MEASURING AND MODELLING THE DYNAMIC RESPONSE OF REMOTE MOUNTAIN LAKE ECOSYSTEMS TO ENVIRONMENTAL CHANGE

A programme of **MO**untain **LA**ke **R**esearch

MOLAR

PROTOCOL FOR GRAIN SIZE MEASUREMENTS OF SEDIMENT CORE SAMPLES

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Protocol for Grain Size Measurements of Sediment Core Samples

1. Introductionary note

Grain size measurements of sediments are important to understand the sedimentological history of a lake. They provide information on the formation of sediments and on the stability or irregularities of a given sedimentary record. However, techniques of grain size measurements are numerous and overspan a whole range of techniques from very simple, low cost methods to highly sophisticated, high precision and expensive techniques.

As some of the MOLAR laboratories may not be suffiently equipped with appropriate instruments, two methods for grain size determinations are recommended within the following GrainSizeManual.

2. General

Grain sizes of unconsolidated sediments can very basically be divided into three different fractions (all grain size values in this manual are given in [µm], which describes the largest diameter of a given particle):

a. Very fine particles, showing particle diameters of
   <2 µm = clay fraction

b. medium particles, showing particle diameters of
   2 to 63 µm = silt fraction
   The silt fraction may be further divided into three sub-fractions:
   fine silt = 2 - 10 µm
   medium silt = 10 - 20 µm
   coarse silt = 20 - 63 µm

c. Coarse particles, showing particle diameters of
   >63 µm = sand fraction

3. Sample preparation

For any technique of grain size determination sediment samples have to be either wet or freeze dried. Using freeze dried samples makes it a bit easier at the end, to calculate percentages of different size fractions.

There is one paramount rule in the grain size business: **NEVER USE OVEN-DRIED OR AIR-DRIED SAMPLES FOR GRAIN SIZE ANALYSES.** Grain size results will then always be arbitrary and wrong.

For most lake sediments it is appropriate to use bulk sediment samples for grain size determination, e.g. without eliminating the organic material from the samples prior to analysis.

Commonly sample preparation is simple for most of the different methods: An aliquot (1 to 5 g) of the wet or freeze dried sample has to be dispersed into a Calgon® solution. This is to avoid koagulation and the formation of larger flocs of smaller particles. To enhance homogeneous dispersion of particles, the suspension should subsequently be placed into an ultrasonic bath or be homogenized with an ultrasonic stirrer.

3.1 Sieving technique (particle fractions of <10 µm to >63 µm)

This basic, low cost grain size determination technique requires three to four measuring sieves, a centrifuge and an ultrasonic bath or an ultrasonic stirrer. If samples are very coarse, one may use additional sieves for sand fractions (e.g. mesh sizes of 125 µm, 250 µm etc.). This 'quick and dirty' technique, does not allow to determine the amount of clay fraction (<2 µm) in a sample; additionally, it is rather inaccurate determining particle fractions between 10 to 30 µm (more accurate determination of small size fractions without automated instrumentation could be performed by time- and labor-intensive methods such as pipette- or Atterberg-cylinders; quicker analyses with an automated sizing method see below).

- Take 5 g of freeze dried sample (or an equivalent amount of wet sample)and disperse sample into 250 ml of a Calgon® solution of 0.01%

- Homogenize the suspension with an ultrasonic stirrer for 3 minutes (US-bath 10 minutes)

- Place three measuring sieves of mesh sizes of 10 µm, 20 µm and 63 µm over a beaker with a volume of approx. 2 l
  (additional sieves with mesh sizes of 30 µm, 40 µm and 50 µm are optional, but would require larger initial sample sizes)

- Rinse sample suspension carefully through the sieves with as little as possible additional water until size fractions are clearly separated on top of the individual sieves

- Keep the effluent water (e.g. with the particles <10 µm) in the beaker

- Carefully transfer particles from sieves into preweighted beakers, ovendry samples and determine weights of individual size fractions of 10-20 µm, 20-63 µm and >63 µm

- Transfer effluent with particles <10 µm into a centrifuge for 10 minutes with 5000 rpm

- Determine weight of residual matter and calculate the amount of particles <10 µm

3.2 Automated particle sizing technique (particle fractions of <0.5 µm to >100 µm)

This sizing technique, based on the principles of Stokes's equation on vertical particle velocities in aqueous media, uses a SEDIGRAPH®, which is an automated, microprocessor controlled particle size analysator, to determine continuous particle distributions between

0.5 µm and 100 µm. Required time for an analysis of size fractions between 1 µm and 63 µm is about 20 minutes. Results can be directly compared to pipette- and Atterberg-techniques. To compare results with Laser® particle sizer, values have to be transformed from mass % (SEDIGRAPH®) to volume % (Laser® instruments).

3.2.1 i If sample is free of sand (>63 µm) take 1-2 g of freeze dried sample (or an equivalent amount of wet sample) and dispers into 250 ml of a Calgon® solution of 0.01%

ii Homogenize the suspension with an ultrasonic stirrer for 3 minutes and transfer dispersed suspension into SEDIGRAPH® for further analyses

iii Use continuous digital or analog data output of SEDIGRAPH® for calculation of particle sizes

3.2.2. i In case that samples contain considerable amounts of coarse particles (>63 µm), take 2-5 g of freeze dried sample (or an equivalent amount of wet sample) and dispers into 250 ml of a Calgon® solution of 0.01%

ii Homogenize the suspension with an ultrasonic stirrer for 3 minutes

iii Place one measuring sieve of mesh size of 63 µm over a beaker of approx. 1-2 l

iv Rinse sample suspension carefully through the sieve with as little as possible additional water until size fraction >63 µm is clearly separated on top of sieve

v Carefully transfer particles from sieve into preweighted beaker, oven-/air-dry sample and determine weight of fractions >63 µm

vi Keep the effluent water (e.g. with all particles <63 µm) in the beaker

vii Transfer effluent with particles <63 µm into a centrifuge for 10 minutes with 5000 rpm

viii Transfer residual matter into 250 ml of a Calgon® solution of 0.01% and perform like above (2A.)

4. Data preparation

Results from SEDIGRAPH® (or similar automated techniques) should be graphically presented both as histogram and as sum curve of the grain size distribution.

Moreover, the results from SEDIGRAPH® (or similar automated techniques) should be used to calculate the following size fractions and some statistical values of the grain sizes of an individual sample:

size fractions: <1 µm, 1-2 µm, 2-4 µm, 4-8 µm, 8-16 µm, 16-32 µm, 32-63 µm, >63 µm (or additional sand fractions if available)
statistical values: median and mean diameter, sorting, skweness.

5. Final note

It is essential that the samples taken for grain size analyses are kept wet or freeze-dried until grain size analyzes is performed. Again (see above): air-dried or oven-dried material

cannot be used for grain size analyses. Just to add: grinded samples can also not be used.

In order to obtain homogenized and comparable data of grain size analyses from the different MOLAR laboratories I recommend that a list of grain size measuring techniques is compiled, where each Institute states, which technique is available and/or can be used for the project. Keep in mind that grain sizes should be measured for all the samples, which are taken of cores from the different MOLAR-sites.

Table 1 The most common size fractions

Sizes of sediment particle fractions (diameter values)

[mm]	[mm]	[µm]	phi-units [-log2 mm]	WENTWORTH-units
1/1024	0.00098	1	10.0	clay
1/512	0.00195	2	9.0	fine silt
1/256	0.00391	4	8.0	fine silt
1/128	0.00781	8	7.0	fine silt
1/064	0.01563	16	6.0	medium silt
1/032	0.03125	31	5.0	coarse silt
1/016	0.06250	63	4.0	coarse silt
1/008	0.12500	125	3.0	fine sand
1/004	0.25000	250	2.0	medium sand
1/002	0.50000	500	1.0	coarse sand
1	1.00000	1000	0.0	coarse sand
2	2.00000	2000	-1.0	fine gravel
4	4.00000	4000	-2.0	fine gravel
8	8.00000	8000	-3.0	medium gravel
16	16.00000	16000	-4.0	medium gravel
32	32.00000	32000	-5.0	coarse gravel

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Equipment for Grain Size Analysis atEAWAG.

sieves [20 µm to 500 µm)
pipette-technique
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