

Determination of Total Sulphate of Aggregate

Lab Test Reference 011

British Standard Reference

BS 812 Part 118 : 1988 Test 6

Principal Apparatus

- i. A thermostatically controlled drying oven capable of maintaining a temperature of 100°-110°C -
Lab Inventory No. xxx.
- ii. Analytical Balance capable of weighing up to 5kg accurate to 5g
Lab Inventory Number xxx.
- iii. BS410 Test Sieves 20mm, 5mm, 1mm and 150 micron
- iv. Mortar and Pestle
- v. Analytical Balance capable of weighing up to 100g accurate to 0.001g
Lab Inventory Number xxx
- vi. 250ml and 500ml glass beakers
- vii. Hotplate
- viii. Two filter funnels of approximately 100mm dia with Whatman No. 40 and No. 42 filter papers of appropriate diameter.
- ix. 10ml dropping pipette
- x. Sintered silica filtering crucibles, porosity grade 4, approximately 35mm in diameter and 40mm in height.

NB These are not required if filtration through No. 42 filter papers is preferred.

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

NB These are not required if the filtering crucibles are used.

- xii. A muffle furnace capable of reaching and maintaining 800°C.
- xiii. Desiccator

Reagents

Reagents of recognised analytical quality and only distilled water shall be used.

1. Barium chloride 5% solution made by dissolving .50 grms of barium chloride in 1 litre of water and filtered before use if necessary.
2. Dilute Hydrochloric Acid, made by diluting 100ml of concentrated hydrochloric acid (SG1.18) to 1 litre of water.
3. Silver Nitrate Solution, made by dissolving 0.5g of silver nitrate in 1000ml of water. Store the solution in an amber coloured reagent bottle.

1.0 Preliminaries

- 1.1 The chemistry laboratory shall be used to perform this test and clear working areas of bench must be prepared before testing proceeds.
- 1.2 Ensure the sample number and test schedule correspond.
- 1.3 Obtain the appropriate test work sheet from the drawer.
- 1.4 Check the calibration status of the equipment and sieves.
- 1.5 Check the conditions of the sieves before testing commences.

2.0 Standard Test Method

2.1 Preparation of Test Portion

Dry the laboratory sample at a temperature of $105 \pm 5^{\circ}\text{C}$ to remove surface water so that the sample is free flowing.

- 2.2 Reduce the laboratory bulk sample using riffle boxes to the masses appropriate to the nominal maximum size of aggregate give below:-

Nominal Max Size of Aggregate (mm)	Minimum Mass of Sub-Sample (kg)
63	50
50	35
40	15
28	5
20	2
14	1
10	0.5
5 or less	0.2

- 2.3 Sieve the sub-sample through a 20mm sieve and crush any oversize aggregate to pass the sieve. Combine, mix thoroughly and where the mass of the sub-sample exceeds 2kg reduce the sub-sample to 2kg.
- 2.4 Dry the sub-sample by heating at a temperature of $100-110^{\circ}\text{C}$ to achieve a constant mass to within 0.1%. Overnight drying is usually sufficient.

- 2.5 Crush all aggregate to pass a 5mm sieve, mix and reduce to not less than 200g. Crush all the aggregate to pass a 1mm sieve and produce a sample of approximately 100g. Grind all of this to pass a 150 micron sieve. This fraction now represents the test portion. (All sample reduction is carried out using clean, appropriately sized riffle boxes).

NOTE Suitable grinding equipment may produce fines which will eliminate some of the intermediate stages.

2.6 Preparation of Acid Extract

Weigh accurately to the nearest 0.001g about 3g of the test portion and record as mass M1g. Place it in a 250ml beaker, and add 100ml of dilute hydrochloric acid and stir.

- 2.7 Repeat with a second duplicate 3g of the test portion and follow the procedure for both.

NOTE 1. Add acid slowly to aggregates containing carbonates to prevent frothing.

NOTE 2. Note whether hydrogen sulphide is given off (characteristic smell).

- 2.8 If the aggregates contain sulphide, place 100ml of the dil HCL in a 250ml beaker and heat to boiling point. Remove from the hotplate and while stirring the acid solution, sprinkle the weighed analytical portion of about 3g mass on to the acid.

- 2.9 Heat to boiling and simmer gently for 15 minutes in a fume cupboard.

- 2.10 Filter through a Whatman No. 40 filter paper, ensuring that the filtrate is clear. If not re-filter using a Whatman No. 42 filter paper and then wash thoroughly with hot distilled water.

- 2.11 Collect the filtrate and washings in a 500ml beaker and dilute to 300ml with distilled water.

- 2.12 Boil and add 10ml of BaCl₂ solution 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 minutes. Leave to cool for 24 hours.

- 2.13 Weigh an ignited and weighed sintered filter crucible if using this apparatus and transfer the precipitate of barium sulphate very carefully using suction. If a glass funnel and filter paper are being used, again transfer the precipitate very carefully into the filter papers by washing with hot distilled water.

- 2.14 Whichever method is chosen the precipitate is washed until the washings are free from turbidity when tested with a drop of silver nitrate solution.

- 2.15 If a sintered crucible is used, remove it from the filter flask and dry at 100-110°C for about 30 minute and then transfer to the muffle furnace where the temperature gradually rises to 800°C. (15 minutes is usually adequate to obtain constant mass).

- 2.16 Cool the crucible in a desiccator and weigh to the nearest 0.001g and calculate the mass of the precipitate M2 from the increase in mass of the crucible.

2.17 If the precipitate is filtered through a filter paper, transfer the filter paper and precipitate to a previously ignited and weighed crucible in a desiccator and weigh as described in para 2.16.

3. Calculations

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

$$\%SO_3 = \frac{M2 \times 34.3}{M1}$$

4. Reporting

4.1 Express the mean total sulphate content of the two test portions to the nearest 0.01% (as SO³) of the dry mass of aggregate. Repeat the test starting with new 3g analytical portions if the individual results differ by more than 0.5% (SO₃).