Aridlands Ecology Lab Protocol
Modified: 2010.05.09, M.Ross

Soil Particle Size: Beaker Method

Carbonates and Organic Matter are removed before texture analysis

Sources: Kettler, T.A., Doran, J.W., Gilbert, T.L. *Simplified method for soil particle-size determination to accompany soil-quality analyses*. Soil Science Society of America Journal. 65:849-852 (2001).

Introduction: Texture analysis with many samples can be time consuming and expensive. Kettler and others (2001) developed a rapid soil texture analysis method to make the process faster and cheaper. This method is an adaptation of Kettler's method with the removal of carbonates and organics as added steps. The method involves weighing ~13g of sample into 50 ml centrifuge tubes and removing carbonates and organics, shaking samples in a Na-HMP flocculating solution, pouring the solution through a sieve to isolate the sand particles, and then pouring off the clay particles after the silts have settled out.

Materials:
50mL centrifuge tubes
3 % by weight solution of Sodium Hexametaphosphate (HMP)
Bleach adjusted to 9.5 pH
Sodium Acetate solution adjusted to 5.0 pH
400mL Beakers
Aluminum weighing tins
Orbital Shaker
Hot Plate
Pyrex Bake Dish or other container for hot water bath
Oven
53 micron (#270) sieve
Squirt bottle with House DI water
Saturated NaCl solution

Solution Recipes:
1M Sodium Acetate: add 136.08g NaOAc per liter of DI water. Adjust to pH 5 with acetic acid or HCl solution. With concentrated Acetic acid it should take ~35mL to adjust the solution. For each sample you should make ~30mL of solution.
Bleach: Household Clorox bleach. Adjust to pH 9.5 with small amount of concentrated HCl. Start with 5mL and increase incrementally. For each sample you should account for using 30-60mL of bleach depending on the organic matter content. *For the Utah PJ soils you will used 60mL.*

HMP solution 3% by weight: 30g Na-HMP for every 1L of solution that you are making. Weigh out Na-HMP into a beaker and fill to the 1L mark. Make this in a big jug with a lid that will screw on tight. Shake the solution until Na-HMP is dissolved. Account for a 3:1

sample:HMP ratio, so ~13g sample will need ~39mL solution.

Sample Prep:
1. Texture samples can be collected with 5cm PVC core and can be used to determine bulk density in addition to soil texture. Analyze sample for texture after the bulk density protocol is complete.
2. If the bulk density procedure has not been done prior to texture analysis, then weigh entire sample. First tare the aluminum weigh tin, add sample and record entire sample weight.
3. Put sample through 2mm x 2mm sieve. Record the weight of the >2mm fraction and then discard rocks.
4. Using the splitter, partition out ~12-13 grams of sieved soil into a labeled and weighed 50mL centrifuge tube. Record tube weight and tube+sample weight to the nearest 0.01 gram.

Carbonate Removal:
1. Make a 1M Sodium Acetate solution and adjust the pH to 5.0 by adding concentrated HCl. (See pH meter instructions).
2. Fill the centrifuge tube up to the 35 mL mark with the adjusted Sodium Acetate solution.
3. Close the tube and shake the sample so that all soil gets dispersed in the solution.
4. Partially unscrew the lids to avoid pressure buildup and allow the sample to react for 30 minutes.
5. Place the samples in a hot water bath for 30 minutes.
6. Hand-shake samples again.
7. Allow samples to cool for 30 minutes.
8. Centrifuge samples in the Medeiros Lab for ten minutes.
9. Decant the Sodium Acetate into an appropriate waste container.
10. Fill the centrifuge tube up to 35 mL with house DI water.
11. Hand-shake the samples
12. Centrifuge for ten minutes and decant the water solution into the sink.
When decanting solutions it is important that soil is not lost from tubes

Organics Removal:
1. After the carbonates have been removed, fill the centrifuge tubes up to 35 mL with pH 9.5 Bleach (see pH meter instructions).
2. Hand-shake the sample so that all the soil is completely dispersed in bleach. Be careful! If samples are high in OM, there is a chance that samples will get foamy and can bubble over when shaken.
3. Allow the sample to react for 30 minutes with lids gently loosened.
4. Again, place the samples in a hot water bath for 30 minutes.
5. Hand-shake the samples periodically to promote oxidation of organic matter.
6. Allow samples to react overnight with loose lids over each sample.
7. After overnight reaction, centrifuge the samples for ten minutes and pour off the bleach into a bleach waste container.
8. If there are still organics in the soil or the soil is still dark in color repeat steps 1-7.
Most samples will need to be reacted two times with bleach

9. If the soil is lightly colored with no organics fill the centrifuge tubes up to 35 mL with house (gray faucet) DI water.
10. Hand-shake the samples.
11. Centrifuge for ten minutes. If solution is murky or has particles floating in it you will need to add saturated NaCl solution to the rinse water. This will remove fine particle from solution. It is really important that these don't get dumped down the drain.
12. Start by adding 10mL of solution to each sample. Gently mix with the rinse water and centrifuge. If solutions are still murky you can add a pinch of salt.
13. Repeat the rinsing step two times.
14. Once soils have been rinsed, decant the rinse water and place centrifuge tubes without lids in the oven with temp set at 105°F and let samples dry overnight. DO NOT LEAVE THE SAMPLES IN THE OVEN FOR MORE THAN 24 HOURS THIS WILL CAUSE TUBES TO BECOME BRITTLE.

Texture analysis:

Deflocculating Sample:

1. Place oven dried sample+tube on scale and record the sample weight (without lid). You already have the tube weight recorded, so you can figure out the sample weight by subtracting the prerecorded tube weight.
2. Shake 3% HMP bottle well to displace any crystallization. Add 3:1 HMP:soil (e.g. 39mL of HMP for 13g of sample).
3. Cap centrifuge tubes hand-SHAKE WELL.
4. Place sample on orbital shaker for 3 hours. Samples tend to shake better if they are horizontally positioned.

Wet Sieving Sample:

1. Record masses of a beaker and aluminum weigh tin for each sample. This is process is easiest with 400 ml beakers.
2. Place 53 micron sieve on top of the silt beaker.
3. Shake up centrifuge tube by hand to make sure none of the sample is stuck to the bottom.
4. Remove centrifuge tube cap and pour solution and sample through sieve and into beaker.
5. Use small squirt bottle to rinse any sample remaining in tube and/or on cap into the sieve (the large squirt bottle has too much pressure and will cause the sample to shoot out of the centrifuge tube too fast).
6. Once all sample is in sieve gently squirt water to wash any silts and clays through the sieve. If sieve is not draining, VERY GENTLY rub your finger around on the screen of the sieve to help all liquid to pass through into the silt beaker. Make sure that all sample is accounted for in sieve and beaker.
7. When water passing through sieve is clear – this usually occurs when the 400mL silt beaker has about 300mL of water in it – then sample washing is complete.
8. Remove sieve (which only has the sand fraction in it now), and wash all of sand from sieve into aluminum weigh boat. Squirt the sides of the sieve so that all the sand goes into the tin. BE CAREFUL to not lose sample.
9. Place the aluminum tin in the oven at 105°C overnight or until dry.

10. Stir the contents of the 400 mL silt beaker to suspend all particles.
11. Let stand for ~5 hours (more than 3 hours but less than 6 hours) – this will allow the silts to settle, while the clays will remain suspended.
12. After carefully decanting the water and suspended clays from the 400mL silt beaker, place beaker in oven 105°C to dry for 24 hours.
13. When dry, record mass of aluminum tin+sample and beaker+sample. Always let samples cool prior to recording the weight.

Calculations:

Clay (g) = Total Mineral Soil Mass (~13g) – Sand (g) – Silt (g)

$$\text{Percent Sand (or Silt/Clay)} = \frac{\text{Sand (or Silt or Clay) (g)}}{\text{Total Mineral Soil (g)}} \times 100$$

$$\text{To calculate the rock percentage} = \frac{\text{Rock (g)}}{\text{Total Unsieved Soil Mass}} \times 100$$