



BSI Standards Publication

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

Part 3: Suspension peroxide oxidation combined acidity and sulfur (SPOCAS) methodology

National foreword

This British Standard is the UK implementation of ISO 14388-3:2014.

The UK participation in its preparation was entrusted to Technical Committee EH/4, Soil quality.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2014. Published by BSI Standards Limited 2014

ISBN 978 0 580 78162 9

ICS 13.080.10

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 September 2014.

Amendments issued since publication

Date	Text affected
------	---------------

INTERNATIONAL STANDARD

ISO
14388-3

First edition
2014-08-01

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

Part 3: Suspension peroxide oxidation combined acidity and sulfur (SPOCAS) methodology

*Qualité du sol — Méthode de comptage acide-base pour les sols
sulfatés acides —*

*Partie 3: Méthode par oxydation au peroxyde en suspension combinée
à l'acidité et aux sulfures (SPOCAS)*



Reference number
ISO 14388-3:2014(E)

AVA Documents
Document Sharing Hub
© ISO 2014



COPYRIGHT PROTECTED DOCUMENT

© ISO 2014

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Principle	2
4.1 Actual acidity and retained acidity	2
4.2 Titratable peroxide acidity, excess acid neutralizing capacity and potential sulfidic acidity	2
5 Reagents	2
6 Apparatus	5
7 Procedure for determination of pH_{KCl} and TAA	6
7.1 Preparation of the suspension	6
7.2 Measurement of pH_{KCl}	6
7.3 Measurement of TAA	7
8 Calculation of TAA	8
9 Procedure for determination of sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) extractable with 1 mol/l potassium chloride	8
10 Calculation of S_{KCl}, Ca_{KCl} and Mg_{KCl}	9
11 Procedure for determination of peroxide pH (pH_{OX}), titratable peroxide acidity (TPA) and excess acid neutralizing capacity (ANC_{E})	9
11.1 Peroxide digestion of sample	9
11.2 Measurement of pH_{OX}	10
11.3 Carbonate modification (HCl titration to pH 4)	11
11.4 Decomposition of excess peroxide	11
11.5 Measurement of titratable peroxide acidity (TPA)	12
12 Calculation of TPA and ANC_{E}	13
12.1 Notation	13
12.2 TPA without carbonate modification	13
12.3 ANC_{E} (or TPA) with carbonate modification	14
13 Procedure for determining peroxide sulfur (S_{p}), calcium (Ca_{p}) and magnesium (Mg_{p})	14
14 Calculation of peroxide sulfur (S_{p}), calcium (Ca_{p}) and magnesium (Mg_{p})	15
15 Procedure for determining acid extractable sulfur in soil residue after peroxide oxidation (S_{RAS})	15
15.1 General	15
15.2 Rinsing of peroxide digested soil residue, extraction with 4 mol/l hydrochloric acid and determination of extractable sulfur	16
16 Calculation of S_{RAS}	16
16.1 Calculation of acidity attributed to S_{RAS} (acid-base accounting)	16
17 Calculation of TSA	17
18 Calculation of S_{POS}, Ca_{A} and Mg_{A}	17
19 Precision	17
20 Test report	18
Bibliography	20

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*:

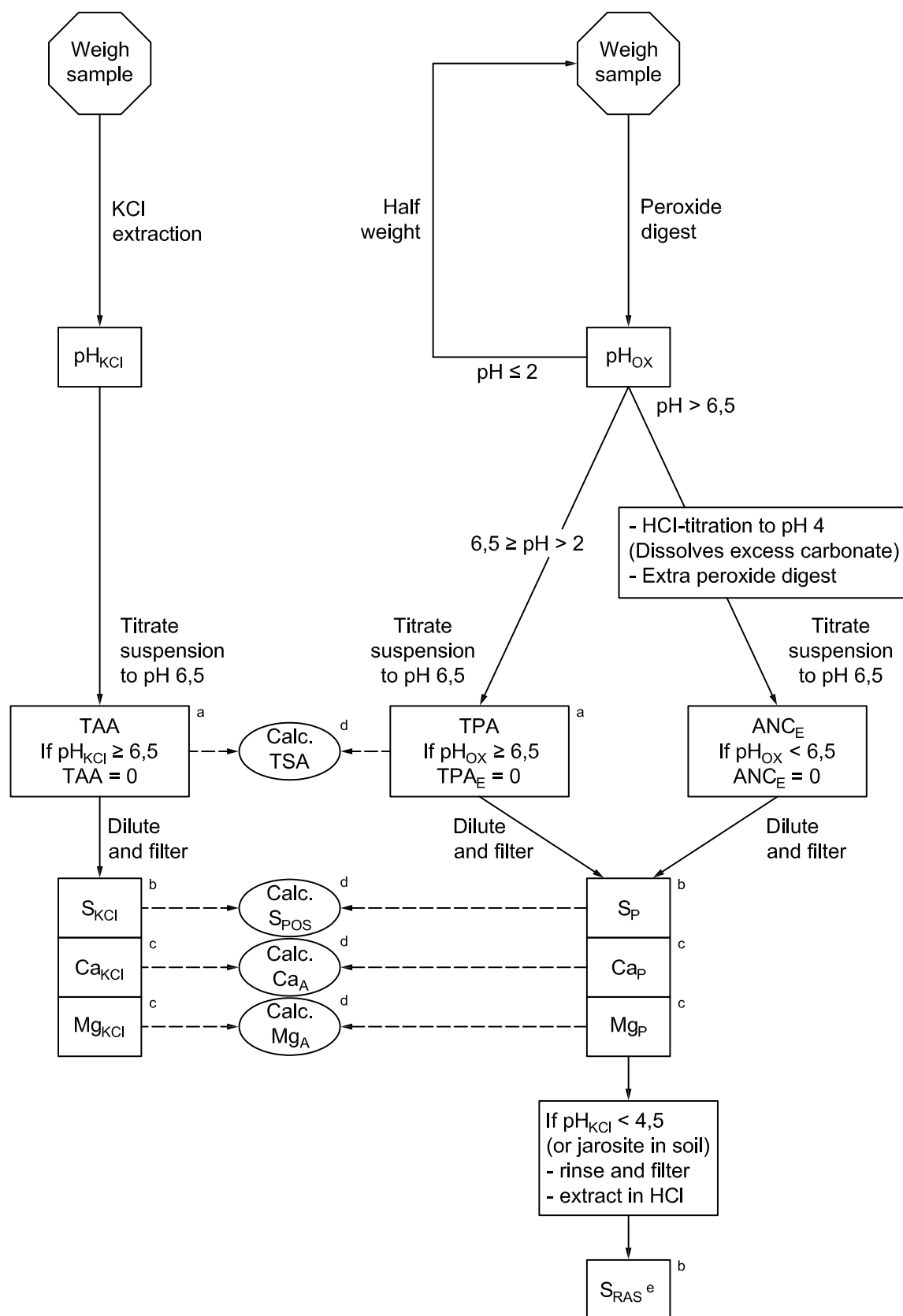
- *Part 1: Introduction and definitions, symbols and acronyms, sampling and sample preparation*
- *Part 2: Chromium reducible sulfur (CRS) methodology*
- *Part 3: Suspension peroxide oxidation combined acidity and sulfur (SPOCAS) methodology*

Introduction

The 'SPOCAS' suite uses the suspension peroxide oxidation combined acidity and sulfur (SPOCAS) method as the basis for determining an acid-base account for acid sulfate soils. Like the chromium suite, a decision-tree type process is followed ([Figure 1](#)). 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 results required to complete the acid-base accounting vary with the soil's existing acidity, as represented by pH_{KCl} . Table A.2 in ISO 14388-1:—¹⁾ shows the analyses required for the SPOCAS suite. This table uses results reported in acidity units. Alternatively, results in equivalent sulfur units may be utilized.

1) To be published.



Key

- | | | | | | |
|---|----------------------|---|--------------------------|---|---|
| a | Acidity titration | c | Cation/acid neutralising | e | Alternatively, S_{NAS} may be substituted for S_{RAS} |
| b | Sulfur determination | d | Calculated parameter | | |

Figure 1 — Flow diagram of SPOCAS suite

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

Part 3: Suspension peroxide oxidation combined acidity and sulfur (SPOCAS) methodology

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 specifies a suite of methods used to determine net acidity in acid sulfate soils. This part of ISO 14388 specifies a method for the determination of pH in a 1 mol/l potassium chloride soil suspension (pH_{KCl}) and where required, titratable actual acidity (TAA). Following this, potassium chloride extractable sulfur (S_{KCl}), calcium (Ca_{KCl}) and magnesium (Mg_{KCl}) are determined. On a separate test portion, following digestion with 30 % hydrogen peroxide, peroxide pH (pH_{Ox}), titratable peroxide acidity (TPA) and excess acid neutralizing capacity (ANC_{E}) are determined. Following this, peroxide sulfur (S_{p}), calcium (Ca_{p}) and magnesium (Mg_{p}) are determined. On samples where jarosite is present, or where pH_{KCl} is $< 4,5$, residual acid soluble sulfur (S_{RAS}) is determined on the soil residue remaining after peroxide digestion. Titratable sulfidic acidity (TSA), reacted calcium (Ca_{A}), reacted magnesium (Mg_{A}) and peroxide oxidizable sulfur (S_{POS}) are then determined by difference. For peat samples containing substantial levels of organic sulfur, the SPOCAS suite is generally unsuitable for estimating sulfide content, and the chromium reducible suite of procedures should be used instead.

2 Normative references

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

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

ISO 835, *Laboratory glassware — Graduated pipettes*

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*

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 8655-5, *Piston-operated volumetric apparatus — Part 5: Dispensers*

ISO 14388-1:—²⁾, *Piston-operated volumetric apparatus — Part 5: Dispensers*

3 Terms and definitions

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

4 Principle

4.1 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** can be determined as residual acid-soluble sulfur (S_{RAS}). This is measured by 4 mol/l hydrochloric acid extraction of the soil residue remaining after the peroxide digestion of the soil following determination of titratable peroxide acidity (after appropriate washing of the residue to remove soluble sulfate). Alternatively, retained acidity is measured as the difference between S_{HCl} (the sulfur measured following overnight extraction of a separate sub-sample with 4 mol/l hydrochloric acid) and S_{KCl} (known as net acid-soluble sulfur, S_{NAS}). The **former** approach is preferable in highly organic soils where S_{NAS} may overestimate retained acidity due to the inclusion of organic S in the result.

4.2 Titratable peroxide acidity, excess acid neutralizing capacity and potential sulfidic acidity

A separate test portion of soil is digested with 30 % hydrogen peroxide and acidity is generated from the oxidation of sulfides (and organic matter where present). Following digestion, pH_{OX} is measured on the digest solution. If the pH after initial peroxide digestion is $\leq 6,5$ then KCl solution is added to the sample which is then titrated to pH 6,5 to determine titratable peroxide acidity (TPA). If after peroxide digestion the pH is $> 6,5$ a HCl titration to pH 4 is performed, followed by an additional peroxide digest step. This allows the calculation of excess acid neutralizing capacity (ANC_{E}), i. e. the available ANC in excess of that needed to neutralize the potential acidity from the presence of sulfides or other acid generating species in the soil.

After appropriate dilution of the soil suspension, peroxide sulfur (S_{P}), calcium (Ca_{P}) and magnesium (Mg_{P}) are measured by suitable analytical technique(s). These analytes allow the calculation of various other soil parameters. Acid neutralizing capacity can also be calculated from reacted calcium (Ca_{A}) and magnesium (Mg_{A}) results (i. e. the difference in soluble calcium and magnesium before and after peroxide oxidation).

Potential sulfidic acidity, (the acidity latent in reduced inorganic iron sulfide compounds), can be determined as peroxide oxidizable sulfur (S_{POS}) (the calculated difference between peroxide sulfur (S_{P}) peroxide and KCl-extractable sulfur (S_{KCl})).

5 Reagents

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).

2) To be published.