

PARTICLE-SIZE ANALYSIS

1 PRINCIPLE

Separation of the mineral part of the soil into various size fractions and determination of the proportion of these fractions.

Most of the following protocol was proposed by **Van Reeuwijk L.P.**, 2002 "**Procedures for soil analysis**" (6th edition). Technical Paper No. 9, ISRIC, Wageningen. <http://www.isric.org/content/technical-papers16-1>

The ISRIC protocol has been adapted and illustrated for use in the GeoScience Laboratory of the Faculty ITC, University of Twente, The Netherlands

The analysis comprises all material, i.e. including gravel and coarser material but the procedure below is applied to **the fine earth <2 mm only.**

If the > 2 mm fraction is significant and the total sample is to be considered, the > 2 mm is to be sieved off, rinsed with demi water, dried at 40°C and weighed to determine the >2mm percentage in the total sample material. This value is to be incorporated in the final calculations.

Of paramount importance in this analysis is the pretreatment of the sample aimed at complete dispersion of the primary particles. Therefore, cementing materials (usually of secondary origin) such as organic matter and calcium carbonate may have to be removed. In some cases also sesquioxides (iron-hydroxide coatings) may need to be removed. It may be argued, however, that for agricultural purposes it is often not relevant or even fundamentally wrong to remove these components. Thus, depending on the aim of study, all pretreatments are to be considered optional. For soil characterization purposes, in the ISRIC laboratory removal of organic matter by H₂O₂ and of carbonates by HCl is routinely carried out.

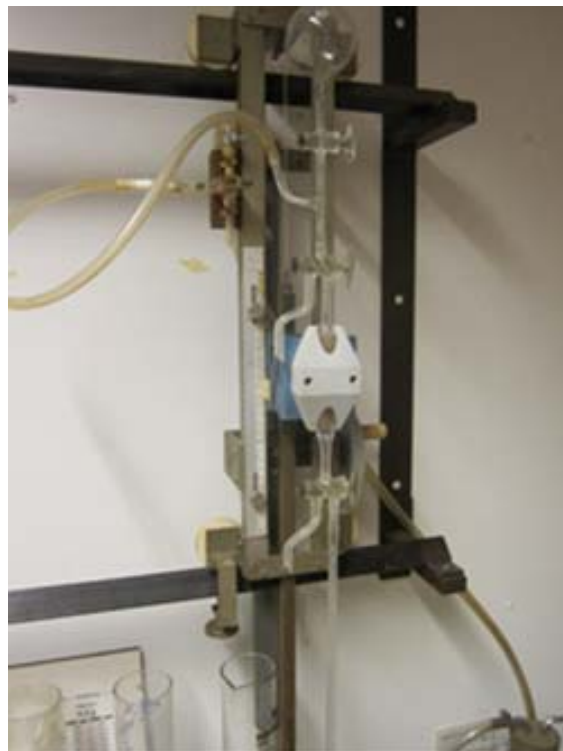
After shaking with a dispersing agent, sand is separated from clay and silt with a 50 µm sieve. In the 1995 ISO/DIS 1127 the 50µm boundary has been changed to a 63 µm boundary. The sand is subsequently fractionated by dry sieving, the clay and silt fractions are determined by the pipette method.

The whole procedure for one batch of ten soil samples requires steps in five days. On the second day a second batch can be started etc. etc. Each batch of ten samples must also contain in addition; one duplicate/repeat sample from an earlier batch, one reference control sample and one blank sample

2. APPARATUS

The following requirements are for one batch

1. Top loading balance (1mg units)
2. Water bath,
3. Hot plate(s),
4. End-over-end shaking machine,
5. Sieving machine, by vibration,
6. Set of sieves, including bottom (diam. 20 cm)
7. Heavy brass funnel (diameter approx. 23 cm) on stand,
8. Small 50 μm or 63 μm sieve (diameter 8 cm),
9. 13 one liter glass sedimentation cylinders,
10. 13 one liter glass beakers & covering watch glasses,
11. Suction pump
12. 13 one liter PVC bottles,
13. Two splash bottles,
14. Rubber policeman 3 cm,
15. 20ml pipetting device, see illustration, →
16. Drying oven with forced air circulation,
17. 4x13 Stainless steel moisture tins,
18. Digital precision balance 0.1 mg units,
19. Stopwatch digital timer,
20. Logbook and sample sheets



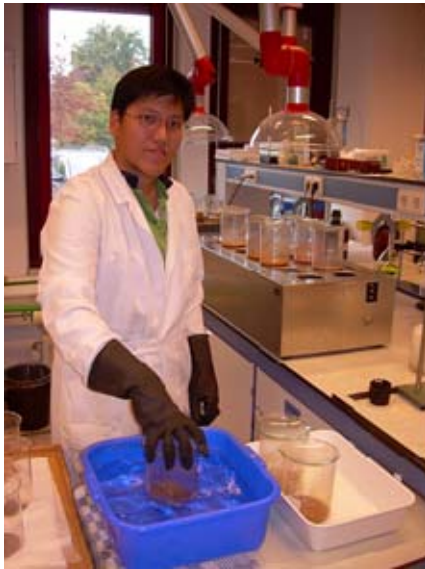
3 REAGENTS

- 1- Hydrogen peroxide H_2O_2 , 30%.
- 2- Dispersing agent: Sodium hexametaphosphate 4% and soda 1% solution ("Calgon"-type). Dissolve 40.0 g $(\text{NaPO}_3)_6$ and 10.0 g NaCO_3 in water in a 1 l volumetric flask and make to volume. Both chemicals should be dried overnight at 105°C prior to use (therefore, hydrated soda qualities may be used).
- 3- Calcium chloride solution, 1 M. Dissolve 147 g $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ in 1 l water.
- 4- Demi water, > 30 liter/ batch

4 PROCEDURE

4.1 Oxidation of organic matter

1. Weigh out approx. 20 g fine earth into all beaker (at carbonate contents exceeding 10% and carbonate is to be removed, weigh out proportionally more soil).



2. **For this step; use protective gloves, a lab coat and safety glasses**

Add 15 ml water and 15 ml H₂O₂ 30%. Cover beaker with watch-glass. In case of strong frothing place beaker in basin with cold water. In addition, frothing can be tempered by adding a few drops of ethanol.

3. Let stand overnight. The beakers are covered with a watch glass.

4. The next day, place beaker on water bath (80°C) and regularly add 5-10 ml increments of H₂O₂ 30% until decomposition of organic matter is completed (usually the supernatant is clear then).
5. Add water to a volume of about 300 ml
6. Place on hot plate and carefully boil for at least one hour to remove any remaining H₂O₂. Do not let boil to dryness!
7. Remove beaker from hot place and allow to cool under a slight angle.



8. Allow material to settle in the beaker and siphon off using a suction pump or water jet pump. Alternatively, transfer to centrifuge tubes, centrifuge and decant .

Note: Flocculation may be enhanced by adding 25 ml 1 M CaCl₂ solution with a measuring cylinder. The washings have to be repeated until the dark residues of the organic matter have gone. Check that the EC of the washings is below 0.4 mScm before attempting to disperse the residue (this would leave a max. of 0.02 g salt in the sample,

corresponding with an error in the correction for dispersing agent of max. 2% which is negligible).

If presence of salts or gypsum is suspected (e.g. from EC check in pH-H₂O extract) measure electrical conductivity of supernatant solution.

- 10 If EC of supernatant solution is higher than 0.4 mS/cm, add about 250 ml water, cap centrifuge tube and shake in end-over-end shaker for one hour (or stir from time to time for one hour) and repeat Steps 8 and 9 until EC of supernatant solution < 0.4 mS/cm.

Proceed with Dispersion (section 4.4) unless carbonates (4.2) and/or iron oxides/hydroxides (4.3) are to be removed first.

4.2 Removal of carbonate (optional)

4.2.1 Reagent

Hydrochloric acid, 1 M Add 87 ml 32% HCl to 900 ml water and make up to 1 liter with demi water (use fume cupboard, wear lab coat, gloves and safety glasses!).

4.2.2 Procedure

1. To the residue of 4.1 add 25 ml HCl 1 M plus 4 ml of the same for each percent of carbonate in the soil (if about 20 g of sample was used). If proportionally more soil was used (3-4.1 Step 1), calculate weight of carbonate in sample and add 25 ml HCl 1 M plus 1 ml of the same for each 50 mg of carbonate. If carbonate is less than 2% then only an initial 25 ml of the acid is required (if in this case flocculation is not adequate, add 20 ml 1 M CaCl₂ solution). Make up to about 250 ml with demi water.
2. Place the suspension on water bath at approx. 80°C for about 15 min., stirring from time to time.
3. Remove suspension from water bath and leave to stand overnight.
4. If the soil flocculates to leave a perfectly clear supernatant, then this can be siphoned off or decanted, otherwise centrifugation will be necessary.
5. Repeat washing with water and siphoning off or decantation until EC of supernatant < 0.4 mS/cm.

Note: A few minerals might not survive this treatment e.g., some zeolites, chlorite, and allophane. If this is suspected to be quantitatively significant, the treatment should be milder. Several means can be considered;

- Omit the heating to 80°C.
- Use a 10% acetic acid solution (Vol / Vol) instead of HCl.
- Use a 1 Mol Na-acetate buffer pH 5.

This consideration is also important when the clay fraction of the particle-size analysis is afterwards used for X-ray diffraction or IR spectroscopy as is done in some laboratories.

4.3 Deferration, removing Feoxide/hydroxide-coating (optional)

If applied, this treatment is usually done after the other pretreatment procedures and prior to dispersion.

4.3.1 Reagents

Buffer solution 0.3 M sodium citrate and 0.1 M sodium bicarbonate. Dissolve 88 g Na-citrate.2H₂O and 8.4 g NaHCO₃ in water and make to up to 1 liter.

Sodium dithionite (powder).

Sodium chloride solution 1 M Dissolve 58.5 g NaCl in water and make to up to 1 liter.

4.3.2 Deferriation procedure

1. Weigh out approx. 20 g fine earth in a 1 liter beaker and add 200 ml buffer solution.
2. Heat on a water bath to 75°C (do *not* exceed 80°C as elemental sulphur will then precipitate).
3. Add approx. 1 g sodium dithionite with a spoon and stir constantly for about a minute and then occasionally for 5 minutes.
4. Repeat Step 3 two more times.
5. Centrifuge and decant or allow to settle and siphon off.
6. For samples containing more than 5% extractable Fe₂O₃ or Fe-hydroxides, repeat the procedure once or twice: a brownish or reddish colour of the sample may indicate still incomplete deferriation.
7. Wash once more with 250 ml 1 M NaCl when centrifuging, or 500 ml when siphoning.
8. Proceed with 4.1 Step 8.

4.4 Dispersion

1. Transfer suspension quantitatively to a 1 liter polythene bottle (if no pretreatment is given, weigh out approx. 20 g fine earth into this bottle).
2. Add 20.00 ml dispersing agent, make the volume to about 400 ml with water and cap the bottle.
3. Shake overnight (16 hrs.) on an end-over-end shaker at a speed of about 30 rpm.



4.5 Separation of fractions



1. Pass the suspension through a 50 μm sieve which is placed in a funnel positioned above a sedimentation cylinder with a stand and clamp. Use a wide (3 cm) rubber policeman.
2. Make to 1 liter mark with water. Proceed with this according to 4.7.

Note: Include a blank (cylinder with water from same source plus dispersing agent) for temperature measurement in clay determination and for correction of dispersing agent addition.



3. Wash the sand fraction remaining on the sieve quantitatively into a porcelain dish, evaporate on water bath and dry at 105°C for at least an hour.

4.6 Determination of sand fractions

1. Transfer the dried sand of 4.5 Step 3 to the top sieve of a stacked set of sieves of the following mesh sizes: 1000 μm ; 500 μm ; 250 μm ; 100 μm ; 50 μm ; bottom. (Or any other set of desired sizes.)
2. Sieve for 10 minutes on the sieving machine at the settings: amplitude 7.0 and interval 4. (At this setting the sieves vibrate at a frequency of 3000x per minute and an amplitude of 2 mm for 4-second periods interrupted for $\frac{1}{2}$ second.)
3. Empty each sieve into a tared weighing dish by tapping it upside down on the brass funnel placed above the dish. Weigh with 0.01 g accuracy (net weights **A** through **E** individual sand fractions).]



4. If any material is collected in sieve bottom ($< 50\mu\text{m}$) transfer this to suspension in sedimentation cylinder mentioned in 4.5.
Note: If pipetting of the silt fraction is done before the sieving, then the collected material (which usually is very little) should be weighed and the weight added to weight **M** (silt fraction 20-50 μm , see Section 3-5) or to weight **P** (silt fraction 2-50 μm , see Section 3-6). These are the fractions where the material is assumed to be mainly derived from.

4.7 Determination of silt and clay

4.7.1 Calibration of pipette

The pipette method described here is based on sampling a 1 l suspension with a 20.00 ml pipette. Therefore, in the calculations a multiplication factor of $1000/20 = 50$ is used (see Section 3-5). Unless a calibrated volumetric pipette is used, calibration of the pipette is necessary. This can be done by pipetting water and weighing the aliquot (accuracy 0.01 g). Repeat this ten times and take the mean (exclude outliers). If the volume is not 20.00 ml, the multiplication factor of 50 should be changed accordingly.

4.7.2 Blank determination

Although the dispersing agent is prepared precisely, a possible error will be multiplied by 50. It is therefore good practice that this is checked in each batch of analyses. This is done by pipetting the blank cylinder as described for the silt and clay fractions below. (Net weight **Z** for dispersing agent.)

4.7.3 Fraction <50 µm

1. After adding material <50 µm possibly collected during sieving (see 4.6, Step 4) close the sedimentation cylinder with a rubber stopper and shake well.
2. Place the cylinder on the table, remove stopper and immediately pipette 20 ml from the center of the cylinder.
3. Transfer the aliquot to a tared moisture tin, evaporate on water bath and dry overnight at 105°C.
4. Remove tin from drying oven, close with lid and cool in desiccator. Weigh with 0.001 g accuracy (net weight **F** for fraction <50 µm),

4.7.4 Fraction <20 µm

- 1 After measuring the temperature of the suspension, again stopper the cylinder and shake well.
- 2 Place the cylinder on a vibration-free table under the pipette-assembly.
- 3 After exactly 5 minutes pipette 20 ml at a depth indicated in Table 3-1.
- 4 Transfer aliquot to tared moisture tin, evaporate on water bath and dry overnight at 105°Celsius.
- 5 Remove tin from drying oven, close with lid and cool in desiccator. Weigh with 0.001 g accuracy (net weight **G** for fraction <20 µm).

4.7.5 Fraction <2 µm


1. After 51h hours measure temperature in blank cylinder and pipette 20 ml at a depth indicated in Table 3-1.
Note: If this temperature differs from initial temperature (measured in 4.7.4 Step 5), use mean of this and initial temperature.
2. Transfer aliquot to tared moisture tin, evaporate on water bath and dry overnight at 105°C.
3. Remove tin from drying oven, close with lid and cool in desiccator. Weigh with 0.001 g accuracy (net weight **H** for fraction <2 µm).

Remark 1: In case only the clay fraction is to be determined (and not the silt) proceed according to Steps 1, 6, 10, 11 and 12 respectively of this section 3-4.7. Measure initial temperature of suspension.

Remark 2: In some cases peptization of the suspension is not or incompletely achieved. This can easily be observed by flocculation in the cylinder. In this case only the determination of the total fraction < 50 µm is possible, whereas clay and silt cannot be determined. This occurs mainly with calcareous soils, and the removal of carbonate (4.2) is then indicated.

Table 1: Depth (in cm) at which fractions <50 μm, <20μm and <2μm are pipetted as a function of the temperature and after indicated settling time

PARTICLE SIZE DETERMINATION

<-----Sedimentation----->  <-----Centrifuging----->

°C	SILT		CLAY	FINE CLAY		°C	
	< 50 μm 1 min cm	< 20 μm 5 min cm	< 2 μm 5½ hrs cm	0.2 μm 1800 rpm min	2500 rpm min		
15	11.8	9.5	6.3		39.7	20.6	15
16	11.2	9.7	6.4		38.7	20.0	16
17	12.5	10.0	6.6		37.7	19.5	17
18	12.8	10.2	6.8		36.7	19.0	18
19	13.1	10.5	6.9		35.8	18.6	19
20	13.5	10.8	7.1		34.9	18.1	20
21	13.8	11.0	7.3		34.1	17.7	21
22	14.1	11.3	7.5		33.2	17.2	22
23	14.5	11.6	7.6		32.5	16.8	23
24	14.8	11.8	7.8		31.7	16.4	24
25	15.1	12.1	8.0		31.0	16.1	25
26	15.5	12.4	8.2		30.3	15.7	26
27	15.8	12.7	8.4		29.6	15.4	27
28	16.2	13.0	8.6		29.0	15.0	28
29	16.6	13.2	8.7		28.3	14.7	29
30	16.9	13.5	8.9		27.7	14.4	30
31	17.3	13.8	9.1		27.1	14.1	31
32	17.6	14.1	9.3		26.6	13.8	32
33	18.0	14.4	9.5		26.0	13.8	33
34	18.4	14.7	9.7		25.5	13.2	34
35	18.8	15.0	9.9		25.0	13.0	35
36	19.2	15.4	10.1		24.4	12.6	36
37	19.5	15.6	10.3		24.0	12.4	37
38	19.9	15.9	10.5		23.6	12.2	38
39	20.3	16.2	10.7		23.1	12.0	39
40	20.7	16.5	10.9		22.7	11.7	40

5 CALCULATIONS

The basis of the calculations is the oven-dry sample weight after all treatments. It is obtained by summation of all individual fractions:

$$\text{Clay (< 2 } \mu\text{m)} = (\text{H} \times 50) - (\text{Z} \times 50) \quad (\text{wt. K})$$

$$\text{Silt (2-20 } \mu\text{m)} = (\text{G} \times 50) - (\text{Z} \times 50) - \text{K} \quad (\text{wt. L})$$

$$\text{Silt ((20-50 } \mu\text{m)} = (\text{F} \times 50) - (\text{Z} \times 50) - \text{K} - \text{L} \quad (\text{wt. M})$$

$$\text{Sand (> 50 } \mu\text{m)} = \text{A} + \text{B} + \text{C} + \text{D} + \text{E}$$

$$\text{Sample weight} = \text{K} + \text{L} + \text{M} + \text{N} \quad \text{all weights in gram)}$$

where

A through E = weight individual sand fractions

F = weight 20 ml pipette aliquot of fraction <50 μm

G = weight 20 ml pipette aliquot of fraction <20 μm

H = weight 20 ml pipette aliquot of fraction < 2 μm

Z = weight 20 ml pipette aliquot of blank

The proportional amounts of the fractions can now be calculated by:

% clay (<2 μm)	= $\frac{\text{K}}{\text{sample wt.}} \times 100$
% silt (2-20 μm)	= $\frac{\text{L}}{\text{sample wt.}} \times 100$
% silt (20-50 μm)	= $\frac{\text{M}}{\text{sample wt.}} \times 100$
% sand (1000-2000 μm)	= $\frac{\text{A}}{\text{sample wt.}} \times 100$
% sand (500-1000 μm)	= $\frac{\text{B}}{\text{sample wt.}} \times 100$
% sand (250-500 μm)	= $\frac{\text{C}}{\text{sample wt.}} \times 100$
% sand (100-250 μm)	= $\frac{\text{D}}{\text{sample wt.}} \times 100$
% sand (50-100 μm)	= $\frac{\text{E}}{\text{sample wt.}} \times 100$

Note: With this calculation, the clay, silt and sand fractions are obtained in percentages of the *fine earth* (minus carbonate and organic matter which have been removed). The coarse fraction >2 mm, if present, is reported in percentage of the *total soil*. If all fractions need to be reported on *total soil* basis convert above obtained figures for clay, silt and sand as follows:

$$\% \text{clay, silt, sand of total soil} = \frac{100 - \%(\text{fraction} > 2\text{mm} + \text{carbonate} + \text{org. matter})}{100} \times \% \text{clay, silt, sand of fine earth}$$

In case deferration was applied the percentage "free iron" (see 12-1) should be included between the parentheses. The determination of the organic matter and carbonate is done by Walkley Black method or by careful ashing & weighing.