Hydrometer

Meet the difference

Contents

On these operating instructions

☞ **If the text follows a mark (as shown on the left), this means that an important instruction follows.**

⚠ **If the text follows a mark (as shown on the left), this means that an important warning follows relating to danger to the user or damage to the apparatus.The user is always responsible for its own personal protection.**

Text **Italic indicated text indicates that the text concerned appears in writing on the display (or must be typed).**

These operating instructions on the hydrometer are based on the ISRIC publication "Procedures for soil analysis" 5th Edition 1995 and the ISO Standard 11277:1998.
Not only the hydrometer method is described (Chapter 4) but also information is given on sample preparation in general (Chapter 3). The hydrometer kit as described in these instructions meets the demands of the ISO/DIS 11277. We refer to these 2 publications for extra information.

1.   Description

To obtain an accurate determination of the particle size distribution of the smallest fractions it is possible to apply the hydrometer method. In this method the sample is cleaned from organic matter after which it is dried and weighed. Next it is suspended in water and sieved. The solution that passes through the sieve is transferred to a measuring cylinder with water. Hydrometer readings are taken after regular intervals. Sedimentation time and hydrometer readings are used to determine the grain sizes according to the Stoke's Law.
The hydrometer kit, among other items, contains: a number of hydrometers (hydrometers, scale 6 0.995 – 1.035 gms/ml, according to BS 1377 and hydrometers, scale -5 to 6 +60 g/l, according to ASTM D 422 and AASHTO T 88), sedimentation cylinders (1000 cc), weighting rings for sedimentation cilinder, a thermometer, a glass container, a heating element with thermostat and stirrer (including fastening materials), a soil stirrer and various accessories.

The heating element provides precise and safe heating, circulation and temperature control of the water in the glass tank. When used correctly, the constant temperature which the hydrometer method requires is guaranteed. The adjusting knob on top of the apparatus enables you to set the temperature to the desired level. Using the knob at the side of the adjusting knob, you can fix the temperature setting. The mounting of the heater is described in chapter 2.
For more extensive information on the heating element: please refer to the seperately enclosed operating instructions of the heating element (M-083010e Heating element).
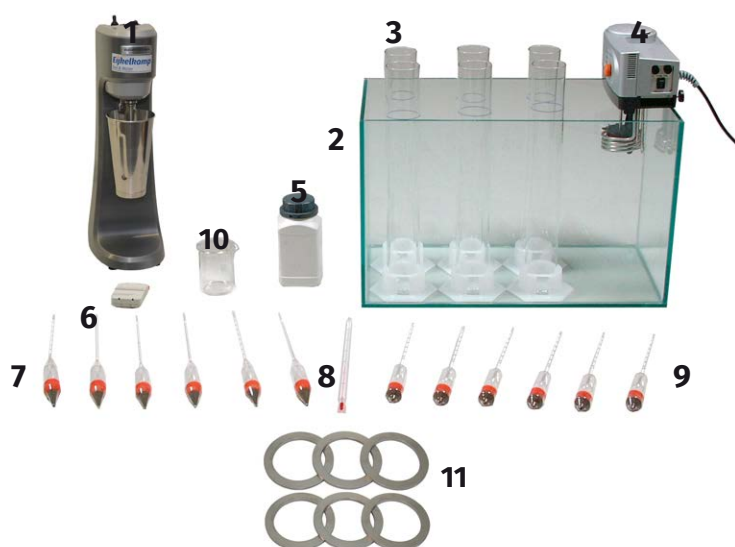
Article numbers:
1 = Soil stirrer (art. no.: 9823)
2 = Glass tank (art. no.: 083008)
3 = Sedimentation cylinder (art. no.: 083012)
4 = Heating element (art. no.: 083010) (installation on support, see page 4)
5 = Sodium Hexametaphosphate (art. no.: 083006)
6 = Stopwatch (art. no.: 090109)
7 = Hydrometer, acc. BS 1377 (art. no.: 08300101)
8 = Thermometer (art. no.: 083003)
9 = Hydrometer acc. ASTM D 422 (art. no.: 08300102)
10 = Beaker (art. no.: 083005)
11 = Weighting rings (art. no.: 08301201 6x)

Not on photo: Rubber stoppers (art. no. 083004) and support for heating element (08301001)

2.   Installation

Every apparatus that comes out of our plant is subjected to a very strict final inspection. Upon receiving the apparatus, please check first whether the apparatus itself or any components have suffered damage during transport. With the help of the enclosed set specification sheet, please ascertain that nothing is missing.

⚠️   **Please read these instructions before using the hydrometer set. Keep the instructions in a place that is accessible for every user.**

☞   **Place the glass tank (container) on a solid table or bench.**

Check if the two stands (1) are installed tight enough.
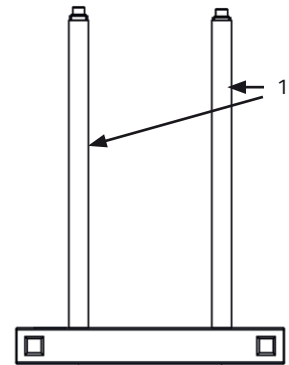
Remove the 2 metal strips (2) by unscrewing the two bolts (3).
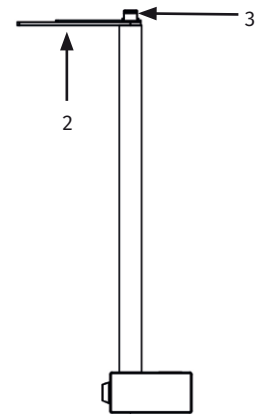
Mount the strips under the heating element by turning in the small screws in the holes (4).
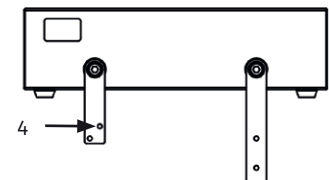
Place the complete support on one side of the glass container. Install it in such a way that the heating element is inside the glass container and the rubber protectives (5) are near the glass container.
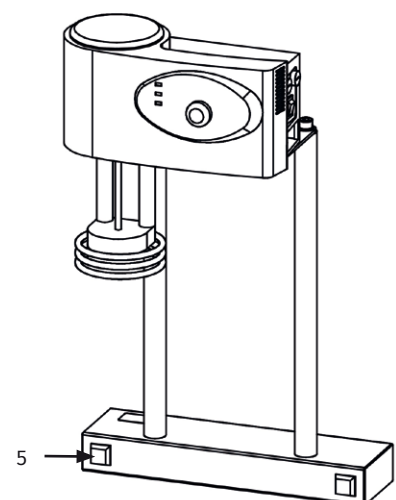
The 1 litre measuring cylinders must be placed in the glass tank (use the weighting ring for a stable set-up). The glass tank is filled with water of a temperature that is kept at as constant a level as possible with the aid of the heating element with thermostat and stirring element (art. nr: 083010).

☞ **The cylinder must be in a vibration-free position.**

☞ **Before measurements can start the samples must be prepared (as is the case when using the pipette apparatus (art. no.: 0816SA).**

3. Preparing samples for particle size analysis

3.1 Principle

Separation of the mineral part of the soil into various size fractions and determination of the proportion of these fractions. 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.
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 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 H_2O_2 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******. The sand is fractionated by dry sieving, the clay and silt fractions are determined by the pipette method or, alternatively, by the hydrometer method.

3.2 Apparatus

Equipment required to prepare the samples:
- Water bath (glass container: included in the set)
- Hot plate (not included)
- End over end shaking machine (not included)
- Sieving machine (e.g. granular composition test set, art. no. 0805)
- Set of sieves, including bottom (diameter 20 cm; included in set 0805)
- Heavy brass funnel (diameter approx. 23 cm) on stand (not included)
- Small 50 μm sieve (diameter 8 cm)
- Glass sedimentation cylinders, marked at 1 litre (included in the set)
- Drying oven (not included)
- Moisture tins (not included)
- Stopwatch (included in the set)

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*   In the ISRIC laboratory carbonates (when present) have always routinely been removed, previously with a Na acetate buffer pH 5 and lately with a 10% acetic acid treatment. The accepted (1995) ISO/DIS (Draft International Standard) 11277 states that removal of carbonate (and oxides) is only done optionally. However, the new Standard 11277 is not accompanied by performance validation data and according to Good Laboratory Practice, a laboratory planning to (drastically) change its procedure should carry out a programme of validation and correlation of the new procedure against the old one, e.g. repeated analysis (min. 10x) of relevant control samples. Therefore, until such validation has been done, the ISRIC laboratory will continue to remove carbonates as a rule, be it with 1 M HCl (see Section 3 4.2). Some other essential steps of Standard 11277 have also been accommodated in the present procedure.

** In the 1995 ISO/DIS 11277 the 50 μm boundary has been changed into a 63 μm boundary. In view of homogeneity of its database (ISIS), ISRIC has decided to postpone introduction of this boundary in soil characterization.

3.3   Reagents

Hydrogen peroxide, 30%.
Dispersing agent: Sodium hexametaphosphate 4% and soda 1% solution ("Calgon" type). Dissolve 40.0 g $(NaPO_3)_6$ and 10.0 g Na_2CO_3 in water in a 1 litre 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).
Calcium chloride solution, 1 *M (= mol/l)* Dissolve 147 g $CaCl_2.2H_2O$ in 1 litre water.

3.4   Procedure

3.4.1   Oxidation of organic matter

1. Weigh out approx. 20 g fine earth into a 1 litre beaker (at carbonate contents exceeding 10% and carbonate is to be removed, weigh out proportionally more soil).
2. Add 15 ml water and 15 ml H_2O_2, 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.
4. The next day, place beaker on water bath (80 °C) and regularly add 5 10 ml increments of H_2O_2 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 1 hour to remove any remaining H_2O_2.
7. Remove beaker from hot place and allow to cool.
8. Centrifuge and decant or, alternatively, allow material to settle in the beaker and siphon off.

   Note: Flocculation may be enhanced by adding 25 ml 1 *M* $CaCl_2$ 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 mS/cm 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-H_2O extract) measure electrical conductivity of supernatant solution.
9. 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 (3.4.4) unless carbonates (3.4.2) and/or iron oxides (3.4.3) are removed.

3.4.2   Removal of carbonate (optional)

3.4.2.1   Reagent

Hydrochloric acid, 1 *M* Add 36 ml conc. HCl to 900 ml water and make to 1 l with water (use fume cupboard!).

3.4.2.2   Procedure

1. To the residue of 3 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 $CaCl_2$ solution). Make up to about 250 ml with water.
2. Place 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:
   1. Omit heating.
   2. Use a 10% acetic acid solution (vol/vol) instead of hydrochloric acid.
   3. Use a 1 M 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 (as is done in some laboratories).

3.4.3   Deferration (optional)

If applied, this treatment is usually done after the other pretreatments prior to dispersion.

3.4.3.1   Reagents

B*uffer solution 0.3 M sodium citrate and 0.1 M sodium bicarbonate*. Dissolve 88 g Na-citrate.2H_2O and 8.4 g NaHCO$_3$ in water and make to 1 litre.
Sodium dithionite (powder).
Sodium chloride solution 1 M. Dissolve 58.5 g NaCI in water and make to 1 litre.

3.4.3.2   Procedure

1. Weigh out approx. 20 g fine earth in a 1 l 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 Fe_2O_3, repeat the procedure once or twice: a brownish or reddish colour of the sample may indicate still incomplete deferration.
7. Wash once more with 250 ml 1 M NaCl when centrifuging, or 500 ml when siphoning.
8. Proceed with 3.4.1 Step 8.

3.4.4   Dispersion

1. Transfer suspension quantitatively to a 1 l 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.

3.4.5   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 litre mark with water. Proceed with this according to 3 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.

3.4.6　Determination of sand fractions

1. Transfer the dried sand of 3.4.5 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 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 µm) transfer this to suspension in sedimentation cylinder mentioned in 3.4.5.

   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.

3.4.7　Determination of silt and clay

3.4.7.1　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.

3.4.7.2　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.)

3.4.7.3　Fraction <50 µm

1 . After adding material <50 µm possibly collected during sieving (see 3.4.6, 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 centre 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).

3.4.7.4　Fraction <20 µm

5. After measuring the temperature of the suspension, again stopper the cylinder and shake well.
6. Place the cylinder on a vibration free table under the pipette assembly.
7. After exactly 5 minutes pipette 20 ml at a depth indicated in Table 3 1.
8. Transfer aliquot to tared moisture tin, evaporate on water bath and dry overnight at 105 °C.
9. 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).

Table 3.1:   Depth (in cm) at which fractions <20 µm and <2 µm are pipetted as a function of the temperature and after indicated settling time.

Temp. °C	5 mins. <20 µm	5½ hrs. <2 µm		Temp. °C	5 mins. <20 µm	5½ hrs. <2 µm
19	10.5	6.9		28	13.0	8.6
20	10.8	7.1		28	13.3	8.8
21	11.0	7.2		30	13.6	9.0
22	11.3	7.4		31	13.9	9.1
23	11.6	7.6		32	14.2	9.3
24	11.9	7.8		33	14.4	9.5
25	12.1	8.0		34	14.8	9.7
26	12.4	8.2		35	15.1	9.9
27	12.7	8.4		36	15.4	10.1

3.4.7.5   Fraction <2 µm

10. After 5½ hours measure temperature in blank cylinder and pipette 20 ml at a depth indicated in Table 3.1.
    <u>Note:</u> If this temperature differs from initial temperature (measured in 3.4.7.4 Step 5), use mean of this and initial temperature.
11. Transfer aliquot to tared moisture tin, evaporate on water bath and dry overnight at 105 °C.
12. 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).

<u>Remark 1</u>: 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.

<u>Remark 2</u>: 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 (3.4.2) is then indicated.

3.5   Calculations

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

Clay	(<2 µm)	=	(H x 50) - (Z x 50)	(wt. K)
Silt	(2-20 µm)	=	(G x 50) - (Z x 50) - K	(wt. L)
Silt	(20-50 µm)	=	(F x 50) - (Z x 50) - K - L	(wt. M)
Sand	(>50 µm)	=	A + B + C + D + E	(wt. N)

Sample weight = K + L + M + N   (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) $\quad$ = $\quad \dfrac{K}{\text{sample wt.}}$ x 100

% silt (2-20 μm) $\quad$ = $\quad \dfrac{L}{\text{sample wt.}}$ x 100

% silt (20-50 μm) $\quad$ = $\quad \dfrac{M}{\text{sample wt.}}$ x 100

% sand (1000-2000 μm) = $\quad \dfrac{A}{\text{sample wt.}}$ x 100

% sand (500-1000 μm)= $\quad \dfrac{B}{\text{sample wt.}}$ x 100

% sand (250-500 μm) $\quad$ = $\quad \dfrac{C}{\text{sample wt.}}$ x 100

% sand (100-250 μm) $\quad$ = $\quad \dfrac{D}{\text{sample wt.}}$ x 100

% sand (50-100 μm) $\quad$ = $\quad \dfrac{E}{\text{sample wt.}}$ x 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:

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

In case deferration was applied the percentage "free iron" should be included between the parentheses.

3.6 Three fractions only (Sand, Silt, Clay)

If only the sand (50-2000 μm), silt (2-50 μm) and clay (<2 μm) fractions are to be determined, the procedure described above is modified as indicated below.

Note: If only the clay fraction is required, then still the silt and sand fractions have to be determined in the present procedure as they are needed for calculating the sample weight. This can be avoided by weighing a precise amount of sample at the outset (in the calculation, if necessary, correct for moisture, organic matter, calcium carbonate and iron oxides).

Down to the last step of Section 3.4.5 no modifications are introduced.

3.6.1 Sand

To Section 3.4.5 (separation of fractions) is added:
4. Weigh sand fraction (net weight **N**, total sand).
   Note: This is the same *N* as obtained by summation of the sand subfractions *A* through *E* of Section 3.4.6.

3.6.2   Silt

For this, proceed as indicated in Section 3.4.7.3 and determine weight **F**. Omit the subsequent Section 3.4.7.4 (fraction <20 μm) but observe instructions of Sections 3.4.7.1 and 2.

3.6.3   Clay

No change, proceed as indicated in Section 3 4.7.5 and determine weight **H.**

3.6.4   Calculations

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

Clay	(< 2 μm)	=	(H x 50) - (Z x 50)	(wt. K)
Silt	(2-50 μm)	=	(F x 50) - (Z x 50) – K	(wt. P)
Sand	(>50 μm)	=	weighed	(wt. N)

Sample weight = K + P + N   (all weights in gram)

where
F = weight 20 ml pipette aliquot of fraction <50 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:

$$\% \text{ clay } (<2 \text{ μm}) = \frac{K}{\text{sample wt.}} \times 100$$

$$\% \text{ silt } (2\text{-}50 \text{ μm}) = \frac{P}{\text{sample wt.}} \times 100$$

$$\% \text{ clay } (50\text{-}2000 \text{ μm}) = \frac{N}{\text{sample wt.}} \times 100$$

<u>Note</u>: The note added to the calculations of Section 3.5 applies here too.

3.7   Fine clay (<0.2 μm)

3.7.1   Principle

Because of the low settling velocity of these small particles, sedimentation in cylinders is not suitable for the determination of this fraction. This is overcome by using a centrifuge to increase the gravity force.

3.7.2   Apparatus

Centrifuge (preferably with refrigeration).

3.7.3   Procedure

1. After pipetting the fraction <2 μm (3.4.7.5, Step 10), stopper in the cylinder and shake well.
2. Allow to stand for an hour and transfer about 200 ml suspension to a 250 ml centrifuge bottle.
   Measure temperature of the suspension. During spinning, the distance between surface of suspension and centre of centrifuge should be 16 cm.

3. Spin at 1800 rpm during the time indicated in Table 3.2 (excluding starting and stopping).

   Note: Spinning at 2500 rpm reduces the time needed. In this case plastic centrifuge bottles should be used. Before spinning a next batch, allow centrifuge to cool for at least an hour or use centrifuge with refrigeration. To gauge temperature increase during spinning, spin a blank batch (water) prior to spinning suspensions. A mean temperature can then be used.

4. Stop centrifuge without using the brake.
5. Gently remove bottles from centrifuge and place under pipette.
6. Pipette 20 ml aliquot at 4.5 cm depth. Measure temperature of suspension.
7. Transfer aliquot to tared moisture tin, evaporate on water bath and dry overnight at 105 °C.
8. Remove tin from drying oven, close with lid and cool in desiccator, weigh with 0.001 g accuracy (net weight **Q**)

Table 3.2: Centrifuge speed and spinning time in minutes as function of the temperature for determination of the fine clay fraction < 0.2 µm.

Temp. °C	1800 rpm	2500 rpm		Temp. °C	1800 rpm	2500 rpm		Temp. °C	1800 rpm	2500 rpm
20	32.0	16.5		27	27.0	14.0		34	23.0	12.0
21	31.0	16.1		28	26.5	13.5		35	22.5	11.8
22	30.0	15.7		29	26.0	13.3		36	22.0	11.5
23	29.5	15.3		30	25.0	13.0		37	22.0	11.3
24	29.0	15.0		31	24.5	12.8		38	21.5	11.1
25	29.0	14.6		32	24.0	12.5		39	21.0	10.9
26	27.5	14.2		33	23.5	12.3		40	20.5	10.6

3.7.4   Calculation

% fine clay (< 0.2 µm)   = $\dfrac{(Q-Z) \times 50}{\text{sample wt.}}$ x100

where
Q = 20 ml aliquot weight of fraction < 0.2 µm
Z = 20 ml aliquot weight of blank (see Section 3.4)
sample wt. is here the same as in Section 3.5 or 3.6.

3.8   Water dispersable clay (or: "natural clay")

3.8.1   Principle

This is the clay content found when the sample is dispersed with water without any pretreatment to remove cementing compounds and without use of a dispersing agent. The proportion of natural clay to total clay is used as a structure stability indicator.

3.8.2   Procedure

1. Weigh about 10 g fine earth (accuracy 0.01 g) into a 1 l polythene bottle.
2. Add 400 ml water and shake overnight in an end over end shaker at about 30 rpm.
3. Transfer to a 1 l sedimentation cylinder and make to the mark with water.
4. Pipette a 20 ml aliquot after 5½ hours at a depth indicated by Table 3.1.
5. Transfer aliquot to tared moisture tin, evaporate on water bath and dry overnight at 105 °C.
6. Remove tin from drying oven, close with lid and cool in desiccator. Weigh with 0.001 g accuracy (net weight **R** g).

3.8.3   Calculation

% water dispersable clay   = $\dfrac{50 \times R}{s}$ x 100 x mcf

where
R = 20 ml aliquot weight of suspension
s = air dry sample weight in gram
mcf = moisture correction factor

A parameter derived from the water dispersable clay is the Index of Structure" ranging from 0 to 100:

Index of structure   =   100 x (1- $\dfrac{\text{% water dispersable clay}}{\text{% total clay}}$)

where *total clay* is the clay content found when pretreatment and dispersing agent are applied (i.e. *% clay* in Section 3.5 or 3.6).

References:
Day, in: Black (1965), p. 545
Gee and Bauder, in: Klute (1986) p. 383
Jackson (1969)
SNLCS, EMBRAPA, 1979 (Natural clay, their method 1.17)
Sombroek (1966, p. 122: Index of Structure)
USDA, SCS (1972, 1982)

4.   Hydrometer method

4.1   Principle

The clay and silt fractions in particle size analysis can conveniently be determined with a hydrometer instead of with the pipette method. It is basically a measurement of the density of the suspension which is a function of the concentration and kind of particles present (after a certain time of settling). The pretreatment of the soil is the same as described for the pipette method. After shaking with the dispersing agent, sand is separated from clay and silt with a 50 µm sieve. The sand is fractionated by dry sieving, the clay and silt fractions are determined by hydrometer readings.

4.2   Requisites

Standard hydrometer, ASTM no. 152H, with Bouyoucos scale in g/l (art. no.: 08.30.01.01)
Stopwatch
Amyl alcohol

4.3   Procedure

1. The suspension <50 µm obtained in 3.4.5 Step 2 is used. (Also use the blank described there: water containing 1 g/l dispersing agent).
2. Allow time for the suspension in the sedimentation cylinder to equilibrate thermally and record temperature.
3. Close the sedimentation cylinder with a rubber stopper and shake well. Add a drop of amyl alcohol if the surface of the suspension is covered with foam. As soon as mixing is completed, carefully lower the hydrometer into the suspension and take a reading when the hydrometer is stable but not later than 50 seconds after completion of the mixing.
4. Remove the hydrometer, rinse, and wipe it dry.
5. Reinsert the hydrometer carefully about 10 seconds before each reading and take readings at 5, 120 and 960 or 1440 minutes. (Readings at other times are possible.)

6. Remove and clean the hydrometer after each reading.
7. Record the reading **R** each time.
8. Place hydrometer in the blank solution, and record the blank reading as **R_{bl}**, and the temperature each time.

4.4 Calculation

1. Determine the *concentration of soil in suspension*, **C** in g/l, by

$$C = R - R_{bl}$$

where
   R = uncorrected hydrometer reading in g/l
   R_{bl} = hydrometer reading of the blank solution.

2. Determine the *summation percentage* **P** for the taken time interval, i.e. the weight percentage of all particles still present at the depth of measurement after the time of settling, by

$$P = \frac{C}{C_0} \times 100$$

where
   $C_0 = C_{50\ sec}$ + total weight sand fractions.

(Note that *C_0 = weight total sample*, *$C_{50\ sec}$ = weight silt + clay fractions*, and that *total weight sand fractions* is weight **N** as calculated in Section 3.5).

3. Determine **X** (*mean particle diameter in μm*) in suspension at time *t*, using:

$$X = 1000 \sqrt{(Bh')} / \sqrt{t}$$

where:
   B = $30\eta / \{g(\rho_s - \rho_1)\}$ (see Table 3.3)
   h' = $-0.164R + 16.3$

and with the terms expressed in the following units:
   h' = effective hydrometer depth, cm
   η = fluid viscosity, poise (= 100 mPa.s)
   g = gravitational constant, 985 cm/s²
   ρ_s = soil particle density, 2.60 g/cm³
   ρ_1 = solution density, g/cm³
   t = time, minutes

      Solution density (g/cm³): $\rho_1 = \rho^0 (1 + 0.630\ Cs)$
                  where ρ^0 = water density in g/cm³ at temperature t
                            Cs = concentration dispersing agent in g/cm³

      Viscosity (cp): $\eta = \eta° (1 + 4.25\ Cs)$
                  where $\eta°$ = viscosity water in centipoise (mPa.s or g.m^{-1}.s) at temperature t

Table 3 3:   The factor B calculated as a function of the temperature.

Temp. °C	B x10^{-4}		Temp. °C	B x10^{-4}		Temp. °C	B x10^{-4}
19	1.90		27	1.57		35	1.32
20	1.85		28	1.54		36	1.30
21	1.81		29	1.50		37	1.27
22	1.76		30	1.47		38	1.25
23	1.72		31	1.44		39	1.22
24	1.68		32	1.41		40	1.20
25	1.64		33	1.38			
26	1.60		34	1.35			

4. Plot a *summation percentage curve* (**P vs. X**; use log scale for X) using the hydrometer readings and the sieve data. From this curve derive silt and clay percentages (and total sand by subtraction from 100%). An example of this procedure is given next in Section 3.8.5.

References:

Gee and Bauder, in: Klute (1986) p. 383
CRC Handbook of Chemistry and Physics, e.g. 69th ed. (1988 1989) or later.

4.5   Calculation example

Temperature   =   220 °C
Blank reading R_{bl} =   2.0
Weight total sand   =   4.50 g

	50 sec.	5 min.	120 min.	1440 min.
Readings (R)	16.0	13.0	6.2	5.6
C (=R-R_{bl})	14.0	11.0	4.2	3.6

Then: total sample weight C_0 = $C_{50\,sec}$ + weight sand = 14.0 + 4.5 = 18.5

And: Calculation of X (particle diameter):

Time (min.)	h'	X
0.83	(−0.164 x 16.0) + 16.3 = 13.68	1000 x √ (1.76 x 10^{-4} x 13.68) / √ 0.83 = 54.0 µm
5	(−0.164 x 13.0) + 16.3 = 14.17	1000 x √ (1.76 x 10^{-4} x 14.17) / √ 5 = 22.3 µm
120	(−0.164 x 6.2) + 16.3 = 15.28	1000 x √ (1.76 x 10^{-4} x 15.28) / √ 120 = 4.7 µm
1440	(−0.164 x 5.6) + 16.3 = 15.38	1000 x √ (1.76 x 10^{-4} x 15.38) / √ 1440 = 1.4 µm

Then: Calculation of **P** (summation percentage) presented with corresponding X

	50 sec.	5 min.	120 min.	1440 min.
P $(= \dfrac{C \times 100}{18.5})$	75.7	59.5	22.7	19.5
X (µm)	54.0	22.3	4.7	1.4

These results have been plotted in the figure below (squares □). Note that for the particle size at 50 seconds (0.83 min.) not 54 µm should be taken but 50 µm since particles >50 µm are not present in the suspension. (This means that in practice the first reading can be taken up to just under 1 minute after mixing.)

In this graph, determine the summation percentages of the fractions wanted, usually 2 µm, 20 µm and 50 µm (indicated by circles ○). These are:

<2 µm: 20.5%,   <20 µm: 57.0%,   <50 µm: 75.7%

Remark: Obviously, the decimals cannot be read accurately and also the interpolation between the determined points by straight lines gives rise to some uncertainty. Therefore, the final results should be rounded off to whole figures.

Hence, the contents of the various fractions are (in % of the total sample):

```
clay   (<2 µm)         = 20.5%                = 21%
silt   (2-20 µm)       = 57.0 - 20.5    = 36.5% = 37%
silt   (2-50 µm)       = 75.7 - 20.5    = 55.2% = 55%
silt   (20-50 µm)      = 75.7 - 57.0    = 18.7% = 19%
sand   (50-2000 µm)    = 100  - 75.7    = 24.3% = 24%
```


ISRIC

Sample