

# Australian Standard<sup>®</sup>

AS 4969.1—2008

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

### Method 1: Pre-treatment of samples

#### 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.

Acid sulfate soil is a special group of soils that, because of the many complex chemical reactions associated with them, require specialized sampling, pre-treatment and analytical methods for their characterization so as to better understand appropriate management practices that will ensure they have minimal impacts on the environment.

The objective of this Standard is to provide a consistent laboratory procedure for the drying, pulverizing and storage of acid sulfate soil prior to chemical analysis.

#### METHOD

##### 1 SCOPE

This Standard provides a procedure for pre-treating acid sulfate soil following sampling, prior to their analysis as dried sample using appropriate methods of test. The procedure includes the handling of samples before delivery to the laboratory, and the drying, grinding and storage of samples. The Standard also provides a procedure for archiving a sample after analysis.

##### NOTES:

- 1 It is recommended to collect a field sample that provides approximately 200 g of dried sample.
- 2 Field personnel may remove coarse fragments (e.g. shell, stones, rock fragments >2 mm) prior to sending the field sample to the laboratory. The presence of such fragments, their size, shape and abundance (and whether they have been removed) should be recorded in field sampling notes.
- 3 For the chemical analysis to most closely reflect the condition of the acid sulfate soil in its natural state at the time of sampling, the handling, preparation and storage of these soils should be such that potential for oxidation of pyrite is minimized.
- 4 This method is not appropriate for preservation of acid volatile sulfur (e.g. iron monosulfides) in acid sulfate soil as oxidation of these compounds is likely to occur. This method, however, is suitable for the purpose of acid base accounting.

## 2 REFERENCED DOCUMENT

The following documents are referred in this Standard:

AS

4969      Analysis of acid sulfate soil—Dried samples—Methods of test  
4969.0    Part 0: Introduction and definitions, symbols and acronyms

AS/NZS

2243      Safety in laboratories  
2243.1    Part 1: Planning and operational aspects  
2243.2    Part 2: Chemical aspects  
2243.3    Part 3: Microbiological aspects and containment facilities

## 3 DEFINITIONS

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

## 4 PRINCIPLE

Field samples are thawed if necessary and extraneous coarse matter, such as large shells and other large fragments, are removed either before or after drying. Samples are rapidly dried in a fan-forced air-extracting oven before being crushed (where required) and then passed through a 2 mm sieve to remove remaining coarse fragments. Samples are then finely ground to a powder ( $<75\ \mu\text{m}$ ).

## 5 APPARATUS

### 5.1 General

Any equipment or apparatus used in soil preparation shall be cleaned to avoid contamination of the soil sample being prepared. It shall be cleaned thoroughly between samples to prevent any carry-over contamination. Detailed descriptions of the apparatus to be employed are not given, but should meet the minimum requirements indicated below.

### 5.2 Balance

A balance capable of measuring up to 1000 g to an accuracy of  $\pm 0.1\ \text{g}$ .

### 5.3 Drying oven

A fan-forced air-extracting drying oven capable of maintaining samples at a temperature of  $85 \pm 5^\circ\text{C}$ .

NOTE: Users should avoid overloading the drying oven, by not placing large quantities of frozen or even wet samples in the oven.

### 5.4 Drying trays

Trays made of polymer or other inert material, capable of withstanding temperatures of up to  $90^\circ\text{C}$  for prolonged periods. Trays should be capable of holding sufficient moist sample to provide approximately 200 g of dried sample, with the thickness of the soil material being no more than 2 cm. Drying trays may also be lined with 'plastic wrap' to alleviate the need to wash sample trays.

### 5.5 Grinding apparatus 1

A grinding mill (typically a hammer type mill) shown not to contribute to the levels of the analytes being determined.

## 5.6 Grinding apparatus 2

A ring mill (or similarly forceful grinding apparatus) capable of reducing a laboratory sample to  $<75\ \mu\text{m}$  and shown not to contribute to the levels of the analytes being determined. (See Notes in Clause 7.4.)

## 5.7 Hammer or mallet

A wooden or other soft-faced, hand held hammer or wood-working mallet.

## 5.8 Mortar and pestle

Made of material shown not to contribute to the levels of the analytes being determined (e.g. glass, porcelain or agate).

## 5.9 Sieve

A sieve with an aperture of 2 mm and shown not to contribute to the level of analyte being determined.

## 5.10 Vacuum sealing apparatus

Capable of removing air from the plastic or foil bag containing the sample and sealing to limit ingress of air and moisture.

# 6 PROCEDURE

## 6.1 Sample pre-treatment in the field

Upon collection in the field, a soil sample shall be immediately placed in a leak-proof container that minimizes the sample's contact with air and avoids moisture loss (e.g. placed in sealable plastic bags, with air excluded). The sample shall be immediately cooled and maintained at less than  $4^{\circ}\text{C}$  until received by the laboratory to reduce the possibility of oxidation.

NOTE: A portable 12 V car freezer or cold box containing dry ice are the most efficient coolers but if not available, ordinary ice may be employed for cooling.

It is most important that sample labelling be indelible.

After collection, field samples should be sent to the laboratory without undue delay. If samples cannot be received by the laboratory within 24 h of collection, or the sample pre-treatment in the laboratory (in Clause 7) is not to be commenced immediately, the samples shall be managed to minimize oxidation. Methods include:

- (a) Freezing the field sample and maintaining it in a frozen state in sealed, air-tight containers.
- (b) Stored in an inert atmosphere (e.g.  $\text{N}_2$ , Ar).
- (c) Vacuum sealing and stored cold (i.e.  $<4^{\circ}\text{C}$ ) or frozen.
- (d) Quick oven drying the sample at  $80\text{--}85^{\circ}\text{C}$  in a fan-forced convection oven. Care should be taken not to overload the oven's moisture removal capacity. The dried sample should then be stored in a sealed container in a low humidity environment (e.g. sealed plastic bags) with as much air removed as possible. If the methods of test are not to be completed within 6 weeks of drying, then the sample shall be stored as per an archive sample (Clause 7.5.2).

# 7 SAMPLE PRE-TREATMENT IN THE LABORATORY

## 7.1 General

Pre-treatment of soil should be carried out in a clean, dedicated preparation area. Care should be taken at all times to avoid contamination of the sample from—

- (a) the ambient atmosphere; and