

# Australian Standard<sup>®</sup>

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

### Method 3: Determination of peroxide pH ( $pH_{OX}$ ), titratable peroxide acidity ( $TPA$ ) and excess acid neutralizing capacity ( $ANC_E$ )

#### 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 laboratory method for the determination of peroxide pH ( $pH_{OX}$ ), titratable peroxide acidity ( $TPA$ ) and excess acid neutralizing capacity ( $ANC_E$ ) in acid sulfate soil.

#### METHOD

##### 1 SCOPE

This Standard specifies a method for the determination of peroxide pH ( $pH_{OX}$ ), titratable peroxide acidity ( $TPA$ ) and excess acid neutralizing capacity ( $ANC_E$ ) in acid sulfate soil following digestion with 30% hydrogen peroxide.

##### NOTES:

- 1 The  $TPA$  measurement does not quantitatively recover retained acidity held in iron and aluminium hydroxy-sulfate minerals such as jarosite  $[KFe_3(SO_4)_2(OH)_6]$  and similar minerals. Methods to determine the retained acidity are given in AS 4969.6 and AS 4969.11.
- 2 The suspension obtained from this method can be analysed subsequently to determine peroxide sulfur, calcium and magnesium (AS 4969.5) followed by the determination of  $S_{RAS}$  on the digested soil residue (AS 4969.6).

##### 2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

1006	Solid-stem general purpose thermometers
2162	Verification and use of volumetric apparatus
2162.1	Part 1: General—Volumetric glassware
2162.2	Part 2: Guide to the use of piston-operated volumetric apparatus (POVA)

AS	
2164	Laboratory glassware—One-mark volumetric flasks
2165	Laboratory glassware—Burettes
2166	Laboratory glassware—One-mark pipettes
2167	Graduated straight pipettes
4969	Analysis of acid sulfate soil—Dried samples—Methods of test
4969.0	Part 0: Introduction and definitions, symbols and acronyms
4969.1	Method 1: Pre-treatment of samples
4969.2	Method 2: Determination of $pH_{KCl}$ and titratable actual acidity ( $TAA$ )
4969.5	Method 5: Determination of peroxide sulfur ( $S_p$ ), calcium ( $Ca_p$ ) and magnesium ( $Mg_p$ )
4969.6	Method 6: Determination of acid extractable sulfur in soil residue after peroxide oxidation ( $S_{RAS}$ )
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 terms and definitions used in AS 4969.0 apply.

### 4 PRINCIPLE

Soil is digested with hydrogen peroxide and acidity is generated from the oxidation of sulfides and organic matter;  $pH_{OX}$  is measured on the digest solution. Resulting acidity ( $TPA$ ) is determined by titration to pH 6.5. For samples with  $pH_{OX} > 6.5$  additional steps are necessary to determine  $ANC_E$ .

### 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.

If the suspension is to be used in methods that determine sulfur, calcium and magnesium (AS 4969.5 and AS 4969.6), the purity of all reagents (for these elements) 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 Calibration solutions for the pH meter

##### 5.2.1 General

For pH determinations, buffer solutions as specified in Clauses 5.2.2 and 5.2.3 are sufficient for calibrating the pH meter. It is the responsibility of the analytical laboratory to verify the accuracy of the buffer solutions.

NOTE: Commercially available buffer solutions covering a comparable pH range may be used.

### 5.2.2 Buffer solution, pH 4.00 at 20°C

Dissolve 10.21 g of potassium hydrogen phthalate ( $C_8H_5O_4K$ ) in water and dilute to 1000 mL at 20°C.

The potassium hydrogen phthalate shall be dried before use for 4 h at 105°C.

### 5.2.3 Buffer solution, pH 6.88 at 20°C

Dissolve 3.40 g of potassium dihydrogen phosphate ( $KH_2PO_4$ ) and 3.55 g of disodium hydrogen phosphate ( $Na_2HPO_4$ ) in water and dilute to 1000 mL at 20°C.

Both salts shall be dried before use for 4 h at 105°C.

### 5.2.4 Buffer solution, pH 9.22 at 20°C

Dissolve 3.81 g of disodium tetraborate decahydrate ( $Na_2B_4O_7 \cdot 10H_2O$ ) in water and dilute to 1000 mL at 20°C.

#### NOTES:

- 1 Disodium tetraborate decahydrate may lose water of crystallization when stored for a long time.
- 2 The buffer solutions as specified in Clauses 5.2.2, 5.2.3 and 5.2.4 are stable for one month when stored in polyethylene bottles.
- 3 The pH 9.22 buffer solution (5.2.4) is used when calibrating the pH meter prior to standardizing the sodium hydroxide solutions (5.9) and/or when alkaline or lime-treated samples are analysed.

### 5.3 Copper chloride solution, 400 mg Cu/L as $CuCl_2 \cdot 2H_2O$

Dissolve 1.073 g of copper (II) chloride dihydrate ( $CuCl_2 \cdot 2H_2O$ ) in water and dilute to 1000 mL at 20°C.

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

**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.**

### 5.5 Hydrochloric acid solution, 0.5 M (Standardized)

Add 50 mL of concentrated HCl (5.4) with stirring to approximately 700 mL of water. Cool to room temperature, transfer to a 1 L volumetric flask and fill to the mark with water.

Standardize against disodium tetraborate decahydrate ( $Na_2B_4O_7 \cdot 10H_2O$ ) and calculate molarity of HCl solution ( $C_3$ ).

Solutions made by diluting commercially available ampoules may also be used.

NOTE: The molarity ( $C_3$ ) of the standardized HCl solution should be used in the calculations (see Clause 8).

### 5.6 Hydrogen peroxide, 30% (w/w) $H_2O_2$ (Bulk peroxide for digestion)

**WARNING: HYDROGEN PEROXIDE IS A CORROSIVE OXIDIZING AGENT. CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE. 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.**