

First radiocarbon dating of pollen and spores from syngenetic ice-wedge ice

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ABSTRACT: The objectives of our study are to 1) elaborate the procedure for pollen concentrate extracting and AMS radiocarbon dating of pollen and spores extracted from syngenetic ice-wedges and surrounding sediments, 2) to obtain AMS radiocarbon dates of pollen concentrate from the ice-wedges ice and from host sediments 3) to work up quantitative criteria for evaluation of the dates obtained in terms of autochthonous and allochthonous matrix ratio in the samples using ancient pollen contents in the sample 4) to remove the non-pollen organic matrix and silicates. Taking into account the variety of techniques and different approaches to pretreatment of sediments of different origin (Brown et al. 1989; Gillespie 1990; Regnell 1992; Richardson & Hall 1994; Zhou et al. 1997; Kretschmer et al. 1999) and also the features of syngenetic ice-wedge formation, we elaborated a modified technique for pollen concentrate extraction from ice-wedge ice for AMS-dating. The first ^{14}C dates of pollen and spores from syngenetic ice-wedge ice using AMS techniques shown that pollen in 3–5 kg of ice-wedge ice is enough for ^{14}C dating, the dates of pollen are in good correlation with ratio of re-deposited and autochthonous pollen in the samples.

1 INTRODUCTION

The objectives of our study is to elaborate the procedure for pollen concentrate extracting and AMS radiocarbon dating of pollen and spores extracted from syngenetic ice-wedges and surrounding sediments, to obtain AMS radiocarbon dates of pollen concentrate from the ice-wedges ice and from host sediments, to work up quantitative criteria for evaluation the dates obtained in terms of autochthonous and allochthonous matrix ratio in the sample using content of ancient pollen in the sample, and to remove the non-pollen organic matrix and silicates. Taking into account different techniques and different approaches to pretreatment of different origin sediments (Brown et al. 1989;

Gillespie 1990; Regnell 1992; Richardson & Hall 1994; Zhou et al. 1997; Kretschmer et al. 1999) and also the features of syngenetic ice-wedge formation, we worked out a modified technique for pollen concentrate extraction from ice-wedge ice for AMS-dating. The first ^{14}C dates of pollen and spores from syngenetic ice-wedge ice using AMS techniques have shown that pollen in 3–5 kg of ice-wedge ice is enough for ^{14}C dating, the dates of pollen are in good correlation with ratio of re-deposited and autochthonous pollen in the samples.

2 METHODS

We have designed a procedure of pollen and spores extraction from Late Pleistocene syngenetic ice-wedge ice for AMS-dating taking into account methods of pollen pretreatment for AMS-dating. The first results of pollen dating from sediments were obtained in 1989 (Brown et al. 1989). Standard techniques of pollen pretreatment were applied. Pollen concentrate was separated to fractions dated separately: fine (20–44 μ) and large (44–88 μ) fractions about 12600–6490 years. The dates of different fractions are very close, and date of bulk sample of peat is older by 700–1000 years. In 1992 the technique of purification of pollen concentrate from all admixtures in samples of lake sediments from hard water lakes

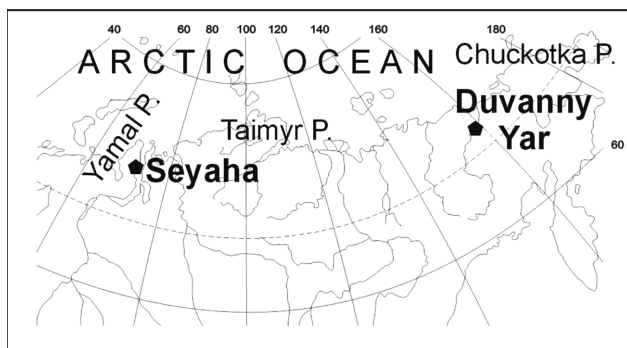


Figure 1. Location map of Seyaha and Duvanny Yar Late Pleistocene syngenetic ice-wedge ice complexes.

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

1. The sample 150 g (residue from ice-wedge melt water).
2. Deflocculating and removing some of the diatom and humic acids by treatment with 10% KOH solution at 85°C for 1 hour.
3. Wash samples through precision-woven polyester meshes of the size range 100, 60, 10 μ to remove larger fraction of the inorganic and organic matrix. A final wash and retention of the samples on 10 μ polyester mesh (due to small size of pollen of tundra plants).
4. Hot 40% HF for 30 min for silicates removal.
5. Hot 1N HCl (removal of SiF₄).
6. Agitation in an ultrasonic tank for a maximum of 15 min for further deflocculating of the matrix.
7. Centrifuging in heavy liquid ZnCl₂ with density 2.1 g/cm³. This density is well suited for the separation of heavy inorganic fraction with density 2.6 g/cm³ mostly contained silicates from the light organic fraction with density 1.3–1.5 g/cm³ mostly consisted of pollen.
8. Washing on a 10 μ mesh.
9. Fraction 60–10 μ 2–3% NaClO₂ bleaching for 3 min.
10. 10% HCl, then rinsed with distilled water.
11. Washing on a 10 μ mesh.
12. Collection of pure pollen concentrate from 10–60 μ fraction by micro glass tubes and drying (according to Mensing and Southon methods 1999).
13. Graphitisation of pure pollen concentrate.

was developed (Long et al. 1992, Regnell 1992). The date of the bulk sample of the lacustrine sediments was also older in comparison with the date of pollen. Every variety of sediments required some modification of methods of sample pretreatment to find the optimal manner of pollen and spore extraction (Richardson & Hall 1994; Kretschmer et al. 1999).

Taking into account these methods and also features of pollen occurrence in ice-wedge ice, such as the size of tundra plants pollen, incoming pollen into frost cracks together with melt water, and dust partially from host sediments we developed the methods of pretreatment ice-wedge ice samples (Table 1).

The main tasks of our methods involved removal of organic admixtures and quartz debris and to prepare a relatively pure pollen concentrate without using carbon-based chemicals. As a result of the pretreatment, organic impurity of non-pollen origin and mineral admixture have been excluded. Fraction 10–60 μ of pure pollen concentrate was combusted to carbon dioxide and dated at Tandetron 4130 AMS of Seoul National University.

3 RESULTS

Along with dating of different kinds of allochthonous organic material, it is very important to obtain dates of

much more informative objects, such as pollen and spores. Dating of pollen concentrate is useful for the evaluation of ratio of synchronous and ancient organic material in close system. This is the first dating of pollen from ground ice (Fig. 2, Table 3). The dates obtained show that pollen concentrate contain re-deposited ancient long preserved pollen and spores. Hence we have a possibility to evaluate the ¹⁴C dates of pollen concentrate in terms of ratio of contemporaneous and ancient organic in the sample using content of ancient pollen in the sample.

In the Groningen AMS laboratory, microorganic remains (fraction more than 100 microns) and alkaline extracts were dated from the same samples for the first time (Vasil'chuk et al. 2000). Pollen and spores concentrate from Late Pleistocene ice-wedge ice has been dated in AMS facility of the Seoul National University. All pollen identifications have been effected after complete treatment, as it was important to specify which pollen was dated.

The sample 363-YuV/27 is collected at the depth of 1.8 m. Microorganic from ice is dated 14550 \pm 100 yr BP, alkaline extract 19920 \pm 130 yr BP. The date of pollen is 25200 \pm 150 yr BP (SNU01-214). From 496 pollen grains and spores 19.3% are re-deposited Pre-Quaternary forms (*Pinaceae*, *Liquidambar*, *Pterocarya*, *Taxodiaceae*, *Nudopollis* sp., *Trudopollis* sp. etc.). Tree pollen 17% of pollen of trees, among which the pollen *Picea* sp. 2%, *Pinus silvestris* 3%, *P. sibirica* 6.5%, *Betula* sp. 4.5%, *Alnus* sp. 1%), pollen of bushes 21% (*Betula* sect. *Nanae* 15%, *Alnaster* sp. 3%, *Salix* 3%), herb pollen 17.5% (*Poaceae* 13%, *Ericaceae* 0.5%, *Polemoniaceae* 1.5%, *Rubus chamaemorus* 0.5%, *Apiaceae* 0.5%, *Lamiaceae* 0.5%, *Draba* sp. 1%), spores 44%.

High contents of re-deposited pollen and spores pointed out that the date is more ancient than the age of ice accumulation and the enclosed sediments.

The sample 363-YuV/108 is collected at depth 4.8 m and is dated 21170 \pm 180 yr BP (SNU01-216). The content of re-deposited forms from 240 counted grains is 9.6% (*Pinus* sp., *Carya* sp., *Taxodiaceae*). Pollen of trees is 12.1% (*Betula* sect. *Albae* 6.6%, *Pinus silvestris* 5.5%), pollen of bushes 37.4% (*Betula* sect. *Nanae* 30.8%, *Alnaster* sp. 6.6%), pollen of herb 38.4% (*Cyperaceae* 22%, *Poaceae* 11.1%, *Liliaceae* 0.9%, *Sparganium* sp. 1.1%, *Papaveraceae* 1.1%, *Caryophyllaceae* 1.1%, *Ericaceae* 1.1%), spores 12.1% (*Bryales* 6.6%, *Sphagnum* sp. 1.1%, *Equisetum* 1.1%, *Lycopodium clavatum* 2.2%, *Selaginella sibirica* 1.1% undetermined spores 1.1%). The ¹⁴C date of this sample is closer to age of microorganic remains.

The sample 363-YuV/87, collected from the depth 12 m. It is dated 22400 \pm 100 yr BP (SNU01-216); microorganic remains in this sample dated 14720 \pm 100 yr BP, and alkaline extract 23620 \pm 160 yr BP.

Depth,

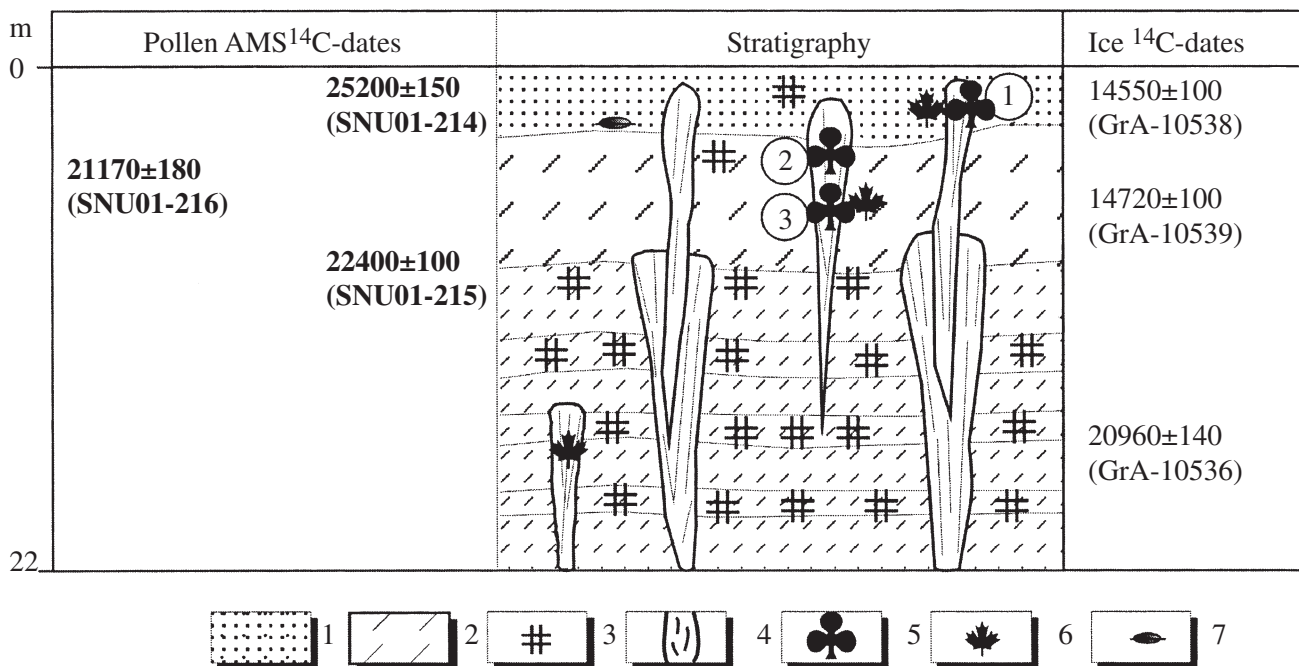


Figure 2. Syngenetic sediments with ice-wedge complexes near the Seyaha settlement: legend 1. sand; 2. loamy sand with small content of allochthonous organic; 3. horizons enriched with autochthonous and allochthonous organic; 4. syngenetic Late Pleistocene large ice wedges; 5. AMS-samples of pollen concentrate from syngenetic Late Pleistocene ice wedges (numbers correspond with Table 3); 6. AMS-samples of total organic micro inclusions from syngenetic Late Pleistocene ice wedges; 7. marine foraminifers.

Table 2. The first ¹⁴C dates of pollen and spores from syngenetic ice-wedge ice using AMS techniques.

Field N	Depth, m (A.S.L., +m)	Lab.N	Age of pollen and spores	δ ¹³ C	Other dates from the same samples
363–YuV/27	1,8 (+20,2)	SNU01-214	25200 ± 150	–25.9	microorganic 14550 ± 100 alkaline extract 19920 ± 130
363–YuV/108	4,8 (+17,2)	SNU01-216	21170 ± 180	–32.7	–
363–YuV/87	12,0 (+10,0)	SNU01-215	22400 ± 100	–25.1	microorganic 14720 ± 100 alkaline extract 23620 ± 160

Table 3. AMS dates of various fractions of allochthonous peat sample from the Duvanny Yar cross-section (316–YuV/9 14 m A.S.L.).

	44200 ± 1100 (ГИИ-4003)– ¹⁴ C	Date of bulk sample
1. Black seed fragments	45700 ± 1200	(SNU01-077) – 32.4
2. Herb remains	39000 ± 1300	(SNU01-076)
3. White twigs without crust	40500 ± 500	(SNU01-078) – 25.6

From 260 grains re-deposited form content is 15.4% (*Pinaceae*, *Taxodiaceae*, *Schizaceae*), tree pollen is 9% (*Betula sect Albae* 6.3%, *Pinus sibirica* 2.7%), pollen of bushes 12.6% (*Betula sect. Nanae* 9%, *Alnaster sp.*

1.8%, *Salix sp.* 1.8), pollen of herb 54.1% (*Cyperaceae* 1.8%, *Poaceae* 4.5%, *Liliaceae* 8.1%, *Sparganium sp.* 34.2%, *Rosaceae* 0.9%, *Polygonum bistorta* 0.9%), spores 23.3% (*Bryales* 21.6%, *Polypodiaceae* 1.8%, *Equisetum* 0.8%). Insignificant content of re-deposited forms confirms the date of this sample is the most authentic.

The application of AMS techniques has allowed to advance considerably in understanding of organic dating and to define a ratio between dates of a bulk sample and separate components of a sample. The separation of samples into components to search most suitable for dating material is very productive for ¹⁴C dating problem of fluvial permafrost sediments.

Sample of yellow allochthonous peat with sandy loam admixture at height 0.2 m above Ob Bay was

collected from cross-section of the third sea terrace (363-YuV/208); it was dated 36800 + 3300/–2100 yr BP (Hel-3950). Age of twig of dwarf birch with crust from this sample is younger. Its date is 31200 + 90 yr BP (Hela-201). It has shown a significant impurity allochthonous material in the sample. The structure of pollen spectra confirms participation of re-deposited organic, as the content of re-deposited forms is 11.2%. Synchronous part of 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%, *Lycopodium sp.* 1%. In order to find the appropriate subject for dating it is very important to obtain dates of various kinds of organic material. For this purpose we have developed the special approach for Duvanny Yar cross-section study (see Fig. 1). We suppose that the lower part of this cross-section contains allochthonous organic material and the most reliable the youngest number of dates. For ^{14}C AMS study the sample 316-YuV/9 at the height +14 m was selected. Hot alkaline extract of the bulk sample dated 44200 ± 1100 yr BP (GIN-4003). Four different fractions of detritus were separated from the bulk sample using a binocular according to standard A-A-A treatment. It was possible to collect seed remains, white twigs without crust (size of 1–0.5 mm), remains of herbs. AMS dating has shown, that: 1) seed remains are the most ancient, they are older than the date of bulk sample; 2) ^{14}C date of white thin twigs is younger than bulk sample, 3) herb remains are the youngest, (Vasil'chuk et al. 2001). These results have confirmed, that most reliable is the youngest sample in a series from the same depth.

It is important to note not only differences in the dates of fractions, but also different $\delta^{13}\text{C}$ values for the seed remains (–32.4‰) and of white twigs (–25.6‰), which demonstrates that the separated fractions have various origin, as the $\delta^{13}\text{C}$ depends on a type of a plant. Hence for dating by a conventional method in this cross-section the remains of herbs are the best material.

4 DISCUSSION

It is very difficult to determine the admixture of more ancient material. For adequate radiocarbon dating it is necessary to select for radiocarbon dating the organic material from the close systems. Syngenetic ice wedges are such close system. Pollen and spores in syngenetic ice wedges are isolated from any influence from environment changes in particular from microbes' activity. Pollen grains get into frost cracks together with snowmelt water in the late winter. The

regional pollen rain is forming mainly from pollen of flowering trees in neighbouring southern areas. At this time up to July the frost cracks are open and when the temperature becomes positive snow cover thaws and snow melt water get into frost cracks. Re-deposited pollen and spores get into frost cracks along with ground particles transported by wind on the surface of snow cover. Pollen concentrate from syngenetic ice wedges is relatively free from organic particles and well preserved. In the ice-wedge ice thin-wall pollen is preserved well.

The most ancient date of pollen in the ice wedge has been obtained in the top part of cross-section. The correlation between dates of pollen and re-deposited pollen and spores contents in spectra is obvious. To the youngest date corresponds to the minor content of re-deposited pollen and spores. The dates of microorganic remains (in our case this is insoluble in alkali an organic material more than 100 μ), their ages are the youngest. It is possible to make a conclusion, that dates of pollen concentrate are older as a result of pollution by “dead” carbon from contemporaneous palynomorphs of Pre-Quaternary age. We have used dependence of ^{14}C age from dead carbon admixture (Olsson 1974, 1991) to establish age of sample with participation of ancient carbon. If the fixed pollution by ancient pollen (or other carbon form) is about 20%, it is possible to suppose, that the mistake of measurements will be about 50%, i.e. if, as in our case, the top sample (363-YuV/27) with the high contents of re-deposited forms is dated 25 thousand years, its real age can be much younger and may be about 11–12 thousand years. It is evident that there is no possibility to use calibrated ages due to high concentration of ancient pollen and spores.

Very interesting results had been obtained in loess sediment of China (Zhou et al. 1997). Series of 6 AMS dates of pollen concentrate shows inversion of ^{14}C dates. The top sample at the depth 0.11 m dated 6950 ± 60 yr BP (AA-12317), the bottom sample at the depth 3.25 m – 11250 ± 80 yr BP (AA-12316). The sample at the depth 1.58 m dated 13260 ± 90 yr BP (AA-12315) contains re-deposited Pre-Quaternary pollen and spores. It is possible that the sediments accumulated in permafrost conditions as evidenced by distribution of ^{14}C dates. Kilian, van der Plicht et al. (2002) have done ^{14}C AMS dating of pollen concentrates from lacustrine sediments of Lake Gosciadz in Poland. The annual lamination of the sediments of Lake Gosciadz has been used as test for ^{14}C dating pollen concentrates. They have found that the pollen (almost all tree pollen) apparently could not be concentrated to the desired purity. The dating results were about 660 years “too old” because of admixtures from other organic remains or maybe because of allochthonous organic molecules, adsorbed to the pollen walls. A series of macrofossils (mainly single-year scales

from Pinus) yields much better results: these dates are about 100 years “too old”, however the error margins in absolute ages of the laminae. The results show that AMS dates of pollen concentrates may show a consistent inter-sample ^{14}C age, but 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. From the other side, Gillespie (1990) shows that pollen concentrates from lacustrine sediments of Lake Tirrel (Australia) become older after oxidation. The date of pollen concentrate after standard procedure is 7215 ± 270 yr BP (NZA-192), but after oxidation the age of the same sample is 7425 ± 445 yr BP (NZA-193). So, some difference between AMS dates of macrofossil and pollen concentrate in could be caused by difference between treatment of macrofossils and pollen concentrate in Killian et al. (2002). We have not used strong oxidation in order to compare number of dates from various fractions.

In Arctic Regions inversion of dates on different fractions of organic material, is more likely a rule than an exception. Radiocarbon dating of 5-m cross-section of horizontal layered well-sorted in Cumberland Peninsula Baffin Island N.W.T. Canada has shown essential admixture of ancient organic material, as the ^{14}C -inversion is more than 7 ka. As a result of the methodical study (Stuckenrath et al. 1979) it was possible to receive a number of dates without inversions only on rather large fraction of organic material, insoluble in alkali (>125 microns). Whereas dating on soluble in alkali fraction, has shown both younger and older age. These data also are very important for understanding of our results of AMS-dating of organic material from ice-wedge ice. Only microorganic remains (organic material, insoluble in alkali $>100 \mu$) gave the youngest and un-inversion number of dates.

The problem of permafrost sediments with allochthonous organic material was studied Nelson with the co-authors (Nelson et al. 1988) at exposure of Holocene sediments in Ipikpuk River valley in Alaska. For definition of pollution sources the allochthonous organic material of peat from a lens has been separated by different fractions and dated. The following number of dates was received: the fraction of peat more than 2 mm is dated 13250 ± 100 yr BP (USGS-2046C), fraction from 1 up to 2 mm 17730 ± 110 yr BP (USGS-2046B), fraction from 0.5 up to 1.0 mm 24740 ± 320 yr BP (USGS-2046C), fraction of 0.25–0.5 mm 30260 ± 530 yr BP (USGS-2046D), fraction less than 0.25 mm 20360 ± 190 yr BP (USGS-2046E). Dating of bulk sample of peat from the same layer is shown 13730 ± 110 yr BP (USGS-883). It may be concluded that the less sizes of fossils, the older dates received. At such disorder of ^{14}C dates

the most reliable represent the youngest dates. Pollen analysis results have shown, that in lenses of peat the contents re-deposited Pre-Quaternary pollen and the spores is about 50% (Nelson et al. 1988).

The differences in ^{14}C dates in permafrost sediments can be caused by effect of reservoir, when the plants use ancient CH_4 or CO_2 contained in permafrost or in the non-frozen ground. Some species of mushrooms which live on the root system of Ericaceae use ancient CH_4 especially active (Pancost et al. 2000). If Ericaceae meets among the plant remains in peat, probably dates the ^{14}C dates can be more ancient in dependence on methane age. The age CH_4 and CO_2 contained in permafrost was determined in lakes of Lower Kolyma valley in Northern Yakutia (Zimov et al. 1997). These measurements have shown that the age stratification of methane is kept even in lake silts. A number of ^{14}C dates without inversions from bottom upwards was obtained such as: 38, 27, 15, 11 and 8 kyr BP. Hence, the ancient methane can affect on radiocarbon age of upper lacustrine and bog permafrost sediments. The reservoir effect varies from lake to lake.

5 CONCLUSIONS

Pollen and spores are well-preserved in ice wedges; their concentration is enough for AMS dating ^{14}C dates of pollen concentrate. Pollen and spores extraction from ice-wedge ice required some modification of samples pretreatment. Relatively pure pollen concentrate enough for AMS ^{14}C dating has been obtained from 30 litres of melt water from ice using 40% HF pretreatment and centrifuging with heavy liquid.

It is obvious from our data of pollen concentrate from syngenetic ice wedges that the contents of pre-Quaternary pollen and spores usually can serve as an indicator of radiocarbon date reliability. The identification of Mesozoic and Paleozoic palynomorphs in sample gives the certain evidence that in a bulk sample could contain the significant amount of “dead” carbon. It is very important, that preliminary pollen analysis can be used to estimate reliability of ^{14}C dates before radiocarbon dating.

Due to good preservation of pollen in permafrost and inside permafrost and common re-deposition especially in permafrost areas it is very complicate to calibrate pollen AMS ^{14}C -dates. Fictive oldering of the ^{14}C dates may be determined by separation of contemporaneous pre-Quaternary pollen and spores in the sample. There is evident correlation between ^{14}C dates of pollen concentrate and re-deposited pollen and spores content. The youngest date corresponds to minimum of re-deposited pollen and spores.

^{14}C dating of pollen concentrate from syngenetic ice wedges allowed to evaluate palaeoreconstructions of vegetation.

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