Test Method

Determination of the Total Sulphur Content of Rock or Soil

RC 353.08

1. Scope

This method is used to determine the total sulphur content (expressed as sulphur trioxide SO_3 of rock or natural soil.

2. Apparatus

(a) Splitters - as in AS 1141.2

(b) Sieve - 200 mm diameter, AS 150 μm

(c) Muffle furnace with temperature gauge.

(d) Gas burner capable of producing a temperature of 1000°C.

(e) Steam bath.

(f) Sand bath on hotplate

(g) Platinum crucible with lid - 50 mL capacity

(h) Balance with limit of performance not exceeding ± 0.5 g.

(i) Analytical balance with limit of performance not exceeding ± 0.0005 g.

(j) Ovens thermostatically controlled and preferably mechanical ventilated to operate at a temperature:

  (i) not exceding 80°C;

  (ii) 105-110°C.

(k) Desiccator.

(l) Screw-top plastic jar of about 2 L capacity, plastic and glass beakers, glass wash bottle, stirring rod, rubber policeman, dishes and trays

(m) Filter funnel with flask.

(n) 500 mL beakers

(o) Filter papers - Whatman grades No. 42 and 540 (hardened)

3. Reagents

All reagents are to be of recognized analytical reagent quality.

(a) Barium Chloride 5% solution: dissolve 50 g of barium chloride ($BaCl_2$,$2H_2O$) in 1 litre of distilled water.

(b) Silver Nitrate Solution: 1% in distilled water.

(c) Sodium Carbonate Solution: 1% in distilled water.

(d) Methyl Red Solution: 0.1% in ethanol.

(e) Hydrochloric Acid: dilute 1:1

4. Sample Preparation

(a) Obtain a bulk sample of the material to be tested using the appropriate procedure detailed in AS 1141.3.1, 3.2, AS 1289.1.2.1 or RC 300.03

(b) As the test procedure requires a test sample that is finer that 150 μm, using procedures detailed in AS 1141.3.1 or Test Method RC 301.01, reduce the mass of the bulk sample as appropriate by crushing, splitting or quartering, and grinding or milling, to provide a representative sample of approximately 50 g which all passes the 150 μm AS sieve.

5. Procedure

(a) Oven dry the well-mixed, representative sample at a temperature not exceeding 80°C (see Note 1).

(b) Obtain a test sample of about 1 g and place in a platinum crucible of known mass. Determine and record the mass of the test sample to the nearest 0.1 mg.

(c) Add about 8 g of sodium carbonate to the test sample and mix thoroughly. No unmixed rock or soil powder must remain in the crucible.

(d) Gently heat the crucible, with lid slightly ajar, over a flame until all moisture is driven off. Gradually raise the temperature until the contents fuse and melt. Swirl the contents of the crucible occasionally to bring the denser particles into the melt (see Note 2).

(e) When fusion is complete, remove the crucible from the flame and swirl the contents until the melt solidifies as a thin layer. Once the red-heat has dissipated, dip the crucible to two-thirds of its depth in distilled water until cold.

(f) Reheat the crucible to about 300-400°C and again dip it into distilled water and allow to cool completely

(g) Transfer, without loss, the solidified melt fragments into a plastic beaker, all solid material still adhering to the crucible being dislodged with hot water and by scrubbing with a rubber policeman.

(h) Dilute to about 150 mL with hot distilled water and place on steam bath until the solids are completely disintegrated and in suspension.

(i) Transfer the solution to a Whatman hardened No. 540 filter paper in the glass funnel and filter into a 400 mL glass beaker. Wash the precipitate well with hot 1% sodium carbonate solution. The solution plus the washings should make up about 250 mL.

(j) Add a few drops of methyl red and acidify the solution with 1:1 hydrochloric acid until the methyl red endpoint is reached. Then add 5mL of the acid in excess.

(k) Bring the solution to the boil and boil for 2 minutes to expel any carbon dioxide then add, with stirring, 20 mL of the barium chloride solution. Boil for five Minutes.

(l) Place above steam bath for at least 2 h then allow the, solution to cool, preferably overnight (particularly if it is known that the sulphate content is low).

(m) Transfer, without any loss, the liquid and precipitate to a Whatman No. 42 filer paper in the glass funnel.

(n) Wash the precipitate several times with cold water until the washings are essentially free from chloride, as indicated by only slight opalescence of the filtrate when a few drops are tested with 1% silver nitrate solution. Do not overwash.

(o) Transfer the filter paper and precipitate to a platinum crucible of known mass and char and consume the paper slowly, without flaming, over the flame of a burner until it is apparent that all carbon has been consumed.

(p) Ignite the residue in a muffle furnace at approximately 900°C for 1 h.

(q) Cool in a desiccator for 10 min and weigh the barium sulphate residue (see Note 3).

6. Calculation

Calculate the sulphur (expressed as SO_3) present in the test fraction as follows:

$$SO_3 = \frac{mass\ of\ ignited\ precipitate\ (g) \times 0.343}{mass\ of\ test\ fraction\ (g)} \times 100$$

7. Reporting

Report the percentage by mass of sulphur (expressed as SO_3) present in the rock or soil sample to the nearest 0.01.

Notes:

Note 1

Soils containing sulphates in the form of gypsum lose water of crystalisation if heated above the specified temperature

Note 2

With some samples a clear melt is obtained after a few minutes indicating that fusion is complete; with others, the melt remains turbid and completion of fusion is indicated when no grains of powder adhere to the bottom of the crucible.

Note 3

If there is any suspicion that the ignited precipitate is contaminated with silica (as suggested by the precipitate not being heavy and granular and/or remaining too long in suspension), add a drop of 1:1 sulphuric acid and a few drops of hydrofluoric acid, evaporate the acids on the steam bath, ignite and reweigh.

Test Method - Revision Summary

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Date	Clause Number	Description of Revision	Authorised by
June 2012	Full document	Re-styled with minor corrections made	Principal Advisor – Pavements & Materials