

Testing aggregates —

Part 103: Method for determination of particle size distribution —

Section 103.2 Sedimentation test

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Committees responsible for this British Standard

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Foreword

This Section of BS 812, prepared under the direction of the Cement, Gypsum, Aggregates and Quarry Products Standards Committee, is a revision of 7.2 of BS 812-1:1975 which is withdrawn. BS 812-2 and BS 812-3 are also being revised and as each of the tests, or collection of related tests, is revised, it is intended to issue it as a separate Part or Section of this standard.

In the case of the test for the determination of clay, silt and dust in fine or coarse aggregate, major changes have been made to the procedures described in BS 812-1:1975. The decantation method for determining the amount of material passing a 75 μm test sieve complying with BS 410 is no longer included, as a procedure for determining this fraction is included in BS 812-103.1, originally published as BS 812-103. The field settling test has also been omitted because of difficulties in interpreting the results. This Section of BS 812 therefore deals only with a revision of the sedimentation test of 7.2.3 of BS 812-1:1975. Although the general principles of the method have been retained, more detail has been included and the procedure has been brought into line with the procedure for sedimentation described in BS 1377.

The terms "clay, silt and dust" which were used in the previous edition of BS 812 have not been retained as they have never been properly defined; the particles are classified solely on the basis of their equivalent particle diameters. The classification is thus solely one of particle size and gives no indication of the composition of the particles or whether or not their presence is likely to be deleterious. It should not therefore be used for this purpose.

It is intended that other British Standards should call up BS 812 test methods as the basis of compliance. Nevertheless it is *not* intended that all aggregates should be subjected regularly to all the listed tests. Requirements in other British Standard specifications will refer only to the relevant test methods.

Some of the tests in other Parts of this standard are of limited application and advice on the use of simpler tests is given, for example, when they can be used for a preliminary sorting of aggregates to see whether more expensive testing is justified.

Reference should be made to BS 812-102 for general guidance on testing aggregates, precision of test methods and variance arising from sampling errors.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 6, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This Section of BS 812 describes a gravimetric method, based on sedimentation, for the determination of the proportion of material finer than 20 μm in particle size in aggregates.

This method is not suitable for particles larger than 20 μm because of their short settling times.

NOTE 1 Methods for determination of particle size distribution of an aggregate down to the passing 75 μm fraction are described in BS 812-103.1. If the proportion of material between 20 μm and 75 μm is required, this may be calculated from the results of the determination carried out in accordance with this Section and BS 812-103.1.

NOTE 2 The titles of the publications referred to in this standard are listed on the inside back cover.

2 Definitions

For the purpose of this Section of BS 812 the definitions given in BS 812-101 and BS 812-102 apply.

3 Principle

Particles of different sizes have different settling velocities; the relationship between particle diameter and settling velocity is given by Stokes' Law from which, when the velocity is measured, the particle diameter can be calculated on the assumption that the particles are spherical in shape. If after a given time from the initial dispersal of the particles in a liquid medium, samples are taken from a given depth below the surface, the suspension will contain only the particles where settling velocities have been insufficient to carry them further. In the method described in this Section of BS 812 samples are taken from an aqueous suspension at various time intervals by means of a pipette; when the samples have been dried and weighed the percentages of different particle sizes are determined.

4 Sampling

The sample used for the test (the laboratory sample) shall be taken in accordance with the procedure described in clause 5 of BS 812-102:1984.

5 Apparatus

5.1 A *sample divider*, of size appropriate to the maximum particle size to be handled, or alternatively a flat shovel and a clean, flat and hard surface, e.g. a metal tray, for use in quartering.

NOTE A suitable divider is the riffle box illustrated in Figure 2 of BS 812-102.

5.2 A *ventilated oven*, fitted with a thermometer¹⁾ or similar device and thermostatically controlled to maintain a temperature of 105 ± 5 °C.

5.3 *Balances*, of suitable capacity, one accurate to 0.1 % of the mass of the sample being weighed, together with one that is accurate to 0.001 g.

5.4 A *variable position sedimentation pipette*, of approximately 25 mL capacity generally as described in BS 3406-2 and of the general form illustrated in Figure 1. This shall be held rigidly in a clamp which can be raised or lowered as required and which is fitted with a scale from which the changes in height of the pipette can be read.

5.5 *Glass sedimentation tubes*, of approximately 1 200 mL capacity graduated at 1 000 mL with rubber bungs to fit.

NOTE The tubes should have a height to diameter ratio of between 3.5 and 7.5. Gas jars are suitable for use provided that they have been carefully calibrated by filling them with 1 kg \pm 1 g of water and the position of the bottom of the meniscus permanently engraved on the outside of the jar.

1 000 mL capacity measuring cylinders complying with BS 604 may also be used but problems arise in placing them in the constant temperature bath (5.7) and with leakages from the gap between the spout and rubber bung.

5.6 *Glass weighing bottles*, of at least 50 mL capacity, fitted with ground-glass stoppers.

NOTE The number required depends on the number of particle sizes below 20 μm that it is desired to measure. A minimum of six bottles is required.

5.7 A *constant temperature bath*, fitted with a thermometer¹⁾ or similar device and capable of being maintained at 25 ± 0.5 °C into which the sedimentation tubes can be immersed up to the 1 000 mL mark. The bath shall not vibrate the sample.

NOTE This item is necessary when sedimentation times in excess of 30 min are required.

5.8 *Two glass bottles*, of 500 mL to 1 000 mL capacity fitted with watertight closures (for fine aggregate only).

5.9 A *device for rotating the bottles about their long axis*, with the long axis horizontal, at a speed of 80 ± 20 r/min (for fine aggregates only).

NOTE If such a device is not available the machine described in 4.5.1(2) in BS 598-2:1974 can be used.

5.10 *Containers*, of a size sufficient to contain the test portions plus five times their volume of water (for coarse aggregates only).

5.11 A *stop-clock or stop-watch*

5.12 A *desiccator*, (a convenient size is from 200 mm to 250 mm in diameter) containing anhydrous silica gel.

¹⁾ Thermometers should comply with BS 593. Other temperature measuring devices should be of equivalent accuracy.

5.13 A pipette, of 50 mL capacity.

6 Reagents

6.1 *Sodium hexametaphosphate solution.* Made by dissolving 33 g of sodium hexametaphosphate

and 7 g of sodium carbonate in distilled water or water of equivalent quality to make 1 L of solution.

This solution is unstable and shall be freshly prepared approximately once a month. The date of preparation shall be recorded on the bottle.

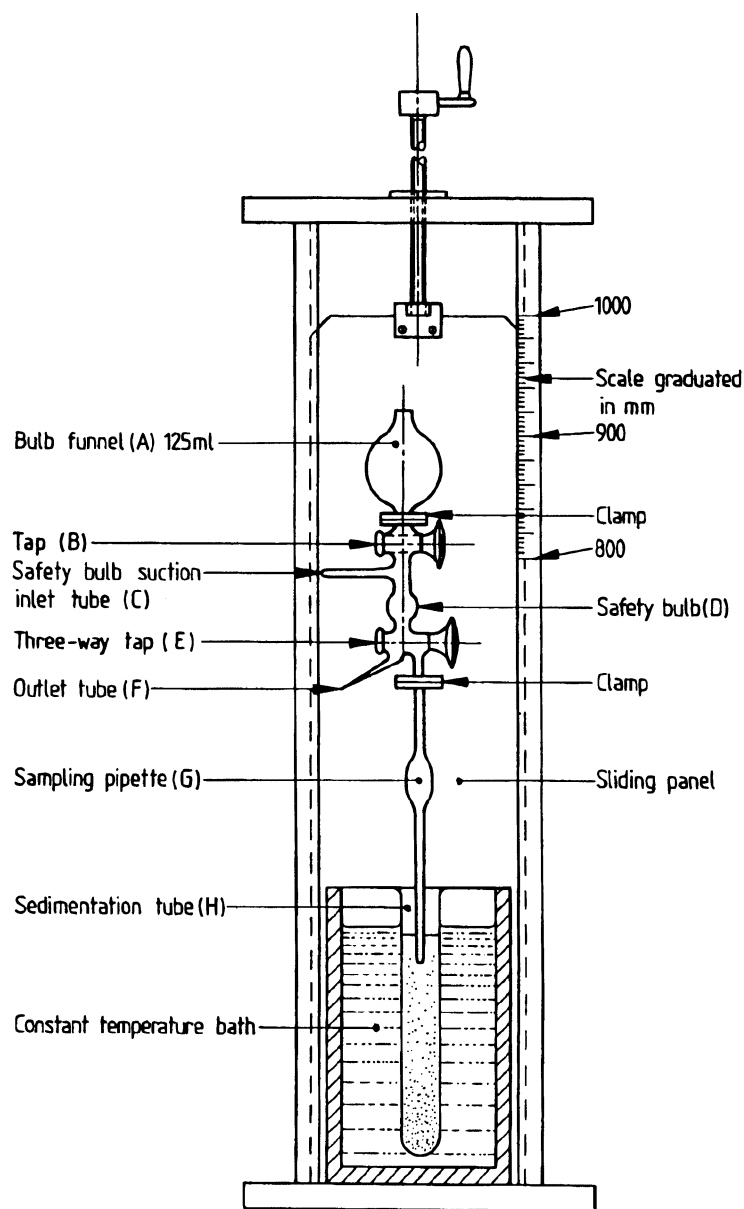


Figure 1 — A suitable arrangement for lowering the sampling pipette into the suspension

6.2 Water. Distilled or water of equivalent quality.

7 Preparation of test portion

7.1 Reduce the laboratory sample by the procedures described in clause 6 of BS 812-102:1984 to produce two test portions with mass not less than the mass given in Table 1 appropriate to the nominal size of the aggregate.

Table 1 — Minimum mass of test portions for materials finer than 20 μm in particle size

Nominal size of aggregate	Minimum mass of test portions
mm	kg
63	6.0
20	1.0
10	0.5
5 or less	0.3

7.2 Dry the test portions by heating at a temperature of 105 ± 5 °C maintaining this temperature until each test portion has reached a constant mass.

NOTE Normally 16 h to 24 h is a sufficient period.

8 Procedure

8.1 Calibration of sampling pipette

8.1.1 Thoroughly clean and dry the sampling pipette and immerse the tip in distilled water. Close tap B and open tap E (see Figure 1).

8.1.2 By means of a rubber tube attached to C, suck water up into the pipette until it rises above E. Close tap E and remove the pipette from the water. Drain off surplus water drawn up into the cavity above E by opening the tap in such a way as to connect bulb D and outlet tube F.

8.1.3 Discharge the water contained in the pipette and tap E into a glass weighing bottle of known mass, and reweigh. Calculate the internal volume (V_p mL) of the pipette and the tap E to the nearest 0.05 mL.

8.1.4 Make three determinations of the volume and calculate and record the average value.

8.2 Preparation of suspensions

8.2.1 General. Prepare a suspension of each of the two test portions, in accordance with either **8.2.2** or **8.2.3**, as appropriate.

8.2.2 Procedure for fine aggregates (5.0 mm nominal size or less). Weigh the dried test portion to the nearest 0.1 g (mass M) and place it in a glass bottle of 500 mL to 1 000 mL capacity together with about 250 mL of water. Add 50 mL of the sodium hexametaphosphate solution by means of the 50 mL

pipette. Fix the closure taking care to ensure watertightness. Then rotate the jar about its long axis, with this axis horizontal at a speed of 80 ± 20 r/min for a period of 15 min. At the end of 15 min, remove the watertight closure and rinse the closure free of adhering particles, collecting the rinsings on a small 75 μm sieve set in a funnel discharging into the sedimentation tube. Pour the suspension from the bottle onto the sieve. Add successive 150 mL portions of water to the bottle, swirl gently and decant the suspension through the sieve. Make up the volume of suspension in the sedimentation tube to 1 000 mL with water.

8.2.3 Procedure for coarse aggregate (larger than 5.0 mm nominal size). Weigh the dried test portion to the nearest 1 g (mass M) and place it in one of the containers described in **5.10**. Cover it with water, agitate vigorously and allow to stand for 24 h with occasional agitation to remove all adherent fine material. Transfer the liquid suspension to another container and repeat this process as necessary until all fine material has been transferred to the second container. Allow the suspension to stand for 24 h and then decant as much of the supernatant liquid as possible without losing any of the solids. Wash the solids into the sedimentation tube, add 50 mL of the sodium hexametaphosphate solution from the pipette and make the volume up to 1 000 mL.

8.3 Sedimentation and gravimetric procedures

8.3.1 Add 50 mL of the sodium hexametaphosphate solution from a pipette to a graduated 1 000 mL sedimentation tube and dilute with distilled water to exactly 1 000 mL. Transfer this sedimentation tube together with the sedimentation tubes containing the suspensions, prepared as described in **8.2**, to the constant temperature bath (see note). Insert the rubber bungs and allow the tubes to stand immersed in water up to the 1 000 mL graduation mark for at least 1 h, or until they have reached the temperature of the bath. When they have attained this temperature remove the tubes and contents from the bath and shake them thoroughly by inverting several times before replacing them in the bath.

NOTE In those cases where only the fraction finer than 20 μm is being measured there is no need to use a constant temperature bath. The sedimentation tubes can therefore be allowed to stand until they have reached room temperature but it is essential that they be kept away from any source of heat such as sunlight or radiators.

8.3.2 At the same instant as the tube containing the suspension is replaced in the bath, start the stop-watch or stop-clock. Remove the rubber bungs carefully and lay them lightly on the top of each tube.

8.3.3 About 30 s before the sample is due to be taken, lower the pipette with the tap E closed until the tip is just in contact with the surface of the suspension. Taking care to avoid causing turbulence, lower the pipette vertically until the tip is 100 ± 1 mm below the surface, taking 10 s to complete this operation. At the required sampling time (see note) open the tap E and draw a sample (V_p mL) up into the pipette. Fill the pipette and the bore in tap E with the solution and then close tap E. Take 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 away this surplus into a beaker down the outlet tube F by opening the tap E in such a way as to connect D and F. Allow distilled water to run from the bulb funnel A into D and out through F until no suspension remains in the system.

NOTE The times at which samples should be removed from the sedimentation tubes by the procedure described in 8.3.3 depend primarily on the particle diameter and particle density. Where the proportion of material finer than $20 \mu\text{m}$ is to be determined, where the particle density is close to 2.70 and where room temperature is close to 25°C , the time of sampling is 4 min 0 s. The sampling time is measured from the moment the tube is replaced in the bath to the moment the pipette starts to fill up with suspension.

Other measurements that may need to be made include the determination of the proportion finer than $6 \mu\text{m}$ and the proportion finer than $2 \mu\text{m}$. For a particle density of 2.70 the sampling times at 25°C for these two fractions are 44 min 30 s and 6 h 42 min respectively. See appendix A for the sampling times at other densities.

8.3.4 Place a weighing bottle (see 5.6) of known mass determined to the nearest 0.001 g 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 a temperature of $105 \pm 5^\circ\text{C}$ and evaporate the sample to constant mass. After cooling in a desiccator, weigh the weighing bottle and contents to the nearest 0.001 g and determine the mass of solid material in the sample (m_1 , m_2 , etc. for each respective sampling time).

8.3.5 During any of the times in which the above sampling is taking place take a sample (V_p mL) from the sedimentation tube containing the sodium hexametaphosphate solution. Take this sample of the solution as described in 8.3.3 and 8.3.4 except that there is no need to time the operation of taking the solution. The depth of sampling is also unimportant. Determine the mass of solid material remaining after drying (m_h).

8.3.6 Repeat the procedure for suspension prepared from the second test portion.

9 Calculation and expression of results

9.1 Calculate the mass of solid material in 1 000 mL of suspension (in g) for each respective sampling time from the following equation:

$$W_x = \frac{m - m_h}{V_p} \times 1\,000 \quad (1)$$

where

W_x	is the mass of solid material finer than $x \mu\text{m}$ in 1 000 mL of suspension;
m	is m_1 , m_2 , m_3 , etc.;
m_1	is the mass of material finer than $20 \mu\text{m}$ in V_p mL from the first sample (in g);
m_2	is the mass of material finer than $6 \mu\text{m}$ in V_p mL from the second sample (in g);
m_3	is the mass of material finer than $2 \mu\text{m}$ in V_p mL from the third sample (in g);
m_h	is the mass of sodium hexametaphosphate in V_p mL (in g);
V_p	is the calibrated volume of the pipette (mL).

9.2 Calculate the percentage of material finer than $20 \mu\text{m}$ in the test portions of mass M (in g) from the following equations:

$$\text{material finer than } 20 \mu\text{m} = \frac{W_{20}}{M} \times 100 \quad (2)$$

If required, calculate the percentage for other sizes (e.g. $6 \mu\text{m}$ and $2 \mu\text{m}$) in the test portions of mass M from the following equations:

$$\text{material finer than } 6 \mu\text{m} = \frac{W_6}{M} \times 100 \quad (3)$$

or

$$\text{material finer than } 2 \mu\text{m} = \frac{W_2}{M} \times 100 \quad (4)$$

9.3 Calculate the mean x of the two determinations from the first sampling and report as the proportion of material finer than 20 μm to the nearest 0.1 %. Use this value of x to calculate r_1 from the equation (5) in 10.4. If the individual results differ by more than r_1 % repeat the test starting with new test portions.

Similarly where required, report the results for the determination of the other size fractions.

10 Precision

10.1 The precision data given in Table 2 were determined from an experiment conducted in 1987 involving nine laboratories. The experiment was designed and the data analysed following the principles set out in BS 5497-1. The materials tested consisted of 10 t stockpiles of a natural dry-screened sand and a crushed rock fine aggregate; 1 kg laboratory samples were prepared by following the procedures described in BS 812-102, and then a single laboratory sample of each material was sent to each participant. The participants produced two test portions from each laboratory sample by following the procedure given in 6.4 of BS 812-102:1984.

10.2 The tests for outliers given in BS 5497:1979 were applied to the test results. The results reported by one laboratory on the natural sand at the 20 μm and 6 μm levels, and by another laboratory on the crushed rock at the 20 μm level, were rejected because the between-test-portion ranges were found to be outliers.

10.3 Definitions of \bar{x} , r_1 , R_1 , R_2 , V_r , V_L , V_S are given in clause 2 and clause 5 of BS 812-101:1984. In this experiment the sample preparation was such that the reproducibility coefficients determined are reckoned to lie closer to R_1 than R_2 .

10.4 The repeatability coefficients in Table 2, for both rock types and for all three sizes, are reasonably well represented by the equation:

$$r_1 = 0.027\sqrt{\bar{x}(100 - \bar{x})} \% \quad (5)$$

The reproducibility coefficients for the natural sand fall around the equation:

$$R_1 = 0.065\sqrt{\bar{x}(100 - \bar{x})} \% \quad (6)$$

and for the crushed rock:

$$R_1 = 0.118\sqrt{\bar{x}(100 - \bar{x})} \% \quad (7)$$

In these equations \bar{x} is the average % finer. These equations may be used to interpolate values of r_1 and R_1 for levels between those which appear in Table 2.

10.5 95 % confidence limits for a test result may be calculated as:

$$\pm \sqrt{\left[\frac{(R_2)^2}{2} - \frac{(r_1)^2}{4} \right]} \%$$

when the test result is calculated as the average of the results of the tests on two test portions.

11 Test report

The test report shall state that the proportion of material finer than 20 μm and/or any other size fractions have been determined in accordance with this Section of BS 812. The report shall also state whether or not a certificate of sampling is available. If available a copy of the certificate shall be provided. The test report shall contain the following additional information:

- sample identification;
- the proportion of material finer than 20 μm ;
- the proportion of material of any other size fraction that was determined;
- particle density of each size fraction (see appendix A).

Table 2 — Precision data

Material	% finer than	Number of laboratories	Overall mean	Precision coefficients		Standard deviations	
			\bar{x}	r_1	R_1	$\sqrt{V_{r1}}$	$\sqrt{V_L}$
	μm		%	%	%	%	%
Natural sand	20	8	6.3	0.9	2.4	0.32	0.78
	6	7	4.6	0.5	1.0	0.18	0.29
	2	8	2.9	0.5	0.8	0.19	0.21
Crushed rock	20	8	9.5	0.7	3.7	0.26	1.28
	6	8	6.1	0.8	2.7	0.27	0.90
	2	8	3.4	0.2	2.1	0.07	0.74

Appendix A The relationship between sampling times for different particle densities and sizes

The relation between sampling time, particle size and particle density for samples removed at a depth of 100 mm in a suspension at 25 °C is given in Table 3.

Table 3 — Sampling times for different particle densities and sizes

Particle density	Times after shaking of starting sampling operation					
	20 µm		6 µm		2 µm	
	minutes	seconds	minutes	seconds	hours	minutes
2.50	4	30	50	30	7	35
2.55	4	20	49	0	7	21
2.60	4	10	47	30	7	7
2.65	4	5	46	0	6	54
2.70	4	0	44	30	6	42
2.75	3	50	43	30	6	30
2.80	3	40	42	0	6	20
2.85	3	35	41	0	6	10
2.90	3	30	40	0	6	0
2.95	3	25	39	0	5	50
3.00	3	20	38	0	5	41
3.05	3	15	37	0	5	33
3.10	3	10	36	0	5	25
3.15	3	5	35	0	5	18
3.20	3	0	34	30	5	10

NOTE 1 Times for other temperatures, particle densities, particle sizes and sampling depths may be calculated from Stokes' Law (see BS 3406-2).

NOTE 2 Methods for the determination of particle densities of the various size fractions are not described. These may differ from the particle density of the complete sample. If this information is not available a particle density of 2.70 may be assumed, but the report should include a note to this effect.

Publications referred to

- BS 410, *Specification for test sieves.*
- BS 593, *Specification for laboratory thermometers.*
- BS 598, *Sampling and examination of bituminous mixtures for roads and other paved areas.*
- BS 598-2, *Methods for analytical testing.*
- BS 604, *Specification for graduated glass measuring cylinders.*
- BS 812, *Testing aggregates.*
- BS 812-1, *Methods for determination of particle size and shape²⁾.*
- BS 812-2, *Methods for determination of physical properties²⁾.*
- BS 812-3, *Methods for determination of mechanical properties²⁾.*
- BS 812-101, *Guide to sampling and testing aggregates.*
- BS 812-102, *Methods of sampling.*
- BS 812-103.1, *Sieve tests.*
- BS 1377, *Methods of test for soils for civil engineering purposes²⁾.*
- BS 3406, *Methods for determination of particle size distribution.*
- BS 3406-2, *Recommendations for gravitational liquid sedimentation methods for powders and suspensions.*
- BS 5497, *Precision of test methods.*
- BS 5497-1, *Guide for the determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

²⁾ Referred to in the foreword only.

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