

Australian Standard[®]

AS 4969.4—2008

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

Method 4: Determination of 1 M potassium chloride extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl})

PREFACE

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

The objective of this Standard is to provide a method for the determination of potassium chloride extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) in acid sulfate soil after the determination of pH_{KCl} and TAA.

METHOD

1 SCOPE

This Standard specifies a method for the determination of potassium chloride extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) in acid sulfate soil after the determination of pH_{KCl} and TAA in AS 4969.2.

NOTES:

- 1 This extraction procedure recovers soluble and exchangeable sulfate, sulfate from gypsum, as well as some sulfate from aluminium hydroxy-sulfate compounds (such as basaluminite). Sulfate from jarosite and similar iron hydroxy-sulfate minerals is not significantly recovered. Methods to determine the residual sulfur held in these iron hydroxy-sulfate minerals are given in AS 4969.6 and AS 4969.11.
- 2 This extraction procedure recovers soluble and exchangeable calcium and magnesium, and calcium from gypsum.
- 3 The KCl extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) measurements can be used in combination with HCl extractable sulfur, calcium and magnesium (AS 4969.8) to determine the net acid-soluble sulfur, calcium and magnesium (AS 4969.11).

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
4969.2 Method 2: Determination of pH_{KCl} and titratable actual acidity (TAA)
4969.6 Method 6: Determination of acid extractable sulfur in soil residue after peroxide oxidation (S_{RAS})
4969.8 Method 8: Determination of hydrochloric acid extractable sulfur (S_{HCl}), calcium (Ca_{HCl}) and magnesium (Mg_{HCl})
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 methods

3 DEFINITIONS

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

4 PRINCIPLE

The soil suspension from AS 4969.2 is diluted, followed by filtration or centrifugation and then potassium chloride extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) 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 similar 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 4969.2.

7 PROCEDURE

The procedure for dilution and filtration of soil suspension following TAA titration shall be as follows:

- (a) After completion of AS 4969.2, quantitatively transfer the contents from titration vessel (6.6) to a tared or weighed beaker (6.1) with water. Subject the solution blanks from AS 4969.2 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), the density of the KCl matrix (which is dependent on the final volume chosen) and the mass of the test portion in AS 4969.2.

- (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.
- (e) Take volumes of centrifuged or filtered solution (sufficient for the instrumental finish to be employed) and determine KCl extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) 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 contain appreciable KCl extractable organic sulfur.
- 3 Codes for analytical techniques used to determine sulfur, calcium and magnesium are shown in Appendix A.

8 CALCULATIONS

Calculate potassium chloride extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) taking into account their respective solution blank concentrations and the mass of test portion used in AS 4969.2 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 (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}), six homogenized acid sulfate soils were analysed in triplicate by 14, 11 and 9 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: