

Determination of Sulphate Content of Concrete

Lab Test Reference 314
British Standard Reference
BS 1881 Part 124 : 1988 Test 10.3

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 5mm, 2.36mm and 150 micron, 600 micron
- iv. Mortar and Pestle
- v. 250ml and 500ml glass beakers
- vi. Hotplate
- vii. Two filter funnels of approximately 100mm dia with ashless filter papers of appropriate diameter.
- viii. 10ml dropping pipette
- ix. Ignition Cubicle
- x. Muffle furnace capable of reaching 800°C
- xi. Desiccator

Reagents

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

1. Barium chloride solution 100g/1: Dissolve 100grms of barium chloride in 1 litre of water.
2. Dilute Hydrochloric Acid, (1+49).
3. Ammonium Hydroxide Solution (1+1).
4. Conc. hydrochloric acid (rel. dens. 1.8).
5. Methyl red indicator solution. Dissolve 1g of methyl red in 600ml of methylated spirits and add 400ml of water.

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

Break up the sample into lumps not larger than about 50mm in size avoiding as far as possible fracture of the aggregate. Dry the lumps in the oven at 100-110°C for 15 to 24 hours and allow to cool to room temperature.

- 2.2 Divide the dried sample into two portions A and B so that portion A is approximately three quarters of the bulk and portion B is the remainder. Ensure as far as possible that each portion remains representative of the whole.
- 2.3 Break portion B by any suitable method down to small pieces and remove any fine material produced by passing through a 150 micron sieve. The aggregate in the concrete cannot be damaged so grinding is not recommended.
- 2.4 Repeat this procedure until sufficient fines passing the sieve has been produced to enable the test to proceed.

2.5 Analytical Procedure

Weigh into a 400ml beaker, 5 ± 0.005 g of the separated sample. Retained in the base pan after sieving (M_d).

- 2.6 Disperse with 50ml of water and add 10ml of conc HCL. If effervescence is considerable, immediately cover the beaker with a watchglass.
- 2.7 Add 50ml of hot water, cover the beaker and boil the solution gently for 5 to 10 mins.
- 2.8 Filter through a medium ashless filter paper using the vacuum apparatus and filter funnel washing the residue thoroughly with hot dilute HCL.
- 2.9 Add three drops of the methyl red indicator and heat the filtrate to boiling. Just neutralise to yellow with dilute ammonium hydroxide solution.

- 2.10 Immediately add 1ml of conc. HCL and then add dropwise to the boiling solution 10ml of the barium chloride solution. If excess ammonium hydroxide was added, 1ml conc HCL may not be sufficient to obtain the required acid solution and the barium sulphate precipitate will then be contaminated. In this case the test shall be repeated.
- 2.11 Boil gently for 5 mins keep the solution at just below boiling for a further 30 mins and then allow to stand at room temperature for 12 to 24 hours.
- 2.12 Filter through a slow ashless filter paper and wash free from chlorides with hot water. Test the washings for milkiness with silver nitrate.
- 2.13 Weigh a clean empty ignition crucible and record as $M^1(g)$.
- 2.14 Transfer the paper and contents to a weighed ignition crucible and burn off the paper without flaming by placing in the muffle furnace at 800oC until constant mass is achieved (M_2)g.
3. Calculations
- 3.1 Calculate the total sulphate content G, expressed as SO_3 as a percentage of the cement from the equation.
- $$G = \frac{M_2 - M_1}{M_d} \times 34.3 \times 100 \quad \text{Where } C_1 \text{ is \% cement}$$
4. Reporting
- 4.1 The sulphate content G (as SO_3) is expressed as a percentage of the cement to the nearest 0.1% (m/m³).