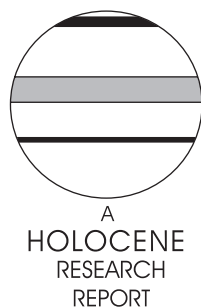


Effects of sample mass and macrofossil type on radiocarbon dating of arctic and boreal lake sediments

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Abstract: Dating lake sediments by accelerator mass spectrometry (AMS) ^{14}C analysis of terrestrial plant macrofossils overcomes one of the main problems associated with dating bulk sediment samples, i.e., the presence of old organic matter. Even so, many AMS dates from arctic and boreal sites appear to misrepresent the age of the sediment. To understand the nature of these apparent dating anomalies better, we conducted a series of ^{14}C dating experiments using samples from Alaskan and Siberian lake-sediment cores. First, to test whether our analytical procedures introduced a sample-mass bias, we obtained ^{14}C dates for different-sized pieces of single woody macrofossils. In these sample-mass experiments, statistically equivalent ages were found for samples as small as 0.05 mg C. Secondly, to assess whether macrofossil type influenced dating results, we conducted sample-type experiments in which ^{14}C dates were obtained for different macrofossil types sieved from the same depth in the sediment. We dated materials from multiple levels in sediment cores from Upper Capsule Lake (North Slope, northern Alaska) and Grizzly Lake (Copper River Basin, southern Alaska) and from single depths in other records from northern Alaska. In several of the experiments there were significant discrepancies between dates for different plant tissues, and in most cases wood and charcoal were older than other macrofossil types, usually by several hundred years. This pattern suggests that ^{14}C dates for woody macrofossils may misrepresent the age of the sediment by centuries, perhaps because of their longer terrestrial residence time and the potential in-built age of long-lived plants. This study identifies why some ^{14}C dates appear to be inconsistent with the overall age–depth trend of a lake-sediment record, and it may guide the selection of ^{14}C samples in future studies.

Key words: Radiocarbon dating, lake sediments, sample mass, plant macrofossils, AMS, chronology, palaeoecology, Siberia, Alaska, Holocene.

Introduction

High-quality chronology is vital in Quaternary sciences, particularly in efforts to understand rates of ecosystem

response to environmental change and feedbacks between the geosphere and biosphere (e.g., Sarnthein *et al.*, 2000). Lake-sediment records are examined widely for the variety of information they contain about the past, including rapid environmental and ecosystem variability (e.g., Allen *et al.*, 1999; Newnham and Lowe, 2000; Clark *et al.*, 2002). In general the chronology of these records is based on ^{14}C dating. Prior to the development of accelerator mass spectrometry

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(AMS), lake records were usually dated via ^{14}C analysis of bulk sediment, which in some circumstances may be complicated by the presence of old organic matter in the sediment matrix (e.g., Olsson, 1974). This problem may be acute at the northernmost latitudes, where organic matter decomposes slowly and may reside in permafrost for long periods of time before being eroded into lake basins (e.g., Nelson *et al.*, 1988; Zimov *et al.*, 1997). The development of AMS ^{14}C dating has made it possible to obtain ages for individual plant macrofossils, potentially avoiding problems associated with dating mixtures of contemporaneous and older organic matter in bulk sediment. When both techniques are applied to the same stratigraphic record, ages for AMS-dated plant macrofossils are often found to be hundreds or even thousands of years younger than ages for bulk sediment from the same core depth (e.g., Cwynar and Watts, 1989; Törnqvist *et al.*, 1992; Bigelow and Edwards, 2001). However, AMS dating is not free of problems, especially in arctic and boreal regions. Many AMS-dated sediment cores from Alaska and northeastern Siberia suffer from age reversals: dates that are anomalously old or young compared with the age–depth relationship for the majority of dates from a core (e.g., Oswald *et al.*, 1999; Brubaker *et al.*, 2001; Lozhkin *et al.*, 2001; Mann *et al.*, 2002).

AMS analyses of standardized laboratory samples have demonstrated that reasonable analytical precision can be obtained for samples <2 mg (e.g., Kirner *et al.*, 1996; Brown and Southon, 1997; Von Reden *et al.*, 1998; Hua *et al.*, 2001). The possibility of obtaining dates from small macrofossils is a major advantage of AMS when dating records from northern lakes, for which in many cases only small plant fragments are preserved in the sediment. However, when age reversals occur at such sites they often involve relatively small samples, and in most cases the problematic dates are younger than would be expected based on the age–depth trend (e.g., Oswald *et al.*, 1999; Andreev *et al.*, 2001). Given these observations, the first set of experiments in this study was used to test whether our analytical procedures result in an age bias at some sample-mass threshold. In these experiments, plant macrofossils found in Alaskan and Siberian sediment cores were split into different-sized pieces and then analysed for ^{14}C age.

Issues related to sample type arise because lake sediments from arctic and boreal regions often have few macrofossils. In most cases a single type of macrofossil is not present throughout a core, and therefore the chronology for the record is based on ages from a variety of different macrofossil types (e.g., seeds, wood, moss, leaves from different taxa). Aquatic plant macrofossils from hard-water lakes in carbonate terrain are usually not dated because of the possibility of old-carbon reservoir effects (e.g., Deevey *et al.*, 1954; MacDonald *et al.*, 1987; Hu *et al.*, 1996), whereas aquatic plants from soft-water lakes have been shown to be equilibrated with atmospheric CO_2 (Abbott and Stafford, 1996; Miller *et al.*, 1999), and thus their macrofossils may be an appropriate target material for ^{14}C dating. However, little is known of systematic biases in the ^{14}C ages of terrestrial plant macrofossils that are commonly analysed. Such biases might arise from differences in taphonomy, ‘in-built age’ (e.g., McFadgen, 1982), or susceptibility of the sample to contamination by young or old carbon. To test the importance of sample type, dates were obtained for several types of plant macrofossil from the same depth in sediment cores. These macrofossil-type experiments were conducted at multiple levels in cores from two sites in Alaska: Grizzly and Upper Capsule lakes. These sites were selected for analysis because (1) the sediment cores contained a variety of macrofossils, and (2) they are representative of boreal forest and arctic tundra ecosystems. The findings from these sites were

supplemented by opportunistic, single-level experiments from several other sediment cores from northern Alaska.

Study sites

Upper Capsule Lake (informal name; $68^{\circ}38'\text{N}$, $149^{\circ}25'\text{W}$) is located in the Arctic Foothills of northern Alaska (Figure 1). This area has a mean July temperature of 11°C , a mean January temperature of -22°C , and 325 mm mean annual precipitation (Zhang *et al.*, 1996). The Upper Capsule watershed is currently dominated by moist dwarf-shrub tussock-graminoid tundra (Walker *et al.*, 1994; Muller *et al.*, 1999), with moist, acidic, organic soils (Bockheim *et al.*, 1998). The site is underlain by continuous permafrost, with a shallow thaw layer (e.g., Walker *et al.*, 2001). The pollen record from Upper Capsule Lake suggests that relatively xeric, discontinuous vegetation occurred during the early Holocene, and that a transition to the modern ecosystem took place as effective moisture increased between $\sim 10\,000$ and 7500 cal. yr BP (Oswald *et al.*, 2003). The timing of an increase in *Alnus* pollen percentages at Upper Capsule and other nearby sites (e.g., Oswald *et al.*, 1999) provides an age–depth reference point to help evaluate the chronology of the sediment record (Figure 2).

Grizzly Lake ($62^{\circ}43'\text{N}$, $144^{\circ}12'\text{W}$) is located in the Copper River Basin of southern Alaska (Figure 1). This area has a mean July temperature of 13.4°C , a mean January temperature of -20.2°C , and 390 mm mean annual precipitation (Western Regional Climate Center, 2002). Permafrost is discontinuous, and moraines near the lake have well-drained soils. Forests near Grizzly Lake are dominated by *Picea glauca*, *Betula papyrifera* and *Populus tremuloides*. *Picea mariana* forms nearly pure stands in areas of wet soils, and *Alnus crispa* occurs on south-facing slopes. The pollen record from Grizzly Lake (W. Tinner, unpublished data) suggests that *Betula papyrifera*, *Betula nana* and *Populus* dominated the vegetation before 9300 cal. yr BP, and that *Picea glauca* forest replaced the open *Betula*–*Populus* stands between 9300 and 8500 cal. yr BP. *Alnus crispa* expanded ~ 8500 cal. yr BP, and *Picea mariana* became abundant after ~ 7000 cal. yr BP, apparently reducing soil erosion in the watershed and decreasing the sedimentation rate. Additional experiments were conducted using samples from several other sites in Alaska and Siberia (Figure 1). All of these sites occur in areas of arctic or boreal vegetation (Table 1).



Figure 1 Map of Bering Strait region with locations of primary study sites (GY, Grizzly, UC, Upper Capsule) and secondary study sites (VP, Vadopadnoye, MK, Malyii Kretchet, AH, Ahaliorak, RG, Red Green, OK, Okpilak)

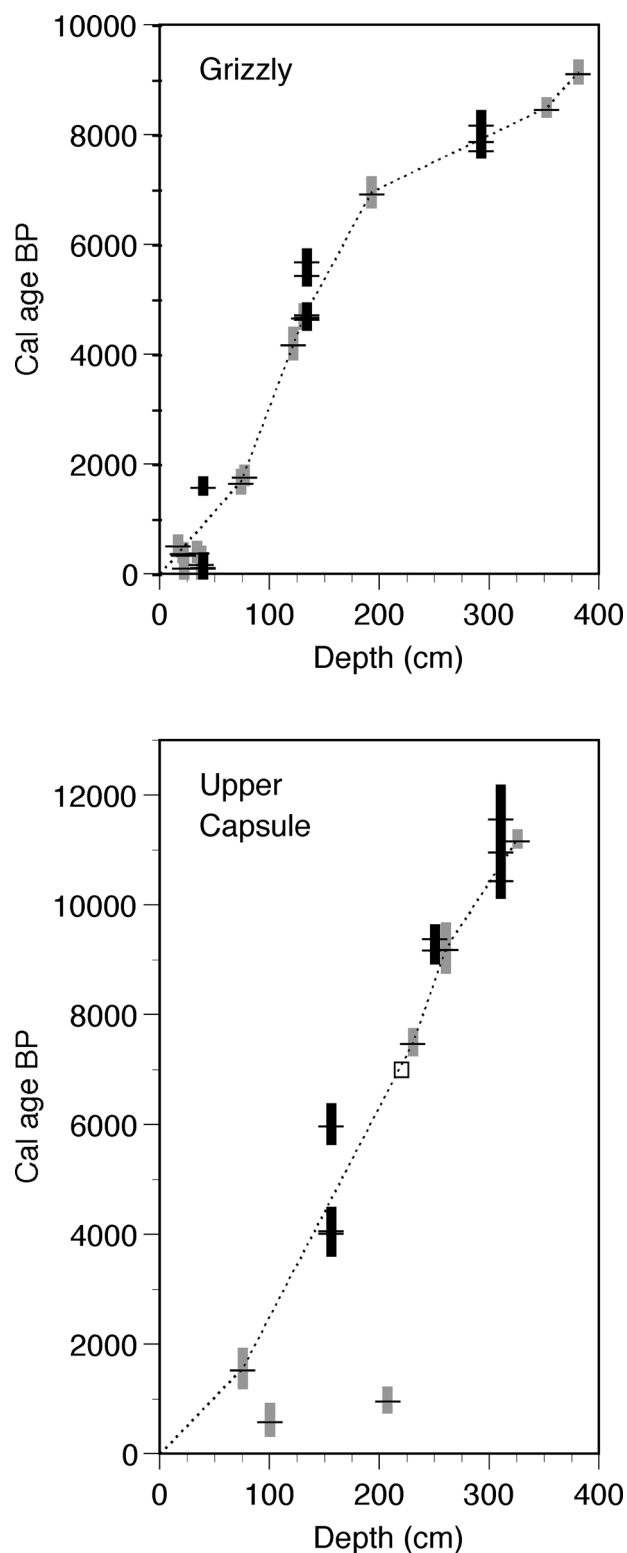


Figure 2 Age–depth plots for Grizzly and Upper Capsule. Bars indicate calibrated ^{14}C range (2σ), with horizontal lines at midpoint of that range. Black bars are samples from the mass and type experiments (Tables 2–4); grey bars are samples from other studies (Oswald *et al.*, 2003; W. Tinner, unpublished data). Open box indicates the depth of the increase in *Alnus* pollen percentages in the Upper Capsule record, which dates to ~ 7000 cal. yr BP in records from nearby sites (Oswald *et al.*, 1999, 2003). The samples at UC 100–101 and UC 207–208 cm (not part of this study) presumably deviate from the age–depth relationship because of contamination by relatively young carbon. The dotted lines are drawn between selected dates to estimate the time elapsed per sample (Table 6) and to illustrate the overall age–depth relationship for the records

Methods

Sampling, pre-treatment and ^{14}C age determination

Sediment cores were collected from the study lakes using a square-rod piston-sampler, 4.5 cm in diameter (Wright *et al.*, 1984). At selected core depths, 1–2 cm sections of the core were washed through a 500 μm mesh screen with distilled water. The >500 μm fraction was examined with a dissecting microscope, and plant macrofossils were removed with clean tweezers. We selected levels with large pieces of wood and abundant macrofossils for the sample-mass and macrofossil-type experiments, respectively. For the sample-mass experiments, woody macrofossils were split into two to six different-sized pieces with a sharp, clean blade. For the sample-type experiments, two to seven different plant macrofossils were chosen from a single sediment sample 1–2 cm thick. To remove exterior contaminants, samples were heated at 70°C for 15 minutes in 1 M HCl, followed by 45 minutes in 1 M KOH and finally 15 minutes in 1 M HCl. Following this pretreatment, samples were stored in 0.1 M HCl in glass vials with teflon-lined screw caps. ^{14}C analyses were conducted at the Center for Accelerator Mass Spectrometry (CAMS) at Lawrence Livermore National Laboratory. ^{14}C ages were determined assuming $\delta^{13}\text{C}$ values of -25‰ (Stuiver and Polach, 1977), and the dates were converted to calibrated (cal.) ages using OxCal 3.9 (Bronk Ramsey, 1995, 2001).

Data analysis

We used a subroutine in CALIB 4.3 (Stuiver and Reimer, 1993) to test for statistically significant ($p < 0.05$) age differences between subsamples from a given depth. The subroutine determines the pooled average (weighted mean) of the ^{14}C dates, calculates the test statistic T from the weighted sum of the differences between each sample age and the pooled average, and compares T with a chi-square distribution for $n - 1$ samples. If T is less than the chi-square value, the dates do not differ statistically (Ward and Wilson, 1978). However, some samples have such large age uncertainties that this test may indicate that they are statistically equivalent even if they differ by several hundred years. The level of acceptable chronological uncertainty depends on the objective of the research; in this case, we limit our analyses to dates with uncertainties less than 250 ^{14}C years. We used a subroutine in OxCal 3.9 to determine the age difference (2σ cal. yr range) between those samples with statistically different ages.

Results

Sample-mass experiments

^{14}C analyses of different-sized pieces of the same woody macrofossil returned statistically equivalent ($p < 0.05$) dates for samples from UC 310–311, MK 121–122, AH 32–33, and VP 48–50 (Table 2). Radiocarbon ages were significantly different for dates on the subsamples of two macrofossils from MK 85–86 (Figure 3). For sample MK 85–86 (Figure 3a), the two ages differed by 1560–3810 (2σ) cal. (1330 ± 510 ^{14}C) years. For sample MK 85–86 (Figure 3b) the dates for the two largest samples were identical ($\sim 3600 \pm 2\sigma$ cal. yr, 3320 ^{14}C years), but the small (0.02 mg C) and very small (0.01 mg C) samples were 360–3650 cal. (910 ± 760 ^{14}C) years younger and 1290–5850 cal. (1870 ± 1160 ^{14}C) years older than this date, respectively. The dates for VP 26–28 (Figure 3) were statistically equivalent, but the two smallest subsamples (mass < 0.04 mg C) had unacceptable uncertainties (280 and 550 ^{14}C years).

Table 1 Study sites

Site	Code	Location	Region	Vegetation
Grizzly	GY	62°43'N, 144°12'W	Copper River, Alaska	<i>Picea</i> boreal forest
Upper Capsule	UC	68°38'N, 149°25'W	North Slope, Alaska	Moist dwarf-shrub tussock graminoid tundra
Red Green	RG	68°39'N, 149°41'W	North Slope, Alaska	Moist graminoid prostrate-shrub tundra
Okpilak	OK	69°25'N, 144°02'W	North Slope, Alaska	Moist graminoid prostrate-shrub tundra
Ahaliorak	AH	68°55'N, 151°20'W	North Slope, Alaska	Moist dwarf-shrub tussock graminoid tundra
Vadopadnoye	VP	59°24'N, 150°39'E	Priokhot'ye, Russia	<i>Larix dahurica</i> forest
Malyii Kretchet	MK	64°28'N, 175°19'E	Anadyr Basin, Chukotka, Russia	<i>Pinus pumila</i> - <i>Alnus</i> shrub tundra

Sample-type experiments

Based on the findings of the sample-mass experiments, samples <0.05 mg C were excluded from the macrofossil-type analyses (Tables 3–5). For the remaining experiments, ^{14}C dates obtained for all different macrofossil types were statistically equivalent in only one case (RG 202–203), whereas at least one date was significantly different in the other eight cases (Figure 4). For UC 155–157, graminoid leaf fragments dated at least 1950–3310 cal. (1530 ± 310 ^{14}C) years older than the other materials, and for UC 250–251, graminoid and moss dates differed by 10–1310 cal. (270 ± 210 ^{14}C) years. For UC 310–311, six of the seven dates (including the three dates on different-sized pieces of the same woody macrofossil) did not differ statistically. The date for moss fragments, however, was younger by at least 140–2620 cal. (440 ± 310 ^{14}C) years. For GY 39–40, five macrofossils had statistically equivalent ages, but the date for charcoal was at least 1410–1900 cal. (1500 ± 100 ^{14}C) years older. Similarly, four of the ages from GY 133–135 were not statistically different, but wood and charcoal macrofossils were older by at least 760–1490 and 930–1590 cal. (575 ± 120 and 730 ± 110 ^{14}C) years, respectively. For GY 292–293, the dates from three different pieces of wood differed by as much as 490–1070 cal. (520 ± 130 ^{14}C) years. In the OK 478–479 experiment, the wood fragment was

550–1300 cal. (430 ± 130 ^{14}C) years older than the moss macrofossil, and for AH 31–32, the seed had the youngest date, the moss fragments were 120–1930 cal. (400 ± 310 ^{14}C) years older than the seed, and the wood macrofossils were 150–1480 and 3190–4710 cal. (310 ± 190 and 2310 ± 200 ^{14}C) years older than the moss.

Discussion

Sample-mass experiments

The finding that ^{14}C dates for >0.05 mg C pieces of the same macrofossil did not differ significantly suggests that 0.05 mg C is the sample-mass threshold for reliable age determination, given the procedures used in this study. These results are consistent with a study by Brown and Southon (1997) in which they observed larger-than-expected scatter in measured values for subsamples smaller than ~ 0.03 mg C from a 6130 ± 20 ^{14}C yr BP sample (Stuiver and Becker, 1993; Brown, 1994). Two analytical factors likely contribute to our findings. First, in our procedures the completeness of the graphitization reaction during sample preparation is uncertain when the samples are very small. Studies of the graphitization process for larger samples show that fractionation occurs during the catalytic reactions of the gaseous constituents (primarily CO_2 and CO),

Table 2 Sample-mass experiments

Sample	Material	Mass (mg C)	CAMS No.	^{14}C age BP	2σ cal. ^{14}C age range
AH 32–33	Wood	0.05	66731	9350 ± 180	11168–10187
	Wood	0.35	66732	9280 ± 50	10636–10245
	Wood	1.03	66733	9320 ± 40	10670–10400
MK 85–86 (a)	Wood	0.03	48500	3170 ± 240	3932–2778
	Wood	0.11	48499	4500 ± 90	5451–4860
MK 85–86 (b)	Wood	0.01	48504	5190 ± 550	7248–4570
	Wood	0.02	48503	2410 ± 330	3269–1692
	Wood	0.04	48502	3320 ± 190	4080–3137
	Wood	0.07	48501	3320 ± 120	3838–3328
MK 121–122	Wood	0.12	66729	8370 ± 80	9530–9132
	Wood	0.50	66730	8230 ± 40	9400–9031
	Wood	0.96	66750	8300 ± 60	9473–9090
VP 26–28	Wood	0.01	49702	2330 ± 550	3646–1172
	Wood	0.03	49701	2830 ± 280	3637–2330
	Wood	0.06	49698	2990 ± 130	3469–2787
	Wood	0.07	49697	2990 ± 120	3466–2848
	Wood	0.07	49699	3040 ± 120	3474–2874
	Wood	0.08	49696	3050 ± 100	3469–2952
	Wood	0.10	49700	3000 ± 90	3386–2922
VP 48–50	Wood	0.06	50792	4170 ± 140	5045–4299
	Wood	0.08	50793	4180 ± 110	4971–4418
	Wood	0.17	50794	4290 ± 60	5028–4654
	Wood	0.22	50795	4270 ± 60	4968–4648
UC 310–311	Wood	0.07	66734	9990 ± 150	12298–11162
	Wood	0.13	66735	9830 ± 90	11549–11115
	Wood	0.29	66736	10030 ± 60	12090–11255

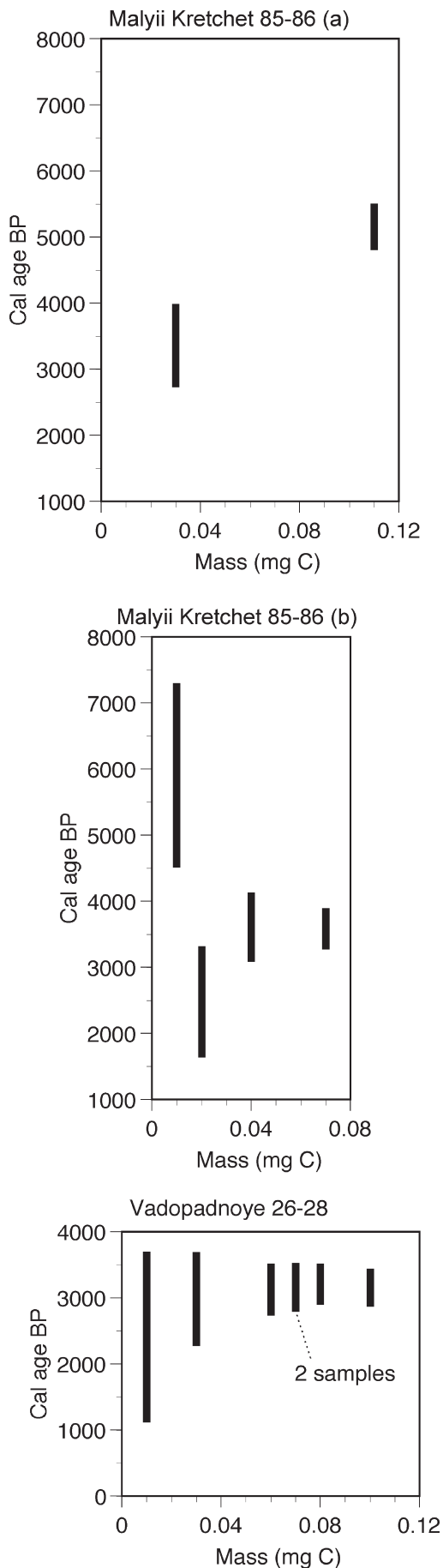


Figure 3 Plots of calibrated ^{14}C age versus sample mass (mg C) for the Malyii Kretchet 85–86 (a), Malyii Kretchet 85–86 (b), and Vadopadnoye 26–28 experiments. Bars indicate calibrated ^{14}C range (2σ)

with the isotopic content of the produced graphite reaching that of the initial CO_2 sample as the reaction goes to completion (e.g., Aerts-Bijma *et al.*, 1997). The fractionation observed between the initial CO_2 $\delta^{13}\text{C}$ values and those of the last CO_2 fraction remaining just before the reaction goes to completion ($\sim 30\text{‰}$) indicates that ^{14}C fractionations of up to $\sim -60\text{‰}$ may occur because of incomplete graphitization. Thus, incomplete graphitization of small samples could result in measured ^{14}C ages being up to ~ 500 years older than the actual age of the sample. Secondly, with decreasing sample mass, any background contaminant carbon introduced during sample processing represents a greater fraction of the total sample. Hence, as sample mass decreases, variations in background contamination have an increasingly larger impact on background corrections of the ^{14}C measurements. Recent graphitization tests at CAMS (T.A. Brown, unpublished data) have shown that the ^{14}C content of background contaminants is consistent with current atmospheric CO_2 values, such that inadvertent introduction of higher-than-normal amounts of such contaminants should result in measured ^{14}C ages being somewhat younger than the actual age of the sample. The increased significance of background contaminant corrections with decreasing sample mass and the larger-than-expected scatter of results obtained for very small samples suggest that larger-than-expected variations are occurring in the background contaminant when sample mass is below some threshold (Brown and Southon, 1997). In addition to these two factors, unusual isotopic fractionation effects in the AMS ion source and introduction of unusual contamination during graphite handling and/or target preparation also may contribute to the unexpectedly large scatter of the very small samples. As shown by Brown and Southon (1997) and by subsequent ongoing tests at CAMS (T.A. Brown, unpublished data), measurements of modern standard materials show similarly larger-than-expected scatter of ^{14}C ages for samples below a sample threshold of several tens of microgrammes of carbon. Thus, while the exact cause of the apparent small sample threshold is not known at present, the 0.05 mg C threshold found in this study is consistent with results of Brown and Southon (1997) and with more recent tests conducted within the CAMS graphitization laboratories.

Sample-type experiments

The results of the sample-type experiments are of concern to current dating procedures for northern lake sediments. In many cases, different types of macrofossil from the same core depth differed by more than 500 ^{14}C years. A number of factors may contribute to these age discrepancies, including (1) carbon source or fractionation differences between plants, (2) the slow sedimentation rates of these lakes, (3) taphonomic or 'in-built age' differences among macrofossil types, and (4) differences in susceptibility to contamination. We consider each factor in more detail below.

Carbon source or fractionation

If plants obtain carbon directly from lake water in carbonate terrain, macrofossil ^{14}C ages might appear too old as the result of a reservoir effect (e.g., Deevey *et al.*, 1954). For example, MacDonald *et al.* (1987) found that ^{14}C ages of aquatic moss macrofossils from western Canada were >1500 years older than their terrestrial counterparts. Although we did not differentiate between terrestrial and aquatic mosses, moss macrofossils never had the greatest ages in the type experiments, suggesting carbon equilibration with the atmosphere, as would be the case for other plant types. In addition to possible carbon-source effects, age discrepancies might also result from

Table 3 Upper Capsule Lake macrofossil-type experiments

Sample	Material	Mass (mg C)	CAMS No.	¹⁴ C age BP	2σ cal. ¹⁴ C age range
UC 155–157	Leaf fragments	0.05	66741	3670 ± 120	4408–3688
	Graminoid leaf fragments	0.05	66742	5250 ± 130	6296–5721
	Semi-woody fragments	0.08	66743	3720 ± 80	4348–3836
UC 250–251	Graminoid leaf fragments	0.11	66745	8470 ± 90	9554–9278
	Moss	1.09	66744	8200 ± 50	9399–9015
UC 310–311	Moss	0.08	66747	9270 ± 120	10745–10211
	Graminoid leaf fragments	0.11	66749	9710 ± 100	11256–10735
	Seed	0.07	66748	9920 ± 150	12099–11088
	Wood	1.04	66746	10010 ± 50	11931–11255

Table 4 Grizzly Lake macrofossil-type experiments

Sample	Material	Mass (mg C)	CAMS No.	¹⁴ C age BP	2σ cal. ¹⁴ C age range
GY 39–40	Moss	0.10	82319	100 ± 70	294–0
	Conifer periderm	0.17	82318	95 ± 45	278–0
	Deciduous periderm	0.30	82317	175 ± 40	300–0
	Wood	0.32	82314	150 ± 40	291–0
	<i>Picea</i> needle	0.37	82313	180 ± 40	302–0
	Charcoal	0.93	82316	1685 ± 30	1692–1524
GY 133–135	<i>Picea</i> conescale	0.37	82322	4125 ± 35	4825–4526
	Moss	0.42	82326	4160 ± 40	4832–4532
	Conifer periderm	0.89	82325	4190 ± 40	4840–4573
	<i>Picea</i> needle	0.94	82320	4225 ± 40	4855–4644
	Wood	0.95	82321	4800 ± 45	5607–5334
	Charcoal	0.97	82323	4955 ± 40	5842–5601
	Deciduous periderm	1.01	82324	4180 ± 40	4836–4571
GY 292–293	Wood	0.94	59340	6910 ± 40	7819–7665
	Wood	0.95	59339	7100 ± 50	8011–7792
	Charcoal	0.99	59341	7430 ± 50	8362–8059

Table 5 Additional macrofossil-type experiments

Sample	Material	Mass (mg C)	CAMS No.	¹⁴ C age BP	2σ cal. ¹⁴ C age range
AH 31–32	Seed	0.07	76814	8680 ± 130	10157–9471
	Moss	0.14	76813	9080 ± 80	10471–9976
	Wood	0.46	76812	9390 ± 50	10742–10430
	Wood	1.00	44522	11390 ± 60	13791–13051
OK 478–479	Seed	0.02*	76817	6750 ± 380	8347–6848
	Moss	0.26	76816	7660 ± 50	8541–8373
	Wood	1.02	76815	8090 ± 40	9236–8988
RG 202–203	Moss	0.04*	66738	7140 ± 220	8387–7573
	Wood	0.07	66739	7190 ± 120	8198–7755
	Leaf	0.08	66737	7060 ± 100	8108–7674

*Samples <0.05 mg C not included in analyses.

differences in ¹⁴C fractionation among plant species (Aitken, 1993). Differences in ¹⁴C depletion can be accounted for by adjusting ¹⁴C values relative to measured ¹³C values (Craig, 1953) or by calculations assuming an expected δ¹³C value. In this study, ¹⁴C dates were calculated assuming δ¹³C values of –25‰ (Stuiver and Polach, 1977), which is almost certainly within 5‰ of the actual δ¹³C value of a terrestrial macrofossil, and within ~9‰ of that of an aquatic macrofossil (Gupta and Polach, 1985; Aitken, 1993). Because the AMS measurements were of the ¹⁴C/¹³C atom ratio of the samples, a δ¹³C departure from –25‰ of 1‰ would correspond to a ¹⁴C age shift of roughly 8 years, and hence the fractionation correction for these dates would be at most 40 years for terrestrial samples and ~80 years for aquatic samples. Because this adjustment is

smaller than the observed age disparities (>650 ± 2σ cal. yr), the fractionation effect is not likely to be the main cause of the ¹⁴C differences among macrofossils from the same core depth.

Timespan of the sample

The slow sedimentation rates of the study lakes could potentially result in the accumulation of macrofossils of widely different ages in a 1–2 cm thick section of the core. To test the importance of this effect, we compared the age discrepancies for the Upper Capsule and Grizzly sample-type experiments with estimates of the timespan of each sample (Table 6). The time elapsed per sample (always <80 cal. yr) was substantially less than the age difference between macrofossils (always >650 ± 2σ cal. yr). Thus slow sedimentation

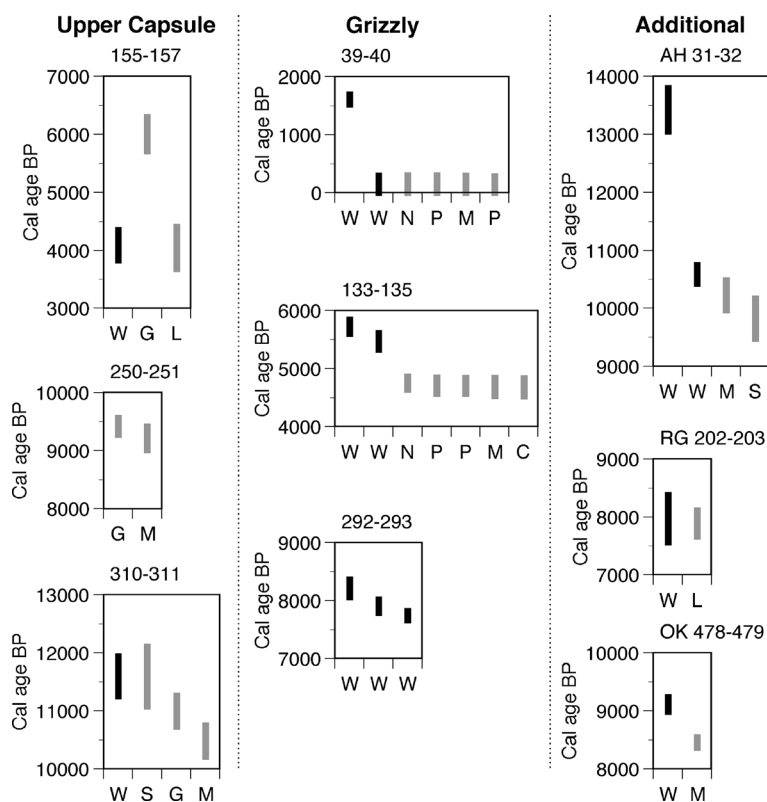


Figure 4 Plots of calibrated ^{14}C ages for the macrofossil-type experiments (Tables 3–5). Bars indicate calibrated ^{14}C range (2σ). Black bars are woody macrofossils (wood and charcoal); grey bars are other types. Codes for macrofossil types: W, wood or charcoal; C, conescale; G, graminoid; L, leaf; M, moss; N, needle; P, periderm; S, seed

does not appear to account for the age discrepancies. On the other hand, the samples from MK 85–86 illustrate the difficulty in dating sediments that have accumulated irregularly. The overall age–depth relationship for the Malyii Kretchet record (Figure 5) is complicated by an interval of peat (60–87 cm), within which macrofossils of substantially different age occur at the same depth. Assuming that the four MK 85–86 samples <0.05 mg C may have erroneous ages (Figure 3), whereas the ages of the two samples >0.05 mg C are likely reliable, it would appear that this 1-cm thick interval contains macrofossils differing by at least 1550–2950 cal. (1180 ± 300 ^{14}C) years.

Taphonomy or in-built age

Age discrepancies might also result from differences in taphonomy or 'in-built age' among macrofossil types. The most striking result of the sample-type experiments is that wood and charcoal are generally older than other macrofossils from the same sample depth. The amount of the offset was inconsistent, with age differences ranging from tens to thousands of years, but this trend occurred in all but one of

Table 6 Comparison of statistically significant age differences with the time elapsed per sample for the Upper Capsule and Grizzly macrofossil-type experiments

Sample	Age difference (2σ cal. yr range)	Years per sample*
UC 155–157	1950–3310	76.7
UC 250–251	10–1310	57.0
UC 310–311	140–2620	30.5
GY 39–40	1410–1900	22.8
GY 133–135	760–1490, 930–1590	73.0
GY 292–293	490–1070	9.7

*Based on age–depth relationships in Figure 2.

the experiments (UC 155–157) in which wood or charcoal was dated. Because woody macrofossils are relatively large and decay-resistant (Hobbie, 1996), they are likely to remain on the landscape longer than smaller, more readily decomposed plant tissues. In addition, dates on wood and charcoal might be older because of an 'in-built age' effect (e.g., McFadgen, 1982; Gavin, 2001). Because woody plants maintain old tissues in their structure, wood in the inner rings of a branch or stem could be substantially older than the outermost layers. Thus, even if the remains of a woody plant were washed into a lake as soon as the plant died, a ^{14}C date on a piece of wood might appear significantly older than the rest of the sediment if the dated tissues came from the inner rings. This type of error should be less important in tundra than in boreal forest ecosystems, as woody tundra plants have been observed to reach 30–55 years in age (Warren Wilson, 1964; Shaver, 1986), whereas boreal forest trees in interior Alaska often live beyond 250 years (e.g., Van Cleve *et al.*, 1983).

Contamination

Another explanation for the tendency of woody samples to be older than non-woody materials is that some macrofossil types may be more susceptible to contamination than others. For example, woody tissues may have some propensity for contamination by old carbon, perhaps because of their rough surface texture. On the other hand, moss and leaf fragments could have younger ^{14}C dates than wood because they are more easily contaminated by modern carbon. These non-woody macrofossils are generally flat or filamentous, and therefore their surface area to volume ratio is larger than that of wood and charcoal pieces. Contamination by modern carbon is much more likely to have an important age effect than that by old carbon, as the substantially higher ^{14}C activity of young carbon creates a larger magnitude dating error than old carbon for the same amount of contaminant (Olsson, 1974). However,

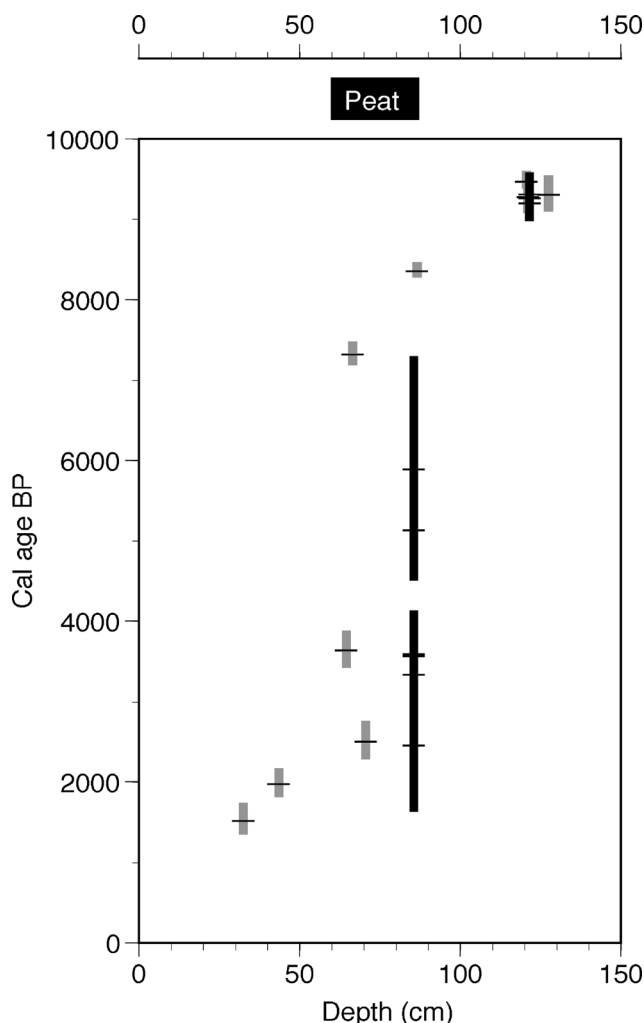


Figure 5 Age–depth plot for Malyii Kretchet. Bars indicate calibrated ^{14}C range (2σ), with horizontal lines at mid-point of that range. Black bars are samples from the mass experiment (Table 2); grey bars are samples from another study (P.M. Anderson, unpublished data). The interval of peat between 60 and 87 cm presumably was deposited during a period of lowered lake level

the sample pretreatment routine should remove impurities from the surface of the macrofossils, such that contamination by young or old carbon is avoided.

Environmental change

A potentially important factor not addressed in depth by this study is the effect of environmental and ecological changes on the age of macrofossils relative to the lake-sediment matrix. As sediment accumulates in a lake basin over time, environmental changes might affect the type or taphonomy of macrofossils that reach the lake and thus become available for dating. For example, the Grizzly pollen record indicates an early Holocene change from *Betula-Populus* stands to *Picea glauca* forest, and a subsequent transition to *Picea mariana* (W. Tinner, unpublished data). The first change might affect the ^{14}C chronology of the sediments by altering the type of woody material entering the lake, whereas the second change reduced the rate of soil erosion and thus may have slowed the delivery of macrofossils to the coring site. Similarly, the Upper Capsule pollen record indicates ecological changes between the early and middle Holocene, including higher overall vegetation cover, increased woody shrub prevalence, and slower decomposition (Oswald *et al.*, 2003). This transition also has

implications for ^{14}C dating because the change in plant-community composition would have increased the availability of woody macrofossils, whereas the change in decomposition would have increased the terrestrial residence time of plant macrofossils. It would be necessary to analyse a greater number of macrofossils and sampling depths to begin to assess the effect of these types of environmental and ecological change.

Conclusions

The results of this study provide insights for dating lake sediments from the northernmost latitudes, but they are also relevant to the general use of ^{14}C analysis in Quaternary sciences. The sample-mass experiments demonstrate that these laboratory and analytical procedures can be used to obtain statistically equivalent ^{14}C dates for lake-sediment macrofossils as small as 0.05 mg C. The ability to date such small materials is crucial for understanding the chronology of macrofossil-poor sediments, including those from many northern lakes, as well as other scenarios where only very small amounts of organic matter are available for dating (e.g., large lakes, glacial-age sediments). However, the experiments involving ^{14}C analysis of different plant tissues from the same sediment depth suggest that some macrofossil types may provide erroneous deposition ages. Wood and charcoal were generally older than other types of plant remains, and in several cases the dates for these materials exceeded other dates by several hundred years. We attribute this pattern to the slower decomposition and longer terrestrial residence time of woody macrofossils in arctic and boreal environments, and perhaps to the ‘in-built age’ effect that may occur in ecosystems with long-lived plants.

Because woody macrofossils are commonly selected for ^{14}C dating (e.g., Oswald *et al.*, 1999; Anderson and Lozhkin, 2001; Lozhkin *et al.*, 2001), the conclusion that they may not provide accurate dates for lake-sediment palaeoenvironmental records is important. If the research objective is to reconstruct past changes at the scale of centuries or decades, as is increasingly the case (e.g., Hu *et al.*, 2001), an error of several hundred years is unacceptable. This type of age bias is not only a potential problem in the northernmost latitudes, but also in mid-latitude regions where intervals of the past were characterized by cold conditions and permafrost. Fortunately, we can use the results of this study and other ^{14}C dating experiments (e.g., Turney *et al.*, 2000; Nilsson *et al.*, 2001) to guide the selection of samples so that the risk of problematic dates is minimized.

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