Test Method

Determination Of The Water-Soluble Sulphate Content Of Crushed Rock

RC 353.07

1. Scope

This method is used to determine the water-soluble sulphate content of the finer than 4.75 mm portion of crushed or ripped rock pavement material. The result obtained gives the sulphate content at the time of testing.

Note: Exposure of the material to the atmosphere may result in further oxidation of any sulphide minerals present to form sulphate salts due to oxidation of any mineral still in the sulphide form.

2. Apparatus

(a) Splitters as in AS 1141.2.

(b) Sieve - 300 mm diameter, AS 4.75 mm and pan.

(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 a limit of performance not exceeding ± 0.5 g

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

(j) Ovens - thermostatically controlledand preferably mechanically ventilated to operate at a temperature of:

  (i) not exceeding 80°C; and

  (ii) 105-110°C.

(k) Desiccator

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

(m) Filter funnel with flask.

(n) Filter papers - Whatman grades No. 40 and 42.

3. Sample Preparation

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

(b) By splitting or quartering, obtain a representative sample of the appropriate mass as detailed in AS 1141.11.

(c) Separate the sample over the 4.75 mm sieve and discard the material retained on the sieve.

(d) By splitting or quartering the material retained in the sieve pan, obtain a test fraction of mass between 300 and 500 g.

4. Procedure

(a) Oven dry the test fraction to constant mass (105 - 110°C)

(b) Weigh the test fraction to the nearest 0.1 g and record.

(c) Transfer, without any loss, the test fraction to a plastic jar and add to it a mass of distilled water equal to the mass of the test sample.

(d) Secure the top of the jar and shake the jar thoroughly three times during 1 h.

(e) Allow to settle and decant the supernatant solution.

(f) Transfer a portion of the solution to a Whatman No. 40 filter paper in the glass funnel and allow to filter.

(g) Measure into a beaker an aliquot of the filtered solution such that it contains 10 to 50 mg of sulphate ion, dilute to 250 mL with distilled water then add 5 mL of 1:1 hydrochloric acid.

(h) Bring the solution to the boil and add, while stirring, 20 mL of the barium chloride solution. Boil for 5 min.

(i) 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).

(j) Transfer the precipitate, without any loss, to a Whatman No. 42 filter paper in the glass funnel.

(k) 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.

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

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

(n) Cool in a desiccator for 10 min and weigh the barium sulphate residue.

5. Calculation

Calculate

(a) The mass of the precipitate (m_p) obtained (to the nearest 0.001 g) from the increase in mass of the crucible

(b) The sulphate (as $SO_4^{=}$) present in the original test fraction.from:

$$Percent\ SO_4^{=} = \frac{m_p \times 0.4115}{V_a} \times 100$$

where V_a = the volume of aliquot in mL.

m_p = mass of ignited precipitate in g.

6. Reporting

Report the percentage by mass of sulphate ($SO_{4=}$) present in the crushed rock fraction passing the 4.75 mm AS sieve to the nearest 0.01.

Test Method - Revision Summary

RC 353.07 Determination Of The Water-Soluble Sulphate Content Of Crushed Rock

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