# INTERNATIONAL STANDARD



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

Part 1:

Introduction and definitions, symbols and acronyms, sampling and sample preparation

# iTeh STANDARD PREVIEW

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

Parti<u>e 1</u>; <u>Introduction</u> et définitions, symboles et acronymes, https://standards.iteh.échantillonnage.et.préparation\_des\_échantillons 0b35a2d0abf9/iso-14388-1-2014



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<u>ISO 14388-1:2014</u> https://standards.iteh.ai/catalog/standards/sist/fa9d39db-1a5f-41b3-adbd-0b35a2d0abf9/iso-14388-1-2014



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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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** add Acid-base accounting procedure for acid sulfate soils: 0b35a2d0abf9/iso-14388-1-2014

- 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) methodology

# Introduction

Acid sulfate soils are a complex group of predominantly low-lying coastal soils and sediments that contain iron sulfides and/or their oxidation products. Typically, the sulfide present is pyrite (FeS<sub>2</sub>); though in some instances, iron 'monosulfides' can be important. Acid sulfate soils are most prevalent in tropical and sub-tropical regions, but also occur in temperate and sub-arctic regions. When maintained in reduced and waterlogged conditions, the pyrite and other sulfides in these soils will not oxidize; and when they are in this state, the soils are generically termed potential acid sulfate soils. When these soils are exposed by whatever mechanism of disturbance to atmospheric and dissolved oxygen, the sulfides they contain oxidize to sulfate and ferric ions, producing acid at the same time. The complete oxidation of pyrite by oxygen is generally represented by Formula (1):

$$FeS2 + \frac{15}{4}O2 + \frac{7}{2}H2O \rightarrow Fe(OH)3 + 4H^{+} + 2SO4^{2-}$$
(1)

However, the oxidation of pyrite does not always go to completion in the short-term, with oxidation products other than ferric hydroxide and sulfuric acid often forming. The iron hydroxy-sulfate mineral *jarosite* [KFe<sub>3</sub>(SO<sub>4</sub>)<sub>2</sub>(OH)<sub>6</sub>], can be a conspicuous product of the oxidation process in acid sulfate soil. These oxidation products, as well as other metal ions from the soil (e. g. aluminium) that are dissolved by the acid can have deleterious environmental, agronomic, and economic impacts. Where the pH of these soils falls to 4 or below (as a consequence of sulfide oxidation), they are generically termed actual acid sulfate soils (AASS). When potential acid sulfate soil contains carbonate or other minerals with an acid neutralizing capacity (ANC) (e. g. calcite in the form of finely divided shell material), they cannot become acidic when they oxidize.

**The STANDARD PREVIEW** Because of the many possible complex chemical reactions, the characterization of acid sulfate soils using analytical methods can be a complex process, but accurate characterization is crucial to the management of these soils. Chemical analysis provides the data necessary to assess the acid-producing potential of these soils. From these data, acid-base accounting can be used to determine the dosing rate of alkaline ameliorants needed to fully treat any net acidity. Once dosed accordingly, these soils should not generate acidity, thereby minimizing potential adverse impacts on the environment and/or infrastructure.

Conceptually, the best way to assess the acid-producing potential of these soils is through an 'acidbase accounting' (ABA) procedure, whereby the 'potential sulfidic acidity', 'existing acidity', and 'acid neutralizing capacity' (ANC) are quantified. These components of the acid-base account can be determined separately by the various methods of test in this series of ISO 14388. Once the individual components are determined, the net acidity can be calculated as:

Net acidity = Potential sulfidic acidity + Existing acidity - ANC

A number of approaches exist for the determination of 'potential sulfidic acidity' (i. e. the acidity that can be generated by the oxidation of sulfides). The sulfide content can be measured by either reduction methods (e. g. chromium reducible sulfur  $S_{CR}$ ) or oxidation methods (e. g. peroxide oxidizable sulfur  $S_{POS}$ ). The sulfidic acidity generated by these soils can then be calculated from the sulfide content determined. Alternatively, the potential sulfidic acidity can be measured directly by titration, following accelerated oxidation of the sulfides with hydrogen peroxide, [e. g. titratable sulfidic acidity (TSA)].

Existing acidity might be present in the soil as a result of previous oxidation of sulfides. If this acidity is in exchangeable and/or soluble forms, it is termed 'actual acidity'. It is measured by titrating a 1 mol/l potassium chloride (KCl) soil suspension to pH 6,5 and is termed 'titratable actual acidity' (TAA). Soil suspensions with pHKCl values  $\geq$  6,5 are deemed to have no actual acidity. Additional existing acidity can also reside in sparingly soluble iron and aluminium hydroxy-sulfate phases such as jarosite. This component of the existing acidity is termed 'retained acidity'. It can be estimated by determining net acid-soluble sulfur (SNAS) or residual acid-soluble sulfur (SRAS); it is usually measured on soil with a pHKCl < 4,5, and/or where jarosite has been identified.

If a soil has a  $pH_{KCl} > 6,5$ , it can contain some ANC (e. g. in the form of CaCO<sub>3</sub> from shell). The ANC can be determined by various methods [e. g. inorganic carbon (C<sub>IN</sub>), using a combustion furnace, or acid digestion followed by titration of unreacted acid (ANC<sub>BT</sub>)].

These individual components can be combined into analytical suites that streamline the process of acid-base accounting. Figure 1 shows the possible options to take for the analysis of acid sulfate soils. The two principal analytical suites, the chromium suite and the SPOCAS suite are conceptualized in Figures 2 and 3 respectively.

The chromium suite combines the measurement of  $S_{CR}$  with various measures of existing acidity and ANC using a decision-tree based on the value of  $pH_{KCl}$ . In soils where  $pH_{KCl} > 6,5$ , and hence there is a chance of ANC being present, ANC can be estimated by various methods such as  $(C_{IN})$  analysis and ANC by back titration (ANC<sub>BT</sub>). Where  $pH_{KCl} < 6,5$ , the soil contains existing acidity. This can be measured by TAA. If  $pH_{KCl} < 4,5$ , the soil can also contain retained acidity in addition to the actual acidity. The retained acidity can be determined as  $S_{NAS}$  in accordance with ISO 14388-2, using the decision-tree approach (Figure 2). In this manner, a full acid-base account can be made of the soil. In many instances, a  $S_{CR}$  measurement by itself does not constitute a proper acid-base account.

The Suspension Peroxide Oxidation Combined Acidity and Sulfur (SPOCAS) method combines the calculation of  $S_{POS}$  and TSA with measurement of alkali cations (calcium and magnesium) solubilised by the peroxide digest. In soils where acid neutralizing capacity exceeds the potential acidity of sulfides present (i. e. soils are 'self-neutralizing'), a titration using hydrochloric acid can be performed to quantify this excess acid neutralizing capacity (ANC<sub>E</sub>). The complete SPOCAS method also measures existing acidity in the form of TAA and estimates retained acidity held in sparingly soluble iron and aluminium minerals from the measurement of  $S_{RAS}$  in the soil residue remaining after peroxide digestion.

The 'SPOCAS' suite uses the SPOCAS method as the basis for determining an acid-base account. Like the chromium suite, a decision-tree type process is followed (Figure 3). Various components of soil acidity and/or alkalinity are determined, depending on pH. Once the decision-tree process is completed, the various components can be used to calculate an acid-base account.

The chromium suite approach has the **advantage of being a cheaper and** faster suite of analyses, and is better at estimating sulfide content at low levels and/or in highly organic samples, whereas the SPOCAS suite gives a more complete set of data (e. g. including alkalications), allowing greater interpretation of what is occurring in a soil or soil profile itenai/catalog/standards/sist/fa9d39db-1a5f-41b3-adbd-

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Figure 1 — Flow diagram of acid sulfate soil analysis



#### Кеу

- a acidity titration
- b sulfur determination
- c acid neutralising determination
- d calculated parameter

Figure 2 — Chromium suite flow diagram



Key

## ISO 14388-1:2014(E)

- a acidity titration
- b sulfur determination
- c cation/acid neutralising
- d calculated parameter
- e alternatively,  $S_{NAS}$  can be substituted for  $S_{RAS}$

Figure 3 — SPOCAS flow diagram

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

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

WARNING — Persons using this International Standard should be familiar with usual laboratory practice. This standard 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 provides a general introduction to acid sulfate soils and the approaches that can be used to measure the various components of this special group of soils and their potential to produce acidity. It provides a compilation of the test methods, identification and definitions of the symbols, terms, and acronyms used in this series of ISO 14388. While part of the decision-making process can involve the analysis of prepared wet or dry samples, this part of ISO 14388 specifically outlines the procedures that are involved for preparing and analysing dried samples (see Figure 1).

This part of ISO 14388 also 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.

This part of ISO 14388 provides an acid-base accounting method to calculate the net acid-producing potential of acid sulfate soil materials by individually assessing the acid-producing capacity due to oxidation of inorganic sulfides, the existing acidity, and the effective acid-consuming capacity using results obtained in ISO 14388-2 and ISO 14388-3.

# 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 14388-2, Soil quality — Acid-base accounting procedure for acid sulfate soils — Part 2: Chromium reducible sulfur (CRS) methodology

ISO 14388-3 Soil quality — Acid-base accounting procedure for acid sulfate soils — Part 3: Suspension peroxide oxidation combined acidity and sulfur (SPOCAS) methodology

# 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

NOTE Symbols prefixed by 'a-' indicate they have been converted to equivalent acidity units (either acid generating or acid neutralizing, assuming the stoichiometry of the pyrite oxidation reaction given in the Introduction).

#### 3.1 actual acid sulfate soil AASS

soil that is severely acidified as a result of inorganic sulfide oxidation

Note 1 to entry: Typically, AASS has resulted from the partial or complete oxidation of PASS due to disturbance, resulting in a soil with a pH of less than 4.

### 3.2

#### actual acidity component of existing acidity

Note 1 to entry: The soluble and exchangeable acidity already present in the soil that can be mobilized and discharged following a rainfall event and arising as a consequence of previous oxidation of sulfides.

Note 2 to entry: The actual acidity does not include the less soluble acidity (i. e. retained acidity) held in hydroxy-sulfate minerals such as jarosite.

3.3

## acid neutralizing capacity

ANC

measurement of a soil's ability to buffer acidity

Note 1 to entry: Expressed as equivalent % CaCO<sub>3</sub>.

3.4

# acid neutralizing capacity by back titration NDARD PREVIEW

ANC<sub>BT</sub> acid neutralizing capacity measured by acid digest followed by back titration of unreacted acid

Note 1 to entry: Expressed as equivalent % CaCO<sub>3</sub>. <u>ISO 14388-1:2014</u>

https://standards.iteh.ai/catalog/standards/sist/fa9d39db-1a5f-41b3-adbd-

#### **3.5** https://standards.tteh.ai/catalog/standards/sist/ta9d39dt acid neutralizing capacity by back titration<sup>22d0abf9/iso-14388-1-2014</sup>

## a-ANC<sub>BT</sub>

acid neutralizing capacity expressed in acid neutralizing units

Note 1 to entry: Calculated as ANC<sub>BT</sub> (%) × 199,8 (Unit: mmol H+/kg).

### 3.6

## excess acid neutralizing capacity

ANC<sub>E</sub> calculated as a-ANC<sub>E</sub> (mmol H<sup>+</sup>/kg)/199,8

Note 1 to entry: Expressed as equivalent % CaCO<sub>3</sub>.

### 3.7

# excess acid neutralizing capacity expressed in acid neutralizing unit $a\text{-}\text{ANC}_{\text{E}}$

SPOCAS HCl titration to pH 4 minus titratable acidity to pH 6,5

Note 1 to entry: Unit: mmol H+/kg.

#### 3.8

### archive sample

dried and ground sample stored in a way to minimize sample oxidation in the long term

Note 1 to entry: The archive sample can be taken from either the laboratory or test sample.

Note 2 to entry: Sample containers can include sealed glass jars or multi-ply, gas-impermeable polymer bags or other containers capable of excluding moisture, and oxygen.