

Australian Standard[®]

AS 4969.5—2008

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

Method 5: Determination of peroxide sulfur (S_P), calcium (Ca_P) and magnesium (Mg_P)

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 peroxide sulfur (S_P), calcium (Ca_P) and magnesium (Mg_P) in acid sulfate soil after hydrogen peroxide digestion and the determination of peroxide pH (pH_{OX}), titratable peroxide acidity (TPA) and excess acid neutralizing capacity (ANC_E).

METHOD

1 SCOPE

This Standard specifies a method for the determination of peroxide sulfur (S_P), calcium (Ca_P) and magnesium (Mg_P) in acid sulfate soil after hydrogen peroxide digestion and the determination of pH_{OX} , and TPA and ANC_E in AS 4969.3.

NOTES:

- 1 This method recovers soluble and exchangeable sulfate, sulfate from gypsum, sulfate formed by oxidation of sulfide, sulfur released by the breakdown of organic matter as well as, variable amounts of sulfate from iron and aluminium sulfate minerals or compounds. Sulfate from jarosite and natrojarosite is not appreciably recovered. Methods to determine the residual sulfur held in these iron hydroxy-sulfate minerals are detailed in AS 4969.6 and AS 4969.11.
- 2 The peroxide extraction procedure recovers soluble and exchangeable calcium and magnesium, and calcium from gypsum. Calcium and/or magnesium released by the acid dissolution of carbonate, oxide or hydroxide minerals are also recovered.
- 3 The peroxide sulfur (S_P) measurement can be used in combination with KCl extractable sulfur (S_{KCl} , AS 4969.4) to calculate the peroxide oxidizable sulfur (S_{POS} , AS 4969.10).
- 4 The peroxide calcium (Ca_P) and magnesium (Mg_P) can be used in combination with KCl extractable calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) (AS 4969.4) to determine 'reacted' calcium (Ca_A) and magnesium (Mg_A) (AS 4969.10).
- 5 The residual acid soluble sulfur (S_{RAS}) (AS 4969.6) can be determined on the entire soil residue following measurement of S_P .

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

- 4969 Analysis of acid sulfate soil—Dried samples—Methods of test
4969.0 Part 0: Introduction and definitions, symbols and acronyms
4969.3 Method 3: Determination of peroxide pH (pH_{OX}), titratable peroxide acidity (TPA) and excess acid neutralizing capacity (ANC_E)
4969.4 Method 4: Determination 1 M potassium chloride extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl})
4969.6 Method 6: Determination of acid extractable sulfur in soil residue after peroxide oxidation (S_{RAS})
4969.10 Method 10: Calculation of peroxide oxidizable sulfur (S_{POS}), reacted calcium (Ca_A) and reacted magnesium (Mg_A)
4969.11 Method 11: Calculation of net acid-soluble sulfur (S_{NAS}), calcium (Ca_{NAS}) and magnesium (Mg_{NAS})

ISO

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

3 DEFINITIONS

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

4 PRINCIPLE

The soil suspension from the peroxide digest of AS 4969.3 is diluted, followed by filtration or centrifugation and then peroxide sulfur (S_P), calcium (Ca_P) and magnesium (Mg_P) are measured by suitable analytical technique(s).

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, calcium and magnesium) should be verified by performing a blank test. Reagents should also be tested for the presence of these elements whenever a change in source is made (e.g. brand or batch).

6 APPARATUS

6.1 Beaker or other container

Of at least 450 mL capacity, made of polyethylene, glass or other inert material.

6.2 Centrifuge

Capable of centrifuging 400 mL of suspension.

6.3 Electronic balance

Capable of weighing up to 500 g to an accuracy of ± 0.01 g.

6.4 Filter paper

Thick medium speed high retention filter paper.

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

6.5 Stirrer

Mechanical stirring apparatus or stirring rod capable of stirring 400 mL of suspension.

6.6 Titration vessel

Of at least 100 mL capacity, made of polyethylene or other inert material.

NOTE: This is the titration vessel used in AS 4969.3.

7 PROCEDURE

7.1 Dilution and filtration of soil suspension following TPA titration

The procedure shall be as follows:

- (a) After completion of AS 4969.3, quantitatively transfer contents from the titration vessel (6.6) to a tared or weighed beaker (6.1) with water. Subject the solution blanks from AS 4969.3 to the same procedure.
- (b) Dilute the suspension with water to a final volume appropriate to the analytical instrumental technique.
NOTE: This can be achieved by using the balance (6.3), taking into account the mass of the container (6.1), density of the KCl matrix (which is dependent on the final volume chosen) and the mass of the test portion in AS 4969.3.
- (c) Homogenize the suspension with a mechanical stirrer or stirring rod (6.5).
- (d) Centrifuge (6.2) or filter (6.4) an appropriate volume of the homogenized soil suspension to obtain a clear solution. If measuring S_{RAS} (AS 4969.6) on the digested residue, the entire suspension shall be filtered.
- (e) Take volumes of centrifuged or filtered solution (sufficient for the instrumental technique used) and determine 'peroxide' sulfur (S_p), calcium (Ca_p) and magnesium (Mg_p) using appropriate analytical techniques. For sulfur measurement, instrumentation that specifically determines sulfate is preferable to that which measures total sulfur in solution.

NOTES:

- 1 An example of a technique that is specific to sulfate is Ion Chromatography (IC). To obtain accurate and reproducible results it is necessary to have an appropriate resin that will handle high levels of chloride introduced by the KCl solution matrix.
- 2 Instruments that determine all sulfur species in solution (e.g. ICP-AES) may give higher results than instruments that specifically measure sulfate. This is particularly the case in soils high in organic matter that may contain appreciable organic sulfur in solution following the peroxide digestion.
- 3 Codes for the analytical techniques used to determine sulfur, calcium and magnesium are shown in Appendix A.

8 CALCULATIONS

Calculate peroxide sulfur (S_p), calcium (Ca_p) and magnesium (Mg_p) taking into account their respective solution blank concentrations and the mass of the test portion used in AS 4969.3 and express as percentages on an oven-dry weight basis.

9 PRECISION

To test the inter-laboratory and within-laboratory precision of the test procedure described in this Standard for measurement of peroxide sulfur (S_p), calcium (Ca_p) and magnesium (Mg_p), six homogenized acid sulfate soils were analysed in triplicate by 11, 10 and 8 laboratories respectively. Samples were assessed prior to dispatch to confirm homogeneity. Before calculation of precision, the data were examined for the presence of statistical outliers using the tests by Cochran to test homogeneity of variances, and Grubbs for mean outliers. Using the data not excluded by these tests, the precision of the test method as determined by a statistical examination of inter-laboratory test results are as follows: