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THE PREPARATION OF LACUSTRINE SEDIMENT SAMPLES FROM CORES FOR USE IN DATING AND PALEOLIMNOLOGY

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Abstract

Holocene sediment cores are used to unravel the paleoenvironmental history of lakes and ponds. Deciphering the history implies that a suitable time framework has been established which represents the true sediment accumulation rate. When the sediment core is collected and processed in a standard way, the sediments can be uncompacted or represented as they were deposited. By using the uncompacted mid-depth, the actual sediment accumulation rate will be higher than the accumulation rate using non-normalized (natural or partially compacted) sediment mid-sample depths.

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INTRODUCTION

The examination of lacustrine sediments for trace metals, fossil shelled invertebrates, or paleobotanical remains requires a time frame with which to relate the results. For lacustrine cores there are a variety of methods used to date a specific horizon. Once these horizons have been identified there remains the task of calculating the sedimentation rate. Care should be taken to ensure the effects of compaction are considered. Often a constant or linear rate of sedimentation is assumed and calculated using uncompacted-depth and time. The assumption is verified from uniform size fractions of the sediments through out the core. The rate of sedimentation is calculated by dividing the depth from the surface to the marker horizon by the number of years elapsed from the present to the time horizon. The marker horizon may be the point of change of a specific pollen type or concentration of ^{210}Pb or ^{137}Cs. The method of calculation may lead to an accumulation rate that could be less than the actual sedimentation rate. This is because the effects of sediment compaction have been ignored. Because of compaction, the thickness of sediment used in the calculation will be less than that originally deposited through loss of pore fluids. To get a more accurate value the measure of sediment thickness or depth should allow for loss of volume by compaction. Robbins & Edgington (1975) have emphasized the importance of the relationship between sediment compaction and porosity in relating depth to age. Robbins (1978) indicated mass sedimentation rates may be calculated using the sample dry weight in the calculations rather than porosity. A more accurate determination of the sedimentation rate then provides a means to estimate age directly by assuming a constant rate of sediment deposition. The purpose of this chapter is to review methods that can be used to normalize depths of lacustrine-core sediments to provide a more realistic sedimentation rate.

CORING PRECAUTIONS

Depending on the requirements for the sediment sample, a variety of coring devices are available to collect lacustrine cores for dating. Generally, the quantity of sediment required will determine the appropriate diameter of the core tube and the type of apparatus to be used. There are, however, several factors that should not vary from one coring device to another. First, and foremost, the coring device should gently penetrate the sediment-water interface. Any disturbance of this interface by a bow/shock wave preceding the device will cause weightless surface sediments to be pushed aside (Sly, 1969). This loss of sediment causes the real quantity of sediment at the sediment-water interface and the porosity to be underestimated. The porosity of the surface sediment sample is the basis for uncompacting the sediment (Robbins & Edgington 1975). This concept is discussed in more detail in the section "Calculations". Inaccurate baseline values would result in calculated low porosity and sediment volume numbers for the remainder of the core. Second, the coring device or tube should be of sufficient diameter to allow for the removal of disturbed or smeared outer portion of the core and still yield an adequate amount of sediment for analysis. Most coring devices, despite the design or size, will carry a small amount of sediment along the sides as it penetrates the sediment. This has the dual effect of contaminating the sides of the sediment core and changing the porosity and sediment volume of the core.

CORE EXTRUSION AND STORAGE

During core extrusion the sample interval is determined. It is during this process that removal of the smeared outer portion of the core takes place. For many purposes a core slice of 1 cm thickness is taken. Regardless of the interval selected, sampling from the top to the bottom of the core should be contiguous to allow for accurate calculations of continuous porosity and sediment volume. Without contiguous sampling neither uncompacted depths nor accurate sedimentation rates can be calculated. For analyses requiring small volume subsamples at distinct core depths (e.g., 1 cm every 10 cm) contiguous sampling should still take place. As the core is extruded each slice of the core should be placed into a pre-weighed, labelled vial or ointment jar. The lid should be applied tightly to prevent moisture loss. If the samples are not to be processed immediately, they should be kept cool or frozen. This will prevent further decay of organic matter and loss of weight by the formation of gases.

The top of the core should be sampled with care. If the water above the sediment-water interface is slightly turbid, the sediments should be permitted to settle until the sediment-water interface is clearly visible. If the top of the core has a high water content, the top intervals should be sampled with some form of suction device such as a large syringe. When the sediment of the next sample interval has a firmer consistency, an apparatus that removes the outer smeared portion should be used. Ideally this should be a stainless-steel ring that "peels off" the outer portion. When using such a stainless-steel ring it is important to keep the sediment moving pass the ring. If the sediment is not moving past the ring then it will pile up producing a longer core than actually present. A thin narrow spatula may be used to keep fibrous organic matter from clogging up the cutting edge of the stainless-steel ring. For later calculations, a note should be made of the internal diameter of the core tube and the internal diameter of the stainless-steel ring. The number of samples for which the suction device was used should be noted. These data are stored at the top of the sediment file (Table 8-1). The examples (Tables 8-1 and 8-2) represent data obtained from Hamilton Harbour, Ontario, core (137) collected by Dr. N. Rukavina (Rukavina, 1988; Turner & Delorme, 1988).

SAMPLE PREPARATION

The dry and wet weight of the sediment samples are required for this procedure. When the core is collected, the vial or jar (without the lid) containing the sediment is weighed and the weight recorded. The next step is to dry the sample. Oven drying methods can be used if the samples are to be ground for later analyses. Freeze drying is the preferred method of many researchers for removing the moisture from sediment samples.

Freeze drying has the advantage of leaving the dried residue in a disaggregated state. In this procedure the slice of the core is placed in a vial, frozen, and then placed into an evacuation chamber under a vacuum. After about 48 hours, depending on the size of the freeze dryer, the ice will have been sublimated leaving disaggregated sediment. With all the sediment retained, analyses for shelled invertebrates (Delorme, et al., 1977), fossil pollen and diatoms (Delorme et al., 1986), grain size analyses (Duncan & LaHaie, 1979), specific weight (Holloway & Delorme, 1987), some geochemical work (Wong et al, 1984), ^{210}Pb (Turner & Delorme, 1988), and ^{14}C dating can be carried out on the same sample. Freeze drying sediments distorts beetle remains (A. Morgan, per. comm.). The dry weight, including the vial weight (without the lid), also should be recorded. Table 8-1 shows the data file as it is set up for later calculations producing the data in Table 8-2. Tables 8-1 and 8-2 can be set up as a spreadsheet for a personal computer.

CALCULATIONS

The **wet weight** ("g"; Table 8-2, column 2) of the core slice is obtained by subtracting the vial weight from the wet weight + vial weight (Table 8-1, column 2 minus column 4).

$$W_w = W_{wv} - W_v \qquad (8\text{-}1)$$

where W_w is the wet weight of the i^{th} sample in grams;
W_{wv} is the wet weight including the vial weight in grams;
W_v is the vial weight (without the lid) in grams.

The **dry weight** ("g"; Table 8-2, column 3) of the i^{th} sample is the vial weight subtracted from the dry weight + vial weight.

$$W_d = W_{dv} - W_v \qquad (8\text{-}2)$$

where W_d is the dry weight of the sediment in grams;
W_{dv} is the weight of the dry sediment including the vial weight in grams.

The **cumulative dry weight** ("g cm^{-2}"; Table 8-2, column 4) is the cumulative sum of the dry weight by sample divided by the surface area of the stainless-steel ring.

$$W_{dc} = \Sigma_{i=n}^{i=1} W_d / A_{cc} \tag{8-3}$$

where W_{dc} is cumulative dry weight in grams;
A_{cc} is the area (cm^2) occupied by the stainless-steel ring.

Samples taken directly from the top of the core tube should have the dry weight of the sample standardized to the same dry weight as those samples extruded through the stainless-steel ring. This is calculated by: {(dry wt. * surface area of the stainless-steel ring)/ surface area of the top sample slice}. Note in Table 8-1 the diameter of the stainless-steel ring is given in line four. Line six (Table 8-1) indicates the number of top samples for which the stainless-steel ring was not used. For example the surface area would be 33.166 cm^2 for the top two samples and 19.625 cm^2 for the remainder of the samples.

The **water content** of the i^{th} sample ("g or cm^{-3}" assuming a specific weight of one for water) is the dry weight subtracted from wet weight (Table 8-2, column 2 from minus 3).

$$W_i = W_w - W_d \tag{8-4}$$

where W_i is the water content of the i^{th} sample in grams or cm^3.

Specific weight or density (Chilingarian & Wolf, 1975) of the dry sediment is obtained by using the Duncan and St. Jacques (1979) method or by using an automated Multivolume Pycnometer (Frazer, 1989). Specific density is the dry weight of the sediment divided by the volume of the sediment in the sample. Several determinations (10 to 15) are made per core and the average used. This value (Turner & Delorme, 1988; 2.49 ± 0.24 g cm^{-3}) is placed in line two of Table 8-1. Where there is a high degree of variability in the sediment density of a core, it is better to determine the density of the sediments for each sample or to interpolate values between every 5 to 10 cm. Density affects the calculations of sediment volume, and therefore porosity, and uncompacted depth. It should be noted that specific sediment density is different from bulk density. Bulk density is the dry weight of the sample divided by the volume of the sample.

Dry sediment volume ("cm^3"; Table 8-2, column 6) is calculated from the dry weight divided by the specific weight.

$$SV_i = W_d/\overline{\rho}_s \tag{8-5}$$

where SV_i is the sediment volume of the i^{th} sample;

$\overline{\rho}_s$ is the specific weight of the sediment.

Attempts to slice the core into a specified thickness accurately to produce a constant volume is rarely attained.

Total volume of the i^{th} sample ("cm^3"; Table 8-2, column 7) is obtained by taking the sum of water content and sediment volume (column 5 + column 6, Table 8-2).

$$TV_i = W_i + SV_i \tag{8-6}$$

where TV_i is the total volume of the i^{th} sample.

The geometric form of the i^{th} sample is a cylinder so the **height or thickness** is the volume divided by the surface area ("cm"; Table 8-2, column 8).

$$t_{ci} = TV_i / A_i \qquad (8\text{-}7)$$

where t_{ci} is the compacted thickness of the i^{th} sample in cm;
A_i is the surface area of the i^{th} sample.

Compacted refers to the natural state of the sediment, when collected, with a given amount of pore fluid expelled relative to the amount originally held. Compacted thickness is then the same as the actual thickness.

The **compacted depth** ("cm"; Table 8-2, column 9) is the cumulative sum of the compacted thicknesses.

$$D_{ci} = \Sigma^{i=1}_{i=n} t_{ci} \qquad (8\text{-}8)$$

where D_{ci} is the cumulative thickness at the compacted i^{th} sample.

The **compacted mid-point or depth** ("cm"; Table 8-2, column 10) is the compacted thickness divided by two and added onto the previous value of the compacted upper depth boundary of the slice.

$$\overline{t}_{ci} = (t_{ci} / 2) + t_{c(i-1)} \qquad (8\text{-}9)$$

where $\overline{t}_{ci}$ is the compacted mid-point or mid-depth in cm.

Porosity is the ratio of the void spaces, usually (some gases may be present) occupied by water in water-saturated sediments, to the bulk or total volume (Chilingarian & Wolf, 1975). Porosity and hence calculated depth values should not be used where there are excessive gases present in the sediments as the volume calculations will be in error. Porosity is expressed as a percent. Sample porosity ("%"; Table 8-2, column 11) is calculated by dividing the water content by total volume (Table 8-2, [column 5/column 7] x 100).

$$\phi_i = (W_i / TV_i) * 100 \qquad (8\text{-}10)$$

where ϕ is the porosity of the i^{th} sample expressed as a percentage.

There are two methods of calculating the **uncompacted thickness**, by using porosity or sediment volume. By uncompacted thickness is meant the thickness of a sample collected at depth <u>i</u> if the sample was to a have a water content equal to that of the surface sample. Equation 8-11 requires the sediment volume and total volume; equation 8-12 requires porosity and total volume for each sample. In determining the uncompacted thickness ("cm"; Table 8-2, column 12), it is assumed the sediments in the top sample are not compacted. It is reasoned that this assumption is valid because of the near absence of loading and therefore compaction or cementation by other sediment particles. Not all cores are suitable for determination of porosity or uncompacted depth. If an erosional surface is detected at the sediment-water interface, the core should not be used for calculating porosity, uncompacted depth, or for ^{210}Pb dating because the results would be misleading. Sometimes a physical disturbance might not be observed until after the calculations have been completed. Lower than expected porosity values (< 85%) may be such an indication. Accepting the core as suitable, a comparison is made between sediment volume of the i^{th}

sample and the sediment volume of the top sample. A comparison is also made of the total volume of the i^{th} sample to the total sample volume of the top sample. This cross product is subtracted from one and added onto the compacted thickness. One of the following two equations may be used to calculate the uncompacted thickness (t_{ui}).

$$t_{ui}=\{(SV_i\ /SV_o)(TV_o\ /TV_i)\}\ -1\ +\ (TV_i\ /V_q) \quad (8\text{-}11)$$

$$t_{ui}=\{(\phi_o\ -\ \phi_i)\ /\ (1\ -\ \phi_o)\}\ +\ (TV_iV_q) \quad (8\text{-}12)$$

where t_{ui} is the uncompacted thickness of the i^{th} sample;
SV_o is the sediment volume of the initial sample;
TV_o is the total volume of the initial sample;
ϕ_o is the porosity of the initial sample;
V_q is the volume of cylinder 1 cm high and surface area equal to either inside of the core tube or stainless-steel ring whichever is appropriate.

The **uncompacted depth**, D_{ui} ("cm"; Table 8-2, column 13), is the cumulative sum of the uncompacted thicknesses.

$$D_{ui}\ =\ \Sigma^{i\ =\ 1}_{i\ =\ n}t_{ui} \quad (8\text{-}13)$$

The **uncompacted mid-point or depth**, $\bar{t}_{ui}$ ("cm"; Table 8-2, column 14), is the uncompacted thickness divided by two and added onto the previous value for the uncompacted depth.

$$\bar{t}_{ui}\ =\ (t_{uci}\ /2)\ +\ t_{uc(i-1)} \quad (8\text{-}14)$$

Time B.P. (Before Present, years; Table 8-2, column 15) is based on a time marker or is calculated after the sedimentation rate has been determined. Present refers to the year the core was collected. Note: sedimentation rate is entered on line 5 of Table 8-1.

DEPTHS USED FOR CALCULATING SEDIMENTATION RATES

The sedimentation rate (cm yr^{-1}) is obtained by taking the depth from the surface or other reference point to another dated point in the core. This number is then divided by the number of years between the two points.

Mass sedimentation rate (g $cm^{-2}yr^{-1}$) is calculated by dividing the cumulative dry weight (Table 2, column 4) by the time in years (Table 8-2, column 15 or column 14/sedimentation rate).

In a core (137) from Hamilton Harbour in Burlington Bay, Ontario, <u>Ambrosia</u> sp. was introduced into the area around 1828 A.D. (Harper & Delorme, 1988). This would be 160 years before the core was collected. Use of the point of change method (Esterby & El-Shaarawi, 1981a, 1981b) shows, in an objective way, this event began at an uncompacted mid-depth of 58.6 cm (Table 8-2, sample 30). The mid-position of the sample, as an uncompacted mid-depth, is used because it is most representative of when the event happened. The sedimentation rate for the top part of this core (about 60 cm) is 0.37 cm yr^{-1} (Turner & Delorme, 1988). This rate is based on a linear sedimentation rate using porosity and uncompacted depth in the equation.

Use of the compacted or natural mid-depth of 31.7 cm (Table 8-2, sample 30), gives a sedimentation rate of 0.20 cm yr^{-1}. This is 46% lower than that calculated using uncompacted depth. For a compacted sample thickness of 0.94 cm (Table 8-2, sample 30), this represents 4.70 years of deposition. An uncompacted thickness of

2.06 cm (Table 8-2, sample 30) represents 5.57 years of deposition or 16% more time to deposit the sediments. Note the difference in sedimentation rate and time span of uncompacted and compacted thickness. Using compacted depth to calculate sedimentation rates ignores the non-linear aspects of the depth measurement and produces erroneous rates.

Mass sedimentation rate is calculated using the cumulative dry weight of 17.02 g cm^{-2} (Table 8-2, sample 30) at an uncompacted depth of 58.6 cm. The rate is 0.11 g cm^{-2} yr^{-1} (17.02/(58.6/ .37)). This compares to 0.10 g cm^{-2} yr^{-1} calculated using the mass sedimentation rate 'ω'from equation 2 of Turner and Delorme (1988) and Robbins and Edgington (1975).

SUMMARY

The preparation and handling of sediments, after a core has been collected, should be done with a protocol in mind. The procedure will vary depending on analyses to be done on the sediments. The sediment-water interface should be intact and the water above be clear. When extruding the core, the smeared outer portion should be removed. Slices of about 1 cm in thickness are placed in vials, weighed, dried, and reweighed. The wet and dry weight of the sediments are used to calculate the uncompacted depth, thickness, and porosity. The uncompacted depth is used to calculate the sedimentation rate, as it most closely represents the conditions at the time of sediment deposition. Using the compacted

depth will give a lower value as compared to the actual sedimentation rate.

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Table 8-1. Wet and dry weights for station 8A (Core 137), Hamilton Harbour collected by Dr. N. Rukavina (Turner & Delorme, 1988).

137 CORE NUMBER
2.486 SPECIFIC GRAVITY G CM^{-3}
33.166 SURFACE AREA, 6.50 CM TUBE DIAMETER
19.625 SURFACE AREA, 5.00 CM CUTTER DIAMETER
0.368 RATE OF SEDIMENTATION CM YR^{-1}
2 NUMBER OF SAMPLES BELOW THE SURFACE BEFORE THE DIAMETER CHANGES TO CUTTER DIAMETER; IF ""ZERO"" SURFACE AREA = 33.166 OR VALUE OF LINE 4.

Samp No.	Wet[1] Wt.	Dry[1] Wt.	Vial Wt.
1	50.27	20.86	11.79
2	49.63	22.93	11.75
3	34.26	19.34	11.57
4	33.45	19.56	11.91
5	35.82	20.85	11.57
6	37.26	21.71	11.68
7	38.57	22.75	11.78
8	37.61	22.38	11.78
9	37.24	22.42	11.64
10	40.38	23.46	11.52
11	37.92	22.93	11.92
12	45.16	25.40	11.66
13	41.00	23.35	11.58
14	42.49	23.60	11.64
15	37.11	21.30	11.52
16	36.91	21.22	11.69
17	38.42	22.22	11.75
18	41.11	23.41	11.57
19	39.40	23.37	11.73
20	40.47	23.23	11.75
21	43.15	24.28	11.67
22	40.91	23.66	11.77
23	36.11	22.16	11.73
24	40.45	24.21	11.68
25	45.26	26.32	11.77
26	46.20	26.78	11.76
27	47.63	27.70	11.57
28	40.24	24.17	11.73
29	42.98	25.32	11.82
30	36.57	22.49	11.79

[1]Includes vial weight

Table 8-2. Calculation of porosity and uncompacted depths given sample wet and dry weights, and specific gravity (Table 8-1) from Station 8A, core 137 Hamilton Harbour (Turner & Delorme, 1988).

Sample Number	Wet Wt. g	Dry Wt. g	Cumm. Dry Wt g cm^{-2}	Water Cont. cm^3	Sed. Vol. cm^3	Total Vol. cm^3	Comp. Thick cm	Comp. Depth cm	Comp. Mid-pt cm	Sample Poros. %	Uncomp. Thick. cm	Uncomp Depth cm	Uncomp. Mid-pt. cm	Time B.P. Years
1	38.48	9.07	0.27	29.41	3.65	33.06	1.00	1.00	0.50	88.96	1.00	1.00	0.50	1
2	37.88	11.18	0.61	26.70	4.50	31.20	0.94	1.94	1.47	85.58	1.25	2.25	1.63	4
3	22.69	7.77	1.01	14.92	3.13	18.05	0.92	2.86	2.40	82.68	1.49	3.74	3.00	8
4	21.54	7.65	1.40	13.89	3.08	16.97	0.86	3.72	3.29	81.86	1.51	5.25	4.50	12
5	24.25	9.28	1.87	14.97	3.73	18.70	0.95	4.67	4.20	80.04	1.76	7.01	6.13	17
6	25.58	10.03	2.38	15.55	4.03	19.58	1.00	5.67	5.17	79.40	1.86	8.87	7.94	22
7	26.79	10.97	2.94	15.82	4.41	20.23	1.03	6.70	6.19	78.19	2.01	10.88	9.88	27
8	25.83	10.60	3.48	15.23	4.26	19.49	0.99	7.70	7.20	78.13	1.98	12.86	11.87	32
9	25.60	10.78	4.03	14.82	4.34	19.16	0.98	8.67	8.18	77.36	2.03	14.89	13.88	38
10	28.86	11.94	4.64	16.92	4.80	21.72	1.11	9.78	9.23	77.89	2.11	17.00	15.95	43
11	26.00	11.01	5.20	14.99	4.43	19.42	0.99	10.77	10.27	77.19	2.06	19.06	18.03	49
12	33.50	13.74	5.90	19.76	5.53	25.29	1.29	12.06	11.41	78.14	2.27	21.33	20.20	55
13	29.42	11.77	6.50	17.65	4.73	22.38	1.14	13.20	12.63	78.85	2.06	23.39	22.36	61
14	30.85	11.96	7.11	18.89	4.81	23.70	1.21	14.41	13.80	79.70	2.05	25.44	24.42	66
15	25.59	9.78	7.61	15.81	3.93	19.74	1.01	15.41	14.91	80.07	1.81	27.25	26.35	72
16	25.22	9.53	8.09	15.69	3.83	19.52	0.99	16.41	15.91	80.36	1.77	29.02	28.14	76
17	26.67	10.47	8.62	16.20	4.21	20.41	1.04	17.45	16.93	79.37	1.91	30.93	29.98	81
18	29.54	11.84	9.23	17.70	4.76	22.46	1.14	18.59	18.02	78.80	2.07	33.00	31.97	87
19	27.67	11.64	9.82	16.03	4.68	20.71	1.06	19.65	19.12	77.39	2.10	35.10	34.05	93
20	28.72	11.48	10.41	17.24	4.62	21.86	1.11	20.76	20.20	78.87	2.03	37.13	36.12	96
21	31.48	12.61	11.05	18.87	5.07	23.94	1.22	21.98	21.37	78.81	2.14	39.27	38.20	104
22	29.14	11.89	11.65	17.25	4.78	22.03	1.12	23.10	22.54	78.29	2.09	41.36	40.31	110
23	24.38	10.43	12.19	13.95	4.20	18.15	0.92	24.03	23.57	76.88	2.02	43.38	42.37	

24	28.77	12.53	12.82	16.24	5.04	21.28	1.08	25.11	24.57	76.31	2.23	45.61	44.49
25	33.49	14.55	13.57	18.94	5.85	24.79	1.26	26.38	25.74	76.39	2.40	48.01	46.81
26	34.44	15.02	14.33	19.42	6.04	25.46	1.30	27.67	27.02	76.27	2.45	50.46	49.23
27	36.06	16.13	15.15	19.93	6.49	26.42	1.35	29.02	28.35	75.44	2.57	53.03	51.74
28	28.51	12.44	15.79	16.07	5.00	21.07	1.07	30.09	29.56	76.26	2.23	55.26	54.14
29	31.16	13.50	16.48	17.66	5.43	23.09	1.18	31.27	30.68	76.48	2.31	57.57	56.41
30	24.78	10.70	17.02	14.08	4.30	18.38	0.94	32.21	31.74	76.59	2.06	59.63	58.60

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