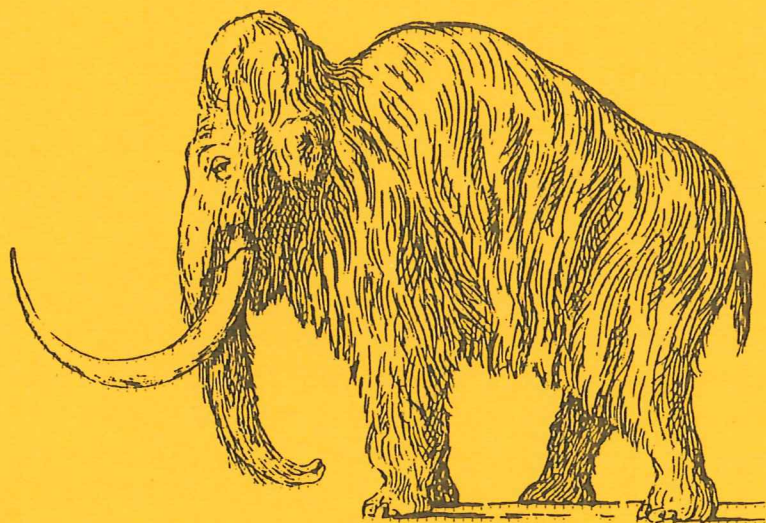


# EXAMENSARBETE I GEOLOGI VID LUNDS UNIVERSITET

Kvartärgeologi

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A comparative study between loss on ignition and  
total carbon analysis on Late Glacial sediments from  
Atteköps mosse, southwestern Sweden, and their tentative  
correlation with the GRIP event stratigraphy

Daniel-Stefan Veres

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Lunds univ. Geobiblioteket



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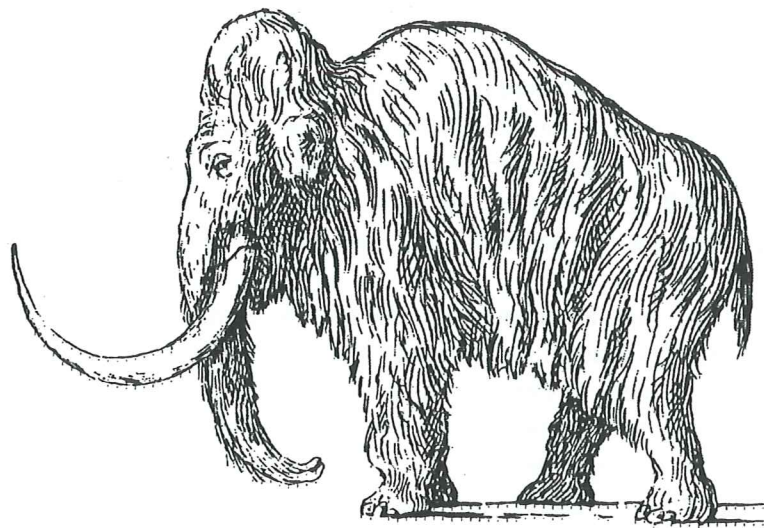
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Geologiska Institutionen, Lunds Universitet

Nr 145

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**A comparative study between loss on ignition and  
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**Daniel-Stefan Veres**

**Veres, Daniel-Stefan, 2001: A comparative study between loss on ignition and total carbon analysis on Late Glacial sediments from Atteköps mosse, southwestern Sweden and their tentative correlation with the GRIP event stratigraphy. *Examensarbete i Geologi vid universitet-Kvartärgeologi, nr 145. 28 pp.***

In this study, a high resolution loss on ignition analysis was performed over the Late Weichselian lacustrine sediments retrieved from Atteköps mosse, southwestern Sweden. The data set was complemented by grain-size measurements, total carbon analysis, magnetic susceptibility and AMS <sup>14</sup>C dates.

The analyzed data set reveals a characteristic pattern in lithology and the loss on ignition curve, where several distinct minerogenic units are overlain by organic rich horizons.

In order to derive a chronology for the studied sediments, the down-core fluctuations in loss on ignition were compared to earlier analysed, close-by sites on Kullen Peninsula, and tentatively to the GRIP event stratigraphy. These correlations imply that sediments started to accumulate during late GS-2, that the studied sequence extends up to the early holocene and that all major Late Glacial climate fluctuations are represented. Based on the investigated parameters, changes in the lake ecosystem, as response to the climate fluctuations are discussed.

Although the absolute values of loss on ignition are subject to several uncertainties, relative down-core changes are useful in interpreting the impact of climatic changes through the sediment sequence. However, if absolute organic determinations are to be made, loss on ignition data must be supported by independent total carbon determination. Carbonate content determination by means of loss on ignition seems to be strongly influenced by fine sediment fractions. Care must therefore be taken when the "carbonate content" via loss on ignition is interpreted in terms of a palaeoenvironmental indicator.

*Keywords:* Atteköps mosse, limnic sediments, Late Glacial, loss on ignition, total carbon.

**Veres, Daniel-Stefan, 2001: En jämförande undersökning av glödförlust- och totalkolanalys av sen-glaciala sediment från Atteköps mosse, sydvästra Sverige, och en möjlig korrelation med GRIP-händelsestratigrafin. Examensarbete i Geologi vid Lunds universitet – Kvartärgeologi, nr 145. 28 pp.**

### **Sammanfattning.**

I denna undersökning har en stratigrafiskt högupplösande glödförluststudie utförts på sen-glaciala och tidigholocena sjösediment från Atteköps mosse i nordvästra Skåne. Studien har kompletterats med kornstorleksanalyser, kolanalyser, mätningar av magnetisk susceptibilitet och AMS  $^{14}\text{C}$ -dateringar.

Undersökningen påvisar ett karaktäristiskt mönster i sedimentens sammansättning och i glödförlustkurvan, på så sätt att ett flertal minerogena enheter överlagras av horisonter rika på organiskt material. För att upprätta en kronologi över de studerade sedimenten har glödförlustkurvan jämförts med motsvarande kurva för en tidigare studerad och närbelägen lokal på Kullaberg. Glödförlustkurvan har dessutom hypotetiskt korrelerats med den grönländska GRIP-iskärnans händelsestratigrafi. Dessa korreleringar visar att sedimenten i Atteköps mosse började avsättas under en sen fas av GS-2 under sen-glacial tid, och att den undersökta delen av lagerföljden tidsmässigt sträcker sig en bit in i tidigholocen tid.

Undersökningen visar också att alla mera omfattande sen-glaciala klimatfluktuationer finns representerade i lagerföljden. Slutligen diskuteras de sen-glaciala klimatfluktuationernas påverkan på fornsjöns ekosystem.

**Veres, Daniel-Stefan, 2001: A comparative study between loss on ignition and total carbon analysis on Late Glacial sediments from Atteköps mosse, southwestern Sweden and their tentative correlation with the GRIP event stratigraphy. *Examensarbete i Geologi vid Lunds universitet-Kvartärgeologi, nr 145. 28 pp.***

### **Popular abstract**

In this study, several sediment core from Atteköps mosse, southwestern Sweden, were investigated with respect to lithostratigraphy, loss on ignition analysis, grain size distribution, magnetic susceptibility, total carbon analysis and  $^{14}\text{C}$  dating.

The first part of the present study foccuses on a detailed analysis of the above mentioned sediment parameters. The results revealed a characteristic pattern with several dominantly minerogenic units separated by units with high organic content. These alternations very likely reflect changing climatic conditions and their impact on the former lake and its catchment.

The available radiocarbon dates, comparisons with an earlier, unpublished investigation of Atteköps mosse and correlations with other published sites from southwestern Sweden imply that the investigated sequence was deposited during the Late Glacial and the early Holocene. By correlating the distinct lithological changes to the GRIP event stratigraphy, and assuming that climatic changes in southern Sweden and on Greenland occurred more or less synchronous, a tentative chronology for the sequence in Atteköps mosse could be established. Based on the chronological framework, the Late Glacial and early Holocene environmental development of the site is discussed.

The second part of the deals with a comparison between organic and carbonate content determinations based on loss on ignition and step-wise total carbon analysis, and a correlation to grain-size.

Although loss on ignition can generally be regarded as an accurate measure of the organic matter content of a sediment, the amount of the fine fraction in the sediments is a limiting factor for an absolute organic carbon determination and influences directly the correction factor. The same holds true for carbonate content determinations based on loss on ignition.

In order to provide accurate organic and carbonate content determinations for a sediment, loss on ignition data must corroborated by standard total carbon analysis.

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# 1 Introduction

The Last Glacial to Holocene transition, (i.e. the last deglaciation), was a complex period during which pronounced but relatively short-lived climatic oscillations occurred. By means of sedimentological methods and temperature sensitive proxies in ice-cores, terrestrial and marine sediments (isotopes, micro- and macrofossils records, various lithological variables), several more or less pronounced climatic oscillations have been identified during this time interval. Important aspects concerning these climatic variables are well known from sites located in northern Europe, particularly Sweden. This period has been the object of a great deal of palaeoclimatic studies over the past decades. Well performed palaeoclimatic investigations based on fossil biological spectra (e.g. Björck & Möller, 1987; Liedberg Jönsson, 1988; Berglund *et al.*, 1994; Andresen *et al.*, 2000) and non-biological proxies (Hammarlund, 1994; Hammarlund *et al.*, 1997) on limnic sediments in southern Sweden, have allowed reconstructing the impact of the climatic changes which characterized the Late Glacial, in both terrestrial and limnic ecosystems.

North-western Skåne and in particular, Hallandsåsen, where Atteköps mosse is located (Fig. 1a), are one of the first ice-free areas in Sweden. Preliminary investigations at Atteköps mosse by Liedberg Jönsson (1988) showed the potential of the site to provide a high resolution sedimentary record starting at around 14,000-15,000 cal yr BP. This is in good agreement with recently presented data from Kullen Peninsula (situated fairly close to Hallandsåsen, and at approximatively the same elevation), where the deglaciation started ~ 17,200 cal yr BP (Sandgren *et al.*, 1999) coincident with the beginning of organic sedimentation at the GS-2/GI-1 transition as a response to the climatic

amelioration, which characterized this time interval.

## 1.1 Aims

In this study, a high resolution loss on ignition analysis was performed over the presumably Late Glacial sediments retrieved from Atteköps mosses, south-western Sweden (Fig. 1b). During the Last Glacial Maximum, the region was covered by the Scandinavian ice-sheet. Around 13,000 <sup>14</sup>C yr BP, the first warm Atlantic waters likely started to penetrate further north, warming up the continent. The only areas in Sweden, which became deglaciated that early are Kullen Peninsula and this area, the western part of the so-called Hallandsåsen.

The first aim of the present study was, therefore, to obtain cores from the deepest part of the basin, to establish a detailed lithostratigraphy and loss on ignition curve, which would make it possible to correlate the sequence to the earlier analysed core (by Liedberg Jönsson) and, as a next step, to suggest a tentative correlation to the GRIP event stratigraphy as proposed by Björck *et al.* (1998).

The second aim of this study was to assess the usefulness of the loss on ignition method in estimating the organic content in minerogenic sediments. The assumption is that the method over-estimates the organic matter and carbonate content of fine-grained sediments because of a weight loss related to various organic and inorganic sediment components. Total carbon analysis with a specific carbon analyser is generally viewed to be a better indicator for those parameters. To test this hypothesis, conventional loss on ignition analysis, total carbon and grain-size measurements were performed over selected sediment intervals and compared with each other.

## 2 Framework

### 2.1 Late Glacial to Holocene environment

The climate shift at the end of the Last Glaciation was driven by an important set of feedback mechanisms involving linkages between ice, ocean, land and changes in greenhouse gas concentration. All of these indirectly controlled other elements of the environment (Roberts, 1998).

The deglaciation of southern Sweden was an unsteady process during which, at several times, more or less pronounced ice-readvances occurred (see Lundqvist & Wohlfarth, 2001). Southern Sweden and in particular, north-western Skåne, where the study site lies, became ice free during the Late Weichselian, at around 17,600-16,000 cal yr BP (Lundqvist & Wohlfarth, 2001).

The GI-1 event (Björck *et al.*, 1998) interpreted as a period of relatively warm temperatures (Liedberg Jönsson, 1988; Berglund *et al.*, 1994) was interrupted by at least three short-lived more or less cool events (Hammarlund, 1994; Andresen *et al.*, 2000). These are superimposed on an overall cooling trend through the later part of GI-1 (e.g. Liedberg Jönsson, 1988; Hammarlund *et al.*, 1997), leading gradually to GS-1 and caused advances or stillstands of the ice-sheet (Lundqvist & Wohlfarth, 2001).

A major episode of sudden climatic cooling, at about 12,650 cal yr BP, is considered to be the beginning of the GS-1 stadial which lasted until ~11,500 cal yr BP. This c. 1000 year long cold phase may have been caused by a slow-down of the thermohaline circulation (Björck *et al.*, 1996). According to Rensen *et al.* (1996), who used simulations with atmospheric general circulation models to reconstruct former annual temperatures, the extended sea-ice cover in the North Atlantic and the presence of the Scandinavian ice-sheet, determined changes in the seasonal wind directions and had a large cooling effect on the surroundings. This is

shown in detail by various proxies data inferred from terrestrial (Björck & Möller, 1987; Berglund *et al.*, 1994), or marine (Jiang & Klingberg, 1996; Sejrup *et al.*, 2001) evidences around Sweden.

The warming at the beginning of the Holocene is recorded in numerous archives around the North Atlantic as an almost instantaneous environmental change (Björck *et al.*, 1996).

However, short-term cooling episodes have been reported, e.g. the Preboreal Oscillation (Björck *et al.*, 1997). This event is seen in many records (Jiang & Klingberg, 1996; Björck *et al.*, 1997; J.Björk & Wastergård 1999; Hammarlund *et al.*, 1997; Sejrup *et al.*, 2001) and marks the transition to interstadial conditions in the early Holocene.

Secular climatic variations of smaller amplitude and shorter duration during the Holocene have continued up to the present day (Snowball & Sandgren, 1996; Nesje *et al.*, 2001).

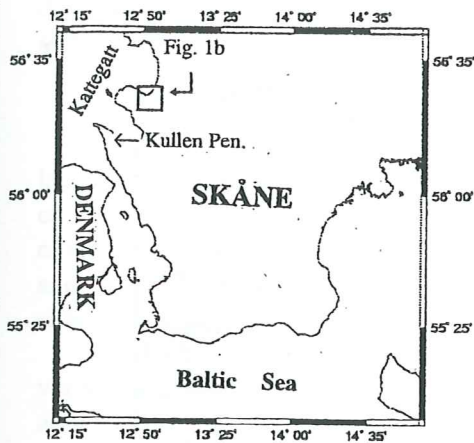
### 2.2 Present environment

North-western Skåne, is a relatively forested area and lies ca. 150-200 m above the highest shoreline. Atteköps mosse is located at 180 m a.s.l (Fig.1b) and the surrounding soils are mainly till derived. The landscape is composed of hills and peat-bogs, and human impact is considerable.

The geographical position of southern Scandinavia at present involves frequent shifts of oceanic and continental air-masses (Hammarlund, 1994). The average July temperature in the study area is between 15-16 °C, while the mean January temperature is about -1 °C. The annual average precipitation is around 1000-1100 l/m<sup>2</sup>, with a seasonal distribution mainly during the summer and autumn. The vegetation length is in order of 200-220 days/yr. The dominant wind directions are from V-SV (National Atlas of Sweden, 1995).

The study area is located in the southern part of the Swedish Granulite region, precisely in the eastern segment of the Sveconorwegian orogen.





**Figure 1a** Map of southern most part of Sweden with location of the study site (square) in north-western part of the province of Skåne.

Geologically, the region is characterised by a dominance of metamorphic and magmatic basement rocks.

The dominant rocks within the region are calc-alkaline orthogneisses (with an age of 1.70-1.66 Ga; Wang, 1996), penetratively deformed by dolerite dykes. Due to a long time exposure under exogenic processes, the

bedrock surface has been levelled into an erosion plateau, the South Swedish Peneplain, with an altitude of 125-175 m. The tectonic movements of the Törnquist tectonic zone, with faults which strike in NW-SE direction, implied for the bedrock in north-western Skåne, a present geomorphologic feature of horst and graben relief-like.

Pleistocene deposits consist mainly of glacial derived sediments (clayey till, sandy till and coarse grained sediments) but some isolated glaciolacustrine sediments occur as well. The till thickness varies between 5-20 m, but it is in some areas susceptible to erosion, with frequent bedrock exposures. The postglacial deposits are represented by scattered fluvial sediments (along the valleys) and lacustrine sediments. The organic deposits are rather well developed, especially where the topography and hydrological features support a wet place. These organic deposits (if not exploited) are mainly developed as peat-bogs (National Atlas of Sweden, 1994).

From a biological point of view, the region belongs to the nemoral zone. The present vegetation cover consists of an association of deciduous species and spruce, the latter one probably favoured by forestry (National Atlas of Sweden, 1996).

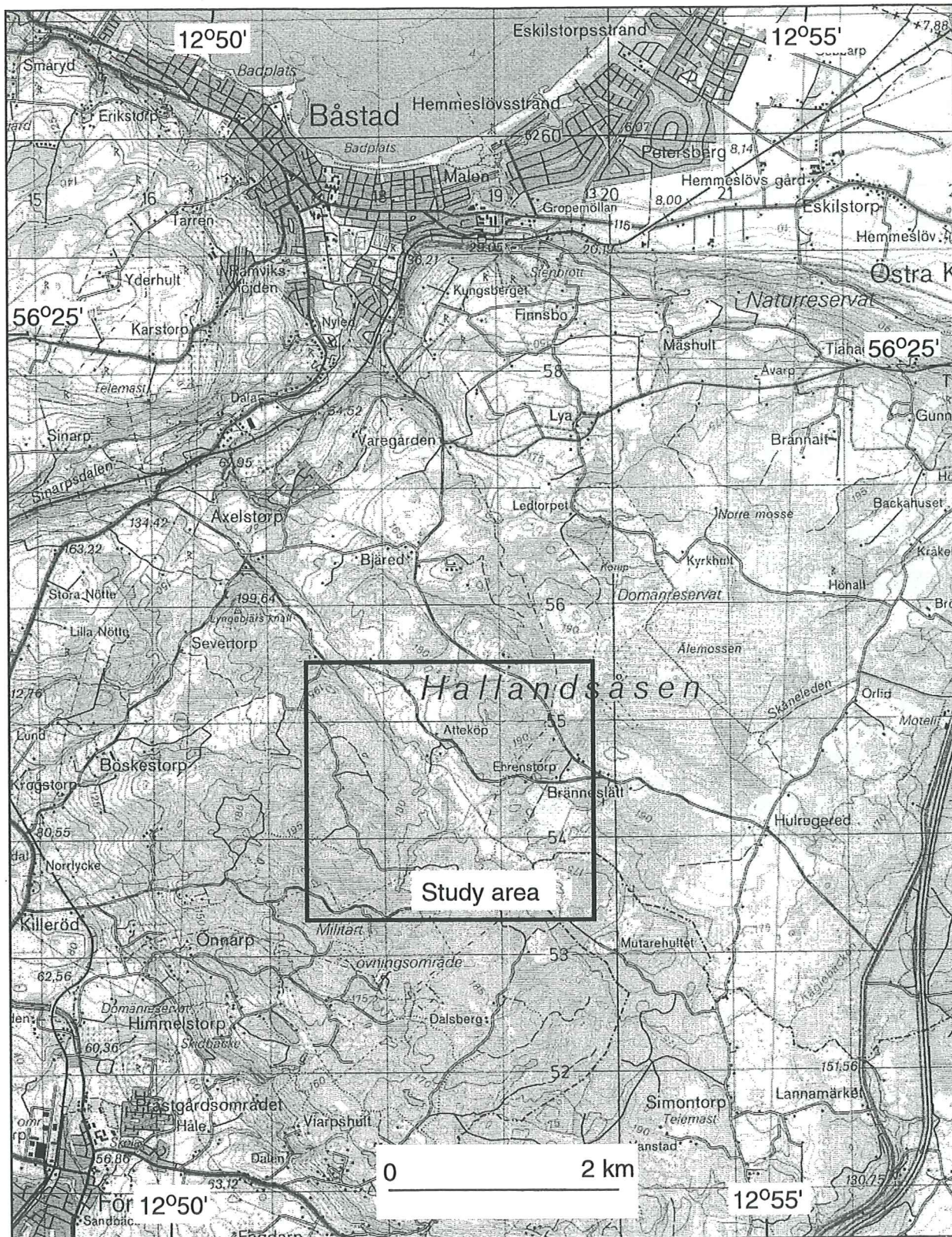


Figure 1b. Topographic map of the Hallandsåsen region and location of the investigated area.

## 3 Theoretical background

### 3.1 Lacustrine sediments as environmental archives

The sedimentological properties of lacustrine sediments can provide valuable data on the nature of former depositional environments, being useful indices of climatic and other local and regional environmental changes (Lowe & Walker, 1997).

Usually, lake sediments consist of variable amounts of autochthonous and allochthonous organic debris, clastic detritus and chemical precipitates. The relative proportion of each other depend upon combined effects of extrinsic and intrinsic factors on hydrological and limnological processes.

Lacustrine biogenic sediments comprise primarily particulate organic carbon and are mostly autochthonous in origin, indicating the level of biological productivity within lake ecosystems. However, some dissolved organic carbon may be incorporated in sediments by adsorption on clays and carbonates (Dean, 1999). Organic petrography on such sediment records may reflect the type of organic matter, its origin and its mode of transport to the sedimentary basin (Meyers & Lallier-Vergés, 1999). Typically, there is a distinction between sources of organic matter productivity based on carbon isotope composition (see Hammarlund, 1994; Hammarlund *et al.*, 1997), whereas microbial reworking of organic matter during early diagenesis can potentially modify its bulk carbon isotopic content (Meyers & Lallier-Vergés, 1999). Important palaeoecological information about origins and palaeoproductivity of sediment organic matter and also of palaeoprecipitation rates may be recognisable from the nitrogen, oxygen and hydrogen isotope content of organic matter (Hammarlund, 1994; Meyers & Lallier-Vergés, 1999).

Biogenic sediments typically contain abundant micro- and macrofossils, being therefore used in a variety of palaeoecological investigations (see e.g. Björck & Möller, 1987;

Liedberg Jönsson, 1988; Coope & Lemdahl, 1995).

The proportion of clastic material in lake sediments varies by a virtue of factors. The inorganic content of its constituent biota, the allochthonous input deposited by means of geomorphological processes and the degree to which the original organic component has been decomposed, are some of these factors (Moore, 1989). The concentration of detrital carbonates in sediments may be used as a proxy for the balance between catchment erosion and lake internal productivity (Hammarlund *et al.*, 1997). Moreover, magnetic susceptibility data may provide a tool to identify a succession of changes in the local environment (e.g. Sandgren & Snowball, 1996; 1999). In addition, some of the lake sediments provide an independent annually laminated or varved chronology.

Anyway, the character and procentual relationship between all lake ecosystem constituents are a result of the powerful influence exerted by climate over geomorphic processes and ecological features, which act in the lake catchment area.

### 3.2 Introduction to the methods

#### *Loss on ignition*

Loss on ignition is a common and widely used method to estimate sediment properties (i.e., water content, organic matter, inorganic carbon and minerogenic residue), because it is the quickest and cheapest among all the methods employed for determining some of these parameters (Dean, 1974; Maher, 1998).

Most sediments are composed of a mixture of clastic silicates and oxides (sand, silt and clay fractions), organic material, carbonates and water. Quantitative determinations of such sediment parameters by means of loss on ignition is based on the sequential heating of the samples (other techniques involve gas collecting and measuring the amount either volumetrically or chromatically (Dean, 1974)) in a muffle furnace (Heiri *et al.*, 2001). After oven-drying

of the sediment to constant weight (or dry matter) and cooling to room temperature, organic matter is combusted in a first reaction to ash and carbon dioxide at temperatures between 500-550 °C. The amount of organic matter is the weight difference between the dry sediment and the 550 °C ash (Dean, 1974; Maher, 1998; Heiri *et al.*, 2001), being assumed to be derived from organic material with the formula  $(CH_2O)_n$ , (Snowball & Sandgren, 1996).

In a second step, the same sample is ignited at temperatures between 925 °C (Bengtsson & Enell, 1986, Heiri *et al.*, 2001) and 1000 °C (Dean, 1974; Maher, 1998) in order to find the amount of carbon dioxide evolved from the breakdown of the carbonate minerals (Dean, 1974; Heiri *et al.*, 2001). The carbonate content in the original sample is then determined by multiplying the weight loss between loss on ignition at 550 °C and loss on ignition at 925 °C with 1.36, which is the difference between the molecular weights of  $CO_3$  (60) and  $CO_2$  (44), according to Bengtsson & Enell (1986).

Dean (1974) and Bengtsson & Enell (1986) pointed out that the method is fast and inexpensive and thus useful for analyses of a large number of samples. It also gives a rough indication of the organic matter and carbonate contents and a good correlation with organic carbon content, despite of several uncertainties (see chapter 7.2), which limit the precision of the method.

#### *Grain-size distribution*

Particle size distribution is a fundamental property of sedimentary material and can be

used to infer the provenance and history of the sediment transport (Buckley & Cranston, 1991).

Size analysis of clay and silt-sized sediment can be inferred by a variety of automated methods based on their physical, electrical and optical properties. The Sedigraph method measures the sedimentation speed of suspended particles by monitoring the rate at which particles settle and are removed from the monitored volume, giving in that way a cumulative size distribution of the sediments (Coakley & Syvitski, 1991). Particle diameters are then calculated in terms of equivalent spherical sedimentation diameter.

#### *Total organic carbon*

Total carbon concentration is a fundamental parameter for describing the abundance of organic matter and inorganic carbon in sediments (Meyers & Lallier-Vergés, 1999).

The total organic carbon concentration is a bulk value that represents the fraction of organic matter that escaped mineralization during sedimentation processes being influenced by both initial biomass production and subsequent degree of degradation. Total organic carbon concentration often varies through the sedimentary sequence, indicating changes in organic deposition under different sedimentary conditions (Meyers & Lallier-Vergés, 1999).

## 4 Methodology

### 4.1 Fieldwork

Corring was performed by John Eriksson and Kalle Ljung in October 2000. Multiple core segments were retrieved with a Russian peat sampler (1m length and 10 cm diameter). The lithostratigraphy of the cores was described both in the field and in the laboratory.

### 4.2 Laboratory work

#### *Core description and sampling*

Because detailed sedimentological analyses mainly focused on the supposed Late Glacial to early Holocene part of the record, only the lowest three core-segments of core C6 (from 767 to 512 cm) were selected for further analysis in this study. Lithofacies and sedimentological structures were described in detail before the core segments were sub-sampled. The upper boundary between sediment units was described as sharp, fairly sharp, gradual and rather gradual according to changes in sediment colour, organic and clastic material concentrations (see Tables 1, 2 and 3). After cross correlation between subcores in the laboratory, contiguous sample slices of 1 cm each were taken, when differences in sediment composition and structure occurred, otherwise 2 cm increments were sampled. Furthermore, every sediment segment was subdivided into three subsamples (for loss on ignition, total carbon and grain-size distribution), put in plastic bags and kept in a cold room until further treatment.

#### *Loss-on-ignition*

In the present study, high resolution loss-on-ignition analysis was performed in the presumably Late Glacial sediments. In total, 226 samples were analysed, according to Bengtsson & Enell (1986) and Heiri *et al.*

(2001). Volumetrical samples were weighed (using a Sartorius-Tillquist model H120 balance), placed in pre-weighted crucibles and dried for 12 h at 105 °C (using a Termarks oven) to determine both the water content (or loss-on-drying) and dry sediment percentages (normally 3-5 g), the latter serving as a base to which ignition losses were compared.

The weight loss during the ignitions (using a Heraeus type 170 muffle furnace) were calculated according to Bengtsson & Enell's (1986) formula, with the exception that in the present study the weight loss is related to dry weight. Samples were gradually heated until they reached temperatures of 550 °C and 925 °C, respectively and afterwards ignited for 4 h at the respective temperatures. The samples were then put into a desiccator and weighed at room temperatures. The carbonate content was determined by multiplying the weight loss obtained between 550 °C and 925 °C with 1.36. Minerogenic residue was calculated by subtraction of organic matter and carbonate percentages from 100 %.

#### *Grain-size distribution*

For the grain-size analysis a Micromeritics Sedigraph 5100 Particle Size Analysis System was used. This sedigraph uses a collimated beam of X-rays to sense the sedimentation speed of suspended particles with time and a device measurement of particles with diameters ranging from 100 to 0,2 micrometers (Coackley & Syvitski, 1991). The samples (2-3 cm<sup>3</sup>) were first split to obtain a subsample of approximately 1,8 g for analysis, deflocculated in 40 ml Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub> and dispersed by means of ultrasonic stirring. Because it had been noted that samples with high organic carbon (>10%) can hinder proper dispersion of the sample (see Coakley & Syvitski, 1991), selected intervals were used where organic matter content was low. Finally, the grain size distribution data is presented as cumulative percentages of several fractions: clay (<2 µm), fine silt (2-6 µm), medium silt (6-20 µm), coarse silt (20-60 µm) and fine sand (60-100 µm).

### *Magnetic susceptibility*

Magnetic susceptibility was continuously measured over the core with 4 mm increments and calibrated to the air between every reading. The sampling and measurements were performed by John Eriksson.

### *Total organic carbon*

The sediments were dried (for 12h at 105 °C), finely ground and homogenised and then stirred and shaken in a tungsten ball container. Due to different amounts of material available, sample size varied between 0.12 and 0.30 g of dry weight. The organic and inorganic carbon content of the selected samples was determined by temperature controlled combustion in pure oxygen (step-wise heating from 100 to 1000 °C) of the homogenous and dried samples with subsequent detection of carbon dioxide by infrared absorption

photometry in a LECO RC 412 Multiphase Carbon Determinator.

The carbon emitted between 105 and 650 °C is interpreted as the content of organic carbon of the samples, otherwise, the carbon evolved between 650 and 925 °C is interpreted as the carbonate content, assuming theoretical carbon contents of 12 % and c. 40% for pure calcium carbonate and organic matter, respectively.

### *Dating*

The radiocarbon dates were carried out at the Radiocarbon Dating Laboratory, Department of Quaternary Geology, Lund University (LuA), following standard procedures for accelerator mass spectrometry (AMS) radiocarbon dating. Calibration of the radiocarbon ages to calendar years BP was done using the calibration program proposed by Stuiver *et al.* (1998).

## 5 Results

### 5.1 Lithology

The general lithology of the investigated cores included a succession of coarse minerogenic sediments (silt and sand fractions) between 767-720 cm, clayey gyttja and gyttja silt between 720 and 683 cm, overlain by fine detritus gyttja and gyttja between 683 and 512 cm. The c. 5 m of coarse detritus gyttja and peat < 512 cm were not analyzed in this study. The section was further subdivided into eleven units (from oldest I to youngest XI) according to the sediment description and the cross-correlation between the cores. Furthermore, changes in loss on ignition parameters and grain-size distribution, which were generally associated with lithological changes, were used for characterising the lithostratigraphical data and to correct the overlap between sub-cores. The differences in depth between the sub-cores, using as standard data the organic matter curve, were in the order of 3.5 cm between sub-core C6-b and C6-a and 1.5 cm between sub-core C6-c and C6-b. For lithological details see the sediment description in Figure 2 and in Tables 1, 2 and 3. Based on the correlation between the different sub-cores (Fig. 2), a "standard" section was established (core C6). This standard section is in the following used to describe the different parameters. The loss on ignition parameters are shown in Figure 3, the grain-size distribution in Figure 4 and magnetic susceptibility in Figure 5.

The bottom sediments of unit I (767-757 cm) consist of alternating layers of fine sand and silt. Within the whole succession, the minerogenic residue content has an absolute maximum in this section (96.2%). The dominant fractions as recorded by grain-size are fine sand (30%) and silt (67%). The magnetic susceptibility curve reaches very high values. The organic matter content is below 1% and shows that this unit is almost inorganic. The carbonate content is relatively high, (~4%). The water content ranges between 6 and 17 %.

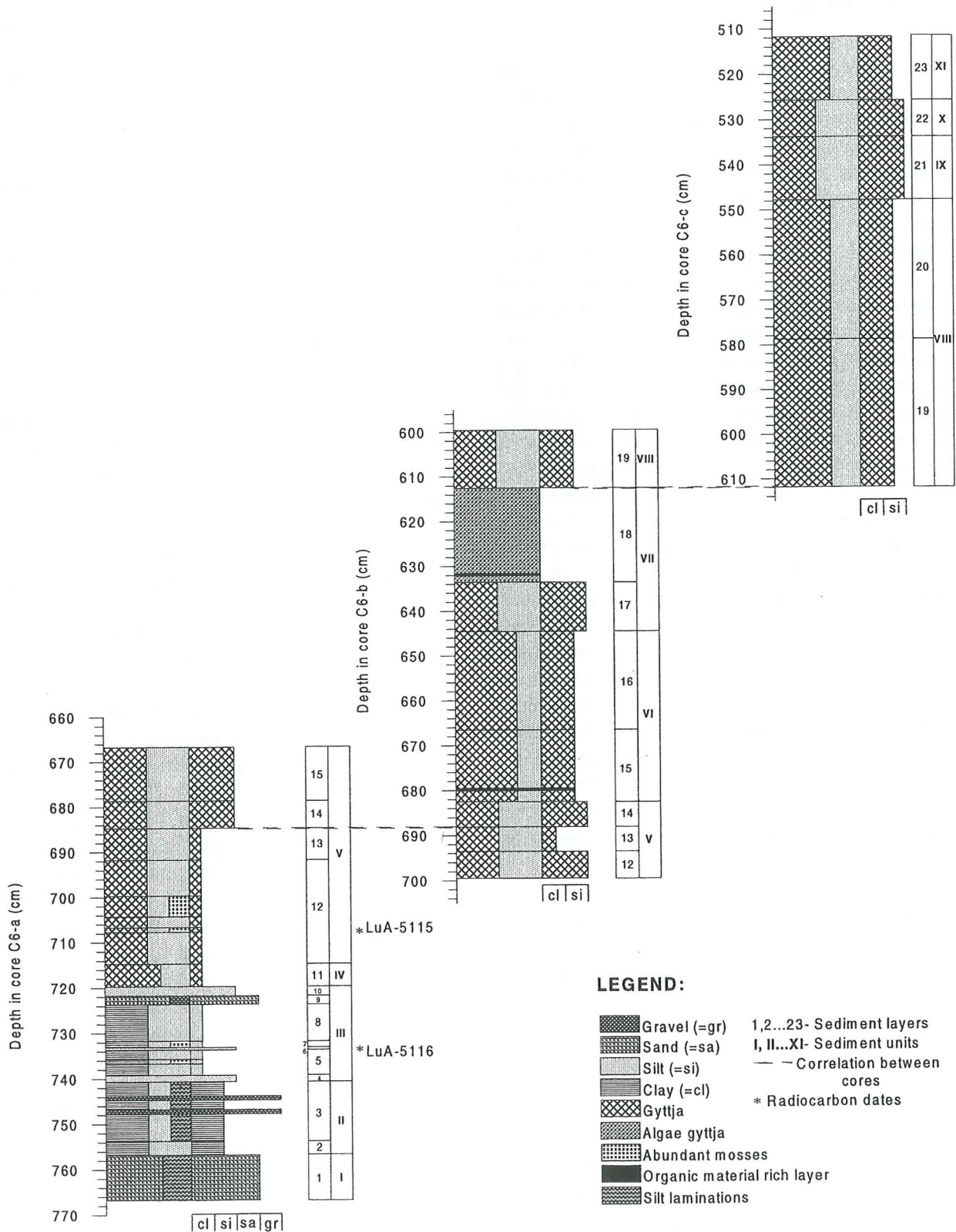
Unit II (757-741 cm) is a succession of silty clay layers, the lower one being

characterized by fine laminations spacing 0.5 cm and by calcareous particles. The sand content is 28 % in layer 2, but drops to 2-5 % in layer 3, while the clay fraction increases to more than 30%. The magnetic susceptibility is moderate, but increases abruptly towards the upper boundary. The carbonate content is higher than 5%. The organic matter content shows increasing values in both layers, but does not exceed 3%. The water content increases from layer 2 (c. 24%) to layer 3 (33%) and the minerogenic residues is still high (90-94%).

The main characteristics of Unit III (741-720 cm) are the extremely fluctuating tendencies for all determined parameters. Organic matter is less than 2% in the coarser layers (4, 6 and 9) and 2-4% in the clayey silt layers (5, 7 and 8). The water content is relatively high (17-35%), with increasing tendencies upwards. The same trend appears in the carbonate content, with values between 1.7 and 6%. The grain-size data suggest an alternation between high percentages of coarse silt and fine sand in the coarser layers and high clay percentages in fine-grained layers. Thus, at 731.5 cm (layer 8), the coarse silt recorded from grain-size data is 60%, while the clay fraction is below 8%. Generally, the clay fraction ranges from 14-18% (layers 4 and 9) to 33-38% (layers 5 and 10). The sand fraction has a slight revert in the range of 3-5% in layers 8 and 9, while coarser silt has fluctuating values, ranging from 37-38% (layers 4, 8, and 9) to 19% (layer 5) or even 10% in layer 10. Medium silt is present in high percentages in the whole unit. Fine silt has the same trend as the clay percentages, but with reduced values. The grain-size data are further supported by the magnetic susceptibility curve, which shows very large fluctuations (Fig. 5).

In Unit IV (720-715 cm) the organic matter content is between 3-4%. Water content is generally high (32-34%). Carbonate content is below 6%, while the minerogenic residue is still high (<93%). The grain-size distribution shows a gradually increasing tendency for the coarser fraction throughout this zone. At the lower boundary the clay fraction attains more

Figure 2 Lithostratigraphical logs and correlations between sub-cores.





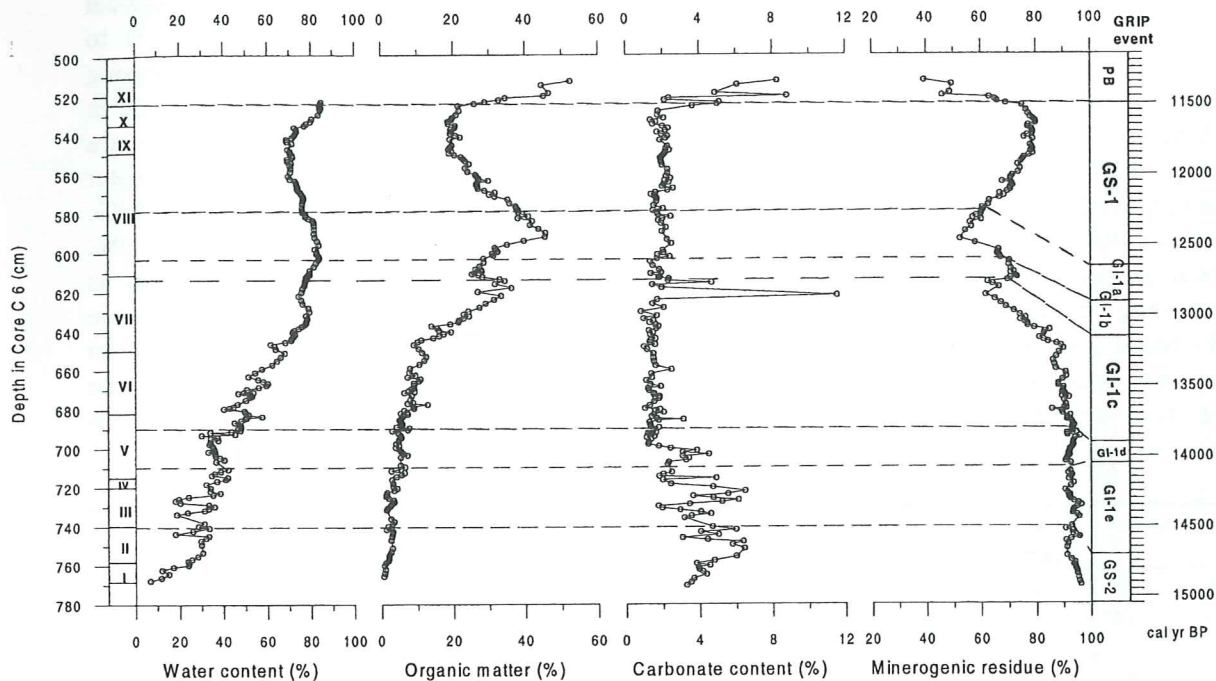
than 44%. Medium silt is high (25-27%), while the fine silt fraction is rather low (< 14%). The magnetic susceptibility starts to decrease in this unit, reaching low values.

With the beginning of unit V (715-679 cm) all parameters change. The alternation between more or less moss rich horizons in the lower part of the unit cause fluctuating values for organic matter, ranging from 2.6% to 7%. The carbonate content is rather low and generally below 3%. The water content is high (<40%), but with oscillating values. The minerogenic residue displays slightly decreasing values (93-90%). A rise in organic

matter at 685 cm corresponds to a lithological change from clayey silt gyttja (layer 13) to gyttja silt (layer 14). The moderately increasing values for organic matter content (from 5% in the gyttja silt to 9% in the silt gyttja) are accompanied by an increase in the water content, which reaches values higher than 40%. The carbonate content decreases below 2%. The grain-size distribution shows that clay is 28% in layer 12, and that the values decrease through layers 13 (16%) and 14 (13%). An inverse relationship with clay is shown by the coarser silt fraction, which rises from 13% (layer 12) to 45-50% in layers 13 and 14.

<i>Unit</i>	<i>Layer</i>	<i>Depth below surface (cm)</i>	<i>Sediment description</i>
VI	15	667-679	Beige grey silt gyttja.
	14	679-685	Beige grey gyttja silt sparse in FeS stains. Upper boundary rather gradual.
	13	685-692	Beige grey clayey silt gyttja, with cladocera remains. Upper boundary rather gradual.
V			685-688 cm more beige, 688-692 cm more dark.
	12	692-715	Beige grey clayey silt gyttja, brown in moss rich areas, laminated by some organic matter layers. Upper boundary rather gradual. 696-700 cm some moss remains, 700-704,5 cm increase in moss remains, 704,5-707 cm few moss remains, 707-708 cm brown layer, rich in mosses.
IV	11	715-720	Brown to beige-grey clayey gyttja silt, organic matter decreases gradually upwards to 718 cm. Upper boundary fairly sharp.
	10	720-722	Very disturbed layer of light grey silt, Upper boundary fairly sharp.
	9	722-724	Dark grey coarse sand/gravels. Upper boundary fairly sharp.
	8	724-732	Dark grey clayey silt with some coarse silt in the bottom. Upper boundary sharp.
III	7	732-733,5	Beige grey clayey silt layer, abundant mosses, laminations. Upper boundary fairly sharp.
	6	733,5-734	Light grey silt layer with some moss remains. Upper boundary rather gradual.
	5	734-739,5	Dark grey clayey silt with mosses (736-737 cm). Upper boundary fairly sharp.
	4	739,5-741	Grey silt with coarse silt and organic material in the bottommost part (dark grey). Upper boundary gradual.
II	3	741-754	Dark grey silty clay laminated by thin silt-layers; gravel particles at 745 and 748 cm, respectively. Upper boundary fairly sharp.
	2	754-757	Layers of dark-grey to black silty clay laminated every 0,5 cm; calcareous particles. Upper boundary fairly sharp.
I	1	757-767	Grey fine-sand to sand with thin silt lamination; calcareous particles and vague lamination from coarse silt to fine sand. Upper boundary rather gradual.

**Table 1.** Lithostratigraphic units and sediment description for Core C6-a.



**Figure 3.** Lithostratigraphic units and water content, organic matter, carbonate carbon and minerogenic matter versus depth in core C6 from Atteköps mosse. The dashed lines suggest a tentative correlation with the GRIP event stratigraphy (Björck et al., 1998) which is in detail discussed in chapter 6.1.

Unit	Layer	Depth below surface (cm)	Sediment description
VIII	19	600-613	Beige silty fine detritus gyttja, FeS stains.
	18	613-645	Greenish grey algae gyttja, FeS stains. Upper boundary fairly sharp.
VII	17	634-645	632-632,5 black organic layer Grey silty gyttja.
	16	645-667	Beige brown silty fine detritus gyttja with cladocera remains. Upper boundary rather gradual.
VI	15	667-683	645-650 light beige brown Beige brown silty fine detritus gyttja with FeS stains, cladocera remains and a black organic layer at 680-680,5. Upper boundary fairly sharp.
	14	683-688,5	Beige grey gyttja silt with some twinkling cladocera remains. Upper boundary rather gradual.
V	13	688,5-694	Beige grey clayey gyttja silt with vague cladocera twinkling and black organic layer at 689,9-690 cm. Upper boundary fairly sharp.
	12	694-700	Dark grey gyttja silt, abundant mosses in layers. Upper boundary rather gradual.

**Table 2.** Lithostratigraphic units and sediment description for Core C6-b

Medium silt is constant with values between 28-33%. Magnetic susceptibility values are low and the fluctuations became less pronounced.

While organic matter content is < 6% in the lower part of Unit VI (679-645 cm), it rises significantly to values higher than 10% upwards. The water content continues to increase (40 to 59%) and its fluctuations became smaller. The carbonate content attains very low (< 2%) and relatively constant values. Minerogenic residues show slightly decreasing values in the whole unit (91-86%). The grain-size data gives 0% for clay in layer 15 and 17% in layer 16. This is in contrast to grain size data from layer 15 of core C6-a, where clay had values higher than 20% (see Fig. 4). Medium silt is decreasing from 71% at the bottom of the unit to 43% upwards. The magnetic susceptibility has low and rather stable values.

Unit VII (645-613 cm) is characterized by a change in sediment type from silty gyttja

(layer 17) to algae gyttja (layer 18), and a rise in all parameters (except for minerogenic residues). Organic matter increases abruptly from 9-19% in layer 17 to more than 35-36% in layer 18 and the water content has increases to >78%. The carbonate content follows the same trend as water curve, increasing abruptly to >10% (a peak with 11.5% at 620 cm depth). Minerogenic content is decreasing (61%). Slightly before the transition to unit VIII, the minerogenic content increases abruptly. The opposite feature is recorded for the organic matter curve.

After a short interval with low values, immediately following the transition to unit VIII (613-548 cm), the organic matter rises abruptly, reaching a maximum of 45.5% at 591 cm depth. This increasing trend is followed by a regular decline between 591 cm (layer 19) and 548 cm (layer 20), showing a minimum of 18.9% at 550 cm.

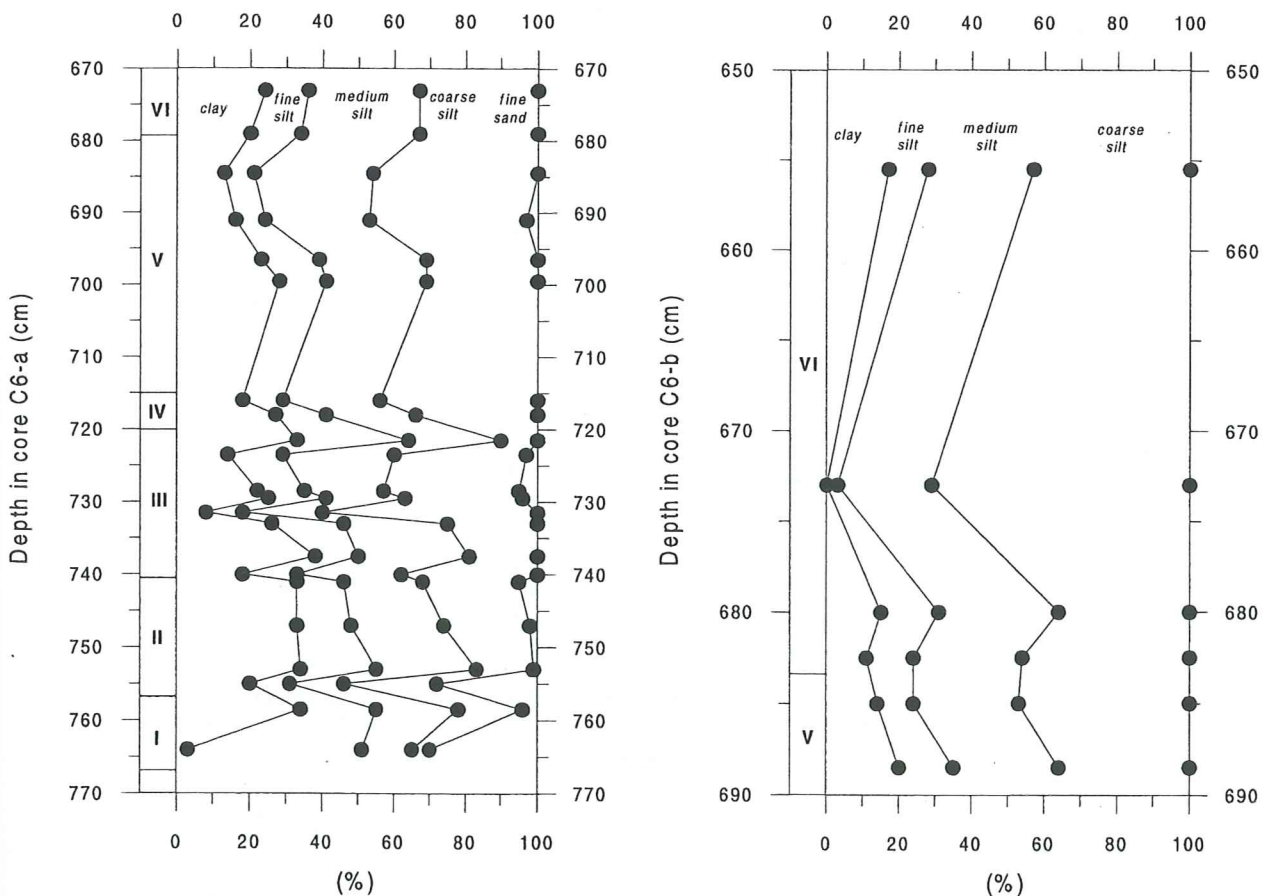


Figure 4 Lithostratigraphic units and cumulative particle size distribution for core C6-a (left) and core C6-b (right), respectively.

Unit	Layer	Depth below surface (cm)	Sediment description
XI	23	512-524	Dark brown silty fine detritus gyttja with some light brown organic matter.
X	22	524-535	Beige grey silty fine detritus gyttja, FeS stains and coarse gravel in the bottom most part. Upper boundary fairly sharp.
IX	21	535-548	Brown silty fine detritus gyttja. Upper boundary fairly sharp.
	20	548-579	Layers of beige grey silty fine detritus gyttja with cladocera remains, vague lamination at 562 cm and increase in organic matter at 561-571cm. Upper boundary rather gradual.
VIII	19	579-612	Beige grey silty fine detritus gyttja. Upper boundary rather gradual.
			612-606 beige brown, FeS stains. Upper boundary rather gradual.
			606-603 beige, FeS stains. Upper boundary rather gradual.
			603-599 beige brown, FeS stains. Upper boundary rather gradual.
			599-594 beige, FeS stains. Upper boundary rather gradual.
			594-579 beige brown, FeS stains. Upper boundary rather gradual.

**Table 3.** Lithostratigraphic units and sediment description for core C6-c.

For minerogenic residue content, the first decreasing values in the lowermost part of the unit (reaching a minimum of 52% at 592 cm) are followed by gradually increasing tendencies, reaching 78% at the upper boundary. The water content is first very high (85%) and drops dramatically to less than 70 % upwards. The carbonate content displays slightly increasing tendencies, but values are generally below 3%.

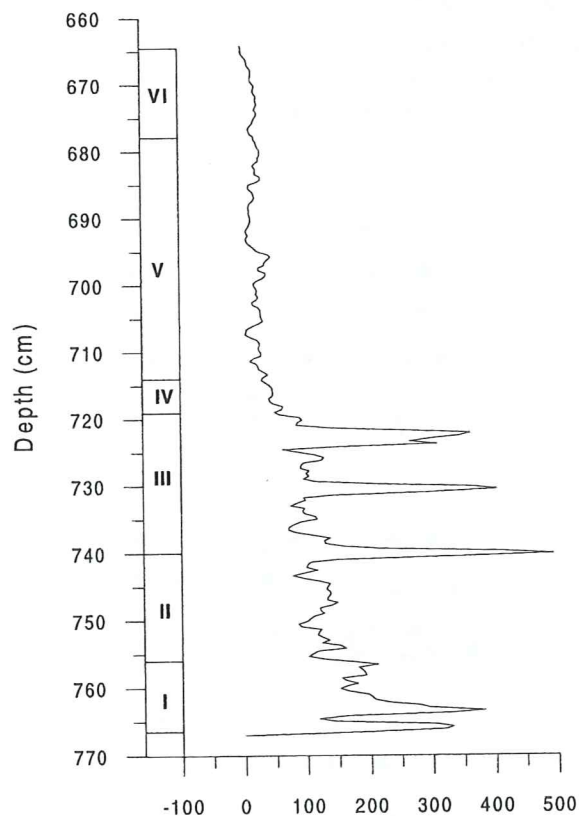
Above 548 cm, in Unit IX (548-535 cm) water content is <70 %. Organic matter is also low with constant values of 18-22%. Carbonate content is fluctuating around 2 % and minerogenic residues are very high (75-80%).

In Unit X (535-524 cm) the organic matter content starts to increase abruptly (30%), while minerogenic residues decrease. Water and carbonate contents have increasing tendencies.

With the beginning of Unit XI (524-512 cm) the sediment becomes highly organic (52%) and has a lower percentage of inorganic material (39%). Water and carbonate contents have oscillating but generally high values of 80-85% and 2-9%, respectively.

## 5.2 Total carbon analyses

With lower resolution than loss on ignition, total carbon analysis (Fig. 6) is used as a standard against which loss on ignition



**Figure 5.** Lithostratigraphic units and magnetic susceptibility data for core C6-a, plotted against depth.

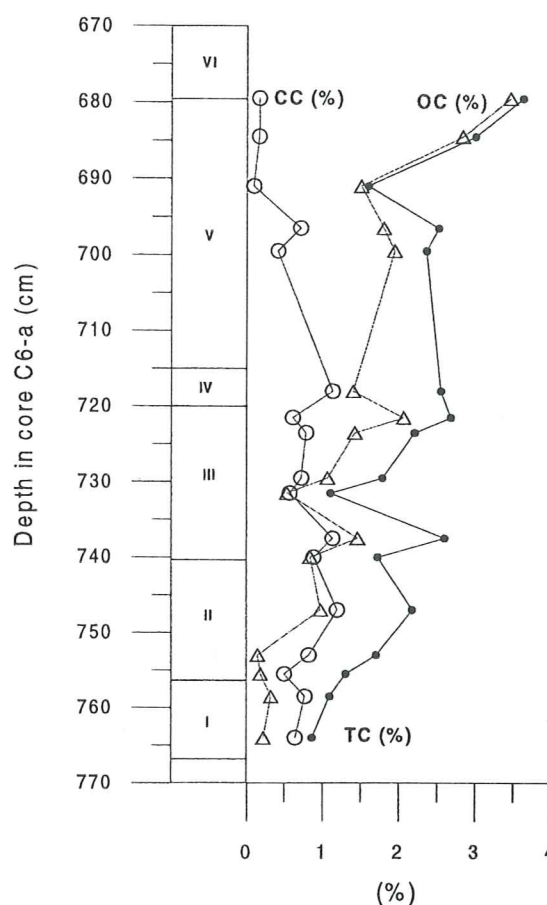
and grain-size data are compared (see Chapter 7.2).

Organic carbon is <1% in unit I and II, fluctuates between values of 0.5 % and 2.5% in units III and IV and starts to increase to >3% at

690 cm in unit V. The carbonate content, however, exhibits increasing values from unit I to unit III (to ~ 1%) and lower values afterwards (< 0.5%). Total carbon (TC) is the composite curve of both parameters, generally with values below 3 % in the whole analysed sequence.

### 5.3 Radiocarbon dates

Two radiocarbon dates were obtained on material from the Atteköp C<sub>6</sub>-a core segment. Samples for <sup>14</sup>C dating were taken at 734 cm depth, at the transition from clayey silt (layer 5) to silt (layer 6) and at 707 cm depth (layer 12, clayey silt gyttja), respectively (see Table 4). The lower sample (LuA-5116) consisted of moss fragments and was dated to 13,580 ± 130 <sup>14</sup>C yr BP. The upper sample (LuA-5115) which was dated to 12,170 ± 130 <sup>14</sup>C yr BP consisted to *Salix* leaves.



**Figure 6.** Lithostratigraphical units and carbon content for core C<sub>6</sub>-a. The carbon values are presented in pure carbon ratio with separate curves for organic carbon (OC-empty triangles), carbonate carbon (CC-empty circles) and total carbon (TC-filled circles). All values are expressed as percentages.

Site locality	Depth (cm)	Material	Laboratory number	<sup>14</sup> C yr BP	δ <sup>13</sup> C	Sample weight	Cal. yr BP 95.4 % probability
Atteköp	707	<i>Salix</i> sp. leaves	LuA-5115	12,170 ± 130		3 mg	14,050 + 1,400
							- 400
Atteköp	734	Aquatic mosses	LuA-5116	13,580 ± 130	- 30.6	20 mg	16,300 ± 650

**Table 4.** Radiocarbon dates from the investigated sequence. The data are presented both in <sup>14</sup>C yr BP and cal yr BP. Calibration of the radiocarbon dates to calendar years BP is according to the age calibration program of Stuiver et al. (1998).

## 6 Interpretations

### 6.1 Chronology

#### *Datings and correlations*

For those familiar with interpreting radiocarbon data derived from lacustrine sediments spanning the Late Glacial, the radiocarbon ages presented in Table 4 may rise several questions. Having a look at the lithostratigraphic description, sediment depth and loss on ignition curves, there seem to be either inconsistencies between the two radiocarbon dates or some special features of palaeosedimentation patterns.

If both radiocarbon dates are regarded as correct, it means that the sediments between 707 cm (LuA-5115:  $12,170 \pm 130$   $^{14}\text{C}$  yr BP) and 734 cm (LuA-5116:  $13,580 \pm 130$   $^{14}\text{C}$  yr BP) are characterized by a low rate of sedimentation. This is in contrast with the general assumption that the Late Glacial was characterized by a high rate of sedimentation (generally clastic particles), due to scarce vegetation cover and increased geomorphological activities. Therefore, a reworking of older organic material, could have caused such a large difference in age between the two dates. Another possibility is that, LuA-5116, obtained on aquatic mosses was subjected to contamination by old carbon, which could have resulted from dissolution of carbonates (e.g. Hammarlund, 1994). Following the above mentioned uncertainties, LuA-5116 has very likely to be regarded as too old.

On the other hand, the younger sample LuA-5115 is situated on a radiocarbon plateau, which leads to large uncertainties for the calibrated age of the sample ( $14,050 \pm 1400$  and  $400$  cal yr BP, respectively). However, the date was obtained on terrestrial macrofossils (leaves of *Salix* sp.), which in general yield reliable ages. It is therefore assumed that LuA-5115 provides a better age estimate than LuA-5116.

An attempt to date the sediments from Atteköps mosse was earlier made by Liedberg Jönsson (unpubl. data), who obtained an age of

$12,980 \pm 120$   $^{14}\text{C}$  yr BP (Lu-2207) on aquatic mosses (S. Björck, pers. comm.). The dated sample was derived from clayey gyttja rich in organic detritus between 670-693 cm depth.

Most probably, this may correspond to layer 12 (692-715 cm, clayey silt gyttja with layers rich in mosses) in the present study. Thereby, probably the same horizon was dated, and the ~20 cm difference between the lithologies may be explained by a more complete stratigraphic sequence recorded here.

Summarising these interpretations, a correction of ~800  $^{14}\text{C}$  yr for Lu-2207 (S. Björck, pers. comm.) should be applied, based on the assumption that the aquatic mosses dated in Lu-2207 probably were subject to contamination by older carbon.

#### *Tentative correlation to the GRIP event stratigraphy*

In an attempt to derive a chronology for the studied sediments, a tentative correlation with the GRIP event stratigraphy (Björck *et al.*, 1998) may be made. Such a correlation rests upon the assumption that the climatic signal registered in the loss on ignition curves corresponds largely to the GRIP events, from the late GS-2 to early Preboreal (see Fig. 3). This may be further supported by the similarities, seen in the loss on ignition curves from Atteköps mosse and those presented by Liedberg Jönsson (1988) from Håkulls mosse (Kullen Peninsula), which span the PreBölling to Preboreal pollen zones. Since both sites are situated within the same area, the similarities, which occur between those two records could be viewed as approximately time-synchronous.

In the following, when the terms GS-2, GI-1 (and subdivisions) and GS-1 are used and expressed as cal. yr BP, they thus not refer to an absolutely dated time interval, but are derived from a tentative correlation.

Based on the arguments presented above and using as a cornerstone the date LuA-5115 ( $12,170$   $^{14}\text{C}$  yr BP), the GRIP events can be separated as follows: Most probably, the sediments around 715 cm correspond to the GI-1e/GI-1d transition and those below 715 cm

would consequently have been deposited during GI-1e (741-715 cm) or even GS-2 (767-741 cm). The GI-1d/GI-1c transition is likely situated at 685 cm, placing the sediments between 715-685 cm into GI-1d. Upwards, the transitions between supposed GRIP events seem to be more

pronounced. Thus, the limit between GI-1c and GI-1b may be set at 613 cm, while the GI-1b/GI-1a transition is fixed at 604 cm. The GI-1a/GS-1 transition is set at 579 cm while the GS-1/Preboreal is clearly evident at around 524 cm depth.

## 7 Discussions

### 7.1 Palaeoenvironmental reconstructions

The analysed sediments are characterized by variations in minerogenic and organic content and comprise from bottom to top sand, silt, silty clay, clayey gyttja and fine detritus gyttja. These sediments probably rest upon glaciofluvial deposits (sand) and underlie Holocene coarse gyttja and peats, indicating in this way, according to Lowe & Walker, (1997) a complete hydroseral evolution. In the following, the main sedimentological properties of the studied sediments are tentatively correlated with the environmental changes characteristic for the Last Glacial-Holocene transition, which are seen in numerous environmental proxies in many previously published palaeoecological studies, mostly from southern Sweden.

The lower most part (767-741 cm, units I-II) most probably marks the transition between glacial derived sediments and organic sediments. In terms of phase, it probably immediately follows the local deglaciation placed at ca. 17,000-16,000 cal yr BP as indicated by recessional moraines (Lundqvist & Wohlfarth, 2001).

The sediments deposited during this time (? late GS-2) are characterized by very low organic content, which could probably result partly from low organic productivity in the lake catchment (see Liedberg Jönsson, 1988) and partly from a substantial dissolution of organic material by mineral matter. The high mineral fluxes (see Fig. 5) and the increased silt fraction (see Fig. 4) indicate intense weathering on land prior to the establishment of a pioneer-like vegetation (this was a colonisation phase, according to Liedberg Jönsson, 1988) and also a low internal productivity, probably because of the high silt content in the water column and hence, low light transmission.

The Late Weichselian isotopic data recorded before 12,400  $^{14}\text{C}$  yr BP derived from Körslättamossen (Hammarlund, 1994) suggests dry summer conditions for this interval. An arctic and highly continental climate probably prevailed

during this time. At the GS-2/GI-1 transition (~14,700 cal yr BP), probably a shift towards more oceanic conditions occurred (e.g. Liedberg Jönsson, 1988), giving rise to organic productivity (see Fig. 3).

The transition from Unit II to Unit IV may be associated with changes in lake productivity and mainly in sedimentation patterns. The GI-1e time period (14,700-14,050 cal yr BP) is generally viewed as a time when a climatic amelioration took place with a positive response in vegetation development (see Liedberg Jönsson, 1988; Berglund *et al.*, 1994) and soil stability (Björck & Möller, 1987). The constant or even decreasing organic matter content and extremely fluctuating values for the other studied parameters between 741 and 715 cm may seem in contrast with the above mentioned records and could be explained either by irregular sedimentation in the lake basin, causing hiatus or, by a high rate of minerogenic inwash (see Fig. 4 and 5), which might have been favoured by an increase in precipitation (e.g. Liedberg Jönsson, 1988) which in turn caused, a dissolution of organic matter. The input of allochthonous carbonates to the lake basin probably points to unfavourable conditions for limnic productivity (e.g. Hammarlund, 1994). Moreover, coleopteran data (Coope & Lemdahl, 1995) suggest a gradually slower rate of temperature increase for southern Sweden during this time (compared to central Europe), likely as a cooling effect exerted by the Fennoscandian ice-sheet and changes in the North Atlantic circulation pattern. On the other hand, isotopic data derived from sites from southern Sweden (Hammarlund, 1994), suggest increasing aquatic productivity at this time, and are interpreted as a change to a more oceanic climatic regime and more humid conditions, as suggested by Björck & Möller (1987).

The decrease in organic matter content and partly also in water content accompanied by a rise in minerogenic matter between 715 (layer 12) and 685 cm (layer 13) could be interpreted as reflecting the GI-1d event (14,050-13,900 cal yr BP).

The GI-1d event has earlier been identified by pollen stratigraphy (Liedberg Jönsson, 1988; Berglund *et al.*, 1994) as an interval characterised



by an open, steppe tundra dominated vegetation (Berglund, 1995). As Hammarlund (1994) pointed out, these conditions were probably caused by a climatic change involving a shortening of the summer seasons, which is also indicated by dendrological data from central Europe (Friedrich *et al.* 2001), and more dynamic geomorphological activities (Björck & Möller, 1987; Berglund *et al.* 1994).

With the beginning of Unit VI, the general trend of all parameters changes. The water content increase continues and fluctuations become smaller. The carbonate content becomes lower than in the preceding units and the minerogenic matter decreases. This may indicate more stable edaphic conditions in concert with a gradually denser vegetation cover. It must be stressed that, the GI-1d/GI-1c (~ 13,900 cal yr BP) transition in the study sediments is not as pronounced as the younger transitions (see Fig. 3). The flat feature of the organic matter curve between 685 and 645 cm may be a response to the cooling effect, caused by last remnants of stagnant ice (see Liedberg Jönsson, 1988). Upwards, the sediment becomes more organic rich, most likely as a result of increased aquatic productivity, as indicated by cladocera remains and algae gyttja deposition. Moreover, a relatively medium carbonate content may indicate an increase in vegetation length, which could imply higher temperatures and the development of a lake ecosystem (Hammarlund, 1994).

The decline in organic matter and water contents correlates to an increase in mineral matter between 613 and 604 cm and may reflect cooler climatic conditions as a result of the cooling, which prevailed during GI-1b (13,150–12,900 cal yr BP). This interval has been recognised as a distinct cooling event with a major impact on both lacustrine and terrestrial ecosystems, as suggested by geochemical and biostratigraphic analyses of a site in Denmark (Andresen *et al.*, 2000).

An abrupt decrease in mineral matter, possibly at the GI-1b/GI-1a (~12,900 cal yr BP) transition, is probably consistent with a sudden climatic amelioration as indicated by the elevated organic content of the sediments between 604 and 591 cm. Most probably, the study sediments

are lacustrine in origin, because the most intensive biological production during the GI-1 interstadial complex, occurred at this time, favoured probably by a long growing season and rising temperatures.

Climatic warming at this stage is indicated by a high concentration of arboreal pollen in pollen diagrams (see Liedberg Jönsson, 1988; Berglund, 1995). Evidence for a climatic deterioration at the later part of GI-1a, gradually leading to arctic or subarctic conditions in early GS-1 is suggested by various climatic proxies from sites in southern Sweden (Björck & Möller 1987; Liedberg Jönsson 1988; Hammarlund 1994; Berglund *et al.*, 1994). The organic productivity as reflected in the sediments probably decreased in the later part of GI-1a and during the early part of GS-1 (the limit between these two stages is fixed at 579 cm depth) at a rate, similar to the rate of cooling (see Fig. 3). Moreover, the relatively high organic content during the early GS-1 (although decreasing) probably reflects in-wash of allochthonous organic and detrital material as an effect of destabilisation of biotic (e.g. Liedberg Jönsson, 1988) and edaphic ecosystems (e.g. Berglund *et al.* 1994) in the lake catchment. Unstable soils susceptible to erosion are also indicated by high values of *Coenococcum geophilum* in sediments of Håkuls mosse (see Liedberg Jönsson, 1988). This increase in nutrient supply may have caused an eutrophication and hence a short enhanced algal production. A similar significant pattern during this period of rapid climatic cooling has been pointed out by Brauer *et al.* (1999). The middle part of the GS-1 stadial was generally characterized by a cold and dry climate with intensive eolian activity (Liedberg Jönsson, 1988; Berglund *et al.*, 1994), even if a slight increase in organic production during this period is recorded in sediments from Torreberga (Hammarlund *et al.*, 1997). A slight increase in organic matter percentages during the later part of GS-1 (see Fig. 3) seems to be in accordance with Liedberg Jönsson's (1988) interpretation, who found a gradual development of the vegetation cover.

The rapid climatic warming at the GS-1/PB transition (< 524 cm depth) led probably to complex responses in the local and regional vegetation, to geomorphologic processes and to

an increase in internal productivity in the lake ecosystem. The decreased erosion rate (see Björck & Möller, 1988) as a result of development of the terrestrial vegetation (see Liedberg Jönsson, 1988) led to enhanced organic productivity (and probably high preservation) as indicated by the highly organic content of the sediments.

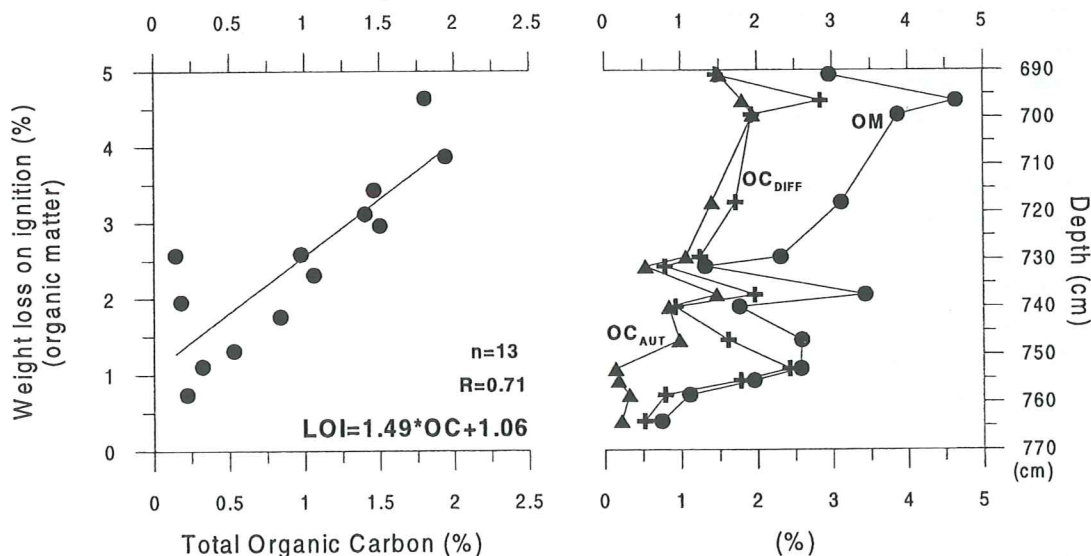
## 7.2 Comparison between loss on ignition and total carbon

Numerous authors from Dean (1974) and onwards have noted that loss on ignition can be considered to be a good tool for determining the organic matter and carbonate contents of sediments, even if several uncertainties limit the absolute precision of the method (see Dean, 1974; Bengtsson & Enell, 1986; Heiri *et al.*, 2001). The relative changes of loss-on-ignition parameters throughout the analysed core sections are regarded as very useful in interpreting the impact of changing climatic conditions on different biogeochemical processes throughout the sediment sequence. In general, the amount of

organic matter determined by loss on ignition at 550 °C is considered to be "approximately" twice the organic carbon content (Dean, 1974, 1999; Snowball & Sandgren, 1996) of a sample. The organic matter content can then be multiplied by a constant, which generally lies between 0.4 and 0.6 (Maher, 1998) in order to estimate the amount of organic carbon in the original sample.

Total carbon determined automatically is a fundamental parameter for describing the abundance of organic and inorganic carbon in sediments. In this way, organic matter values, via loss on ignition, are, as already mentioned, presumed to be equivalent to about twice the total organic carbon values (Meyers & Lallier-Vergés, 1999). Total organic carbon can be both diluted by clastic sediment particles and concentrated by dissolution of carbonate minerals.

Various conversion factors have been proposed for converting organic carbon to original organic matter content of a sample. For example, Dean (1974) found a conversion factor of 2.13, Zolitschka & Löhr (1999, cited by Ramrath *et al.*, 1999) and Ramrath *et al.*, (1999) have proposed a factor of 1.82 and 1.81, respectively, while Digerfeldt *et al.*, (2000)



**Figure 6.** Left: Scatter diagram of percent total organic carbon determined automatically and percent organic matter at 550 °C measured on samples from Atteköps mosse. Number of data points ( $n$ ), correlation coefficient ( $R$ ) and the regression line are given.

Right: Percentage organic carbon determined automatically (OC<sub>AUT</sub>-triangles), percentage organic matter (OM<sub>LOI</sub>-circles), and the difference in results between those parameters (OC<sub>DIFF</sub>-crosses), plotted against depth.

obtained a factor of 1.45.

The accuracy of organic carbon determination by means of loss on ignition is hampered by some factors, which limit the precision of the method, leading to an overestimation of the true organic content. It was generally noted that for determining organic carbon content of minerogenic sediments by means of organic matter (via loss on ignition), the results can strongly be overestimated because of various losses, which belong to volatile salts, organic compounds (e.g. Bengsston & Enell, 1986), structural water (e.g. Dean, 1974), sulphide oxidation (Ramrath *et al.*, 1999) or inorganic carbon. For example, if siderite minerals ( $\text{FeCO}_3$ ) are present they will evolve  $\text{CO}_2$  at temperatures as low as c.  $490^\circ\text{C}$  (Hedvall, 1925, cited by Hammarlund, 1994). Moreover, as Aaby (1983, cited by Andersen, 1986) pointed out, the volume of organic matter cannot be determined exactly, as pore space enters the volume of the ignitable matter. In this case, changes in the organic content can be detected by determination of organic carbon (Andersen, 1986). On the other hand, technical factors such as sample size, exposure time, position of the sample in the furnace may affect the loss on ignition results, with loss on ignition at  $550^\circ\text{C}$  being more susceptible to these factors than loss on ignition at  $925^\circ\text{C}$  (see Heiri *et al.* 2001). As a consequence, any bias of loss on ignition at  $550^\circ\text{C}$  will influence the results of loss on ignition at  $925^\circ\text{C}$  (see Heiri *et al.* 2001).

One of the major aims of this study is to test the accuracy of organic carbon determination by loss on ignition, since selected values from loss on ignition at  $550^\circ\text{C}$  are believed to represent either typically organic matter, but also a weight loss related to various organic or inorganic sediment components. Therefore, organic carbon values determined automatically and on the same sample are regarded as representative for the organic carbon of the original sediment matter, and are compared to

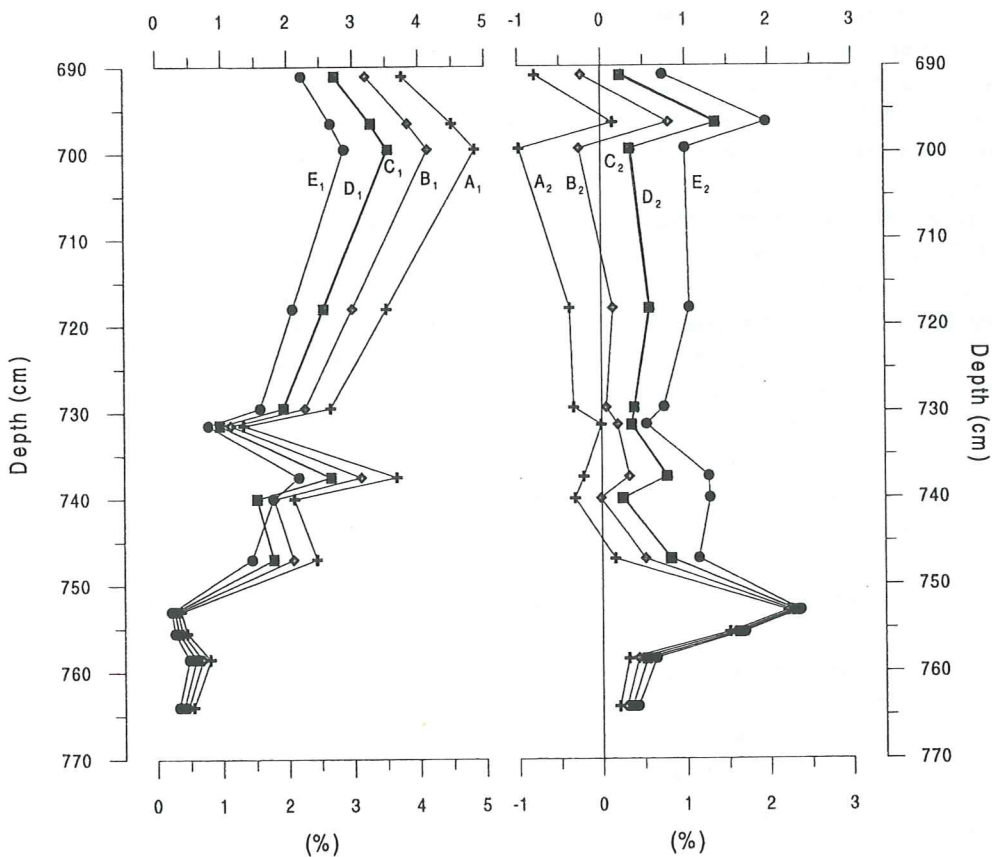
corresponding loss on ignition values. The results of this test are plotted in Figure 6 (left panel). Surprisingly, the resulting regression line crosses the y-axis at a loss on ignition value of 1.06 %. This probably corresponds to a weight loss which should be related to the already mentioned extra-loss generators. The true organic matter content in the analyzed samples as suggested by the equation presented in Figure 6 (left) may be obtained by multiplication of the total organic carbon determined automatically with a factor of 1.49. Though this low factor suggests a good correlation between these different organic carbon determinations, indicating that loss on ignition is an accurate measurement of the amount of organic matter in a sample, the resulting conversion factor is not in good agreement with the published factors presented above. This confirms the idea that the amplitude in difference between the results of those two methods may depend on type and chemical composition of the analyzed sediments.

In order to test the accuracy of the organic carbon determination and to find out a proper correction factor, the ignition loss results were compared with total organic carbon multiplied by different conversion factors (see Fig. 7 (left panel)) :

- A<sub>1</sub>- automatically determined organic carbon multiplied by 2.5 (according to the chemical formula  $\text{CH}_2\text{O/C}$ )
- B<sub>1</sub>- automatically determined organic carbon multiplied by 2.13 (according to Dean, 1974)
- C<sub>1</sub>- automatically determined organic carbon multiplied by 1.82 (Zolitschka & Löhner, 1999 cited by Ramrath *et al.*, 1999)
- D<sub>1</sub>- automatically determined organic carbon multiplied by 1.81 (Ramrath *et al.*, 1999)
- E<sub>1</sub>- automatically determined organic carbon multiplied by 1.45 (Digerfeldt *et al.*, 2000).

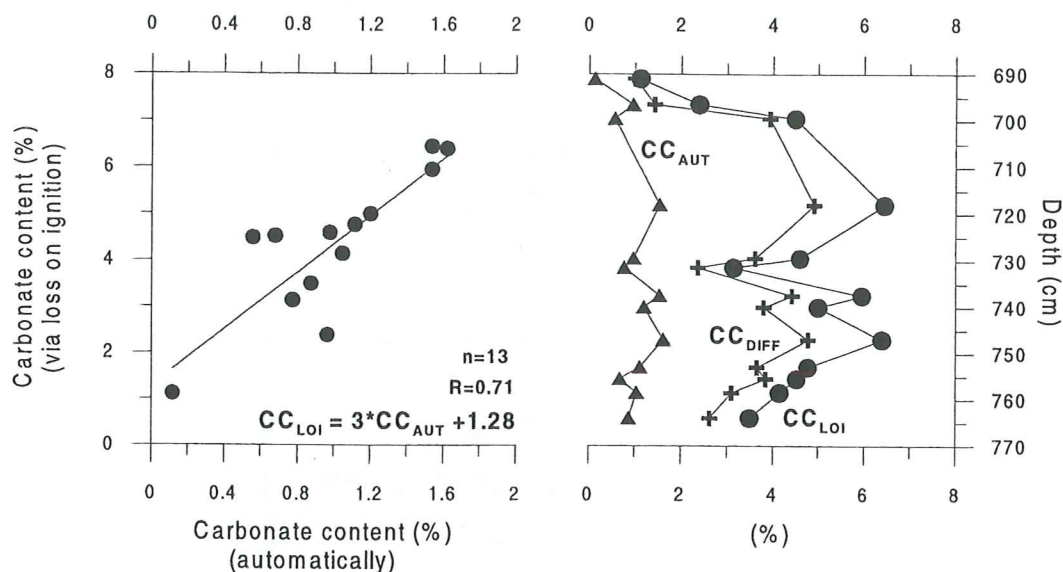
The variables which resulted as differences between loss on ignition at 550 °C and organic carbon multiplied by different constants are labelled from A<sub>2</sub> (as a variable produced by the A<sub>1</sub>-chemical formula) to F<sub>2</sub> (a variable induced by the F<sub>1</sub>- Digerfeldt *et al.*, 2000) and plotted in Figure 7 (right). The vertical zero line implies a perfect correlation between the values. The positive trend suggest that the loss on ignition values at 550 °C are higher than presumably supposed, while the negative values, indicate higher values for organic carbon. As easily may be seen from the curves in Figure 7 (right), the A<sub>2</sub>

and B<sub>2</sub> variables yield generally negative values, implying in this way an overestimation of primary organic matter content determined by loss on ignition. The C<sub>2</sub> and D<sub>2</sub> variables are relatively close to zero, whereas E<sub>2</sub> leads to increased differences. To summarize these results, it is evident that the conversion factors mentioned above are strongly influenced by the type of sediment. Moreover, if an absolute determination of the organic content is to be done, then the loss on ignition data must be supported by independent total organic carbon determination.



**Figure 7.** Left: Organic carbon determined automatically and multiplied by various conversion factors: 2.5 (A<sub>1</sub>-crosses), 2.13 (B<sub>1</sub>-rhombus), 1.82 (C<sub>1</sub>-triangles), 1.81 (D<sub>1</sub>-squares) and 1.45 (E<sub>1</sub>-circles) plotted against depth.

Right: The differences (A<sub>2</sub>, B<sub>2</sub>, C<sub>2</sub>, D<sub>2</sub>, E<sub>2</sub>) which result from a subtraction of organic carbon determined automatically and multiplied by the already mentioned factors from loss on ignition at 550 °C.



**Figure 8.** Left: Scatter diagram of percent carbonate content determined automatically and percent carbonate content derived from loss on ignition at 925 °C measured on samples from Atteköps mosse. Both values are multiplied by 1.36. Number of data points ( $n$ ), correlation coefficient ( $R$ ) and the regression line are given.

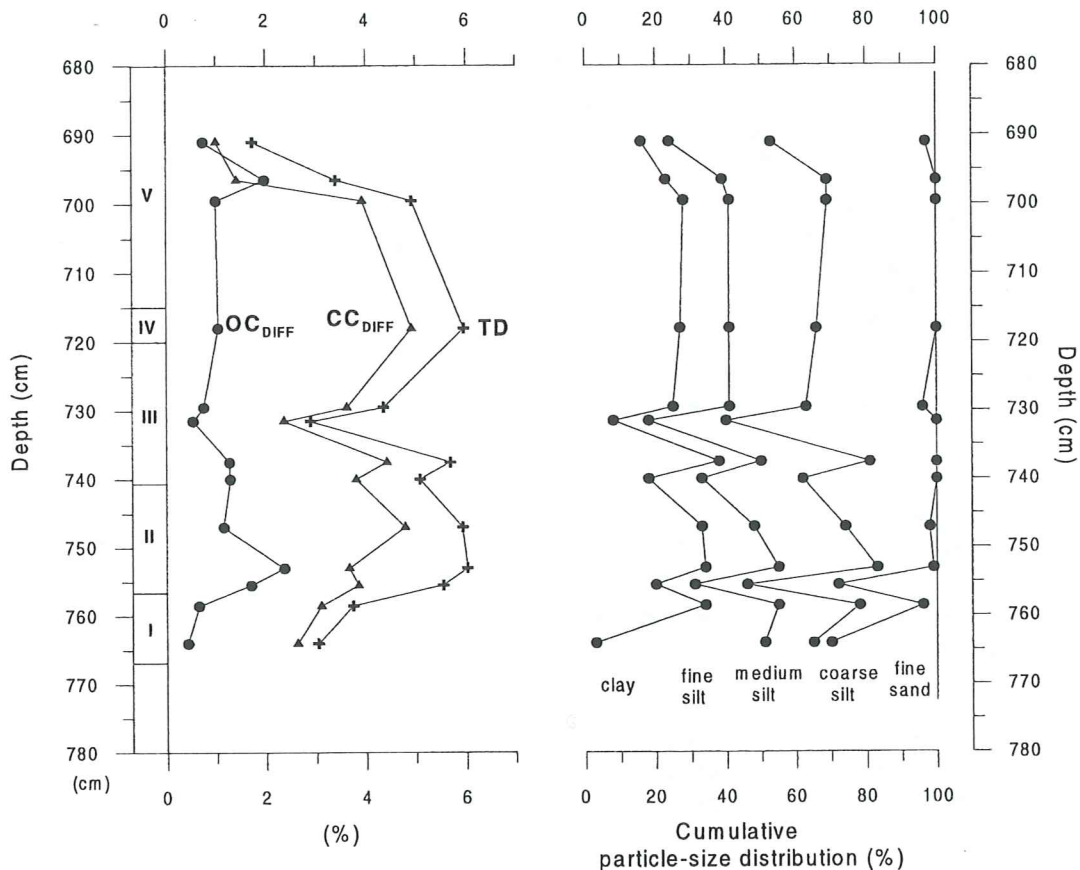
Right: Selected values of carbonate carbon determined automatically ( $CC_{AUT}$ -fill triangles), carbonate content calculated from loss on ignition at 925 °C ( $CC_{LOI}$ -fill circles) and the difference in results between those parameters ( $CC_{DIFF}$ ) plotted against depth. Both carbonate contents are multiplied by 1.36.

The main difference between the two methods are shown by the curves presented in Figure 8. Thus, if the carbonate content determined automatically ( $CC_{AUT}$ ) in an order of <2 % is viewed as the proper carbonate content of the analyzed sediments, then the high carbonate percentages (<7%) derived from loss on ignition at 925 °C ( $CC_{LOI}$ ) lead to a large difference ( $CC_{DIFF}$ ) between those two methods (for further discussions see Fig. 9)

In order to check the assumption that higher percentages of organic carbon are commonly associated with fine-grained sediments, because of the higher adsorption capacities of finer particles and also because the oxidation of organic matter in fine-grained sediments can be retarded due to less penetration of oxygen (see Buckley & Cranston, 1991), the above discussed differences between loss on ignition and total carbon (see Fig. 9a) were related to grain size data at the corresponding levels (Fig. 9b). Moreover, if a fine fraction is present in relatively high quantity, but has a low

carbonate content, because the clay contains up to 5% hydroxyl groups ( $OH^-$ ) which are liberated as water when the sample is heated up over 550 °C, part of the loss (3-4 %) may result from the chemically bound water loss in such samples with high clay and low carbonate content (see Maher, 1998). Thereby, the assumption that the ignition loss between 550-925 °C represents  $CO_2$  evolved from carbonates will be in error by an amount, which is directly proportional to the amount of clay present, and inversely proportional to the amount of carbonates present (see Dean, 1974).

It is evident from Figure 9 that the  $OC_{DIFF}$  curve has more or less the same trend as the curve for the finer fraction. The difference is not so large because the clay fraction is only around 20-30% (compared with 50-70 % in Digerfeldt *et al.*, 2000). For example, Ramrath *et al.* (1999) and Digerfeldt *et al.* (2000) found a weight loss related to the above mentioned loss generators of 5.3% and 7.8%, respectively, whereas in this study the weight loss is 1.06 %.



**Figure 9.** Left: Organic carbon difference ( $OC_{DIFF}$ -circles), carbonate content difference ( $CC_{DIFF}$ -triangles) and total difference (TD-crosses).  $TD = OC_{DIFF} + CC_{DIFF}$   
 Right: Cumulative particle size distribution (%) for selected values.

The most evident correlation between the variable features and the grain-size distribution is suggested by the  $CC_{DIFF}$  curve. It is obvious that a rise in clay content leads to a difference between the methods. This seems to be valid for fine silt as well. For example, in the lower most sample, where the clay content is extremely low and fine silt dominates the grain-size distribution, the  $CC_{DIFF}$  is in an order of  $\sim 3\%$ . The coarse fraction (medium silt, coarse silt and fine sand) has generally the same trend as the finer fraction

and seems not to influence the amplitude of the difference, at least, not as much as the fine fraction.

Total difference (TD) is a composite curve combining the values of both variables presented above. It increases where the fine fraction is high.

To summarize, it is suggested that organic carbon determination by means of loss on ignition should be interpreted with care. If an absolute organic carbon determination is to be made, loss on ignition data must be sustained by total organic carbon determination.

## 8. Conclusions

1. The analysed sedimentary sequence from Atteköps mosse, southwestern Sweden, shows evidence for several climatic fluctuations. During prevailing cold climates the organic matter content decreases, likely as a response to decreasing temperatures, which were accompanied by large inputs of detrital material. The grain size and magnetic susceptibility further support these interpretations. Climatic ameliorations are indicated by an increased organic content of the sediments. These interpretations are in accordance with classical studies dealing with Late Glacial sediments from southern Sweden.
2. The overall trend of all loss on ignition parameters allow a tentative correlation with the GRIP event stratigraphy (Björck *et al.* 1998). A radiocarbon date which places the sediment around 707 cm depth most probably at the GI-1e/GI-1d transition, support this supposition.
3. Loss on ignition is an accurate measurement of primary organic content of sediments, if the fine fraction is present in low percentages.
4. Sediment composition seems to be the main cause which determines the value of the correction factor between loss on ignition and total organic carbon. If an absolute organic carbon determination is to be done, loss on ignition data must be sustained by organic carbon determination.
5. Carbonate carbon determination by means of loss on ignition is strongly influenced by the distribution of the fine fraction. A rise in clay content is closely followed by a rise in extra weight-loss. Care must be taken in the interpretation of carbonate content via loss on ignition as a paleoenvironmental indicator.

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