

DETERMINATION OF SULPHATE CONTENT OF ROAD SALT

Lab Test Reference 10
British Standard Reference BS3247 : 1984

Principal Apparatus:- Use the same apparatus as described on

P.188 with additionally a 500ml volumetric flask
Oven - Invent xxx (BS2648)

Reagents:-These must be of recognised analytical quality and only distilled water shall be used.

- 1 Barium Chloride 5% solution made by dissolving 50g of barium chloride 1 litre of water and filtered before use if necessary.
- 2 5N Hydrochloric Acid.
- 3 Silver Nitrate Solution.
- 1 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 worksheet from the drawer.
 - 1.4 Check the calibration status of the equipment and sieves.
 - 1.5 Check the condition of the sieves before testing commences.
- 2 Standard Test Method
 - 2.1 Preparation of Test Portion

Obtain a bulk sample of about 15Kg of representative material by means of increments taken from the stock in accordance with BS812 - Part 102.
 - 2.2 Using appropriately sized riffle boxes, subdivide until about 3Kg is obtained from which a specimen of not less than 500g is taken.
 - 2.3 Wash this sub sample with methylated spirits to remove the moisture, spread out in a shallow tray to a depth not exceeding 10mm and dry in the oven set at 55-65°C for 2 hours.
 - 2.4 Allow the sample to cool at 15° to 20° C for 1 hour.

NBWhilst the sample is drying, open the windows, switch on the fume cabinets and vacate the room.
 - 2.5 Weight 50 + 0.01g of the sample and record the weight as M¹(g).
 - 2.6 Dissolve this in about 400ml of distilled water in the volumetric flask and when completely dissolved make up a 500ml with further distilled water. Shake the flask to ensure thorough mixture.

- 2.7 Set up a filter funnel connected to vacuum apparatus and flask, insert an appropriately sized filter paper of medium porosity and filter 50ml of the solution, measured by pipette. Wash the filter paper with distilled water and make up the filtrate and washings to 100ml in a volumetric flask using further distilled water.
- 2.8. Neutralise with 5N HCL and add 1ml in excess
- 2.9 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 30mins. Leave to cool for 24 hours.
- 2.10 Weight an ignited and weighed sintered filter crucible (M₂) and transfer the precipitate of barium sulphate very carefully using suction. The precipitate is washed until the washings are free from turbidity when tested with a drop of silver nitrate solution.
- 2.11 Remove the sintered crucible from the filter flask and dry at 100-110°C for about 30 mins and transfer to the muffle furnace allowing the temperature to gradually rise to 800°C. (Leave in for at least 15 mins).
- 2.12 Cool the crucible in a desiccator and weigh to the nearest 0.001g (M₃) and calculate the mass of precipitate from the increase in mass of the crucible.
- 3 Calculations
- 3.1 Calculate the sulphate content (as Calcium Sulphate) as a percentage by mass of the dry road salt from the equation.

$$\% \text{ SO}_3 = M_3 - M_2 \times \frac{1}{0.583}$$

M₁