

Particle Size Analysis by Hydrometer

Scope

This document describes the measurement of the abundance of sand-, silt-, and clay-sized particles in soil samples using the principle of sedimentation. Clay content is calculated using the density of a dispersed suspension (of water and clay particles) as measured with a hydrometer; sand content is measured directly by wet sieving; silt is calculated by adding sand and clay content and subtracting from 100. The procedure gives two separate estimates of silt and sand content, which can be compared to verify the effectiveness of sample dispersion. Results are reported as percent sand, silt, and clay on an oven-dry (105 °C) soil mass basis. We note that different labs may have different dispersion procedures, and SHI is flexible but would like to discuss dispersion methods different from those suggested below.

Equipment

2-mm (10 mesh) sieve
53- μ m (270 mesh) sieve (47- μ m/300 mesh also acceptable; specify which sieve is used in final report)
Air-drying facility (minimum temperature: 30 °C)
Coarse-grinding apparatus (e.g., rolling pin, flail mill)
Drying tins
Drying oven
Desiccator
Analytical balance (± 0.1 g sensitivity)
500 mL beaker (1 per sample)
1000 mL sedimentation cylinder (1 per sample + 1 for blank)
Stirring rod with rubber stopper on bottom
Dispersion cup
Milkshake mixer or sample shaker
Hydrometer (units g/L)
Thermometer

Reagents

Sodium hexametaphosphate dispersant

Weigh out 35.7 g sodium hexametaphosphate and 7.94 g sodium carbonate. Place in a volumetric flask and bring to 1.0 L volume with distilled water. In case of incomplete dissolution, dissolve sodium hexametaphosphate and sodium carbonate separately, mix, and bring to 1.0 L volume.

Amyl alcohol

Procedure

1. Air-dry the bulk soil sample at 30-40 °C. Transfer the sample to a paper bag or spread out on a tray before drying. Forced air circulation is recommended to remove humidity. Soils that form very strong aggregates upon drying (e.g., Andisols) should be dried at lower temperatures (<25 °C). When in doubt, consult the sample submitter.
2. Break up the bulk soil sample with a rolling pin or coarse-grinding mill (e.g., flail mill) and pass through a 2-mm sieve. When working with large samples (e.g., composite samples), a subsample may be taken prior to sieving using a sample splitter or by spreading the soil sample across a flat surface, dividing the soil into sections (e.g., a rectangular grid or pie), and collecting all material within multiple non-adjacent sections. Visually verify that the proportions of large to small aggregates/particles are similar for the subsample and the bulk soil sample, as soil aggregates will separate by size during sample handling.
3. Some soils require extra pretreatments to remove organic matter and/or mineral phases that impede dispersion. Following Soil Survey Staff (2014) guidelines, SHI recommends chemical oxidation of organic matter using hydrogen peroxide or sodium hypochlorite (bleach) when analyzing samples with >2% organic carbon. Additional pretreatments may be necessary for samples with >0.25% inorganic carbon (=2% CaCO₃) or high concentrations of iron oxides, gypsum, or soluble salts. Consult SHI or the sample submitter when in doubt about pretreatments.
4. Weigh and record the mass of an empty, labeled drying tin. Add 10.0±1.0 g sieved soil to drying tin and record total mass of tin and soil. Dry the 10.0 g subsample in an oven at 105 °C for a minimum of 24 hours. After drying, place sample in desiccator to cool to room temperature, and record the total oven-dry mass of the tin and soil.
5. Weigh 40.0±4.0 g of soil in 500 mL beaker. For soils with abundant clay and/or silt, sample masses may need to be reduced (minimum 20.0±2.0 g) so that the density of the soil + sodium hexametaphosphate suspension is within the range of the hydrometer scale (e.g., 0.995-1.038 g/L for a standard NIST hydrometer).¹ Increasing sample masses (maximum 100.0±10.0 g) may improve estimation of silt + clay in sands and loamy sands.
6. Shake or stir carboy of sodium hexametaphosphate dispersant in case reagent has precipitated. Add 100 mL deionized water and 100 mL sodium hexametaphosphate dispersant to soil in 500 mL beaker (note: determine dispersant volume following Calculation #1 below if sodium hexametaphosphate concentration in dispersant differs from 35.7 g/L or if air-dry soil mass differs from 40.0±4.0 g). Swirl and allow to soak overnight. If a sample shaker is available, conduct steps 4 and 5 in a sealable bottle rather than beaker and shake overnight.
7. Transfer soil suspension to dispersion cup and mix for 5 minutes with milkshake mixer.
8. Transfer soil suspension to 1000 mL sedimentation cylinder and bring to 1000mL volume with deionized water.
9. To prepare reagent blank, add 100 mL sodium hexametaphosphate dispersant to 1000 mL sedimentation cylinder and bring to volume with deionized water.
10. Manually stir sample up and down for 30 seconds. Ensure complete suspension with several strong upward strokes just above sediment.² Record time that stirring stops. If sample surface is foamy, add 2 mL (a few drops drops) amyl alcohol to the foam on top of the suspension.

11. Carefully insert hydrometer into suspension and read density at 40 seconds after mixing stopped. Remove hydrometer, rinse, and wipe dry. Record temperature of suspension. Measure density and temperature of reagent blank at 40 seconds.
12. Reinsert hydrometer into soil suspension and record density after 7 hours. Again, record temperature of suspension and density and temperature of reagent blank.
13. Transfer soil and soil suspension from 1000mL cylinder to prewetted 53- μ m sieve. Wash with tap water and gently rub material retained on sieve, continue washing until only sand remains in the sieve (water runs clear) and wash into weighed and labeled evaporation dish. Dry at 105 °C for a minimum of 24 hours and record mass after cooling to room temperature in a desiccator.

Calculations

1. $v_{\text{NaHMP}} = 0.09 m_{\text{AD}} / c_{\text{NaHMP}}$
 where:
 v_{NaHMP} = volume (mL) of sodium hexametaphosphate needed to achieve a consistent ratio of dispersant mass to soil mass (0.09 g sodium hexametaphosphate:1 g soil)
 m_{AD} = mass (g) of soil used for hydrometer analysis, air-dry basis
 c_{NaHMP} = concentration (g/mL) of sodium hexametaphosphate in dispersant (0.0357 g/mL if reagent is prepared following this SOP)
2. $\text{OD/AD} = (\text{mass of oven-dry soil and tin} - \text{mass of empty tin}) / (\text{mass of air-dry soil and tin} - \text{mass of empty tin})$. Commonly referred to as a moisture correction.
3. $m_{105} = m_{\text{AD}} \times \text{OD/AD}$
 where:
 m_{105} = mass (g) of soil used for hydrometer analysis, oven-dry basis
4. $\rho_{y,t,20} = \rho_{y,t,T} + 0.36 \times (T - 20)$
 where:
 $\rho_{y,t,20}$ = density (g/L) of suspension y (sample or blank) adjusted to 20 °C
 T = suspension temperature (°C) at time t
 $\rho_{y,t,T}$ = suspension density (g/L) at time t and temperature T
5. $\% \text{ clay} = (\rho_{s,7h,20} - \rho_{b,7h,20}) / m_{105} \times 100$
 where:
 $\% \text{ clay}$ = mass percent of particles < 2 μ m in diameter
 $\rho_{s,7h,20}$ = density (g/L) of sample suspension at 7 hours adjusted to 20 °C
 $\rho_{b,7h,20}$ = density (g/L) of blank at 7 hours adjusted to 20 °C
6. $\% \text{ sand}_{\text{sieve}} = \text{sand}_{105} / m_{105}$
 where:
 $\% \text{ sand}_{\text{sieve}}$ = mass percent of particles > 53 μ m in diameter as determined via sieving
 sand_{105} = mass of material retained on 53- μ m sieve, oven-dry basis
7. $\% \text{ sand}_{\text{hydrometer}} = 100 - (\rho_{s,40s,20} - \rho_{b,40s,20}) / m_{105} \times 100$
 where:
 $\% \text{ sand}_{\text{hydrometer}}$ = mass percent of particles > 50 μ m in diameter as determined via sedimentation
 $\rho_{s,40s,20}$ = density (g/L) of sample suspension at 40 seconds adjusted to 20 °C

$\rho_{b,40s,20}$ = density (g/L) of blank at 40 seconds adjusted to 20 °C

8. $\% \text{ silt}_{\text{sieve}} = 100 - \% \text{ clay} - \% \text{ sand}_{\text{sieve}}$

where:

$\% \text{ silt}_{\text{sieve}}$ = mass percent of particles with diameters between 2 and 53 μm , with upper diameter limit determined via sieving

9. $\% \text{ silt}_{\text{hydrometer}} = 100 - \% \text{ clay} - \% \text{ sand}_{\text{hydrometer}}$

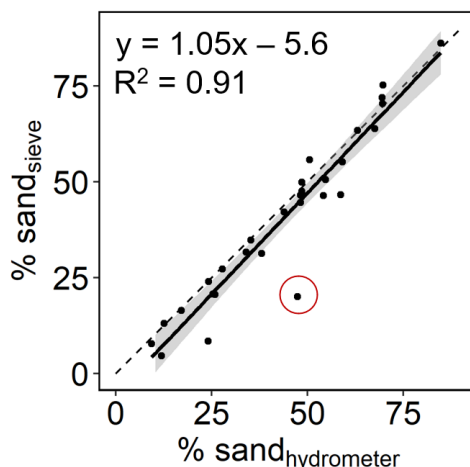
where:

$\% \text{ silt}_{\text{hydrometer}}$ = mass percent of particles with diameters between 2 and 50 μm , with upper diameter limit determined via sedimentation

QA/QC

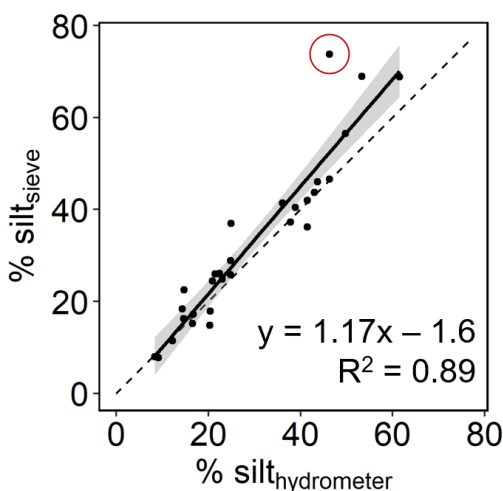
1. After every 50 samples, run a duplicate analysis on one of the preceding 100 samples selected at random. Duplicate readings should differ by no more than 8%.³
2. Inspect scatterplots of the following quantities to verify expected positive relationships and diagnose potential bias. Major scatter and/or outliers may indicate procedural errors.

$\% \text{ sand}_{\text{hydrometer}}$ vs. $\% \text{ sand}_{\text{sieve}}$



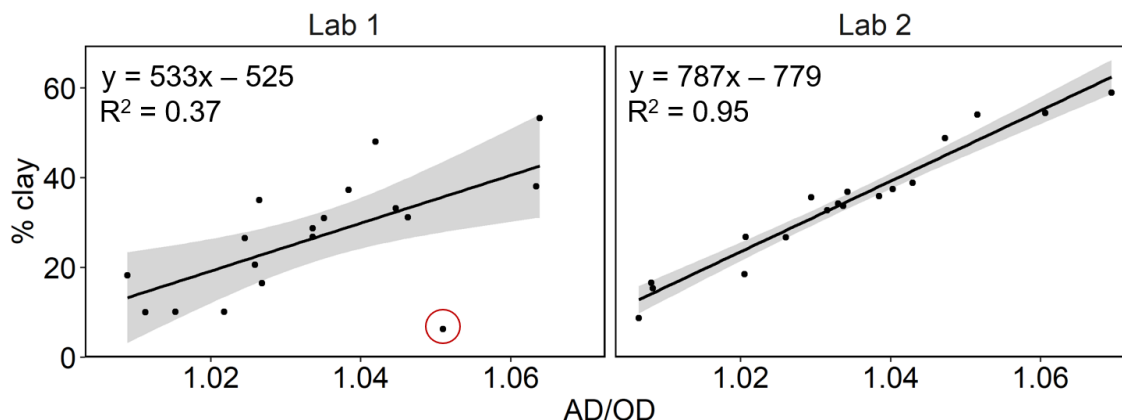
For this set of samples from Texas and Arkansas, sand concentrations estimated using sedimentation (x-axis) agree reasonably well with sand concentrations estimated by sieving (y-axis). The slope of the relationship between the two methods is not significantly different from the dashed 1:1 line ($p=0.4$). Overestimation of sand concentrations via sedimentation was relatively minor on average (5.6% more sand for hydrometer vs. sieving, $p = 0.07$) but reached 27% for the outlier circled in red. These diagnostics suggest that sand-sized aggregates were not fully dispersed in several of these samples.

$\% \text{ silt}_{\text{hydrometer}}$ vs. $\% \text{ silt}_{\text{sieve}}$



This scatterplot depicts silt concentrations for the same set of samples in the previous scatterplot, with silt estimated using sedimentation only (x-axis) vs. a combination of sedimentation and sieving (y-axis). A slope significantly greater than 1 ($p=0.04$) indicates that silt concentrations were underestimated for the siltiest samples via sedimentation. When interpreted in conjunction with the previous plot, this plot suggests that silt-sized particles were included in the material that fell out of suspension within the first 40 seconds of the measurement sequence.

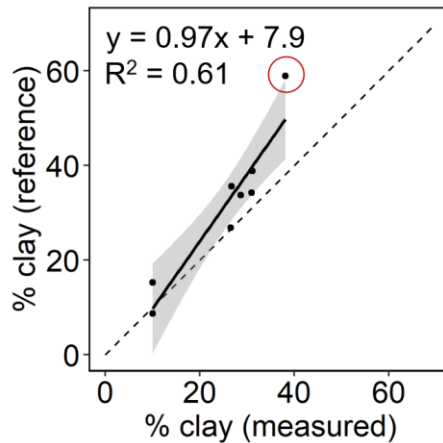
% clay vs. AD/OD



Here, we compare the relationship between the ratio of air-dry to oven-dry mass (i.e., 1 / moisture correction factor) to clay concentration for a single set of samples analyzed by two different labs. The lower slope and greater spread (gray envelope) of data from Lab 1 suggest inconsistent and incomplete dispersion compared to Lab 2. This relationship also allows identification of outliers (e.g., point in red circle) when data from independent laboratories are unavailable for verification.

- Analysis of a minimum of eight soil standard reference materials ranging in texture is recommended every six months and after any procedural modifications. Soil standards that have been analyzed at an external laboratory will be provided by SHI. Additional soil standards

can be developed in-house but must be analyzed at least once at an external laboratory to obtain independent reference values.



Periodically analyzing a set of soil standards allows quantification of measurement reproducibility (i.e., consistency of results for a single sample measured multiple times) and may help reveal systematic errors for individual particle size fractions. For this set of samples, the slope and intercept for the relationship between lab measurements and reference values were not significantly different from 1 or 0, respectively, as expected in the absence of bias.

References

1. Soil Survey Staff. 2014. Soil Survey Field and Laboratory Methods Manual. Soil Survey Investigations Report No. 51, Version 2.0. R. Burt and Soil Survey Staff (ed.). U.S. Department of Agriculture, Natural Resources Conservation Service.
2. Kroetsch, D., and Wang, C. 2007. Particle Size Distribution in *Soil Sampling and Methods of Analysis* (2nd ed.). M.R. Carter and E.G. Gregorich (ed.). Boca Raton, FL: CRC Press.
3. Gavlak, R., D. Horneck, and R. Miller. 2005. Plant, Soil and Water Reference Methods for the Western Region. Western Regional Extension Publication (WREP) 125, WERA-103 Technical Committee, <http://www.naptprogram.org/files/napt/western-states-method-manual-2005.pdf>.

NOTE

This SOP was developed by SHI, for SHI communication, and developed after Method 3.2.1.2.1.1–3.1 of the Soil Survey Field and Laboratory Methods Manual (v2, 2014), Gavlak et al. 2005, Kroetsch and Wang 2007, and American Agricultural Laboratory SOP#01073.04 (2012). For any specific questions, contact contact Dr. Liz Rieke erieke@soilhealthinstitute.org.