Australian Standard®

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

# Method 8: Determination of hydrochloric acid extractable sulfur ($S_{HCl}$), calcium ($Ca_{HCl}$) and magnesium ($Mg_{HCl}$)

AS 4969.8—2008

PREFACE

This Standard was prepared by the Australian members of the Joint Standards Australia/Standards New Zealand for Committee EV-009, Sampling and Analysis 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 hydrochloric acid extractable sulfur ($S_{HCl}$), calcium ($Ca_{HCl}$) and magnesium ($Mg_{HCl}$) in acid sulfate soil.

METHOD

## 1 SCOPE

This Standard specifies a method for the determination of hydrochloric acid extractable sulfur ($S_{HCl}$), calcium ($Ca_{HCl}$) and magnesium ($Mg_{HCl}$) in acid sulfate soil following extraction with 4 M hydrochloric acid.

NOTES:

1 This extraction procedure recovers soluble and exchangeable sulfate, sulfate from gypsum, the relatively insoluble iron and aluminium hydroxy-sulfate compounds (e.g. jarosite, natrojarosite, basaluminite) as well as some sulfur from organic matter. It also recovers soluble and exchangeable calcium and magnesium, calcium from gypsum, as well as calcium and magnesium from oxides, hydroxides and carbonates of these two elements.

2 The $S_{HCl}$, $Ca_{HCl}$ and $Mg_{HCl}$ measurements can be used in combination with KCl extractable sulfur ($S_{KCl}$), calcium ($Ca_{KCl}$) and magnesium ($Mg_{KCl}$) (AS 4969.4) to determine the net acid-soluble sulfur ($S_{NAS}$), calcium ($Ca_{NAS}$) and magnesium ($Mg_{NAS}$) (AS 4969.11).

## 2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

| | | |
|---|---|---|
| AS | | |
| 4969 | Analysis of acid sulfate soils—Dried samples—Methods of test | |
| 4969.0 | Part 0: | Introduction and definitions, symbols and acronyms |
| 4969.1 | Method 1: | Pre-treatment of samples |

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AS
4969.4 Method 4: Determination of 1 M potassium chloride extractable sulfur ($S_{KCl}$), calcium ($Ca_{KCl}$) and magnesium ($Mg_{KCl}$)
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 purpose of this Standard the definitions used in AS 4969.0 apply.

## 4 PRINCIPLE

Soils are extracted with 4 M HCl, then filtered or centrifuged to obtain a clear extract and after appropriate dilution, hydrochloric acid extractable sulfur ($S_{HCl}$) calcium ($Ca_{HCl}$) and magnesium ($Mg_{HCl}$) 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).

### 5.2 Hydrochloric acid ($\rho_{20}$) 1.16 g/mL

**WARNING: CONCENTRATED HYDROCHLORIC ACID IS A CORROSIVE AGENT. AVOID CONTACT WITH THE 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.

## 6 APPARATUS

### 6.1 Centrifuge

Capable of centrifuging 80 mL of suspension.

### 6.2 Dispenser (manual or automatic)

Capable of accurately dispensing 80 ±0.5 mL.

### 6.3 Electronic balance

Capable of weighing to an accuracy of ±0.01 g.

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

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

### 6.6 Shaking or mixing machine

Capable of keeping solid particles continuously in suspension.

NOTE: For example end-over-end shaker.

## 7 PROCEDURE

### 7.1 Extraction with 4 M HCl and determination of extractable sulfur, calcium and magnesium

The procedure shall be as follows:

(a) Weigh a test portion of 2.0 ±0.1 g (6.3) from the test sample prepared in accordance with AS 4969.1 into a sample bottle (6.5). At least one solution blank should be subjected to the same procedure as the test portion in each analytical run.

(b) In a fume cabinet using the dispenser (6.2), add 80 ±0.5 mL of 4 M HCl (5.3) to the sample bottle to make a 1:40 soil solution ratio. Swirl and place stopper in sample bottle.

CAUTION: FOR SAFETY REASONS STEP 7.1 (b) SHOULD BE CARRIED OUT IN A WELL VENTILATED FUME CUPBOARD IN ACCORDANCE WITH AS/NZS 2243.8.

NOTE: Soils high in carbonate have the potential to react vigorously at this stage and generate carbon dioxide gas. Wait until this initial reaction subsides before placing stopper on sample bottle.

(c) Shake or mix the suspension for 16 ±0.5 h, using the mechanical shaker or mixer (6.6).

(d) Filter soil suspension through filter paper (6.4) or centrifuge (6.1) to obtain a clear extract.

(e) After appropriate dilution, determine hydrochloric acid extractable sulfur ($S_{HCl}$), calcium ($Ca_{HCl}$) and magnesium ($Mg_{HCl}$) using suitable analytical technique(s). For sulfur measurement, instrumentation that specifically determines sulfate is preferable to that which measures total sulfur in solution.

NOTES:

1 The high acidity and chloride concentration may preclude the use of certain analytical techniques for determining sulfur.

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 with high organic matter that contain appreciable HCl-extractable organic sulfur.

3 Codes for the analytical techniques used to determine sulfur, calcium and magnesium are shown in Appendix A.

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