

NEW ZEALAND STANDARD

METHODS OF TESTING SOILS
FOR CIVIL ENGINEERING
PURPOSES

Part 2

Soil classification tests

2.8

DETERMINATION OF THE PARTICLE-SIZE DISTRIBUTION

TEST 2.8.3

Standard method for fine soils (Pipette method)

2.8.3.1

Scope

This method covers the quantitative determination of the particle-size distribution in a soil from the coarse sand size down (see Note (1)). This test is not normally carried out if less than 10 % of the material passes the 63 μm test sieve, as measured in Test 2.8.1.

2.8.3.2

Related documents

The provisions of Part 1 of this Standard are applicable to, and shall be read in conjunction with this method of test. Reference is made to Tests 2.1, 2.8.1, 3.3.1 and 3.3.2 of this Standard.

2.8.3.3

Apparatus

- (a) A sampling pipette of the type illustrated in fig. 2.8.2 fitted with a pressure and suction inlet, and having a nominal capacity of 10 ml. The pipette shall be so arranged that it can be inserted to a fixed depth into a sedimentation tube (see 2.8.3.3(b)) when the latter is immersed in a constant temperature bath (see fig. 2.8.3).
- (b) Two glass sedimentation tubes, of nominal diameter 50 mm and 350 $\pm$ 25 mm long graduated to 500 ml volume, with rubber bungs to fit.
- (c) Nine glass weighing bottles, 25 to 30 ml capacity fitted with ground glass stoppers. The masses of the bottles shall be known to the nearest 0.001 g.
- (d) A constant temperature bath capable of being maintained at 25 $\pm$ 0.1 $^{\circ}\text{C}$ into which the sedimentation tube can be immersed up to the 500 ml mark. The bath must not vibrate the sample.

- (e) A water bath capable of being maintained at 100 $^{\circ}\text{C}$ for heating solutions and suspensions.
- (f) A mechanical shaker capable of keeping 20 g of soil and 150 ml of water continuously in suspension. (The type of shaker should be one that gives a gentle mixing action, for example an end-over-end shaker or a vibratory shaker).
- (g) A flat glass or other smooth non-absorbent mixing plate (a convenient size is 10 mm thick and 500 mm square).
- (h) Two palette knives with blades about 100 mm long and 10 mm wide.
- (j) Test sieves 2.00 mm, 600 μm , 212 μm , 63 μm and a receiver.
- (k) A hand lens of $\times 10$ power.
- (m) A balance readable and accurate to 0.001 g.
- (n) A drying-oven complying with the requirements of 1.4.2 of Part 1.
- (o) A timing device readable and accurate to 1 s.
- (p) A desiccator (a convenient size is 200 mm to 250 mm in diameter) containing anhydrous silica gel.
- (q) A 650 ml or 1 litre tall-form beaker and a cover glass to fit, and a smaller beaker.
- (r) A source of vacuum, for example a filter pump.
- (s) A Buchner or Hirsch funnel of nominal diameter 70 mm hardened filter papers to fit, for example Whatman No. 542, a cover glass and a 1 litre filter flask.
- (t) A shaking bottle, for example, a 300 ml cream bottle and rubber stopper to fit.
- (u) A 100 ml measuring cylinder and a 25 ml pipette.
- (v) A glass filter funnel of nominal diameter 100 mm.
- (w) A wash bottle, preferably plastics, containing distilled water.

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- (x) A suitable stirrer such as a length of glass rod 150 mm to 200 mm long and 4 mm to 5 mm in diameter fitted at one end with a rubber policeman.
- (y) A length of suitable tubing to connect the vacuum source to the filter flask.
- (z) A means of measuring pH (see Tests 3.3.1 and 3.3.2).

2.8.3.4

Reagents

Use reagents of recognized analytical reagent quality:

- (a) *Hydrogen peroxide*. A 20 volume solution (see Note (2)).
- (b) *Amyl alcohol*. Commercial grade (optional).
- (c) *Sodium hexametaphosphate solution*. Dissolve 66 g of sodium hexametaphosphate and 14 g of sodium carbonate in distilled water to make 1 litre of solution.

NOTE – This solution is unstable; prepare a fresh solution approximately once a month and record date of preparation on the bottle (see Note (3)).

2.8.3.5

Procedure

2.8.3.5.1

Calibration of sampling pipette

- (a) Thoroughly clean and drain the sampling pipette and immerse the nozzle in distilled water at room temperature. Close the tap B and open tap E to the pipette (see fig. 2.8.3).
- (b) By means of a rubber tube attached to C, suck water up into the pipette until it rises above E. Close the tap E and remove the pipette from the water. Pour off surplus water drawn up into cavity D by turning tap E to discharge through F into the small beaker.
- (c) Discharge the water contained in the pipette and tap E into a glass weighing bottle of known mass and determine the mass of water. From this mass calculate the internal volume (V_p ml) of the pipette and the tap to the nearest 0.02 ml. Make 3 determinations of the volume and if the range of values is less than 0.05 ml take the average, otherwise repeat the calibration.

2.8.3.5.2

Pretreatment of soil

- (a) Take the sub-sample obtained in accordance with the method specified in 1.6.7 of Part 1 or 2.8.1.5 of Test 2.8.1 (see Notes (4) and (5)) and sieve through the 2.00 mm test sieve suitably protected by larger sieves if necessary. The sample must be sufficient to yield equivalent to 25 g to 50 g of oven-dry mass passing the 63 μ m test sieve.
- (b) Clean any particles retained on the sieve by brushing or washing until the individual particles are clean of any finer material, taking

care especially with weak materials to ensure that the treatment is not removing parts of larger particles.

- (c) Discard the material retained (see Note (4)) and transfer the material passing the 2.00 mm test sieve together with any washings into the tall beaker. Adjust the volume of the soil suspension to about 100 ml by evaporation or by adding distilled water.
- (d) Add 50 ml of 20 volume hydrogen peroxide (see Note (2)) to the suspension at room temperature, and stir. Allow the reaction to proceed for about 30 min, stirring from time to time to reduce frothing and to break down aggregates.
- (e) Warm the beaker on the boiling water bath. The reaction may become vigorous so that frequent stirring, temporary removal from the bath, or the use of a few drops of amyl alcohol may be required to reduce excessive frothing. Leave the beaker on the water bath until the reaction has subsided, when the suspension may safely be boiled for a few minutes. When the organic matter has been oxidized to a sufficient degree, the soil is usually a pale grey or buff colour. Some organic soils may require additions of further increments of hydrogen peroxide (see Note (6)) repeating steps (d) and (e).
- (f) Assemble the Buchner or Hirsch funnel and filter flask and connect the flask to the source of vacuum (see Note (7)). Wet a hardened filter paper with distilled water, place it on the funnel ensuring that it seals properly and apply the vacuum.
- (g) Slowly pour the contents of the beaker into the funnel. Wash the beaker with a jet of distilled water and scrub its sides with the rubber policeman. Make sure that all the soil is transferred into the funnel.
- (h) Maintain the soil surface wet by adding further distilled water to the funnel as required. Except when adding water, keep a cover glass on the funnel.
- (j) Continue until nearly 1 litre of filtrate has been collected, finally allowing all free water in the funnel to be drawn through the soil, but do not allow the surface of the soil to dry. Release the vacuum and remove the funnel from the flask.
- (k) Carefully remove the filter paper together with all the soil from the funnel and place on the mixing plate. Scrape the filter paper gently with a palette knife and finally wash the paper with a jet of distilled water using a minimum of water.
- (m) Thoroughly mix the soil to a uniform consistency, taking care not to grind coarser particles. Working quickly, transfer sufficient to contain about 10 g oven-dry mass of material passing the 63 μ m test sieve to a weighed shaking bottle for dispersion and weigh to 0.01 g. Calculate by difference the wet mass of soil taken

DETERMINATION OF THE PARTICLE-SIZE DISTRIBUTION

Standard method for fine soils (Pipette method)

for dispersion (M_w). Use all the remaining material to make two determinations of water content as specified in Test 2.1.

2.8.3.5.3

Dispersion of soil

(see Note 8):

- (a) Add about 100 ml of distilled water to the shaking bottle, stopper and shake the bottle vigorously until all the soil has been brought into suspension. Using the pipette, add 25 ml of the sodium hexametaphosphate solution. Place the bottle on the mechanical shaking device for at least 4 h (when convenient the suspension may be left shaking overnight (see Note (9))).
- (b) Transfer the suspension to the 63 μm test sieve placed on the receiver and wash the soil on the sieve using *only* a jet of distilled water until all the fine material has passed the sieve and been collected. Do not use more than about 150 ml of distilled water during this operation. Inspect the material retained on the sieve with the hand lens to check that it contains only clean sand grains (see Note (10)). Transfer all the suspension passing the sieve to the sedimentation tube using the glass funnel and a jet of distilled water and make the volume up to 500 ml with distilled water. Shake vigorously until a uniform suspension is formed. Measure the pH of the suspension and record (see Note (3)).
- (c) Transfer the material retained on the 63 μm test sieve to an evaporating dish and dry in the oven at 105 °C to 110 °C. After drying, re-sieve this material on the 600 μm , 212 μm and 63 μm test sieves (see Note (11)). Transfer the material retained on the 600 μm , 212 μm and 63 μm sieves after the second sieving to glass weighing bottles of known mass, and weigh to 0.001 g. Record the mass of dry material retained on these sieves as the mass of coarse, medium and fine sand in the sample (M_{cs} , M_{ms} , M_{fs}). Add the material passing the 63 μm sieve to the suspension.

2.8.3.5.4

Sedimentation:

- (a) Add 25 ml of the sodium hexametaphosphate solution from a pipette to a graduated 500 ml sedimentation tube and dilute with distilled water to 500 ml. Agitate this solution thoroughly and transfer this reference tube together with the sedimentation tube containing the soil suspension to the constant temperature bath, and insert the rubber bungs. Allow them to stand immersed in water up to the 500 ml graduation mark for at least 1 h or until they have reached the temperature of the bath. Inspect for signs of flocculation and again measure the pH of the suspension and record (see Note (3)). When they have

attained the bath temperature, take the tube containing the soil suspension, thoroughly shake until a uniform suspension is formed, and finally invert end-over-end several times and immediately replace in the bath.

- (b) At the same instant as the tube containing the soil suspension is replaced in the bath, start the timing device. Remove the rubber bungs carefully. Carry out the above sampling procedure at the 3 times shown in table 2.8.2, corresponding to the appropriate solid density of the silt and clay particles. Begin the sampling operation at the elapsed times shown from the time the suspension was replaced in the bath after shaking (see Notes (12) and (13)).
- (c) About 15 s before the sample is due to be taken, lower the pipette, with the tap E closed, vertically into the soil suspension until the end is 100 ± 1 mm below the surface of the suspension. Take approximately 10 s to complete this operation and do not cause any turbulence in the suspension. Open the tap E and draw up a sample (V_p ml) into the pipette. Fill the pipette and bore in the tap E with the suspension and close tap E. Take approximately 10 s to complete this sampling operation. Withdraw the pipette from the suspension, taking approximately 10 s to complete the operation. During the sampling a small amount of the suspension may have been drawn up into the bulb D above the bore of the tap E. Wash this surplus through F into the small beaker. Allow distilled water to run from the bulb funnel A into D and out through F until no suspension remains in the system.
- (d) Place a tared weighing bottle under the end of the pipette and open the tap E so that the contents of the pipette are delivered into the bottle. Wash any suspension left on the inner walls of the pipette into the weighing bottle by allowing distilled water from the bulb A to run through B, D and E into the pipette. Place the weighing bottle and contents in the oven maintained at 105 °C to 110 °C and evaporate the sample to dryness. After cooling in a desiccator weigh the weighing bottle and contents to 0.001 g and determine the mass of solid material in the sample (M_1 , M_2 and M_3 for each respective sampling time).
- (e) Between any of the times in which the above sampling is taking place take a sample (V_p ml) from the reference tube containing the sodium hexametaphosphate solution. Take this sample of the solution as in (c) and (d) above except that there is no need to time the operation of sampling the solution, and the depth of sampling is also unimportant. Determine the mass of solid material in the sample of sodium hexametaphosphate solution (M_4).

2.8.3.6

Calculations

(see Form 2.8.3).

2.8.3.6.1

Dry mass

Calculate the dry mass of the pretreated soil (M) from the formula:

$$M = \frac{100M_w}{100 + w} \dots\dots\dots \text{g}$$

where M_w = wet mass of the pretreated soil (g)
 w = water content of the pretreated soil (%)

2.8.3.6.2

Fine sieving

(see Note (14)):

- (a) Calculate the percentage of coarse sand in the pretreated sample from the formula:

$$\text{Percentage coarse sand (2.0 mm to 0.6 mm)} = \frac{100 M_{CS}}{M} \dots\dots\dots \%$$

- (b) Calculate the percentage of medium sand in the pretreated sample from the formula:

$$\text{Percentage medium sand (0.6 mm to 0.2 mm)} = \frac{100 M_{ms}}{M} \dots\dots\dots \%$$

- (c) Calculate the percentage of fine sand in the pretreated sample from the formula:

$$\text{Percentage fine sand (0.2 mm to 0.06 mm)} = \frac{100 M_{fs}}{M} \dots\dots\dots \%$$

2.8.3.6.3

Sedimentation

- (a) Calculate the volume proportion of the original 500 ml suspension taken at each sampling from the formula:

$$S = \frac{500}{V_p}$$

where V_p = calibrated volume of the pipette (ml)

- (b) Calculate the percentage of medium silt in the pretreated sample from the formula:

$$\text{Medium silt (0.02 mm to 0.006 mm)} = \frac{S (M_1 - M_2)}{M} \times 100 \dots\dots\dots \%$$

- (c) Calculate the percentage of fine silt in the pretreated sample from the formula:

$$\text{Fine silt (0.006 mm to 0.002 mm)} = \frac{S (M_2 - M_3)}{M} \times 100 \dots\dots\dots \%$$

- (d) Calculate the percentage of clay in the pretreated sample from the formula:

$$\text{Clay (less than 0.002 mm)} = \frac{S (M_3 - M_4)}{M} \times 100 \dots\dots\dots \%$$

- (e) Calculate the percentage of coarse silt in the pretreated sample from the formula:

Coarse silt (0.06 mm to 0.02 mm) =

$$100 \frac{M - (M_{CS} + M_{ms} + M_{fs} + S (M_1 - M_4))}{M} \dots\dots \%$$

2.8.3.7

Reporting of results

2.8.3.7.1

Report the following values:

- (a) Results in the form of a table showing to the nearest 1 % the percentage by mass of each of the size fractions defined above.
(b) If required, the results on a semi-logarithmic chart of the type shown in fig. 2.8.1 of Test 2.8.1 (see Note (15)).

2.8.3.7.2

State the history of the sample, for example natural state, air-dried, oven-dried or unknown.

2.8.3.7.3

State the pH of the suspension and if other than sodium hexametaphosphate were used, state the dispersant.

2.8.3.7.4

State that the pipette method was used.

2.8.3.7.5

State that the result was obtained in accordance with this Standard Test Method.

NOTES ON TEST 2.8.3

- (1) If the sample has been wet sieved down to the 63 μm test sieve size, then only that material passing the 63 μm sieve need be used to carry out this test, thus eliminating the necessity of repeating the determinations of the same fractions.
(2) If the organic content of the soil is very low there is no need for peroxide pretreatment.

Hydrogen peroxide is a fairly efficient oxidant and 20 volume has been specified as a reasonably stable, effective, and safe concentration. Even at this concentration it should not be allowed to come in contact with clothes or skin. Any accidental contact should be washed off reasonably quickly. Sometimes a higher concentration such as 100 volume may be used with advantage but greater care is required in handling and of course quantities must be reduced pro rata.

- (3) *Dispersing agent.* Sodium hexametaphosphate has been specified as the dispersing agent in view of the success with which it has been employed in the analysis of a large number of soils. However, it has been found to be ineffective when dealing with some soils derived from volcanic ash or rock and also certain highly aggregated soils, especially those of tropical origin. When using sodium hexametaphosphate, it is important to maintain the pH of the soil suspension reasonably high, for example, above 8.0. If after mechanical shaking the pH is below this level, a further 25 ml increment of sodium hexametaphosphate solution should be added and the suspension again shaken. If the pH still falls below this level, it may be maintained with a small amount of sodium hydroxide. However, there will still be occasions when partial or complete flocculation per-

sists as shown by large crumbs or "flocs" of soil settling out rapidly and perhaps leaving a sharply defined relatively clear layer above the suspension. The sediment of a well dispersed soil remains firm at the bottom of the sedimentation tube when tilted but flocculated material flows to maintain a horizontal line. Under these conditions some other method of pretreatment or dispersion or both shall be tried*. Of course if any modification to the standard amount or type of dispersing agent is made, similar changes shall be made to the reference solution (see 2.8.3.5.4 (a)).

- (4) In this method it has been assumed that a sample obtained in accordance with the method specified in 1.6.7 of Part 1 instead of 2.8.1.5 of Test 2.8.1 will contain no significant material retained on the 2.00 mm test sieve. If there is such material, but insufficient to justify Test 2.8.1, it shall be dried after the initial separation, weighed, and allowed for in the final calculations. With certain volcanic and tropical soils it may be difficult to remove all the finer fractions from the material retained on the 2.00 mm sieve. In such cases it may be necessary to use a dispersing agent as in Test 2.8.1 before wet sieving on the 2.00 mm sieve.
- (5) *Size of sedimentation sample.* Stokes' Law is derived for a single sphere falling in a large body of liquid. It is not applicable to a highly concentrated suspension when the particles are close enough to influence each other. It has been established that if the mass of solid matter in the suspension is not too large, the deviation from Stokes' Law is negligible.
- (6) A rough guide to the amount of hydrogen peroxide which may be required is 5 ml to 10 ml of 20 volume hydrogen peroxide per 10 g of dry soil per 1 % organic matter.
- (7) Filtration has been specified as the means of washing and recovering the soil after pretreatment since the equipment is widely available. Centrifuging which is a very rapid and convenient method is an acceptable alternative.
- (8) Extreme care is needed with the procedure from this point on as any loss of material is recorded in the coarse silt fraction, this fraction being obtained by difference.
- (9) Other means of mechanical dispersion may be used provided they are as effective as 4 h end-over-end shaking without causing undue degradation of coarser particles.
- (10) If any aggregates are found, they must be broken down and dispersed, but not on the sieve. Transfer the material retained on the sieve to a mortar with a little distilled water, and gently rub with a rubber covered pestle, taking care not to break down individual grains. After re-sieving, the further material passing must be added to the suspension.

(11) *Wet sieving of sand.* In the wet sieving operation for the separation of sand particles, grains with diameters just slightly smaller than the apertures of the 63 μ m test sieve tend to be retained due to the surface tension of the water held between the sieve meshes. The absence of such particles from the suspension should be of no consequence to the sedimentation analysis since, by definition, they will fall into the grade of coarse silt, which is the fraction determined by difference. However, as a precaution the material passing the 63 μ m sieve should be added to the suspension. In order that the various percentages of the sand fractions can be accurately determined, it is necessary to dry the material retained on the 63 μ m sieve, and re-sieve it. The particles retained on the sieve after this second operation are subsequently classified as "coarse", "medium" and "fine" sand. The effect of surface tension can be lessened by placing the sieve in a tray of water so that the wire mesh and the soil particles are completely immersed.

(12) *Sedimentation time and sampling depth.* In order to define the shape of the cumulative curve, at least 3 samples shall be taken at appropriate sedimentation times. The times quoted in 2.8.3.5.4 (b) correspond to equivalent particle diameters of 0.02 mm, 0.006 mm and 0.002 mm. It is not necessary to measure the solid density of soil particles for every sample used for fine analysis. An average value for samples from the same area or soil type in an area may be used without loss of accuracy. It is essential that the solid density used is that of the silt and clay fraction and not that of the whole sample of soil.

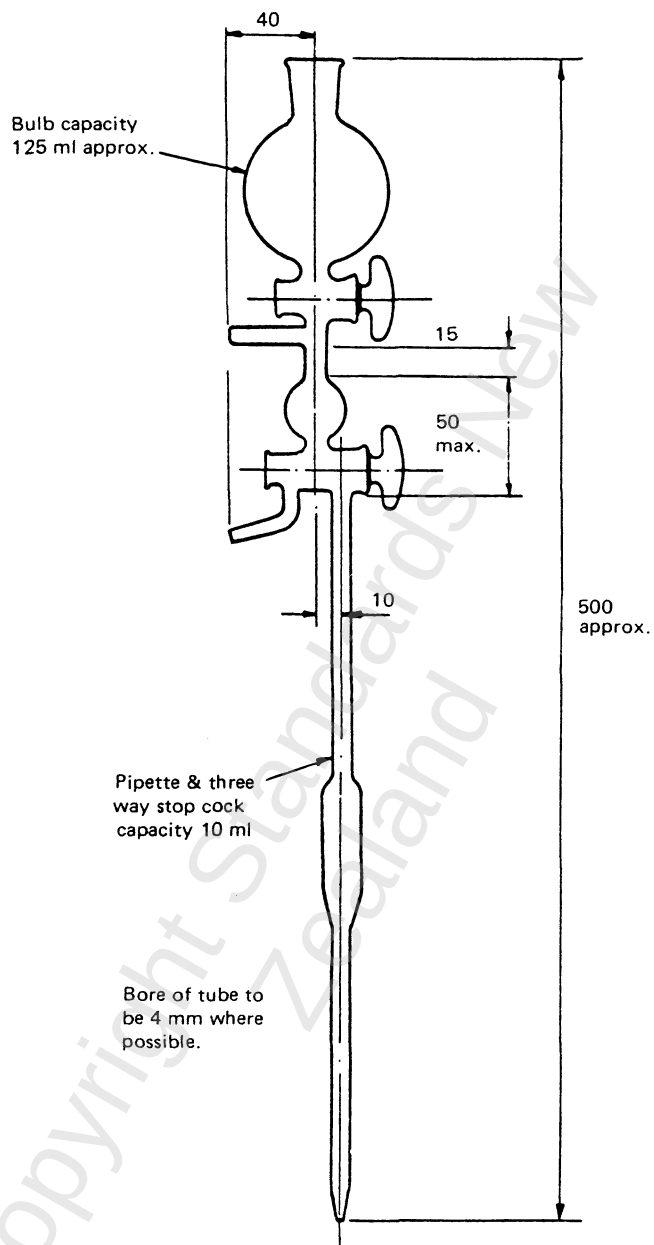
(13) If it is more convenient, the sedimentation tube can be shaken up again after each sample is taken, provided that the timing device is re-started at the instant the tube is replaced in the bath. The time of taking the next sample is then reckoned from this instant.

(14) The sieves used do not relate exactly to the sizes of the sand fractions but the differences are negligible for all practical purposes.

(15) If a coarse analysis has been carried out on the sample, the complete analysis should be shown on the chart. Allowance should be made for material greater than the 2.00 mm test sieve size in the calculation of the percentages.

* Birrell, K.S. *Determination of clay contents in soils containing allophane.* New Zealand Journal of Agricultural Research, 9 (3), 554-64 (1966), 17 (3), 333-8 (1974).

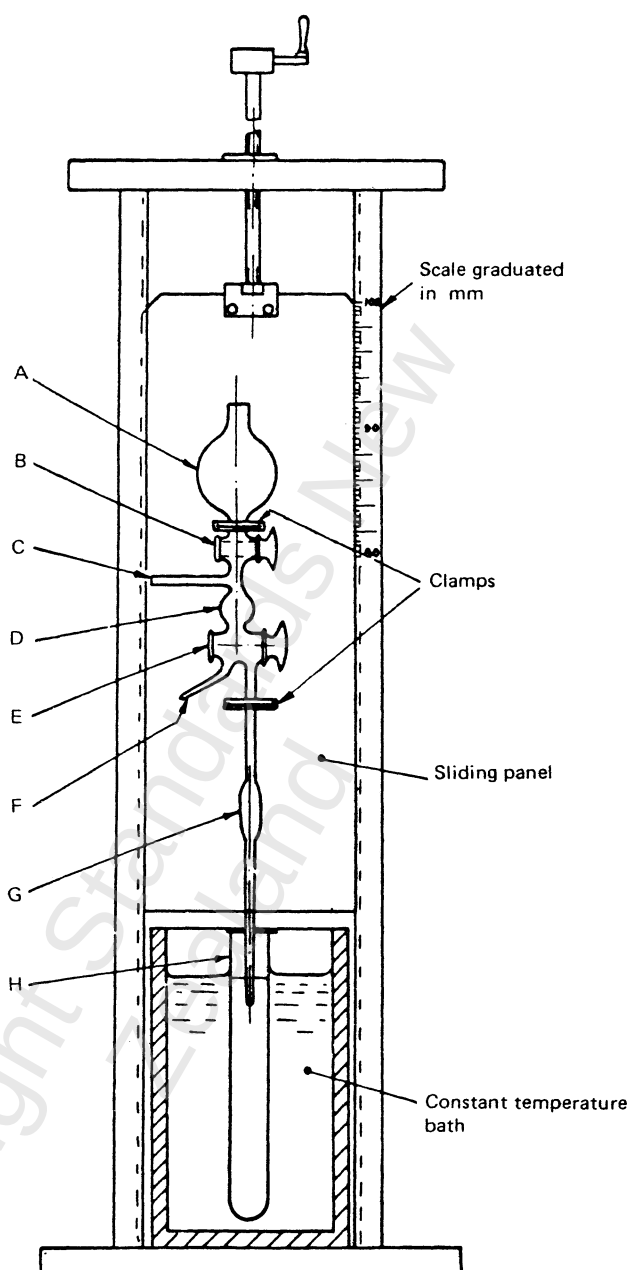
Wintermeyer, A.M., and Kinter, E.B. *Dispersing agents for particle-size analysis of soils.* Highway Research Board Bulletin 95 (1955) (National Research Council Division, Engineering and Industrial Research, Washington, D.C.), 1-14.



This design has been found satisfactory, but alternative designs may be employed provided that the essential requirements are fulfilled.

Fig. 2.8.2
SAMPLING PIPETTE FOR SEDIMENTATION APPARATUS

All dimensions in millimetres.



This design has been found satisfactory, but alternative designs may be employed provided the essential requirements are fulfilled.

Fig. 2.8.3
AN ARRANGEMENT FOR LOWERING THE SAMPLING PIPETTE INTO THE SOIL SUSPENSION

Table 2.8.2
TIMETABLE FOR SAMPLING SOIL SUSPENSION

Solid density of soil particles (t/m^3) of silt and clay fractions	Elapsed times after shaking for starting sampling				
	1st sample		2nd sample	3rd sample	
	min	s	min	h	min
2.50	4	35	50.5	7	36
2.55	4	25	49	7	21
2.60	4	15	47.5	7	07
2.65	4	5	46	6	54
2.70	4	0	44.5	6	42
2.75	3	55	43.5	6	30
2.80	3	50	42	6	20
2.85	3	40	41	6	10
2.90	3	35	40	6	0
2.95	3	30	39	5	50
3.00	3	25	38	5	41
3.05	3	20	37	5	32
3.10	3	15	36	5	24
3.15	3	10	35	5	17
3.20	3	5	34.5	5	10
Column 1	2		3	4	

DETERMINATION OF THE PARTICLE-SIZE DISTRIBUTION **Standard method for fine soils (Pipette method)**

NZS 4402 : 1986
Test 2.8.3

Form 2.8.3

DETERMINATION OF THE PARTICLE-SIZE DISTRIBUTION – PIPETTE METHOD **(Test 2.8.3)**

Job: _____ Sample no.: _____
 Location: _____ Tested by: _____
 Depth: _____ Date: _____
 Test details: * _____ Checked by: _____
 Test made on fraction passing test sieve. _____ Date: _____
 History: Natural/air-dried/oven-dried/unknown. _____ Volume of pipette ml
 Solid density of soil particles in silt and clay _____ Volume proportion of each sampling
 fraction t/m³ measured/assumed. _____
 Dispersant used: Sodium hexametaphosphate/
 other (specify) $S = \frac{500}{V_p} = \dots\dots\dots$

Determination of water content of wet soil for dispersion

Container number _____
 Mass of container and wet soil g _____
 Mass of container and dried soil g _____
 Mass of container g _____
 Mass of water g _____
 Mass of dried soil g _____
 Water content % _____
 Mass of wet soil for dispersion M_w g
 Mass of dry pretreated soil $M = \frac{100 M_w}{100 + w}$ g

Sieving

	600 μm	212 μm	63 μm
Standard test sieve			
Container number			
Mass of container and soil retained g			
Mass of container g			
Mass of soil g	$M_{CS} =$	$M_{MS} =$	$M_{FS} =$
Percentage of soil retained %			
Percentage of soil finer %			

Sedimentation

pH of suspension initial final

Pipette sampling		1st	2nd	3rd
		min s	min s	h min
Time of taking pipette sample				
Weighing bottle number				
Mass of bottle and dried pipette sample g				
Mass of bottle g		$M_1 =$	$M_2 =$	$M_3 =$
Mass of dried pipette sample (V_p ml suspension) g				
Weighing bottle number				
Mass of bottle and sodium hexametaphosphate g				
Mass of bottle g				
Mass of sodium hexametaphosphate (V_p ml solution) M_4 g				
		Percentage %	Maximum size (mm)	Percentage finer than size %
Coarse sand (2 mm to 0.6 mm) $100 M_{CS}/M$			2.0	100
Medium sand (0.6 mm to 0.2 mm) $100 M_{MS}/M$			0.60	
Fine sand (0.2 mm to 0.06 mm) $100 M_{FS}/M$			0.212	
Coarse silt (0.06 mm to 0.02 mm) $100 \left(\frac{M - (M_{CS} + M_{MS} + M_{FS} + S(M_1 - M_4))}{M} \right)$			0.063	
Medium silt (0.02 mm to 0.006 mm) $100S (M_1 - M_2)/M$			0.020	
Fine silt (0.006 mm to 0.002 mm) $100S (M_2 - M_3)/M$			0.006	
Clay (less than 0.002 mm) $100S (M_3 - M_4)/M$			0.002	

* Delete inappropriate words.

NOTES