

Research Papers

No.26

**PALAEOECOLOGICAL EVALUATION OF
THE RECENT ACIDIFICATION
OF SUSCEPTIBLE LAKES:
Methods utilised under DoE contract PECD 7/7/139
and the Royal Society SWAP Project**

A.C. Stevenson, S.T. Patrick, A. Kreiser, R.W. Battarbee

Editors: S.T. Patrick & A.C. Stevenson

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1.0 Introduction

Palaeoecology is an interdisciplinary science and uses a great variety of techniques. Lake acidification studies carried out in the Royal Society Surface Water Acidification Project (SWAP) and Department of the Environment (DoE) Regional Acidification Programme have been especially wide ranging, involving specialists from different laboratories working on material from common sites and common cores. In addition, a number of different laboratories have been involved in performing similar analyses (eg. diatom analysis). In these cases a standard approach to key aspects of the work has been developed but minor differences between laboratories and between countries exist.

This report introduces and describes the methods used and the analyses performed upon lake sediment core material obtained from sites in the UK. Other, non core-based methods, are also briefly described (Section 13). This report therefore complements the results-based reports issued separately in the DoE and SWAP research programmes. A list of reports currently available can be found at the end of this report. Where methods have been altered these alterations will be identified within the individual site-report concerned.

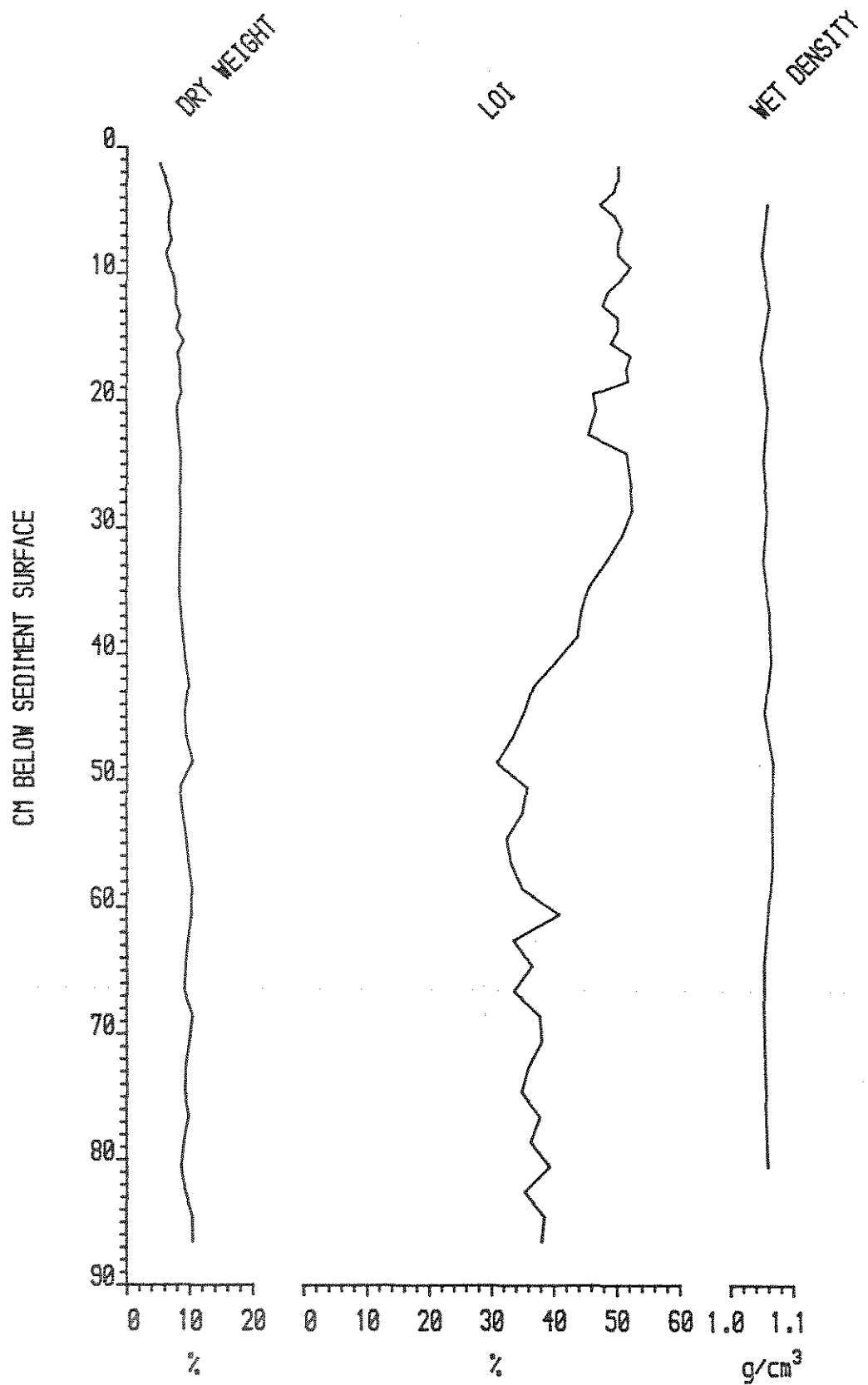
2.0 Coring (PRU University College London; FBA Windermere)

The longer cores which comprise the basis of most of the analyses described in this paper are taken using a Mackereth mini-corer (Mackereth 1969) operated from an inflatable boat. In some cases a portable raft is used so that additional or longer sediment cores can be collected by means of a Livingstone corer. Preliminary and surface sediment samples are collected by means of a Kajak corer and/or Eckman grab.

3.0 Core extrusion & routine measurements of sediment characteristics (PRU University College London; FBA Windermere)

Cores are extruded in the laboratory and sliced at 0.5 cm or 1 cm intervals. The slices are then subsampled for percentage dry weight, percentage loss on ignition and wet density measurements. Marked changes in these parameters can indicate catchment disturbance. For example, peatland drainage may result in greatly increased loss on ignition values, whereas sub-soil erosion would increase the wet density and percentage dry weight values. Sediment bulk density is also required for calculating accumulation rates. The percentage dry weight is obtained by weighing approximately 2 g of wet sediment from each slice in a pre-weighed crucible, drying the sediment at 105°C for 24 hours and re-weighing the crucible. The percentage loss on ignition is determined by placing the crucible containing the dried sediment in a muffle furnace at 550°C for 2 hours. At UCL wet density in g cm^{-3} is obtained by filling a pre-weighed 2cm^3 container with wet sediment which is then re-weighed. An alternative method of determining density is the volumetric displacement of water by a wet sub-sample. This method is employed at the FBA. Results of these analyses are plotted in the form depicted in Fig. 1.

Fig. 1. Example plot of sediment characteristic data (L. Laidon)



4.0 Diatoms (A. Kreiser, S. Fritz, V.J. Jones, R.J. Flower and E.Y. Haworth)

In order to assess whether lake acidification has occurred it is necessary to ascertain the magnitude and rate of change of lake acidity in the past. This information may be contained to some extent in past chemistry measurements and records of fishery status although both these sources have limitations. Lake chemistry records are either not available or tend to be inadequate in that sampling is often too infrequent to provide a true value for mean annual pH. The impact of the more acidic episodes of precipitation which occur during the winter may be missed, particularly if the lake is remote and difficult to sample. In the longer term inaccuracies in the data may exist due to the less accurate methods of pH measurement used in the past. Evidence for acidification may be found in reports of declining fish catches although these records should also be interpreted with caution since other factors, such as increased angling pressure or loss of spawning habitat may be responsible.

It has been known for some time that the structure of diatom communities is related to water chemistry and in particular to pH (Hustedt, 1937-1939). This relationship can be quantified by examining the diatom communities present in lakes where the pH of the water is known. The siliceous valves of the diatom cells have the advantage of being well preserved and abundant in the sediments of acid, oligotrophic lakes where silica dissolution is minimal and sediment accumulation slow. When examined under a light microscope each diatom valve has a distinctive combination of features characteristic of a particular taxon. It is therefore possible to use these preserved diatom assemblages in a dated core to reconstruct changes in the lake water pH in the past. This technique has advantages over other sources of water quality history in that the record is continuous and the working methods are consistent over the period represented by the sediment. Diatom analyses has been performed primarily by researchers at UCL. In general it is the preparation and counting techniques used at UCL that are described below. Colleagues in other laboratories employ variations on some of these techniques. Taxonomic and interpretation methodologies have been harmonised at workshops at UCL involving all diatomists working on the DoE and SWAP projects.

4.1 Preparation

Between 0.05 g and 0.1 g of dried sediment is weighed into a 250 ml beaker and oxidised with approximately 20 ml of 30% hydrogen peroxide warmed on an electric hotplate set at 90°C. The time taken to remove all organic matter varies but the process is usually completed in 2-3 hours. The samples are washed by concentration in a centrifuge set at 1200 rev min⁻¹ for four minutes followed by resuspension in distilled water. This is repeated at least three times. Coarse mineral material can be removed by decanting off the diatom and fine mineral suspension. If there is clay present this is largely removed by keeping the centrifuge time to the minimum required to separate the diatoms, leaving the clay in suspension. This suspension is checked with a microscope for fine diatom fragments before being discarded. After washing the sample is diluted with enough distilled water to produce a suitable working concentration. Approximately 0.5 ml of suspension is placed on a round 19 mm diameter cover slip and allowed to dry overnight in an undisturbed location away from dust sources. The cover slip is mounted on a glass slide using Naphrax or Mikrops 163 mounting mediums.

The method utilised at the FBA differs in that wet sediment is oxidised using a chromic acid and hydrogen peroxide mixture. In addition, the fraction of the prepared sample used to make the final slide is measured with particular accuracy as this is used to calculate diatom concentrations.

The diatoms are identified and counted using a research quality microscope with phase contrast and a 100x oil immersion objective, providing a magnification of >1000x. At least 500 valves are counted from each sample. The counts for individual taxa are expressed as percentages of the total count, and plotted for each sample level down the sediment core (Fig. 2). Principal floras used in identification are Hustedt (1930), Hustedt (1930-1966), Cleve-Euler (1951-1955) and Patrick and Reimer (1966). The unpublished floras compiled by the USA Palaeoecological Investigations of Recent Lake Acidification (PIRLA) Project are also utilised.

4.2 Diatom concentration ←

If the rate of sediment accumulation in a lake is known, then the annual rate of diatom accumulation can also be calculated provided the concentration of diatoms in the sediment is determined. This is useful in assessing changes in diatom productivity independent of variations in sediment accumulation rate. Absolute diatom counts are also valuable in the interpretation of percentage data since they enable real changes in the abundance of a particular taxon to be distinguished from the apparent changes caused by fluctuations in other taxa. The concentration of diatoms in the dry sediment can be determined by the addition of markers of similar size and concentration to the diatoms in the sample preparation procedure (Battarbee and Kneen 1982).

The concentration of diatoms in the sediment is calculated by the addition of a known number of 6.4 μ polydivinyl benzene microspheres, from a stock suspension calculated on a Coulter counter to contain 4.4×10^6 spheres ml^{-1} . This suspension is shaken thoroughly and sonicated for 30 seconds before use to ensure even distribution of the spheres throughout the suspension. Initially 1.5-2.0 ml of suspension are added to the prepared complete sample and a test slide made to examine the diatom/microsphere ratio. The two components should be present in roughly equal proportions. More microspheres can be added to the sample suspension if required prior to making up the final slides for counting.

The spheres are counted at the same time as the diatoms. They can be distinguished from chrysophyte cysts by their denser appearance and lack of any surface features. The total number of diatoms contained in the sample processed can be calculated thus:

$$\text{Diatoms in sample} = \frac{\text{total No. spheres in sample}}{\text{No. of spheres counted}} \times \text{No. diatoms counted}$$

At the FBA total diatom concentrations are determined by counting the total number of valves in one transect of known area and calculating this as a proportion of the total number of diatoms on the slide. The concentrations of taxa that are of specific interest are calculated by scanning the whole slide.

4.3 pH reconstruction

The classification of diatoms by pH preference was first proposed by Hustedt (1937-1939) who allocated diatom taxa to one of five pH categories according to the pH of the water in which they were found growing. These categories were defined as:

1. Alkalibiontic (alb): occurring at pH values >7.
2. Alkaliphilous (alk): occurring at pH about 7 with widest distribution at pH >7.
3. Indifferent (circumneutral) (ind): equal occurrences on both sides of pH 7.
4. Acidophilous (acf): occurring at pH about 7 with widest distribution at pH <7.
5. Acidobiontic (acb): occurring at pH <7, optimum distribution at pH 5.5 and under.

This classification forms the basis of most studies of diatom pH preference and pH reconstruction.

4.3.1 pH and surface sediment assemblages

Before the past pH of a lake can be reconstructed from the sub-fossil diatoms it is necessary to quantify the relationship between diatoms and pH. This can either draw on pH preferences described in the literature or on studies of diatom distributions in surface sediments. The latter involves the construction of a data set of surface diatom assemblages and pH values from as many lakes as possible within the study region. Ideally the data set should be used to test pH reconstruction techniques on cores taken from lakes within the same region to eliminate any regional differences.

4.3.2 Index B

Based on earlier work by Nygaard (1956) and Merilainen (1967), (Renberg & Hellberg (1982) devised an index (Index B) whereby the degree of pH preference in a diatom assemblage is weighted as follows:

$$\text{Index B} = \frac{\% \text{ind} + (5 * \% \text{acf}) + (40 * \% \text{acb})}{\% \text{ind} + (3.5 * \% \text{alk}) + (108 * \% \text{alb})}$$

The model was tested on 30 lakes in Scandinavia of known pH and the Log Index B was shown to have a linear relationship with pH:

$$\text{pH} = 6.4 - 0.85 \text{ Log Index B} \quad r^2 = 0.91$$

This equation has also proved satisfactory in reconstructing core pH in Galloway (Flower and Battarbee, 1983) and can be used with pH preferences from literature sources. However, a greater degree of accuracy was achieved by calibrating Index B with a surface sediment data set from 34 Galloway lochs in the Galloway region. The equation was redefined thus:

$$\text{pH} = 6.3 - 0.86 \text{ log Index B} \quad \text{where } r^2 = 0.82$$

based on the regression of Index B with mean summer lake water pH values.

4.3.3 Multiple regression

The data set has also been used to carry out a multiple regression analysis of both preference groups and individual key taxa against mean summer pH. The multiple regression of preference groups against pH in Galloway produces the equation:

$$\text{pH} = 7.82 - 0.037\%acb - 0.035\%acf - 0.013\%ind - 0.015\%alk + 0.1\%alb.$$

where $r^2 = 0.82$

These pH reconstruction methods are currently employed at UCL to reconstruct pH in both Welsh and Scottish lakes. The surface sample data set is currently being expanded to include lakes from Wales, thus increasing the accuracy of pH reconstruction.

5.0 Core dating - radiometric methods (P.G. Appleby)

For each site an empirically tested and carefully evaluated chronology of sedimentation for at least the past 150 years is constructed. As far as is possible these chronologies accommodate any evidence for sediment mixing, sediment source shifts and post depositional sediment redistribution.

5.1 ^{210}Pb

^{210}Pb occurs naturally in lake sediments as one of the radioisotopes in the ^{238}U decay series. Its half-life of 22.26 years makes it well suited to dating sediments laid down over the past 100-150 years. The total ^{210}Pb activity in sediments has two components. Supported ^{210}Pb is that component of the activity which derives from in situ decay of the parent isotope ^{226}Ra within individual soil particles. The second component, called unsupported ^{210}Pb , derives from dissociation of ^{210}Pb from ^{226}Ra and the through diffusion of the intermediate gaseous isotope ^{222}Rn . Pathways by which unsupported ^{210}Pb accumulates in lake sediments are discussed in detail in Oldfield and Appleby (1984). In most samples the supported ^{210}Pb can be assumed to be in radioactive equilibrium with ^{226}Ra and the unsupported activity at any level is obtained by subtracting the ^{226}Ra activity from the total ^{210}Pb . Fig 3a shows the total and supported ^{210}Pb activity v depth for a sediment core from Llyn Llgi in north Wales. The unsupported ^{210}Pb activity in the core is shown in Fig. 3b.

^{210}Pb dates for each sediment core are calculated using both the constant rate of ^{210}Pb supply (CRS) model and the constant initial ^{210}Pb concentration (CIC) model (Appleby and Oldfield 1978). The first of these models is perhaps the most widely accepted. It assumes that the ^{210}Pb supply is dominated by direct atmospheric fallout, resulting in a constant rate of supply of ^{210}Pb from the lake waters to the sediments irrespective of changes in the net dry mass accumulation rate. This model will not be valid where there are interruptions to the ^{210}Pb supply owing to, for example, sediment focusing or a sediment hiatus. In these cases dates are calculated either by the CIC model (where there is evidence for a constant primary accumulation rate) or by using a composite of both models. Factors governing model choice are set out in Appleby and Oldfield (1983) and Oldfield and Appleby (1984). Fig 4 shows ^{210}Pb dates v depth and dry mass accumulation rates for Llyn Llgi calculated by the CRS and CIC models. The accumulation

rate appears to have been reasonably uniform over the past 140 years and for this reason both models give similar chronologies.

5.2 ^{137}Cs and ^{241}Am dating

^{137}Cs activity in sediments predating the 1986 Chernobyl accident derives mainly from atmospheric weapons testing fallout. Where this isotope is strongly adsorbed on to sediments the shape of the activity v depth profile is presumed to reflect varying fallout rate and the onset of fallout in 1954 and peak fallout in 1963 provide useful additional chronological markers. In practice many recent studies (eg. Davis et al. 1984) have shown that under conditions of low pH ^{137}Cs is relatively mobile within the sediment column with maximum concentrations too close to the surface to represent 1963 and residual values at levels too deep to represent 1954. In these cases ^{241}Am measurements may provide a useful alternative. This isotope also derives from nuclear weapons fallout and a growing data set from lakes with a wide range of pH values suggests that it is considerably less mobile than ^{137}Cs . Fig. 5 shows the ^{137}Cs and ^{241}Am profiles for Llyn Llgi. Both profiles have a well defined maximum at 4.25 cm. This level is dated by ^{210}Pb to 1965-1968 and confirms the association of this feature with the 1963 fallout peak.

5.3 Detector system

Sediments from each core are measured for ^{210}Pb , ^{226}Ra , ^{137}Cs and ^{241}Am by direct gamma assay using an Ortec HPGe GWL-80210-S well-type coaxial low background intrinsic germanium detector (Appleby et al. 1986). Background suppression is achieved by means of a 100 mm thick lead castle, a 305 mm diameter * 305 mm long sodium iodide (NaI(Tl)) escape suppression shield and a 3 mm thick copper cylinder fitting over the end of the detector and sample. The configuration is shown in Fig. 6. Samples (with masses ranging from 0.5 g to 3 g) are placed in specially fabricated plastic sample holders designed to fit inside the well recess of the detector.

Data are acquired using an ADC (analogue to digital computer) interfaced to a BBC microcomputer and then transferred to an IBM 3083 computer for analysis. The system achieves a resolution of 1.5 KeV at 100 KeV and 2 KeV at 1 Mev. Fig. 7 shows a typical gamma spectrum. ^{210}Pb is measured by its gamma emissions at 46.5 KeV and ^{226}Ra by the 352 KeV gamma rays emitted by its daughter isotope ^{214}Pb . ^{137}Cs and ^{241}Am are measured by their emissions at 662 KeV and 59.5 KeV respectively. The absolute efficiency of the detector has been measured using a series of calibrated sources and sediment samples of known activity. The effect of self-absorption of low energy gamma rays has been estimated by determinations of the relative efficiency at 46.5 KeV and 352 KeV, using samples of different weights in which ^{210}Pb and ^{226}Ra are known to be in radioactive equilibrium. The background counts are carried out at regular intervals to ensure maintenance of the low background characteristics.

Total ^{210}Pb Conc v Depth

LAI1



Unsupp ^{210}Pb Conc v Depth

LAI1

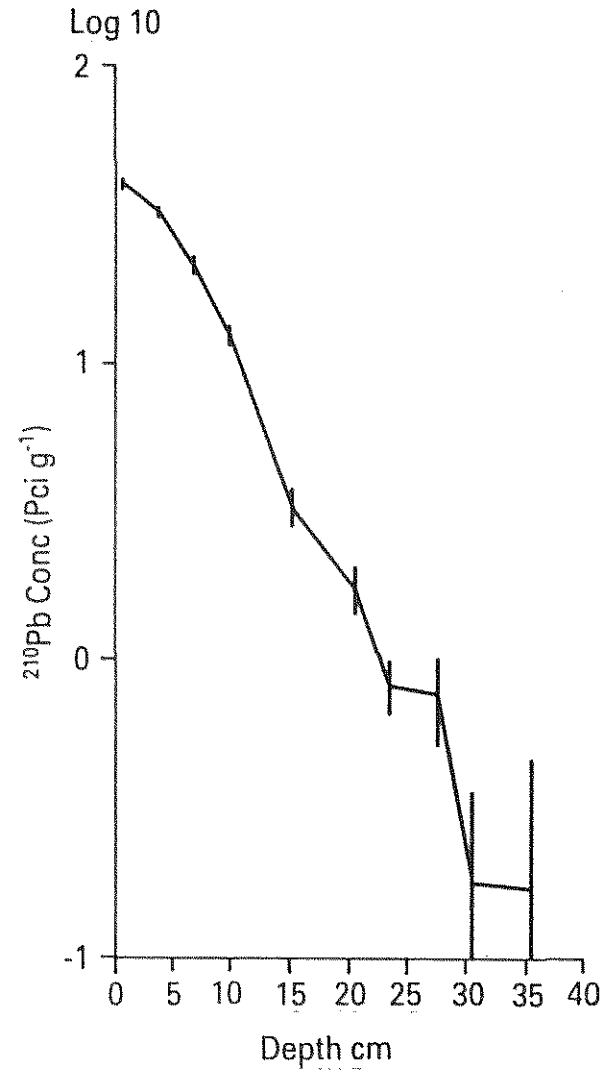
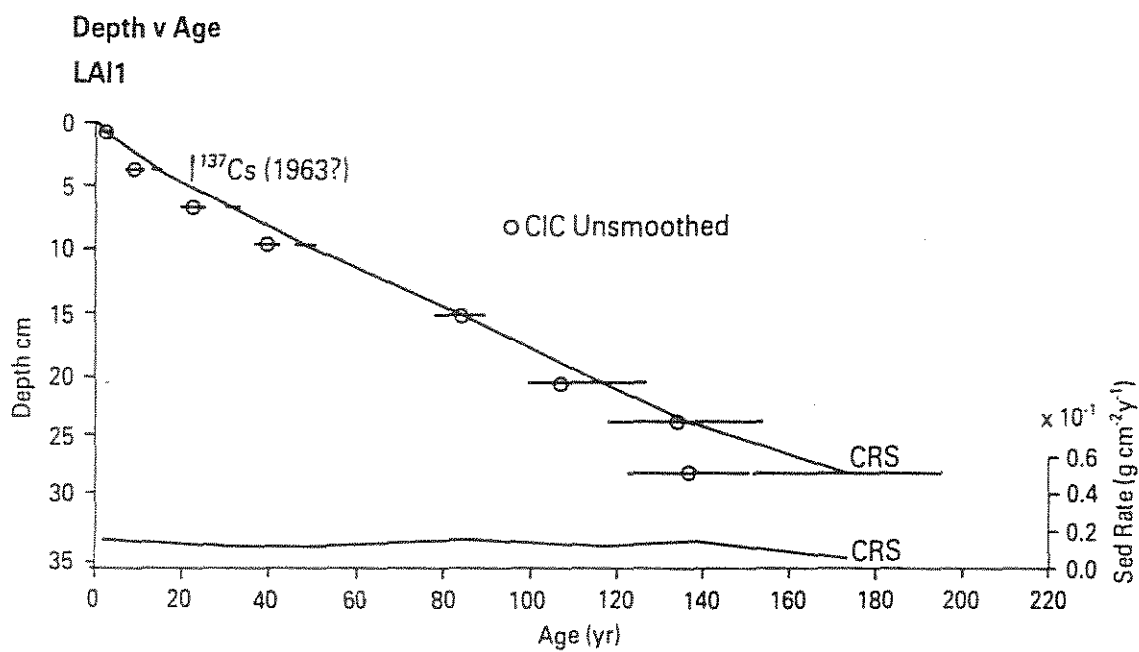
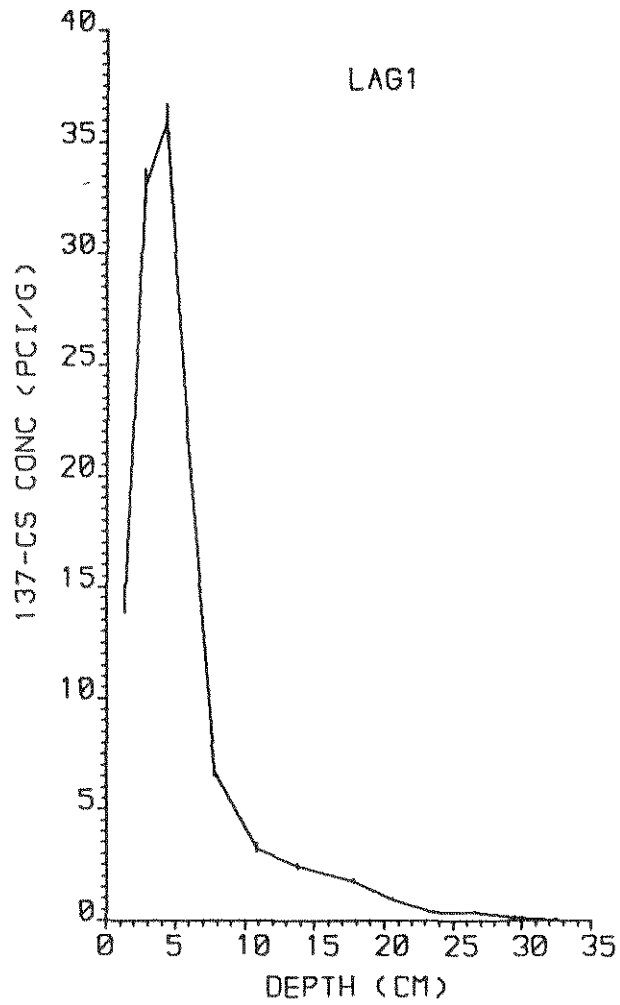


Fig. 3. ^{210}Pb profiles (L. LAI1):
(a) Total and supported ^{210}Pb , and
(b) unsupported ^{210}Pb

Fig.4. Depth v ^{210}Pb age and dry mass accumulation rates given by the CRS and CIC models, (L. Llagi)



(a) LLYN LLAGI
CS-137 CONC V DEPTH



(b) LLYN LLAGI
AM-241 CONC V DEPTH

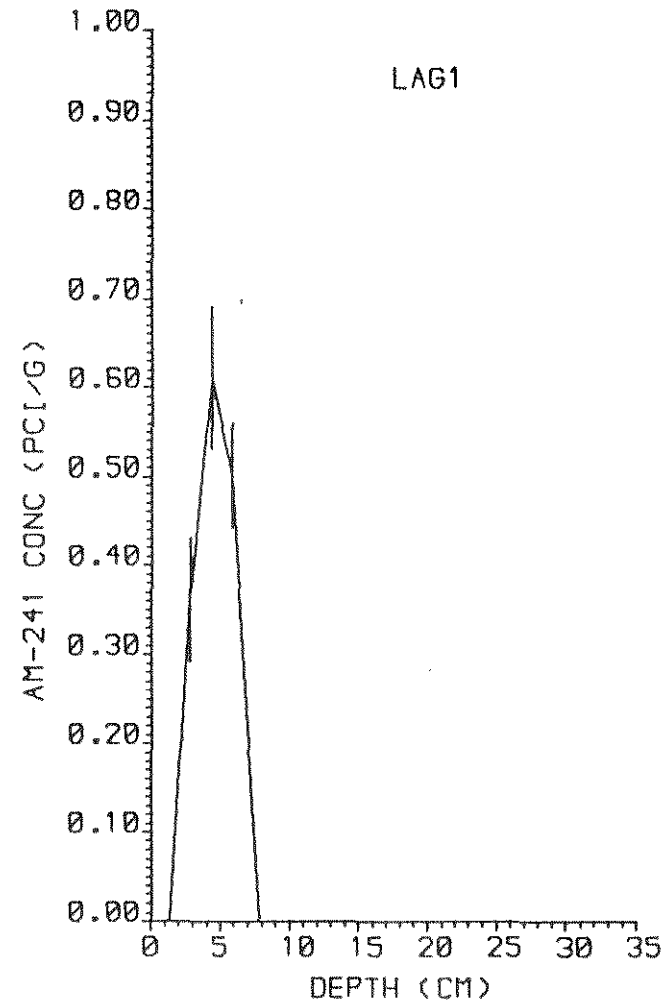
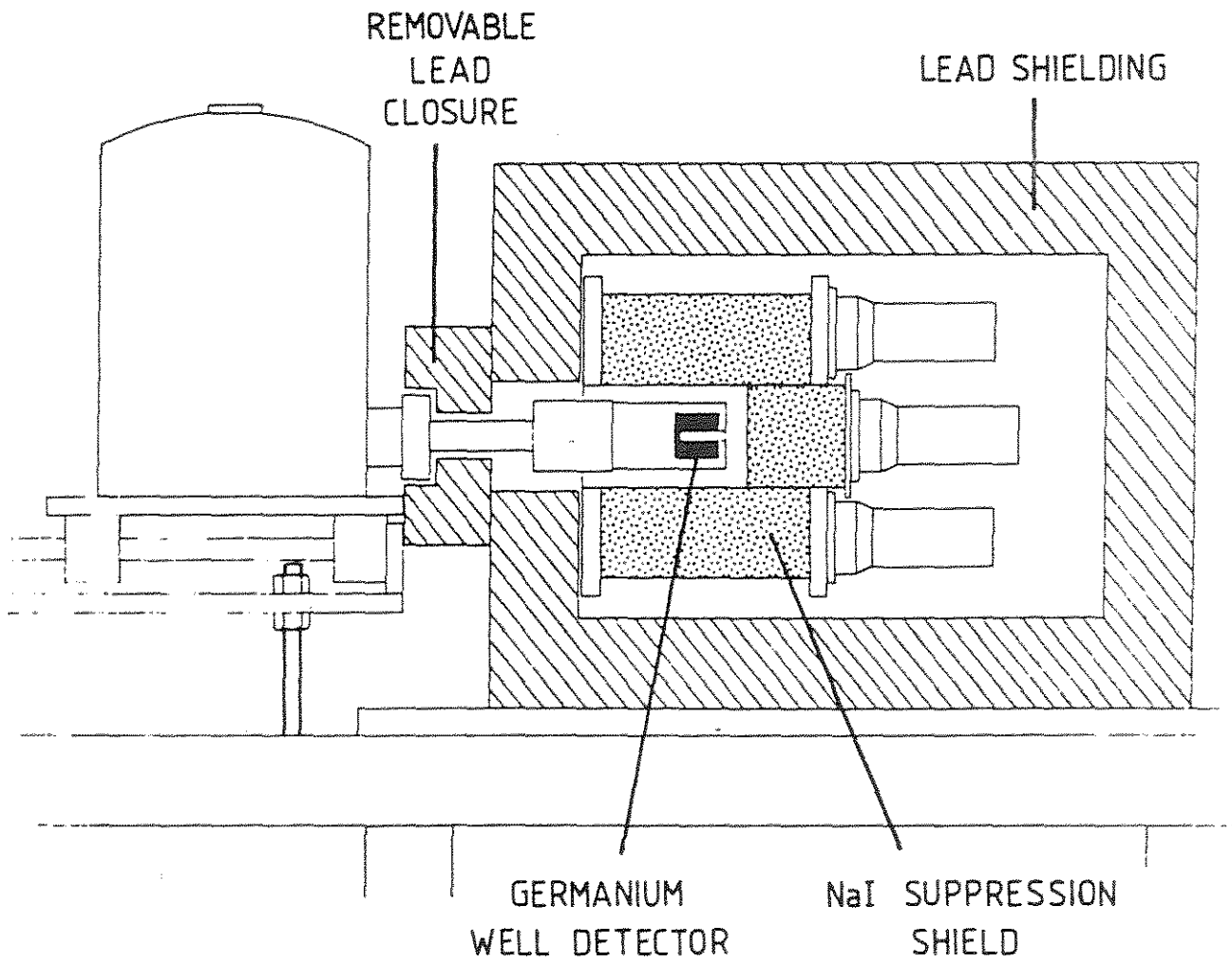


Fig.5. L. Llagi: (a) ¹³⁷Cs and (b) ²⁴¹Am profiles

Fig.6. The configuration of the Liverpool gamma based ^{210}Pb dating system



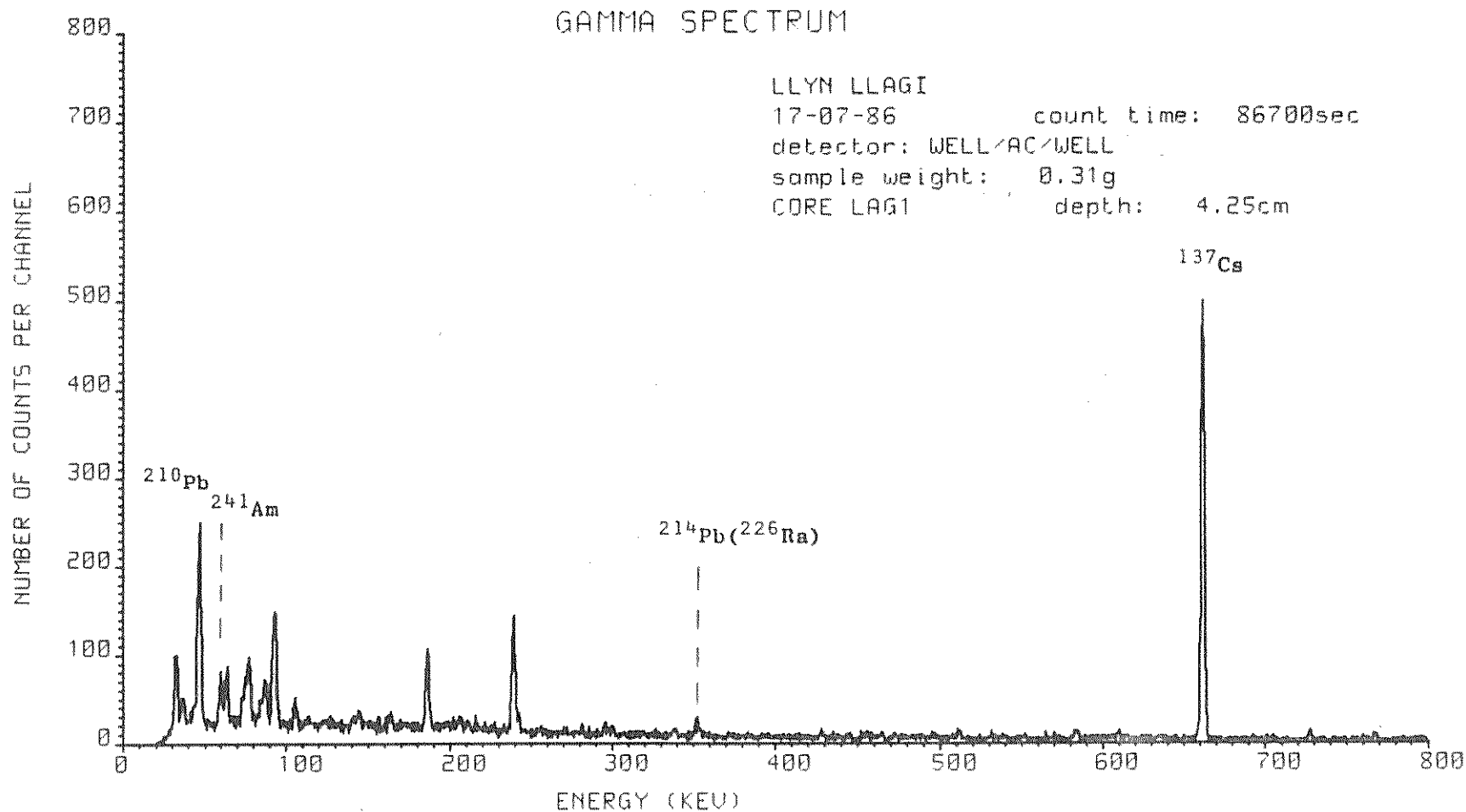


Fig. 7. Gamma spectrum for a sediment sample measured on the well detector

6.0 Magnetic measurements of lake sediment (F. Oldfield and S.R. Higgitt)

Several studies (Oldfield *et al.* 1978, 1979, 1984; Jones 1985; Jones 1986; Thompson and Oldfield 1986) have confirmed that recent peats accumulating in mires which have been dependent entirely on atmospheric sources for mineral inputs, contain a record of particulate pollution readily detectable by means of magnetic measurements. A range of industrial processes, including iron and steel manufacture, solid fuel-fired power generation and non-ferrous metal smelting give rise to particulate emissions with high ferrimagnetic concentrations (Jones 1985). Scanning Electron Microscope studies confirm that in peats post-dating c. 1950 AD, the magnetic particles are predominantly spherical and, in terms of size, shape and surface patterning most are indistinguishable from power station fly ash (Hunt in prep., Jones 1985). Magnetic measurements can therefore contribute to the reconstruction of particle emissions associated with major SO₂ generating industrial processes.

For each site a full mineral magnetic characterisation of the recent sediments, of the lake catchment and of atmospheric deposition in the area is provided. These studies contribute a preliminary basis for core correlation and for selecting/rejecting cores for further study; evidence for shifts in allochthonous sediment sources resulting from changes in land use or catchment hydrology; evidence for sediment redistribution, disturbance or focusing within the lake; and a record of the atmospheric deposition of magnetic particles resulting from fossil fuel combustion and industrial processes during the last 200 years. Where fresh undisturbed sediment is available from lake cores in oriented 'cubes', measurements of natural remnance (NRM) can be obtained. Where a sufficiently strong, stable and repeatable NRM trace is present, it can be used to supplement chronologies based on radiometric methods.

Studies of lake and estuarine sediments at a wide variety of sites (Thompson *et al.* 1975, Dearing 1979, Higgitt 1985, Oldfield *et al.* 1985, Thompson and Oldfield 1986) show that in many types of geological context, the magnetic properties of most late Holocene sediments are dominated by catchment-derived, erosive input. They can therefore often be used as evidence for catchment disturbance and resulting changes in allochthonous sediment sources. In this way, variations in magnetic mineral concentrations and assemblages can indicate the effects of human activity within the catchment.

6.1 Noise and contamination

Magnetic characterisation of lake sediment samples has been largely based on room temperature remanence measurements. This is a response to the problems posed by variable and often very small sample size (frequently <0.5 g) and generally low ferrimagnetic concentrations. This combination precludes reliable susceptibility measurements, since many of the readings obtained fall within the same range as the noise level on the most sensitive instruments; moreover they are comparable in magnitude to the diamagnetic effect of the styrene sample holder plus any included plastic foam packing. Susceptibility measurements have therefore been used only at sites with relatively high ferrimagnetic concentrations.

Given such small, weakly magnetic samples, even measuring remanences can pose serious problems of noise and contamination. During the measuring procedures, sample holders (10 ml) and packing materials (cellophane film and plastic foam) are inevitably magnetised along with the sample; consequently any magnetic contamination will contribute to the measured remanence. In order to minimise this effect, all sample pots and packing materials are screened before use by means of isothermal remanence (IRM) measurements carried out after magnetisation in a field of 300 mT. Only pots with a total IRM (after washing in distilled water and subsequent magnetisation) of less than $1 \times 10^{-6} \text{Am}^2$, or in the case of sites with the lowest sample sizes, less than $0.5 \times 10^{-6} \text{Am}^2$, are accepted for the project. In preparation for the sequence of measurements outlined in Fig. 8, sediments are immobilised by 'bedding down' in 10 cm³ pots with plastic foam. As a further precaution against particle movement, bearing in mind that samples were often very small, the sediments were initially wrapped in cellophane film. Packing materials are harder to screen with complete confidence than are the sample pots themselves. The cellophane film used to wrap the sediments has been found to have a consistently negligible IRM after magnetisation and is therefore not extensively screened during this project. However, a 10 cm³ pot full of the type of plastic foam used would typically have an IRM of $1-1.5 \times 10^{-6} \text{Am}^2$ after being magnetised in a field of 300 mT. Plastic foam is screened in pots for each sample and only pots and foam with a combined IRM_{+300 mT} below $1.0 \times 10^{-6} \text{Am}^2$ are used. Isothermal remanence (IRM and SIRM) measurements are all at least an order of magnitude above the maximum combined pot-plus-packing contamination level and between one and two orders of magnitude above the noise level of the Minispin Magnetometer employed in the study. Anhysteretic remanence (ARM) measurements pose rather more complex problems. The AF field of 100 mT used during anhysteretic magnetisation has the effect of demagnetising virtually all the isothermal remanence grown through the magnetisation of any impurities during the screening procedure. However ARM values for actual samples are typically one to two orders of magnitude less than IRMs. As a result of these differences, whereas the effective limits of reliable detection for IRM measurements reflect contamination rather than instrument noise levels, in the case of ARM measurements the reverse is the case. Moreover, the low ARM readings for many pre-20th century samples are actually within the instrument noise range. The effect of this, especially on calculated remanence quotients (SIRM/ARM), has limited the significance of variations in these values for the smallest and the most weakly magnetic samples.

6.2 The measurements and their interpretation

Fig. 8 depicts the procedures which provide the following measurements and quotients that form the basis for the main parameter vs depth plots (see eg. Fig. 9):- ARM, SIRM, SIRM/ARM, IRM_{-20mT}/SIRM, IRM_{-40 mT}/SIRM, IRM_{-100 mT}/SIRM, IRM_{-300mT}/SIRM.

ARM and SIRM are plotted as $10^{-6} \text{Am}^2 \text{kg}^{-1}$. IRM_{-n mT}/SIRM values are graphed as percentage reverse saturation. Thus 50 is the point at which IRM_{-n mT}/SIRM is zero and 100 is the point at which IRM_{-n mT}/SIRM is -1.

ARM measurements are especially sensitive to ferrimagnetic grains of true stable single domain dimensions. For magnetite, this implies a diameter of between c. 0.03 and 0.1 μm . Previous experience has shown that fine grains within this narrow size range are more likely to be typical of secondary

sources (eg. soils and weathering products) or of authigenic bacterial magnetite, than of either primary-bedrock or particulate pollutant-derived material. SIRM measurements can include significant contributions from the full range of remanence carrying minerals, both ferrimagnetic (eg. magnetite and maghaemite) as well as imperfect anti-ferromagnetic (eg. haematite and goethite). Thus it is very important to find some convenient way of characterising the different components and their relative contributions. This can be done through the $IRM_{-n\text{mT}}/SIRM$ plots which are closely related to the concept of coercivity of remanence - $(B_0)_{CR}$. In simple terms, as the plots within the box shift to the right, they reflect the increasing relative importance of magnetic components with a 'soft' remanence, eg. coarse or even very fine ($\sim 0.02 - 0.03 \mu\text{m}$ diameter) ferrimagnets such as magnetite. As the plots shift to the left, especially if the shift involves the $IRM_{-300\text{mT}}/SIRM$ curve, they are likely to reflect the increasing relative importance of the magnetic components with a 'hard' remanence eg. fine grained haematite. In practice the $IRM_{-n\text{mT}}/SIRM$ plots are often both more complex and more informative than this and they help to provide a good basis for a first appraisal of magnetic mineral and grain size variations in the sample set. $SIRM/ARM$ may reflect the influence of many variables, but in sample sets where the magnetic properties are dominated by ferrimagnets, the quotient will often respond to magnetic grain size variations, with high quotients indicating relatively higher multidomain vis a vis stable single domain contributions and vice versa.

A further stage in the use of the data has been the separation of the two extreme ('soft' and 'hard') components in the SIRM and their calculation on a mass specific basis. This involves calculation of $SIRM - IRM_{-20\text{mT}}$ and $SIRM + IRM_{-300\text{mT}}$ respectively. The first may be regarded as reasonably proportional to the total ferrimagnetic ie. 'magnetite' concentration in the sample, although it is likely to favour multidomain components. The second is the best indicator of changing imperfect anti-ferromagnetic ('haematite') concentrations available within the data set. These two components can be regarded as essentially independent of each other, thus their variations can be used as rough indications of the changing concentrations of the two different magnetic mineral types. These same values, like SIRM, can also be recalculated as annual magnetic accumulation rates thus giving estimates of changing net depositional flux of magnetic minerals to the lake bed (cf. Fig. 10).

The data set also provides further options for sample discrimination using cross-plots, both normalised and non-normalised, between pairs of measurements or quotients. These can help to discriminate particular sample assemblages and source types as well as to infer variations in the proportions of the contributing magnetic mineral types.

A general introduction to magnetic studies of this type and to the interpretation of the results, is given in Thompson and Oldfield (1986) which also includes a comprehensive glossary and reference list.

Fig.8. The magnetic measuring sequence

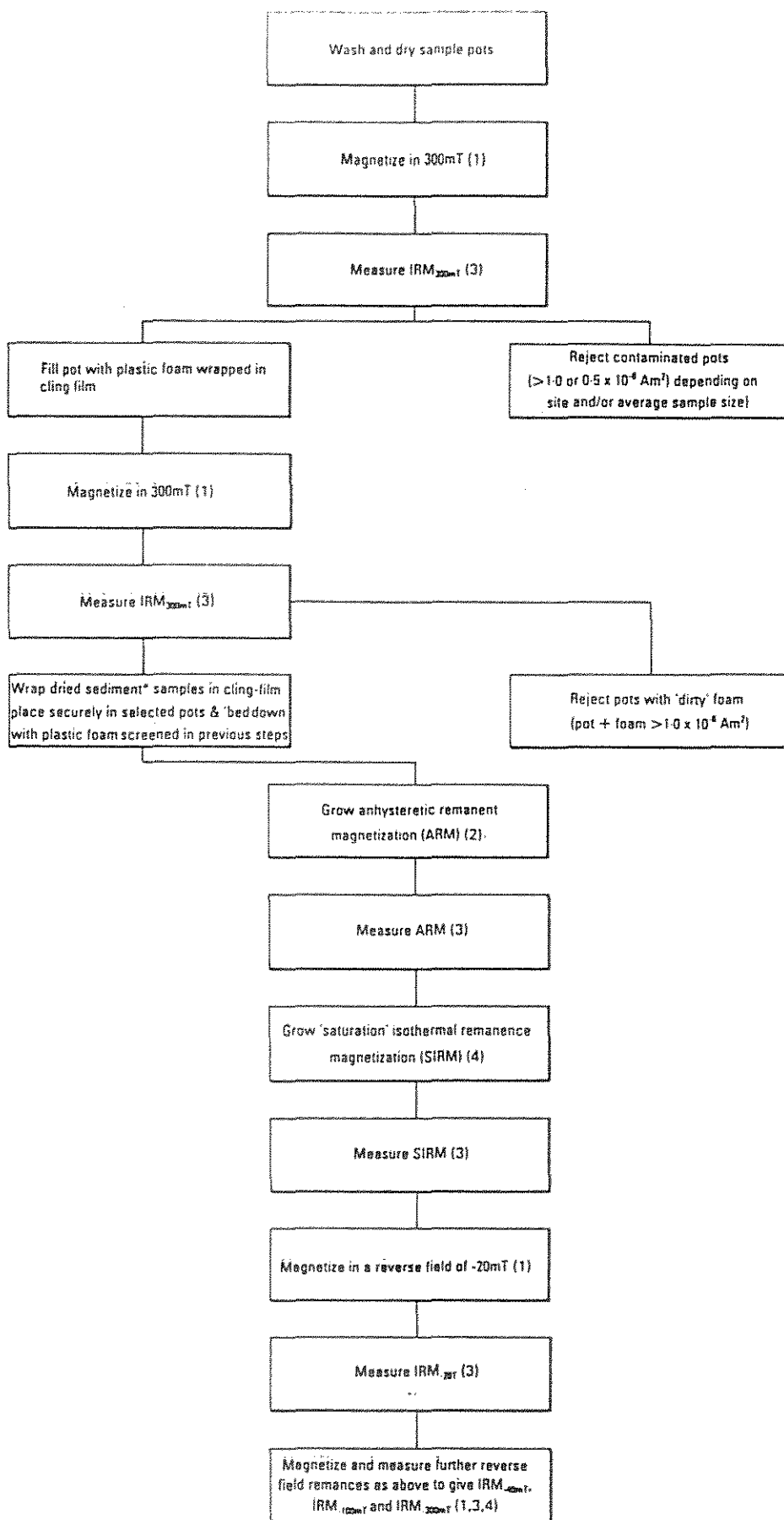


Fig.9. Magnetic remanence measurements for a core from L. na Achlaise

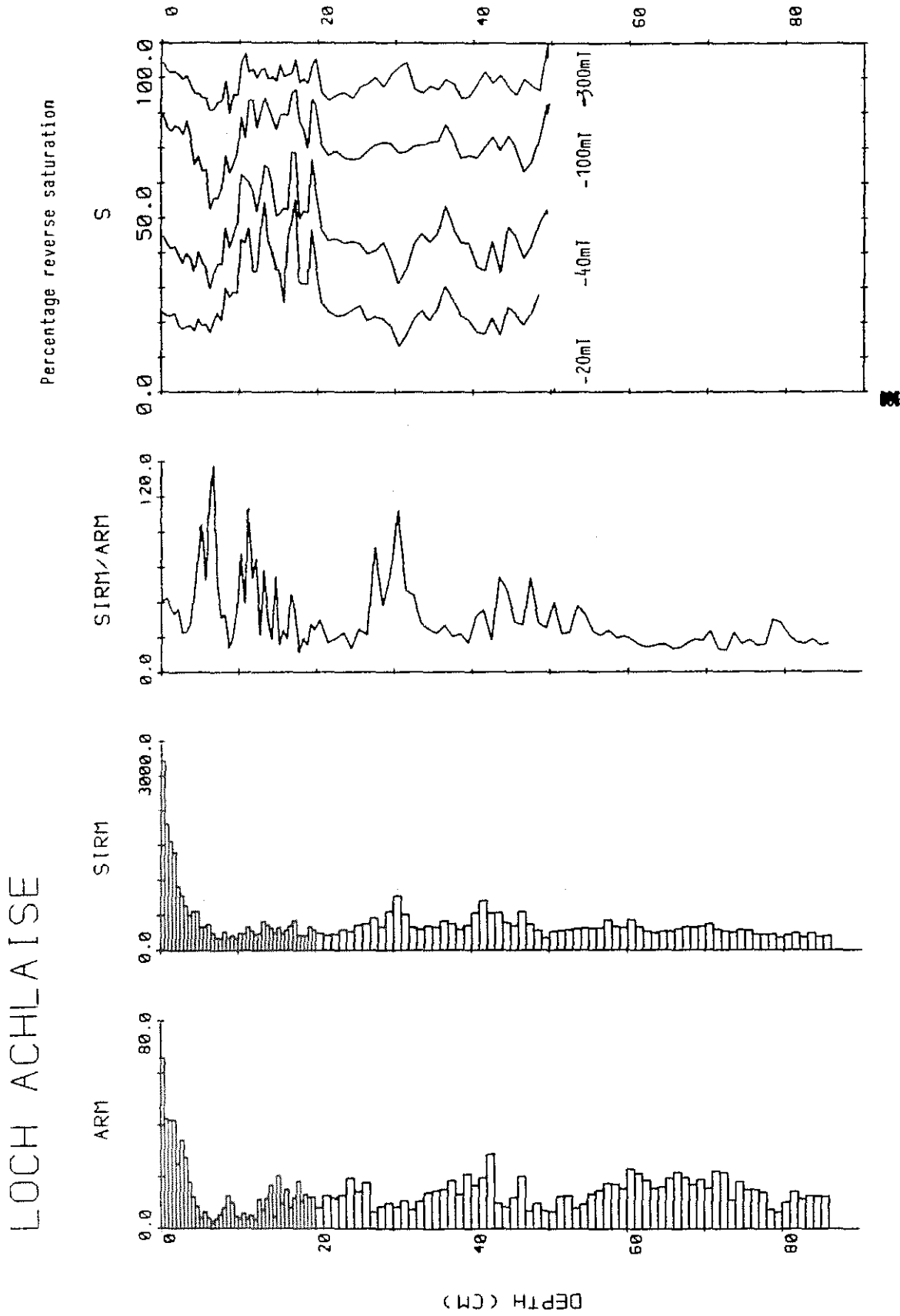


Fig.10. Magnetic accumulation rates for a sediment core from L. na Achlaise. All values are 10^{-6} Aa^{-1}

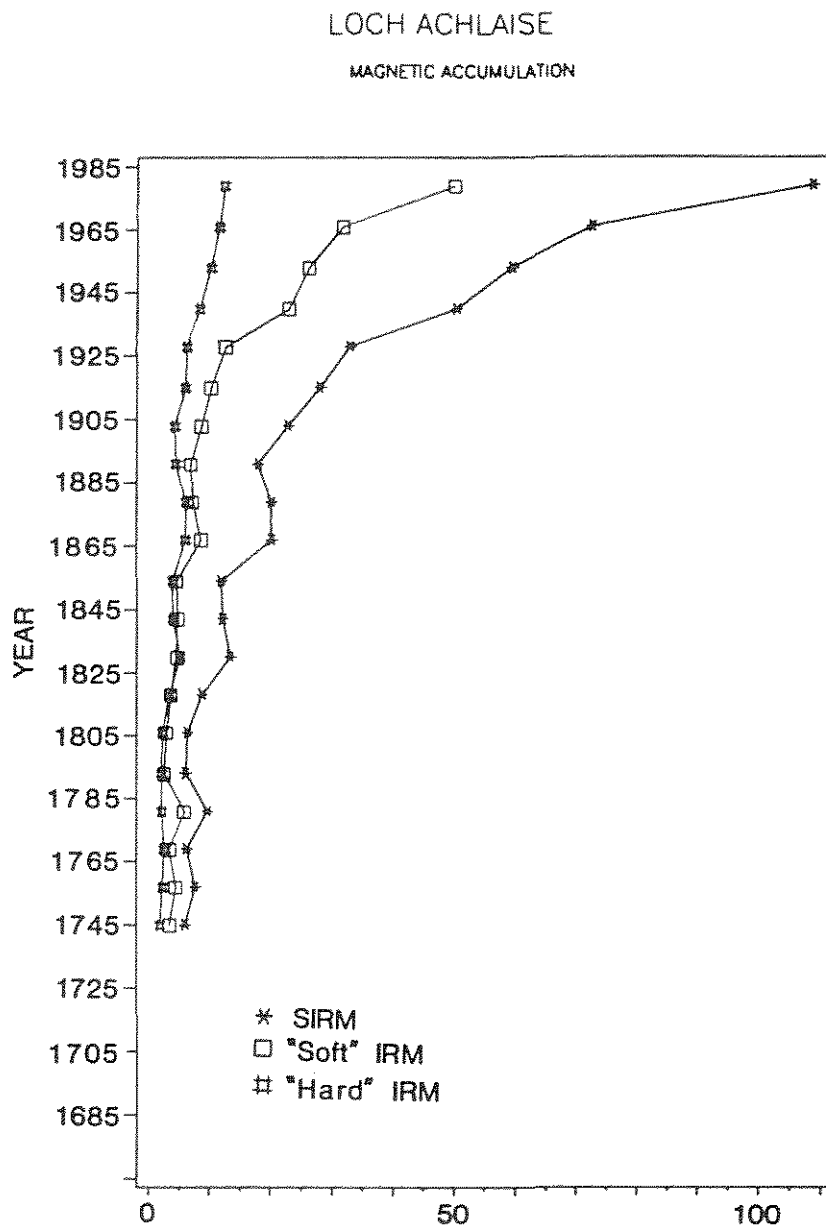


Table 1 Key to Fig. 8. (The magnetic measuring sequence)

- (1) Samples magnetized in a Molspin Pulse Magnetizer with a maximum field of 300 mT.
 - (2) Samples magnetized in a Molspin AF demagnetizer with ARM attachment using a peak AF field of 100 mT and a DC bias of 0.04 mT.
 - (3) Measurements carried out in a Minispin portable slow speed spinner fluxgate magnetometer.
 - (4) Samples magnetized in a Molspin Pulse Magnetizer with a maximum field of either 850 mT or 1000 mT.
- * Where possible the sediments are gently ground before packing into cellophane. In the case of samples which are to be used afterwards for 'soot particle' analysis, sediment fragments are not ground but are simply wrapped tightly in cellophane.

7.0 Sediment Chemistry (B. Rippey)

7.1 Major cations and trace metals

Sediment samples are lightly ground and dried overnight at 105°C. Between 0.3 and 0.5 g are weighed to 0.0001 g accuracy into teflon beakers. The beakers are cleaned by scrubbing and soaking in a cleaning mixture (HCl and Decon 90).

In turn 10 ml of hydrofluoric acid, 10 ml of nitric acid and 5 ml of perchloric acid (all Aristrar grade) are added and evaporated to dryness. Occasionally more perchloric acid has to be added to completely digest the organic matter. When all the silicates and organic matter have been decomposed 1 ml of Aristrar HCl and some distilled water (Gelman's Water I) are added and the salts dissolved. This is transferred with washings to a 25 ml volumetric flask and made up to volume. The samples are stored in cleaned polypropylene bottles.

All metals are determined by flame atomic absorption spectrophotometry (Perkin-Elmer 2380). Deuterium lamp background correction is used and the samples are diluted 50-fold with 0.1% lanthanum chloride for the major cations. Curve correction is used for the major cations and zinc. Five blanks are included with each core to check for contamination. This whole analytical method gives good recoveries and precision (Rippey et al. 1982).

7.2 Total Sulphur

This is determined by potassium nitrate/sodium carbonate half fusion (Grant and Yeung 1971) with the sulphate by turbidity (Tabatabai 1974). The sample size is 0.5 g. Before instituting this method it was checked for accuracy and precision.

The accuracy was determined by recoveries. In three trials the mean recovery was not significantly different from 100% ($0.10 < P < 0.05$, $n=5$; $0.60 < P < 0.80$, $n=5$; $0.40 < P < 0.50$, $n=5$). The precision ranged from 0.25 to 0.07 mg S g⁻¹ and the mean value was 0.15 mg S g⁻¹.

8.0 Polycyclic Aromatic Hydrocarbons (PAH) in lake sediments (B. Rippey)

The main sources of PAH at the earth's surface are the combustion of carbonaceous particles, contamination by petroleum or coal and diagenesis of certain biogenic compounds. Analysis of PAH in dated lake sediment cores provides a record of change in the flux and type of anthropogenic combustion products deposited at the site from the atmosphere. In lakes where there is no input from urban or industrial sources within the catchment, the following characteristics in recently deposited sediment are evidence for contamination by combustion products: a complex mixture of PAH; higher concentrations in recently deposited sediments; and decreasing abundance as the degree of alkyl substitution increases.

PAH analysis is by solvent extraction and high performance liquid chromatography (HPLC) with fluorescence detection. The PAH are removed from the wet sediment by Soxhlet extraction with dichloromethane for eight hours. This method removes non-polar compounds quantitatively from sediments (Sporstol *et al.* 1983). The samples are cleaned up by adsorption chromatography using Waters Sep-Pak Alumina with hexane elution.

The PAH are separated by HPLC (Spectra Physics B700) using a reverse phase column (Chrompac PAH) under isocratic conditions (75% acetonitrile and 25% water; Rathburn solvents) with fluorescence detection (Kontron SFM 25; Das and Thomas 1978). Detection is at 280/350 nm changing to 300/400 nm after 11 minutes. Data handling is by a computer integrator (Trivector Trio). The identity of the peaks is confirmed by coelution with standards and their fluorescence spectra.

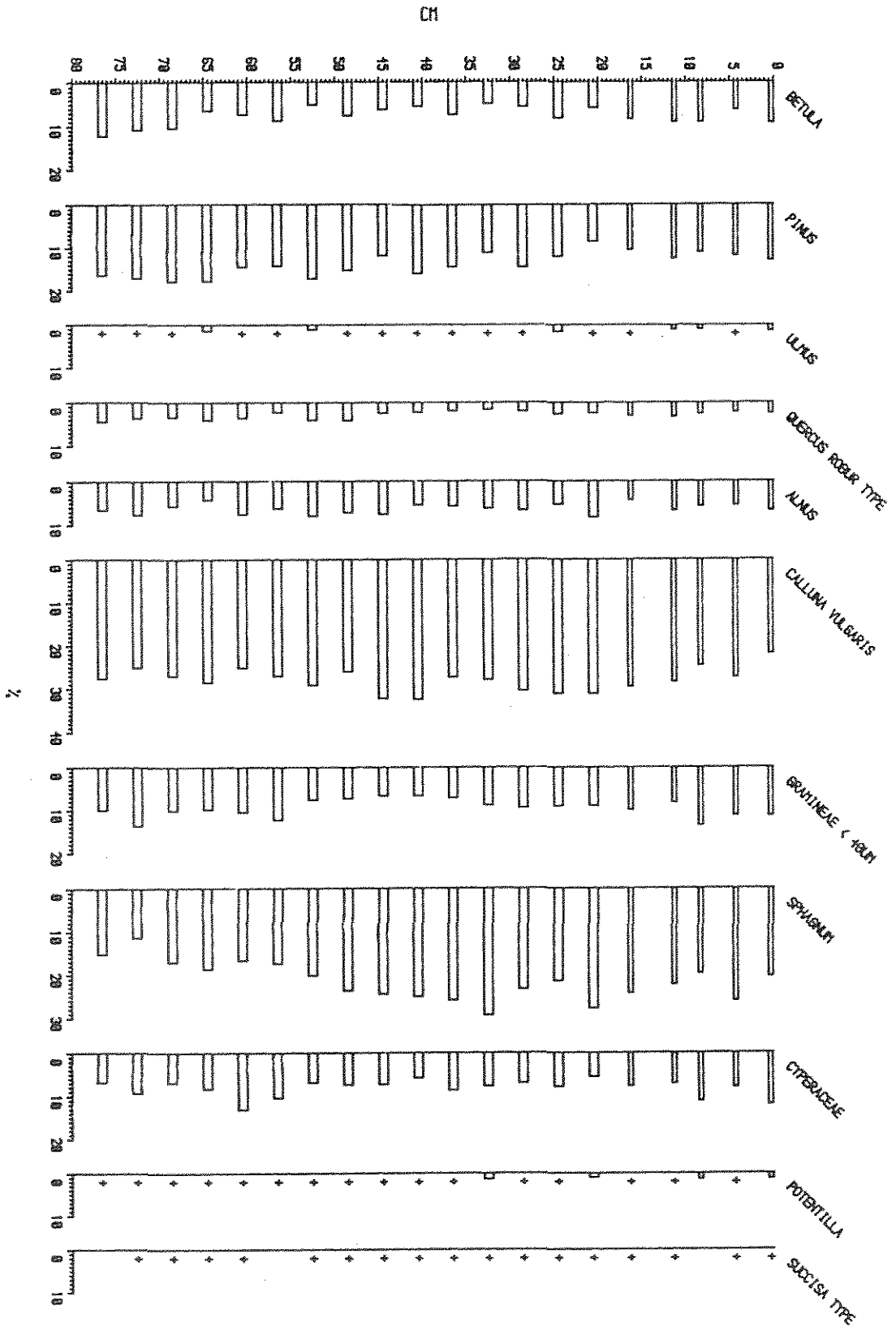
9.0 Pollen (A.C. Stevenson)

Pollen analysis of sediments allows a long to medium term assessment of vegetation change within the catchment. Documentation of such change is important in its own right - it is fundamental in investigating the 'land use hypotheses' of lake acidification of Rosenqvist (1978, 1980) and Krug and Frink (1983) - and it often indicates whether a conformable sequence of lake sediment is present in sample cores.

9.1 Methods

Subsamples from selected levels in each sediment core are prepared for pollen analysis by KOH digestion followed by HF and acetolysis treatments (Birks and Birks 1980). The samples are mounted in glycerol jelly and at least 500 grains of land pollen counted for each sample. Pollen identifications follow Moore and Webb (1978). In most cases pollen concentration data are obtained by the addition of Lycopodium tablets to a known dried weight of sediment (Stockmarr 1971). Pollen counts from sample levels down the sediment core are expressed as percentages and presented in the format exemplified in Fig. 11.

Fig.11. Example plot of pollen species counts (L. Laidon)



10.0 Chironomid analysis (Y.W. Brodin)

The Chironomidae (Diptera) is a species-rich family of small midges with larvae living in all types and conditions of aquatic habitats. The species composition of the benthic chironomid fauna, especially that of the profundal zone, is generally specific for a particular environmental condition. This has made chironomids useful in classifying the trophic status of lakes and in this study permits them to be used to indicate and interpret environmental change caused by acidification.

Acidification of lakes results in a well-defined composition of the chironomid fauna and further generally gives rise to: a less diverse fauna; a characteristic change in the dominance of different subfamilies and subgroupings; an increase in relative numbers of carnivorous chironomid species; a characteristic change in the trophic level of the fauna; and a dominance of certain groups of species which may be considered indicators of acidification.

10.1 Sediment sample treatment, sorting and slide preparation

The principal aim of the methods employed for treating sediment samples is to provide high quality larval head capsules of the midge families Chironomidae, Ceratopogonidae and Chaoboridae. Samples, each with a volume of about 10 cm³, are taken at intervals of 1-2 cm down the sediment cores. A solution of potassium hydroxide and distilled water (KOH concentration 5-10%, depending on sediment texture) is added and each sample directly heated at 70-80°C for 4-8 minutes. The digested samples are poured into a sieve with a mesh size of 50 µm. The washed samples are then stored in vials with 75% ethanol.

After the sieving procedure samples are transferred to a sorting dish containing water. A stereomicroscope is employed to pick out all forms of larval insect and mite remains. This technique is employed quantitatively only for third and fourth larval instars of Chironomidae, Ceratopogonidae (head capsules) and Chaoboridae (mandibles).

After sorting, the insect and mite remains are transferred to baths of 75% and 95% ethanol and then mounted on microscope slides.

10.2 Taxonomic identification

The identification of different species or species types of the midge families Chironomidae, Ceratopogonidae and Chaoboridae is achieved primarily by comparison of laboratory specimens reared to adults with modern taxonomic literature. Some specimens can also be identified directly with reference to taxonomic literature.

10.3 Measurements of fauna stability and diversity

All calculations of fauna parameters are based on 50 randomly chosen specimens from each sampling level of the cores. The Shannon Weiner Diversity Index is used to calculate temporal changes in species diversity (Southwood 1971):

$$H' = - \sum_{i=1}^s (p_i \ln p_i)$$

Where s = number of taxa

p_i = relative proportion of the i th taxa

The evenness with which the individuals are distributed among the specimens are calculated according to Pielou (1975):

$$J' = \frac{H'}{\ln s}$$

Temporal changes in the turnover rate of the insect fauna are calculated using the index described by Brodin (1985):

$$PS' = \frac{1}{4} (PS_{x,v} + PS_{x,w} + PS_{x,y} + PS_{x,z})$$

Where k = number of categories

$PS_{x,v} \dots x,z$ = percentage similarity between the species composition of a particular sample (x) and a sample of its second closest older (v), closest older (w), closest younger (y) and second closest younger sampling level (z).

11.0 Chrysophyte analysis (G. Cronberg)

A characteristic feature of chrysophytes is their ability to produce resting stages in the form of endogenously formed siliceous cysts. The chrysophyte cyst is characteristic of the species and even within the same genus different species produce cysts of unique size, form and ultrastructure. Chrysophyte cells are covered with loosely attached silica scales the ultrastructure of which is also characteristic of the species. The silica scales and cysts are well preserved in lake sediments and as chrysophytes are primarily planktonic and possess restricted environmental requirements, they can be good indicators of water quality.

11.1 Methods

To date full working methods have not been finalised.

12.0 Carbonaceous particle (soot) analysis (J. Darley)

When fossil fuels are combusted spherical carbonaceous particles (SCPs) are emitted into the atmosphere along with other pollutants. The amount released depends on several factors such as type of fuel, type of combustion system and type of emission control procedure. Both coarse and fine particles are produced during combustion. The morphology and surface texture, particularly of coarse particles, are indicative of their origin. When the particles are deposited from the atmosphere they accumulate in lake sediments where they are preserved and form a stratigraphic record of the history of the deposition of pollutants from fossil fuel combustion.

12.1 Sampling and preparation

Sediment is subsampled from the relevant core from each study lake. Up to 40 subsamples of 0.2-0.4 g are taken at regular intervals down the core to a depth of about 30 cm, which for most cores covers sediment dating from the early industrial period - c. 1850s.

Each subsample is placed in a clean 250 ml glass beaker and 10 ml 32% H₂O₂ is added to remove organic matter by oxidation. Each beaker is covered with a plastic lid and left to stand for 24 hours.

For the following two weeks the beakers are kept in a warm oven at 55°C, to assist oxidation and 10 ml H₂O₂ added at regular intervals until each sample has received 110 ml.

Beakers are then removed from the oven and filled with distilled water to begin a wash process by sedimentation. The beakers are left to stand for at least 24 hours to allow the oxidised sediment to settle, before decanting up to 66% of the fluid and filling again with clean distilled water. This process is repeated four times.

As much fluid as possible is decanted before transferring the residue to pre-weighed 100 ml conical flasks and sealed with stretch plastic tape. A suspension of 75 ml is considered the appropriate concentration for pouring.

For each sample a subsample of the prepared suspension is poured into a petri dish ready for counting. Each flask is unsealed, weighed to 0.001 g and resealed. The suspension is homogenised by vigorous shaking and a small amount (c. 8-10 ml) poured into a clean petri dish placed on a level surface. The suspension is poured out to leave a level, thin layer of sediment when dry. Each flask is re-weighed to determine the fraction of suspension that has been poured out. The dishes are then left at room temperature for evaporation to take place.

12.2 Counting

The SCPs are counted under a stereomicroscope at 40x magnification. The black spherules are seen to stand out against a background of bleached sediment. Spherules with a diameter in excess of c. 10 µm can be distinguished. The maximum diameter observed has been in the range 80-90 µm. A uniform distribution of sediment is assumed so that only 50% of the contents of each dish are counted. This proportion is determined by placing a grid beneath the dish. The result for each dish is multiplied by two to obtain a figure for the subsample poured out.

12.3 Calculations

12.3.1. Calculation of SCP concentration:

The results are expressed in terms of the number of particles per gramme dry sediment using the formula $A - \text{SCPs g dry sediment}^{-1} = \frac{a * 2X}{b.c}$

where a = weight of total suspension prepared
 b = weight of suspension fraction poured out
 c = weight of dry sediment prepared
 X = number of SCPs counted in the half dish.

12.3.2. Loss on ignition data:

Results are also expressed in terms of the organic fraction of the sediment as determined from loss on ignition values, using the formula B -
 SCPs g organic fraction of dry sediment⁻¹ = $S * \frac{100}{L}$

where S = SCPs g dry sediment⁻¹ (from formula A)
 L = loss on ignition value for the appropriate sediment level.

Results expressed in this way are considered to give a more reliable picture of SCP concentrations than values expressed solely in terms of sediment dry weight. This is particularly the case where the mineral matter input to the sediment has been widely variable through time.

Graphs are constructed from the results to obtain the SCP patterns which are used in the interpretation of the results (cf. Fig. 12).

13.0 Non core-based methods

13.1 Surveying and mapping techniques (A. Nicholson, A. Kreiser, S. Patrick)

Computer drawn bathymetries and/or core location maps are produced for each lake.

Polar alidades, sited at fixed locations on the lake shore, are utilised to record the position of an inflatable boat aboard which a graphical echo sounder records lake water depths. Spot depths are recorded on the continuous echo sound trace and coordinated with alidade sightings by means of citizen band hand radios.

Contour maps and calculations of lake surface area and volume are produced using proprietary mapping software (MAPICS 1985). Lake outlines are taken from 1:10000 maps and enlarged to a suitable scale. Surveyed depth and core points are transposed on to these outlines from the polar alidade field tracings. The following data are digitised for each site: lake outline, outline of islands (if present), core locations and surveyed depths including a sample of points from lake outlines recorded as zero depth.

The MAPICS graphics system produces high quality bathymetric contour maps of the lake basins from height-gradient grids interpolated from the surveyed depths and lake/island outlines. Contour maps are drawn (cf. Fig. 13) using a Hewlett-Packard vector plotter. Surface areas and volumes are estimated using MAPICS grid conversion facilities. Essentially this process involves converting vector information into grid form of a suitable density. Areas are then calculated as the number of grid cells multiplied by the grid cell area. Volumes are calculated as the sum of the individual grid cell volumes. Grid density can be adjusted to any suitable resolution and the volumes and surface areas of individual contour levels can be calculated for hypsographic generation.

Fig.12. Example of a SCP analysis plot (L. Laidon)

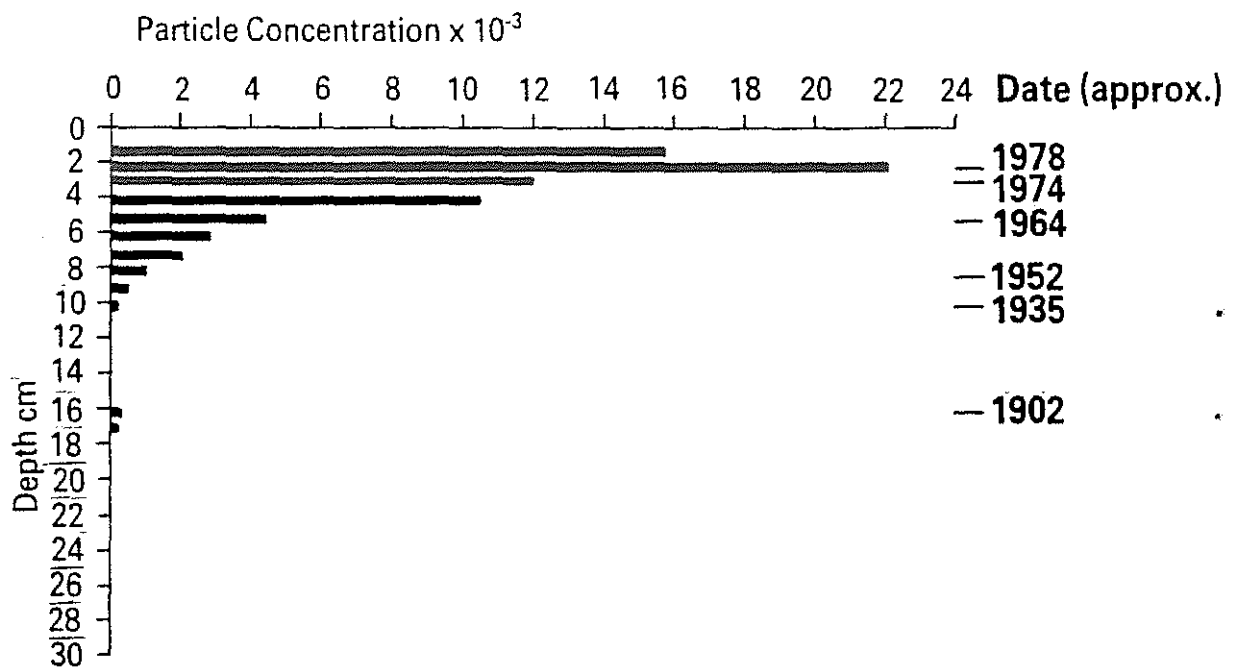
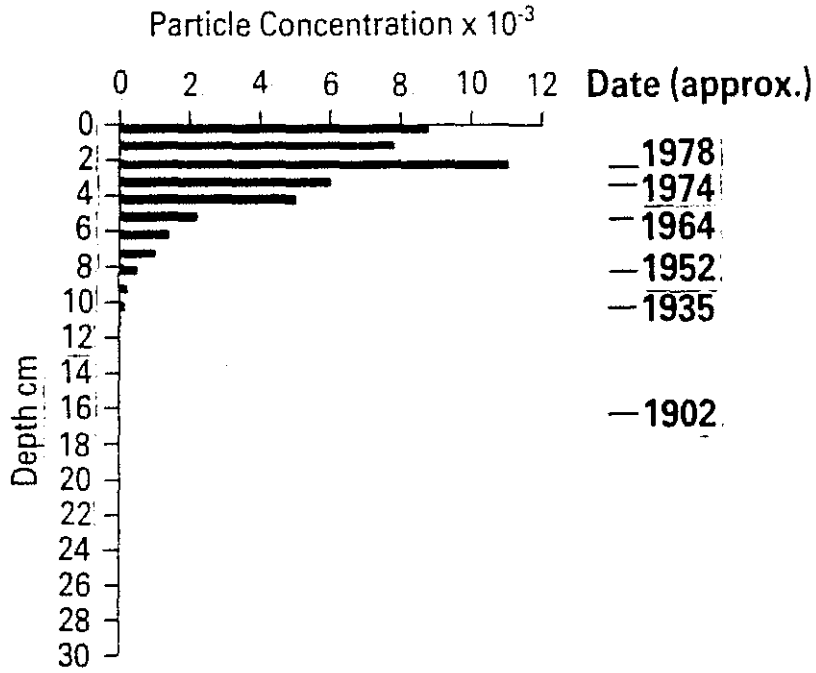
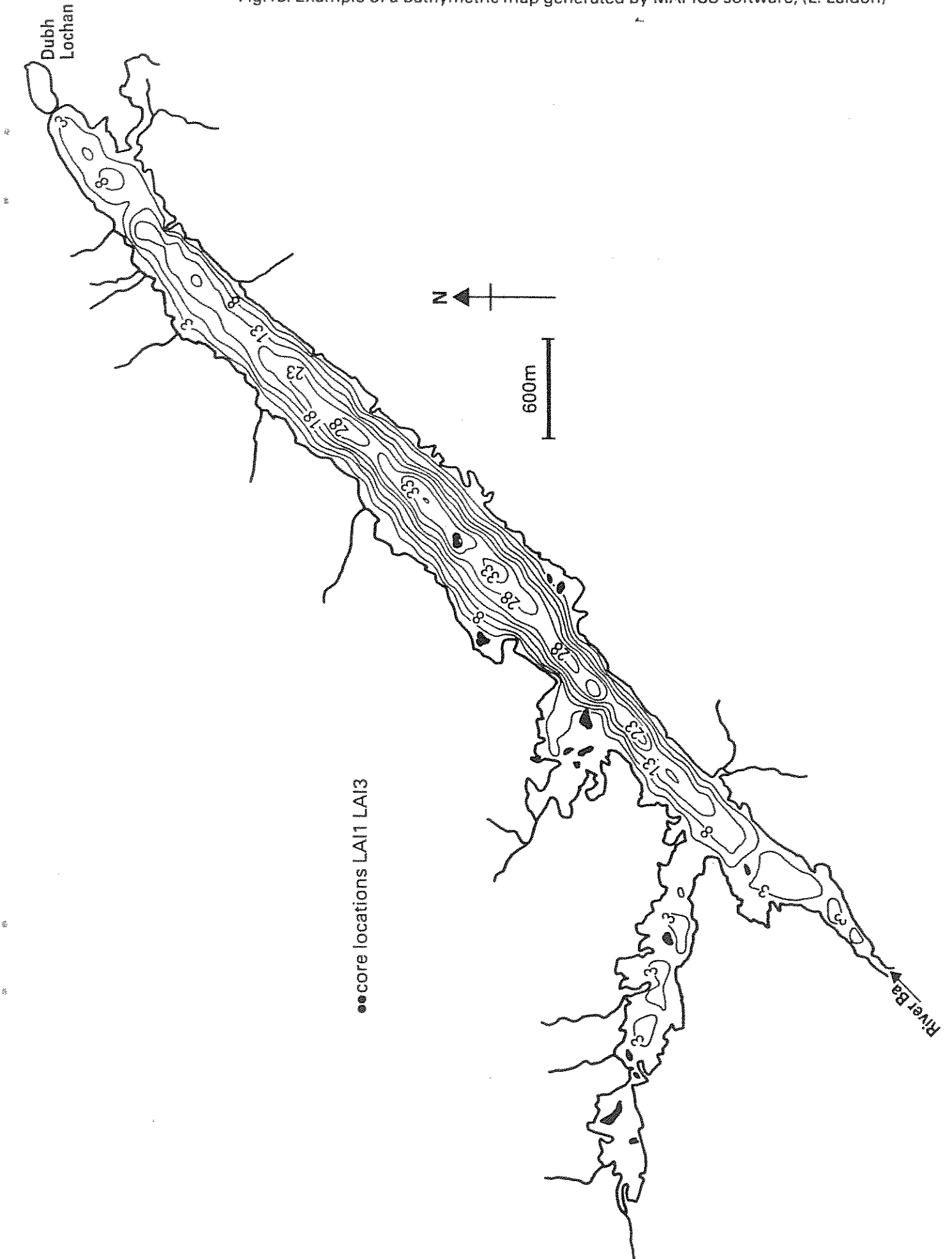


Fig.13. Example of a bathymetric map generated by MAPICS software, (L. Laidon)



13.2 Water chemistry (WWA Llanelli and DAFS Pitlochry)

All water samples are collected from immediately below the water surface in the littoral (preferably from the outflow) in about 40 cm depth of water. During sampling care is taken to avoid collecting re-suspended particulate material.

Samples for water pH and conductivity measurement are collected in acid pre-washed polypropylene 1 litre bottles and stored immediately in a dark plastic sack. Chemical analyses are carried out at the Welsh Water Authority's laboratory at Llanelli and at the DAFS laboratory in Pitlochry. The parameters measured are listed in Table 2.

13.3 Catchment land use and management and fishery histories (S. Patrick)

Documentary and ethnographic sources are used to provide background information about the lakes and their catchments and to examine evidence that may indicate the influence of acidifying factors other than acid deposition - liming, drainage, afforestation, mining, changing stocking densities and burning regimes etc.

The timescale covered by this investigation is limited by the availability of documentary material and broadly encompasses the past 200 years with the emphasis on the past century. Certain sources are common to investigations in Wales and Scotland - large scale Ordnance Survey maps, air photographs, estate plans and records, annual parish agricultural returns, diaries and topographies and land utilisation surveys. Certain sources are specific to each country, notably Tithe Survey documents (Wales) and the 'Statistical Account' editions (Scotland). The personal evidence, particularly of farmers and anglers, provides much important evidence of more recent change. The detail with which catchment and fishery histories can be compiled varies greatly between lakes and depends primarily on the remoteness of the site and the intensity with which the catchment resources are managed and the lake fished.

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