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Soil quality - Acid-base accounting procedure for acid sulfate soils —

Part 2: Chromium reducible sulfur (CRS) methodology

Qualité de l'eau — Méthode de comptage acide-base pour les sols sulfatés acides —

Partie 2: Méthode de sulfato réduction au chrome

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

ISO 14388 consists of the following parts, under the general title *Soil quality — Acid-base accounting procedure for acid sulfate soils*:

— *Part 1: Introduction and definitions, symbols and acronyms, sampling and sample preparation*

— *Part 2: Chromium reducible sulfur (CRS) methodology*

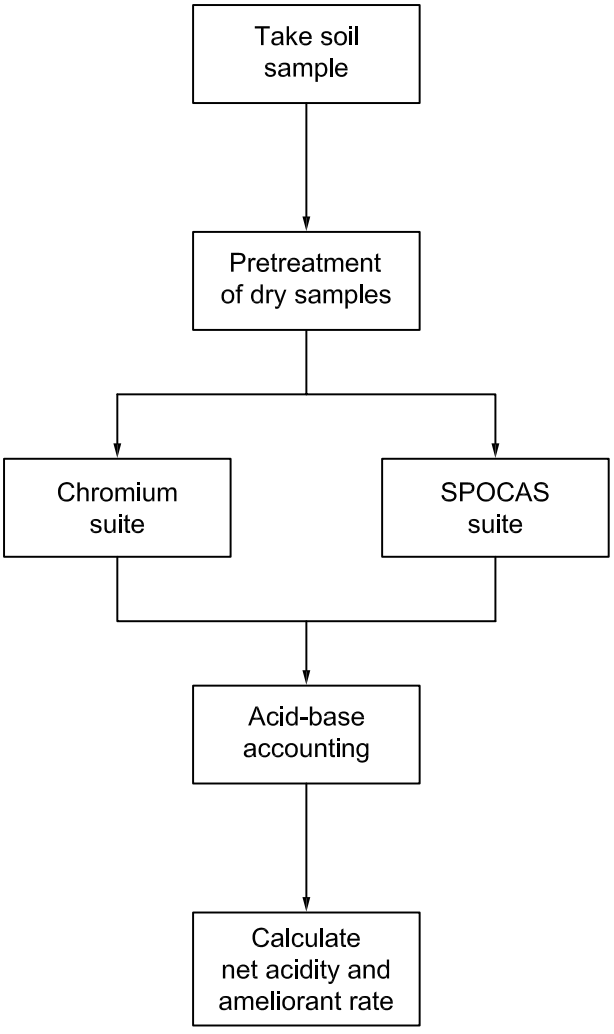
— *Part 3: Suspension peroxide oxidation combined acidity and sulfur (SPOCAS) suite analysis*

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Introduction

The objective of this part of ISO 14388 is to determine the net acidity (or alkalinity) of acid sulfate soils by providing a streamlined approach for determination of the various components of soil acidity and/or alkalinity, depending on pH. The chromium suite combines the measurement of chromium reducible sulfur (S_{CR}) with various measures of existing acidity and acid neutralizing capacity (ANC) using a decision-tree based on the value of pH_{KCl} (Figure 1) as the basis for determining an acid-base account for acid sulfate soils.

The results required to determine net acidity vary with the soil's actual acidity, as represented by pH_{KCl}. Table A.1 of ISO 14388-1 shows the analyses required for the Chromium suite. This table uses results reported in acidity units. Alternatively, results in sulfur units can be utilized.



Key

a acidity titration

b sulfur determination

c acid neutralising determination

d calculated parameter

Figure 1 — Chromium suite flow diagram

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Soil quality - Acid-base accounting procedure for acid sulfate soils —

Part 2:
Chromium reducible sulfur (CRS) methodology

WARNING — Persons using this part of ISO 14388 should be familiar with usual laboratory practice. This part of ISO 14388 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This part of ISO 14388 specifies a suite of methods used to determine the net acidity in acid sulfate soils. This part of ISO 14388 specifies a method for measuring chromium reducible sulfur (S_{CR}) by iodimetric titration of distilled hydrogen sulfide trapped as zinc sulfide, following acidic chromous chloride digestion. 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.

On a separate test portion of soil, the pH in a 1 mol/l KCl soil suspension (pH_{KCl}) is determined. When pH_{KCl} is < 6,5, titratable actual acidity (TAA) is then determined. Subsequently, potassium chloride extractable sulfur (S_{KCl}), calcium (Ca_{KCl}), and magnesium (Mg_{KCl}) can also be determined. Where jarosite is identified in the soil (or where pH_{KCl} is < 4,5), net acid-soluble sulfur (S_{NAS}) is determined by the difference between hydrochloric acid extractable sulfur (S_{HCl}) and potassium chloride extractable sulfur. On samples where pH_{KCl} is < 6,5, acid neutralizing capacity is determined by measuring either inorganic carbon (C_{IN}) by combustion furnace, or ANC_{BT} (ANC measured by back-titration of acid remaining following an acid digest).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385-1, *Laboratory glassware — Burettes — Part 1: General requirements*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 835-1, *Laboratory glassware — Graduated pipettes — Part 1: General requirements*

ISO 835-2, *Laboratory glassware — Graduated pipettes — Part 2: Pipettes for which no waiting time is specified*

ISO 835-3, *Laboratory glassware — Graduated pipettes — Part 3: Pipettes for which a waiting time of 15 s is specified*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 1770, *Solid-stem general purpose thermometers*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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ISO 8655-1, *Piston-operated volumetric apparatus — Part 1: Terminology, general requirements and user recommendations*

ISO 8655-2, *Piston-operated volumetric apparatus — Part 2: Piston pipettes*

ISO 8655-3, *Piston-operated volumetric apparatus — Part 3: Piston burettes*

ISO 10694, *Soil quality — Determination of organic and total carbon after dry combustion (elementary analysis)*

ISO 14388, *Soil quality — Acid sulfate soil — Part 1: Introduction, terminology, soil preparation and acid-base accounting*

3 Terms and definitions

For the purposes of this document the terms, definitions, symbols, units and abbreviated terms given in ISO 14388-1 apply.

4 Principle

4.1 Potential sulfidic acidity

The **potential sulfidic acidity** is determined as chromium reducible sulfur (S_{CR}). In this method, sulfur is distilled as H_2S from acid sulfate soil digested in an acidic chromous chloride solution produced by the reaction of powdered chromium metal and hydrochloric acid. The evolved H_2S gas is precipitated as ZnS in a zinc acetate/ammonia trapping solution with the trapped sulfur, then quantified by iodimetric titration.

4.2 Actual acidity and retained acidity

To determine **actual acidity**, a 1:40 suspension of soil in 1 mol/l 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). Where pH_{KCl} is < 4,5 (or where the presence of jarosite has been noted in the submitted sample), **retained acidity** is determined as net acid-soluble sulfur (S_{NAS}). Net acid-soluble sulfur is the difference between 4 mol/l hydrochloric acid extractable sulfur (S_{HCl}) and potassium chloride extractable sulfur (S_{KCl}). In highly organic soils, S_{NAS} might overestimate retained acidity due to the inclusion of organic S in the result.

4.3 Acid neutralizing capacity

In soils where pH_{KCl} > 6,5, and hence where there is a chance of acid neutralizing capacity (ANC) being present, ANC can be estimated by various methods such as inorganic carbon analysis (e. g. C_{IN} by combustion furnace according to ISO 10694), and ANC by back titration (ANC_{BT}). In this manner, a full acid-base account can be made of the soil.

5 Reagents for analysis of chromium reducible sulfur

All reagents shall be of analytical grade. Deionised or glass distilled water of grade 2 as defined in ISO 3696 shall be used throughout.

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

Commercially available ampoules of standardized solutions can also be used where available.

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