84-026 PARTICLE SIZE DISTRIBUTION (Filter Candle System)

1. Application

1.1 Particle-size analysis is the measurement of the proportions of the various sizes of primary soil particles as determined usually either by their capacities to pass through sieves or various mesh size or by their rates of settling in water. The proportions are usually represented by the relative weights of particles within stated size classes. The limits of these size classes differ in various commonly used systems of soil particle-size classification (Fig. 12). In this Manual the CSSC system is used. For engineering interpretations, the AASHO and the Unified systems are used.

There is no general 'best' method of doing particle-size analysis. The best method in a particular case depends upon the nature of the soil being analyzed, the purpose of the analysis, time constraints, and the equipment available. The following method was adapted from the U.S. soil survey Lincoln, Nebraska and reduces the time required to do the analysis by replacing the numerous centrifuge washing steps with a filter candle system. The data determined using the filter candle system corresponds well with the data from the pipette method and centrifuge washing.

2. Apparatus

2.1 Fleakers - 300 mL plus plastic caps.

2.2 Filter candles - FP-88-02.

2.3 Shakers 1. end-over-end (40-60 rpm)
2. sieve shaker (500 oscillations per minute).

2.4 Cylinder - soil suspension (1205 mL) marked at 1000 mL.

2.5 Racks 1. Custom built metal frame to hold 4 motor driven stirrers equipped with propeller type stirrer and teflon guard.
2. Shaw pipet rack modified to hold four 25 mL Lowy pipets.
3. Custom built wood frame to support fleakers, filter candles, and vacuum system.

2.6 Styrofoam pipe insulating cover.

2.7 100 mL beakers or wide mouth glass pill bottles.

2.8 Balance (0.1 mg sensitivity).

2.9 Sieves 1. 300 mesh 6".
2. Set of sieves brass 2 1/2". U.S. series and Tyler screen scale equivalent designations as follows:

Opening mm	U.S.A. No.	Tyler Mesh Size
1.00	18	16
0.50	35	32
0.25	60	60
0.105	140	150
0.046	300	300

3. Reagents

3.1 Hydrogen peroxide (30 or 50%).

3.2 Hydrochloric acid 1N.

3.3 Citrate-bicarbonate buffer. Prepare a 0.3M solution of sodium citrate (88.4 g/L) and add 125 mL of 1M sodium bicarbonate (84 g/L) to each liter of citrate solution.

3.4 Sodium hydrosulphite (dithionite).

3.5 Saturated sodium chloride solution.

3.6 Prepare a solution of sodium metaphosphate with enough sodium carbonate added to bring the pH to 10 ($Na\ PO_3$) 35.7 g/L + Na_2CO_3 7.9 g/L is suitable.

4. Procedure

4.1 Removal of Carbonates

4.1.1 Weigh 10 g of 2 mm air dry soil into a 300 mL fleaker (tared to 1 mg). If the sample appears to be sandy, weigh a larger sample (e.g. 30 g).

4.1.2 Add 50 mL of water, mix and add 1N HCl slowly until the pH falls between 3.5 and 4.0 and remains there for 10 minutes. Stronger HCl can be used to avoid having a large volume of solution in soils high in carbonate content. Soils requiring a large amount of HCl to adjust the pH are washed several times with water to remove excess acid by using the filter candle system.

4.2 Removal of Organic Matter

4.2.1 Add 10 mL of hydrogen peroxide (H_2O_2 30 or 50%) to the fleakers, cover and allow to stand. If a violent reaction occurs, repeat the cold H_2O_2 treatment until no more frothing occurs.

4.2.2 When frothing subsides heat to about 90°C. Continue adding H_2O_2 and heating until most of the organic matter is destroyed (as observed by the color and the rate of reaction of the sample).

4.2.3 Rinse down the sides of the reaction vessel occasionally. Continue heating the sample for about 45 minutes after the final addition of hydrogen peroxide to remove excess hydrogen peroxide.

NOTE: 1. It may be necessary to transfer samples containing high amounts of organic matter (>5%) to large beakers (e.g. 1000 mL tall form).

2. If excessive frothing occurs cool the container either with cold water or by the addition of methyl alcohol to avoid sample loss.

4.3 Removal of Soluble Salts.

4.3.1 Place the fleakers in a rack and filter the remaining peroxide and water off from step 4.23 using a filter candle system.

4.3.2 Add 150 mL water in a jet strong enough to stir the sample and filter the suspension through a filter candle (FP-88-02) system. Five such washings and filterings are usually enough except for soils containing much coarse gypsum. To test for salts check with silver nitrate ($AgNO_3$) for Cl and barium chloride for SO_4^{2-}.

4.3.3 Remove soil adhering to the filter candle by applying back pressure gently, and using a rubber tipped finger as a policeman.

NOTE: If iron oxides are to be removed DO NOT complete step 4.34 at this time.

4.3.4 Place the sample in an oven overnight at 105°C, cool in a dessicator, and weigh to the nearest milligram. Use the weight of the oven-dry treated sample as the base weight for calculating percentages of the various fractions.

4.4 Removal of Iron Oxides (Optional)

NOTE: Iron oxides should be removed from samples to permit the determination of phyllosilicate minerals by x-ray diffraction but this is not necessary for most samples. However, if the interest is in iron oxides in the clay fraction, pretreatments with dithionite-citrate-bicarbonate must be avoided.

4.4.1 Add 150 mL of citrate-bicarbonate buffer to the samples in the fleakers. Stir and add 3 g of sodium hydrosulfite ($Na_2S_2O_4$) gradually as some samples may froth.

4.4.2 Put the fleakers in a water bath at 80°C and stir intermittently for 20 minutes.

4.4.3 Remove the fleakers from the bath, place in the holding rack and filter the suspension through the filter candle system. If a brownish color remains repeat steps 4.4.1 to 4.4.3 inclusive. If the samples are completely gleyed (gray) proceed to step 4.4.4.

4.4.4 Wash five times with a jet of water strong enough to stir the sample and filter the suspension through the filter candle system.

4.4.5 Do step 4.3.4 to determine the oven dry weight.

4.5 Dispersion of Sample.

4.5.1 Add 10 mL of sodium metaphosphate dispersing agent to the fleakers containing the oven dry treated samples. Make the volume to 200 mL with distilled water.

4.5.2 Stopper tightly and shake end-over-end (50-60 rpm) overnight.

4.6 Separation of Sand Fractions.

4.6.1 Pour the suspensions through a 300 mesh (47 μm) sieve into a sedimentation cylinder marked at one liter. The 300 mesh sieve of about 14 cm diameter is placed in a large funnel held above the cylinder (2.4) by a retort stand.

4.6.2 Wash the sand retained on the sieve thoroughly with a fine jet of water and collect the washings in the cylinder until the volume in the cylinder is about 950 mL. Remove sieve and make final volume to 1000 mL.

4.6.3 Transfer the sand to a 100 mL beaker and oven dry at 105°C. Weigh the sand and record weight at this time if only total sand is being determined. Otherwise proceed with sand fractionation.

4.6.4 Transfer the dried sand to a set of sieves (6 cm diameter) arranged as follows from top to bottom: 1 mm, 0.5 mm, 0.25 mm (60 mesh), 0.105 (140 mesh), 0.047 (300 mesh) and pan. Pour sand on the top sieve, put the cover in place and shake the sieves on a sieve shaker. The time of shaking depends on the type of shaker and volume of sand (usually 5 to 10 minutes is sufficient). Weigh each sand fraction and record weight.

4.7 Determination of Clay (0-2 μm)

4.7.1 Before placing cylinders in sedimentation room (any vibration free area equipped with Shaw pipet rack), stir the material in the sedimentation cylinders for 4 minutes with a motor driven stirrer (8 minutes if suspension has stood for longer than 16 hours).

4.7.2 Remove from stirrer, slip a length of styrofoam pipe-insulating cover over sedimentation cylinder. Stir the suspension for 30 seconds with a hand stirrer, using an up and down motion. Note the time at the completion of stirring.

4.7.3 Sample the 2 μm fraction after a predetermined settling time (usually 4.5 to 6.5 hrs), varying depth according to time and temperature. About 1 minute before sedimentation is complete, lower the tip of a closed Lowy 25 mL pipet slowly into the suspension to the proper depth with a pre-calibrated shaw pipet rack. Regulate the filling time of the pipet to about 12 seconds. Fill the pipet and empty it into a tared 90 mL wide mouth bottle (or 100 mL beaker) and rinse the pipet into the bottle once.

4.7.4 Evaporate the water and dry in an oven at 105°C for at least 24 hours. Cool in a desiccator containing phosphorus pentoxide (P_2O_5) as a desiccant. Weigh and record the weight.

4.8 Determination of Clay 0.2 μm (optional)

4.8.1 Pour about 200 mL of suspension from the sedimentation cylinders into 250 mL centrifuge bottles. Shake the suspensions, and centrifuge at the appropriate speed for the time necessary to sediment particles coarser than 0.2 μm to a depth of 5 cm (54 minutes at 1500 RPM on an IEC Model V Centrifuge). The formula to use is based on Stokes' law:

$$t = \frac{63.0 \times 10^8 \, n \log R/S}{N^2 D^2 \Delta s}$$ where

n = viscocity in poises at the existing temperature

R = radius of rotation (cm) of the top of the sediment in the tube

S = radius of rotation (cm) of the surface of the suspension in the tube

N = revolutions per minute

D = particle diameter in μm

Δs = difference in specific gravity between the solvated particle and the suspending liquid (usually use s = 1.65)

t = time in minutes
(see Jackson, 1956 for tables of centrifuging times and speeds)

4.8.2 Withdraw a 25 mL aliquot from a depth of 5 cm. Discharge the sample into a tared weighing bottle or beaker, rinse the pipette, add the rinsing to the weighing bottle, dry at 105°C, cool in a desiccator and weigh.

5. Calculations

5.1 A = weight (g) of pipetted fraction (2 μm or 0.2 μm)

B = weight correction for dispersing agent (g)

NOTE: To determine the correction factor, add 10 mL of the sodium metaphosphate solution (3.6) to a 1 liter cylinder, make to volume, stir thoroughly, withdraw duplicate 25 mL samples, dry and weigh (about 0.012 g).

$$K = \frac{1000}{\text{pipet (mL)}}$$

$$D = \frac{100}{H_2O_2 \text{ treated oven dry total sample (g)}}$$

5.2 Sand fraction(s):
Percentage of sand fraction (s) = weight (g) of fraction on sieve times D.

Pipetted fraction(s):
Percentage of pipetted fraction(s) = (A-B)KD.

Silt fraction:
Percentage of silt = 100 - (0-2 μ clay + sand).

6. Precision

6.1 Within the LRRI analytical service lab the coefficients of variation at sand levels of 56%, silt levels of 31% and clay levels of 12.2% were 6.9%, 9.2% and 9.0% respectively.

7. References

7.1 McKeague, J.A. Ed. 1978. Manual on soil sampling and methods of analysis. 2nd Edition. Can. Soc. Soil Sci. Suite 907, 151 Slater St., Ottawa, Ont.

Table 5. Settling Depths for Specific Times and Temperatures for Particle Size = 2 μm.

Temp. C°	Time			
	4 1/2 hrs. Depth-cm.	5 hrs. Depth-cm.	5 1/2 hrs. Depth-cm.	6 1/2 hrs. Depth-cm.
20.0	5.79	6.44	7.08	8.37
20.3	5.81	6.48	7.13	8.43
20.5	5.86	6.52	7.17	8.47
20.7	5.89	6.55	7.20	8.51
21.0	5.93	6.59	7.25	8.57
21.3	5.97	6.64	7.30	8.63
21.5	6.01	6.68	7.33	8.68
21.7	6.04	6.72	7.39	8.73
22.0	6.09	6.75	7.43	8.78
22.3	6.13	6.80	7.49	8.85
22.5	6.15	6.83	7.51	8.88
22.7	6.18	6.86	7.55	8.92
23.0	6.22	6.91	7.60	8.98
23.3	6.27	6.96	7.66	9.05
23.5	6.29	6.98	7.68	9.08
23.7	6.33	7.04	7.74	9.15
24.0	6.37	7.08	7.78	9.20
24.3	6.40	7.12	7.83	9.25
24.5	6.43	7.15	7.86	9.29
24.7	6.45	7.18	7.89	9.33
25.0	6.51	7.24	7.96	9.41
25.3	6.56	7.28	8.01	9.47
25.5	6.58	7.31	8.04	9.50
25.7	6.61	7.35	8.08	9.55
26.0	6.66	7.40	8.14	9.62
26.3	6.69	7.44	8.18	9.67
26.5	6.72	7.47	8.22	9.72
26.7	6.76	7.51	8.26	9.76
27.0	6.81	7.56	8.32	9,83
27.3	6.85	7.61	8.37	9.89
27.5	6.87	7.64	8.40	9.93
27.7	6.91	7.68	8.44	9.98
28.0	6.97	7.74	8.51	10.06
28.3	7.01	7.79	8.57	10.13
28.5	7.04	7.82	8.61	10.17
28.7	7.07	7.86	8.65	10.22
29.0	7.12	7.91	8.70	10.28
29.3	7.16	7.95	8.75	10.34
29.5	7.19	7.99	8.79	10.39
29.7	7.22	8.02	8.82	10.43
30.0	7.27	8.08	8.88	10.50

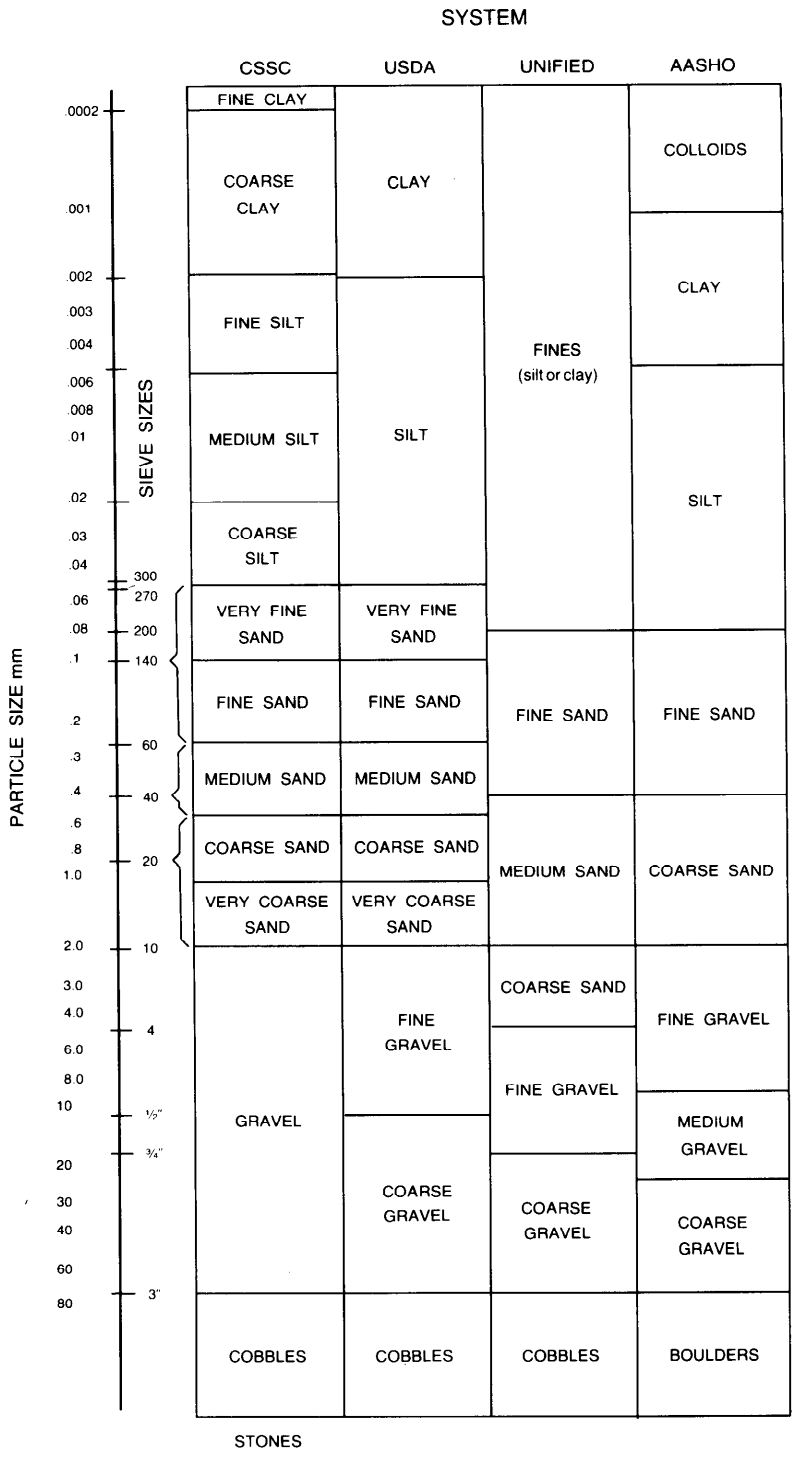

Fig. 12 A comparison of particle size limits in 4 systems of particle size classification.

AASHO - American Association of State Highway Officials
USDA - United States Department of Agriculture
CSSC - Canada Soil Survey Committee