

Australian Standard[®]

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

Method 2: Determination of pH_{KCl} and titratable actual acidity (*TAA*)

PREFACE

This Standard was prepared by the Australian members of the Joint Standards Australia/Standards New Zealand 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 pH_{KCl} and titratable actual acidity (*TAA*) in acid sulfate soil.

METHOD

1 SCOPE

This Standard specifies a method for the determination of pH in a 1 M KCl soil suspension and where required titratable actual acidity (*TAA*) in acid sulfate soil.

NOTES:

- 1 The *TAA* 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 from this method can be analysed subsequently to determine KCl extractable sulfur, calcium and magnesium (AS 4969.4).

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)
2164	Laboratory glassware—One-mark volumetric flasks
2165	Laboratory glassware—Burettes
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

AS

- 4969.4 Method 4: Determination of 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})
- 4969.11 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

A suspension of soil in KCl solution is shaken for 4 h and allowed to stand overnight. The suspension is then briefly shaken prior to determining pH (pH_{KCl}). If pH_{KCl} is less than 6.5, titratable actual acidity (TAA) is determined by titrating the soil suspension to pH 6.5.

5 REAGENTS

5.1 General

All reagents shall be of analytical grade (AR grade). 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 (e.g. AS 4969.4), 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 ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) 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 (5.4) and/or when alkaline or lime-treated samples are analysed.

5.3 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.

5.4 Standardized sodium hydroxide (NaOH) solutions

CAUTION: SOLID SODIUM HYDROXIDE IS CAUSTIC AND HYGROSCOPIC AND SHOULD BE STORED AWAY FROM WATER. DILUTE SODIUM HYDROXIDE SOLUTIONS ABSORB CARBON DIOXIDE. AVOID UNNECESSARY CONTACT OF THE SOLUTIONS WITH THE ATMOSPHERE.

5.4.1 0.25 M NaOH

Dissolve 10.1 ± 0.1 g of NaOH in water, transfer quantitatively to a 1 L volumetric flask and fill to the mark with water.

Standardize against potassium hydrogen phthalate previously dried for 4 h at 105°C and stored in a desiccator.

The procedure shall be as follows:

- (a) Accurately weigh 0.25 ± 0.05 g (6.5) of potassium hydrogen phthalate and dissolve in water.
- (b) Titrate (6.3) potassium hydrogen phthalate solution with NaOH solution (5.4.1) and record equivalence point volume.
- (c) Calculate the molarity of NaOH solution (C_1).

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

5.4.2 0.05 M NaOH

Dissolve 2.05 ± 0.05 g of NaOH in water, transfer quantitatively to a 1 L volumetric flask and fill to the mark with water.

Standardize against potassium hydrogen phthalate previously dried for 4 h at 105°C and stored in a desiccator.

The procedure shall be as follows:

- (a) Accurately weigh 0.10 ± 0.02 g (6.5) of potassium hydrogen phthalate and dissolve in water.
- (b) Titrate (6.3) potassium hydrogen phthalate solution with NaOH solution (5.4.2) and record equivalence point volume.
- (c) Calculate molarity of NaOH solution (C_2).