BSI Standards Publication

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

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

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National foreword

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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.

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Amendments issued since publication

Date	Text affected

INTERNATIONAL STANDARD

ISO
14388-1

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

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

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

Partie 1: Introduction et définitions, symboles et acronymes, échantillonnage et préparation des échantillons

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Contents

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) 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_2); 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):

$$FeS_2 + {}^{15}/_4\, O_2 + {}^7/_2\, H_2O \rightarrow Fe(OH)_3 + 4\, H^+ + 2\, SO_4^{2-} \tag{1}$$

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* [$KFe_3(SO_4)_2(OH)_6$], 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.

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 'acid-base 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 ≥ 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 $CaCO_3$ from shell). The ANC can be determined by various methods [e. g. inorganic carbon (C_{IN}), using a combustion furnace, or acid digestion followed by titration of unreacted acid (ANC_{BT})].

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_{BT}). 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_E). 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 alkali cations), allowing greater interpretation of what is occurring in a soil or soil profile.
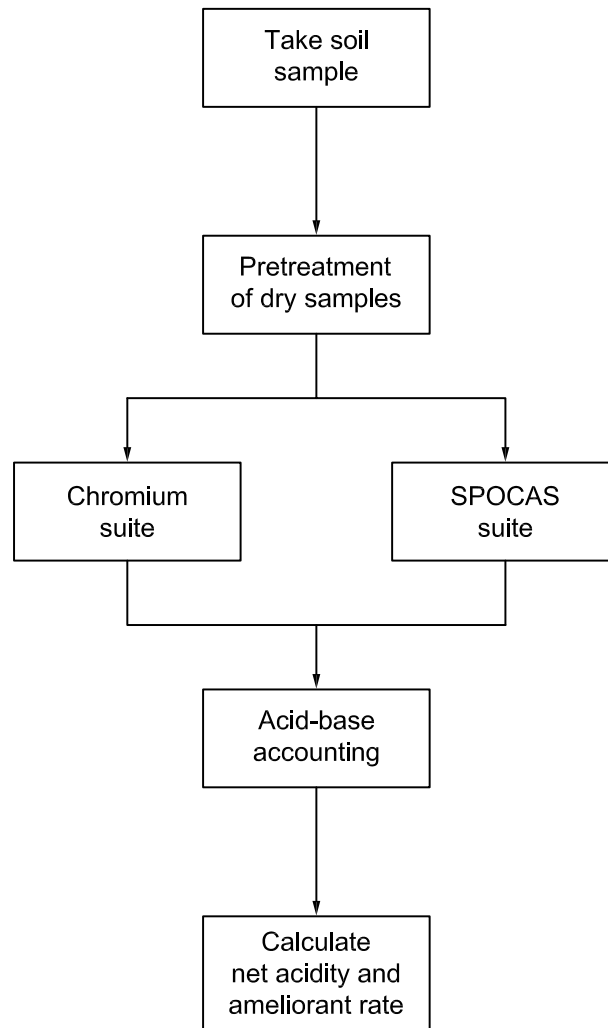
Figure 1 — Flow diagram of acid sulfate soil analysis

Key
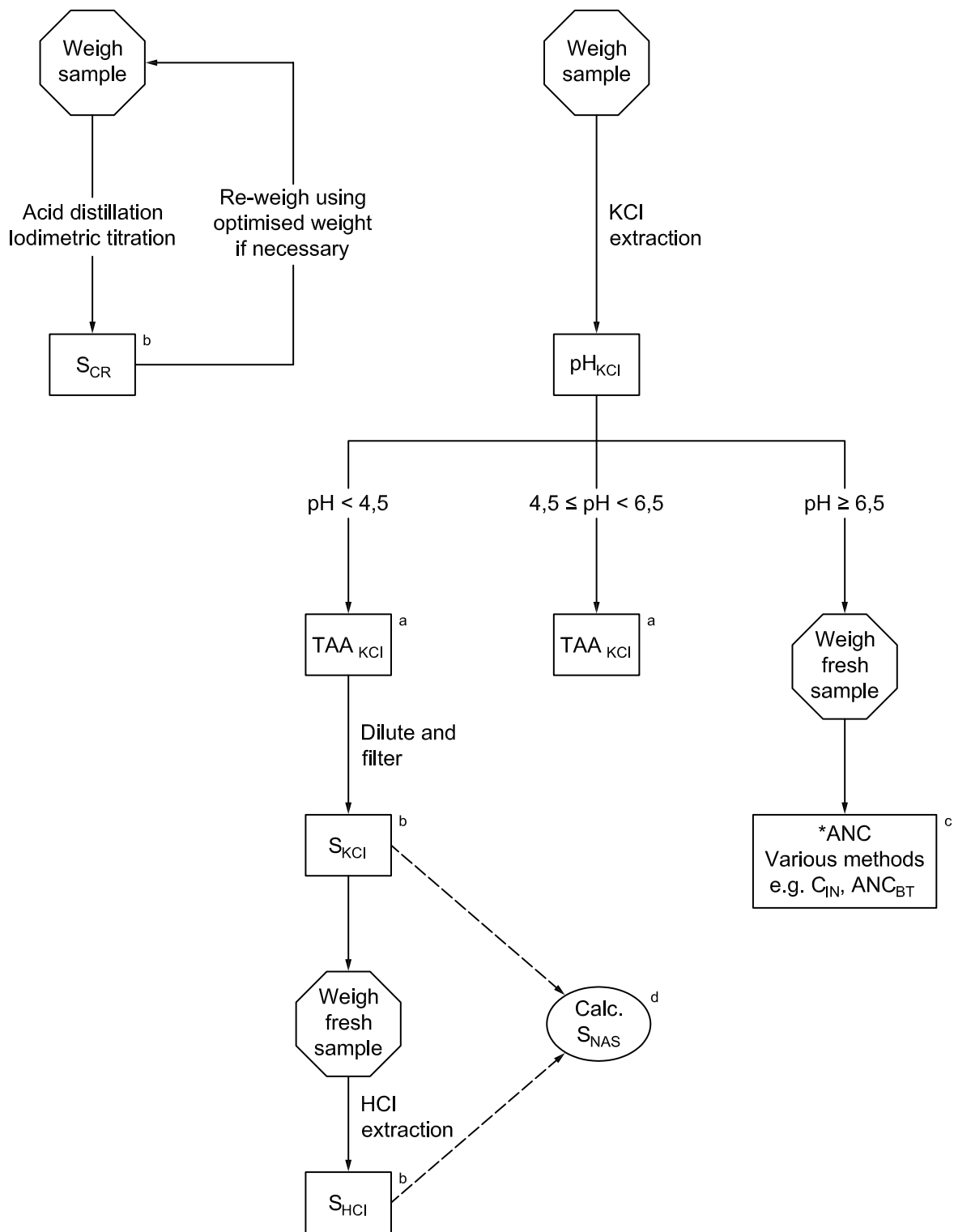
a   acidity titration

b   sulfur determination

c   acid neutralising determination

d   calculated parameter

Figure 2 — Chromium suite flow diagram

Weigh sample

Weigh sample

KCl extraction

Half weight

Peroxide digest

pH_{KCl}

pH_{OX}

pH ≤ 2

pH > 6,5

- HCl-titration to pH 4
(Dissolves excess carbonate)
- Extra peroxide digest

6,5 ≥ pH > 2

Titrate suspension to pH 6,5

Titrate suspension to pH 6,5

Titrate suspension to pH 6,5

TAA [a]
If pH_{KCl} ≥ 6,5
TAA = 0

Calc. TSA [d]

TPA [a]
If pH_{OX} ≥ 6,5
TPA_E = 0

ANC_E
If pH_{OX} < 6,5
ANC_E = 0

Dilute and filter

Dilute and filter

Dilute and filter

S_{KCl} [b]

Calc. S_{POS} [d]

S_P [b]

Ca_{KCl} [c]

Calc. Ca_A [d]

Ca_P [c]

Mg_{KCl} [c]

Calc. Mg_A [d]

Mg_P [c]

If pH_{KCl} < 4,5
(or jarosite in soil)
- rinse and filter
- extract in HCl

S_{RAS} [e] [b]

Key

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

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 % $CaCO_3$.

3.4
acid neutralizing capacity by back titration
ANC_{BT}
acid neutralizing capacity measured by acid digest followed by back titration of unreacted acid

Note 1 to entry: Expressed as equivalent % $CaCO_3$.

3.5
acid neutralizing capacity by back titration
a-ANC_{BT}
acid neutralizing capacity expressed in acid neutralizing units

Note 1 to entry: Calculated as ANC_{BT} (%) × 199,8 (Unit: mmol H^+/kg).

3.6
excess acid neutralizing capacity
ANC_E
calculated as a-ANC_E (mmol H^+/kg)/199,8

Note 1 to entry: Expressed as equivalent % $CaCO_3$.

3.7
excess acid neutralizing capacity expressed in acid neutralizing unit
a-ANC_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.

3.9
acid sulfate soil
ASS
complex group of predominantly low-lying coastal soils and sediments that contain iron sulfides and/or their oxidation products

3.10
inorganic carbon
C_{IN}
calculated as the difference of total carbon (C_T) minus total organic carbon (C_{TO})

Note 1 to entry: Unit: % C.

3.11
inorganic carbon expressed in equivalent acid neutralizing unit
$a\text{-}C_{IN}$
calculated as C_{IN} (%) × 8,333 × 199,8

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

Note 2 to entry: Assumption is that all inorganic carbon is in the form of carbonate.

3.12
total carbon
C_T
carbon measured by combustion

Note 1 to entry: Unit: % C.

3.13
total organic carbon
C_{TO}
carbon measured by combustion following pre-treatment with acid

Note 1 to entry: Unit: % C.

3.14
reacted calcium
Ca_A
calcium made soluble by acid generated from 30 % hydrogen peroxide digestion and calculated as (Ca_P - Ca_{KCl})

Note 1 to entry: Unit: % Ca.

3.15
reacted calcium expressed in equivalent acid neutralizing unit
$a\text{-}Ca_A$
calculated as Ca_A (%) × 499

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

Note 2 to entry: Assumption is that all such calcium is in carbonate, oxide or hydroxide forms.

3.16
hydrochloric acid extractable calcium
Ca_{HCl}
calcium extracted in 4 mol/l HCl (1:40, soil: solution for 16 h)

Note 1 to entry: Unit: % Ca.

3.17
potassium chloride extractable calcium
Ca_{KCl}
calcium soluble in 1 mol/l potassium chloride (after TAA titration)

Note 1 to entry: Unit: % Ca.

3.18
net acid-soluble calcium
Ca_{NAS}
calculated as (Ca_{HCl} - Ca_{KCl})

Note 1 to entry: Unit: % Ca.

3.19
net acid-soluble calcium expressed in equivalent acid neutralizing unit
a-Ca_{NAS}
calculated as Ca_{NAS} (%) × 499

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

Note 2 to entry: Assumption is that all such calcium is in carbonate, oxide or hydroxide forms.

3.20
peroxide calcium
Ca_P
calcium extracted in 1 mol/l potassium chloride and determined following 30 % hydrogen peroxide digestion and TPA titration

Note 1 to entry: Unit: % Ca.

3.21
existing acidity
acidity already present in acid sulfate soils, usually as a result of oxidation of sulfides, but which can also be from organic material or acidic cations

Note 1 to entry: It can be further subdivided into actual and retained acidity, i. e. Existing acidity = Actual acidity + Retained acidity

3.22
field sample
field condition sample, prepared by sampling personnel for sending to the laboratory, from which a test sample can be prepared

3.23
fineness factor
factor applied to the acid neutralizing capacity result in the acid-base account to allow for the poor reactivity of coarser carbonate or other acid neutralizing material

3.24
laboratory sample
dried (and if required pre-ground) field sample that has not undergone the final grinding to < 75 μm

3.25
reacted magnesium
Mg_A
magnesium solubilised by acid generated from 30 % hydrogen peroxide digestion and calculated as (Mg_P - Mg_{KCl})

Note 1 to entry: Unit: % Mg.

3.26
reacted magnesium expressed in equivalent acid neutralizing unit
a-Mg$_A$
calculated as Mg$_A$ (%) × 822,6

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

Note 2 to entry: Assumption is all magnesium is in carbonate, oxide or hydroxide forms.

3.27
potassium chloride extractable magnesium
Mg$_{KCl}$
magnesium extracted in 1 mol/l potassium chloride (after TAA titration)

Note 1 to entry: Unit: % Mg.

3.28
net acid-soluble magnesium
Mg$_{NAS}$
calculated as (Mg$_{HCl}$ - Mg$_{KCl}$)

Note 1 to entry: Unit: % Mg.

3.29
net acid-soluble magnesium expressed in equivalent acid neutralizing unit
a-Mg$_{NAS}$
calculated as Mg$_{NAS}$ (%) × 822,6

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

Note 2 to entry: Assumption is all magnesium is in carbonate, oxide or hydroxide forms.

3.30
peroxide magnesium
Mg$_P$
magnesium extracted in 1 mol/l potassium chloride and determined following 30 % hydrogen peroxide digestion and TPA titration

Note 1 to entry: Unit: % Mg.

3.31
monosulfides
highly reactive iron sulfide minerals found in acid sulfate soil that have the approximate formula 'FeS'

3.32
net acidity
result obtained when the values for various components of potential and existing soil acidity and acid neutralizing capacity are substituted into the acid-base accounting equation

Note 1 to entry: Calculated as:

Net acidity = Potential sulfidic acidity + Existing acidity − (Acid neutralizing capacity/Fineness factor)

3.33
potential acid sulfate soil
PASS
acid sulfate soil that contains sufficient inorganic sulfide to cause severe acidification upon oxidation

Note 1 to entry: Typically, undisturbed PASS has a soil pH that is near-neutral.

3.34
potassium chloride suspension pH
pH_{KCl}
pH in a 1:40 (*W/V*) suspension of soil in a solution of 1 mol/l potassium chloride

3.35
peroxide oxidized suspension pH
pH_{OX}
pH in a suspension of soil after initial digestion by 30 % hydrogen peroxide

3.36
potential sulfidic acidity
acidity in acid sulfate soil that is released if the sulfide minerals they contain (e. g. pyrite) are fully oxidized

Note 1 to entry: It can be estimated by titration (i. e. TSA) if no acid neutralizing material is present, or calculated from S_{POS} or S_{CR} results.

3.37
retained acidity
'less available' fraction of the existing acidity (not measured by the TAA) that can be released slowly into the environment by hydrolysis of relatively insoluble sulfate salts (such as jarosite, natrojarosite, and other iron and aluminium hydroxy-sulfate minerals)

3.38
chromium reducible sulfur
S_{CR}
sulfide measured by iodimetric titration after acidic chromous chloride digestion

Note 1 to entry: Unit: % S.

3.39
chromium reducible sulfur expressed in acid equivalent unit
$a\text{-}S_{CR}$
calculated as S_{CR} (%) × 623,7

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

Note 2 to entry: Assumption is that each mole of chromium reducible sulfur releases 2 moles of acidity.

3.40
hydrochloric acid extractable sulfur
S_{HCl}
sulfur extracted in 4 mol/l hydrochloric acid (1:40, soil:solution for 16 h)

Note 1 to entry: Unit: % S.

3.41
potassium chloride extractable sulfur
S_{KCl}
sulfur soluble in 1 mol/l potassium chloride (after TAA titration)

Note 1 to entry: Unit: % S.

3.42
net acid-soluble sulfur
S_{NAS}
calculated as (S_{HCl} - S_{KCl})

Note 1 to entry: Unit: % S.

3.43
net acid-soluble sulfur expressed in acid equivalent unit
a-S$_{NAS}$
estimate of acidity produced on hydrolysis of jarositic and other low solubility hydroxy-sulfate minerals, calculated as S$_{NAS}$ (%) × 0,75 × 623,7

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

Note 2 to entry: Assumption is that one mole of sulfur (e. g. in jarosite) produces 1,5 moles of acidity.

3.44
oxidizable sulfur
S$_{OX}$
broad term encompassing sulfur compounds that oxidize to produce acid

Note 1 to entry: Unit: % S.

3.45
peroxide sulfur
S$_P$
sulfur extracted in 1 mol/l potassium chloride and determined following 30 % hydrogen peroxide digestion and TPA titration

Note 1 to entry: Unit: % S.

3.46
peroxide oxidizable sulfur
S$_{POS}$
sulfur oxidized by peroxide digestion and calculated as (S$_P$ - S$_{KCl}$)

Note 1 to entry: Unit: % S.

3.47
peroxide oxidizable sulfur expressed in acid equivalent unit
a-S$_{POS}$
calculated as S$_{POS}$ (%) × 623,7

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

Note 2 to entry: Assumption is that each mole of peroxide oxidizable sulfur releases 2 moles of acidity.

3.48
residual acid-soluble sulfur
S$_{RAS}$
sulfur extracted in 4 mol/l hydrochloric acid (1:40, soil: solution for 16 h) from washed soil residue used for TPA and S$_P$ determination as part of SPOCAS

Note 1 to entry: Unit: % S.

3.49
residual acid-soluble sulfur expressed in acid equivalent unit
a-S$_{RAS}$
estimate of acidity produced on hydrolysis of jarositic and other 'insoluble' hydroxy-sulfates, calculated as S$_{RAS}$ (%) × 0,75 × 623,7

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

Note 2 to entry: Assumption is that one mole of sulfur (e. g. in jarosite) produces 1,5 moles of acidity.

3.50
titratable actual acidity
TAA
acidity titration with standardized sodium hydroxide on 1:40, suspension in 1 mol/l potassium chloride to pH 6,5

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

3.51
test portion
amount of soil material taken from the test sample on which the actual analysis or method of test is performed

3.52
test sample
sample prepared by appropriate drying, (sieving where necessary) and fine grinding of the laboratory sample to < 75 μm from which the test portion is taken

3.53
titratable peroxide acidity
TPA
acidity titration with standardized sodium hydroxide to pH 6,5 on 1:40 suspension in 1 mol/l potassium chloride after 30 % hydrogen peroxide digestion

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

3.54
titratable sulfidic acidity
TSA
calculated as (TPA - TAA)

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

4   Abbreviated terms

CRS   chromium reducible sulphur

          NOTE   The analytical method is symbolized by S_{CR}.

RIS   reduced inorganic sulphur

          NOTE   Includes disulfide sulfur, acid volatile sulfur and elemental sulfur.

SPOCAS   suspension peroxide oxidation combined acidity and sulfur method

5   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 at 85 °C ± 5 °C to kill bacteria and minimize further pyrite oxidation 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 μm).

6   Apparatus

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. The material of the apparatus used shall not contribute to the levels of the analytes

being determined. Detailed descriptions of the apparatus to be employed are not given, but should meet the minimum requirements indicated below.

6.1   Balance, capable of measuring up to 1 000 g to an accuracy of ± 0,1 g.

6.2   Drying oven, fan-forced, air-extracting, and capable of maintaining samples at a temperature of 85 °C ± 5 °C.

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

6.3   Drying trays, made of polymer or other inert material, capable of withstanding temperatures of up to 90 °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 can also be lined with 'plastic wrap' to alleviate the need to wash sample trays.

6.4   Grinding apparatus 1, grinding mill (typically a hammer type mill).

6.5   Grinding apparatus 2, ring mill (or similarly forceful grinding apparatus) capable of reducing a laboratory sample to < 75 μm (see also 8.4.)

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

6.7   Mortar and pestle, e. g. glass, porcelain, or agate.

6.8   Sieve, with an aperture of 2 mm.

6.9   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.

7   Sampling and 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 °C until received by the laboratory to reduce the possibility of oxidation.

NOTE 1   A portable 12 V car freezer or cold box containing dry ice are the most efficient coolers, but if not available, ordinary ice can be employed for cooling.

It is very 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 (see Clause 8) is not to be commenced immediately, the samples shall be managed to minimize oxidation. Methods include the following:

a)   freezing the field sample and maintaining it in a frozen state in sealed, air-tight containers;

b)   stored cold (i. e. < 4 °C) in an inert atmosphere (e. g. N_2, Ar);

c)   vacuum sealing and stored cold (i. e. < 4 °C) or frozen;

d)   quick oven-drying the sample at 85 °C ± 5 °C in an oven (6.2). 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 six weeks of drying, then the sample shall be stored as per an archive sample (see 8.5.2).

This preparation methodology 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.

NOTE 2   It is recommended to collect a field sample that provides approximately 200 g of dried sample.

NOTE 3   Field personnel can 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 during sampling) should be recorded in field sampling notes.

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.

8   Sample pre-treatment in the laboratory

8.1   General

Samples shall be processed so as to minimize any possible temporal changes in soil properties.

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

b)   stored samples, or samples being processed nearby.

8.2   Drying

8.2.1   General

The entire field sample shall be dried in accordance with either of the procedures specified in 8.2.2 and 8.2.3.

WARNING — Dried acid sulfate soils can be dusty and contain chemical contaminants, spores or pathogens and strongly acidic substances, and can pose health hazards.

8.2.2   Oven-drying

Spread on a drying tray in as thin a layer as possible (and to a maximum thickness of 2 cm). Where possible, cloddy or plastic clay samples should be broken into lumps no more than 1 cm to 2 cm in diameter. If an estimate of field moisture is required, then retain a representative portion of the soil in a sealed polyethylene bag or 'moisture container'. An 'as received moisture content' determination can then be made.

Dry in an oven (6.2) at a temperature of 85 °C ± 5 °C for 48 h, or after an initial drying period of 12 h until the weight loss from a sample, over a 6 h interval is less than 1 % of the final weight.

NOTE 1   Samples are rapidly dried in a fan-forced air-extracting oven at 85 °C ± 5 °C to kill bacteria and to minimize further pyrite oxidation.

NOTE 2   Typically, pH decreases of 0,25 to 1 units have been recorded on oven-drying, without any measurable oxidation of sulfides, although larger pH decreases on oven-drying large samples and some oxidation equating to 2 % of average TPA have been described.[1]

NOTE 3   It was demonstrated that oxidation of between 3 % and 5 % of the reduced inorganic sulfur (as measured by S_{CR}) occurred in a wide variety of materials even when dried quickly in a fan-forced oven, and that this was accompanied by large increases in water-soluble sulfate.[2]

8.2.3   Freeze-drying

As an alternative to oven-drying, freeze-drying can be used to minimize sample oxidation (e. g. ISO 16720[4]). Freeze-dry soil sample according to the manufacturer's instruction manual for your specific freeze-dryer.

NOTE   While freeze-drying soil samples can cause minor changes in S constituent analysis when compared to fresh, field-moist, refrigerated samples, greater change is caused by oven-drying.[3]

8.3   Removal of > 2 mm size material

8.3.1   Preliminary separation

After drying the field sample; shells, stones, and any other particles greater than 2 mm shall be removed.

Do not remove soil aggregates, roots, and partially decomposed organic remnants such as small roots which can contain sulfides.

Removed coarse materials (fraction A) shall be identified, recorded, and if necessary, retained; if these materials contain significant sources of existing or potential acidity (e. g. sulfides) or alkalinity (e. g. carbonate), this should be considered separately. The remaining dried material (<2 mm) is termed fraction B.

Fine material adhering to the removed coarse material should be returned to the sample.

NOTE   Appreciable contents of shell and stone material can effectively 'dilute' field material and affect management, such as calculation of liming rates based on fraction B.

8.3.2   Soil fractionation and initial grinding

Fraction B shall be processed as follows:

a)   Crush any aggregates with a hammer or mallet (6.6) or a mortar and pestle (6.7) and pass soil through a 2 mm sieve (6.8) (fraction C). Fraction C is known as the laboratory sample. If required for calculating the percentage of > 2 mm material, fraction C is weighed (m_2).

b)   Avoid crushing any coarse shell material to less than 2 mm.

c)   If required, the material retained on the sieve (fraction D) shall be identified, noted, and then added to fraction A; and the combined material weighed (m_1).

NOTE   Fraction C can be passed through a grinding mill (6.4) to facilitate representative sub-sampling and/or to enhance the fine grinding operation. This ground sample is still called the laboratory sample.

8.4   Final grinding of the laboratory sample

A representative sub-sample (at least 50 g) of the laboratory sample shall be ground to a powder (<75 μm) in a ring mill or similarly forceful grinding apparatus (6.5). This sample is termed the test sample. The ring mill shall be cleaned between samples by grinding acid-washed silica sand and then completely removing the ground silica sand (e. g. using dry compressed air).

Grinding the test sample to a particular particle size can impact on the results from certain analytical methods. Until new analytical data determines otherwise, test samples should be ground to < 75 μm.

As a guide, for 50 g of sample, mineral soils should be ground for at least 1 min and organic rich soils (such as peats) for at least 5 min. Longer grinding times might be necessary depending on the sample

size and the capacity of the grinding apparatus. However, to avoid excessive heating of sample, grinding time should not be prolonged. It is recommended that the grinding apparatus is timer-controlled.

8.5   Storage and archiving of samples

8.5.1   Storage of samples

Ground samples shall be stored in a cool, dry place in an air-tight plastic or other inert container (e. g. sealable plastic bag, or plastic/glass jar) to minimize oxidation. All methods of test shall be performed on a test sample within six weeks of initial drying. If it is not possible to conduct all methods of test in this period, then the sample shall be stored following the procedure in 8.5.2.

8.5.2   Archiving samples

If required, laboratory and/or test samples shall be archived in a manner that minimizes absorption of moisture and diffusion of atmospheric oxygen into the sample. Procedures to achieve this include:

a)   storing sample in an inert atmosphere (e. g. N_2, Ar);

b)   vacuum-sealing the sample (preferably after being purged with inert gas, e. g. N_2) in multiply gas-impermeable polymer (or foil-lined) bag.

NOTE   Air is capable of diffusing through commonly available polyethylene bags, and the bag should be of sufficient thickness to maintain the vacuum and to limit the rate of oxygen diffusion. Bags constructed with layers of polymer less permeable to oxygen are also available (e. g. ethylene vinyl alcohol (EVOH)). Bags with a foil covering which present a better barrier to oxygen diffusion are also available.

9   Calculation of extraneous material

The proportion of extraneous material (W_{ext}) (fractions A and D), in the 'dried' laboratory sample shall be calculated using Formula (2):

$$W_{ext} \ (\%) = \left[m_1 / \left(m_1 + m_2 \right) \right] \times 100 \tag{2}$$

where

$\quad W_{ext}$ is the proportion of extraneous/coarse material in 'dried' sample, expressed as percentage by mass (%);

$\quad m_1$ is the mass of 'dried' shell or stone material > 2 mm (fractions A and D), expressed in grams (g);

$\quad m_2$ is the mass of 'dried' sample after removal of extraneous/coarse material (fraction C), expressed in grams (g).

10   Pre-treatment report

The report shall contain the following information:

a)   reference to this part of ISO 14388;

b)   sample identification as submitted to the laboratory;

c)   date on which the sample was taken (if known);

d)   date on which the pre-treatment procedure was carried out;

e)   whether the sample was oven-dried or freeze-dried;

f)   details of any variation to the sample pre-treatment procedure;

g)   all masses determined;

h)   any unusual observations made during the course of the pre-treatment processes which can have had an effect on the subsequent test result.

11 Calculation of the acid-producing potential of acid sulfate soil using an acid-base accounting method

11.1 General

The method calculates the net acidity of acid sulfate soil. The properties required for such a calculation include:

a)   the potential sulfidic acidity (acidity that can develop upon oxidation of inorganic sulfides);

b)   the existing acidity, which is the sum of titratable actual acidity (consisting of readily soluble and exchangeable acidity) and retained acidity (consisting of sparingly soluble acidity residing in iron and aluminium hydroxy-sulfate phases such as jarosite);

c)   the acid neutralizing capacity, consisting principally of exchangeable alkalinity and Mg and Ca carbonates, oxides or hydroxides.

A fineness factor is used to modify acid neutralizing capacity to allow for the often poor reactivity of coarser carbonate or other acid neutralizing materials. The minimum value for the fineness factor is 1,5 for finely divided pure agricultural lime, but can be as high as 3,0 for coarser shell material.

The Net acidity in the soil material is calculated as given in 11.3.

If Net acidity is positive, then the acid sulfate soil material is considered to be capable of causing adverse environmental effects due to acidification.

A number of approaches exist for the determination of most of the components within the acid-base accounting procedure. The individual components can be combined into analytical suites that streamline the process of acid-base accounting. The two principal analytical suites are the Chromium suite and the SPOCAS suite. These suites are detailed in ISO 14388-2 and ISO 14388-3.

The results required to complete the acid-base account vary with the soil acidity, as represented by pH_{KCl} Tables A.1 and A.2 in Annex A show the analyses required for the Chromium and SPOCAS suites respectively. These tables use results reported in acidity units. Alternatively, results in sulfur units can be used after appropriate conversion.

11.2 Procedure

11.2.1 Determination of potential sulfidic acidity

Potential sulfidic acidity, expressed in acid equivalent units, shall be measured as either:

a)   chromium reducible sulfur ($a\text{-}S_{CR}$), or

b)   peroxide oxidizable sulfur ($a\text{-}S_{POS}$).

11.2.2 Determination of pH_{KCl}

The pH_{KCl} shall be determined according to ISO 14388-2 and ISO 14388-3.

If $pH_{KCl} < 4,5$, then both the titratable actual acidity and the retained acidity need to be determined.

If $4,5 \leq pH_{KCl} < 6,5$, then the titratable actual acidity (see ISO 14388-2 and ISO 14388-3) needs to be determined.

If $pH_{KCl} \geq 6,5$, then the acid neutralizing capacity needs to be determined.

11.2.3 Determination of existing acidity

Existing acidity = titratable actual acidity + retained acidity

Titratable actual acidity (TAA) is determined according to ISO 14388-2 and ISO 14388-3.

Retained acidity expressed in acid equivalent units, is either determined as residual acid-soluble sulfur (a-S_{RAS} see ISO 14388-3) or calculated as net acid-soluble sulfur (a-S_{NAS} see ISO 14388-2).

If jarosite is visibly identified in the sample, then retained acidity needs to be determined regardless of the pH_{KCl} of the sample.

11.2.4 Determination of acid neutralizing capacity

Acid neutralizing capacity, expressed in acid equivalent units, shall be measured where $pH_{KCl} \geq 6,5$ by determining:

a)   acid neutralizing capacity by back titration (a-ANC_{BT}, see ISO 14388-2);

b)   excess acid neutralizing capacity (a-ANC_E, see ISO 14388-3) in combination with peroxide oxidizable sulfur (a-S_{POS}, ISO 14388-3), where pH_{OX} (see ISO 14388-3) is $\geq 6,5$;

c)   calculating titratable sulfidic acidity (TSA, see ISO 14388-3), in combination with peroxide oxidizable sulfur (a-S_{POS}, ISO 14388-3), where pH_{OX} (see ISO 14388-3) is $< 6,5$.

NOTE 1   ANC can be determined by alternate methods such as inorganic carbon (C_{IN}) or the sum of reacted calcium (a-Ca_A) plus magnesium (a-Mg_A) (see ISO 14388-3).

NOTE 2   a-ANC_{BT} is usually utilized to calculate the ANC component in the chromium suite for acid-base accounting (see Table A.1 and ISO 14388-2).

NOTE 3   a-ANC_E, TSA, a-Ca_A and a-Mg_A are usually utilized to calculate the ANC component in the SPOCAS suite for acid-base accounting (see Table A.2).

11.2.5 Fineness factor

The minimum value for the fineness factor is 1,5 for finely divided pure agricultural lime and finely divided carbonates in soil, but can be as high as 3,0 for coarser shell material.

A default value of 1,5 is typically used for the fineness factor but the appropriate regulatory authority should be consulted regarding the application of the fineness factor.

11.3 Calculation

The calculation of net acidity could be expressed as follows:

a)   In acid equivalent units

The Net acidity in the soil material is calculated (mmol H_+/kg) as:

Net acidity = Potential sulfidic acidity + Existing acidity – (Acid neutralizing capacity/Fineness factor)

b)   As equivalent % $CaCO_3$

The Net acidity can alternatively be expressed in terms of the equivalent amount $CaCO_3$ the soil would neutralize using the following conversion:

Net acidity (equivalent % $CaCO_3$) = Net acidity {in mmol H^+/kg}/199,8

NOTE   Tables A.1 and A.2 in Annex A indicate the acid-base accounting results in acidity units utilized using the chromium suite and SPOCAS suite respectively. Table A.3 provides conversions between units for acid-base accounting purposes.

ISO 14388-1:2014(E)

Annex A
(normative)

Chromium suite and SPOCAS suite acid-base accounting

Table A.1 — Chromium suite acid-base accounting – acidity units. Analyses required

Preliminary analysis results	Potential acidity		Actual acidity		Retained acidity		ANC
$pH_{KCl} \geq 6,5$	$a\text{-}S_{CR}$					–	$[a\text{-}C_{IN}/FF^a]$
	$a\text{-}S_{CR}$					–	or
							$[a\text{-}ANC_{BT}/FF^a]$
$4,5 \leq pH_{KCl} < 6,5$	$a\text{-}S_{CR}$	+	TAA				
$pH_{KCl} < 4,5$	$a\text{-}S_{CR}$	+	TAA	+	$a\text{-}S_{NAS}$		
[a] FF Fineness factor (at least 1,5)							

Table A.2 — SPOCAS suite acid-base accounting – acidity units. Analyses required

Preliminary analysis results	Potential acidity		Actual acidity		Retained acidity		ANC
TPA = 0	$a\text{-}S_{POS}$					–	$[(a\text{-}S_{POS} + a\text{-}ANC_E{}^b)/FF^a]$
$pH_{KCl} \geq 6,5$	$a\text{-}S_{POS}$					–	or
							$[(a\text{-}Ca_A + a\text{-}Mg_A)/FF^a]$
TPA > 0	$a\text{-}S_{POS}$					–	$[(a\text{-}S_{POS} - TSA^c)/FF^a]$
$pH_{KCl} \geq 6,5$	$a\text{-}S_{POS}$					–	or
							$[(a\text{-}Ca_A + a\text{-}Mg_A)/FF^a]$
$4,5 \leq pH_{KCl} < 6,5$	$a\text{-}S_{POS}$	+	TAA				
TPA > 0	$a\text{-}S_{POS}$	+	TAA	+	$a\text{-}S_{RAS}{}^d$		
$pH_{KCl} < 4,5$							

[a]   FF   Fineness factor (at least 1,5).

[b] ANC_E represents the ANC left after any acidity, represented by $a\text{-}S_{POS}$, is neutralised. Therefore the full ANC equals the sum of $a\text{-}S_{POS}$ and ANC_E.

[c]   Where TPA > 0, TSA will only be less than $a\text{-}S_{POS}$ where some neutralizing capacity has been used in neutralizing acidity. If the TSA is greater than the $a\text{-}S_{POS}$, the result from $[(a\text{-}S_{POS} - TSA)/FF]$ is added to the potential sulfidic acidity.

[d] $a\text{-}S_{RAS}$ can alternatively be substituted by $a\text{-}S_{NAS}$ if available.

Table A.3 — Conversions for SPOCAS and Chromium suite acid-base accounting

Symbol and units	Conversion
s-TAA (equiv. % pyrite S)	(TAA/623,7) (TAA in mmol H^+/kg)
s-TPA (equiv. % pyrite S)	(TPA/623,7) (TPA in mmol H^+/kg)
s-TSA (equiv. % pyrite S)	(TSA/623,7) (TSA in mmol H^+/kg)
a-S_{POS} (mmol H^+/kg)	(S_{POS} x 623,7) (S_{POS} in %S)
a-S_{CR} (mmol H^+/kg)	(S_{CR} x 623,7) (S_{CR} in %S)
a-S_{NAS} (mmol H^+/kg)	(S_{NAS} x 0,75 × 623,7) (S_{NAS} in %S)
s-S_{NAS} (equiv. % pyrite S)	(S_{NAS} x 0,75) (S_{NAS} in %S)
a-S_{RAS} (mmol H^+/kg)	(S_{RAS} x 0,75 × 623,7) (S_{RAS} in %S)
s-S_{RAS} (equiv. % pyrite S)	(S_{RAS} x 0,75) (S_{RAS} in %S)
a-Ca_A (mmol H^+/kg)	(Ca_A x 499,0) (Ca_A in %Ca)
s-Ca_A (equiv. % pyrite S)	(Ca_A x 0,800) (Ca_A in %Ca)
a-Mg_A (mmol H^+/kg)	(Mg_A x 822,6) (Mg_A in %Mg)
s-Mg_A (equiv. % pyrite S)	(Mg_A x 1,319) (Mg_A in %Mg)
a-C_{IN} (mmol H^+/kg)	(C_{IN} x 1665) (C_{IN} in %C)
s-C_{IN} (equiv. % pyrite S)	(C_{IN} x 2,67) (C_{IN} in %C)
a-ANC_{BT} (mmol H^+/kg)	(ANC_{BT} x 199,8) (ANC_{BT} in %$CaCO_3$ equivalent)
s-ANC_{BT} (equiv. % pyrite S)	(ANC_{BT} x 3,121) (ANC_{BT} in %$CaCO_3$ equivalent)
a-ANC_E (mmol H^+/kg)	(ANC_E x 199,8) (ANC_E in %$CaCO_3$ equivalent)
s-ANC_E (equiv. % pyrite S)	(ANC_E x 3,121) (ANC_E in %$CaCO_3$ equivalent)

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