Australian Standard®

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

Method 7: Determination of chromium reducible sulfur (S_{CR})

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 method for the determination of chromium reducible sulfur (S_{CR}) in acid sulfate soil.

METHOD

1 SCOPE

This Standard specifies a method for the determination of chromium reducible sulfur (S_{CR}) by iodometric titration of distilled hydrogen sulfide trapped as zinc sulfide.

NOTE: This method determines inorganic sulfides (e.g. pyrite, marcasite, greigite, mackinawite) and elemental sulfur in acid sulfate soil without interferences from organic sulfur and oxidized forms of sulfur such as sulfate.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS
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

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 in an acidic chromous chloride solution, produced by the reaction of powdered chromium metal and hydrochloric acid. Chromium reducible sulfur is released and distilled as H_2S. The evolved H_2S gas is trapped as ZnS in a zinc acetate/ammonia solution and the trapped sulfur then quantified by iodometric titration.

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.

The purity of all reagents for sulfur should be verified by performing a blank test. Reagents should also be tested for the presence of sulfur whenever a change in source is made (e.g. brand or batch).

5.2 Chromium powder

NOTE: Different sources or batches of chromium powder may yield different blank values.

CAUTION: CHROMIUM DUST MAY BE TOXIC IF INHALED AND MAY REPRESENT A COMBUSTION RISK. AVOID THE USE OF VERY FINE CHROMIUM POWDER.

5.3 Ethanol 95% solution

WARNING: DO NOT USE ABSOLUTE ALCOHOL TO MAKE 95% ALCOHOL SOLUTION, AS ABSOLUTE ALCOHOL MAY CONTAIN ADDITIVES THAT ARE CARCINOGENIC AND THEREFORE HAZARDOUS IF INHALED OR INGESTED.

5.4 Hydrochloric acid (ρ_{20}) solution 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, 6 M

Add 600 mL of hydrochloric acid (5.4) with stirring to approximately 300 mL of water. Cool to room temperature, transfer to a 1 L volumetric flask and fill to the mark with water.

5.6 Iodine solution, 0.0125 M (standardized)

Dissolve 22.5 ±0.1 g of potassium iodide in water and add 3.2 ±0.01 g of iodine. After the iodine has dissolved, dilute to 1 L with water. Standardize iodine solution against the standardized 0.025 M sodium thiosulfate solution (5.9), using the starch solution (5.10) as an indicator. Calculate the molarity (C) of the iodine solution, in moles per litre as follows:

$$C = \frac{F \times D}{2 \times E}$$

where

D = titration volume of standard sodium thiosulfate solution, in millilitres

E = volume of iodine solution titrated, in millilitres

F = molarity of sodium thiosulfate solution used, in mol/L

The iodine solution should be standardized on a daily basis.

5.7 Nitrogen gas

High purity grade.

5.8 Sodium hydroxide solution, 6 M

CAUTION: SOLID SODIUM HYDROXIDE IS CAUSTIC AND HYGROSCOPIC AND SHOULD BE STORED AWAY FROM WATER.

Dissolve 240 ±1 g of sodium hydroxide in water, transfer quantitatively to a 1 L volumetric flask. Cool to room temperature and fill to the mark with water.

5.9 Sodium thiosulfate solution, 0.0250 M (standardized)

Accurately weigh 6.205 ±0.001 g (6.4) of $Na_2S_2O_3.5H_2O$ and dissolve in water. Transfer quantitatively to a 1 L volumetric flask. Add 1.5 ±0.01 mL of 6 M NaOH (5.8) and fill to the mark with water. Standardize against potassium iodate or potassium dichromate solutions.

NOTE: Commercially available ampoules of standardized sodium thiosulfate solution may also be used.

5.10 Starch indicator solution

Dissolve 2.0 ±0.1 g of arrowroot starch and 0.20 ±0.01 g of salicylic acid in 100 mL of hot water. Allow to cool to room temperature before use.

5.11 Zinc acetate/2.8% ammonia solution (trapping solution)

Dissolve 30 ±0.5 g of zinc acetate dihydrate in 750 mL water. Add 100 mL of 28% ammonia solution. Transfer to 1 L volumetric flask and fill to the mark with water.

6 APPARATUS

6.1 General

Grade A volumetric glassware shall be used throughout. Volumetric flasks shall comply with AS 2164 and pipettes shall comply with AS 2166 and AS 2167. The use of volumetric glassware shall conform to AS 2162, Parts 1 and 2.

6.2 Burette

A-grade, 10 mL capacity, graduated at 0.02 mL intervals, complying with Class A according to AS 2165. Alternatively, a similarly accurate digital burette or a suitably calibrated burette from an automatic titration instrument may be used.