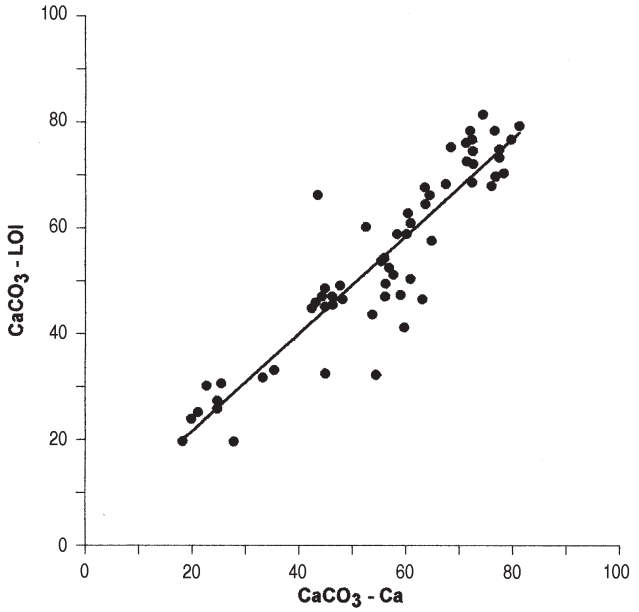

Fig. 4.3. Comparison of calcite content calculated from LOI and from Ca concentration. r = 0.92, y = 6.21 + 0.96·x.

tion analyses. Other bulk samples were taken into solution by the mixture of nitric and hydrofluoric acids. For samples treated by the latter procedure, major elements (Ca, Mg, Mn, Fe, Al, Ti) and minor ones (Zn, Cu, Cr, Ni, Sr, and Ba) were determined by means of a Perkin Elmer atomic absorption spectrometer (AAS). Cr, Ni, and Cu concentrations were close to the detection limits of these elements in the method applied: Cr ≤10 ppm, Ni≤ 10 ppm, Cu≤ 5 ppm. The fluorescence caused by high concentration of Mn prevented the precise determination of Ti content. The concentrations of K and Na were analysed by optical flame emission spectrophotometry.

For determination of biogenic SiO_2,, separate portions of the sediments were dissolved in 5% Na_2CO_3 solution and analyzed by the colorimetric method. Concentrations of P and total S were analyzed also from separate portions of the sediment after their dissolution with *aqua regia*. S and P contents were determined by gravimetric methods.

Total carbon content was determined with a LECO carbon analyser, and the content of organic carbon was estimated from the difference between the total carbon and carbonate carbon concentration calculated from the TG curves. But, as mentioned above, the carbonate carbon concentrations probably are correct only for samples poor in Mn and Fe.

For 25 samples the analysis of organic material was performed on the Perkin Elmer elemental CHN analyser.

Similar analytical procedures were used for element-content determination in 1M HCl dissolved sediment fraction. Mg, Mn, Fe, Sr, Cu, Zn, Ni, and Al concentrations were determined by AAS, and sulphate-sulphur by the gravimetric method. Because of the rather high Ca content within all samples, the EDTA titration method for its determination was applied.

4.6. PALAEOBOTANICAL ANALYSES

4.6.1. Palynological analysis (pollen and extra-palynomorphs)

Magdalena Ralska-Jasiewiczowa & Bas van Geel

The samples used for analysis of pollen and other plant microfossils were of two different volumes: samples of 1 cm^2 per known number of laminae couplets in the case of annually laminated sediments, or samples of 1 cm^3 volume in case of other sediment types (see Ralska-Jasiewiczowa et al., Chapter 4.1.3).

The reference profile G1/87 was mostly subsampled with the routine 50 yr time resolution, with samples embracing 10 couplets, except for the bottom part, where samples of 6-couplets were collected and analysed with a denser time resolution, and the upper 6 m, where the subsampling intervals were not quite regular due to the lamination disturbances. At the contacts between subsequent sediment segments (of 2 m), where the continuity of lamination was slightly destroyed, the subsampling was completed from a twin core G2/87. This core was divided into segments at different depths and correlated precisely with G1/87 by "year-to-year" laminae analysis (Goslar, Chapter 6.1).

The other profiles were subsampled with 10-couplets samples but with varying time resolution for laminated sediments (G1/90, T1/90 – Late-Glacial part), and for non-laminated sediments with 1 cm^3 samples, either continuously (G1/90-bottom part), or in intervals of 10 cm, 5 cm, and smaller intervals (G28/92 and T1/90 – Holocene part).

The chemical preparation of samples was based on the Faegri & Iversen (1975) acetolysis method with two different ways of pre-treatment: gravity separation of organic and mineral matter by heavy-liquid mixture of bromoform and alcohol (specific gravity 2) was applied to the samples from G1/87 profile prepared at the Hugo de Vries Laboratory of the Amsterdam University; the samples from all other profiles prepared at the Palaeobotanical Department of the Institute of Botany in Cracow were pre-treated with cool HF and HCl to remove mineral matter and carbonates.

One to four *Lycopodium* pellets containing 13,500; 12,077; 11,300±400 or 10,850±200 *Lycopodium* spores were added to all samples to enable the calculation of pollen concentration according to the method of Stockmarr (1971, 1973). The samples were embedded in pure glycerine, and no staining was applied.

The pollen counts of samples from profile G1/87 and from the frozen cores (GF) were performed by two persons separately on each sample using the same sample

material. M. Ralska-Jasiewiczowa counted pollen sums from over 500 P (in Late-Glacial spectra) to 2000 P and rarely more, on average around 1000 P per sample. B. van Geel counted mostly 300–500 P and all the non-pollen microfossils found on the same slide surface. The pollen counts were then summed up in each sample.

This procedure had some advantages, e.g. possibilities to compare the qualitative and quantitative composition of pollen spectra generated from the same material by two different pollen analysts, and then to check possible differences once again; to compare the composition of pollen spectra when based on different pollen sums; and to get a pollen diagram based on a P-sum higher than commonly produced.

Some difficulties were caused by the sometimes different approaches of the two analysts to pollen identification, taxonomy, and nomenclature. When possible, such identifications were then checked and harmonized. In rare cases when this was impossible, different pollen curves representing counts of the two analysts within the same taxonomic group appear in the diagram (e.g. *Secale cereale* and other cereal types, and *Secale*/Cerealia pollen curves in the GF profile). An attempt was made to adopt the recommendations of the European Pollen Database (Gaillard et al. msc.) concerning nomenclature of pollen taxa, but this was not always possible.

The calculation of the pollen-analytic results and construction of pollen-percentage and pollen-influx diagrams follow generally the recommendations proposed by Berglund and Ralska-Jasiewiczowa (1986). All diagrams except for the Late-Glacial G28/92 profile and the bottom part of T1/90 profile are based on calendar time scales. The data obtained by M. Ralska-Jasiewiczowa and B. van Geel were handled by A. Walanus and T. Goslar, and most pollen diagrams were drawn using the POLPAL-for Windows program (Walanus 1995, Walanus & Nalepka 1996). Some diagrams were produced by D. Demske with the plotting program TILIA-GRAPH version 1.25 (Grimm 1992), and the zonation of the diagrams was supported by the CONISS cluster analysis belonging to the TILIA program.

4.6.2. Plant-macrofossil analysis

Dieter Demske

Samples of 25 or 50 cm^3 were dispersed in water and washed on a sieve with 0.2 mm mesh. In case of calcareous material samples were pre-treated with 10% HCl. After segregation in water, the material was preserved in a mixture of water, alcohol and glycerine 1:1:1 with addition of thymol (Wasylikowa 1986). After the taxa were determined the data were recalculated for a standard volume of 25 cm^3. The diagram was plotted with the TILIA-GRAPH program.

4.7. CLADOCERA ANALYSIS

Krystyna Szeroczyńska

The sediments of cores G1/87 and G1/90 were sampled as described in Chapter 4.1.3. The Late-Glacial part was sampled every 6 varves. In the Holocene part sampled with the time resolution of 50 years each sample includes 10 varves. The core T1/90 was sampled in cubic centimetres. Samples used for analysis of cladoceran remains were treated with a few drops of 4% formalin and stored in a refrigerator to prevent the growth of bacteria and fungi. The cladoceran remains were prepared according to Frey's method (1986a, b). Each sample was boiled for half an hour in a 10% solution of KOH to remove humic matter after elimination of carbonates by HCl. The residue was washed with distilled water over a 50 μm sieve. The final residue was filled up to 15 cm^3 with distilled water, containing a few drops of formalin and safranin. For preparation of microscope slides 0.05 ml of this solution was used. All skeletal parts were counted: headshields, shells, postabdomen, postabdominal claws, antennules, antennal segments, mandibles, and ephippia. 3–7 slides were counted from each sample, depending on the abundance of remains. When a sample was very rich in remains and frequency exceeded 500 specimens, only two slides were examined, and the average result calculated. The results of qualitative and quantitative analyses are presented in a percentage diagram and in a concentration diagram, in which for each sample an absolute number of specimens was calculated for 1 cm^3 of sediment. The next stages of research were based on the number of specimens recorded. Sources by Flössner (1972) and Smirnov (1971, 1978) and the systematical analyses by Frey (1958, 1959, 1962, 1980) and Goulden (1964a, b) were used to determine the species found.

The method as described above was applied to samples from all cores to enable comparison.

4.8. DIATOM ANALYSIS

Barbara Marciniak

The samples for diatom analysis were macerated according to methods applied to sediments rich in calcium carbonate and organic substances (Siemińska 1964). 1000 diatoms were counted in each sample, and the diagram showing percent content (i.e. relative frequency) of the dominant and subdominant diatoms as well as less frequent ones was plotted. The groups of euplanktonic, littoral-planktonic, and periphytic diatoms were presented in a separate diagram. The diagrams were plotted with a computer program (POLPAL) elaborated earlier for the purposes of palynological studies (Ralska-Jasiewiczowa & Walanus 1989, 1991, Walanus 1989) and then adapted for diatom analysis by A. Walanus.