

Australian Standard[®]

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

Method 12: Complete suspension peroxide oxidation combined acidity and sulfur (SPOCAS) method

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 and Working Group EV-009-02-01, Analysis of Acid Sulfate Soil.

The objective of this Standard is to encompass the determination of the various components of soil acidity and/or alkalinity, depending on pH, into a combined method to streamline the process of acid base accounting. It specifies methods for the determination of pH_{KCl} , titratable actual acidity (TAA), potassium chloride extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) in acid sulfate soil (ASS), as well as for the determination of pH_{OX} , titratable peroxide acidity (TPA), excess acid neutralizing capacity (ANC_E), peroxide sulfur (S_P), calcium (Ca_P), magnesium (Mg_P) and residual acid soluble sulfur (S_{RAS}). These results can be used to calculate titratable sulfidic acidity (TSA), reacted calcium (Ca_A), reacted magnesium (Mg_A) and peroxide oxidizable sulfur (S_{POS}).

The term 'informative' has been used in this Standard to define the application of the appendix to which it applies. An 'informative' appendix is only for information and guidance.

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. Following this, potassium chloride extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) are determined. On a separate test portion, peroxide pH (pH_{OX}), titratable peroxide acidity (TPA) and excess acid neutralizing capacity (ANC_E) are determined following digestion with 30% hydrogen peroxide. Following this, peroxide sulfur (S_P), calcium (Ca_P) and magnesium (Mg_P) are determined. On samples where jarosite is present, or $pH_{KCl} < 4.5$, residual acid soluble sulfur (S_{RAS}) is determined on the soil residue remaining after peroxide digestion.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

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| 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 |
| 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 |
| 4969.8 | 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}) |

AS/NZS

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| 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

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| 3696 | Water for analytical laboratory use—Specification and test methods |
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3 DEFINITIONS

For the purpose of this Standard the terms and definitions set out 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. After appropriate dilution of the soil suspension, potassium chloride extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) are measured by suitable analytical technique(s).

A separate test portion of soil is digested with 30% hydrogen peroxide and acidity is generated from the oxidation of sulfides (and organic matter where present). Following digestion, pH_{OX} is measured on the digest solution. If the pH after peroxide digestion is greater than 6.5, a HCl titration is performed, followed by an additional peroxide digest step. This allows the calculation of excess acid neutralizing capacity (ANC_{E}). Titratable peroxide acidity (TPA) is then determined by titration to pH 6.5 in a KCl suspension. After appropriate dilution of the soil suspension, peroxide sulfur (S_{P}), calcium (Ca_{P}) and magnesium (Mg_{P}) are measured by suitable analytical techniques. After appropriate washing of the soil residue, the sulfur is extracted with hydrochloric acid and the residual acid soluble sulfur (S_{RAS}) is measured by a suitable analytical technique on samples where $pH_{\text{KCl}} < 4.5$, or where the presence of jarosite has been noted in the submitted sample.

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.

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 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 solution (5.11) 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, ρ_{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.