Sediment Grain Size Analysis for K-12 Teachers: Materials, Safety, Standards
Instructional Protocol

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This procedure provides relative proportions of sand, silt, and clay in beach sediments that contain a lot of silt and clay, such as we find on Louisiana's barrier islands in the Mississippi River delta. I adapted it in 2007 for classroom teachers of Earth, Environmental and Physical Sciences. Please send suggestions and criticisms for future revisions.

Adapted from
Folk, R.L., 1980. *Petrology of Sedimentary Rocks*. Hemphill Publishing Co., Austin, TX 78703;
http://pasternack.ucdavis.edu/protocols/grainsize.doc;
http://pubs.usgs.gov/of/2000/of00-358/text/contents.htm;
http://pubs.usgs.gov/of/2000/of00-358/text/chapter1.htm;
http://csmres.jmu.edu/geollab/fichter/SedRx/readternary.html;
http://www.geology.iupui.edu/research/SoilsLab/procedures/psd.
Environmental Indicators for Agriculture – Vol. 3: Methods and Results, OECD, 2001, glossary, p. 389-391.
http://stats.oecd.org/glossary/detail.asp?ID=2419

If you have questions after reading this, or would like to analyze samples unlike those described here, Pasternack provides steps for a wider array of samples formatted as a recipe. Folk has the literature review, helpful tips, and good discussion of why the procedures are as they are. The US Geological Survey (USGS) website provides video demonstrations of laboratory methods. The James Madison University website provides an excellent discussion of how to use a ternary diagram, a link to a site where you can download free software to do this on the computer, and a presentation that discusses using ternary diagrams to classify your samples. I strongly suggest you review it (and consider using pieces of it in your class, with appropriate attribution). The withdrawal time table used here is excerpted from the one published on the Soils Lab website. The definition of sediment was provided by the Environmental Indicators for Agriculture reference on the glossary website.

Sediment is defined as material of varying size, both mineral and organic that is being, or has been, moved from its site of origin by the action of wind, water, gravity, or ice, and comes to rest elsewhere on the earth's surface. Grain size distribution is one of the most important characteristics of sediment. Grain size allows an investigator to interpret the speed of fluid (water, air) from which a sample of particles was deposited. High speed flows can carry coarser sediment than lower speed flows. A flow that is increasing in speed will pick up progressively coarser particles, while a flow that is decreasing speed will drop progressively finer sediment particles.

Beach sediments consist of particles from the fine end of the of the size continuum (the range of possible sediment sizes). Terms are assigned to particles within specific size ranges. Gravel consists of particles larger than 2mm. Sand includes particles between 2mm and 62.5μm. Silt includes particles between 62.5μm and 2μm (or 4μm). When used in reference to size, clay refers to sediment smaller than 2μm (or 4μm). The term mud is used here (as is frequently done in sediment literature) to refer to all sediment smaller than sand, that is the fraction consisting of silt and clay.

Note 1: The distinction between clay and silt is subject to discussion because of the behavior and composition of these very small grains. Some scientists use the 2μm cutoff, some use 4μm. I have always used the 2μm cutoff. Whatever you do, be consistent to avoid confusing your students.

Note 2: The term clay refers to size as stated above, but it is also a term that scientists use to refer to the composition or mineralogy of a particle. Clay minerals are fine grained, sheet-like (or flaky) aluminosilicate particles that have a negative surface charge that makes them join in aggregates of particles.

	Finer than	Coarser than
Gravel	-	2mm
Sand	2mm	62.5μm
Silt	62.5μm	2μm (4μm)
Clay	2μm (4μm)	-

Sediments typically include a broad distribution of sizes. Upon measuring the number or weight of particles in each size class or size fraction, geologists graph a sediment distribution from which they can calculate many statistics that allow them to interpret the depositional environment of the sample. This procedure lists the steps to get a less detailed size distribution than is sometimes used in geology; it does not provide the statistics. However, the sand, silt, and clay ratios this method returns provide much useful information and are less time consuming to obtain. Plotting them on a triangular (ternary) diagram helps in the interpretation and comparison of different samples.

NOTES ON SAFETY, BACKGROUND, AND TECHNIQUE

TECHNIQUES FOR SEDIMENT SIZE ANALYSIS

There are a wide variety of techniques for measuring grain size. Because of the broad distribution of sediments (from larger than a house to smaller than the diameter of a human hair) and because their composition and behavior change from coarser to finer particles, the technique analysts choose depends on the size range of interest. For the beach sediment considered here, two different techniques will be used, one that works well for sand and gravel, and one that works well for silt and clay.

Sieve Separation of Gravel to Sand-sized fraction.

- This method is direct – it measures dimensions directly. A sample is placed into a sieve, which has a screen of precise mesh size. Particles that go through the screen are smaller than the size of the mesh. Particles retained on the mesh are coarser.
- Weigh the sediment retained on each sieve and get a distribution of sediment in two size fractions, 1) particles coarser than the sieve mesh size, 2) particles finer than the sieve mesh size.
- This method is appropriate for particles from fine sands to coarse gravels (at which point, it is easy enough to measure individual particle dimensions with a ruler).

Pipette Analysis is performed on the Mud fraction.

- Sieving is not practical because the viscosity of water does not allow particles and water to fall easily through the fine mesh required to measure. Also, when particles get below 2-4μm, the electrical charges on their surfaces cause them to flocculate, or aggregate they will not occur as individual particles. At even smaller diameters the effects of Brownian motion become important.
- Pipette technique is an indirect measurement. Size is calculated using the settling velocity of particles. Particles of uniform density settle at a velocity proportional to their diameter or grain size, so larger particles like sand will fall out of a mixed column of sediment more quickly than finer particles.
- Start with a sample that is completely uniform through the height of a 1000mL column.
- Draw off 25mL from a specific depth (settling distance), and a specific time (distance divided by time equals velocity). This 25mL is a subsample with mass equivalent to $1/40^{th}$ of the mass in 1000mL that is finer than diameter associated with that fall velocity. This is because all particles coarser than that diameter will have fallen past that depth, by that time.
- The first aliquot is withdrawn at almost initial time (time depends on room temperature) and deep enough (10cm) to represent the whole sample before settling. Therefore, multiplying the dry weight of sediment withdrawn initially (M) by 40 estimates the amount of sediment in the whole cylinder sample. This sample includes everything smaller than finest sand, i.e. mud, i.e. the mass of silt and clay.

- The second aliquot is withdrawn from a depth and time appropriate to 2 µm (or 4µm). Multiplying the dry weight of sediment in this subsample gives the mass of sediment finer than 2µm (or 4 µm). This is the mass of clay.

PIPETTE TECHNIQUE

- Prepare the cylinder (adapted from Folk 1980):
  - Stir the cylinder vigorously, starting at the bottom and working up until even the coarsest material is suspended uniformly through the column. As the stirring rod emerges from the cylinder for the last time, start the timer. Do not stir again.
- Prepare to withdraw samples:
  - Place the bulb on the pipette. Squeeze air from the bulb using the valve. At 15 seconds before time, insert the pipette into the cylinder with the tip at the appropriate depth. Hold the pipette and bulb steady with one hand. Rest your hand gently on the rim of the cylinder if you must steady it. Hold the valve that will allow sample into the pipette with the other hand, making sure you will be able to release the valve comfortably and smoothly.
- Withdraw samples:
  - At time insert pipette to a depth of 10cm and withdraw sample to approximately 2cm higher than the 25mL mark.
    Release the sample slowly to within 2mL of the 25mL mark.
    Remove the pipette from the cylinder, expel the solution into a weighed dish for drying.
    Suction 25mL of distilled water into the pipette, then expel it into the same dish to rinse the pipette.

PIPETTE SAFETY CONCERNS

- Pipetting by mouth was the standard for grain size analysis back in days of yore. In recent years it has lost favor in the research community for a variety of reasons. In the classroom, it is strictly forbidden – Do not pipette by mouth!
- Pipette use has been linked to laboratory accidents and injuries related to glass shattering, for example as the pipette is inserted into the pipette bulb. Therefore I strongly recommend using polypropylene for pipettes and polypropylene or polymethylpentene for graduated cylinders. Polymethylpentene is nice for cylinders because it is very clear and allows you to watch the suspension settle. If you have access to glass pipettes and do not anticipate doing this often, it helps to wrap the pipette in plastic wrap to keep the pipette together if it does break.

CHEMICALS THAT MAY BE USED IN SIZE ANALYSIS

- Hydrogen Peroxide: In this application, hydrogen peroxide (H_2O_2) is used to remove organic material from sediment. It helps to understand this by realizing that peroxide is just water (H_20) with an extra oxygen. The oxygen oxidizes the organic material – remember the equation for respiration ($C_6H_{12}O_6+6O_2 \rightarrow 6H_20+6CO_2$); The free oxygen ($O_2$) oxidizes the organic carbohydrate to produce water and carbon dioxide.
  - I did not recommend this step for very sandy samples for several reasons.
    - This requires a 30% solution of hydrogen peroxide, which does not store well (it must be sealed tightly, and kept at cool/stable temperatures in the dark). The peroxide you can buy at the drugstore is ~3%. If you decide you would like to remove organic material, you may order it from a chemical supply company, or check with your local salon supply store (this is the peroxide used to bleach hair).
    - Sands are not typically as rich in organic material as muds by virtue of their chemistry. Sand contains fewer clay minerals, and is frequently composed of silica (SiO_2). As a result, sand particles are usually roughly equidimensional (like a ball) and do not have a net chemical charge. Mud contains abundant clay minerals, hydrous aluminosilicates (for example, $Al_2Si_2O_5(OH)_4$). In addition to

causing the clay particles to be flat and flaky, the complex chemistry leads to a net negative charge, which can be neutralized by connecting to similar charges in organic molecules and other clay particles. The clay-organic aggregates have a different size distribution from the individual particles. If you plan to analyze mud samples, plan to remove organic material.

- If you use hydrogen peroxide, you may download the MSDS from this site: http://www.sciencelab.com/xMSDS-Hydrogen_Peroxide_30_-9924299.

- Sodium Hexametaphoshate, also known as SHMP or $Na(PO_3)_6$: In this application, SHMP is used to disperse sediment, or keep particles separate, so that the pipette analysis measures the size of individual grains and not that of multiple-particle aggregates. SHMP is equivalent to the laundry water softener Calgon before it was reformulated to eliminate phosphates, which occurred after phosphates in laundry detergents were linked to nutrient enrichment and algal blooms.
  - The charges on clay particles cause them to attract each other, and mud is famous for flocculating when it is suspended in a graduated cylinder for pipette analysis. SHMP is used to disperse clay particles and prevent flocculation (mostly), because the sodium (NA^+) binds into the clay mineral matrix.
  - SHMP is not considered as dangerous as some chemicals (strong acids, carcinogens, etc.), and is actually used as a food additive. However, it is a contact irritant, and there are guidelines for its transportation and storage. It must be used with care.
  - I have recently been using this method frequently with samples from Mississippi barrier islands and marshes, which contain more sand and less clay. I have found that for classroom demonstrations, SHMP is not necessary for these very sandy samples.
  - If you use SHMP, you may download the MSDS from this site: http://www.sciencelab.com/xMSDS-Sodium_hexametaphosphate-9924995

MISCELLANEOUS SAFETY AND BACKGROUND

- Drying ovens:
  - Drying oven settings do not always specify temperature, so be careful not to set the dial too high. Check the thermometer in the top of the oven to make sure the temp is <100°C before you use it.
  - I have heard of teachers using home ovens to dry sediment. Keep in mind that you might spill your sample. However, the primary concern I see with this is not knowing EXACTLY what is in your sample, particularly if it spills. Who knows what dog marked that as his territory? Or what neighbor disposed of motor oil? Or when was the last time public services sprayed herbicide? You would be taking a chance, although it might be very small. If you do not have access to a drying oven, an alternative to using your home oven might be to dry your sample next to a heater or under a warm light bulb (incandescent). If you decide to go with a home oven, it does not need to be too hot. Dry samples at 70-100°C (that is 160-212°F).
- Thermometer:
  - Temperature determines the rate at which particles settle; cold water is more viscous, so particles settle more slowly. Therefore you must record the temperature and use it to decide at which times pipette aliquots should be removed.
  - If you overheat the oven, the thermometer may break. Use a thermometer that does not contain mercury.

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MATERIALS
Listed in order of use in the procedure

Datasheets
3 heat-tolerant lightweight dishes (4 to include gravel in distribution)*
25mL volumetric pipette with bulb
Substitute 20mL volumetric pipette, or 25mL transfer pipette
2 1L bottles
Sodium Hexametaphosphate (SHMP)
Distilled Water
Balance, accurate to 0.1g
500 mL squeeze bottle (marked at exactly 500mL)
2mm mesh sieve
Sediment sample, ~50g, do not dry*
~500mL jar with tight fitting lid*
Spatula or spoon
62.5µm sieve, (#230 mesh)
Shallow dish (to catch sample below the sieve)
Plastic funnel
~2L plastic bottle (marked at 1L)*
1000mL graduated cylinder*
Thermometer
Clock with second indicator
Cylinder mixer (narrow paddle, meter stick; something to lift material from the bottom)
Drying oven

*Each sample will require its own set of these items

ANALYSIS
PREPARATION

1) Print datasheets – one for each sample you will analyze.
2) Label three dishes with Sample Number and designation of Sand, Mud or Clay (S, M, C, or 1, 2, 3). Weigh and record their masses.
3) Mark pipette at the following distances from the tip: 10cm, 20cm.
4) PREPARE SHMP (0.5%)
   a. First make a *STOCK* solution of 5% SHMP. Measure out 50g of SHMP and pour it into a 1L bottle (clearly marked at 1L level) that already contains 500ml of distilled water. Cap the bottle tightly and shake for one minute to aid dissolution of the powder. Add distilled water to bring the total volume up to 1L. Shake again and leave overnight.
   b. Pour 100ml of the 5% Stock into a 1L bottle and then fill the bottle up to the 1L line with distilled water.
   c. Fill your squeeze bottle with 0.5% SHMP. Keep track of how much you use at each step.
5) If you have a few gravel particles in your sample, pick them out. If you have more than you want to pick out, use a 2mm sieve to separate them. This will require you to separate the gravel into a 2mm sieve resting above the 62.5µm sieve, which adds a few more steps and requires one more weighing dish.
6) Weigh out ~50g of moist sediment.
7) Place sediment sample in 500mL jar.

REMOVE ORGANIC MATERIAL – optional, usually less important in sand than mud

1) Add about a teaspoon of 30% H_2O_2 to the sample.
2) Using a spatula, carefully break up the sample to allow the H_2O_2 to mix with it and react with and decompose any organics present. H_2O_2 reacts slowly, so allow it time to react. Once the reaction begins, the sample will foam.

3) Use the spatula to stir the sample and knock down bubbles to avoid a spill. If sample spills, clean up and start over. The remaining sediment may not have the same distribution as the original.
4) Keep adding H_2O_2 in small amounts until no more reaction occurs. Leave sample uncovered overnight to ensure that the reaction is complete.

DISPERSE AGGREGATES

1) Weigh out 2.5g of SHMP and add it to the sample jar.
2) Fill the jar with distilled water up to the 500mL line.
3) Tighten the lid on the jar and shake it vigorously.
4) Leave the sample for several hours (preferably overnight) for SHMP to dissolve completely.

SIEVE SEPARATION

1) Shake the sample jar for 2 minutes.
2) Place the 62.5μm sieve into the shallow dish.
3) Pour in the sample, shaking the sieve gently to allow fine particles to flow into the dish below.
4) Use the 0.5% SHMP squeeze bottle to wash remaining finer particles through the sieve, leaving only the sand.
5) Periodically use the funnel to empty the bottom dish into a 2L plastic bottle.
6) Rinse the sand remaining in the sieve with distilled water to remove remaining SHMP.
7) Wash this sand into the pre-weighed dish labeled Sand.
8) Place Sand dish in the oven.
9) Move remaining sample from the dish to the 2L bottle. Rinse pan with SHMP.

PIPETTE SEPARATION

1) Record the temperature of the room (to 0.5°C) and enter it on the data sheet.
2) Refer to Withdrawal Time Table to find appropriate times and depths of withdrawal for Mud and Clay. Enter these on the data sheet.
3) At 5 minutes before start time, shake the first sample vigorously and carefully pour it into a 1L cylinder until the cylinder contains exactly 1L of sample.
   a. If there is <1L, add 0.5% SHMP to make 1L of solution.
   b. If there is >1L, pour the excess into another cylinder to measure the total volume. Record the actual total volume on the pipette analysis spreadsheet and use that volume through all calculations. Dump the excess after recording the volume.
4) Mix the sample well and continue mixing to keep all sediment in suspension.
5) At start time, remove the stirrer.
6) Use the pipette to withdraw fluid from the appropriate cylinder depth at time for mud aliquot.
7) Expel fluid in the Mud dish. Rinse residues from the pipette into the Mud dish with distilled water. Place the Mud dish into the oven.
8) Use the pipette to withdraw fluid from the appropriate cylinder depth at time for clay aliquot.
9) Expel fluid in the Clay dish. Rinse residues from the pipette into the Clay dish with distilled water. Place the Clay dish into the oven.
10) When all pipetting is complete, dispose of the remaining fluid and clean the cylinders.

WEIGHING

1) Dry in an oven for at least 24 hours @~100°C. Remove all dishes from the oven at the same time.
2) Allow dishes to equilibrate with atmospheric humidity (this will cause rapid weight gain initially) for several hours.
3) Weigh subsample + beaker to 0.1g. Enter data onto data sheet.

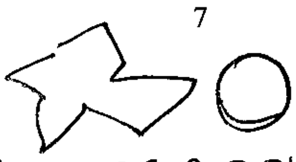
Withdrawal Time Table

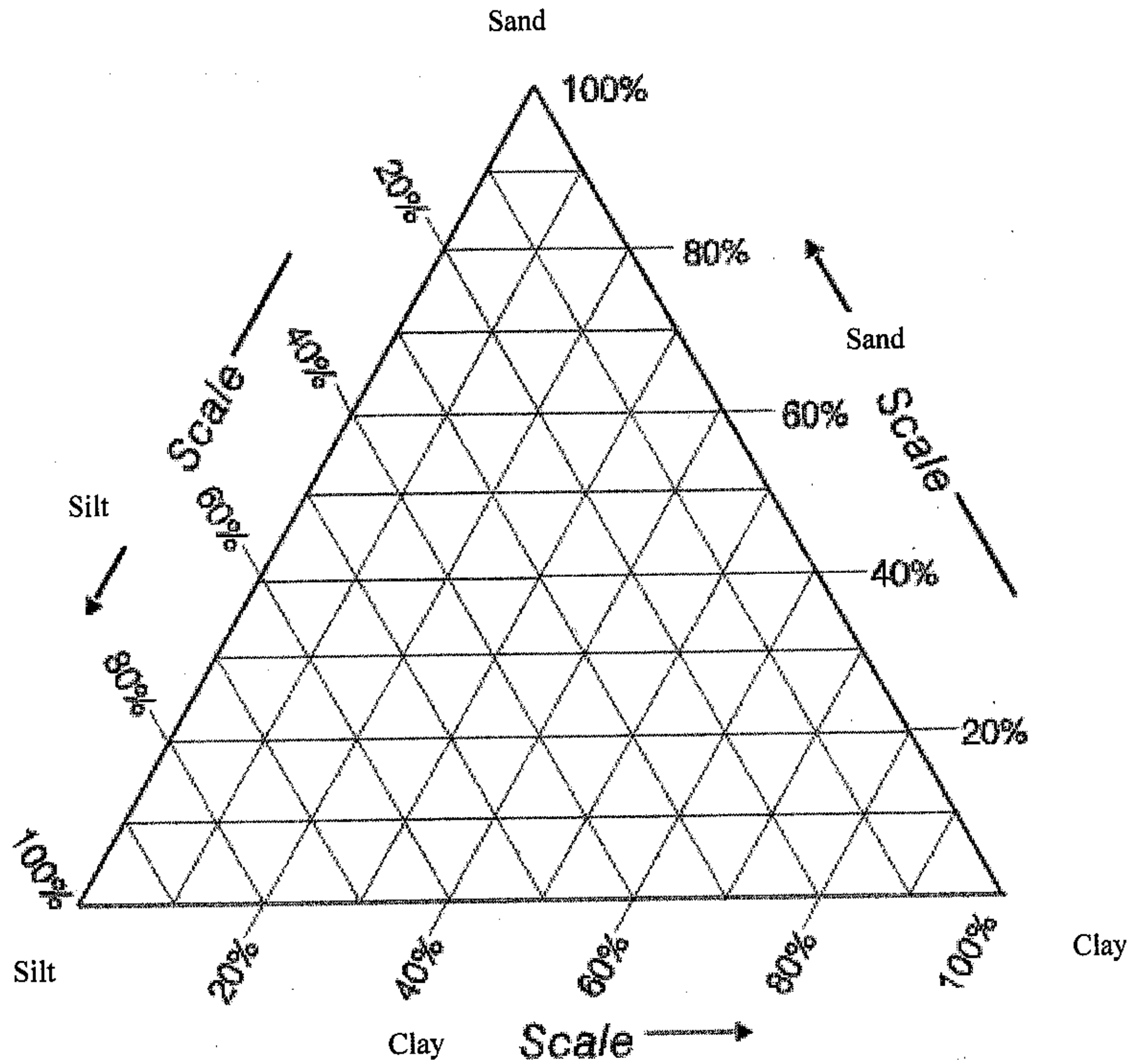
Note: Times are provided time for both accepted size cutoffs between silt and clay, 2μm and 4μm. Use what works best for your class time limits. Be consistent to avoid confusing your students.

Temperature	20°C	68°F	
Diameter (μm)	Name	Withdrawal Depth cm	Withdrawal Time h=hour m=minute s=second
62.5	Silt	10	29s
4	Clay1	5	61m, 19s
2	Clay2	5	4h, 5m
Temperature	24°C	75°F	
Diameter (μm)	Name	Withdrawal Depth	Withdrawal Time
62.5	Silt	10	26s
4	Clay1	5	55m, 41s
2	Clay2	5	3h 43m
Temperature	30°C	86°F	
Diameter (μm)	Name	Withdrawal Depth	Withdrawal Time
62.5	Silt	10	23s
4	Clay1	5	48m, 42s
2	Clay2	5	3h, 5m

Terminology
Composition

Quartz (Qtz)
Feldspar
Rock fragments → Volcanic, Sedimentary, Metamorphic

Texture
Size
roundness 
well sorted poorly sorted
Sorting → homogenous vs hetrogeneous



Ternary Diagram – print and use and please maintain reference to this website and author:
Lynn S. Fichter, James Madison University, Department of Environmental Sciences
http://csmres.jmu.edu/geollab/fichter/SedRx/readternary.html