

## AMS $^{14}\text{C}$ DATING OF POLLEN CONCENTRATE FROM LATE PLEISTOCENE ICE WEDGES FROM THE BISON AND SEYAHA SITES IN SIBERIA

Alla Vasil'chuk<sup>1</sup> • Jong-Chan Kim<sup>2</sup> • Yurij Vasil'chuk<sup>1</sup>

**ABSTRACT.** Accelerator mass spectrometry (AMS) radiocarbon dates of pollen concentrate were obtained from multistage syngenetic ice wedges of cross-sections from the Late Pleistocene Bison site, located along the Lower Kolyma River (68°34'N, 158°34'E), from ~43,600 to ~26,200 BP, and 3 AMS  $^{14}\text{C}$  dates of pollen concentrate in ice wedges from the Seyaha site cross-section, located on the east coast of the Yamal Peninsula (70°10'N, 72°34'E), from ~22,400 to ~25,200 BP. Pollen concentrate samples were prepared using a special pretreatment procedure. Pollen and spores from ice-wedge ice signalize a regional pollen rain. Therefore,  $^{14}\text{C}$ -dated extracts of pollen and spores from ice-wedge ice enable an adequate reconstruction and chronology of landscape dynamics on a regional scale. The pollen and spores were well preserved despite numerous redepositions in the penecontemporaneous structure in which they were found. Thus, a comparison with dates on other fractions from the same sample is necessary. The youngest date is the most reliable among the intersample AMS  $^{14}\text{C}$  dates from the ice and permafrost sediments.

### INTRODUCTION

Ice-rich permafrost sediments are widespread in northern Siberia. The sediments are one of the best climatic archives because their invariable frozen state keeps organic materials well preserved for thousands of years. Despite this, permafrost sediment often contains both freshly deposited organic material and redeposited "old" material. Therefore, the purpose of this paper is to compare  $^{14}\text{C}$  dates on pollen concentrate, taken from Late Pleistocene ice wedges from northwest Siberia and northern Yakutia, with AMS  $^{14}\text{C}$  dates of microorganic inclusions and alkaline extract obtained from the same samples, and also to establish the reliability of AMS  $^{14}\text{C}$  dates of different fractions.

### Location and Description of the Cross-Sections

The Bison site is located along the eastern bank of the Kolyma River (68°34'N, 158°34'E) in the mouth of Lakeevskaya Stream, near the northern limit for larch forest. The site Seyaha is located on tundra on the eastern coast of the Yamal Peninsula (70°10'N, 72°34'E) in the Seyaha (Zelyonaya) River mouth, and extends more than 4 km along the coastline of Ob Bay (Figure 1).



Figure 1 Location of the Bison and Seyaha sites in Siberia

<sup>1</sup>Departments of Geology and Geography, Lomonosov's Moscow State University, Leninskie Hills, Moscow, Russia, 119992. Email: vasilch@geol.msu.ru.

<sup>2</sup>Department of Physics, Seoul National University, Kwanak-ku, Seoul, 151-742, South Korea. Email: jckim@phy.snu.ac.kr.

The Bison cross-section is more than 15 m high and is located in a larch forest. The sandy loam sediments contain lenses of peat and are ice-rich and dissected by ice wedges up to 2 to 2.5 m wide and 7 to 9 m high.

The Seyaha cross-section is more than 24 m high. It is located in moss-dwarf, shrub-sedge mezc tundra near the boundary between Arctic and sub-Arctic tundra. The sandy loam sediments have layers of peat and are ice-rich and dissected by 3-stage ice wedges. Lower-stage ice wedges are up to 3 m wide, while middle- and upper-stage ice wedges are up to 1.5 m wide. Laminated sediments are covered by sand layers containing sub-saline foraminiferans.

### **Nature of Ice Wedges**

Syngenetic ice wedges grow as the upper permafrost surface rises in response to the addition of material on the ground surface. The added material may be alluvium or lacustrine-alluvium, as in river and lake floodplain deposits (e.g. Bison); tidal marine deposits, as in tidal marsh (e.g. Seyaha); peat, as in a tundra polygon (organic-rich layers in Seyaha and Bison); solifluction deposits, as at the bottom of a slope; and so on. The age of a new ice veinlet in a syngenetic wedge therefore corresponds closely with that of the new material in which it grows, hence the term "syngenetic." In the Arctic, this polygonal structure is widespread as a type of permafrost. Usually the syngenetic ice wedges consist of atmospheric water frozen within frost cracks resulting from the spring snow melt (<80% by volume of ice wedge). Minor sources include hoarfrost and the melt of active layer ice (<5–10%). The frost cracks penetrate up to 2–3 m in the permafrost, and are <1 cm wide at the top and become narrower, closing to 1–3 mm at 1 m deep (Vasil'chuk 1992). The melted water freezes in the upper part of the crack due to the colder temperature within the crack, and blocks the further penetration of water. In late winter or early spring, the water in the crack does not exceed a few grams of water per 1–3 m of crack depth. The upper part of the crack (approximately 0.5–0.8 m) is located in the soil, while the lower part (approximately 1.5–2 m) is in the ice wedge. The temperature of the cracks' walls was no higher than –20 °C (winter temperatures were simulated through isotope analyses of ice-wedge ice: see Vasil'chuk 1992; Vasil'chuk et al. 2000). As some of the flow water erodes, the frozen soil and its organic material (a negligible admixture of organic material) enters the ice-wedge body from the soil walls. Thus, the syngenetic ice wedges grow higher, often vertically nested in a chevron pattern (Mackay 1990) due to both the horizontal and vertical growth rates.

Growth of syngenetic ice-wedge occurs cyclically. The long sub-aerial phase leads to a short sub-aqueous phase (Vasil'chuk and Vasil'chuk 1996, 1998b). Thus, the pollen and soil particles enter the ice wedges in different ways. In the sub-aqueous phase (the top part of the Seyaha ice-wedge complex), most of the pollen and ground particles have been reworked and redeposited by water from older deposits; however, during the sub-aerial phase, when peaty layers are formed, most of the pollen and ground particles are contemporaneous with the ice growth.

### **Ways of Organic Material Penetration**

It is very difficult to determine the admixture of older material; thus, for adequate <sup>14</sup>C dating it is important to select organic material from closed systems like syngenetic ice wedges. Pollen, spores, and other organic material in syngenetic ice wedges are isolated from environmental changes—in particular, from microbial activity and the rootlets of modern plants—since the time of their inclusion into the ice wedge. The majority of organic material in ice-wedge ice originates from the pollution that accumulates in the snow during spring melting, which then solidifies as the crack walls freeze under much lower temperatures. In the upper part of the crack, the water freezes, forming an

ice neck that blocks the further penetration of water and organic material (Vasil'chuk et al. 2000). The organic remains therefore reflect the composition of organic material in the snowmelt water at the time of ice-wedge formation. The obtained <sup>14</sup>C date shows the age of the organic material that accumulated at the site during the winter. We suppose that among the obtained AMS <sup>14</sup>C dates of the same sample, the youngest is the most reliable. The sample with the youngest date has only minor admixtures of old organic material because the ice and host sediments are preserved in a frozen state and the system is closed from the incorporation of young <sup>14</sup>C contamination in syngenetic permafrost. Thus, when dating permafrost, contamination by material older than the sample is more important than contamination by material younger than the sample.

Pollen grains permeate the frost cracks together with snowmelt water during late winter (May to June). The regional "pollen rain" originates mainly from the high pollen productivity of flowering trees and herbs in neighboring southern areas. Up until July, the frost cracks are open and snowmelt water permeates the frost cracks, while the temperature is above freezing at the surface.

Most of the pollen and spores enter the ice wedges from the snowmelt; fewer spores enter from the dry walls of the frost crack and the ground particles (Vasil'chuk and Vasil'chuk 1998a; Vasil'chuk et al. 2000, 2003). Microorganic inclusions could also enter ice wedges with snowmelt during the winter when the cracks are open. Thus, the collection method for the separation of pollen and organic microinclusions is based on different-sized particles and different reactions to acids. Organic microinclusions are separated from pollen using 100–200- $\mu$ m sieves in laboratory pretreatment, because pollen grains are smaller than microinclusions. Thus, almost all organic microinclusions are collected in 100- $\mu$ m sieves, whereas all pollen grains are collected as filtered residue. Pollen grains are not destroyed in hot hydrofluoric acid (HF), though nearly all types of organic material, and even silica particles, are destroyed in hot HF. All charcoal particles and organic pieces that were not dissolved in HF are visible in a microscope and can be removed using microtubes.

Pollen and spores from ice-wedge ice are characteristic of the regional pollen rain. Thus, the dated extracts of pollen and spores from ice-wedge ice provide an adequate simulation and chronology of landscape dynamics on a regional scale. However, one must compare the date of the pollen concentrate with the dates of the alkali extract and microorganic fractions from the same sample, given that pollen and spores have good preservation attributes and ample opportunity for redeposition. Redeposited pollen and spores enter frost cracks along with ground particles transported by wind on the surface of the snow cover. Pollen from syngenetic ice wedges is well preserved and relatively free of other organic admixtures.

In order to reduce contamination, we collected the samples from permafrost sediments and ice wedges which never melted since the time of their formation. We isolated every sample from the environment during the ice-melting stage and controlled every step using a microscope.

## METHODS

We have designed a procedure to pretreat pollen and spores from Late Pleistocene syngenetic ice-wedge ice for AMS <sup>14</sup>C dating (Vasil'chuk et al. 2003) by considering the methods of pollen pretreatment for AMS <sup>14</sup>C dating from peat deposits (Brown et al. 1989; Richardson and Hall 1994), lacustrine and marine deposits (Long et al. 1992; Regnell 1992; Mensing and Southon 1999; Kretschmer et al. 1999; Kilian et al. 2002), and loess (Zhou et al. 1997). The main goal of our pretreatment is to exclude quartz and other mineral particles of sizes comparable to pollen grains from the pure pollen concentrate. The critical stages are treatment in hot 40% HF for silicate removal, fol-

lowed by centrifugation in heavy liquid. The technique has 14 preparatory steps before the graphitization of the pure pollen concentrate (see Table 1).

The microorganic fraction was obtained by filtering through 100- $\mu\text{m}$  precision-woven polyester mesh for the Seyaha samples, and 200- $\mu\text{m}$  mesh for the Bison cross-section. A standard acid-alkali-acid procedure was used for the pretreatment of this fraction, as well as the alkali extract fraction.

Table 1 Pretreatment method of ice-wedge ice samples (after A Vasil'chuk).

1.	150-g sample taken from residue from ice-wedge meltwater;
2.	Deflocculate and remove some of the diatom and humic acids by treatment with 10% KOH solution at 85 °C for 1 hr;
3.	Wash samples through precision-woven polyester meshes (100 $\mu\text{m}$ , 60 $\mu\text{m}$ , and 10 $\mu\text{m}$ ) to remove a larger fraction of the inorganic and organic matrix. A final wash and retention of the samples with 10- $\mu\text{m}$ polyester mesh (due to the small size of pollen from tundra plants);
4.	Hot 40% HF for 30 min for silicate removal;
5.	Hot 1N HCl (to remove $\text{SiF}_4$ );
6.	Agitation in an ultrasonic tank for a maximum of 15 min for further deflocculation of the matrix;
7.	Centrifugation in heavy liquid $\text{ZnCl}_2$ with density 1.9 $\text{g}/\text{cm}^3$ . This density is well suited for the separation of any heavy inorganic fraction with a density of 2.6 $\text{g}/\text{cm}^3$ from the light organic fraction with a density of 1.3–1.5 $\text{g}/\text{cm}^3$ (consisting mostly of pollen);
8.	Washing with 10- $\mu\text{m}$ mesh;
9.	10–60- $\mu\text{m}$ fraction bleached in 2–3% $\text{NaClO}_2$ for 3 min;
10.	10% HCl, then rinse with distilled water;
11.	Washing with 10- $\mu\text{m}$ mesh;
12.	Collection of pure pollen concentrate from 10–60- $\mu\text{m}$ fraction by micro-glass tubes and drying (Mensing and Southon 1999);
13.	Combustion of sample and collecting of $\text{CO}_2$ gas;
14.	Graphitization of pure pollen concentrate (Kim et al. 2000).

Pollen and spore concentrates from Late Pleistocene ice-wedge ice were dated in the AMS facility of Seoul National University. All pollen identifications were done after complete treatment, as it was important to specify which pollen was dated. The  $^{14}\text{C}$  dates of the pollen and spore concentrates were much older than the dates of the microinclusions. We assumed that the pollen and spore concentrates also contain reworked pre-Quaternary and Quaternary palynomorphs.

#### Indicators of Penecontemporaneous Pollen Spectra

We propose palynologic and  $^{14}\text{C}$  dating indicators to determine palynomorphic redeposition. Palynologic indicators include the following: Penecontemporaneous pre-Quaternary pollen and spores indicate a different degree of palynomorphic preservation. Charcoal particles also could be used as an indicator of redeposition, because the  $^{14}\text{C}$  dates of recent lenses of charcoal particles on the river or bay banks range from the present day to 5000 yr old. The charcoal may be older or contemporaneous as a result of fires during ice formation. Charred palynomorphs are a result of diagenesis, and could also be penecontemporaneous. The difference between AMS  $^{14}\text{C}$  dates of different fractions (alkali extract, microorganic inclusions, and pollen concentrate) from the same sample is also an indicator of redeposition. If the date of the pollen concentrate is not the youngest date, the

pollen concentrate contains penecontemporaneous palynomorphs. Non-palynologic admixtures, such as charcoal particles or charred palynomorphs, may affect the <sup>14</sup>C age of the pollen concentrate.

Some pollen grains in ice wedges are enveloped with clay particles. We suggest that this feature is caused by melting and re-freezing processes out of the ice-wedge body. We believe that this pollen is contemporaneous with the ice.

**RESULTS**

**Bison Ice-Wedge Ice**

This ice-wedge ice complex was dated using microinclusions of organic material and alkali extract from the ice. Samples were collected from 3-stage ice wedges from the main wall of the exposure (see Figure 2, wedges 1 and 2) and from the thermo-eroded part (see Figure 2, ice wedge 3). Seven samples from this section were analyzed at the Centre for Isotope Research, Groningen University (the Netherlands), and we obtained 14 <sup>14</sup>C dates (from 26,000 BP to more than 38,000 BP) from microinclusions (>200 μm in size) and from alkali extracts (Vasil'chuk et al. 2001a, 2002).

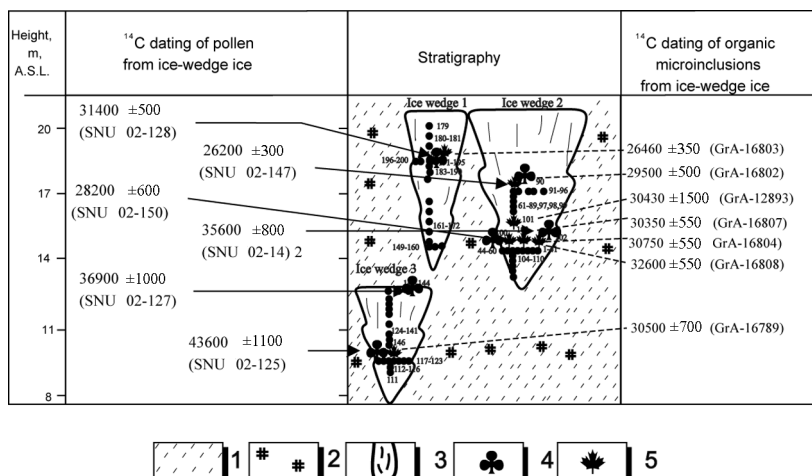


Figure 2 Syngenetic sediments with ice-wedge complexes in the Bison cross-section. Legend: 1 = loamy sand; 2 = horizons enriched with autochthonous and allochthonous organic material; 3 = large syngenetic Late Pleistocene ice wedges; 4 = AMS samples of pollen concentrate from syngenetic Late Pleistocene ice wedges (numbers correlate with Tables 2 and 3); and 5 = AMS <sup>14</sup>C samples of total organic microinclusions from syngenetic Late Pleistocene ice wedges.

Pollen concentrates were obtained in 6 samples using a special procedure for pollen and spore extraction for AMS <sup>14</sup>C dating (Table 2).

Six dates from ~43,600 to ~26,200 BP were obtained on pollen concentrate of ice wedges from the Bison cross-section (see Figure 2). The content of reworked pollen in this cross-section is not more than 1% of the total pollen. This is caused by the low content of reworked palynomorphs in all facies. In some samples, charred pollen grains and spores were found.

- The uppermost sample, 378-YuV/195, was collected from a narrow vein at 18.0 m asl and gave a <sup>14</sup>C age of 31,400 ± 500 BP (SNU02-128). The low concentration (164 grain/L) of pollen and local components of pollen spectra corresponds to landscapes of Arctic tundra (Table 3). On the

Table 2 Direct  $^{14}\text{C}$  AMS dating of pollen and spore concentrate from ice-wedge ice in the Bison cross-section, lower Kolyma River valley, north of Yakutia.

Field nr	Lab nr	Height (m, asl)/Depth (m)	$^{14}\text{C}$ age (BP)
<b>Ice wedge 1</b>			
378-YuV/195	SNU02-128	18.0/2.6	31,400 ± 500
<b>Ice wedge 2</b>			
378-YuV/90	SNU02-147	16.6/4.0	26,200 ± 300
378-YuV/100	SNU02-150	13.0/7.6	28,200 ± 600
378-YuV/102*	SNU02-124	13.0/7.6	35,600 ± 800
<b>Ice wedge 3</b>			
378-YuV/144	SNU02-127	12.5/8.1	36,900 ± 1000
378-YuV/146*	SNU02-125	9.6/11.0	43,600 ± 1100

other hand, the high content of *Pinus pumila* (42%) and the high concentration of dust in the ice is evidence of intensive wind transport of dust and pine pollen. Given that the date of the micro-organic material is the youngest, some portion of *Pinus pumila* pollen possibly was redeposited—sufficient evidence does not exist to prove otherwise. We have marked maximum percentages of *Pinus pumila* pollen in many pollen diagrams of permafrost sediments in northern Siberia (Vasil'chuk 2003). This event occurred about 31,000 BP.

- Sample 378-YuV/90 was collected at 16.6 m asl in a wide ice wedge, and the pollen concentrate dated to 26,200 ± 300 BP (SNU02-147). A pollen spectrum from the sample corresponds to landscapes of typical (hypo-Arctic) tundra, without any signs of redeposition. All pollen grains and spores have a similar state of preservation, and there are no charcoal particles or charred palynomorphs. The date of pollen concentrate most likely is authentic.
- Sample 378-YuV/100 was collected at 13.0 m asl from the same wide ice wedge as the sample above. The pollen concentrate dated to 28,200 ± 600 BP (SNU02-150). A pollen spectrum from the sample is characterized by high content of *Betula* sect. *Nanae* (38.4%) and corresponds to landscapes of birch shrub tundra. There are no indications of pollen redeposition; thus, the date for the pollen concentrate most likely is authentic.
- Sample 378-YuV/102 was collected at 13.0 m asl from the same wide ice wedge as above. The pollen concentrate dated to 35,600 ± 800 BP (SNU02-124). Palynomorphs here show various degrees of preservation: Pollen of *Betula* sect. *Albae*, *Pinus pumila*, and *Artemisia* are often destroyed, while herb pollen was well preserved. There are several grains of dark charred palynomorphs. We suppose that the date of pollen concentrate is older than the ice because there are indicators of redeposition. Some components of the pollen spectra, such as charred palynomorphs, are probably penecontemporaneous.
- Sample 378-YuV/144 was collected at 12.5 m asl from a wide ice wedge from the lowest stage. The pollen concentrate dated to 36,900 ± 1000 BP (SNU02-127). Here, ice is transparent due to its low dust content. Components of pollen spectrum are probably penecontemporaneous. There were pre-Pleistocene pollen grains of *Pinaceae* and *Quercus sibirica*. The content of charcoal particles was 9.9%; they also could be penecontemporaneous. The pollen concentrate is slightly older than the ice. The pollen of *Larix* (0.3%) was present, which is evidence of forest tundra or larch forest. Larch pollen would have been destroyed very quickly in the sediments.
- Sample 378-YuV/146 was collected at 9.6 m asl from the same ice wedge of the lowest stage as the sample above. The pollen concentrate dated to 43,600 ± 1100 BP (SNU02-125). The pollen spectrum is rich, with *Larix* (3%) and various species of herbs found here, and corresponds to larch woodland. But there also exists 25% of dark charred undetermined palynomorphs, and

Table 3 Percentages of pollen and spores in comparison with AMS <sup>14</sup>C data.<sup>a</sup>

Field nr	378-YuV/195	378-YuV/90t	378-YuV/100	378-YuV/102 <sup>b</sup>	378-YuV/144 <sup>c</sup>	378-YuV/146 <sup>d</sup>
Height (m asl)/ depth (m)	18.0/2.6	16.6/4.0	13.0/7.6	13.0/7.6	12.5/8.1	9.6/11.0
<sup>14</sup> C age (BP) pollen concentrate; Lab #	31,400 ± 500 SNU02-128	26,200 ± 300 SNU02-147	28,200 ± 600 SNU02-150	35,600 ± 800 SNU02-124	36,900 ± 1000 SNU02-127	43,600 ± 1100 SNU02-125
<sup>14</sup> C age of organic microinclusions	26,460 ± 350 <sup>e</sup>	29,500 ± 500	32,600 ± 700	30,750 ± 550	None	30,500 ± 550
<sup>14</sup> C age of alkaline extract	27,790 ± 400	32,200 ± 650	36,300 ± 1000	33,500 ± 750	None	>38,400
AP Tree pollen	4.8	—	—	0.8	1.2	5.0
Scrub pollen	50.4	28.6	40.8	8.7	26.1	26.3
NAP Herb pollen	21.6	49.3	48.6	55.7	41.2	35.1
Spores	23.2	22.1	10.6	34.8	31.5	33.6
<i>Pinus silvestris</i>	1.2	—	—	—	—	—
<i>Pinus sibirica</i>	1.2	—	—	—	—	—
<i>Betula</i> sect. <i>Albae</i>	2.4	—	—	0.8	0.6	1.9
<i>Larix</i>	—	—	—	—	0.6	3.1
<i>Pinus pumila</i>	42.0	10.4	—	1.6	2.7	5.6
<i>Betula</i> sect. <i>Nanae</i>	8.4	15.6	38.4	4.0	23.4	16.9
<i>Alnaster</i>	—	—	—	1.6	—	2.5
<i>Salix</i>	3.6	2.6	2.4	1.6	—	1.3
<i>Poaceae</i> (small)	1.2	—	—	1.6	6.4	4.4
<i>Poaceae</i> (large)	0.8	—	—	5.6	—	—
<i>Cyperaceae</i> ( <i>Carex</i> )	13.6	23.8	25.2	20.5	21.3	6.9
<i>Cyperaceae</i> ( <i>Eriophorum</i> )	2.4	10.0	—	3.8	—	0.6
<i>Ericaceae</i>	—	1.3	—	0.8	—	1.9
<i>Artemisia</i>	—	—	—	1.6	—	—
<i>Compositae</i>	—	—	—	—	1.8	—
<i>Varia</i>	3.6	6.5	18.0	19.4	7.2	13.1
<i>Chenopodiaceae</i>	—	—	0.9	1.6	—	—
<i>Polygonaceae</i>	—	—	—	—	0.9	0.6
<i>Polemoniaceae</i>	—	—	0.9	—	—	1.3
<i>Rosaceae</i>	—	5.1	—	—	2.7	1.9
<i>Dryas</i> sp.	—	—	3.6	—	—	1.3
<i>Brassicaceae</i>	—	—	—	0.8	—	—
<i>Draba</i> sp.	—	—	—	—	—	0.6
<i>Saxifragaceae</i>	—	—	—	—	—	1.9
<i>Liliaceae</i>	—	1.3	—	—	—	0.6
<i>Juniperus</i>	—	1.3	—	—	0.9	—
<i>Bryales</i>	3.6	11.7	6.0	25.2	14.4	9.2
<i>Sphagnum</i> sp.	—	—	—	3.2	1.8	0.6
<i>Polypodiaceae</i>	1.2	—	—	0.8	4.5	0.6
<i>Equisetum</i>	2.8	10.4	4.6	4.2	4.5	1.3
<i>Selaginella sibirica</i>	15.6	—	—	1.4	6.3	21.9
Nr of grains	295	280	311	382	342	722
Ice-sample vol (L)	1.8	1.8	1.9	1.2	1.5	1.5

Table 3 Percentages of pollen and spores in comparison with AMS  $^{14}\text{C}$  data.<sup>a</sup> (Continued)

Field nr	378-YuV/195	378-YuV/90t	378-YuV/100	378-YuV/102 <sup>b</sup>	378-YuV/144 <sup>c</sup>	378-YuV/146 <sup>d</sup>
Grains per liter	164	156	164	318	228	481
Redeposited pollen and spores	—	—	1% <i>Ulmus</i> ; <i>Diervilla</i>	—	0.4% <i>Quercus sibirica</i> ; <i>Pinaceae</i>	1% <i>Sporites durabilis</i> ; <i>Trudopollis</i> sp.; <i>Shchizaceae</i> ; <i>Leotriletes</i> sp.

<sup>a</sup>Obtained for dating different organic fractions from the same samples of ice-wedge ice in the Bison cross-section.

<sup>b</sup>Date of the sample from the same depth (378-YuV/182).

<sup>c</sup>Six charred pollen grains were found; no charcoal particles were found; the herb pollen aggregated into clay.

<sup>d</sup>Many mushroom spores were found; almost all herb pollen aggregated into clay.

<sup>e</sup>25% charred pollen and spores were found, as well as phytoliths of sedge, grasses, and *Artemisia*.

also pre-Pleistocene *Sporites durabilis*, *Trudopollis* sp., and *Shchizaceae*. Thus, the pollen characteristics correlate to the landscape, but the pollen concentrate could be older than the ice. According to the pollen data, the landscapes changed from forest tundra to hypo-Arctic tundra and then to Arctic tundra. A detailed comparison with the pollen data of host sediments will provide more information.

### Seyaha Ice-Wedge Ice

We first dated the pollen from ground ice we collected from the Seyaha ice-wedge ice. The dates obtained show that the pollen concentrate contains redeposited, long-preserved pollen and spores (Figure 3; Tables 4–5). Hence, we have the ability to evaluate the  $^{14}\text{C}$  dates of pollen concentrate in terms of the ratio of contemporaneous and ancient organic material in the sample by using the content of ancient pollen in the sample. In the radiocarbon laboratory of Groningen University, organic microinclusions (more than 100 microns) and alkaline extract from the same samples were AMS dated (Vasil'chuk et al. 2000) for the first time. The  $^{14}\text{C}$  dates of microinclusions (from ~21,000 to ~14,000 BP) were younger than the  $^{14}\text{C}$  age of alkaline extract by 9000–5000 yr. This indicates that the alkaline extract contains ancient organic material.

- Sample 363-YuV/27 was collected at 1.8 m asl. Microorganic fractions from the ice dated to  $14,550 \pm 100$  BP, the alkaline extract to  $19,920 \pm 130$  BP, and the pollen to  $25,200 \pm 150$  BP (SNU01-214). From the 496 pollen grains and spores counted, 19.3% are redeposited pre-Quaternary palynomorphs, while charcoal particles make up 12% of the total pollen and spore sum (see Table 4). The majority of the older organic materials exist in the fraction at 10–60  $\mu\text{m}$ .
- Sample 363-YuV/108 was collected at 4.8 m asl and dated to  $21,170 \pm 180$  BP (SNU01-216). Redeposited pollen and spores are 6.6% of the total, while charcoal particles are 5% of the total pollen and spore sum. The  $^{14}\text{C}$  date of this sample is more reliable because indicators of redeposition pointed to less redeposition of organic materials.
- Sample 363-YuV/87 was collected at 12 m asl and dated to  $22,400 \pm 100$  BP (SNU01-216). Organic microinclusion remains in this sample dated to  $14,720 \pm 100$  BP, and the alkaline extract to  $23,620 \pm 160$  BP. The redeposited grain content is 15.4% of the total sum. The age of the alkaline extract is the oldest. We conclude that the ancient organic material is contained both in the 10–60- $\mu\text{m}$  fraction and the alkali fraction.

The use of AMS has allowed us to advance considerably in understanding the dating of organics and to define a ratio between the dates of a bulk sample and its separate components. The separation of samples into components to search for those most suitable for dating material is important for the  $^{14}\text{C}$  dating problem of fluvial permafrost sediments.



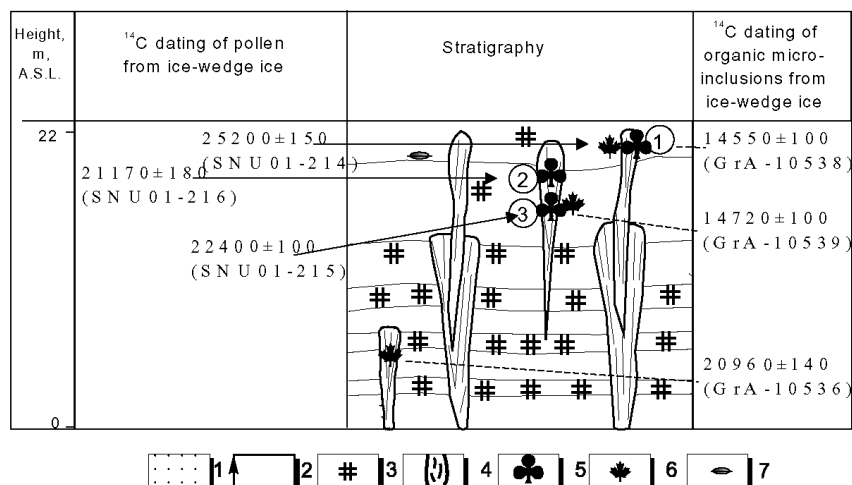


Figure 3 Syngenetic sediments with ice-wedge complexes near the Seyaha settlement. Legend: 1 = sand; 2 = loamy sand with a small amount of allochthonous organic matter; 3 = horizons enriched with autochthonous and allochthonous organic; 4 = large syngenetic Late Pleistocene ice wedges; 5 = AMS <sup>14</sup>C samples of pollen concentrate from syngenetic Late Pleistocene ice wedges (numbers correlate with Tables 4–5); 6 = AMS <sup>14</sup>C samples of total organic microinclusions from syngenetic Late Pleistocene ice wedges; and 7 = marine foraminiferans.

Table 4 Direct AMS <sup>14</sup>C dating of pollen and spore concentrate from ice-wedge ice in the Seyaha cross-section, Yamal Peninsula, northwest Siberia.

Field nr	Height (m, asl)/Depth (m)	<sup>14</sup> C age (BP)	Lab nr
<b>Ice wedge 1</b>			
363-YuV/27	20.2/1.8	25,200 ± 150	SNU01-214
<b>Ice wedge 2</b>			
363-YuV/108	17.2/4.8	21,170 ± 180	SNU01-216
<b>Ice wedge 3</b>			
363-YuV/87	10.0/12.0	22,400 ± 100	SNU01-215

A sample of yellow allochthonous peat with a sandy loam admixture was collected at 0.2 m asl above Ob Bay from a cross-section of the third sea terrace (363-YuV/208). It dated to 36,800 +3300/–2100 BP (Hel-3950). However, a dwarf birch twig from this sample was younger in age, 31,200 ± 90 BP (Hela-201), revealing significant impurities of allochthonous material in the sample. The structure of the pollen spectra confirms the presence of redeposited organic material, as the content of redeposited pollen and spores is 11.2% of the total. The synchronous part of the pollen spectra shows typical tundra features and consists of *Pinus silvestris* 1%, *P. sibirica* 5%, *Betula* sect. *Albae* 7%, *Salix* 7%, *Betula* sect. *Nanae* 16%, *Alnaster* 1%, *Poaceae* 13%, *Cyperaceae* 7%, *Varia* 9%, *Ericaceae* 5%, *Artemisia* 10%, *Bryales* 10%, *Sphagnum* 1%, *Polypodiaceae* 3%, and *Lycopodium* sp. 1%.

Table 5 Percentages of pollen and spores and comparison of AMS  $^{14}\text{C}$  data obtained from dating different organic fractions of the same samples of ice-wedge ice in the Seyaha cross-section.

Field nr	363-YuV/27	363-YuV/108	363-YuV/87
Height (m asl)/depth (m)	20.2/1.8	17.2/4.8	10.0/12.0
$^{14}\text{C}$ age (BP) of pollen concentrate; lab nr	25,200 $\pm$ 150 SNU01-214	21,170 $\pm$ 180 SNU01-216	22,400 $\pm$ 100 SNU01-215
$^{14}\text{C}$ age of organic microinclusions	14,550 $\pm$ 100	—	14,720 $\pm$ 100
$^{14}\text{C}$ age of alkaline extract	19,920 $\pm$ 130	—	23,620 $\pm$ 160
AP Tree pollen	17.0	12.1	9.0
Scrub pollen	21.0	37.4	12.6
NAP Herb pollen	25.5	40.7	54.1
Spores	36.5	9.8	24.3
<i>Pinus silvestris</i>	3.0	5.5	2.7
<i>Pinus sibirica</i>	6.5	—	—
<i>Picea</i> sp.	2.0	—	—
<i>Betula</i> sect. <i>Albae</i>	4.5	6.6	6.3
<i>Alnus</i> sp.	1.0	—	—
<i>Betula</i> sect. <i>Nanae</i>	15.0	17.6	9.0
<i>Alnaster</i>	3.0	6.6	1.8
<i>Salix</i>	3.0	13.2	1.8
<i>Poaceae</i> (small)	7.0	3.0	4.5
<i>Poaceae</i> (large)	6.0	10.2	—
<i>Cyperaceae</i> ( <i>Carex</i> )	8.0	12.0	1.8
<i>Cyperaceae</i> ( <i>Eriophorum</i> )	—	10.0	—
<i>Ericaceae</i>	0.5	1.1	—
<i>Polygonum bistorta</i>	—	—	0.9
<i>Polemoniaceae</i>	1.5	—	—
<i>Rosaceae</i>	—	—	0.9
<i>Rubus chamaemorus</i>	0.5	—	—
<i>Draba</i> sp.	1.0	—	3.6
<i>Apiaceae</i>	0.5	—	—
<i>Caryophyllaceae</i>	—	1.1	—
<i>Lamiaceae</i>	0.5	—	—
<i>Papaveraceae</i>	—	1.1	—
<i>Sparganium</i>	—	1.1	40.6
<i>Liliaceae</i>	—	1.1	1.8
<i>Bryales</i>	16.0	5.6	21.6
<i>Sphagnum</i> sp.	2.0	1.2	—
<i>Polypodiaceae</i>	12.0	—	1.8
<i>Equisetum</i>	4.0	0.9	0.9
<i>Lycopodium clavatum</i>	0.5	1.2	—
<i>Lycopodium</i> sp.	2.0	—	—
<i>Selaginella sibirica</i>	—	0.9	—
Nr of grains	496	340	425
Volume of ice sample (L)	1.0	0.8	0.8
Grains per liter	496	272	321
Redeposited pollen and spores (%)	19.3 ( <i>Picea</i> ; <i>Abies</i> ; <i>Liquidambar</i> ; <i>Pterocarya</i> ; <i>Taxodiaceae</i> ; <i>Nudopollis</i> sp.; <i>Trudopollis</i> sp.)	6.6 ( <i>Pinaceae</i> ; <i>Taxodiaceae</i> ; <i>Carya</i> sp.)	15.4 ( <i>Pinaceae</i> ; <i>Taxodiaceae</i> ; <i>Alnus</i> ; <i>Ulmus</i> ; <i>Plantaginaceae</i> )

## DISCUSSION

Our results from AMS dating the pollen concentrate show that the content of penecontemporaneous reworked pollen in permafrost sediments and ground ice varies greatly in light of facial conditions, vegetation zone, and regional features. The highest concentration of penecontemporaneous pollen and spores is found in the marine and fluvial sediments, and the lowest in the autochthonous peat. In the tundra, the relative content of penecontemporaneous pollen and spores is higher than in the forest, due to the low pollen productivity of tundra plants. In the forest and forest tundra, microorganic remains in the syngenetic ice wedges are often contemporaneous with ice formations, because organic particles as a rule originated from nearby shrubs or trees that are above the snow cover.

In the Lower Kolyma region, the content of pre-Quaternary palynomorphs in recent pollen spectra varied from 0.5–1% in moss pollsters to 2–3% in modern alluvial sediments. In the Bison section, ice wedges in <sup>14</sup>C data sets for samples 378-YuV/102 and 378-YuV/195 are the oldest among other organic fractions, and there is no evidence of redeposited palynomorphs. Different degrees of preservation of Quaternary pollen of *Betula* sect. *Albae*, *Pinus pumila*, and *Artemisia* may indicate possible redeposition. In the <sup>14</sup>C data set of sample 378-YuV/100, the pollen concentrate date is the youngest. All palynomorphs are well preserved, including pollen of *Ulmus*, which also could be contemporaneous. *Ulmus* pollen found in the stomach of a Selerican horse in the Indigirka River valley dated to ~38,000 BP (Ukrainseva 1993).

In northwest Siberia, the content of pre-Quaternary palynomorphs in recent pollen spectra varied from 3–5% in moss pollsters to 20–33% in modern beach sediments. If we take the uppermost sample (363-YuV/27) from the Seyaha ice wedges as an example and suppose that 14,550 ± 100 BP is the age of ice accumulation, adding 19.3% of “dead” carbon to the contemporary carbon will reduce the age to ~16,100 BP, which is still much younger than the age of the pollen concentrate (25,200 ± 150 BP). In order to achieve a pollen concentrate age beginning at 14,550 BP, we need to add as much as 260% of dead carbon (Olsson 1974, 1991). Evidently, that pollen spectrum almost completely consists of reworked Quaternary palynomorphs, which are very difficult to separate. Only AMS dating of pollen concentrate indicates redeposition. Thus, in this case the content of pre-Quaternary palynomorphs is the only indicator of the general process of sediment and pollen spectra accumulation. A low concentration of local pollen and spores often corresponds to very cold conditions.

The correlation between the dates of pollen and the content of redeposited pollen and spores in spectra is obvious. The youngest date corresponds to the least amount of redeposited pollen and spores. The dates of organic microinclusions (in our case, this is organic material insoluble in alkali and larger than 100 μm) are the youngest. It is possible to conclude that dates of pollen concentrate are older as a result of pollution by dead carbon from palynomorphs of pre-Quaternary age and also from Quaternary penecontemporaneous palynomorphs, which are impossible to separate.

A comparison of dates of pollen concentrate from Seyaha and Bison ice wedges shows that, as a rule, the dates of alkaline extract are the oldest, and the organic microinclusion dates are the youngest. Yet, in 2 cases the date of pollen concentrate was the youngest (SNU02-147 and SNU02-150, see Figure 2). The age depends on the surrounding landscape. We suppose that in the forest or forest tundra, the pollen spectra are often contemporaneous with the ice-wedge ice pollen. This is due to the lower wind speeds in the forest and forest tundra and also because of the higher background concentration of organic material in those regions. Higher pollen productivity of forest landscapes results in more pollen entering the ice wedges (e.g. the Bison cross-section); thus, in these conditions the <sup>14</sup>C dates of pollen concentrate are often the youngest. In the Arctic tundra and polar desert,

however, the local pollen concentration is less than the reworked pollen and spores. Thus, the  $^{14}\text{C}$  dates of pollen concentrate in the tundra and polar desert are often older than the ages of other fractions of organic material.

A series of 6 AMS dates of pollen concentrates from loess in China shows an inversion of  $^{14}\text{C}$  dates. The top sample (0.11 m asl) dated to  $6950 \pm 60$  BP (AA-12317), and the bottom sample (3.25 m asl) to  $11,250 \pm 80$  BP (AA-12316) (Zhou et al. 1997). Sample AA-12315 at 1.58 m dated to  $13,260 \pm 90$  BP and contained redeposited pre-Quaternary pollen and spores. It is possible that the sediments were accumulated in permafrost conditions, as evidenced by the distribution of  $^{14}\text{C}$  dates.

Kilian et al. (2002) conducted AMS  $^{14}\text{C}$  dating of pollen concentrates from lacustrine sediments of Lake Gościąg in Poland. The annual lamination of the lake's sediments has been used as a test for  $^{14}\text{C}$  dating of pollen concentrates. Kilian et al. found that the pollen (almost all tree pollen) apparently could not be concentrated to the desired purity. The dating results were about 660 yr too old because of admixtures from other organic remains or possibly due to allochthonous organic molecules that had adsorbed to the pollen walls. A series of macrofossils (mainly single-year bud scales from *Pinus*) yielded much better results, though. These dates are about 100 yr too old according to the absolute ages of the laminae. The results show that AMS dates of pollen concentrates may have a consistent intersample  $^{14}\text{C}$  age, but may nevertheless be many centuries too old. Without the time control based on annual laminae and without the AMS dates of macrofossils, the interpretation of the AMS dates of pollen concentrates would have resulted in a mistake. On the other hand, Gillespie (1990) shows that pollen concentrates from lacustrine sediments of Lake Tirrel (Australia) become older after oxidation. The date of pollen concentrate using standard procedure is  $7215 \pm 270$  BP (NZA-192), but after oxidation the age of the same sample is  $7425 \pm 445$  BP (NZA-193). Thus, some discrepancy between AMS dates of macrofossil and pollen concentrates could be caused by differences between the treatment of macrofossils and pollen concentrates in Kilian et al. (2002).

In the Arctic, inversions of dates on different fractions of organic material more likely are the rule than the exception.  $^{14}\text{C}$  dating of a 5-m cross-section of horizontally layered well-sorted sand and sandy loam in Cumberland Peninsula (Baffin Island, Canada) has shown an admixture of ancient organic material, as the  $^{14}\text{C}$  inversion is more than 7000 yr. As a result of the methodical study of Stuckenrath et al. (1979), it was possible to achieve a number of dates without inversions only on a rather large fraction of organic material which is insoluble in alkali ( $>125 \mu\text{m}$  in size), whereas dating the soluble part of the alkali fraction has shown both a younger and an older age. These data also are very important in understanding our results of AMS dating of organic material from ice-wedge ice. Only the organic remains of microinclusions (organic material insoluble in alkali,  $>100 \mu\text{m}$  in size) gave the youngest dates without  $^{14}\text{C}$  age inversions.

The problem of permafrost sediments with allochthonous organic material was studied by Nelson et al. (1988) at an exposure of Holocene sediments in the Ipikuk River valley in Alaska. To define the sources of contamination, the allochthonous organic material of peat from a lens was separated into different fractions and dated, resulting in the following dates: the  $>2$ -mm fraction of peat dated to  $13,250 \pm 100$  BP (USGS-2046A); the 1–2-mm fraction was  $17,730 \pm 110$  BP (USGS-2046B); the 0.5–1.0-mm fraction was  $24,740 \pm 320$  BP (USGS-2046C); the 0.25–0.5-mm fraction was  $30,260 \pm 530$  BP (USGS-2046D); and the  $<0.25$ -mm fraction was  $20,360 \pm 190$  BP (USGS-2046E). Dating a bulk sample of peat from the same layer resulted in a date of  $13,730 \pm 110$  BP (USGS-883). One may conclude that the smaller the fossil size, the older the date. Given the wide array of  $^{14}\text{C}$  dates, the youngest dates are most reliable. Pollen analysis results have shown that in lenses of peat, the content of redeposited pre-Quaternary pollen and spores is about 50% of the total (Nelson et al. 1988).

Differences in <sup>14</sup>C dates in permafrost sediments can be caused by reservoir effect when the plants use ancient CH<sub>4</sub> or CO<sub>2</sub> contained in permafrost or in the unfrozen ground. Some species of mushrooms that live on *Ericaceae* roots use ancient CH<sub>4</sub> (Pancost et al. 2000). If *Ericaceae* is among the plant remains in the peat, <sup>14</sup>C dates are probably older, depending on the methane age. The age of CH<sub>4</sub> and CO<sub>2</sub> contained in permafrost was determined in lakes of the Lower Kolyma River valley (Zimov et al. 1997). Their measurements show the age of methane in lacustrine sediments. A number of <sup>14</sup>C dates without inversions from the bottom-up were obtained: 38,000, 27,000, 15,000, 11,000, and 8000 BP. Hence, it is clear that ancient methane can affect the <sup>14</sup>C age of upper lacustrine and bog permafrost sediments. The reservoir effect varies from lake to lake.

## CONCLUSION

Pollen and spores are well preserved in ice wedges and their concentration is enough for AMS dating. The extraction of pollen and spores from ice-wedge ice requires some modifications in sample pretreatment. It is obvious from our data of pollen concentrates from syngenetic ice wedges that the contents of pre-Quaternary pollen and spores could be used as an indicator of the <sup>14</sup>C date reliability. The identification of Mesozoic and Paleozoic palynomorphs in samples provides evidence that a bulk sample could contain a significant amount of “dead” carbon. It is very important that a preliminary pollen analysis be used to estimate the reliability of <sup>14</sup>C dates before dating.

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