

Australian Standard®

AS 4969.6—2008

Analysis of acid sulfate soil—Dried samples— Methods of test

Method 6: Determination of acid extractable sulfur in soil residue after peroxide oxidation (S_{RAS})

PREFACE

This Standard was prepared by the Australian members of the Joint Standards Australia/Standards New Zealand for Committee EV-009, Sampling and Analysis of Soil and Biota, Working Group EV-009-02-01, Analysis of Acid Sulfate Soil,

The objective of this Standard is to provide a method for the determination of residual acid soluble sulfur (S_{RAS}) in acid sulfate soil after peroxide oxidation.

METHOD

1 SCOPE

This Standard specifies a method for the determination of residual acid soluble sulfur (S_{RAS}) in acid sulfate soil after peroxide digestion (AS 4969.3) and the determination of peroxide sulfur (S_p) (AS 4969.5).

In order to perform this method, AS 4969.3 and AS 4969.5 shall have already been performed on the same test portion.

NOTES:

- 1 This extraction procedure recovers the relatively insoluble iron and aluminium hydroxy sulfate compounds (e.g. jarosite, natrojarosite, basaluminite) which are not measured to any significant extent in S_p .
- 2 S_{RAS} , unlike S_{NAS} , (AS 4969.11) does not measure significant sulfur from organic matter as this is removed during rinsing of the soil residue. Some laboratories may prefer to perform S_{NAS} analysis for operational reasons.
- 3 This procedure would normally only be performed on a soil with a potassium chloride pH < 4.5 (pH_{KCl} , (AS 4969.2) or where jarosite/natrojarosite and similar minerals have been identified in the soil when it was sampled.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

- 2164 Laboratory glassware—One mark volumetric flasks
- 4969 Analysis of acid sulfate soil—Dried samples—Methods of test
- 4969.0 Part 0: Introduction and definitions, symbols and acronyms
- 4969.2 Method 2: Determination of pH_{KCl} and titratable actual acidity (TAA)
- 4969.3 Method 3: Determination of peroxide pH (pH_{OX}), titratable peroxide acidity (TPA) and excess acid neutralizing capacity (ANC_E)
- 4969.5 Method 5: Determination of peroxide sulfur (S_p), calcium (Ca_p) and magnesium (Mg_p)
- 4969.11 Method 11: Calculation of net acid-soluble sulfur (S_{NAS}), calcium (Ca_{NAS}) and magnesium (Mg_{NAS})

AS/NZS

- 2243 Safety in laboratories
- 2243.1 Part 1: Planning and operational aspects
- 2243.2 Part 2: Chemical aspects
- 2243.8 Part 8: Fume cupboards

ISO

- 3696 Water for analytical laboratory use—Specification and test methods

3 DEFINITIONS

For the purposes of this Standard the terms and definitions used in AS 4969.0 apply.

4 PRINCIPLE

After appropriate washing of the soil residue from AS 4969.5, the sulfur is extracted with 4 M hydrochloric acid then filtered or centrifuged to obtain a clear extract and the residual acid soluble sulfur (S_{RAS}) is measured by a suitable analytical technique.

5 REAGENTS

5.1 General

All reagents shall be of analytical grade (AR). Deionized or glass distilled water of Grade 2 as defined in ISO 3696 shall be used throughout.

The purity of all reagents for sulfur should be verified by performing a blank test. Reagents should also be tested for the presence of sulfur whenever a change in source is made (e.g. brand or batch).

5.2 Hydrochloric acid (ρ_{20}) 1.16 g/mL

WARNING: CONCENTRATED HYDROCHLORIC ACID IS A CORROSIVE AGENT. AVOID CONTACT WITH SKIN AND EYES. SAFETY GLASSES AND GLOVES AND OTHER SUITABLE PROTECTIVE CLOTHING AND FOOTWEAR SHALL BE WORN AND SHALL COMPLY WITH AS/NZS 2243, PARTS 1 AND 2.

5.3 Hydrochloric acid solution, 4 M

Add 400 mL of hydrochloric acid (5.2) with stirring to approximately 400 mL of water. Cool to room temperature, transfer to a 1 L volumetric flask and fill to the mark with water.

5.4 Potassium chloride solution, 1 M

Dissolve 74.55 g of KCl in water, transfer to a 1 L volumetric flask and fill to the mark with water.

6 APPARATUS

6.1 General

Grade A volumetric glassware shall be used throughout. Volumetric flasks shall comply with AS 2164.

6.2 Centrifuge

Capable of centrifuging 80 mL of suspension.

6.3 Dispenser (manual or automatic)

Capable of accurately dispensing 80 ± 0.5 mL.

6.4 Filter paper

Thick medium speed, high retention filter paper.

NOTE: Whatman No. 3 paper has been found to be suitable.

6.5 Sample bottle

Of 100 to 250 mL capacity to allow efficient mixing and also to minimize the headspace, made of polyethylene or other inert material, with a tightly fitting cap or stopper to prevent leakage.

NOTE: Sample bottle and stopper should be made of a material not containing sulfur.

6.6 Shaking or mixing machine

Capable of keeping soil particles continuously in suspension.

NOTE: For example end-over-end shaker.

7 PROCEDURE

7.1 Rinsing of peroxide digested soil residue, extraction with 4 M HCl and determination of extractable sulfur

The procedure shall be as follows:

- (a) Ensure that the entire suspension from Clause 7.1 in AS 4969.5 has been filtered, so that complete recovery of the soil residue is achieved.

CAUTION: TAKE CARE TO ENSURE THAT NO SOIL RESIDUE IS LOST IN THE FOLLOWING RINSING STEPS.

- (b) Rinse filter paper containing the soil residue with $(2 \times 10) \pm 1$ mL aliquots of 1 M KCl solution (5.4). Subject the filter papers from the blank solution to the same procedure.

NOTE: A pipette, piston operated volumetric apparatus (POVA) or other suitable apparatus may be used for rinsing.

- (c) Rinse filter paper containing the soil residue with $(4 \times 10) \pm 1$ mL aliquots of water.

NOTE: A pipette, piston operated volumetric apparatus (POVA) or other suitable apparatus may be used for rinsing.

- (d) Place filter paper containing the soil residue into the sample bottle (6.5).