

DETERMINATION OF PARTICLE SIZE DISTRIBUTION (GRAVEL, SAND, SILT AND CLAY) IN SEDIMENT SAMPLES

Scott Laswell⁺, Susanne J. McDonald*, Alan D. Watts* and James M. Brooks*

*TDI-Brooks International/B&B Laboratories Inc.

College Station, Texas 77845

⁺Laser Geo-Environmental

Austin, Texas 78748

ABSTRACT

Grain size distribution in marine sediments is determined using the Wentworth scale. The major size classes determined are gravel (-2 phi to -5 phi), sand (+4 phi to -1 phi), silt (+5 phi to +7 phi) and clay (+8 phi and smaller). Determining particle size in sediments is important due to potential correlations with contaminant levels. Sediments are pre-treated with hydrogen peroxide to remove organic matter prior to particle size determination.

1.0 INTRODUCTION

Higher contaminant concentrations are often associated with finer grain sediments. Consequently, useful correlations can be made between particle size distribution and contaminant concentrations. Particle size distribution is a cumulative frequency distribution, or a frequency distribution of relative amounts of particles in a sample within specified size ranges (phi scale is normally used). The size of a discrete particle is characterized as a linear dimension, and is designated as a particle diameter. The use of sieves and settling tubes, as described below, results in a sediment description based on particle size and density, reported as percentages of the total sample weight.

2.0 APPARATUS AND MATERIALS

2.1 EQUIPMENT

- Balance, analytical accurate to 0.1 mg
- Calibrated weights, certified
- Oven, electric convection, capable of maintaining stable temperatures of at least 200°C
- Desiccator, cabinet style
- Spatulas, stainless steel

- Beakers, glass 50 mL and 150 mL
- Glass pipette, 25 mL
- Wide mouth jars, 32-ounce
- Wet sieve, 8-inch #230 (63 μm)
- 9-inch plastic funnel
- Metal ring stand
- Graduated cylinder, 1000 mL
- Plunging device
- Sieve shaker, Humboldt H-4325
- Testing sieves, 8-inch diameter, #10 (2000 μm , -1 phi), #230 (63 μm , +4 phi), sieve cover, sieve bottom pan

2.2 REAGENTS

- Acetone, reagent grade
- Dichloromethane, pesticide grade or equivalent
- Deflocculent solution (5 g/L sodium hexametaphosphate in DI water)
- Hydrogen peroxide, 30%

3.0 PROCEDURE

Sediments are collected in plastic bags, plastic bottles or glass bottles and stored refrigerated (4°C) until analysis.

Samples are thoroughly mixed. Approximately 45 –50 g of sandy sediment or 20 to 25 g of muddy sample is weighed into a 32-ounce wide-mouth jar. Sufficient water is added to cover the sample. The jars are placed under a hood and small quantities of 30% hydrogen peroxide (H_2O_2) are carefully added until all reactions (bubbling) have ceased. Once the bubbling has stopped, the jars are placed into a 65°C water bath and small amounts of H_2O_2 continue to be added until no more reaction is observed. The jars are removed from the bath and cooled. Once the jars are cool, 20 mL of deflocculent solution and 30 mL of deionized water are added. The jars are sealed and shaken until the sample is disaggregated. The sample and deflocculent solution are poured through a 63 μm sieve into a 1,000 mL graduated cylinder using deionized water to rinse all material out of the jar. The sediment remaining on the sieve is thoroughly rinsed with deionized water to ensure that the particles smaller than 63 μm pass through the sieve. The volume in the graduated cylinder must remain under 1,000 mL. The coarse sediment remaining on the sieve is gently concentrated against the bottom lip of the sieve. The sediment on the sieve is rinsed with deionized water into a labeled 150 mL beaker. The coarse sediment in the beaker is dried in an oven at 70 °C to 90 °C. . Once the coarse fraction is dry, the material from the 150 mL beaker is transferred to the top sieve in a sieve stack that is arranged in descending order (i.e., -1 phi and +4 phi). A bottom collection pan and top is added to the sieve stack and shaken for 15 minutes on the shaker. The top sieve (-1 phi) is removed

and emptied onto a large piece of clean paper, using a brush to remove as much sediment as possible. The sediment from the paper is poured into a tarred weighing boat and weighed to the nearest 0.0001 g. Next the bottom sieve (+4 phi) is inverted and brushed clean over the paper and added to the weighing boat. The total weight of the sediment from both sieves to is recorded to obtain a cumulative weight. The difference in the values of the cumulative weight, and the weight of the gravel fraction alone, represents the weight of the sand fraction. Any remaining sediment in the bottom pan (<63µm) is added to the corresponding graduated cylinder.

The silt/clay fraction is determined by filling the corresponding graduated cylinder to 1,000 mL with deionized water. The cylinder is covered and maintained at 24 °C for approximately 24 hours. After 24 hours, the contents of the cylinder are mixed for 1 minute with the plunger. The contents of the cylinder are allowed to sit undisturbed for 20 seconds, after which a 25 mL aliquot is withdrawn with a calibrated pipette from a depth of 20 cm. The 25 mL aliquot is placed in a labeled pre-weighed 50 mL beaker. The pipette is washed with deionized water and the wash is added to the sample beaker. This first aliquot represents 1/40 of the total fine sediment (silt/clay) in the sample. In order to separately report the clay fraction (+8 phi and smaller) and the silt fraction (+5 phi to +7phi), a second 25 mL aliquot is withdrawn with a calibrated pipette from a depth of 10 cm at intervals determined by the temperature of the liquid in the cylinders. The aliquot is placed into a second labeled pre-weighed 50mL beaker. The pipette is washed with deionized water and added to the sample beaker. This second aliquot represents 1/40 of the total clay fraction in the sample. The difference in the dry weights of the first aliquot (silt/clay) and the second aliquot (clay) represents 1/40 of the total silt fraction in the sample. All beakers are placed into an oven at 70 °C to 90 °C until dry. Once samples are dry they are transferred to a desiccator. Samples are allowed to cool and are then weighed.

4.0 CALCULATIONS

4.1 SAMPLE WEIGHT

The weight of each fraction is calculated using the following equation:

$$\text{Dry Wt.} = (\text{Vessel Wt.} + \text{Dry Wt.}_{\text{sample}}) - (\text{Vessel Wt.})$$

4.2 PIPETTE CALIBRATION FACTOR

A pipette calibration factor is determined for each analyst to minimize weighing errors associated with imprecise pipetting of sample aliquots. A 25 mL volumetric pipette is filled with deionized water, emptied into a clean pre-weighed 50 mL beaker and weighed (which represents 1/40 of the total volume in a 1,000 mL graduated cylinder). This

process is repeated nine more times. The mean water weight is determined and the following equation is used to calculate the pipette calibration factor for each analyst.

$$\text{Pipette Calibration Factor} = (40) \frac{\text{Mean Water Wt.}}{25}$$

5.0 QUALITY CONTROL (QC)

A duplicate analysis is performed for every 20 samples analyzed with a QC batch. A second aliquot of a sample is processed identical to all other samples. The relative percent difference (RPD) is calculated for all duplicate samples and should be no more than 25%. If this criterion is not met after re-weighing the duplicate samples, corrective action is taken which may result in re-processing all samples in the QC batch.

The RPD for each duplicate is calculated using the following equation:

$$\text{RPD} = \frac{(\text{Particle Size Wt.}_{\text{sample1}} - \text{Particle Size Wt.}_{\text{sample2}})}{(\text{Particle Size Wt.}_{\text{sample1}} + \text{Particle Size Wt.}_{\text{sample2}})(0.5)} \times 100$$