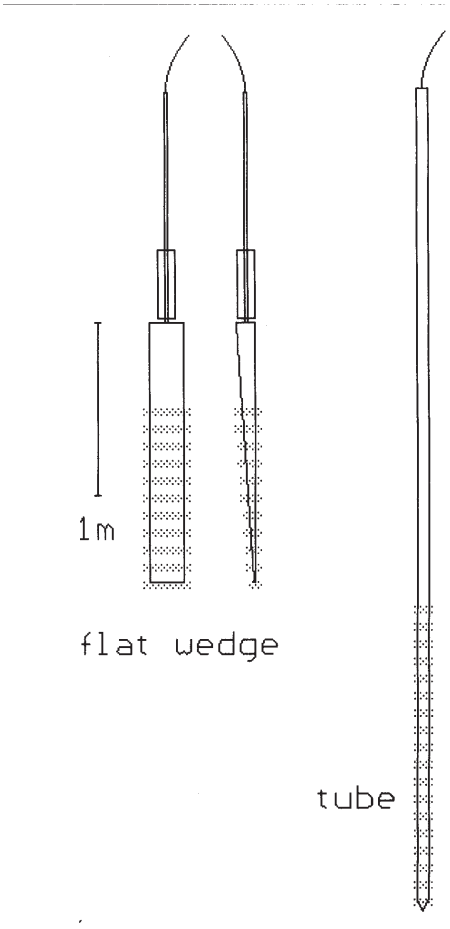

Fig. 4.1. Two types of freezing samplers.

and the age of samples are given by Goslar (Chapter 9.2.2).

4.1.3. Sediment subsampling

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In the case of annually laminated sediments the samples to be used for the majority of analyses (mineralogy, chemistry, stable isotopes, pollen, cladocera, diatoms, etc.) were collected from the same sediment slices, and their resolution followed the sediment laminations. Slices of a known number of laminae couplets were cut with a sharp knife or scalpel through the whole or half of the cleaned core. Laid flat, they were then divided into 1 cm^2 squares by pressing in a metal grid (Fig. 4.2). The full sediment squares were then used for the volumetric (influx) analyses. Routine sampling included 10 couplets, with 40 couplets left inbetween, but some sediments sections were sampled additionally for special purpose with higher time resolution, or continuously. The samples from non-laminated sections were collected in a similar way from slices 1 cm thick or with a 1 cm^3 volumetric device.

The samples were packed in plastic tubes and stored in a cold room. In profile T1/90 the halves of slices were packed in 25 cm^3 or 50 cm^3 samples for macrofossil analysis (Demske 1995).

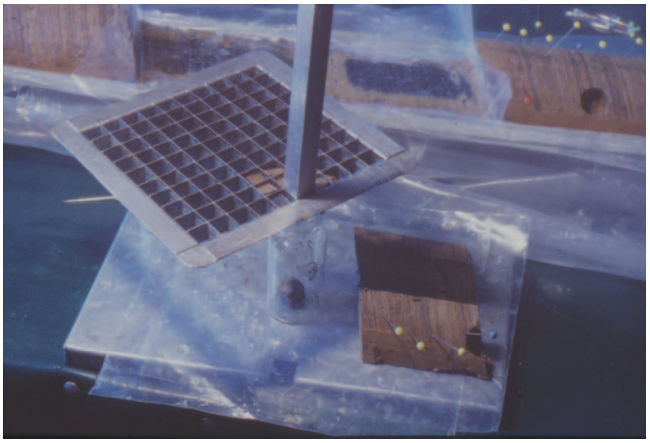

Fig. 4.2. Subsampling of laminated core section. The sediment slices of known number of yearly couplets are divided into 1 cm^2 samples to enable later calculation of microfossils influx values (Phot. T. Goslar).

The subsampling of frozen cores was done by two different methods, depending on device (tubular or wedge) used for freezing *in situ*. The subsamples from tubular cores were cut out with a scalpel from the core surface air-dried in a deep-freezer. This procedure, however, had two serious drawbacks. First, in order to get sufficient amount of dried sediment, the cores had to be stored in a freezer for several months. Besides, the freeze-dried sediment was very light and loose, and it was extremely difficult to handle it without any loss or mixing material from adjacent subsamples. The subsampling of cores collected with a wedge sampler was much easier, for two large flat pieces of sediment could be cut out and melted with no risk of collapse. The subsamples were then taken from melted surface of core pieces with a small brass trough. All subsamples were collected continuously, with a time resolution of 1–2 years, sometimes 3 years, depending on sampling difficulties. Their volume, attempted to be 1 cm^3 per year, appeared in practice not quite uniform.

4.2. CHRONOLOGICAL METHODS

Tomasz Goslar

4.2.1. Radiocarbon dating

Since its introduction (Libby 1946) radiocarbon dating has become well established and the generally accepted method of age determination of organogenic sediments. The discovery that the concentration of radiocarbon in the atmosphere (and hence in the biosphere) was not con-

stant over time (Suess 1970) led to the definition of the so-called "conventional radiocarbon age" (Stuiver & Polach 1977) which has to be distinguished from the "true" i.e. calendar age. The development of dendrochronology as well as the techniques of radiocarbon detection enabled the precise reconstruction of the dependence between conventional and calendar age (calibration curve) for nearly the whole Holocene (see Calibration 1993).

To avoid confusion, the ages quoted with respect to radiocarbon or calendar time scales must be precisely distinguished. Most of ages quoted in this book are calendar ages. They are expressed in years AD (Anno Domini), BC (before Christ), cal BP (before present, e.g. 1000 cal BP = AD 950), or sometimes in kyr BP (1 kyr BP = 1000 cal BP). The conventional radiocarbon ages are usually expressed as ^{14}C BP. The notation "BP" was avoided, but its use in some graphs was clearly stated each time.

An important consequence of natural ^{14}C variations is that the error of a calendar age determined after calibration of a radiocarbon date depends not only on the laboratory precision but also on the shape of calibration curve (Aitchinson et al. 1989, Michczyńska et al. 1990). For example, the age of a sample descending from the period of plateau of the calibration curve cannot be determined by the radiocarbon method with an error less than the length of the plateau.

Conventional ^{14}C dating of bulk samples

The bulk samples of peat and gyttja were dated at the Gliwice Radiocarbon Laboratory, Silesian Technical University. The peat samples were treated with 4% HCl at 80°C overnight. The samples of carbonate-rich gyttja were treated with 4% HCl, and the CO_2 from the carbonate fraction was collected. The CO_2 from the organic fraction was produced by combustion in a stream of oxygen. The specific activity of ^{14}C was measured in the CO_2-filled proportional counters. One serious complication in dating bulk samples from lacustrine environments is that the ^{14}C concentration in precipitating carbonates and living lacustrine organisms is usually lower than that in the contemporaneous atmosphere, so the ages of bulk lacustrine samples must always be interpreted with a great caution.

AMS dating of terrestrial macrofossils

The most suitable materials for dating lake sediments are macrofossils from terrestrial plants. Dates free of hard water (or reservoir) effect (Olsson 1986) obtained on short-living parts of land plants incorporated into sediments provide the best estimate of the radiocarbon age for the time when they were deposited.

Prior to the collection of macrofossils the sediment carbonate was dissolved in HCl. The residual fraction was then suspended in distilled water, and the macrofossils were hand-picked from the suspension, botanically identified, and stored in hermetically closed small vials with some water. The majority of samples were dated in the Centre des Faibles Radioactivites, CNRS-CEA, in Gif-sur-Yvette, France. In pretreatment, the standard AAA method (e.g. Stuiver & Pearson 1986) was used. The samples were then combusted, and the CO_2 produced was converted to graphite and deposited on an iron powder. The capsules of iron+carbon were used as targets in the ion source of the tandem accelerator. The details of Gif-sur-Yvette AMS facility were given by Arnold et al. (1989). Some samples were dated in the ETH Zürich, Switzerland (Hajdas et al. 1995).

4.2.2. Varve counting

During the varve counting several techniques were used. The tape-peel technique (Simola 1977, see also Goslar, Chapter 9.2.2) was used for the cores taken by freezing *in situ* (see Walanus, Chapter 4.1.2) from the uppermost 2.5 m of sediment. The thin-section method (Merkt 1971) was used to recognize the basic varve structures for the whole profile. In most of Holocene section the very regular character of laminations enabled counting and measuring the thickness of varves directly on the core surface and/or on good-quality photo-negatives with a low-magnification binocular (4–10x) and the equipment used in tree-ring width measurements (Goslar 1987). In the whole Late-Glacial part of the profile, where the lamination was less regular, the varves were counted on thin sections under the microscope. In parts of the profile above ca. 8 m, where the varves were often bent and in many fragments hardly visible, the conventional X-ray radiography was applied (Koivisto & Saarnisto 1978).

The varves in all available cores were correlated with one another, using the photographs of the core surface. The correlation is precise in the sense that for each varve in a given core the corresponding (i.e. deposited in the same year) one has been found in other cores. In this correlation, characteristic patterns of laminae brightness and thickness were sought. The characteristic varves were marked, and the number of varves counted between corresponding pairs of markers had to be the same in all cores. Such a correlation was made for the entire profile. This method enabled filling the gaps and overcoming disturbances of laminated sequences in one core by using other cores and counting many varves which, while not quite clear in one core, were well developed in other cores. Finally, correlation of cores coming from different parts of the lake enabled us to find and exclude the missing and/or duplicated fragments in the continuous laminated sequences. The details are given in the forthcoming chapters.