British Standard

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BRITISH STANDARD

BS 812-118:
1988

Testing aggregates —

Part 118: Methods for determination of sulphate content

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Aggregate Concrete Block Association
Association of Consulting Engineers
Association of Consulting Scientists
Association of Lightweight Aggregate Manufacturers
Brick Development Association
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Foreword

This Part of BS 812 has been prepared under the direction of the Cement, Gypsum, Aggregates and Quarry Products Standards Committee. In the revision of BS 812-2:1975, BS 812-3:1975 and BS 812-4:1976 each test, or collection of related tests, is being revised separately and is being issued as a separate Part or Section of BS 812.

This Part of BS 812 contains methods for determination of total sulphate content and water-soluble sulphate content of aggregates, neither of which was previously included in BS 812, but are included now because of the problems that may arise if aggregates containing high concentrations of sulphates are used in concrete or placed close to concrete structures.

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 aggregates should be subjected regularly to all the listed tests. Specifications in other standards, should call up only relevant test methods.

Advice on the use of simpler qualitative tests is given for a preliminary sorting of aggregates to see whether more precise and expensive testing is justified.

Reference should be made to BS 812-101 for general guidance on testing aggregates, precision of test methods and variance arising from sampling errors.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, 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.

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1 Scope

This Part of BS 812 describes two methods for determining the sulphate content of aggregates.

The first method determines the water-soluble sulphate content of aggregates, which applies to natural and synthetic aggregates to be used as drainage materials or for fill or hard core, e.g. unbound roadbases or sub-bases or foundations.

The second method determines the total sulphate content of aggregates, which applies to aggregates used in concrete and cement bound materials.

NOTE 1 The second method for the determination of total sulphate by acid extraction is specified as it is impractical to produce a method for the determinatin of total water-soluble sulphate because of low water solubility of calcium sulphate. For practical purposes for aggregates in concrete the total sulphate content determined by this method is taken to be the same as the total water-soluble sulphate content.

NOTE 2 In Appendix A two procedures for a semi-quantitative test are described. It is strongly recommended that one of these is used as a preliminary check before resorting to the test described in the main text, which may be needed for compliance with a specification.

NOTE 3 The titles of the publications referred to in this standard are listed on the inside back cover.

2 Definitions

For the purposes of this Part of BS 812, the definitions given in BS 812-101 and BS 812-102 apply.

3 Principle

3.1 Method for water-soluble sulphates

An aggregate sample is extracted with twice its own mass of distilled or demineralized water to remove water-soluble sulphate ions. The water-soluble sulphate content is determined by either an ion-exchange method or a gravimetric method in one of the following ways.

a) Some of the extract is transferred to an ion-exchange column and the eluate is titrated with standardized sodium hydroxide solution.

b) Some of the extract is treated with diluted hydrochloric acid. Barium chloride solution is added and the precipitate is collected, dried and weighed.

The ion-exchange method measures the presence of sulphate ions. When other anions (see note) are present this method is precluded. The gravimetric method is applicable when aggregates containing sulphides (i.e. slags) are being analysed.

NOTE Anions of strong acids, e.g. chlorides, nitrates and phosphates, interfere with the ion-exchange method and are apparently measured as sulphate. However, when chloride is the only other ion present, and it has been quantitatively determined, the ion-exchange method described can be used provided the result is corrected as shown in **5.6.2**.

3.2 Method for total sulphate content

The sulphates are extracted from a crushed sample of aggregate by dilute hydrochloric acid. Barium chloride solution is added to the extract. The precipitate of barium sulphate is collected, dried and weighed and the total sulphate content is calculated.

4 Sampling

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

Ensure that the sample is representative of the moisture content as well as the solids by preventing loss of moisture, except through evaporation, throughout the sampling procedure and at any subsequent stage up to the point of test.

NOTE This is necessary because where the aggregate to be tested carries surface water the sulphates will normally be dissolved, at least partly, in both this and the absorbed water and hence the sulphate content will be affected by migration of water through the bulk materials.

5 Determination of the sulphate content of a 2 : 1 water/aggregate extract

NOTE Calcium sulphate is relatively harmless because of its limited solubility and the extraction conditions have been chosen so that aggregates containing only calcium sulphate are classified as falling in class 1 of Table 6.1 in BS 8110-1:1985.

5.1 Apparatus

5.1.1 *Apparatus for preparation of test portion and extraction of the sulphate* (see **5.4** and **5.5.1**)

5.1.1.1 *Well ventilated oven*, capable of being controlled to maintain a temperature of 105 ± 5 °C.

5.1.1.2 *Balance*, capable of weighing up to 5 kg accurate to 5 g. The balance shall be regularly calibrated as specified in BS 5781-1.

5.1.1.3 *Test sieve*, 20 mm square hole perforated plate complying with BS 410.

5.1.1.4 *Pestle and mortar*, or suitable mechanical crusher.

NOTE This is not required for aggregate all passing the 20 mm sieve.

5.1.1.5 *Two plastics or metal bottles*, wide-mouthed, screw-capped. The bottles shall be 5 L capacity when testing coarse aggregates or 2 L when testing fine aggregate.

5.1.1.6 *Mechanical shakers or rollers*, to take the extraction bottles (**5.1.1.5**).

5.1.1.7 *Two filter funnels*, of approximately 100 mm diameter with medium grade[1] filter papers of a diameter appropriate to the size of the funnel.

5.1.1.8 *Beakers.*

5.1.2 *Apparatus for the ion-exchange method* (see **5.3** and **5.5.2.1**)

5.1.2.1 *Pipette*, 50 mL size, grade B in accordance with BS 1797.

5.1.2.2 *An ion-exchange column and reservoir.*

NOTE A typical design is shown in Figure 1.

5.1.2.3 *Conical flasks*, 500 mL capacity. At least two are required.

5.1.2.4 *Graduated measuring cylinder*, capacity 100 mL, grade B in accordance with BS 1797.

5.1.2.5 *Burette*, 50 mL size, grade B in accordance with BS 1797.

5.1.2.6 *Beakers.*

5.1.3 *Apparatus for the gravimetric method* (see **5.5.2.2**)

5.1.3.1 *Pipette*, 50 mL size, grade B in accordance with BS 1797.

5.1.3.2 *Apparatus* as listed in **6.1.2.1** to **6.1.2.10**.

5.1.4 *Apparatus for preparation of reagents* (see **5.2.2**)

5.1.4.1 *Plastics container*, airtight.

5.1.4.2 *Amber-coloured glass reagent bottle.*

5.2 Reagents

5.2.1 *General.* Reagents of recognized analytical quality and only distilled water or water of equivalent quality shall be used.

NOTE 1 Where accurately standardized solutions are required it may be more convenient to obtain them already standardized to be used in accordance with the manufacturer's instructions.

NOTE 2 If the ion-exchange column method is not used, the reagents described in **5.2.2.1** to **5.2.2.7** are not required.

5.2.2 *Reagents*

5.2.2.1 *Cation-exchange resin*, strongly acid[2].

5.2.2.2 *Hydrochloric acid solution* (c(HCl) = approximately 4 mol/L), made by diluting 360 mL concentrated hydrochloric acid (relative density 1.18) to 1 L with water.

5.2.2.3 *Hydrochloric acid solution*, made by diluting 200 mL concentrated hydrochloric acid (relative density 1.18) to 1 L with water.

5.2.2.4 *Nitric acid* (c(HNO_3) = approximately 1 mol/L), made by diluting 15 mL of concentrated nitric acid (relative density 1.42) to 100 mL with water.

5.2.2.5 *Sodium hydroxide solution* (c(NaOH) = approximately 0.1 mol/L), made by dissolving 2 g of sodium hydroxide in 500 mL of water. Determine the exact concentration (in mol/L) by titration against potassium hydrogen phthalate or with a standardized acid solution, and keep the solution in an airtight plastics container (**5.1.4.1**).

5.2.2.6 *Indicator*, giving a distinct colour change in the pH range 4 to 5.

NOTE Screened methyl orange is suitable.

5.2.2.7 *Silver nitrate solution*, made by dissolving 0.5 g of silver nitrate in 100 mL of water. Store the solution in an amber-coloured glass reagent bottle (**5.1.4.2**).

5.3 Preparation of the ion-exchange column

NOTE This procedure is not required if the ion-exchange column method is not used (see **3.1**).

5.3.1 Empty a quantity of cation-exchange resin (**5.2.2.1**), sufficient to half-fill the column (**5.1.2.2**), into a beaker (**5.1.2.6**) and stir with water. Empty this suspension of resin in water into the column so that when the resin has settled, there is approximately 20 mm depth of water above the resin when the surplus water has drained away. Maintain this depth of water above the resin at all times as it is important that the resin is not allowed to dry out.

5.3.2 Activate the cation-exchange resin by leaching with 100 mL of the hydrochloric acid solution (**5.2.2.2**) followed by washing with distilled water. Place the acid in the reservoir and leave the apparatus until the acid has passed through the column. Rinse and fill the reservoir with distilled water and leave the water to percolate through until the liquid coming from the column gives no turbidity when tested with about 1 mL of silver nitrate solution (**5.2.2.7**) acidified with about five drops of nitric acid (**5.2.2.4**).

5.3.3 Regenerate the cation-exchange resin in the manner described in **5.3.2** after it has been used for four consecutive determinations.

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[1] Whatman No. 40 have been found suitable.

[2] A suitable material is Zerolit 225 or Amberlite 1R-120.

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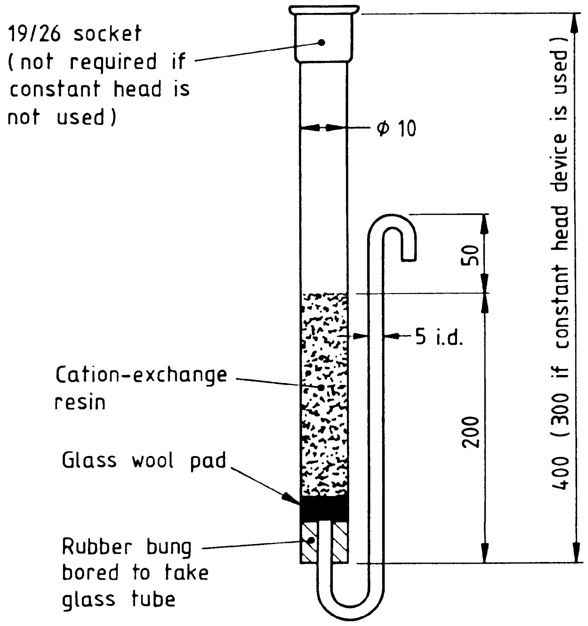
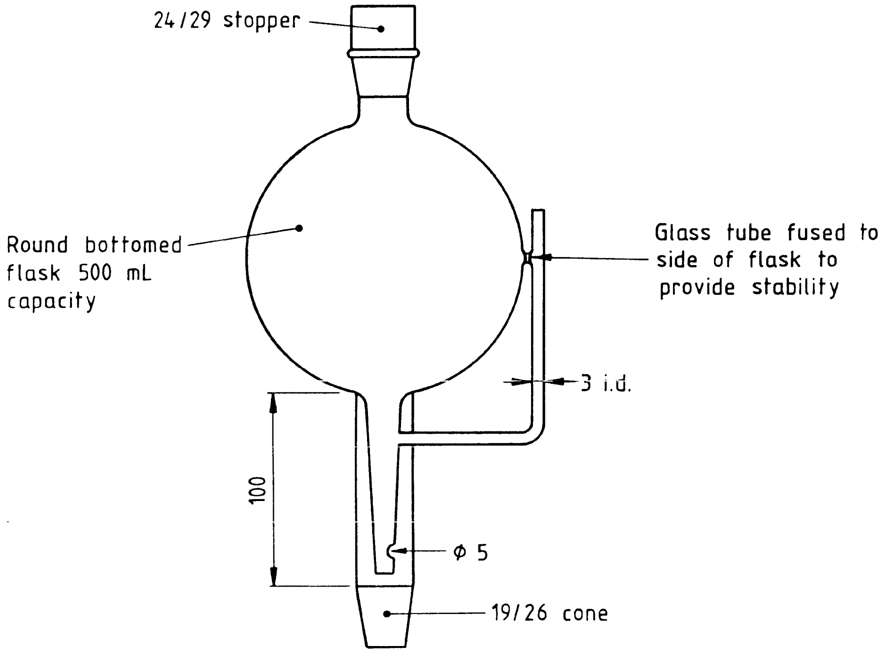


(a) Ion-exchange column



(b) Constant head device for use with ion-exchange column

All dimensions are in millimetres.

Figure 1 — Ion-exchange column for water-soluble sulphates

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5.4 Preparation of test portion

5.4.1 Dry the laboratory sample at a temperature of 105 ± 5 °C to remove surface water so that the sample is free flowing.

5.4.2 Reduce the laboratory sample by the procedures described in clause **6** of BS 812-102:1984 to an amount not less than the mass given in Table 1 appropriate to the nominal size of the aggregate.

Table 1 — Minimum mass of preliminary sub-sample for water-soluble sulphate method

Nominal maximum size of aggregate	Minimum mass of sub-sample
mm	kg
63	50
50	35
40	15
28 or less	5

5.4.3 Sieve the sub-sample through a 20 mm sieve (**5.1.1.3**) and crush any oversize aggregate to pass the sieve avoiding excessive grinding. Combine, mix thoroughly and by suitable sample reduction techniques produce two test portions each of about 2 kg mass for coarse aggregate or two test portions each of about 500 g mass for sand. Dry the test portions by heating at a temperature of 105 ± 5 °C to achieve a dry mass which is constant to within 0.1 %.

5.5 Analytical procedure

5.5.1 *Preparation of extracts*

5.5.1.1 For coarse aggregates use the two plastics or metal bottles of 5 L capacity and for fine aggregate use the two bottles of 2 L capacity (**5.1.1.5**). Weigh each bottle and record its mass to the nearest 5 g.

5.5.1.2 Transfer the test portions obtained as described in **5.4** to the bottles, weigh bottle and contents and record their mass to the nearest 5 g. Calculate the mass of aggregate in each bottle by difference.

5.5.1.3 Add to each bottle a mass of distilled or demineralized water equal to twice the mass of the test portion. Fasten the water-tight bottle closures and mix the contents by shaking or rolling continuously for a minimum of 24 h.

NOTE If mechanical agitation equipment is not used and the sample and water are merely allowed to remain in contact with occasional shaking there is a possibility (particularly if the source of sulphate is, for example, large crystals of gypsum) that the sulphate which could theoretically dissolve will not all be extracted in 24 h.

5.5.1.4 Filter the extracts through dry, medium-grade filter papers (**5.1.1.7**) until at least 100 mL of clear filtrates have been collected in clean dry beakers (**5.1.1.8**).

5.5.2 *Determination of the sulphate in the extracts*

5.5.2.1 *Ion-exchange method.* With a pipette (**5.1.2.1**) transfer 50 mL of filtered extract to the top of the ion-exchange column and allow to drain through the resin.

Rinse the resin with two 75 mL increments of distilled water collecting all the solution and washings in a conical flask (**5.1.2.3**) placed under the outlet of the column.

Add three to five drops of the indicator (**5.2.2.6**) to the contents of the conical flask and titrate to the colour change with the standardized sodium hydroxide solution (**5.2.2.5**).

Replace the flask under the outlet of the column and pass a further 75 mL of distilled water through the resin. If the indicator colour changes to show that more acid has been washed from the resin, titrate again to neutrality.

Record the total volume of standardized sodium hydroxide solution used (v_1).

Repeat with another 50 mL aliquot of the filtrate and record the volume of standardized sodium hydroxide solution used (v_2).

Take the mean value of v_1 and v_2 (V).

If the values of v_1 and v_2 differ by more than 0.5 mL repeat the procedure.

Repeat the complete procedure with a second test portion.

5.5.2.2 *Gravimetric method.* Transfer 50 mL of filtered extract to a 500 mL beaker, dilute to 300 mL with distilled or demineralized water, add 10 mL of hydrochloric acid solution (**5.2.2.3**) and carry out the procedure described in **6.4.2** to **6.4.9**.

NOTE When aggregates containing sulphide are being analysed see the note to **6.4.2**.

Repeat the procedure with a second test portion.

5.6 Calculation and expression of results

5.6.1 If no other anions are present, calculate the concentration of sulphate ions, (as SO_3 in g/L), present in the extracts, from the equation:

$$\text{concentration of } SO_3 = 0.8mV$$

where

V is the volume of sodium hydroxide solution (in mL);

m is the concentration of sodium hydroxide solution (in mol/L).

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5.6.2 If chlorides are the only other ions present, calculate the concentration of sulphate ions, (as SO_3 in g/L), present in the extracts from the equation:

$$\text{concentration of } SO_3 = 0.8mV - 5.64\ C$$

where

C is the chloride ion content as a percentage by mass of dry aggregate.

5.6.3 When the gravimetric method has been used, calculate the sulphate concentration of sulphate ions, (as SO_3 in g/L), present in the extracts from the equation:

$$\text{concentration of } SO_3 = 6.86m_2$$

where

m_2 is the mass of precipitate obtained in **6.4.9** (in g).

5.6.4 Express the mean sulphate contents of the 2 : 1 extracts to the nearest 0.1 g/L. If the individual results differ by more than 0.1 g/L and r_1, (calculated from **B.5**) repeat the test starting with new analytical test portions (see **5.4**).

NOTE With aggregates containing discrete crystals of gypsum it may be difficult to obtain close agreement between individual test portions.

6 Determination of the total sulphate content by acid extraction

6.1 Apparatus

6.1.1 *Apparatus for preparation of test portion* (see **6.3**)

6.1.1.1 *Well ventilated oven*, capable of being controlled to maintain a temperature of 105 ± 5 °C.

6.1.1.2 *Balance*, capable of weighing up to 5 kg accurate to 5 g. The balance shall be regularly calibrated as specified in BS 5781-1.

6.1.1.3 *Test sieves*, 20 mm, 5 mm, 1 mm and 150 µm complying with BS 410.

6.1.1.4 *Pestle and mortar*, or suitable mechanical crusher.

6.1.2 *Apparatus for analysis of test portion*

6.1.2.1 *Balance*, capable of weighing up to 100 g accurate to 0.001 g. The balance shall be regularly calibrated as specified in BS 5781-1.

6.1.2.2 *Beakers*, capacity 250 mL and 500 mL.

6.1.2.3 *Electric hot-plate*, capable of being controlled to boil the contents of beakers without causing undue overheating.

6.1.2.4 *Fume cupboard.*

6.1.2.5 *Two filter funnels*, of approximately 100 mm diameter with medium grade[3] and fine grade[4] filter papers of a diameter appropriate to the size of the funnels.

6.1.2.6 *Dropping pipette*, capacity 10 mL.

6.1.2.7 *Sintered silica filtering crucibles*, porosity grade 4, approximately 35 mm in diameter and 40 mm in height.

NOTE These are not required if filtration through fine grade[4] filter papers is preferred.

6.1.2.8 *Ignition crucibles*, approximately 35 mm in diameter and 40 mm in height and capable of maintaining a constant mass when heated to 800 °C.

NOTE These crucibles are not required if the filtering crucibles (**6.1.2.7**) are used. Porcelain, silica or platinum are suitable materials for ignition crucibles.

6.1.2.9 *A suitable means of igniting the precipitate*, preferably an electric muffle furnace capable of reaching and maintaining 800 °C.

6.1.2.10 *Dessicator.*

6.1.3 *Apparatus for preparation of reagents* (see **6.2.2**)

6.1.3.1 *Amber-coloured glass reagent bottle.*

6.2 Reagents

6.2.1 *General.* Reagents of recognized analytical quality and only distilled water or water of equivalent quality shall be used.

6.2.2 *Reagents*

6.2.2.1 *Barium chloride*, 5 % solution, made by dissolving 50 g of barium chloride in 1 L of water, and filtered before use if necessary.

6.2.2.2 *Dilute hydrochloric acid*, made by diluting 100 mL of concentrated hydrochloric acid (relative density 1.18) to 1 L with water.

6.2.2.3 *Silver nitrate solution*, made by dissolving 0.5 g of silver nitrate in 100 mL of water. Store the solution in an amber-coloured glass reagent bottle (**6.1.3.1**).

6.3 Preparation of test portion

6.3.1 Dry the laboratory sample at a temperature of 105 ± 5 °C to remove surface water so that the sample is free flowing.

6.3.2 Reduce the laboratory sample by the procedures described in clause **6** of BS 812-102:1984 to an amount not less than the mass given in Table 2 appropriate to the nominal maximum size of aggregate.

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[3] Whatman No. 40 is suitable, but fine filter paper, e.g. Whatman No. 42 may be necessary if the filtrate is not completely clear.

[4] Whatman No. 42 is suitable.

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Table 2 — Minimum mass of preliminary sub-sample for total sulphate content method

Nominal maximum size of aggregate	Minimum mass of sub-sample
mm	kg
63	50
50	35
40	15
28	5
20	2
14	1
10	0.5
5 or less	0.2

6.3.3 Sieve the sub-sample through a 20 mm sieve (**6.1.1.3**) and crush any oversize aggregate to pass the sieve. Combine, mix thoroughly and where the mass of the sub-sample exceeds 2 kg reduce the sub-sample to 2 kg. Dry the sub-sample by heating at a temperature of 105 ± 5 °C to achieve a constant mass to within 0.1 %. Crush all the aggregate to pass a 5 mm sieve, mix and reduce to not less than 200 g. Crush all the aggregate to pass a 1 mm sieve and produce a sample of approximately 100 g and grind all this to pass a 150 µm sieve. This is the test portion. Carry out all sampling reduction by the method described in clause **6** of BS 812-102:1984.

NOTE With suitable mechanical grinding equipment it is possible to by-pass some of these stages. For example, a jaw-crusher could be used to crush all the sub-sample to pass 5 mm without the preliminary sieving and crushing at the 20 mm size. Similarly the 200 g sub-sample portion passing the 5 mm sieve could be crushed to pass 150 µm, by-passing the 1 mm size operation, in, for example, a disc mill. The intention is to produce a test portion which is fully representative of the laboratory sample.

6.4 Analytical procedure

6.4.1 Weigh accurately to the nearest 0.001 g about 3 g of the test portion (prepared as described in **6.3**) and record the mass (m_1). Place it in a 250 mL beaker (**6.1.2.2**), add 100 mL of the diluted hydrochloric acid (**6.2.2.2**) and stir. Repeat with a second duplicate 3 g of the test portion, and follow the procedure for both.

NOTE 1 Aggregates containing significant amounts of carbonates will froth at this stage. In these cases add the acid slowly while continuously stirring.

NOTE 2 Aggregates containing sulphide will release H_2S on acidification and this will be noticeable by its smell. In these cases there is a danger that this procedure will overestimate the sulphate content because of sulphide oxidation.

If the aggregates contain sulphide, place 100 mL of the diluted hydrochloric acid in a 250 mL beaker and heat to boiling point. Remove from the source of heat and, while stirring the acid solution, sprinkle the weighed analytical portion of about 3 g mass on to the acid.

6.4.2 Heat to boiling and simmer gently for 15 min in a fume cupboard (**6.1.2.4**). Filter through a medium filter paper (**6.1.2.5**). Ensure that the filtrate is clear. If not, refilter with a fine filter paper. Wash thoroughly with hot distilled water. Collect the filtrate and washings in a 500 mL beaker (**6.1.2.2**) and dilute to 300 mL with distilled water.

NOTE When analysing extracts for water soluble sulphate (see **5.5.2.2**) filtration will not normally be necessary unless the aggregate contains sulphides (e.g. slags). When such aggregates are being tested, stand the solution in a warm place for 30 min after simmering. If a white precipitate develops, filter through a medium filter paper and wash thoroughly with hot distilled water and continue as described in **6.4.3**.

6.4.3 Boil and add 10 mL barium chloride solution (**6.2.2.1**) dropwise with constant stirring. Continue boiling until the precipitate is properly formed and then let the solution stand at just below boiling point for at least 30 min then leave to cool for 24 h or overnight.

6.4.4 Transfer the precipitate of barium sulphate with extreme care to a previously ignited and weighed sintered silica filter crucible (**6.1.2.7**) using suction. Alternatively transfer the precipitate with extreme care to a suitable filter paper in the glass funnel and filter.

6.4.5 In either case wash the precipitate several times with hot distilled water until the washings are free from chloride as indicated by an absence of turbidity when a drop is tested with the solution of silver nitrate (**6.2.2.3**). If a sintered silica filter crucible is used remove it from the filter flask and dry at 105 ± 5 °C for approximately 30 min and gradually raise the temperature to 800 °C either in an electric muffle furnace or by other suitable means (**6.1.2.9**) until no further loss in mass occurs; 15 min at 800 °C should suffice.

6.4.6 Cool the crucible in a desiccator (**6.1.2.10**) and weigh to the nearest 0.001 g, and calculate the mass of the precipitate (m_2) from the increase in mass of the crucible.

6.4.7 If the precipitate is filtered through a filter paper, transfer the filter paper and precipitate to a previously ignited and weighed crucible (**6.1.2.8**).

6.4.8 If an electric muffle furnace is used, place the crucible and contents in it at room temperature and gradually raise the temperature to 800 °C. If a bunsen or other burner is used, first dry the filter paper and precipitate slowly over a small flame taking care to char the filter paper slowly rather than to allow it to inflame, otherwise some of the precipitate may be lost, and gradually raise the temperature to red heat.

NOTE Ignition for about 15 min should be sufficient in all cases.

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6.4.9 Calculate the mass of the precipitate obtained (m_2) from the increase in mass (to the nearest 0.001 g) of the crucible.

6.5 Calculation and expression of results

6.5.1 Calculate the total sulphate content, as a percentage by mass of the dry aggregate, from the equation:

$$\text{percentage of } SO_3 = \frac{m_2 \times 34.3}{m_1}$$

6.5.2 Express the mean total sulphate content of the two test portions to the nearest 0.01 % (as SO_3) of the dry mass of aggregate. Repeat the test starting with new 3 g analytical portions if the individual results differ by more than 0.05 % (SO_3).

7 Precision

7.1 A precision experiment was carried out on five aggregates by 10 laboratories. Details of the experiment and the precision data are given in Appendix B.

7.2 The uses of the precision data are described in clause **5** of BS 812-101:1984.

8 Test report

The test report shall affirm that the sulphate content was determined in accordance with this Part of BS 812 and state whether or not a certificate of sampling is available. If available, a copy of the certificate of sampling shall be provided. The test report shall include the following information:

a) sample identification;

b) the sulphate content in g/L of a 2 : 1 water/aggregate extract expressed in g/L; and/or

c) the total sulphate content expressed as a percentage by mass of the dry aggregate.

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Appendix A Semi-quantitative test for the presence of sulphate ions

A.1 Apparatus and reagents

A.1.1 *Apparatus for method 1 or method 2* (see **A.2.2**)

A.1.1.1 *Plastics bucket*, capacity 10 L.

A.1.1.2 *Beaker or plastics drinking cup.*

A.1.2 *Additional apparatus for method 2*

A.1.2.1 *Filter paper,* medium grade[5].

A.1.2.2 *Beaker*, capacity 500 mL.

A.1.2.3 *Test tube.*

A.1.3 *Reagent for method 1*

A.1.3.1 *Sulphate test strips*[6].

A.1.4 *Reagents for method 2*

A.1.4.1 *Hydrochloric acid solution*, made by diluting 200 mL of concentrated hydrochloric acid (relative density 1.18) to 1 L with water.

A.1.4.2 *Barium chloride* 5 % *solution*, made by dissolving 50 g of barium chloride in 1 L of water, and filtered before use, if necessary.

A.1.4.3 *Measuring cylinder.*

A.1.4.4 *Stop watch.*

A.2 Procedure

A.2.1 Place approximately 5 kg of coarse aggregate or 1 kg of sand in the bucket (**A.1.1.1**) and add an equivalent mass of water of low sulphate content. Agitate the contents intermittently for 7 h and then pour some solution into a beaker or plastics drinking cup (**A.1.1.2**).

A.2.2 Carry out one of the following procedures.

Method 1. Allow any solids to settle and briefly dip the sulphate test strip (**A.1.3.1**) into the clear supernatant liquid. After 2 min observe the colouration of the three test zones. If none of the test zones has changed from red to yellow the sulphate in the aggregate can be taken to be less than 0.02 %.

Method 2. Filter the solution through a medium grade filter paper (**A.1.2.1**) until approximately 50 mL solution has been collected. Measure 50 mL into a 500 mL beaker (**A.1.2.2**), add 150 mL water and stir thoroughly. Pour some of the diluted solution into a test tube (**A.1.2.3**) so that it is half filled, add 5 mL hydrochloric acid (**A.1.4.1**) and 5 to 10 drops barium chloride solution (**A.1.4.2**) and mix. If there is no turbidity or it has not formed within 5 s, assume that the aggregate contains less than 0.02 % sulphate as SO_3.

NOTE The barium chloride test is very sensitive and an observable turbidity will eventually form (after approximately 1 min), even when the SO_3 content of the aggregate is only 0.004 %. It is also necessary to ensure that the water used for testing does not contain sufficient sulphates (< 20 mg/L) to give a positive result.

Appendix B Details of experiments for determining precision of tests for water-soluble sulphate and total sulphate contents of aggregates

B.1 The precision data given in Table 3 and Table 4 were determined from an experiment conducted in 1984/5 involving 10 laboratories. The experiment was designed, and the data analysed following the principles set out in BS 5497-1:1979.

B.2 The materials used were approximately 200 kg lots. With each material, twenty 5 kg to 10 kg laboratory samples were prepared and two sent to each laboratory. The laboratories divided each laboratory sample into four test portions: one was used for the chloride determination described in BS 812-117, one for the water-soluble sulphate determination and two for the total sulphate determination described in this Part of BS 812.

B.3 The tests for outliers given in BS 5497-1:1979 were applied to the data. A summary of the results shown to be outliers by these tests is given in Table 5 and Table 6.

B.4 Because of the small quantities of the aggregates obtained for this experiment, the estimates of V_S measure only that variability which arises when dividing 100 kg to 200 kg of material into 5 kg to 10 kg laboratory samples. Likewise the contribution of sample reduction variability to the estimates of V_{r_1} consists only of the variability which arises when dividing 5 kg to 10 kg laboratory samples into two test portions. In practice one might expect V_S and perhaps V_{r_1} to be larger than found in this experiment.

B.5 The precision data from Table 3 (water-soluble sulphate) for four of the materials (limestone, shale, gravel and sand) fit the following relationships:

$$r_1 = 0.02 + 0.05\ \bar{x}$$

$$R_1 = R_2 = 0.06 + 0.11\ \bar{x}$$

These may be used to interpolate values of r_1, R_1 and R_2 for levels of sulphate ion ($\bar{x}$ g/L) between those which appear in Table 3. The blastfurnace slag gave a higher value of R_1 and R_2 that that predicted by the relationship. This is attributed to the presence of sulphides: this difficulty is overcome by following the procedure described in the note to **6.4.2**.

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[5] Whatman No. 40 has been found suitable.

[6] Merck quant test strips have been found suitable.

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B.6 The precision data from Table 4 (total sulphate) fit the following relationships:

$r_1 = 0.03 + 0.01\bar{x}$

$R_1 = 0.06 + 0.49\bar{x}$

$R_2 = 0.05 + 0.58\bar{x}$

These may be used to interpolate values of r_1, R_1 and R_2 for levels of sulphate ion ($\bar{x}$ %) between those which appear in Table 4.

Table 3 — Precision data, water-soluble sulphate determination

Aggregate	Sulphate ion g/L							
	n	$\bar{x}$	r_1	R_1	R_2	$\sqrt{V_{r_1}}$	$\sqrt{V_L}$	$\sqrt{V_S}$
14 mm light grey limestone (D)	9	0.01	0.01	0.03	0.003	0.003	0.010	0.003
14 mm sea-dredged gravel and sand (B)	9	0.14	0.04	0.05	0.06	0.015	0.012	0.004
5 mm medium grey limestone crushed sand/shell mixture (E)	9	0.17	0.03	0.13	0.13	0.011	0.045	0.000
14 mm blastfurnace slag (A)	8	1.31	0.10	0.60	0.62	0.035	0.210	0.065
28 mm colliery shale (C)	8	2.13	0.12	0.29	0.29	0.041	0.095	0.000

NOTE n is the number of laboratories whose results were included in the calculation of the precision data. Definitions of $\bar{x}$, $r1$, $R1$, R_2, V_{r1}, V_L and V_S are given in **5.2** of BS 812-101:1984.

Table 4 — Precision data, total sulphate determination

Aggregate	Sulphate ion %							
	n	$\bar{x}$	r_1	R_1	R_2	$\sqrt{V_{r_1}}$	$\sqrt{V_L}$	$\sqrt{V_S}$
14 mm light grey limestone (D)	9	0.02	0.02	0.06	0.06	0.008	0.018	0.006
14 mm sea-dredged gravel and sand (B)	9	0.08	0.05	0.18	0.18	0.017	0.062	0.000
5 mm medium grey limestone crushed sand/shell mixture (E)	9	0.28	0.03	0.14	0.14	0.012	0.049	0.014
14 mm blastfurnace slag (A)	9	0.52	0.04	0.28	0.30	0.015	0.100	0.034
28 mm colliery shale (C)	9	0.80	0.04	0.50	0.57	0.015	0.177	0.098

NOTE n is the number of laboratories whose results were included in the calculation of the precision data. Definitions of $\bar{x}$, r_1, R_1, R_2, V_{r1}, V_L and V_S are given in **5.2** of BS 812-101:1984.

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Table 5 — Results excluded from calculation of precision data in Table 3

Laboratory	Aggregate	Laboratory average	Between-sample range	Between-test-portion range	
				Laboratory sample 1	Laboratory sample 2
L2	C		Outlier		
L3	A	Also excluded			
L3	B	Outlier			
L3	C	Outlier			
L3	D	Also excluded			
L3	E	Outlier			
L4	E			Outlier	
L7	D			Outlier	
L9	A		Outlier (spoiled samples)		

Table 6 — Results excluded from calculation of precision data in Table 4

Laboratory	Aggregate	Laboratory average	Between-sample range	Between-test-portion range	
				Laboratory sample 1	Laboratory sample 2
L1	A			Outlier	
L3	A	Outlier			
L3	B	Also excluded			
L3	C	Also excluded			
L3	D	Outlier			
L3	E	Outlier			
L10	C				Outlier

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Publications referred to

BS 410, *Specification for test sieves.*
BS 812, *Testing aggregates.*
BS 812-2, *Methods for determination of physical properties*[7].
BS 812-3, *Methods for determination of mechanical properties*[7].
BS 812-4, *Methods for determination of chemical properties*[7].
BS 812-101, *Guide to sampling and testing aggregates.*
BS 812-102, *Methods for sampling.*
BS 812-117, *Method for determination of water-soluble chloride salts.*
BS 1797, *Tables for use in the calibration of volumetric glassware.*
BS 5497, *Precision of test methods.*
BS 5497-1, *Guide for the determination of repeatability and reproducibility for a standard test method by interlaboratory tests.*
BS 5781, *Measurement and calibration systems.*
BS 5781-1, *Specification for system requirements.*
BS 8110, *Structural use of concrete.*
BS 8110-1, *Code of practice for design and construction.*

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[7] Referred to in the foreword only. Under revision.

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