

Hydrometer Method for Soils with <5% Organic Matter

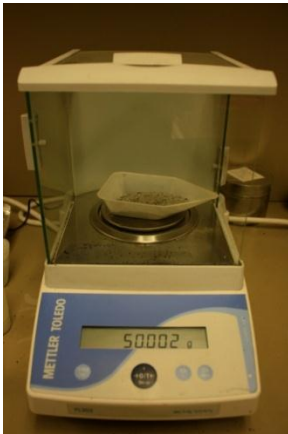
This method should be used to calculate particle size distribution for samples with <5% Organic Matter. When testing a single sample, the process should take about 2.5h. If you are analyzing more than one sample, multiple hydrometer tests can be run simultaneously in order to save time. Adding a second sample will increase the timeframe by approximately 5-10minutes and each pair of samples after that will take an additional 20-30minutes. Eventually you can run as many tests as you feel comfortable with, but it isn't recommended that you run more than 4 at a time right away.

You Will Need:

- Hydrometer
- Sedimentation Cylinder (1L graduated cylinder)
- Plunger
- Milkshake Mixer and Dispersion Cup (metal milkshake cup)
- Stopwatch
- Thermometer
- Weigh Scale
- Weigh boat
- Two Beakers, one at least 500mL
- Calgon Dispersing Agent
- Spoons/Stir sticks (for transferring solids and stirring)
- 2mm Sieve
- Amyl Acid
- pipette

Procedure

1. Separate out all coarse fragments of your soil sample using a 2mm sieve.



2. Weigh out 50g of your fine-textured material (+/-0.02g).



3. Add the 50g sample to a 500mL beaker, and then add 250 mL of distilled water.



4. Add 5g (+/- 0.02g) of Calgon dispersing agent to a *separate* beaker, then add distilled water to 50mL and stir until the calgon is dissolved. (If you are doing particle-size analysis for multiple samples, you may want to mix up the calgon solution for all of the samples simultaneously. If so, the solution should have a concentration of 100g Calgon/L solution).



5. Add the 50mL of calgon solution and the soil solution to a dispersion cup (metal milkshake cup). If some of the sample remains in the beaker, which it often does, pour some water from the dispersion cup into the beaker, swirl it around, and quickly dump it back into the dispersion cup. Continue doing this until the entire sample is in the dispersion cup.



6. Place the dispersion cup in the milkshake mixer and stir for 15 minutes. Make sure the cup is secure before turning on the machine. (Note: the green milkshake mixer will turn on immediately upon securing the cup into place, and plugging it in. Make sure the dispersion cup is in place before plugging in the machine.)



7. Transfer the soil suspension to a sedimentation cylinder and add water to the 1L mark. (Getting the sand out of the dispersion cup can be difficult. After the initial pour into the cylinder, continue adding water in small amounts to the dispersion cup, swirling it around and dumping it into the cylinder. Do this until no sediment remains in the dispersion cup. Use a funnel if necessary). A drop of amyl alcohol should clear up any foam on the liquid surface. See Appendix A for amyl alcohol methodology.
8. Take a temperature reading of the water – it should be approximately room temperature. If not, cover the cylinder and allow the temperature to equilibrate overnight.



9. Use a plunger to stir the suspension for 2 minutes, moving it up and down through the cylinder (25-30 plunges).



10. Remove the plunger and, quickly but gently, lower the hydrometer into the suspension. This should occur simultaneously with step 11. but be careful.
11. Start your stopwatch immediately upon removal of the plunger from the cylinder. Take a reading of the hydrometer at 40 seconds, and again after 2 hours. If measuring multiple samples, rinse the hydrometer between readings of the different samples.
12. Take another temperature reading at the end of 2 hours.

Making Calculations:

1. Adjust all hydrometer values by subtracting the blank hydrometer reading from both the 40s and 2h readings. See Appendix B for blank hydrometer method. Make this adjustment before moving on to step 2.
2. Adjusting hydrometer values: For each degree your sample is below the temperature at which you took your blank reading, subtract 0.36 from your hydrometer reading. For every degree above that temperature, add 0.36 to your hydrometer reading. This applies to both the 40s and 2h readings.
3. Calculation for Silt + Clay %: Divide corrected 40s hydrometer reading by sample weight (g), and multiply by 100 ($[\text{Adjusted 40s reading}/\text{Sample weight (g)}] * 100 = \text{Silt+Clay\%}$). In this case, the sample weight is 50g, so you are effectively multiplying your adjusted readings by 2 (100/50).
4. Calculation of Clay %: Same as for Silt + Clay, but using adjusted 2h reading ($[\text{Adjusted 2h reading}/\text{Sample weight (g)}] * 100 = \text{Clay\%}$).
5. Calculation of Silt %: Subtract Clay % from Silt + Clay % ($[\text{Silt + Clay\%}] - \text{Clay\%} = \text{Silt\%}$).
6. Calculation of Sand %: Subtract the Silt + Clay % from 100 ($100 - [\text{Silt+Clay\%}] = \text{Sand\%}$).

Clean Up:

After completing each test, make sure to:

1. Wipe off remaining sediments from milkshake mixers.
2. Rinse out metal cups, making sure that no sediments go down the drain.
3. Rinse Plungers, hydrometers, and beakers. Take care that sediments do not go down the drain.

After all tests are complete for a given day:

1. Turn off weigh scale, and wipe off any spilled soil.
2. Unplug milkshake mixers and wipe off any sections that look dirty.
3. Return hydrometers to their cases, and to the drawer they came from. The thermometer should also be returned to this drawer.
4. Pour contents of sedimentations cylinders into container, not down the sink.
5. Rinse the cylinders, along with other glassware.
6. Once rinsed, leave all glassware and spoons to dry on a tray.

Available Materials for Multiple Simultaneous Particle Size Analyses:

- 4 Hydrometers
- 2 Milkshake mixers
- 4 Dispersion Cups
- 21 Sedimentation Cylinders
- 5 Plungers

Hydrometer Method for Soils with >5% Organic Matter

This pretreatment procedure is more complicated and takes a significant amount of time. It may take an entire day and night to complete (or longer for certain samples). I recommend using this method for two samples simultaneously. Though treating more than two samples is possible, it is difficult to work within the fume hood when it is cluttered with many hotplates and beakers. An important note is that, though the initial addition of H₂O₂ is only 50mL, this method can easily use 100-200mL per sample.

Additional Materials:

- Hotplate
- 0.05mm or 0.053mm Sieve
- H₂O₂ (30%) – This is painful to touch, so care should be taken
- 1L Beaker (at least this large)
- Watch glass
- Oven
- Gloves/goggles

Procedure:

1. Separate out all coarse fragments of your soil sample using a 2mm sieve.
2. Weigh out 50g of your fine-textured material, and add to 1L beaker
3. Add 50mL of water.
4. Donning gloves and goggles, add 50mL of H₂O₂ in 10mL increments. Adding slowly will help prevent excessive foaming.
5. Stir and place watch glass over beaker, then move the beaker under a fume hood.
6. Watch for excessive frothing for the next 15 minutes. If this occurs, lower the beaker into a bucket of cold water and evenly disperse several drops of amyl alcohol across the surface of the liquid using a pipette.
7. Any soil that sticks to the watch glass should be rinsed back into the beaker.
8. The foam may take several hours to completely settle. When frothing has subsided enough that the foam is about 1cm thick or less, add about 10mL H₂O₂, and watch for any further reaction. Repeat until little or no reaction occurs (few 10mL additions should be necessary to achieve this).
9. Put the beaker on a hot plate and heat to 80°C (using thermometer to gauge temperature). Keeping a consistent temperature requires continual adjustments at first, but maintaining a stable 80°C can be accomplished after a few minutes.
10. If foam is approaching the top of the beaker, remove the beaker from the hot plate and allow it to cool. Repeat steps 8 and 9 until the foam no longer approaches the top of the beaker when heating.

11. Add 10mL H₂O₂ to beaker, and then wait for the reaction to slow. Repeatedly add small amounts of H₂O₂ in this way while heating at 80°C. When little or no foaming occurs upon further additions of H₂O₂, and the sample and liquid appear bleached, remove from heat.
12. Using a wash bottle, add water to 400mL, making sure to rinse any soil from the edge of the beaker and into the solution and disperse sediments at the bottom of the beaker.
13. Boil on hot plate for 1h, but do not allow all water to boil away. This will remove remaining H₂O₂ from the solution.
14. Remove the beaker from the hotplate and allow cooling and settling. (Leave the beakers overnight to allowing cooling to approximately room temperature). The settled solution should be somewhat clear above the sample.
15. Transfer the suspension to the metal dispersion cup (as in the <5% OM method).
16. Follow steps 4-12 from **Hydrometer Method for Soils with <5% Organic Matter**.
17. After completing steps 4-12 from **Hydrometer Method for Soils with <5% Organic Matter**, pass the suspension through the 0.05mm/0.053mm sieve and rinse with water until only sand remains. The sieve should be placed over a plastic bucket during this step, so that sediments do not go down the drain. Allowing the sand to dry in the sieve somewhat will make it easier to remove.
18. Transfer the sand to a beaker.
19. Dry sand in oven at 105°C for 24h. See Appendix D for oven use instructions.
20. Cool and then weigh sand.

Making Calculations:

1. Adjust for blank hydrometer readings, as in **Hydrometer Method for Soils with <5% Organic Matter** (Appendix B).
2. Adjusting hydrometer values: For every degree your sample is below the temperature at which you took your blank reading, subtract 0.36 from your hydrometer reading. For every degree above that temperature, add 0.36 to your hydrometer reading. This applies to both the 40s and 2h readings.
3. Sample Weight: Add weight of sand (g) to corrected hydrometer reading at 40s ([corrected 40s hydrometer reading] + [weight of sand (g)] = sample weight (g)).
4. Calculation for Silt + Clay %: Divide corrected 40s hydrometer reading by sample weight (g), and multiply by 100 ([Adjusted 40s reading/Sample weight (g)] *100=Silt+Clay).
5. Calculation of Clay %: Same as for Silt + Clay, but using adjusted 2h reading ([Adjusted 2h reading/Sample weight (g)] *100=Clay%).
6. Calculation of Silt %: Subtract Clay % from Silt + Clay % ([Silt + Clay%]-Clay%=Silt%).
7. Calculation of Sand %: Multiply weight of sand by 100, and divide by sample weight ([weight of sand (g)]*100)/ [sample weight (g)]. The value should be equivalent to 100%-[Silt + Clay%].

Additional Clean Up:

- Unplug hotplates when finished
- Rinse all beakers and watch glasses/petri dishes

APPENDIX A: Method for the use of Amyl Alcohol

Amyl alcohol has a very strong odour that can be pervasive in the lab, though it is non-toxic. Hopefully these suggestions will help avoid the persistence of any unpleasant fumes.

1. Don't open the bottle until it is under the fume hood. The smell will be far less substantial.
2. You may want to transfer some amyl alcohol to a smaller bottle (there is one already labeled "amyl alcohol" with a dropper lid), for use throughout your analyses. Some samples will require no amyl alcohol, while others will require a few drops, so a small bottle will be sufficient for many samples.
3. Any applications should be done under the fume hood. Even a few drops in a pipette will be odorous once removed from the hood.
4. Use as few drops as is necessary. Don't over apply, because it will not be of any further help.

APPENDIX B: Running a blank hydrometer reading

The hydrometer readings you will use to calculate particle size distribution will be affected by the blank hydrometer reading. The blank hydrometer reading can be found by running a hydrometer test with water and calgon solution, but no sample.

1. Weigh out 5g of calgon dispersing agent.
2. Add the calgon to a beaker, and then add water to 50mL.
3. Stir.
4. Transfer the calgon solution to a 1L sedimentation cylinder and add water to 1L.
5. Take a temperature reading with a thermometer. The temperature should be approximately the same as that of the samples and the room temperature.
6. Use a plunger to stir calgon and water for 2 minutes.
7. Place a hydrometer in the water, and wait about a minute for it to stabilize before taking a reading. This reading should then be adjusted according to temperature, and subtracted from all 40s and 2h readings for all samples.
8. Do this for all hydrometers and make note of which blank readings correspond to which hydrometers

Adjusting Blank Reading for Temperature:

-For every 1°C that your sample is below the blank reading, you should subtract 0.36 from your reading.

-For every 1°C that your sample is above the blank reading, you should add 0.36 to your reading.

Sample calculation:

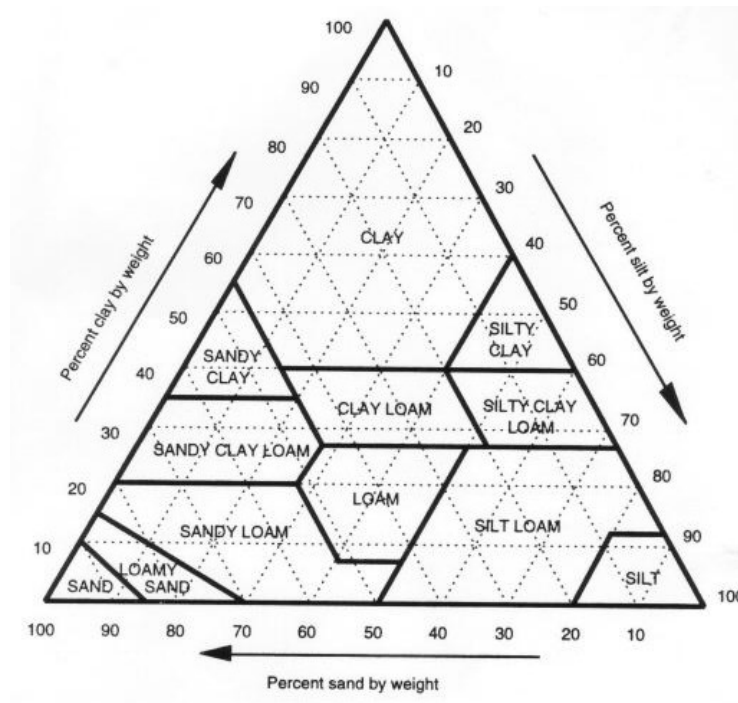
Suppose you took a blank reading of 3.5 at 23°C, and a 40s reading of 22 at 21°C.

Corrected reading = Reading - Sample calculation - [blank temp - read temp] * 0.36 = 22 - 3.5 - [2 * 0.36] = 17.78

APPENDIX C: Soil texture classification using the textural triangle

The goal of your particle size analysis may be to classify your soil sample into a specific textural class, such as a sand, silt, clay, loam, etc. The soil textural triangle can allow one to visually assess textural class based on the three percentage values obtained through particle size analysis.

Figure 1 - Soil Textural Triangle



Example of Methodology for Assessing Textural Class

Assuming you've found, through particle size analysis, that your soil sample contains 55% sand, 23% silt, and 22% clay.

1. First, become accustomed with the textural triangle's three axes.
 - Percent sand is along the bottom of the triangle, and increases towards the left.
 - Percent clay is on the left axis, and increases towards the top.
 - Percent silt is on the right axis and increases towards the bottom.
2. Now, follow the sand axis to halfway between 50 and 60%.
3. Dotted lines travelling upwards and to the left from each of these two values represent 50 and 60% sand anywhere along their length.

4. The lines travelling straight across the triangle, from left to right, represent 10% graduations for percent clay.
5. Follow the lines for 50 and 60% sand up to slightly past the dotted line for 20% clay, to approximately 22%. This point will also coincide with 23% silt if you were to travel downwards and to the left from between the 20 and 30% silt graduations. This point, therefore, is the point on the graph where your sand, silt, and clay percentages add to 100%.
6. In this case, the bold lines around this point contain the textural class of “sandy clay loam.”

Optional Alternative:

I would recommend a visit to the Natural Resources Conservation Service Website:

<http://soils.usda.gov/technical/aids/investigations/texture/>, where you will find a soil texture calculator which automatically plots percent sand, silt, and clay upon a textural triangle, and gives you a texture reading. Simply input your sand and clay percentages, click “Get Type,” and read the output of the “Texture:” box.

Appendix D: Use of the oven

This method applies to the oven in RCB 6303.

1. Note that the dial is on a Fahrenheit scale, but you want a temperature of 105°C. On the oven there is a conversion for this temperature (221F=105°C).
2. Turn the dial to 221F (105°C), then place the beaker containing your sample in the oven.
3. Wait 24h.
4. Turn the dial back to 0F.
5. You can wait for the beaker to cool before removal, or use tongs or a heat resistant glove to remove the sample.