

# Australian Standard®

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

### Method 13: Determination of acid neutralizing capacity by back-titration ( $ANC_{BT}$ )

AS 4969.13—2009

#### 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 provide a method for the determination of acid neutralizing capacity by back titration ( $ANC_{BT}$ ) in acid sulfate soil.

#### METHOD

##### 1 SCOPE

This Standard specifies a method for the determination of acid neutralizing capacity ( $ANC_{BT}$ ) in acid sulfate soil by back titration of excess acid following hydrochloric acid digestion. This determination is used to estimate acid neutralizing capacity (ANC) as part of the acid base accounting (ABA) procedure (AS 4969.14).

##### 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
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
- 4969.14 Method 14: Calculation of the acid-producing potential of acid sulfate soil using an acid base accounting method

**AS/NZS**

- 2243 Safety in laboratories
- 2243.1 Part 1: Planning and operational aspects
- 2243.2 Part 2: Chemical aspects

**ISO**

- 3696 Water for analytical laboratory use—Specification and test methods

**3 PRINCIPLE**

Soil is digested with a known amount of hydrochloric acid. The acid remaining after digestion (i.e. not neutralized by components of the soil) is then titrated with dilute NaOH solution to pH 7. The acid neutralizing capacity ( $ANC_{BT}$ ) is then determined by difference.

**4 DEFINITIONS**

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

**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 calcium carbonate ( $CaCO_3$ ) should be verified. Reagents should be tested 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 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 (5.6).

#### 5.3 Calcium carbonate

Dried at 105°C for 4 h and stored in a desiccator prior to use.

#### 5.4 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.**

#### 5.5 Hydrochloric acid solution, 0.10 M (Standardized)

Add 10 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 ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ ) and calculate molarity.

The procedure shall be as follows:

- (a) Accurately weigh  $0.40 \pm 0.05$  g (6.6) of sodium tetraborate decahydrate and dissolve in water.
- (b) Titrate sodium tetraborate solution with HCl solution and record equivalence point volume.
- (c) Calculate molarity of the HCl solution ( $C_1$ ).

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

#### 5.6 Sodium hydroxide solution, 0.10 M (Standardized)

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

Dissolve  $4.10 \pm 0.10$  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  $0.20 \pm 0.04$  g (6.6) of potassium hydrogen phthalate and dissolve in water.
- (b) Titrate potassium hydrogen phthalate solution with NaOH solution (5.6) and record equivalence point volume.
- (c) Calculate molarity of the NaOH solution ( $C_2$ ).

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