



Transport
Roads & Maritime
Services

Test method T1011

Quantitative determination of sulphates in soil

NOVEMBER 2012



Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
		Reformatted and Revision Summary Added	D.Dash	June 2001
Ed 2/ Rev 0	All	Reformatted RMS template	J Friedrich	November 2012

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

The most recent revision to Test method T1011 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T1011

Quantitative determination of sulphates in soil

1. Scope

This test method sets out the procedure for the determination of sulphates in soil by precipitation, as barium sulphate, from a solution of the soluble ions extracted from the soil. The method is derived from "Quantitative Inorganic Analysis" by Arthur I. Vogel.

2. Apparatus

- (a) A 2.36 mm woven wire AS sieve.
- (b) 1 litre plastic bottle with liquid tight seal.
- (c) A drying oven capable of maintaining a temperature of 75°-85°C
- (d) Filter paper, 110mm diameter (Whatman, No. 542 is suitable).
- (e) Electric muffle furnace capable of maintaining a temperature of 700°C, or other suitable means of igniting the precipitate.
- (f) Laboratory glassware including pipettes, beakers, measuring cylinder, etc.
- (g) Buchner funnel, about 120mm diameter and readable to 0.01 g.
- (h) Analytical balance of 200g capacity and readable to 0.0001g.
- (i) Balance of 2000g capacity and readable to 0.01g.
- (j) Porcelain crucible of 15-20 mL capacity.

3. Reagents

- (a) Ammonium nitrate A.R. Grade.
- (b) Hydrochloric acid AR grade
- (c) Picric Acid, AR grade, saturated solution
- (d) Barium chloride AR grade, 5% solution

4. Extraction of Soluble Salts

- (a) Dry the sample in the oven at 75°-85°C
- (b) Sieve the material to pass the 2.36mm sieve, breaking up by hand any aggregations of material.
- (c) Weigh to the nearest 0.1 g, 200 g of soil passing a 2.36 mm sieve into a suitable 1 litre plastic bottle.
- (d) Add exactly 500 mL distilled water and shake well for 1 minute (500 mL is conveniently measured using a volumetric flask).
- (e) Allow to stand for 1 hour with occasional shaking, making sure that the soil is well broken up.
- (f) After the soil has settled (up to 2 hours after the last shaking) pipette off a 100 mL aliquot into a beaker for analysis.

NOTE: The water layer above the soil should be substantially free from suspended matter, otherwise errors in volume will be introduced. If it is not clear, the addition of 0.2 g of ammonium nitrate after the first shaking is often helpful in settling out difficult soils. The solution must be clear for the determination of sulphates. In cases of obstinate turbidity the solution must be clarified by centrifuging

5. Procedure

- (a) Take the 100 mL aliquot from 4 (f) above and acidify slightly by adding 0.5mL concentrated hydrochloric acid.

CAUTION: HYDROCHLORIC ACID IS CORROSIVE, HANDLE WITH CARE. SAFETY GLASSES MUST BE WORN WHEN HANDLING ACID.

- (b) Add 10mL saturated picric acid solution followed by a slight excess of 5% barium chloride solution.

CAUTION: PICRIC ACID MUST ALWAYS BE KEPT MOIST. DO NOT ALLOW PICRIC ACID TO BE DEPOSITED AT THE MOUTH OF THE CONTAINER WHERE IT MAY DRY OUT AND/OR BE SUBJECT TO FRICTION WHEN REPLACING THE CONTAINER LID.

- (c) Cover the beaker with watch glass, gently boil the solution for 5-10 minutes and cool to ambient temperature.
- (d) Filter the solution in a Buchner funnel and thoroughly wash the precipitate with hot water till free of chloride.
- (e) Fold the filter paper with precipitate and place in a tared crucible. With a bunsen slowly char the filter paper without flaming then burn off the carbon at a low temperature with free access of air.
- (f) Ignite the crucible and precipitate for 30 minutes in the furnace at 700°C, remove, cool in a desiccator and weigh.

6. Calculations

Calculate the sulphate content in the original sample as follows:

$$\text{Sulphate Content (ppM } SO_4) = 0.4115 \times \frac{M_p}{M_s} \times \frac{V}{V_a} \times 10^6$$

Where M_p = mass of $BASO_4$ precipitate, g

Where M_s = mass of soil for extraction, g

Where V = volume water added to soil, mL

Where V_a = volume of aliquot, mL

7. Reporting

Report the sulphate content in ppM to the nearest whole number.