

### 4.3. ANALYSIS OF MAIN SEDIMENT COMPONENTS

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For quantitative analysis of the main sediment components along the whole profile a few simple standard methods were used. These methods provide proxies rather than direct estimates for the concentrations of particular components. Nevertheless, they were useful for some interpretations given in subsequent chapters.

As the proxy for organic matter content, loss on ignition (LOI) was used. Loss on ignition is defined as a loss of sample mass after heating to 550°C, with respect to the mass of sample dried to 120°C. The content of calcium carbonate was determined by simple Scheibler method. Such an approach seems justified as the carbonates in the Lake Gościąg sediments consist almost entirely of calcite (Łącka et al., Chapters 7.3 and 8.2). The amount of iron in the sediments was measured by the iodometric method, and it was displayed in the form of Fe<sub>2</sub>O<sub>3</sub> content.

### 4.4. STABLE ISOTOPE ANALYSIS

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The samples of the core material 0.5 to 1.0 cm<sup>3</sup> in volume collected for stable-isotope analyses were sealed in plastic vials and transported to the laboratory, where they were dried (12 hours, +90°C), ground to a fine powder, homogenized, and stored in tightly closed plastic vials. A portion between 60 and 120 mg was used for individual analysis, depending on carbonate content in a given core. Chosen samples were analysed twice. Decomposition of carbonates to gaseous CO<sub>2</sub> and phosphates for isotopic measurement was done by the standard procedure (McCrea 1950) at controlled temperature and pressure. The analyses were performed using the Micromass 602C mass spectrometer equipped with computer data output.

The results are reported as per mille deviations of <sup>18</sup>O/<sup>16</sup>O (<sup>13</sup>C/<sup>12</sup>C) isotopic ratios in the analysed carbonates from those in the PDB carbonate standard, following the generally accepted notation (Craig 1957):

$$\delta^{18}\text{O} = \left\{ \frac{(^{18}\text{O}/^{16}\text{O})_{\text{sample}}}{(^{18}\text{O}/^{16}\text{O})_{\text{standard}}} - 1 \right\} \times 1000\text{‰}$$

$\delta^{13}\text{C}$  – analogous definition.

The overall analytical uncertainty of the isotope analyses (one sigma for single measurement) was around 0.2‰ for  $\delta^{18}\text{O}$  and 0.1‰ for  $\delta^{13}\text{C}$ .

### 4.5. MINERALOGICAL AND GEOCHEMICAL METHODS

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Mineral composition of sediment samples was determined by X-ray diffraction (XRD) and differential thermal analysis (DTA). The morphology and chemical composition of individual mineral phases as well as the variation in the structure and composition of the varve sequences were investigated with scanning electron microscope (SEM) and energy dispersive spectrometry (EDS).

Every sample was air-dried and ground in an agate mortar to 200 mesh size for XRD, DTA, and chemical analysis.

The diffractograms of untreated samples were recorded by the reflection method, using quartz-filtered CuK $\alpha$  radiation (Sigma 2040). For the very small samples, the XRD transmission method was applied with the INEL position goniometer. In both cases the INEL computer registration programs were used. The fluorescence from high concentration of amorphous Mn and Fe hydroxides excluded the detection of minor and poorly crystallized mineral components of the sediments.

DTA was performed in air on 200 mg samples with Al<sub>2</sub>O<sub>3</sub> as a standard (MOM, type Q1500D). The samples were heated 10°C/min. up to 1000°C. The sensitivity of TG determination was 10 mg over the whole temperature range. In addition to mineral identification, the carbonate carbon was determined from the weight loss between 550 and 1000°C (the temperature range of carbonate decomposition). CO<sub>2</sub> content recorded in this way was recalculated to determine semiquantitative content of carbonate minerals. However, for the Mn-rich samples the CO<sub>2</sub> concentrations were too low even to fix the total Ca with calcite. This incompatibility of the Ca and CO<sub>2</sub> content is probably caused by the gain in weight due to the oxidation of Mn and Fe species during the ignition. Comparison of the calcite content calculated from loss on ignition between 550 and 1000°C (Wicik, Chapter 3.4) and from the Ca content (Fig. 4.3) shows that the first values are slightly lower than those calculated from total Ca content reduced by the value corresponding to the gypsum content in sediments.

The samples for SEM examination were air-dried, and damage of delicate sediment structure as well as of neoformed minerals could have taken place. In all cases, the chipped fragments of the core were mounted perpendicularly to the varve succession and coated with carbon and then with gold. SEM examination was performed with JSM 840A microscope (JEOL). The qualitative chemical analyses of minerals and varves were carried out by AN 10000/85S EDS (Link) in the 2000  $\mu\text{m}^2$  microarea and, in special cases, in the spot mode.

Some of samples were chosen for acid-dissolved frac-