

Australian Standard®

AS 1141.36:2017

Methods for sampling and testing aggregates
Method 36: Sulfur in metallurgical slag, crushed rock or other pavement materials

This Standard incorporates Amendment No. 1 (August 2019). The changes required by the Amendment are indicated in the text by a marginal bar and amendment number against the clause, note, table, figure or part thereof affected.

WARNING: SOME OF THE CHEMICALS OR PROCEDURES SPECIFIED IN THIS STANDARD COULD LEAD TO A HAZARDOUS SITUATION IF CORRECT SAFETY MEASURES ARE NOT FOLLOWED. PERSONS USING THIS AUSTRALIAN STANDARD NEED TO BE FAMILIAR WITH NORMAL LABORATORY PRACTICE. THIS STANDARD DOES NOT ADDRESS THE SAFETY PROBLEMS, IF ANY, ASSOCIATED WITH ITS USE. IT IS THE RESPONSIBILITY OF THE LEGAL ENTITY USING THIS STANDARD TO ESTABLISH APPROPRIATE SAFETY AND HEALTH PRACTICES, TO ENSURE COMPLIANCE WITH ANY NATIONAL, STATE OR LOCAL REGULATORY REQUIREMENTS, AND TO INSTRUCT IN AND ENFORCE COMPLIANCE OF THE SAFETY AND HEALTH PRACTICES.

1 SCOPE

This Standard sets out the method for the determination of the amount of sulfur in metallurgical slag, crushed rock or other pavement materials expressed as total sulfur content and acid soluble sulfate content. It includes a test for total sulfur and a test for acid soluble sulfate. The method assumes that personnel conducting the procedure are trained and competent in safely conducting chemical tests and in the preparation of standard solutions.

2 PRINCIPLE

In the total sulfur test, sulfur is oxidized and brought into solution as sulfate. Soluble silica is precipitated by boiling with acid. Mixed trioxide (R_2O_3) contamination is removed by precipitation and filtration. Sulfur present is precipitated as barium sulfate, the mass of which is then determined. The content of sulfur containing compounds is expressed as percentage total sulfur (percent S).

Acid soluble sulfate is determined by a similar procedure to the total sulfur procedure; however, the oxidation step is omitted. Acid soluble sulfate content is expressed as percentage sulfur trioxide (percent SO_3 , sulfite).

3 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

- 1141 Methods for sampling and testing aggregates
- 1141.1 Part 1: Definitions
- 1141.2 Method 2: Basic testing equipment

AS

A1 | [Text deleted]

2243 Safety in laboratories

2243.8 Part 8: Fume cupboards

4 DEFINITIONS

For the purpose of this Standard the definitions in AS 1141.1 apply.

5 SAFETY PRECAUTIONS

Procedures using concentrated mineral acids, including the preparation of dilute solutions of these acids, should be carried out in a fume cupboard.

WARNING: CONCENTRATED ACIDS AND BROMINE ARE EXTREMELY HAZARDOUS AND CAN RESULT IN OFTEN IRREVERSIBLE DAMAGE TO THE SKIN OR EYES. INGESTION OF THE CHEMICALS, INHALATION OF THE VAPOUR, OR SKIN CONTACT CONSTITUTES A SERIOUS HEALTH HAZARD.

Appropriate documentation should be consulted as to safety precautions.

6 APPARATUS

A1 | The following apparatus, conforming to the relevant provisions of AS 1141.2 and the requirements given below, is required:

- (a) *Analytical balance*—with a limit of performance not exceeding ± 0.5 mg.
- (b) *Bath*—a steam bath.
- (c) *Bunsen burner*.
- (d) *Container*—airtight, for storage of test portion.
- (e) *Crucible*—platinum.
- (f) *Crushing equipment*—to crush to pass 2.36 mm, 600 μ m and 150 μ m sieves.
- (g) *Desiccator*—with dried silica gel.
- (h) *Glassware*—beakers, flasks and other items, as necessary.
- (i) *Oven*—thermostatically-controlled, complying with AS 1141.2 and capable of operating at a temperature within the range of 105°C to 110°C.
- (j) *Muffle furnace*—capable of maintaining a temperature of 800°C.
- (k) *Hotplate*.
- (l) *Fume cupboard*.
NOTE: Advice on suitable fume cupboards can be found in AS 2243.8.
- (m) *Centrifuge*—capable of at least 2000 rpm.
- (n) *Water-operated vacuum pump*.
- (o) *Filter paper*—white double fine grade.
- (p) *Sample divider*—complying with AS 1141.2.
- A1 | (q) *Sieves*—in woven wire, aperture sizes 2.36 mm, 600 μ m and 150 μ m.

7 REAGENTS

All reagents shall be of analytical grade. Distilled or de-ionized water shall be used for all stages of the test. Reagents shall be specified by name or formula and shall consist of the following:

- (a) *Hydrochloric acid* (HCl) (density = 1.16 kg/L).
- (b) *Nitric acid* (HNO₃) (density = 1.42 kg/L).
- (c) *Ammonium solution* (NH₃, 1:3) (density = 0.90 kg/L).
- (d) *Methyl red* Dissolve 0.1 g methyl red in 3.7 mL of 0.1 Mol per litre solution NaOH and dilute to 250 mL with water.
- (e) *Sulfuric acid* (H₂SO₄) (density = 1.84 kg/L).
- (f) *Barium chloride* (BaCl₂) solution (0.1 kg/L).
- (g) *Solutions* Dilute the volume of the reagent with the stated volume of water, for example, HCl (1:2).
- (h) *Bromine* (Br₂).

8 PREPARATION OF SAMPLE FOR TEST

The procedure for preparation of the test sample shall be as follows:

- (a) Dry a representative portion of the sample (about 500 g) at 105°C to 110°C and crush so that it will all pass a 2.36 mm test sieve.
- (b) Thoroughly mix and subdivide by quartering or riffing to obtain a portion of about 100 g. Grind this portion so that it will all pass a 600 µm test sieve.
- (c) Again thoroughly mix and subdivide by quartering or riffing to obtain a portion of about 20 g and grind the portion so that it will all pass a 150 µm test sieve.
- (d) Mix thoroughly, dry at 105°C to 110°C to constant mass and store in an airtight container as the test portion for analysis.

9 PROCEDURES

9.1 General

One or both of the procedures given in Clauses 9.2 and 9.3 shall be performed as required.

9.2 Total sulfur

The test procedure for total sulfur shall be as follows:

- (a) Measure out about 1 g of the test portion, the mass of which has been accurately determined to the nearest 0.1 mg and recorded as *C*, into 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 min to prevent undue formation of lumps.
- (b) Slowly add through the funnel 15 mL of HNO₃ of concentration as given in Clause 7 and heat on a steam bath for 1 h, 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.
- (c) Add 15 mL of HCl (1:2) and reduce by boiling down. Add a further 15 mL of HCl (1:2) and again reduce by boiling. 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.
- (d) Add two or three drops of the methyl red solution (already prepared). Make alkaline to methyl red by means of NH₄OH (1:3).