

Loss on ignition as a method for estimating organic and carbonate content in sediments: reproducibility and comparability of results

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Abstract

Five test runs were performed to assess possible bias when performing the loss on ignition (LOI) method to estimate organic matter and carbonate content of lake sediments. An accurate and stable weight loss was achieved after 2 h of burning pure CaCO₃ at 950 °C, whereas LOI of pure graphite at 530 °C showed a direct relation to sample size and exposure time, with only 40–70% of the possible weight loss reached after 2 h of exposure and smaller samples losing weight faster than larger ones. Experiments with a standardised lake sediment revealed a strong initial weight loss at 550 °C, but samples continued to lose weight at a slow rate at exposure of up to 64 h, which was likely the effect of loss of volatile salts, structural water of clay minerals or metal oxides, or of inorganic carbon after the initial burning of organic matter. A further test-run revealed that at 550 °C samples in the centre of the furnace lost more weight than marginal samples. At 950 °C this pattern was still apparent but the differences became negligible. Again, LOI was dependent on sample size.

An analytical LOI quality control experiment including ten different laboratories was carried out using each laboratory's own LOI procedure as well as a standardised LOI procedure to analyse three different sediments. The range of LOI values between laboratories measured at 550 °C was generally larger when each laboratory used its own method than when using the standard method. This was similar for 950 °C, although the range of values tended to be smaller. The within-laboratory range of LOI measurements for a given sediment was generally small. Comparisons of the results of the individual and the standardised method suggest that there is a laboratory-specific pattern in the results, probably due to differences in laboratory equipment and/or handling that could not be eliminated by standardising the LOI procedure.

Factors such as sample size, exposure time, position of samples in the furnace and the laboratory measuring affected LOI results, with LOI at 550 °C being more susceptible to these factors than LOI at 950 °C. We, therefore, recommend analysts to be consistent in the LOI method used in relation to the ignition temperatures, exposure times, and the sample size and to include information on these three parameters when referring to the method.

Introduction

Sequential loss on ignition (LOI) is a common and widely used method to estimate the organic and carbonate content of sediments (e.g., Dean, 1974; Bengtsson & Enell, 1986). In a first reaction, organic

matter is oxidised at 500–550 °C to carbon dioxide and ash. In a second reaction, carbon dioxide is evolved from carbonate at 900–1000 °C, leaving oxide. The weight loss during the reactions is easily measured by weighing the samples before and after heating and is closely correlated to the organic matter and carbonate

content of the sediment (Dean, 1974; Bengtsson & Enell, 1986). Dean (1974) evaluated the method and concluded that LOI provides a fast and inexpensive means of determining carbonate and organic contents of clay-poor calcareous sediments and rocks with precision and accuracy comparable to other, more sophisticated geochemical methods. Bengtsson & Enell (1986), in their instructions to the technique, mention that the method gives a rough indication of the organic matter and carbonate content of sediments. Depending on the ignition temperature, various losses of volatile salts, structural water and inorganic carbon may occur (Dean, 1974; Bengtsson & Enell, 1986; Sutherland, 1998) and it is important to check the ignition temperature carefully for organic matter determination. Nonetheless, a short survey of recently published palaeolimnological studies shows that there are still substantial differences in the methods used. Exposure times vary from 1 to 4 h (e.g. Spaulding et al., 1997; Henderson & Last, 1998) and ignition temperatures from 500 to 550 °C (e.g. Virkanen et al., 1997; Korsman et al., 1999). Cautioned by the discovery that a defective furnace produced a strong and immediately noticeable change of LOI results, we decided to run several tests to estimate possible bias of the method with a new digital-display furnace. In particular, we wanted to answer the following questions:

- i) How long must samples be exposed in the furnace for a complete reaction?
- ii) Does the position of crucibles in the furnace influence the LOI results?
- iii) Does sample size affect the results?
- iv) Are LOI results measured in different laboratories comparable?

This last point is all the more important as palaeolimnological projects reach regional or even continental scales (e.g. Wathne et al., 1995; Korhola et al., 1999) and LOI as a quick and inexpensive estimate of sedimentary organic matter is increasingly measured at different institutes. Interpretation of results is then only possible if an estimate of a laboratory specific error is available and real trends in the data can be viewed in relation to measurement errors.

The basic LOI method

Determination of weight percent organic matter and carbonate content in sediments by means of LOI is

based on sequential heating of the samples in a muffle furnace (see Dean, 1974; Bengtsson & Enell, 1986 for more details on the method, but note that in the equations for calculation of LOI in the latter publication the weight loss is related to the wet weight of the sediment instead of the dry weight). After oven-drying of the sediment to constant weight (usually 12–24 h at ca. 105 °C) organic matter is combusted in a first step to ash and carbon dioxide at a temperature between 500 and 550 °C. The LOI is then calculated using the following equation:

$$\text{LOI}_{550} = ((\text{DW}_{105} - \text{DW}_{550}) / \text{DW}_{105}) * 100 \quad (1)$$

where LOI_{550} represents LOI at 550 °C (as a percentage), DW_{105} represents the dry weight of the sample before combustion and DW_{550} the dry weight of the sample after heating to 550 °C (both in g). The weight loss should then be proportional to the amount of organic carbon contained in the sample and Dean (1974) showed a strong correlation between LOI at 550 °C and organic carbon content determined chromatographically in lake sediments.

In a second step, carbon dioxide is evolved from carbonate, leaving oxide and LOI is calculated as:

$$\text{LOI}_{950} = ((\text{DW}_{550} - \text{DW}_{950}) / \text{DW}_{105}) * 100 \quad (2)$$

where LOI_{950} is the LOI at 950 °C (as a percentage), DW_{550} is the dry weight of the sample after combustion of organic matter at 550 °C, DW_{950} represents the dry weight of the sample after heating to 950 °C, and DW_{105} is again the initial dry weight of the sample before the organic carbon combustion (all in g). Assuming a weight of 44 g mol⁻¹ for carbon dioxide and 60 g mol⁻¹ for carbonate (CO₃²⁻), the weight loss by LOI at 950 °C multiplied by 1.36 should then theoretically equal the weight of the carbonate in the original sample (Bengtsson & Enell, 1986). Again, LOI shows a good correlation with other methods of determining carbonate content of lake sediments (Dean, 1974).

Experimental setting

All LOI analyses were carried out in a Nabertherm® Controller C6 muffle furnace with digital temperature display and thermostatic temperature control (Nabertherm®, Lilienthal/Bremen, Germany). For this study we used LOI temperatures close to those proposed by

Dean (1974) and Bengtsson & Enell (1986), i.e. 530 and 550 °C for organic matter and 950 °C for carbonate. Care was taken that no humidity remained in samples before weighing. Therefore, empty crucibles and wet sediment were dried at 105 °C overnight and all samples were cooled to room temperature in a desiccator before any measurements were made. To avoid overheating, crucibles were put into the furnace only after a constant temperature was reached. For the longer time series crucibles were heated, cooled to room temperature, measured, and the same sediment was then again exposed to the respective temperature.

For the test runs, three artificial sediments were produced: Cores from several lakes on the Swiss Plateau were mixed to produce the first test sediment standard with a low organic matter and intermediate carbonate content. A short Kajak core of Nydalasjön, northern Sweden, provided the source for a sediment standard with a high organic matter content. A third sediment standard with a high carbonate content was produced from a mix of mid-Holocene sediment from Rotsee, central Switzerland. The three sediments are referred to as 'mixed sediment', 'high organic matter sediment' and 'high carbonate sediment' respectively. The sediments were dried, finely ground and homogenised by hand stirring and shaking in a closed container. Due to different amounts of material available sample size varied between 0.4 and 3.9 g dry weight (see figure legends for average dry weight and number of samples per test run).

Results and discussion

Exposure time and sample size

In the first two test runs, we exposed samples of pure graphite (Merck #104206, Merck KGaA, Darmstadt, Germany) and of pure calcium carbonate (CaCO_3 , Merck #102064) to 530 and 950 °C to assess if the reactions are complete within the 2 h we previously used for LOI analysis. Theoretically, graphite, as a source of totally combustible carbon, should give a weight loss of 100% at 530 °C. Assuming a weight of 99.96 g/mol for CaCO_3 and 55.96 g/mol for CaO, calcium carbonate should produce a weight loss of 44.02% at 950 °C (molar weights according to Mortimer, 1983). Only 40–70% of the graphite was combusted in most samples after 2 h of exposure at 530 °C (Figure 1), whereas 5 h of exposure were needed for a median weight loss of 98.3%. The range

of 26.7–99.0% LOI was unexpectedly high after 2 h of exposure but decreased to 63.8–99.8% after 5 h of exposure. This high variability is only partly due to the fact that larger samples needed more time to combust, as can be seen if the samples are separated into weight classes and plotted vs. time (Figure 2). A median of 99.8% of the total weight loss (range: 99.7–99.9%) was reached after exposing the samples for 2 h to 950 °C, confirming that the graphite we used is entirely combustible and that in many samples the reaction at 530 °C was not finished even after 5 h of exposure. However, graphite is an inadequate substitute for sedimentary organic matter, as it has different chemical properties and is presumably more refractory than autochthonous algal remains that commonly constitute most of the lacustrine sedimentary organic carbon. Still, our results indicate that 1 or 2 h of exposure as generally recommended for combustion of organic matter (Dean, 1974; Bengtsson & Enell, 1986) may not be sufficient for larger sediment samples with a high organic content and that LOI at 550 °C may depend on weight (Figure 2).

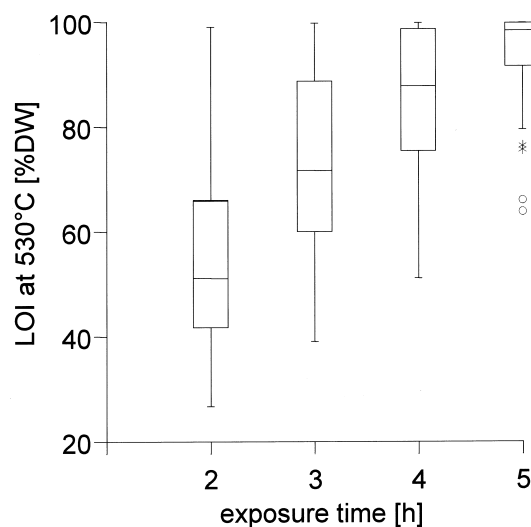


Figure 1. Box plots of LOI values of 56 samples of graphite heated for 2, 3, 4 and 5 h to 530 °C (average sample dry weight \pm S.D.: 1.12 ± 0.22 g). The central horizontal line in the box marks the median of the samples, the box edges (hinges) the first and third quartile. The interquartile range within the box includes the central 50% of the values. The whiskers show the range of observed values that are not within the first and third quartile but not further away than 1.5 times the interquartile range from the hinges. Values between 1.5 and 3 times the interquartile range from the nearest hinge are marked by asterisks, values farther than 3 times the interquartile range from the next hinge are marked by open circles.

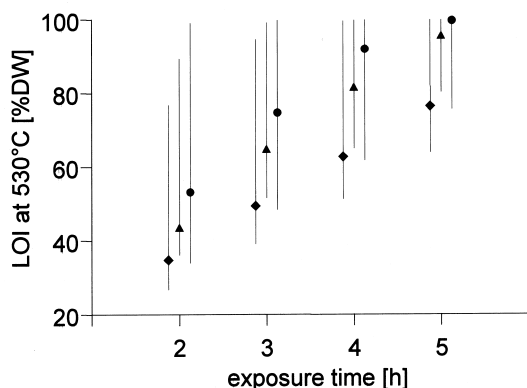


Figure 2. Median and range of LOI at 530 °C of three weight classes of samples in the graphite experiment. Circles: 44 samples between 0.7 and 1.2 g dry weight; triangles: 7 samples between 1.2 and 1.5 g dry weight; diamonds: 5 samples between 1.5 and 1.8 g dry weight.

Using CaCO_3 , no significant weight loss was detected at 530 °C, whereas at 950 °C a median of 42.7% of the total weight loss was reached after 2 h of exposure. This represents the equivalent of 97% of the theoretically possible weight loss, indicating that the evolution of carbon dioxide from the carbonate was largely finished within the exposure time.

In a third test we combusted samples of the ‘mixed sediment’ at 550 °C, exposing them for 0.5–64 h to assess how long the organic matter needs to be completely ashed. In order to test the reproducibility of the results, this series was repeated in three separate runs of short (0.5, 1, 1.5, 2, 2.5 h), intermediate (4.5, 6.5, 8, 10 h) and long (16, 24, 33, 40, 48, 56, 64 h) time intervals. Afterwards, each set of samples was heated to 950 °C for 2.5 h to calculate the total carbon content. The results indicate that LOI at 550 °C was slightly lower in the samples used for the intermediate time intervals (approximately 0.15%; Figure 3). Still, the method is precise enough to give a clear overall impression of the weight loss through time. Continuing weight loss was registered up to the last measurement at 64 h of exposure (Figure 3). As expected, LOI was strongest in the first 2–2.5 h whereupon weight loss per time diminished to approach a more or less constant value of approximately 0.02% per h.

This small but continuing weight loss at 550 °C was an unexpected result and may be explained by the high inorganic content of the mixed sediment. Different authors (e.g. Ball, 1964; Dean, 1974; Bengtsson & Enell, 1986) warn that clay may lose structural water during LOI. According to Ball (1964) this may happen at temperatures as low as 500 °C, thus causing a weight

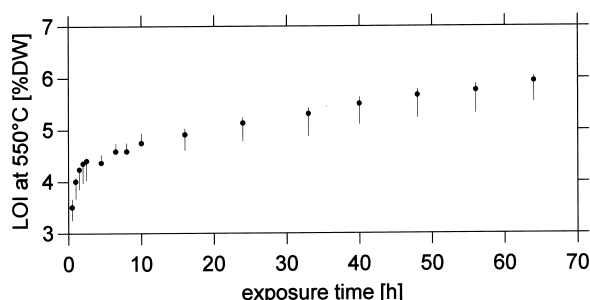


Figure 3. Median and range of LOI of 5 samples of the ‘mixed sediment’ exposed to 550 °C in irregular intervals up to 64 h (average sample dry weight \pm S.D.: 3.93 ± 0.03 g).

loss of up to 20% in clay minerals. Sutherland (1998) mentions that structural water may be lost by metal oxides at temperatures as low as 280–400 °C and that inorganic carbon may be lost at temperatures between 425 and 520 °C in minerals such as siderite, magnesite and rhodochrosite (Weliky et al., 1983; Sutherland, 1998). Finally, Bengtsson & Enell (1986) mention the possible influence of loss of volatile salts at 550 °C on LOI results. In our opinion one or several of these processes taking place at a lower reaction speed than the combustion of organic matter is the most likely cause of the continuing weight loss observed in our experiment.

Two hours of exposure at 950 °C were sufficient for the total loss of carbon dioxide to be completed and further exposure led to no detectable subsequent reduction in weight. The total weight loss (at 550 and 950 °C) proved to be reproducible with an average (\pm S.D.) of $20.32 \pm 0.03\%$ for short time intervals, $20.31 \pm 0.03\%$ for the intermediate, and 20.38 ± 0.04 for the long time intervals.

Position in the furnace

In a fourth test run, again using the ‘mixed sediment’, we assessed the influence of the position of the crucibles in the furnace on LOI (see Figure 4a for the experimental setup). LOI at 550 °C was consistently highest in the centre of the furnace (exemplified for 2 and 18 h of exposure in Figures 4b & 4c). To compare how these differences change with increasing exposure time, the median LOI value and range of the twelve outermost samples and the six innermost samples are plotted in Figure 5a (see Figure 4a for the experimental setup). Again samples lost weight up to the maximum exposure time of 18 h. Differences due to the position

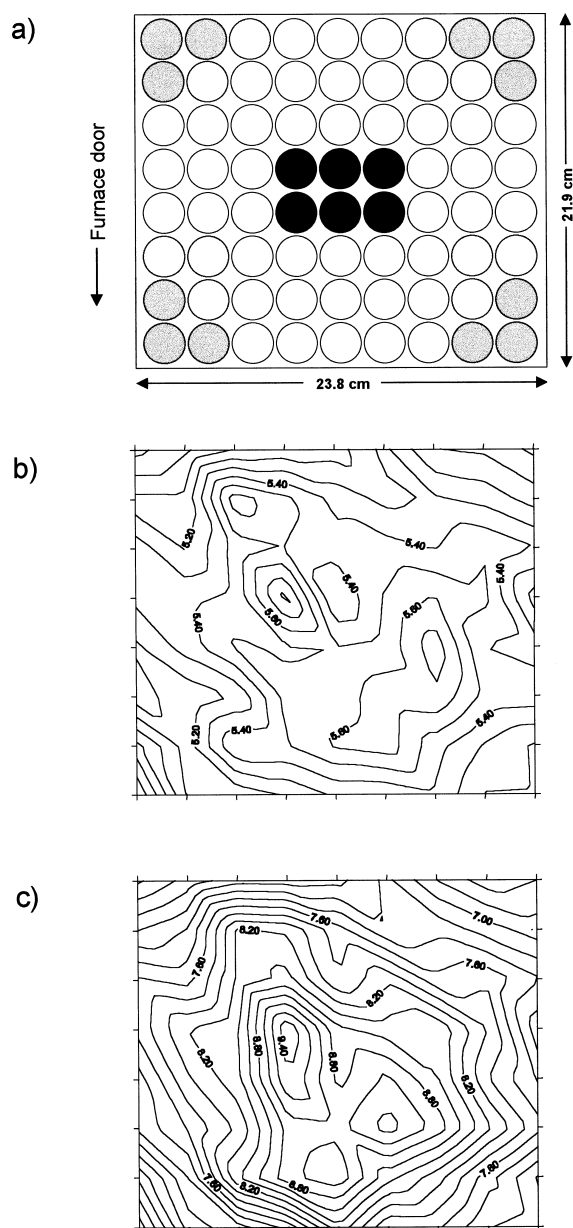


Figure 4. Spatial distribution of LOI at 550 °C within the furnace. (a) Experimental setup and position of the crucibles on the tray: The measurements were carried out on 72 samples of the 'mixed sediment' (average sample dry weight \pm S.D.: 1.30 ± 0.24 g). Black and shaded circles indicate the position of the six innermost and twelve outermost samples used to calculate LOI for Figure 5; (b) LOI after 2 h of exposure; (c) LOI after 18 h of exposure. For (b) and (c) LOI is interpolated using the program SURFER® and triangulation/linear interpolation.

of the crucibles became more distinct with longer exposure to 550 °C, starting with 0.42% difference of median weight loss between the innermost and outer-

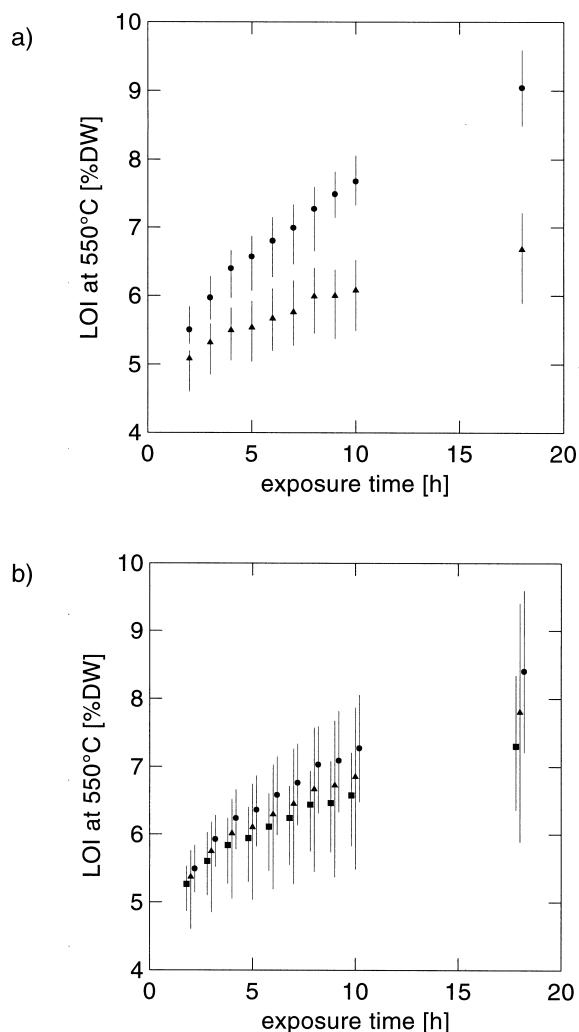


Figure 5. (a) Median and range of LOI of the six innermost (circles) and the twelve outermost samples (triangles) on a tray of 72 samples of the 'mixed sediment' exposed for 2, 3, 4, 5, 6, 7, 8, 9, 10 and 18 h to 550 °C (see Figure 4a for the experimental setup); (b) Median and range of LOI at 550 °C of three weight classes of samples in the same experiment. Circles: 0.8–1.0 g dry weight (7 samples); triangles: 1.0–1.5 g dry weight (48 samples); squares: 1.5–1.8 g dry weight (17 samples).

most samples after 2 h (maximum difference between crucibles 1.24%) to 2.36% difference after 18 h (maximum difference 3.70%). At 950 °C weight loss was also highest in the middle of the tray but the difference of 0.08% between median values of the innermost and outermost crucibles (maximum difference 0.48%) was negligible at an average total weight loss of 20.10%. As in the experiment using graphite, smaller samples again lost weight faster than larger ones (Figure 5b).

This difference was tolerable after 2 h of exposure to 550 °C, when the median values of the smallest (0.7–1.0 g DW) and largest size class (1.5–1.8 g DW) were only about 0.23% weight loss apart. Yet, the differences increased with exposure time and reached a maximum of 1.1% after 18 h of exposure (Figure 5b).

Again, higher loss of structural water, volatile salts or inorganic carbon due to warmer temperatures in the centre of the furnace and quicker heating of smaller samples are deemed to be the most likely explanation for the differential weight loss due to positioning of samples in the furnace and to sample size. As indicated by one of our old furnaces, certain ovens may lead to even more heterogeneous results due to uneven interior heat dispersions. A simple test using a standardised sediment may help detect such heat inhomogeneities and may be appropriate if a furnace yields very noisy LOI results.

Analytical quality control: the comparability of results

Two batches of five samples of the ‘mixed sediment’, the ‘high carbonate sediment’ and ‘high organic matter sediment’ were sent to ten different European laboratories. Participants in this LOI quality control exercise were asked to measure LOI on batch 1 using the method routinely used in their laboratory. All laboratories used LOI temperatures of 550 and 925–980 °C, but exposure times varied from 1–6 h for organic matter and from 0.5–6 h for carbonate determination (Table 1). Batch 2 was processed using a standardised method (Table 1). Given our results of the time-series for 550 °C (Figure 3) we proposed an exposure time of 4 h at 550 °C, since by this time the reaction reached a plateau after the rapid initial weight loss, whereas for a longer time interval sample size and

the positioning of samples in the furnace had an increasing influence on results (Figures 4 & 5). For carbonate measurement we deemed 2 h of exposure at 950 °C to be sufficient. We proposed a protocol that closely follows the method of Bengtsson & Enell (1986) with the exception that empty crucibles were dried at 105 °C overnight (instead of burning at 550 °C for 1 h). After every heating, sediments and crucibles were to be cooled down in a desiccator, thus ensuring that no moisture increased the dry weight measurements. Moreover, we asked participants to use porcelain lids on the crucibles for the first LOI step to prevent losing material by explosive combustion and to only move crucibles using tongs.

The results of the first batch of 15 samples measured were surprisingly consistent between different laboratories, given the differences in exposure times used. At 550 °C median values showed a maximum difference of 1.7, 1.6 and 4.2% LOI for the ‘mixed’, the ‘high carbonate’ and the ‘high organic matter sediment’, respectively (Figure 6). Using the standardised method, the maximum difference of medians decreased slightly or stayed the same for the ‘mixed’ and the ‘high carbonate sediment’ (1.7, 1.2%) but was more than halved for the ‘high organic matter sediment’ (1.9%), mainly due to the fact that laboratory I, that had previously measured an exceptionally high LOI value, was now more consistent with the other participants. The within-laboratory range of measurements was generally rather small (0.06–2.55% for the first batch, 0.11–1.23% for the second batch) although some laboratories tended to a larger range of values than others (e.g. C, F vs. H, I, J).

Compared to LOI at 550 °C, LOI for carbonate determination tended to have lower maximum differences of median values between laboratories when they used their own method (‘mixed’ 2.2%; ‘high

Table 1. Methods used by the ten institutes involved in the LOI laboratory comparison for the first batch of samples (A–J) and the standard method used by all for the second batch (S). Laboratory D left the samples in the furnace during the initial heating (no preheating), but exposure time was measured only after the desired temperature had been attained (x)

Institute	A	B	C	D	E	F	G	H	I	J	S
Temperature for first LOI step (°C)	550	550	550	550	550	550	550	550	550	550	550
Exposure time (h)	5.5	3	6	1	2	1	2	1	2	4	4
Temperature for second LOI step (°C)	950	950	950	980	950	925	950		950		950
Exposure time (h)	2	2	6	1	2	0.5	2		2		2
Furnace preheated to a constant temperature	+	+	–	x	+	+	+	+	+	+	+
Porcelain lids used for organic matter determination	–	+	–	–	–	–	–	–	–	–	+
Desiccator used for cooling crucibles and sediments	+	+	+	+	+	–	+	+	+	+	+
Cold crucibles moved by hand (h)/with tongs (t)	t	t	h	t	h	h	t	h	h	t	t

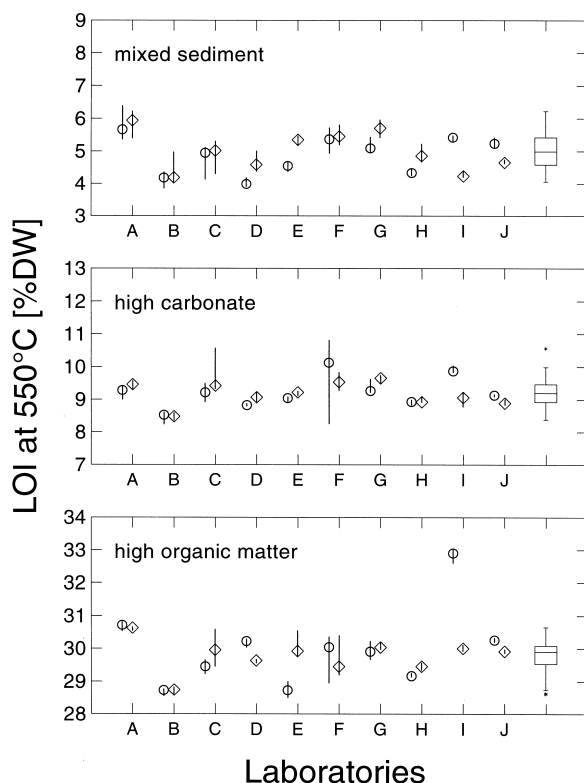


Figure 6. Median and range of LOI at 550 °C in the intercomparison experiment of 10 laboratories (A–J) for the ‘mixed’, ‘high carbonate’ and ‘high organic matter sediment’. Circles: laboratories used their own method; diamonds: all laboratories used the same standardised method; the small box plot shows the distribution of LOI using the standardised method within all 50 samples of the second batch (see legend of Figure 1 for an explanation of the symbols in the box plots).

carbonate’ 1.1%; ‘high organic matter’ 1.7%; Figure 7). Again, using the standardised method, maximum differences stayed the same (‘high carbonate’ 1.1%) or decreased (‘mixed’ 1.6% and ‘high organic matter’ 1.2%). Also, the within laboratory range of values was smaller for carbonate measurements (range of 0.04–1.39% and 0.03–1.00% for the first and second batch, respectively).

Total weight loss after both LOI steps (Figure 8) revealed that the between laboratory differences in LOI can be separated into two groups: most laboratories were consistent with each other concerning the total weight loss (approximately 19.5, 45.5 and 32% for the ‘mixed’, ‘high carbonate’ and ‘high organic matter sediment’, respectively), and the differences in organic matter and carbonate measured between these laboratories using the standardised method is probably

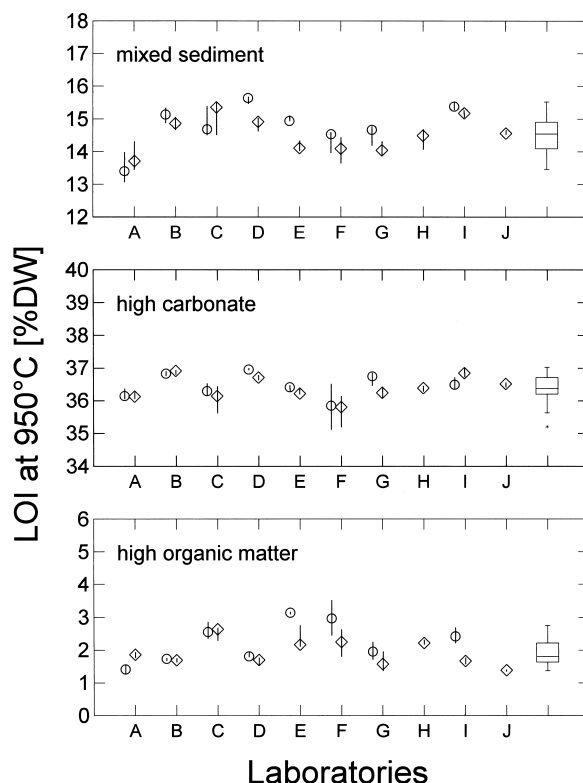


Figure 7. Median and range of LOI at 950 °C in the intercomparison experiment of 10 laboratories (A–J) for the ‘mixed’, ‘high carbonate’ and ‘high organic matter sediment’. Circles: laboratories used their own method; diamonds: all laboratories used the same standardised method; the small box plot shows the distribution of LOI using the standardised method within all 50 samples of the second batch (see legend of Figure 1 for an explanation of the symbols in the box plots).

mainly due to small differences of furnace temperature (e.g. spatial temperatures differences or initial over-heating) leading to differential organic matter combustion or loss of volatile salts, structural water or inorganic carbon. Two institutes showed inconsistent total LOI values (laboratory I using the ‘mixed’ and ‘high organic matter sediment’ in the first batch and laboratory B using the ‘high organic matter sediment’ in both batches; Figure 8). Possible reasons could be a weight increase of sediments or crucibles due to uptake of humidity before weighing, although the values are surprisingly consistent within themselves (i.e. they have a small range). One of the unexpected results of the comparison of different laboratories is that there seems to be a ‘laboratory specific’ error. Even when using the standardised method the same laboratories seemed to measure consistently ‘too high’ or ‘too low’

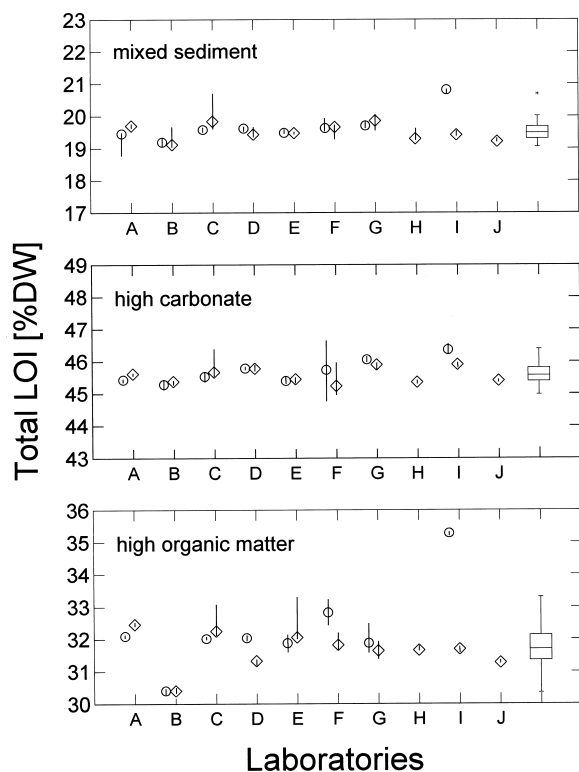


Figure 8. Median and range of total LOI in the intercomparison experiment of 10 laboratories (A–J) for the ‘mixed’, ‘high carbonate’ and ‘high organic matter sediment’. Circles: laboratories used their own method; diamonds: all laboratories used the same standardised method; the small box plot shows the distribution of LOI using the standardised method within all 50 samples of the second batch (see legend of Figure 1 for an explanation of the symbols in the box plots).

LOI values (e.g. see the pattern between laboratory A, B and C in Figure 6). This is probably due to differences in handling and/or equipment of different laboratories.

Conclusions

Even though LOI is generally considered to be a simple method to estimate organic matter and carbonate contents of sediments, our results indicate that factors as difference as exposure time (Figures 1, 3 & 5), position of crucibles in the furnace (Figures 4 & 5), sample size (Figures 2 & 5), and the laboratory that measures LOI may have an influence on results (Figures 6, 7 & 8).

Determination of carbonate seemed to be less influenced by these factors than organic matter measure-

ments (Figures 6 & 7), although any bias of LOI at 550 °C will, of course, also influence the calculation of LOI at 950 °C (see equation 2). Also, sediment with a high organic matter content seemed more prone to inconsistencies than sediment with a low organic matter content (Figure 6).

The results presented here lead us to the following conclusions important for palaeolimnologists and sedimentologists working with the LOI method:

1. LOI can be strongly dependent on the exposure time. The fact that some authors do not mention exposure times at all (e.g. Belis et al., 1999; Lamoureux, 1999) suggests that a common assumption is that at 550 °C organic matter is burned rapidly and thereafter no further weight loss takes place. This is clearly not the case in the ‘mixed sediment’ we used. Analysts should be aware that other reactions than burning of organic matter can take place at 550 °C, e.g. dehydration of clay minerals or metal oxides, loss of volatile salts, or loss of inorganic carbon in minerals such as siderite, magnesite or rhodochrosite. Also, the experiment with graphite suggests that for samples with a very high organic matter content the 2 h commonly used for LOI at 550 °C (e.g. Bengtsson & Enell, 1986) may not be sufficient. For the ‘mixed sediment’ used in the time-series experiment a reasonable exposure time at 550 °C would be 4 h: the fast initial weight loss due to burning of organic matter was largely completed and the differences in weight loss due to the positioning of crucibles in the furnace or sample size are still rather small. This optimal exposure time may, however, well depend on the sediment. If LOI is to be a major part of a sedimentological study, a preliminary test run with a standardised sediment of similar composition as the samples of interest may be useful to optimise quick measurement with a low variability of LOI at 550 °C. For LOI at 950 °C, 2 h of exposure was sufficient to evolve all the carbon dioxide from the carbonate in our experiments.
2. LOI at 550 °C is dependent on the sample size. An effort should, therefore, be made to keep samples roughly the same size when measuring LOI. Also, for results to be reproducible, next to the temperature and exposure time an estimate of sample size should be given when referring to the method.

3. The intercomparison of laboratories revealed that the maximum differences of LOI results measured at different institutes can be reduced in many instances if analysts closely follow a standardised LOI protocol. Still, in our experiment, a maximum error of approximately 2% LOI was detected when comparing results of different laboratories following a standard method. Therefore, small differences in LOI of sediments should not be over-interpreted.
4. The within-laboratory range of measurements was generally rather small, suggesting that LOI is a useful tool for correlating different sediment cores with a distinct LOI signature (as e.g. in Walker et al., 1993; Virkanen et al., 1997) or, if the analyst is aware of the possible bias of the method, as a proxy of carbon content of sediments.

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References

- Ball, D. F., 1964. Loss-on-ignition as an estimate of organic matter and organic carbon in non-calcareous soils. *J. Soil Sci.* 15: 84–92.
- Belis, C. A., A. Lami, P. Guilizzoni, D. Ariztegui & W. Geiger, 1999. The late Pleistocene ostracod record of the crater sediments from Lake Albano (Central Italy): changes in trophic status, water level and climate. *J. Paleolim.* 21: 151–169.
- Bengtsson, L. & M. Enell, 1986. Chemical analysis. In Berglund, B. E. (ed.), *Handbook of Holocene Palaeoecology and Palaeohydrology*. John Wiley & Sons Ltd., Chichester, 423–451.
- Dean, W. E. Jr., 1974. Determination of carbonate and organic matter in calcareous sediments and sedimentary rocks by loss on ignition: Comparison with other methods. *J. Sed. Petrol.* 44: 242–248.
- Henderson, P. J. & W. M. Last, 1998. Holocene sedimentation in Lake Winnipeg, Manitoba, Canada: implications of compositional and textural variations. *J. Paleolim.* 19: 265–284.
- Korhola, A., A. F. Lotter, H. J. B. Birks & N. G. Cameron, 1999. Climate history as recorded by ecologically sensitive arctic and alpine lakes in Europe during the last 10,000 years: a multi-proxy approach (CHILL-10,000). In *Proceedings of the European Climate Science Conference*, Vienna, City Hall, 19–23 October 1998 (in press).
- Korsman, T., M. B. Nilsson, K. Landgren & I. Renberg, 1999. Spatial variability in surface sediment composition characterised by near-infrared (NIR) reflectance spectroscopy. *J. Paleolim.* 21: 61–71.
- Lamoureux, S., 1999. Spatial and interannual variations in sedimentation patterns recorded in nonglacial varved sediments from the Canadian high arctic. *J. Paleolim.* 21: 73–84.
- Mortimer, C. E., 1983. *Chemie – Das Basiswissen der Chemie in Schwerpunkten*. Georg Thieme Verlag, Stuttgart, 637 pp.
- Spaulding, S. A., D. M. McKnight, E. F. Stoermer & P. T. Doran, 1997. Diatoms in sediments of perennially ice-covered Lake Hoare, and implications for interpreting lake history in the McMurdo Dry Valleys of Antarctica. *J. Paleolim.* 17: 403–420.
- Sutherland, R. A., 1998. Loss-on-ignition estimates of organic matter and relationships to organic carbon in fluvial bed sediments. *Hydrobiologia* 389: 153–167.
- Virkanen, J., A. Korhola, M. Tikkanen & T. Blom, 1997. Recent environmental changes in a naturally acidic rocky lake in southern Finland, as reflected in its sediment geochemistry and biostratigraphy. *J. Paleolim.* 17: 191–213.
- Walker, I. R., E. D. Reavie, S. Palmer & R. N. Nordin, 1993. A paleoenvironmental assessment of human impact on Wood

- Lake, Okanagan Valley, British Columbia, Canada. *Quat. Int.* 20: 51–70.
- Wathne, B. M., S. T. Patrick, D. Monteith & H. Barth (eds), 1995. AL:PE—Acidification of mountain lakes: Palaeolimnology and Ecology. AL:PE 1 report for the period April 1991–April 1993. Ecosystem Research Report 9, European Commission, Directorate-General for Science, Research and Development, Luxembourg, 296 pp.
- Weliky, K., E. Suess, C. A. Ungerer, P. J. Müller & K. Fischer, 1983. Problems with accurate carbon measurements in marine sediments and particulate organic matter in sea water: a new approach. *Limnol. Oceanogr.* 28: 1252–1259.