



Test method T218

Total sulphur content in metallurgical slag, crushed rock or other pavement materials

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Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
		Reformatted and Revision Summary Added	D.Dash	May1999
		Date on Test Method Revised to Agree with Date on Revision Summary	D.Dash	Feb 2001
Ed 2/ Rev 0	All	Reformatted RMS template	J Friedrich	October 2012

Note that Roads and Maritime Services is hereafter referred to as 'RMS'.

The most recent revision to Test method T218 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T218

Total sulphur content in metallurgical slag, crushed rock or other pavement materials

1. Scope

This test method sets out the procedure for determination of the sulphur content in metallurgical slag, crushed rock or other pavement materials. The method conforms in general to the method described in Australian Standard AS1141.

2. Reagents

All chemicals are to be accepted analytical grade purity. Distilled or deionized water to be used throughout the procedure.

- (a) Hydrochloric Acid (HCl) ($d = 1.16 \text{ g/mL}$)
- (b) Nitric Acid (HNO₃) ($d = 1.42 \text{ g/mL}$)
- (c) Ammonium hydroxide (NH₄OH) ($d = 0.90 \text{ g/mL}$)
- (d) Bromine (Br₂)
- (e) Methyl red. Dissolve 0.1 g methyl red in 3.7 mL of NaOH and dilute to 250 mL with water
- (f) Sulphuric Acid (H₂SO₄) ($d = 1.84 \text{ g/mL}$)
- (g) Solutions. Dilute 1 volume of the reagent with the stated number of volumes of water. For example, HCl (1:2)
- (h) Barium Chloride (BaCl₂) solution (100 g/L)

3. Apparatus

- (a) A thermostatically controlled oven, with good air circulation capable of maintaining a temperature within the range of 105°C to 110°C
- (b) An analytical balance readable and accurate to 0.2 mg within the operating range
- (c) Porcelain mortar, approximately 180 mm dia, with porcelain pestle
- (d) 2.36 mm, 600 μm and 150 μm AS sieves
- (e) Sieve brushes
- (f) Steam bath
- (g) Hot plate
- (h) Laboratory glassware including beakers, flasks, porcelain crucibles, filter funnels centrifuge tubes etc
- (i) Filter paper
- (j) A laboratory crusher capable of crushing the slag, crushed rock or other pavement materials to pass 2.36 mm sieve
- (k) Fume cupboard
- (l) Desiccator
- (m) Centrifuge, capable of at least 2000 rpm
- (n) Muffle Furnace capable of maintaining temperature at 800°C
- (o) Water operated vacuum pump and tubing

4. Preparation

- (a) Dry a representative portion of the sample (about 500 g) at 105°C to 110°C and crush so that all material passes a 2.36 mm sieve.
- (b) Mix the portion and sub-divide by quartering or riffing to obtain a portion of about 100 g. Grind so that all material passes a 600 µm sieve.
- (c) Reduce the portion by quartering or riffing to obtain a portion of about 20 g and grind so that all material passes a 150 µm sieve.
- (d) Thoroughly mix the material passing 150 µm sieve and dry at 105°C to 110°C and store in an airtight container for analysis.

5. Procedure

CAUTION: Procedure (a)-(c) should be performed in the fume cupboard.

- (a) Weigh about 1g of the test portion to the nearest 0.2 mg and place in a wide mouthed CO₂ flask. Add 3 mL of water and 1 mL of Br₂. Insert a short-stemmed funnel into the flask and gently agitate the mixture for about 1 minute to prevent undue formation of lumps.
- (b) Slowly add through the funnel 15 mL of HNO₃ and heat on a steam bath for 1 hour, breaking up the gel at intervals with a flattened glass rod. Add 30 mL of water and gently boil on a hotplate until evolution of dense brown fumes ceases.

CAUTION: Nitric acid is corrosive, handle with care. Safety glasses must be worn when handling acid.

- (c) Add 15 mL of HCl (1:2) and boil down to a small bulk. Add a further 15 mL of HCl (1:2) and again boil down to a small bulk. Transfer the contents of the flask to a 250 mL beaker and wash with water until the total volume in the beaker is about 100 mL. Add a little filter paper pulp and bring contents of the beaker almost to the boil.

CAUTION: Hydrochloric acid is corrosive, handle with care. Safety glasses must be worn when handling acid.

- (d) Make alkaline to methyl red by means of NH₄OH (1:3). Simmer for 0.5 minutes, filter under gentle suction using a filter paper of medium porosity and wash once with a little hot water, reserving the filtrate. Transfer the filter paper back into the beaker, add 5 mL of HCl and 70 mL of water and boil. Repeat the process of precipitation with NH₄OH (1:3) filtering and washing, reserving the filtrate and rejecting the precipitate.
- (e) Combine the filtrate and washings (which should now total about 220 mL). Acidify with HCl, bring to the boil and add drop by drop 10 mL of BaCl₂(100g/l). Keep the mixture at about 30°C for 3 to 4 hours.
- (f) Alternative (1) - Filter off the BaSO₄ through a small double-fine grade paper, wash with hot water until the washings are free from chloride. Dry the paper and precipitate and heat slowly to 800°C in a tared crucible avoiding ignition of the filter paper. Cool in a desiccator and moisten the precipitate with a few drops of concentrated H₂SO₄. Reheat the precipitate slowly to 800°C. Cool in a desiccator and determine the mass.

CAUTION: Sulphuric acid can cause severe burns. Avoid contact with eyes, skin and clothing. Safety glasses must be worn when handling acid.

- (g) Alternative (2) - Oven dry the centrifuge tubes, cool in a desiccator and weigh. Centrifuge the mixture at speed of at least 2000 rpm for a minimum period of 5 minutes. Decant the clear supernatant liquid and repeat the centrifuging process until all the mixture has been processed. Wash and centrifuge the precipitate till the decanted wash is free from chlorides. Oven dry, cool in a desiccator and determine the mass of Barium Sulphate.
- (h) Make a blank determination, following procedures (a) to (e) and the alternative (f) to (g), using the same amounts of all reagents.

- (i) In the event of a dispute between the supplier and purchaser the method described in (f) shall be used in any subsequent retests.

6. Calculations

- (a) Calculate the percentage of sulphur present as follows:

$$\text{S percent} = \frac{(A - B) \times 0.1374 \times 100}{C}$$

Where

- A = Mass of BaSO₄(g)
B = Correction for blank, in grams
C = Mass of test portion used (g)

7. Reporting

Report the sulphur content in metallurgical slag, crushed rock or other pavement materials to the nearest 0.02%.