Soil Texture analysis: Hydrometer Method with Carbonate removal and Sieving

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Introduction

Soil texture, or particle-size distribution (PSD), affects cation exchange capacity, soil fertility, soil structure, organic matter storage, water infiltration rates and water holding capacity, susceptibility to erosion, and many other soil characteristics of interest to ecologists, geologists, agronomists, environmental scientists, and engineers. Furthermore, soil texture is very difficult to change or improve with active management.

The US Department of Agriculture (USDA) uses the following categories for soil particle sizes:

Sand: 2.0 mm to 0.05 mm
Silt: 0.05 mm to 0.002 mm
Clay: less than 0.002 mm

There are three commonly used methods for determining soil texture: 1) sieving, which works for sand subcategories only, 2) the pipette method, which can be more accurate but is not often used, and 3) the hydrometer method, which is simple, can be accurate ***if*** done properly, and the most universally used method. This protocol has been optimized for carbonate rich, minimally sandy soils and combines the hydrometer method with sieving to accurately measure the sand fraction.

Theory:

The pipette and hydrometer methods take advantage of the fact that different sized soil particles have different sedimentation rates. Stokes' law describes the relationship between the velocity and size of a soil particle settling in a liquid medium:

$$v = (g/18\eta)*(\rho_s - \rho_l)d^2$$

Where: v is the velocity of the particle (cm/s)
g is the acceleration of gravity (cm/s^2)
η is the viscosity of the settling medium (poise) (g/cms)
ρ_s is the density of the solid particle (2.65 g/cm^3)
ρ_l is the density of the settling medium (g/cm^3)
d is the effective diameter of the soil particle (assumed to by spherical) (cm)

In general, this equation tells you that the settling velocity is proportional to the square of particle diameters (we assume soil particles are spheres -- is this a good assumption?) so bigger particles settle much more quickly than smaller particles. As particles settle out of solution, the density of that solution changes predictably. We use the change in solution density with settling soil particles to estimate the percent sand, silt, and clay in a soil. Given the size of sand particles, we know that 40 seconds after soil has been evenly suspended in deionized water, all of the sand particles should have settled out of the solution. A density measurement at this time (40 sec) using a hydrometer tells us the mass of silt plus clay particles in the cylinder. After 7 hours, we know that all of the silt particles should have settled out of solution. A density measurement at this time tells us the mass of clay suspended in solution. Thus, by taking density readings (hydrometer) at 40 seconds and ≥7 hours, we can calculate the grams of silt + clay and the grams of clay in the soil, respectively. This method assumes that soils are evenly dispersed throughout the cylinder and no aggregation of clays is present. Therefore a dispersal agent is always used to destroy aggregates and disperse the soil. In most cases the dispersal agent is sodium hexametaphosphate because sodium is a deflocculating cation due to its large hydration sphere (alternately, calcium promotes aggregation and is a flocculating cation).

Additional Information:

The method described above is the simplest and most common procedure for the hydrometer analysis of soil texture. However, some methods use times of 2 or 24 hours (2 hours has been shown to be largely inaccurate and only appropriate for classroom demonstrations). Hydrometer readings between 7 and 24 hours are not statistically different although consistency is always recommended. It is also possible to take many hydrometer readings and plot changes in density to figure out the exact time at which the suspension contains only silt + clay or only clay for a given soil.

There are 2 factors that affect soil density that must be tested for and corrected prior to texture analysis. The first is high concentrations of organic matter (≥5%), which can be oxidized and removed with hydrogen peroxide (H_2O_2), see *Appendix A*. The second and most common in dryland environments is high concentrations of carbonates (≥1%), which are removed using sodium acetate adjusted to a pH of 5.0.

The method of dispersal can also vary. While everyone uses sodium hexametaphosphate to disperse soils, some shake overnight, while others use a blender to disperse soil. In addition, depending on the fraction of silt and clay, as well as salts in the soils the amount of dispersal agent can vary in concentration.

Finally, the accuracy of the hydrometer method described here can be improved by sieving the soil for sand content (using a 53 µm sieve, No. 270) after the last hydrometer reading has been taken. Then a 7 or 24 hour hydrometer reading can be used for clay content, the mass of soil on a 53 µm sieve is used for the sand content, and silt is determined by difference. In this case a 40 second hydrometer reading would be unnecessary.

Equipment needed

*An example spreadsheet entitled Soil Texture Data Sheet is available with all columns matched to the protocol.

Materials for Texture Analysis Only:

250 mL Nalgene bottles
Large aluminum soil tins (VWR 25433-085)
Shaker table
Squirt bottle
Solution of sodium hexametaphosphate (50g/L)
Mixing rod (rubber stoppers are an alternative)
Sedimentation cylinders (1 L)
Stopwatch
Hydrometer, ASTM 152H with scale in g/L
Coarse balance (0.01 g)
Thermometer
53 micron Sieve

Carbonate removal Materials:
1000 mL Pyrex Erlenmeyer flasks
250 mL bottles (must be autoclavable, regular bottles will melt and/or lose weight!!!)
    **Alternatively you may try 250 mL Erlenmeyer flasks
Thin metal spatula
Aluminum foil
Vacuum manifold (VWR 28198-910) or equivalent
Vacuum pump, tubing, and moisture traps
Plastic Buchner funnels, 110 mm (VWR 30305-109)
Glass fiber filters, GF/F (0.7um), 110 mm (VWR 28497-973)
Black rubber or silicone stoppers (No. 8) with large hole for funnel stems
Solution of 1.0 M sodium acetate (pH 5.0, using acetic acid)
Balance to 3 digits (grams)

Additional Materials for Organic Matter Removal:
Solution of drugstore grade (3%) hydrogen peroxide
Heat plate or hot water bath
Fume hood

Procedure

Carbonate Extraction Procedure

Note: If your soils are coarse textured, it is possible to skip the following filtration procedure. If this is the case, ignore filter funnel and filters in step 5 and steps 9-11. If there is still suspended sediment after 12 hour settling period, continue with the entire filtration procedure.

Basic carbonate removal:

1. Record the weight for the 250 mL bottle or Erlenmeyer flask in the ***Bottle Weight*** column and tare the container. Take 40 ± 0.5 grams of oven dry soil (soil should be oven dried at 105°C for 24 hours) and place in bottle or flask making sure to record the weight in the ***Weight of soil*** column. Add 100 mL of DI water and 10 mL of 1.0 M sodium acetate (pH 5.0, using acetic acid). Shake vigorously for 1 minute and then place on shaker table for 1 hr at 180 rpm.
    - Note: while on the shaker table, the ***caps should be loosened*** to prevent the bottles from exploding due to the evolved CO_2.
2. Allow samples to settle overnight (~12 hr) in oven at 60°C so that supernatant is free of suspended sediment. The heat increases the dissolution and evolution of the carbonates and CO_2 respectively. ***Again, make sure to unscrew caps slightly so the CO_2 can escape.***

Supernatant vacuum procedure (for clear supernatant):

3. Open 250 mL Nalgene sample bottle and place next to the **sample trap**. Place bottle cap or other item underneath bottle on one end so the bottle is sitting at an angle on the bench top. Turn on vacuum pump and using the vacuum line, slowly remove the supernatant from the bottle without disturbing the settled material (you should be able to remove ~100 mL of solution).
    - Note: only insert tip of vacuum line 1-2 mm into supernatant at moderate vacuum to prevent disturbing the settled material.
4. Fill a 100 mL beaker with DI water and vacuum rinse the line with water to remove any residual soil or organic matter from the line.

5. Us a 10% HCl solution to drop test the soil in the bottle to look for strong effervescence. If excess CO_2 is evolved, a second sodium acetate treatment is required.

   Note: If filtering supernatant, continue with steps 6-8. Otherwise, continue to Rinsing Steps.

Filtration procedure (for cloudy supernatant):

6. Setup the vacuum apparatus using the following items:
   - Bench-top vacuum pump with 1L side-arm E-flask attached as the **"water trap"**
   - One 500 mL side-arm E-flask with vacuum line for supernatant (will attach to water trap), this is the **"sample trap"**
   - One 47mm **filter funnel** plus clamp (set to side for filtering supernatant later)
   - Pack of 47mm filter papers with <0.47 micron pore size (e.g. Whatman GF/F or GF/B, Pall 66078). ***Record the dry filter weight*** before placing it on the funnel base. Make sure filter paper is centered inside the funnel and remain mostly dry prior to sample filtration.
   - You will also need Aluminum foil, tweezers, one beaker filled with DI water (200-500 mL), a squirt bottle with DI-water, a Sharpie, and paper towels.
7. Remove the vacuum/stopper from the **sample trap** and disconnect from vacuum. Set **sample trap** to the side. Take the **filter funnel** and flask and attach to vacuum line. Pour supernatant into **filter funnel.** Rinse **sample trap** with minimal DI water and pour into **filter funnel** to remove any residual soil or organic material. Use squirt bottle sparingly to rinse soil and organic material down off funnel sides onto the filter. Allow sample to filter.
8. Turn off vacuum pump and remove funnel top. Make sure to run finger along funnel base to check for residual material to add to filter by hand. Using the tweezers, remove the filter and place on Aluminum foil next to corresponding sample ID#.
9. Dry filter paper in oven at 105°C 24 hours. The difference in filter weight should be subtracted from the total soil added for texture analysis -- this will be your new total soil weight.
10. Rinse sample trap and filter funnel with DI water. Dry filter funnel and load new filter onto funnel. Reattach sample trap onto vacuum line and repeat process for each sample.

Rinsing procedure:

11. After removing the sodium acetate supernatant, add 100 mL of DI water to each sample to rinse the soils. Shake vigorously for 1 minute and then place on shaker table for 1 hr at 180 rpm.
12. Allow samples to settle overnight (~12 hr) in oven so that the supernatant is free of suspended sediment (won't ever be entirely clear). There may be a waxy film on the surface of the water that can be discarded with the supernatant.

    Note: Again, if you observe suspended sediment, follow the filtration procedure outlined above (steps 6-9). Otherwise, continue to step 13.

13. Repeat steps 3-4 to remove supernatant.
    - Note: After 1 DI water rinse cycle, there is still a residual amount of sodium acetate in each sample (~1.12 g/40 g soil). This should not interfere with the

hydrometer readings during texture analysis but you can perform a second DI rinse if desired (~0.1 g remaining).

14. Assuming you were more or less consistent about the amount of water removed from the bottles, you can proceed to the standard hydrometer texture procedure below.

Soil Texture Procedure:

*If the previous steps for carbonate removal were done start with Step 2.

1. Take 40 ± 0.5 grams of oven dry soil (soil should be oven dried at 105°C for 24 hrs) and place in 250 mL Nalgene bottle.
2. Add 100 mL of sodium hexametaphosphate solution (50g/L). Shake overnight (12 hours).
3. Number the sedimentation cylinders (1 L graduated cylinders) according to sample number # and include a blank. Remove the samples from the shaker and make sure each lid is tightened securely. One at a time, shake each 250 mL Nalgene bottle vigorously until soil particles are suspended and then quickly and quantitatively transfer the sample into a cylinder. Rinse all the remaining soil particles into the cylinder using a squirt bottle filled with DI water. Fill the cylinder to the 1000 mL mark with DI water. Transfer **ALL** of the soil particles!!
    - Note: DI water needs to be at room temperature for all the cylinders or the density of water will change during the measurements and between samples – a correctable but annoying complexity (see calculations section). Typically a 20-50 L carboy is filled the night before with DI water and allowed to adjust to room temperature for use the following day.
4. Thoroughly mix the contents of the settling cylinder using the mixing rod. Mixing time should be consistent between samples; standard time is 10 to 15 seconds and involves several short up and down strokes at the bottom of the cylinder to suspend the sand followed by several strokes the entire length of the cylinder for even distribution. Time starts immediately after removing the mixing rod and must be precise!
5. After the soil suspensions are settling, prepare a blank column. In a sedimentation cylinder add 100 mL of sodium hexametaphosphate and fill to the 1000 mL mark with DI water.
6. Take temperature and hydrometer readings at 7 hours of all cylinders (**DO NOT REMIX CYLINDERS!!**). If the clay content of your soil is >50 see Elliot et al., 1999 for procedure.
    - Place thermometer into cylinder and let sit for 40 seconds. Record temperature under ***7 hour Temp***. Remove thermometer from the cylinder, rinse, and move the thermometer to next cylinder. Be careful to not disturb the solution when inserting or removing the thermometer.
    - Place hydrometer in the cylinder. The hydrometer should not be bobbing when measurement is recorded. Record hydrometer reading under ***7 hour hydrometer***. If some residual foam is still present and obscures the hydrometer a few drops of amyl alcohol will disperse it.

Sieving Sand Fraction Procedure:

This procedure follows the 7 hour hydrometer reading as described above. Once the 7 hour temperature and hydrometer measurements have been recorded, the soils are ready to be sieved for the sand fraction using a 53 µm sieve.

1. Prepare a large aluminum tin and record ***Tin ID*** and ***Tin Weight***.

2. Set the 53 µm sieve in the sink on a 2 gal white bucket or sweater box. Slowly pour the water and sediment from the cylinders into the sieve being careful **not to splash** to avoid losing material**.** Use tap or DI water from the sink (with hose) to gently spray out the remaining sediment from the cylinder to the sieve. **Make sure all sediment and organic particles are removed from the cylinder before moving forward.**
    - Note: The purpose of a small bucket or sweater box placed upside down in the sink is to 1) raise the sieve closer to the faucet to reduce the potential for splashing, and 2) if the container is white, it is much easier and quantitative to determine when all the clay and silt has passed through the sieve.
3. Massage the sediment through the sieve until clay and silt are removed. For a loamy soil this often takes 5-10 minutes. You will know you are done when sediment no longer feels "slimy" (due to the silt) and water coming from the sieve is no longer murky and leaves little or no particulate on the bucket.
4. Use DI water from the sink (with hose) to consolidate all sediment and organic particles into one sector of the sieve. Use a squirt bottle with DI water to further consolidate particles into one sector. **Be careful not to splash sediment out of the sieve during this step.**
5. Tilt the sieve on its lip above the large aluminum tin and scoop particles into the tin with one finger. Spray the particles from your finger back into the sieve. Reconsolidate the few remaining particles into a small area with the bottle again.
6. Again, tilt sieve on its lip above the aluminum tin. Use the squirt bottle with DI water to spray the remaining particle into the tin. This step *must be done assertively* to get the remaining particles into the tin without overflowing the tin with rinse water.
7. Do not move forward until all particles are removed from sieve.
    - Note: Make sure to check the rim on the sieve.
    - It is often helpful to do a final reconsolidation to make sure no small grains have stuck to the sieve elsewhere.
8. Place tins in the oven at 105°C for 24 hours to remove moisture. Record the weight of the tin and oven dry sand in ***Tin Wt. + OD Sand Wt.*** column.

Soil Organic Particle Correction to Total Soil Weight:

The muffle furnace is used to bake the sieve sand samples to remove remaining organic material that passed through the 2 mm sieve. In this way we get a final sand only weight.

9. Turn on the muffle furnace and set the temperature to 450°C. Also, at ISU turn the furnace hood on low to vent any smoke from the samples. Load samples while the furnace begins to warm.
10. Bake the samples in the muffle furnace for 8 hours. Note: the furnace does not turn off automatically so plan to start and stop the furnace accordingly.
11. When finished, turn off the furnace. Leave the hood on and crack the furnace door for one hour to cool. CAREFUL NOT TO PLACE FACE OR HANDS NEAR OPENING!!!
12. Remove samples from the furnace and place in 105°C for 24 hours. Remove samples and place in desiccator for 15 minute to all samples to cool (make sure desiccant is fresh). Weigh samples in batches of 10 so they don't sit out for prolonged periods of time and absorb water from the atmosphere. Record weight under ***Tin Wt. + Ashed Wt.*** Set samples aside; do not throw away until data has been entered and QCed.

Calculations

Because sodium hexametaphosphate changes the viscosity of water the blank is measured and the hydrometer reading subtracted from all the sample measurements.

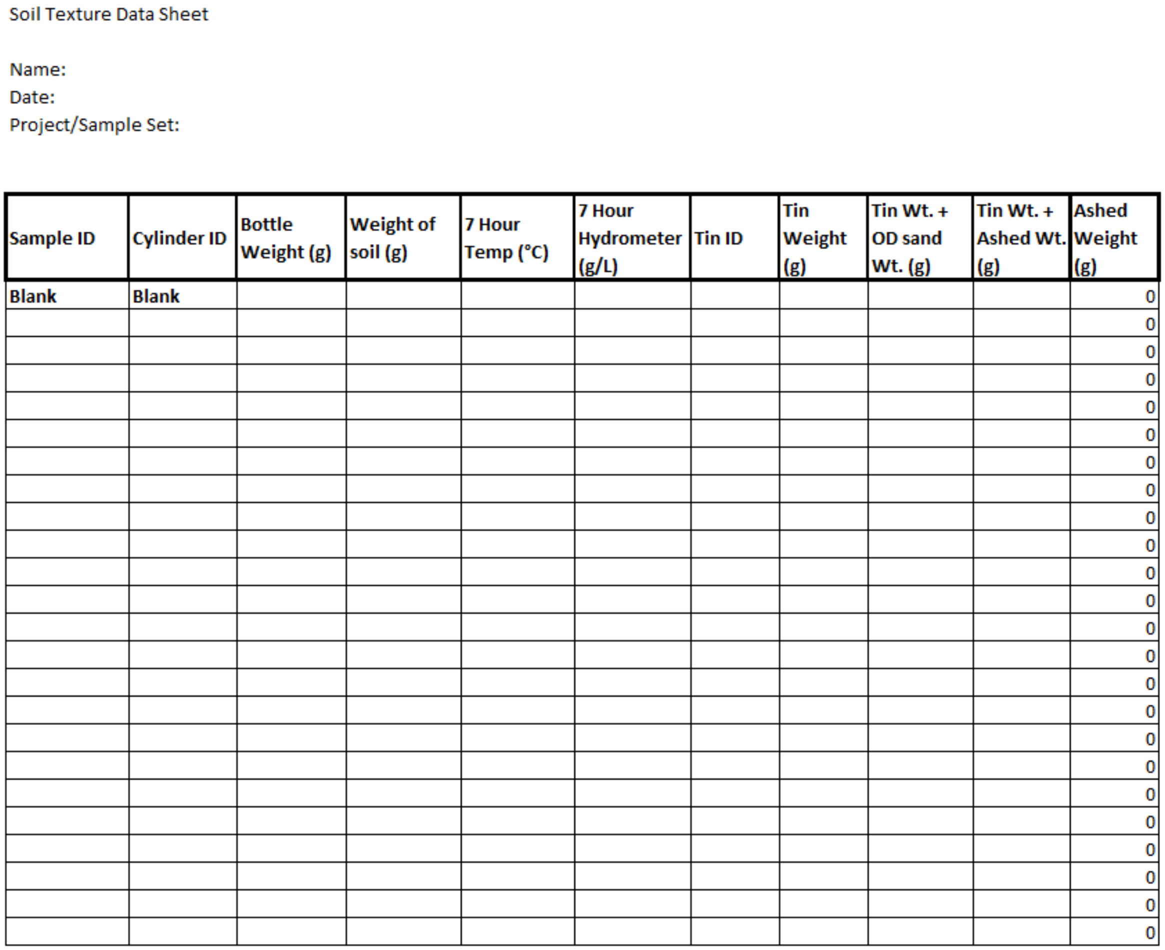
In addition, all readings must be corrected for deviations from the temperature at which the hydrometer is calibrated. As the temperature of the liquid increases, the soil particles fall faster than if the liquid were cooler; the hydrometer is calibrated at 20°C so you should add 0.4 g/L for each degree above 20°C or subtract 0.4 g/L for each degree below 20°C.

The blank-corrected 7 hour reading tells you the grams of clay per liter of suspension. Sand is calculated by mass sieved. Thus using the original dry weight of soil added to the Nalgene bottles, one can calculate the percent sand, silt, and clay of the sample.

Appendix A: Soil organic matter removal protocol

1. Take 40 ± 0.5 grams of oven dry soil (soil should be oven dried at 105°C for 24 hours) and place in 1000 mL Pyrex Erlenmeyer flask. Add 25 mL of DI water and 5 mL of drugstore grade (3%) hydrogen peroxide. Swirl gently for 1 minute and let sit until frothing ceases. Add another 5 mL and repeat until frothing abates. In the fume hood, heat sample to 90°C swirling occasionally until soil organic matter breaks down and the soil becomes pale colored (Gee and Bauder, 2002). The time required for this can be hours to days depending on the type and amount of organic material.
2. Setup the filtration apparatus as described in the carbonate extraction protocol of Appendix A above. After 1 DI rinse there will be 0.27% residual H_2O_2 or 0.025% after 2 DI rinses; this again will have no significant impact on the hydrometer readings.

Appendix B: Soil Texture Data Sheet



Soil Texture Data Sheet

Name:
Date:
Project/Sample Set:

Sample ID	**Cylinder ID**	**Bottle Weight (g)**	**Weight of soil (g)**	**7 Hour Temp (°C)**	**7 Hour Hydrometer (g/L)**	**Tin ID**	**Tin Weight (g)**	**Tin Wt. + OD sand Wt. (g)**	**Tin Wt. + Ashed Wt. (g)**	**Ashed Weight (g)**
Blank	**Blank**									0
										0
										0
										0
										0
										0
										0
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