.38	24(3)	5730 ± 330[c]	–
3132	Dönhyltagyl	*Dryas octopetala* (Lf, F), *Salix polaris* (Lf)	1.42	–	7(7)	12 090 ± 185	–
11514	Skälgylet	*Salix polaris* (Lf), *Salix reticulata* (Lf), *Salix/Betula* (Lf)	1.09	0.90	13(10)	9522 ± 125[c]	–
11515	Metegylet	*Salix polaris* (Lf), *Salix/Betula* (Lf)	0.43	0.30	13(10)	9811 ± 130[c]	–
11516	Långasjön	*Salix polaris* (Lf), *Salix/Betula* (Lf)	2.2	0.51	13(10)	7201 ± 100[c]	–
11517	Mossjön	*Salix polaris* (Lf), *Salix/Betula* (Lf)	1.07	0.60	13(10)	7814 ± 110[c]	–
11522	Farslycke	*Salix/Betula* (Lf, F)	0.71	0.30	7(7)	8719 ± 105[c]	–28.76
11523	Sandsjön	*Salix/Betula* (Lf), *Dryas octopetala* (F)	0.39	0.15	7(7)	3154 ± 150[c]	–
11524	Skälgylet	*Salix/Betula* (Lf)	0.7	0.20	7(7)	9147 ± 130[c]	–
11525	Skälgylet	*Salix polaris* (Lf), *Salix reticulata* (Lf) *Salix/Betula* (Lf), *Dryas octopetala* (Lf)	6.59	3.03	7(7)	11 354 ± 140[c]	–28.82
Småland group							
11519	Lillsjön	*Salix/Betula* (F), *Betula nana* (Lf), *Dryas octopetala* (F, Lf)	0.6	–	3(2)	9572 ± 90[c]	–
Östergötland group							
2543	Hargsjön	*Salix/Betula* (B, Lf), *Salix polaris* (L, Lf), *Dryas octopetala* (Lf)	1.12	0.32	2(1)	9910 ± 140[c]	–
11518	Tynn	*Salix/Betula* (Lf), *Dryas octopetala* (Lf, F)	0.4	–	3(7)	10 511 ± 130	–
11526	Hargsjön	*Salix/Betula* (Lf), *Betula nana* (Lf)	1.1	0.80	11(7)	8525 ± 130[c]	–29.33
11527	Hargsjön	*Salix* indet. (Lf)	1.81	0.91	11(7)	10 384 ± 130	–27.82
11528	Hargsjön	*Salix polaris* (Lf), *Salix/Betula* (Lf), *Dryas octopetala* (Lf)	1.02	0.54	11(7)	7694 ± 120[c]	–
11529	Adlerskogssjön	*Salix polaris* (Lf), *Salix/Betula* (Lf), *Betula nana* (Lf)	1.43	1.06	11(7)	8395 ± 100[c]	–26.57
11530	Adlerskogssjön	*Salix/Betula* (Lf)	0.82	0.50	11(7)	6584 ± 80[c]	–
Västergötland group							
2741	Mullsjön	?*Dryas octopetala* (Lf)	1.23	0.22	60(3)	9640 ± 190	–
2742	Mullsjön	*Betula/Salix* (Lf), brown mosses (Lf, F, stems)	2.47	0.77	60(3)	9945 ± 115	–

coring locality, we therefore know the exact varve-year age of each sample.

Macrofossils were extracted from the varved clays by sieving the sediment under running water through a 0.5 mm mesh. After identification under a binocular microscope, terrestrial macrofossils, consisting mainly of leaves and leaf fragments of *Salix* species, *Betula nana* and *Dryas octopetala*, were selected for radiocarbon dating (Table 1a–c). In some cases the samples also contained leaves, seeds or flowers of *Saxifraga, Arenaria, Oxyria, Ranunculus ?glacialis* and undifferentiated Caryophyllaceae and Ericacae.

In the first and second dating series (Table 1a and b), the selected macrofossils were submitted to the AMS facility in de-ionised water, which was acidified by a few drops of

Table 1 Continued

(b) Sample series 2, submitted in slightly acid distilled water, pre-treatment carried out: Lf = leaves and leaf fragments, S = seeds, B = budscales

Laboratory number Ua-	Site name	Macrofossils submitted	Dry weight (mg)	Carbon content (mg)	Preparation time[a,b] (months)	AMS radiocarbon date (yr BP)	$\delta^{13}C$ (‰) PDB
Blekinge group							
4245	Skälgylet	*Salix polaris* (Lf), *Salix/Betula* (Lf)	0.77	0.3	1(1)	12 330 ± 370	−29.66
4246	Skälgylet	*Salix polaris* (Lf), *Salix/Betula* (Lf)	6.28	3.3	1(1)	12 590 ± 130	−28.72
4247	Farslycke	*Salix/Betula* (Lf)	0.7	0.3	1(1)	12 595 ± 360	−29.77
4248	Farslycke	*Salix polaris* (Lf), *Salix/Betula* (Lf)	3.44	1.51	1(1)	12 310 ± 145	−28.59
4249	Skälgylet	*Salix polaris* (Lf)	0.9	0.8	1(1)	11 045 ± 170[c]	−29.20
Västergötland group							
4212	Mullsjön	*Salix herbacea* (Lf), *Salix* indet. (Lf)	2.76	1.68	4(3)	10 160 ± 115	−28.81
4213	Mullsjön	*Salix herbacea* (Lf), *Salix* indet. (Lf), *Betula nana* (Lf)	0.45	0.4	4(3)	8740 ± 185[c]	−27.50
4214	Mullsjön	*Salix* indet. (Lf, B), *Salix/Betula* (Lf), *Betula nana* (Lf)	0.48	0.4	4(3)	10 170 ± 195	−28.37
4215	Mullsjön	*Salix* indet. (Lf), *Salix herbacea* (Lf), *Betula nana* (Lf), *Salix/Betula* (Lf), *Oxyria* (S), Caryopyllacea (S)	1.05	0.8	4(3)	10 140 ± 155	−30.00
4216	Mullsjön	*Salix* indet. (Lf), *Betula nana* (Lf), *Oxyria* (S)	1.8	1.10	4(3)	10 620 ± 155	−27.97
4217	Mullsjön	*Salix herbacea* (Lf)	1.11	0.7	4(3)	10 330 ± 175	−30.85

(c) Sample series 3, submitted dried, pre-treatment applied: Lf = leaves and leaf fragments, S = seeds, F = fruits

Laboratory number Ua-	Site name	Macrofossils submitted	Dry weight (mg)	Carbon content (mg)	Preparation time[a,b] (months)	AMS radio carbon date (yr BP)	$\delta^{13}C$ (‰) PDB
Småland group							
4635	Lillsjön	Salix polaris (Lf), *Salix/Betula* (Lf), *Dryas octopetala* (Lf)	1.2	0.75	6(2)	11 895 ± 860	−25.71
4945	Lillsjön	*Salix/Betula* (Lf)	2.8	1.29	8(4)	11 530 ± 130	−29.75
4946	Lillsjön	*Dryas octopetala* (Lf), *Salix/Betula* (Lf)	2.17	1.30	8(4)	10 515 ± 165[c]	−29.16
Östergötland group							
4358	Hargsjön	*Salix polaris* (Lf)	4.26	2.39	2(1)	10 980 ± 100	−29.19
4359	Hargsjön	*Dryas octopetala* (Lf), *Salix polaris* (Lf)	2.53	1.50	2(1)	10 610 ± 110	−29.01
4493	Adlerskogssjön	*Salix polaris* (Lf), *Dryas octopetala* (Lf, F)	3.59	2.26	4(2)	10 830 ± 165	−27.80
4496	Glottern	*Salix/Betula* (Lf)	3.0	0.24	4(2)	10 585 ± 465	−29.33
10181	Gummmetorpasjön	*Saxifraga* (F), *Salix* indet. (Lf), *Silene* (Lf)	1.5	1.13	2(1)	11 450 ± 240	−30.47
10182	Gummmetorpasjön	Ericacea (Lf, F), *Salix/Betula* (Lf), *Dryas octopetala* (Lf)	5.4	3.02	2(1)	11 470 ± 130	−28.23

Table 1 Continued

Laboratory number Ua-	Site name	Macrofossils submitted	Dry weight (mg)	Carbon content (mg)	Preparation time[a,b] (months)	AMS radiocarbon date (yr BP)	$\delta^{13}C$ (‰) PDB
10183	Gummmetorpasjön	*Dryas octopetala* (Lf), *Betula/Salix* (Lf)	4.2	2.60	2(1)	11 030 ± 120	−28.57
10184	Gummmetorpasjön	*Salix/Betula* (Lf), *Dryas* octopetala (Lf)	8.7	4.52	2(1)	10 970 ± 90	−28.58
10185	Gummmetorpasjön	*Salix reticulata* (Lf), *Salix* polaris (Lf), *Salix* indet. (Lf), *Ericacea (Lf), Dryas octopetala* (Lf)	5.3	3.29	2(1)	11 230 ± 100	−28.60
10186	Gummmetorpasjön	*Dryas octopetala* (Lf), *Salix/Betula* (Lf)	7.5	4.35	2(1)	11 040 ± 110	−28.99
10187	Gummmetorpasjön	*Dryas octopetala* (Lf), *Salix/Betula* (Lf)	1.5	1.19	2(1)	10 420 ± 220	−31.10
11233	Nedre Emmaren	*Salix polaris* (Lf), *Salix* indet. (Lf), *Dryas octopetala* (Lf, F), *Betula nana* (Lf)	1.7	0.5	14(8)	10 745 ± 240	−28.88
11234	Nedre Emmaren	*Salix polaris* (Lf), *Salix* indet. (Lf), *Dryas octop etala* (Lf, S), *Arenaria* (Lf)	2.5	0.4	14(8)	10 885 ± 250	−29.27
11236	Nedre Emmaren	*Dryas octopetala* (Lf, F)	1.9	0.5	14(8)	8915 ± 180	−29.30
11610	Gummmetorpasjön	*Salix polaris* (Lf), *Salix* indet. (Lf), flower stem (?*Dryas octopetala*)	3.7	1.5	3(2)	10 415 ± 105	−28.50
11611	Gummmetorpasjön	*Salix polaris* (Lf), *Salix* indet. (Lf), *Dryas octopetala* (Lf), flower stem (?*Dryas octopetala*)	4.0	1.6	3(2)	10 280 ± 125	−28.64
11612	Gummmetorpasjön	*Salix polaris* (Lf), *Salix* reticulata (Lf), *Salix/ Betula* (Lf), *Dryas octopetala* (Lf, F, S), ?*Ranun-culus glacialis* (Lf)	13.9	3.2	3(2)	9575 ± 75	−27.69

[a]Time interval between coring and measurement.
[b]Time interval between sample preparation and measurement.
[c]Radiocarbon ages too young.

10% HCl. The glass bottles in which the samples were stored had first been rinsed in de-ionised water. The samples were kept in a dark and cool (5°C) environment prior to ^{14}C measurement. For the first sample series, the time interval between fieldwork and AMS ^{14}C measurement was between 2 and 60 months and between sample selection and measurement 1 to 10 months (Table 1a). In the second sample series, we reduced the time interval between fieldwork, sample selection and measurement to between 1 and 4 and 1 and 3 months, respectively (Table 1b). In contrast to the second series, samples of the first series were not pre-treated in the AMS laboratory (see description below). This was justified by the fact that the sediments contained no calcium carbonate and that an infiltration of humic acids into these heavy and thick clays could only have been of minor importance. The macrofossil samples selected for the third dating series (Table 1c) were, after identification, immediately dried in an aluminium foil package at 50°C overnight. The dried samples were then stored in a glass bottle, which had been sterilised at 105°C. The pre-treatment performed in the radiocarbon laboratory on series 2 and 3 included 1% HCl (6 h < 100°C) and 0.5% NaOH (1 h at 60°C). The samples in series 3 were measured, at the latest, within 14 months after fieldwork and within 8 months after sieving. In all three series, the dried material was acidified with HCl (pH 4), burnt to CO_2 and converted to graphite through a Fe-catalytic reaction. The standard procedures and the corrections for background ^{14}C of our samples have been described earlier (Wohlfarth *et al.*, 1993).

Discussion

On the basis of replicate radiocarbon measurements on the same sample intervals at the same localities (Farslycke,

Skälgylet in Blekinge; Lillsjön in Småland; Hargsjön, Adlerskogssjön, Gummetorpasjön in Östergötland; Mullsjön in Västergötland, see Table 1a–c), we know the expected age range in radiocarbon years for each sample. Furthermore, through the correlation of varve-thickness diagrams between close-by localities within each area (e.g. Metegyl, Långasjön, Mossjön, Sandsjön, Farslycke in Blekinge), we are confident that the samples measured correspond to the same varve-year intervals. Our age estimates are corroborated by pollen-stratigraphic investigations, which had been performed on time-synchronous varve sequences (Björck and Möller, 1987; Björck and Digerfeldt, 1989; J. Ising, in preparation; J. Björck, in preparation). These investigations provide an independent estimate of age through a comparison with radiocarbon-dated pollen zones in southern Sweden. The expected radiocarbon age of the oldest samples from Blekinge, where varved clays were deposited during the end of the Bølling, lies between 12 000 and 12 600 ^{14}C yr BP (mean 12 300 ^{14}C yr BP) (Wohlfarth *et al.*, 1994). The samples from Småland range between 11 500 and 12 000 ^{14}C yr BP (mean ca. 11 750 ^{14}C yr BP), which corresponds to the early Allerød. In Östergötland, varved clay deposition started during late Allerød and ended in the Younger Dryas. Therefore, the correct radiocarbon ages of these samples fall into two different categories, i.e. in the later part of the Allerød (10 900–11 500 ^{14}C yr BP, mean 11 200 ^{14}C yr BP) and in the early Younger Dryas (10 300–10 800 ^{14}C yr BP, mean 10 550 ^{14}C yr BP). The samples from Västergötland are from varved clays that were deposited during the later part of the Younger Dryas (10 000–10 600 ^{14}C yr BP, mean 10 300 ^{14}C yr BP) (Björck and Digerfeldt, 1989).

The apparently young radiocarbon ages obtained for Blekinge extended from ca. 6000 to ca. 11 000 ^{14}C yr BP and for Småland extended from ca. 9600 to ca. 10 500 ^{14}C yr BP. In samples from Östergötland and Västergötland, the young ages range from ca. 6600 to ca. 9900 ^{14}C yr BP. As shown in Table 1a–c, most samples that resulted in anomalously young ages were obtained in sample series 1. Contamination by Holocene plant material can be excluded because only typical Late Weichselian macrofossils from plants that were not growing in this part of Sweden during the last ca. 9000 ^{14}C yr BP were selected for radiocarbon measurements (Table 1a–c).

The most obvious difference between the 51 samples can be found in the sample storage and the pre-treatment prior to radiocarbon dating and in sample size. The macrofossils in sample series 1 and 2 were stored wet and cool, whereas the plant material was dried immediately after identification in sample series 3. The time interval between fieldwork, subsampling, identification of the macrofossils and radiocarbon measurement also differed significantly between the three series (Table 1a–c). Furthermore, sample size was in general larger in sample series 3, which resulted in a higher carbon content in these samples. To evaluate the contribution of these different factors on our samples, we correlated the deviation from the expected radiocarbon age for each sample series separately to (i) the time interval between coring and measurements, (ii) the time interval between preparation and measurement and, (iii) to the carbon content (Table 2). The age deviation of the apparently young radiocarbon dates was calculated by assuming the following 'correct' mean radiocarbon age: 12 300 for the samples from Blekinge; 11 750 for the samples from Småland; 11 200 and 10 550 for the samples from Östergötland; and 10 300 for the samples from Västergötland. Expected radiocarbon ages were set to '0'.

When each sample series is compared separately with the different variables, none of the correlations is significant, even when samples Ua-2741 and Ua-2742, which had an exceptionally long storage time, are excluded. A significant correlation can be found only when the three sample series are compared jointly with the time interval between sample identification and measurement ($r = 0.55$) and to the carbon content ($r = -0.38$) (Table 2). In contrast, the time interval between coring and measurement is significant only when samples Ua-2741 and Ua-2742 are excluded ($r = 0.58$) (Table 2, Fig. 2a). Obviously, the storage time of the identified macrofossil samples and the carbon content are strongly correlated with the radiocarbon ages obtained (Fig. 2b and c). Long storage time and/or low carbon content seem to result in radiocarbon ages that are too young.

Long-term sample storage

Most anomalously young ages were obtained in sample series 1, where the time period between coring and measurement and between selection of plant macrofossils and measurement was considerably longer than in sample series 2 (Fig. 2a and b). Furthermore, the macrofossil samples in series 1 were stored in de-ionised water and were not subject to any pre-treatment prior to radiocarbon measurement, whereas the wet-stored samples in series 2 were pre-treated. The samples in series 3 were dried immediately after identification and pre-treated prior to radiocarbon measurement.

In one sample, which was submitted together with samples Ua-11514 to Ua-11518 and Ua-11522 to Ua-11530 from the first dating series, and where the macrofossils had been stored wet and cool for >7 months prior to the radiocarbon measurement, we observed a dense white cover on the leaf surfaces. The macrofossils in this sample were not dated, but the coating was cultivated and could be determined as the fungus *Paecilomyces farinosus* (Holm ex Gray) (O. Constantinescu, pers. comm., 1994). This fungus is an insect parasite, which is equally common in temperate and tropical zones, with forest soils as the preferred habitat (Domusch *et al.*, 1980). As samples Ua-11514 to Ua-11518 and Ua-11522 to Ua-11530 were prepared at approximately the same time, the possibility exists that they may have become contaminated by the same fungus during sieving and/or identification. Even when stored in slightly acid water (pH ~2) and under cool and dark conditions, *Paecilomyces farinosus* may easily grow and absorb CO_2 from the remaining air in the glass bottle and from the ambient water (O. Constantinescu, pers. comm.).

Experiments performed on the assimilation of $^{14}CO_2$ by the fungus *Fusarium oxysporum* under oligotrophic conditions, have shown that 0.78% of the cell's carbon is derived from CO_2 and 99.22% originates from other sources, which include pre-formed cell material and trace organic contaminants in the medium and in the atmosphere (Parkinson *et al.*, 1990, 1991). Compared with the usual handling of sediment and plant macrofossil samples in a laboratory, these experiments were carried out under strictly controlled conditions, which excluded exogenous organic contaminants during preparation (Parkinson *et al.*, 1991). Macrofossil samples that are used for AMS ^{14}C measurements are usually not handled in a sterile environment, and during all steps in the preparation process, samples can easily become contaminated. If we consider fungal growth as the main contamination source for our samples, then it is likely that the fungi took up CO_2 from the fossil leaf material, but that its metabolism also included assimilation of CO_2 from

Table 2 Correlation coefficient calculated between age deviation, storage time and carbon content for the different sample series. The correlation analysis indicates that none of these correlations is significant, except for the last comparison, where all sample series are compared jointly

Sample series	Time interval between coring and measurement	Time interval between identification and measurement	Carbon content
1	$r = -0.24/r = 0.41$[a] (20/18 observations)	$r = 0.39$ (20 observations)	$r = -0.30$ (19 observations)
2	$r = 0.01$ (11 observations)	$r = 0.01$ (11 observations)	$r = -0.25$ (11 observations)
3	$r = 0.39$ (20 observations)	$r = 0.27$ (20 observations)	$r = -0.14$ (20 observations)
1 + 2 + 3	$r = 0.13/r = 0.58$[a] (51/49 observations)	$r = 0.55/r = 0.54$[a] (51/49 observations)	$r = -0.38$ (50 observations)

[a]Samples Ua-2741 and Ua-2742, where the cores were stored for 60 months prior to sieving, are excluded.

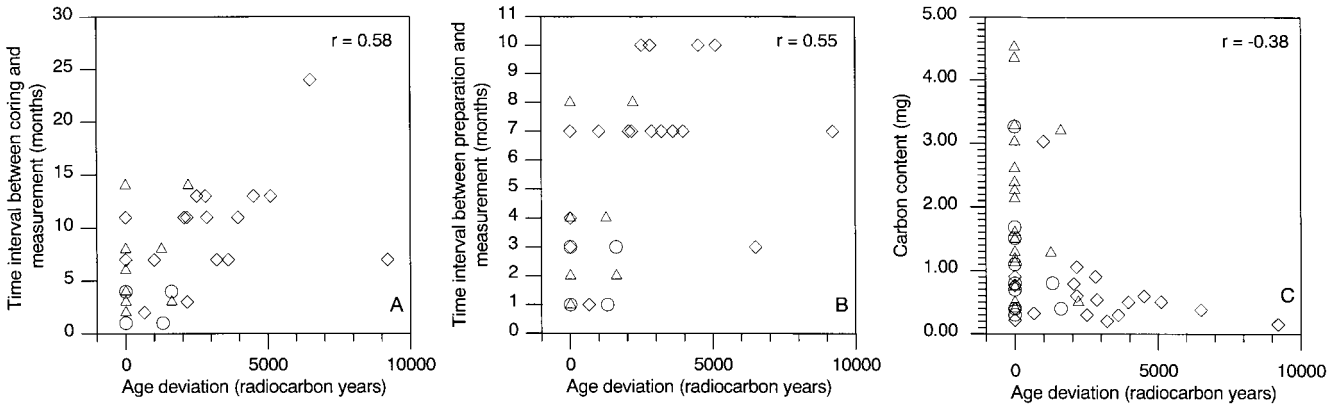

Figure 2 Comparison between age deviation and (A) the time interval between coring and sample measurement, (B) the time interval between sample preparation and measurement and, (C) the carbon content of the three sample series (sample series 1, open diamonds; sample series 2, open circles; sample series 3, open triangles). Samples Ua-2741 and Ua-2741, which were stored ca. 60 months prior to subsampling and sieving, are not included in Fig. 2a. When these two samples are taken into account, the correlation coefficient is not significant ($r = 0.13$).

contaminating nutrients, which were present in the de-ionised water and in the air enclosed in the glass bottle.

Based on the identification of one fungal species in one single sample, we can, of course, not draw the conclusion that all other samples were contaminated by fungi, in general, or by the same fungal species. Micro-organisms, such as bacteria, which are present in the air, also could have contaminated the fossil leaves and the ambient water during sample preparation. Earlier investigations have pointed to the fact that bulk sediments from marine cores, which had been stored for many years at a temperature of 4°C, produce unreliable radiocarbon dates due to contamination by recent carbon from modern, terrestrial bacteria (Geyh *et al.*, 1974). Only recently, it was shown that several bulk sediment radiocarbon dates from cores, which had been stored for a longer time period and were only partly kept refrigerated, resulted in ages that were too young (Colman *et al.*, 1997).

To test whether long-term and wet storage of plant macrofossils may lead to anomalously young radiocarbon dates, we performed additional AMS ^{14}C dates on a series of plant macrofossil samples from a lake sediment sequence. Coring at the peat bog Bjärsjölagård (Fig. 1) was carried out in May 1994. Subsampling of the sandy clay gyttja, silty clay and fine sand, sieving and identification of the plant macrofossils were performed within one week in early June 1994. The macrofossils identified were divided into two subsamples; one half was dried on aluminium foil at 50°C immediately after sieving and identification, whereas the other half was stored in a glass bottle in slightly acid, de-ionised water at normal room temperature. Each subsample contained the same type of terrestrial macrofossils, i.e. leaves of *Salix polaris* and *Dryas octopetala*. The AMS ^{14}C dates obtained on the dried samples ranged from 12 750 ± 130 ^{14}C yr BP for the bottom sediment to 11 855 ± 220 ^{14}C yr BP for the top of the core (Table 3). Two of the samples, which had been stored for more than 2 years, were measured in February and March 1997. The resulting ages differed by 410 and 680 years from the samples measured earlier (Table 3). Even when the quoted errors are taken into account, there is a statistically significant difference between the two dating series. The probability that the dried sample B11 had a lower ^{14}C content than the long-stored, wet sample B11a is 98% and >99.9% for sample B5, compared with B5a. As we did not sample the leaf surfaces for fungi or bacteria, we cannot determine which type of microorganisms may have been responsible for the age difference, or when con-

Table 3 Comparison of AMS radiocarbon dates obtained on immediately dried samples (B5–B12) and on samples stored humid in room temperature (*) between June 1994 and March 1997 (B5a, B11a): L = leaves, S = seeds

Laboratory number Ua-	Site name	Macrofossils submitted	Dry weight (mg)	Carbon content (mg)	AMS ^{14}C date (yr BP)
4835	Bjärsjölagård B5	*Salix polaris* (L)	44.1	1.6	12 705 ± 130
4836	Bjärsjölagård B8	*Salix polaris* (L)	10.1	0.6	12 490 ± 235
4837	Bjärsjölagård B10	*Salix polaris* (L)	9.2	0.9	12 250 ± 170
4838	Bjärsjölagård B11	*Salix polaris* (L)	25.2	2.6	12 295 ± 120
4839	Bjärsjölagård B12	*Dryas octopetala* (L), *Betula* (S), *Salix* (L)	4.8	0.6	11 855 ± 220
*11693	Bjärsjölagård B5a	*Salix polaris* (L)	2.0	1.2	12 025 ± 135
*11602	Bjärsjölagård B11a	*Salix polaris* (L)	2.0	0.9	11 885 ± 150

tamination occurred. Nevertheless, the age difference between the samples shows that the wet-stored samples must have been considerably contaminated by recent material, which, in turn, increased the ^{14}C content of the fossil plant material. Accordingly, the risk of obtaining anomalously young radiocarbon ages seems considerably higher for long-stored, wet samples than for dried samples.

Small samples and low carbon content

As shown by the comparison between age deviation and carbon content for the three different dating series (Table 2, Fig. 2c), the carbon content of a sample seems to be another factor that can influence a radiocarbon age. Those samples in our dating series that exhibit low carbon content values generally show larger age deviations, whereas samples with values of > 1.4 mg carbon gave expected ages. Because the carbon content of a sample is related to sample dry weight, small samples are more likely to give ages that are too young. As shown in Table 1a–c and Fig. 2c, the samples submitted in the first dating series had the lowest sample weight and carbon content, with a mean of 0.66 mg carbon (compared with a mean of 1.03 mg, series 2 and 1.88 mg, series 3). The number of anomalously young ages is considerably lower in dating series 2 and 3 (Table 1b and c), where only 2 out of 9 samples and 3 out of 17 samples show large age deviations. Contamination of these samples by microbiological activity possibly can be excluded because the macrofossils were either measured shortly after subsampling or dried immediately after identification. Contamination by exogenic particles, which may fall on the sample during the identification of the plant material or during the handling of the sample, has to be regarded as another likely source of error. The possibility that such contamination has a larger influence on the radiocarbon age of a small sample compared with a larger sample is illustrated in Fig. 3a and b. If, for example, samples with an expected age of 12 300 or 10 250 ^{14}C yr BP and with a dry weight of 1 mg become contaminated by 0.1 mg recent carbon, the error will be in the order of 2700 or 2100 radiocarbon years, respectively (Fig. 3a). Although the age error clearly decreases with increasing sample weight, it still ranges between 500 and 1000 yr for samples with a measured age of 12 300 or 10 250 ^{14}C yr BP. If recent contamination of samples with the same ages as cited above and 1 mg dry weight is only 0.02 mg, then the errors will be less, but still significant, at around 500 or 700 yr respectively (Fig. 3b). Increased sample weight (e.g. 4 mg dry weight) also reduces the age error by 450 or 550 yr and results in radiocarbon ages of significantly lower precision.

Contamination by exogenic particles can hardly be avoided when samples are prepared and identified in non-sterile environments. However, to increase the precision of a radiocarbon measurement, it seems that, in addition to careful handling, only samples with an organic carbon content > 1.4 mg should be measured.

Conclusions

From a set of 51 AMS ^{14}C dates obtained on terrestrial plant macrofossils, 20 measurements resulted in ages that were several hundred to several thousand radiocarbon years younger than expected. An evaluation of this data set showed a significant statistical relationship between sample preparation, sample size and the anomalous radiocarbon measurements. Long-term and cool storage (> 6 months) of wet macrofossil samples prior to the radiocarbon measurement has significant consequences on the radiocarbon age obtained. The preparation and identification of small macrofossil samples is usually carried out in a non-sterile environment. Thus, fungal spores and microorganisms may be easily incorporated in the sample. CO_2 may then be assimilated by the fungi from the organic-rich medium and from contaminating nutrients in the ambient water during growth. This recent carbon, when added to the sample, may account for the large errors of several hundred to several thousand years encountered in our data series. Although this study relates to macrofossils extracted from clay sediments, it is equally probable that sediment cores that are stored for several years prior to the radiocarbon measurement can become affected by fungi or microorganisms during storage (Geyh *et al.*, 1974; Colman *et al.*, 1997). To avoid the contaminating effects of fungi and/or microorganisms, it is advisable to dry plant macrofossils before they are submitted to the radiocarbon laboratory at around 70°C (O. Constantinescu, pers. comm.)

Samples with low sample dry weight and low carbon content seem to be more susceptible to contamination than larger samples. If small macrofossil samples (~< 1.4 mg carbon content) cannot be prepared with extreme care and in a sterile environment to avoid any impurities, larger samples may have to be submitted if significant errors in the subsequent age determination are to be avoided.

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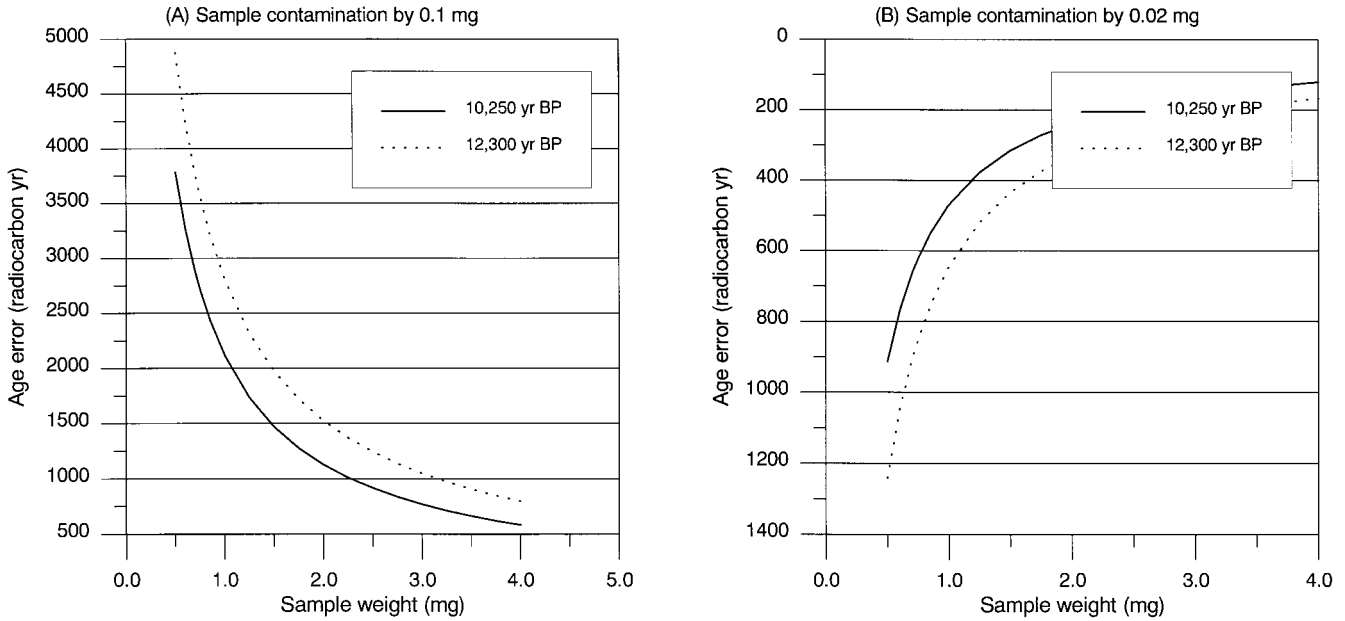

Figure 3 Resulting age error of samples with expected radiocarbon ages of 10 250 and 12 300 ^{14}C yr BP and with a varying dry sample weight when contaminated by recent material in the order of 0.1 mg (A) and 0.02 mg (B).

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