

BS EN 15875:2011

Incorporating corrigendum August 2012



BSI Standards Publication

Characterization of waste — Static test for determination of acid potential and neutralisation potential of sulfidic waste

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National foreword

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The UK participation in its preparation was entrusted by Technical Committee B/508, Waste Management, to Subcommittee B/508/3, Characterization of waste.

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

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ISBN 978 0 580 80587 5

ICS 13.030.10

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 April 2012.

Amendments/corrigenda issued since publication

Date	Text affected
30 April 2013	Implementation of CEN corrigendum August 2012: Modification to Table 1

English Version

Characterization of waste - Static test for determination of acid potential and neutralisation potential of sulfidic waste

Caractérisation des déchets - Essai statique pour la détermination du potentiel de génération d'acide et du potentiel de neutralisation des déchets sulfurés

Charakterisierung von Abfällen - Statische Prüfung zur Bestimmung des Säurebildungspotenzials und des Neutralisationspotenzials von sulfidhaltigen Abfällen

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Foreword

This document (EN 15875:2011) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2012, and conflicting national standards shall be withdrawn at the latest by April 2012.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/395), which assigned the development of standards on the characterization of waste from extractive industries.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

This document has been developed primarily to support the implementation of the Directive 2006/21/EC of the European Parliament and of the council on the management of waste from the extractive industries, especially relating to technical requirements for waste characterization as sulfide bearing materials may generate sulfuric acid when subjected to weathering.

Test methods for the determination of acid generation behaviour can be divided in static and kinetic tests. A static test is usually relatively fast to perform, but gives only indicative information based on total composition of the waste material. The kinetic test gives more detailed information on behaviour based on reaction rates under specified conditions. This standard only covers static testing.

The application of this test method alone may not be sufficient to determine the actual potential in the field for the formation of acidic drainage as site specific conditions will affect the behaviour in the field and require a more detailed assessment.

To carry out a more precise assessment of the acid generation potential and buffering capacity mineralogical information is required. A number of special cases can be identified: e.g. presence of sulfate (e.g. gypsum), non-acid producing sulfides or carbonates with no buffering capacity. Acid neutralisation behaviour as obtained by other methods can provide additional information in circumstances of uncertainty.

1 Scope

This European standard specifies methods to determine the potential of sulfide bearing materials for the formation of acidic drainage. Specified are methods for determining both the acid potential (AP) and the neutralisation potential (NP) of the material. From these results the net neutralisation potential (NNP) and the neutralisation potential ratio (NPR) are calculated.

This European standard is applicable to all sulfide bearing wastes from the extractive industries excluding wastes which will have pH < 2 in the initial step of the procedure described in 8.2.3.

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.

EN 13137:2001, *Characterization of waste — Determination of total organic carbon (TOC) in waste, sludges and sediments*

EN 14346, *Characterization of waste — Calculation of dry matter by determination of dry residue or water content*

EN 14582, *Characterization of waste — Halogen and sulfur content — Oxygen combustion in closed systems and determination methods*

EN 14899, *Characterization of waste — Sampling of waste materials — Framework for the preparation and application of a Sampling Plan*

EN 15002, *Characterization of waste — Preparation of test portions from the laboratory sample*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 15178, *Soil quality — Determination of total sulfur by dry combustion*

ISO 16720, *Soil quality — Pretreatment of samples by freeze-drying for subsequent analysis*

3 Terms and definitions

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

3.1

laboratory sample

sample sent to or received by the laboratory

3.2

test sample

sample, prepared from the laboratory sample, from which test portions are removed for testing or analysis

3.3

test portion

quantity of material of proper size, for measurement of the concentration or other properties of interest, taken from the test sample

NOTE The test portion may be taken from the laboratory sample directly if no preparation of sample is required (e.g. samples of proper homogeneity, size and fineness).

3.4

acid potential

maximum potential acid generation from a sample assuming that all sulfur occurs as pyrite and that acidity will result from its complete oxidation

3.5

neutralisation potential

capacity of a sample to neutralise the generated acidity

3.6

carbonate rating

carbonate content of the sample used to specify the volume(s) of acid to be added during the procedure

NOTE In this European standard the carbonate rating to specify the volume(s) of acid to be added during the procedure is described in 8.2.3.

3.7

net neutralisation potential

difference between neutralisation potential and acid potential

3.8

neutralisation potential ratio

ratio of neutralisation potential and acid potential

4 Symbols and abbreviations

AP	acid potential
NP	neutralisation potential
CR	carbonate rating
M_d	dry mass of the test portion
M_w	un-dried mass of the test portion
m_d	mass after drying at 105 °C
m_w	mass before drying
M_s	molecular weight of sulfur
NNP	net neutralisation potential
NPR	neutralisation potential ratio
$t = 0$	time at the start of the test (after 15 min \pm 5 min stirring)
$V_{A/B}$	volume of acid or base added
$V_{A, t=0}$	volume of acid added at $t = 0$
$V_{A, t=22h}$	volume of acid added at $t = 22$ h
w_{dr}	dry residue of the sample

5 Principle

This test method consists of four steps:

- Determination of total sulfur by bomb (EN 14582) or high temperature combustion (ISO 15178) and calculation of acid potential (AP). Instead of total sulfur, sulfides may be determined using techniques described in the informative Annex C.
- Determination of carbonate content by dry combustion (EN 13137:2001, method A) to give the carbonate rating (CR).
- Determination of the neutralisation potential (NP) by hydrochloric acid addition to reach pH = 2 to 2,5 and back titration with sodium hydroxide to reach pH = 8,3 after reaction time of 24 h.
- Calculations of the net neutralisation potential (NNP) and the neutralisation potential ratio (NPR) based on AP and NP.

AP and NP are expressed as H⁺ content in mol/kg. The conversion factor is given for expression as carbonate equivalents (CaCO₃) in kg/t.

6 Reagents and laboratory devices

6.1 Reagents

- 6.1.1 Distilled or demineralised water
- 6.1.2 Hydrochloric acid (analysis grade), $c(\text{HCl}) = 1 \text{ mol/l}$
- 6.1.3 Sodium hydroxide (analysis grade), $c(\text{NaOH}) = 0,1 \text{ mol/l}$

6.2 Laboratory devices

- 6.2.1 Analytical balance, with an accuracy of 0,05 g
- 6.2.2 Bottles or vessels (250 ml) made of inert material such as glass or high density polyethylene (HDPE) or polypropylene (PP) and supplied with a lid of inert material (e.g. PTFE). Rinsing is compulsory. When using magnetic bar in stirring (see 6.2.4) it is crucial to use a test vessel or bottle with flat bottom in order to guarantee good mixing.
- 6.2.3 Size reducing equipment, e.g. a jaw crusher, rotary swing mill, ball mill or similar device.
- 6.2.4 Stirring device or magnetic stirring device with magnetic bar coated with PTFE. The parts in contact with the sample and reagents shall be made of materials not affecting the outcome of the test like glass, PTFE.
- 6.2.5 pH meter with a measurement accuracy of at least $\pm 0,05 \text{ pH units}$.
- 6.2.6 Sample dividers (e.g. rotary splitter or riffle divider)
- 6.2.7 Sieves, conforming to the requirements of ISO 3310-1, with screen size of 0,125 mm.

7 Sampling and sample preparation

7.1 Laboratory sample

Perform sampling in accordance with EN 14899 in order to obtain a representative laboratory sample.

The laboratory sample shall have a mass of at least 1 kg (dry mass).

NOTE The mass of the laboratory sample is dependent on its maximum particle size and homogeneity. Further information on sample masses can be obtained from EN 15002.

7.2 Test sample

The test sample shall have a particle size of 95 % less than 0,125 mm. For material with larger particle sizes the following shall apply: Crush the laboratory sample to < 2 mm following the procedures given in EN 15002. Take a subsample from the crushed material by using a suitable divider (6.2.6) or by coning and quartering. The subsample of approximately 100 g is then milled to a particle size of 95 % less than 0,125 mm.

Moist material that is not possible to sieve is dried prior to sieving and/or crushing. The drying temperature shall not exceed 40 °C in order to avoid unwanted reactions. Alternatively, freeze drying according to ISO 16720 can be used.

The crushed material can change upon storage due to ageing of fresh surfaces. It is therefore recommended to test the material as soon as possible after crushing. If short-term storage is needed, crushed material should be stored cold and dark. For long-term storage material should be dried (at temperatures not exceeding 40 °C) prior to storage to prevent acid generating reactions.

7.3 Determination of dry residue of the sample

The whole test sample, complying with the size criteria in 7.2 shall not be dried any further. The dry residue (w_{dr}) of the test sample shall be determined on a separate test portion according to EN 14346.

The dry residue of the sample shall be determined at 105 °C ± 5 °C according to EN 14346. The dry residue expressed as mass fraction in percent is calculated according to Equation (1):

$$w_{dr} = 100 \times \frac{m_d}{m_w} \quad (1)$$

where

w_{dr} is the dry residue of the sample expressed as mass fraction in percent;

m_d is the mass after drying expressed in grams (g);

m_w is the mass of the sample after sample pretreatment as described in 7.2 and before drying expressed in grams (g).

7.4 Test portion for the determination of neutralisation potential

Prepare a representative test portion in accordance with EN 15002.

Calculate the mass of the test portion M_w in grams to be used for the test in accordance with Equation (2):

$$M_w = \frac{M_d}{w_{dr}} \times 100 \quad (2)$$

where

M_d is the dry mass of the test portion expressed in grams (g);

M_w is the total mass of the test portion expressed in grams (g).

8 Test procedures

8.1 Determination of acid potential

8.1.1 General

The acid potential (AP) can either be based on the determination of total sulfur or on a species analysis where sulfide sulfur is determined.

Analysis of total sulfur is a well established method with a high potential of automatization resulting in short analysis times. Total sulfur determination can therefore be regarded as a default method which is described in 8.1.2.

If either through mineralogical or chemical analysis it is shown that substantial parts of total sulfur is sulfate, then a more appropriate (realistic) AP is obtained by analyzing sulfur species (see 8.1.3).

NOTE The main source for acidity is the oxidation of sulfides. Total sulfur is used as a conservative approximation of sulfide content. If a significant part of total sulfur is sulfate this will lead to a significant overestimation of AP, in which case a more realistic assessment will be achieved if the AP determination is based on sulfide content.

8.1.2 Total sulfur content

Total sulfur content is determined either by bomb combustion according EN 14582 or by high temperature combustion according ISO 15178.

The bomb combustion method described in EN 14582 usually gives high recoveries when inorganic salts are analysed. However, depending on the material to be analysed, suitable pure inorganic compounds with similar sulfur binding shall be used to test recovery rates.

When using high temperature combustion techniques, appropriate operating conditions of the analysis shall be chosen, to ensure the detection of all sulphur components, including temperature stable inorganic sulphates (e.g. calcium sulfate). This can be achieved by increasing the temperature (e.g. up to 1 500 °C) or the use of tin capsules (which burn in an exothermic reaction resulting in high temperatures). Also measuring time may be increased as these stable compounds react slower. Calibration has to be made with calibrants having the same range of sulfur concentration as the material to be analysed.

8.1.3 Determination of sulfur species

The main purpose of all species analyses is the determination of sulfides, mainly pyrite. This can be done either by direct determination of pyrite or by subtraction of the sulfate sulfur fraction from the total sulfur content (assuming no other sulfur species like elemental sulfur is present). The choice of direct or indirect approach depends on the mineralogical composition of the sample.

There are no CEN or ISO standards for the determination of sulfur species (sulfate-, sulfide-, disulfide-, sulfur) in waste described in this standard. However, guidance is given in the informative Annex C.

NOTE Some sulfides do not contribute to AP, this can only be assessed through more detailed characterisation, such as mineralogy.

8.1.4 Calculation

The acid potential (AP) is calculated based on the sulfur content (either total or sulfide sulfur) as follows:

NOTE 1 Calculation of acid potential assumes all sulfur to appear as pyrite

a) expressed as H⁺ content in mol/kg (Equation (3))

$$AP = 0,625 \times w_s \quad (3)$$

where

0,625 is the conversion factor (taking into account the conversion of units and that 1 mol of sulfur in pyrite creates 2 moles of H^+) (for further explanation see Annex D);

w_s is the sulfur (either total or sulfide sulfur) content as mass fraction in percent.

b) expressed as carbonate equivalents ($CaCO_3$) in kg/t (Equation (4))

$$AP = 31,25 \times w_s \quad (4)$$

where

31,25 is the conversion factor (ratio of molecular masses of calcium carbonate (100 g/mol) and sulfur (32 g/mol)) (for further explanation see Annex D);

w_s is the sulfur (either total or sulfide sulfur) content as mass fraction in percent.

NOTE 2 AP and NP values expressed as H^+ content in mol/kg can be converted to carbonate equivalents ($CaCO_3$) in kg/t by multiplying by 50. Likewise a AP or NP expressed in carbonate equivalents ($CaCO_3$) in kg/t can be converted to H^+ content in mol/kg by multiplying by 0,02.

8.2 Determination of neutralisation potential

8.2.1 General

The amount of acid added for the test is crucial for the results obtained. Therefore, the carbonate content is determined first to give the carbonate rating. From this, the required hydrochloric acid addition is taken from Table 1.

8.2.2 Carbonate rating

Determine the carbonate content following the procedure in EN 13137:2001, method A.

The carbonate carbon content obtained from EN 13137:2001 (there named "inorganic carbon") is expressed as mass fraction in percent, it is used as carbonate rating in 8.2.3 (for further explanation see Annex D).

8.2.3 Neutralisation potential

Weigh test portion M_w corresponding to $2,00 \text{ g} \pm 0,10 \text{ g}$ of dry mass (M_d) into a test vessel or bottle (6.2.2). Record the exact weight of the sample.

Add $90 \text{ ml} \pm 5 \text{ ml}$ of demineralised water (6.1.1). Start mixing using an appropriate stirring device (6.2.4). Maintain the temperature at $20 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$ during the whole test.

NOTE 1 Some materials may form gas when in contact with water in acidic conditions. Therefore, it is highly recommended to allow possible gas exit by making a small hole in to the lid of the vessel or bottle.

Test portion and water are stirred for $15 \text{ min} \pm 5 \text{ min}$ and the pH of the slurry is measured and recorded before the acid addition. If $\text{pH} < 2$ already, this sample cannot be characterised with this test.

After $15 \text{ min} \pm 5 \text{ min}$ of stirring hydrochloric acid (6.1.2) is added based on the carbonate rating (8.2.2) of the test sample (see Table 1). This is considered the starting point of the test ($t = 0$). Stir the slurry throughout the test.

Table 1 — Addition of acid based on carbonate rating

Line number	Carbonate rating expressed as CO ₃ -C mass fraction %	Volume c(HCl) = 1 mol/l (6.1.2) to be added in ml
		at $t = 0$ ($V_{A, t=0}$)
1	0 - 0,3	0,5
2	0,3 - 0,6	1,5
3	0,6 - 0,9	2,5
4	0,9 - 1,2	3,5
5	1,2 - 1,8	5
6	1,8 - 2,4	7
7	2,4 - 3,6	10
8	3,6 - 4,8	14
9	4,8 - 6,0	18
10	> 6,0	20

Measure and record the pH of the slurry after 22 h \pm 15 min. If the pH is above 2,5 add HCl (6.1.2) to adjust the pH between 2,0 and 2,5. It is strongly recommended to adjust the pH as close as possible to 2,0 to ensure being in the range of pH = 2,0 to 2,5 at $t = 24$ h. Record the exact volume added ($V_{A, t=22h}$) and the pH after the acid addition.

If the pH at $t = 22$ h before acid addition is below pH = 2,0, too much acid was added in the beginning of the test and the test shall be repeated adding a smaller volume of acid. For the repeated test use the acid addition at start ($t = 0$) in table 1 one line above the one used in the discarded test.

EXAMPLE 1 In the test the added HCl was $V_{A, t=0} = 1,5$ ml which corresponds to line 2 in table 1. At $t = 22$ h the measured pH was pH < 2,0 and the test was discarded. A new test is started with acid addition one line above compared to the discarded one which is line 1. Therefore the added HCl is $V_{A, t=0} = 0,5$ ml.

If $V_{A, t=22h}$ exceeds 50 % of total volume of acid ($V_{A, t=0} + V_{A, t=22h}$), terminate the test at $t = 22$ h and start again as follows: Based on the total volume of acid added, choose a new volume for acid addition at $t = 0$ from table 1 so that $V_{A, t=0}$ in the new test corresponds to the highest volume in the table that does not exceed the total volume of acid used in the discarded test (this is usually the volume of HCl addition one or two lines below in Table 1 of the discarded test).

EXAMPLE 2 $V_{A, t=0} = 1,5$ ml is used corresponding to line 2 in Table 1. 3 ml of acid is needed at $t = 22$ h to reach pH 2,0. $V_{A, t=0} + V_{A, t=22h} = 4,5$ ml and the addition at $t = 22$ h is 66, 7% of the total addition. Therefore the test is terminated at $t = 22$ h. Based on total consumption of 4,5 ml acid addition for the next test is $V_{A, t=0} = 3,5$ ml corresponding to line 4 in Table 1.

If the volume of acid added at $t = 22$ h ($V_{A, t=22h}$) does not exceed 50 % of total volume of acid, proceed further in testing as instructed in the next paragraph.

After 24 h \pm 15 min, terminate the test and add demineralised water to the bottle to bring volume to approximately 125 ml. Measure and record the pH, making sure it is in the required range between pH = 2,0 and pH = 2,5. If the pH is outside the required pH range repeat the test applying a reduced (if pH < 2,0) or increased volume of acid (if pH > 2,5).

If neither a smaller nor a bigger volume of initial acid addition from Table 1 leads to completion of the test due to problems described in examples above it is allowed to use an initial volume of acid deviating from Table 1. This deviation should be documented in the test report together with information of the acid volumes used in the preceding trials.

EXAMPLE 3 $V_{A, t=0} = 10$ ml is used corresponding to line 7 in Table 1. At $t = 22$ h pH is adjusted to 2,0. Still, pH of the slurry is above 2,5 at $t = 24$ h. In the next trial $V_{A, t=0} = 14$ ml is used corresponding to line 8 in table 1 leading to pH of the slurry being below 2,0 at $t = 22$ h. For the following trial e.g. $V_{A, t=0} = 12$ ml may be tried.

NOTE 2 Materials usually contain other neutralizing compounds than calcium carbonate, which may react slower than calcium carbonate and thus leads to underestimation of NP. (See Annex B). Titrate the slurry to a pH of 8,3 using a sodium hydroxide solution (6.1.3). Record the exact volume of NaOH consumed in titration.

NOTE 3 The end point of the back-titration is 8,3, being the usual endpoint for acidity titrations, corresponding to the stoichiometric equivalence point for carbonate/bicarbonate in natural waters in which carbonic acid is the most dominant weak acid.

NOTE 4 An example of a data sheet for the recording of test results is given in Annex A, Table A.1.

8.2.4 Calculation

Calculate the NP of the sample expressed as H⁺ content in mol/kg and as carbonate equivalents (CaCO₃) in kg/t as follows:

a) expressed as H⁺ content in mol/kg (Equation (5))

$$NP = \frac{c(\text{HCl}) \times V_A(\text{HCl}) - c(\text{NaOH}) \times V_B(\text{NaOH})}{M_d} \quad (5)$$

where

$c(\text{HCl})$ is the concentration of HCl in mol/l;

$V_A(\text{HCl})$ is the volume of HCl added ($V_{A, t=0} + V_{A, t=22h}$) in ml;

$c(\text{NaOH})$ is the concentration of NaOH in mol/l;

$V_B(\text{NaOH})$ is the volume of NaOH used in back titration in ml;

M_d is the dry mass of the test portion expressed in grams (g).

b) expressed as carbonate equivalents (CaCO₃) in kg/t (Equation (6))

$$NP = 50 \times \frac{c(\text{HCl}) \times V_A(\text{HCl}) - c(\text{NaOH}) \times V_B(\text{NaOH})}{M_d} \quad (6)$$

9 Calculation of neutralisation potential ratio and net neutralisation potential

The acid potential (AP) and neutralisation potential (NP) are to be calculated in H⁺ content in mol/kg. The resulting neutralisation potential ratio (NPR) (potential for neutralisation of acidic drainages) is assessed using the formula in Equation (7):

$$NPR = \frac{NP}{AP} \quad (7)$$

NOTE 1 A NPR < 1 means that there is not enough neutralisation capacity to neutralise all potentially released acidity. A significant excess of NP means that there is enough neutralisation capacity to neutralise all potentially released acidity. In theory, a NPR > 1 should be enough to avoid acidic drainage. However, the reaction rates may differ between AP minerals and NP minerals. Different mineralogy and crystalline structure may also lead to different availability. Thus, there is a range of NPR-values which will be interpreted as uncertain. (see Annex B).

NOTE 2 In some countries the acid potential is expressed as pyrite in mol/kg instead of H⁺ in mol/kg, these figures differ by a factor of 4. Consequently, the value for insufficient neutralisation capacity would be NPR < 4.

The net neutralisation potential (NNP) is calculated from AP and NP using the formula in Equation (8):

$$NNP = NP - AP \quad (8)$$

A NNP expressed as H⁺ content in mol/kg can be converted to carbonate equivalents (CaCO₃) in kg/t by multiplying by 50. Like wise a NP expressed as carbonate equivalents (CaCO₃) in kg/t t can be converted to H⁺ content in mol/kg by multiplying by 0,02.

NOTE 2 While NPR is the preferred value in the screening and classification of mineral wastes, the NNP can be used in the interpretation of test results, e.g. it gives an indication of how sensitive the result is to variations in NP and AP.

10 Performance characteristics

Performance characteristics for the determination of neutralisation potential NP according to 8.2.3 have been determined in a European inter-laboratory comparison that was carried out in 2010. They are presented in Table 2.

Table 2 – Performance characteristics for the determination of neutralisation potential NP of sulfidic waste

Material	<i>p</i>	<i>N</i>	<i>O</i>	Mean mol H ⁺ /kg	<i>s_r</i> %	<i>s_R</i> • %
Waste rock from mining of Zn-, Cu-, Au- and Ag-ore	18	36	2 ¹⁾	0,750	1,28	10,5
Coal mining waste	17	34	2 ²⁾	0,653	2,07	16,5
Tailings from enrichment of Zn- and Pb-ore	18	36	2 ²⁾	1,69	1,36	12,9
Tailings from enrichment of Ni-ore	17	34	2 ²⁾	0,870	2,90	40,6 *
Tailings from enrichment of Au-ore	17	34	0	3,65	1,56	10,8

p is the number of participating laboratories
N is the number of observed values
O is the number of rejected laboratories
s_r is the relative repeatability standard deviation
s_R is the relative reproducibility standard deviation
 1) statistical outliers
 2) rejections based on non-compliance with the protocol

* High reproducibility standard deviation was judged to be caused by the presence of serpentine – a silicate mineral - as the main neutralising mineral of this material. Silicate minerals are in general slower to react than carbonates in the conditions of this test method (see informative annex B and especially B.3.2). Therefore, special attention should be paid to proper mixing of the test slurry (see 6.2) in order to improve the reaction of slow reacting minerals.

11 Test report

The following information shall be documented in such way that they are immediately available on request. In the following list, at least, the items marked with an asterisk (*) shall be included in the test report:

- a reference to this European standard *;
- date of receipt of the laboratory sample *;
- date of the test (beginning and end) *;
- a complete identification of the sample *;
- pretreatment (e.g. method of size-reduction, drying, sub-division) and storage conditions;
- pH values of the slurry before acid addition at start, before and after acid addition at *t* = 22 h and at the termination of the test *;
- dry residue of the laboratory sample (mass fraction in %) *;

- h) added acid volumes (ml) and concentrations (mol/l), and the corresponding amounts of H^+ or OH^- (mol/kg);
- i) any deviation from the test method and the reason for this deviation together with all circumstances that have influenced the results *;
- j) analytical results for total-S (optionally sulfide-S) and CO_3 -C-content and calculated AP, NP, NNP and NPR values *

Annex A (informative)

Example of a data sheet for the recording of test results according to 8.2.3

The user of this European standard is allowed to make use of this present form.

Table A.1 — An example of a data sheet for the recording of test results

Parameter	Unit	Sample code
Dry residue of the laboratory sample (w_{dr})	%	
Un-dried mass of the test portion (M_W)	g	
Volume demineralised water added	ml	
$c(\text{HCl})$	mol/l	
pH at $t = 0$ before acid addition		
Volume HCl added at $t = 0$ ($V_{A, t=0}$)	ml	
pH at $t = 22$ h before acid addition		
Volume HCl added at $t = 22$ h ($V_{A, t=22h}$)	ml	
pH at $t = 22$ h after acid addition		
Total HCl volume added (V_A)	ml	
pH at $t = 24$ h after water addition		
$c(\text{NaOH})$	mol/l	
Volume NaOH used in titration (V_B)	ml	

Annex B (informative)

Operation and uses of the test: influence of parameters

B.1 Sulfur determination

Determination of sulfur and sulfur species depends strongly upon the methods chosen. Analysis for total sulfur by combustion and infrared detection is a routine method. However, care has to be taken that a complete combustion is guaranteed and that a calibration has been made in the appropriate range of sulfur content. Especially when sulfur concentrations are high (e.g. mass fractions above 10 %), longer combustion times and/or higher temperatures may be necessary. It is advised to check the apparatus with standards (e.g. ores) of known composition. Concentrations of sulfur species may strongly depend on the method chosen.

B.2 Particle size

Depending on the static test method the waste rock samples are crushed and ground variably into particle size of < 0,24 mm (Sobek [6]), < 0,074 mm (Lawrence and Wang [3]), and < 0,044 mm (Lapakko [8]) . Tailings samples are usually only broken up to reduce aggregation.

Issues that should be considered for particle size are:

- a) A decreasing particle size increases exponentially the surface area of the materials and thus, increases also the potentially reactive surface of the samples;
- b) Vigorous crushing and grinding exposes all the minerals for the acid attack – including those that are naturally armoured, practically inert or non-reactive;
- c) Certain minerals are more susceptible to grinding than others. (Nevertheless, excluding platy minerals such as micas, susceptibility to grinding normally follows the weathering rate of the minerals);
- d) Decrease in particle size facilitates shorter testing times.

Thus, it follows that usage of too fine particle size in ABA determination may result in overestimation of the NP of the sample, since also those minerals that are actually nonreactive influence on the NP due to e.g. broken edges. Jambor [2] has shown that decrease in particle size from < 0,25 mm to < 0,074 mm may increase the NP significantly due to larger amount of fines, even though, in general, the increase is usually smaller (e.g. White et al. [7]). Thus, the amount of fines can be a significant factor in causing NP enhancement (Jambor [2]). It may cause differences in NP results, if grinding is done with different intensities in test laboratories.

B.3 Mineralogy

B.3.1 Sources of acidity

B.3.1.1 General

Two key sources contribute to acid generation from extractive wastes: primary sources (sulfide oxidation) and secondary sources (sulfate dissolution). This European standard assumes all acid is generated from primary sources, although the back-titration used to determine NP neutralises any acidity produced by the sample during the time of the test.

Inorganic sulfur in the natural environment occurs mainly as sulfide and sulfate minerals. The total sulfur content will overestimate the actual AP of samples containing substantial non acid-producing sulfate minerals

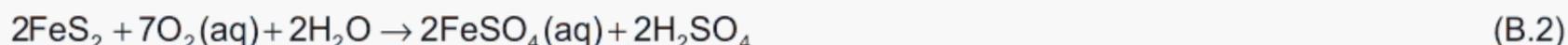
(e.g. barite or gypsum) and/or sulfur from organic matter. Knowledge of sample mineralogy is essential for correct interpretation of data from analysis using this standard.

B.3.1.2 Primary sources of acidity

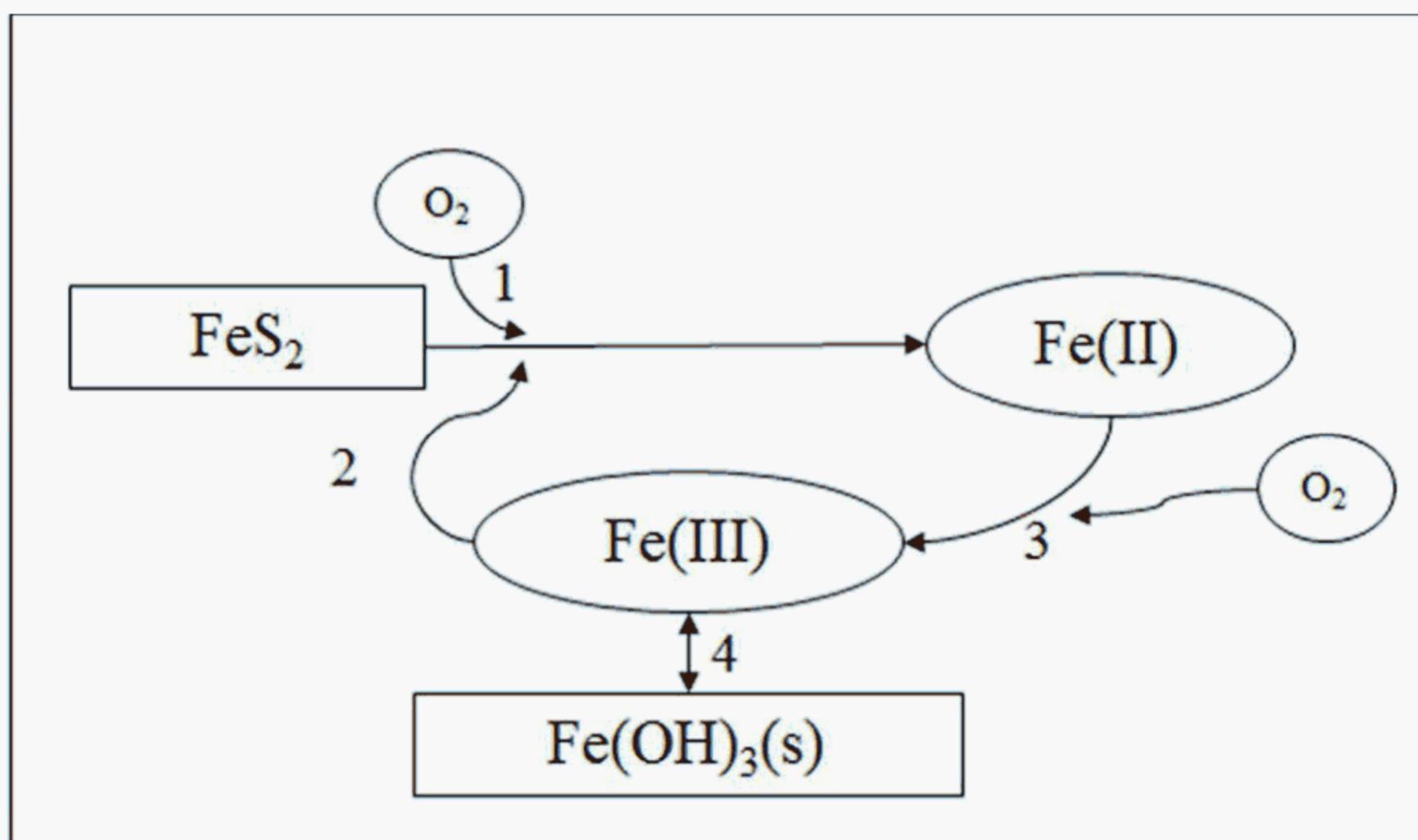
Primary acid-generating minerals are sulfides of the type, MS₂, the most common being pyrite (FeS₂). Oxidation of pyrite is often simplified by the following stoichiometric equations:



or



However, a better representation is given in Figure B.1.



Oxygen path	$\text{FeS}_2 + 7/2\text{O}_2(\text{aq}) + \text{H}_2\text{O} \rightarrow \text{Fe}^{2+} + 2\text{SO}_4^{2-} + 2\text{H}^+$ (1)
Ferric ion path	$\text{FeS}_2 + 14\text{Fe}^{3+} + 8\text{H}_2\text{O} \rightarrow 15\text{Fe}^{2+} + 2\text{SO}_4^{2-} + 16\text{H}^+$ (2)
Oxidation of ferrous ion	$2\text{Fe}^{2+} + 2\text{H}^+ + 0,5\text{O}_2 \rightarrow 2\text{Fe}^{3+} + \text{H}_2\text{O}$ (3)
Hydration of ferric ion	$\text{Fe}^{3+} + 3\text{H}_2\text{O} \leftrightarrow \text{Fe}(\text{OH})_3 + 3\text{H}^+$ (4)

Figure B.1 — Iron redox cycling. Pyrite oxidation by 1) dissolved molecular oxygen and 2) aqueous ferric iron; 3) Oxidation of aqueous ferrous iron by oxygen. 4) Solubility equilibrium between aqueous solution and Fe(OH)₃(s) (Salmon, [5])

In the natural environment, the slow oxidation of pyrite leads to release of Fe(II) as shown by Equation (1) in Figure B.1. Under most conditions in the presence of dissolved molecular oxygen, Fe(III) is the thermodynamically more stable species. However, kinetic limitations mean that oxidation of Fe(II) to Fe(III) is relatively slow (Equation (3) in Figure B.1). Fe(III) is also a powerful oxidant for sulfides and is itself reduced to

Fe(II), leading to iron redox cycling (Equation (2) in Figure B.1). The availability of aqueous Fe(III) for reaction can be limited by relatively fast precipitation of secondary Fe(III) phases, such as amorphous ferric hydroxide (Equation (4) in Figure B.1). Availability of ferrous and ferric iron for slow reactions and equilibrium, such as Equation (2) in Figure B.1, depends on solution pH and the presence of any complexing agents.

When acid mine water, rich in ferric iron, reaches the surface it will fully oxidize, hydrolyze and may precipitate iron hydroxides. This process will also produce acid except at very low pH, where Fe^{3+} is stable.

Sphalerite (ZnS) and galena (PbS) are considered as non-acid or low acid producing sulfides, because they usually contain no iron. However, if iron substitutes for zinc, sphalerite will be an acid generator in a similar way to iron-bearing sulfides. In the case of dissolution, the possible coating of galena by secondary phases with low solubilities may increase the apparent resistance because they protect the sulfides from direct contact with oxidizing agents.

B.3.1.3 Secondary sources of acidity

On weathering, sulfides can release all their acid potential producing a range of hydroxides and oxides such as goethite. Alternatively, they can, in the non-saturated zone release only a portion of their acid potential and store the rest in secondary salts which are stable in oxidizing acidic environments (Bowell et al., [1]). The most common of these sulfate salts are given in Table B.1. Not all necessarily release hydrogen and sulfate on dissolution, but all release sulfate anions. These minerals are highly soluble so they can represent an instantaneous source of acidic sulfate-rich water upon dissolution and hydrolysis, for example the dissolution of jarosite:

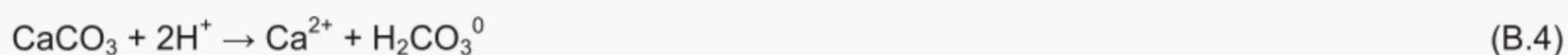


Subsequent oxidation of ferrous iron and hydrolysis of ferric iron at $\text{pH} > 2$ provides an additional source of acidity. These minerals are therefore important as both sinks and potential sources of acidity.

B.3.2 Neutralisation potential

The determination of neutralisation potential (NP) is dependent on a number of parameters. Apart from sample mineralogy, the most important have been identified as being sample pre-treatment, temperature, testing time, particle size, end pH and back-titration pH. Some of them have already been defined in existing international method descriptions (Lawrence and Wang [3]), with levels set where results have been empirically proven to be appropriate. Other important parameters have been defined in this standard to increase the reproducibility and comparability of results.

All NP is assumed to react like calcite in acidic conditions



- Fe and Mn carbonates are not neutralising under aerobic conditions (i.e. siderite, FeCO_3 , and rhodochrosite, MnCO_3);
- Silicates (and some other minerals) will contribute to the neutralisation to some extent (slower reaction than calcite);
- In sulfidic soils (clays) organic matter may contribute significantly to the neutralising capacity.

When using the test described in this European standard it is assumed that all neutralising capacity is determined within the testing time of 24 h. However, some minerals react faster, others slower. To give an indication of reactivity of some carbonate minerals the following sequence may be used:

monohydrocalcite > aragonite > calcite > dolomite > magnesite > siderite > rhodochrosite

In Table B.1, a more detailed list of carbonate minerals is shown.

Table B.1 – Mineralized environment, carbonate minerals.

Mineral group	Mineral	Chemical Formula
Hydrated carbonates	Monohydrocalcite	$\text{CaCO}_3 \cdot \text{H}_2\text{O}$
Aragonite group: Orthorhombic	Aragonite	CaCO_3
Calcite group: Trigonal	Calcite	CaCO_3
	Magnesite	MgCO_3
	Rhodochrosite	MnCO_3
	Siderite	FeCO_3
Dolomite group: Trigonal	Ankerite	$\text{CaFe}(\text{CO}_3)_2$
	Dolomite	$\text{CaMg}(\text{CO}_3)_2$
Carbonates with hydroxyl or halogen	Azurite	$\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$
	Malachite	$\text{Cu}_2\text{CO}_3(\text{OH})_2$

If slowly reacting carbonates or silicates are present in higher concentrations the application of a dynamic test may be advisable to give a more realistic forecast of waste behaviour. CEN/TS 14429, CEN/TS 14497 and CEN-ISO/TS 21268-4 can provide more detailed acid neutralisation behaviour of materials over wider pH ranges and assess the contributions of non-carbonate neutralisation and non-neutralising carbonates. Geochemical modelling of major elements analysed in sulfidic mining waste have also been shown to provide mineral and other phases contributing to ANC over pH ranges from pH 2 to 8.

Annex C (informative)

Speciation of sulfur compounds

For the analysis of sulfur species, no international standards are available. Speciation of sulfur compounds is a difficult task and is dependent on the material to be analyzed. A number of different methods have been developed and are used in the mining industry worldwide.

The main purpose of all species analyses is the determination of sulfides, mainly pyrite. This can be done either by direct determination of pyrite or by difference of total and sulfate sulfur (assuming no other sulfur species like elemental sulfur is present). The choice of direct or indirect approach depends on the mineralogical composition of the sample.

Sulfur species have different thermal stabilities when being combusted. Many sulfides, especially pyrite, need lower combustion temperatures than sulfates. This fact is used for direct determination of pyrite at a temperature of about 800 °C in an automated sulfur analyzer (method code in Figures C.1 and C.2: LTIR). Alternatively, in ASTM E1915 the sulfide sulfur is determined by roasting (pyrolysis) the sample in an oven at a temperature of 550 °C (PYR1) or 650 °C (PYR2). After roasting, the sulfur content of the pyrolysis residue is determined. Sulfide sulfur is calculated by subtracting sulfur in the roasted sample from sulfur in the untreated sample (= total sulfur).

Another speciation method for coal is given in ISO 157 Coal - Determination of forms of sulfur (H₂S). Samples are treated with HCl for sulfides and with HCl plus Cr³⁺ for disulfide (pyrite) to form hydrogen sulfide (H₂S) which is collected and its concentration determined by titration.

Other methods (see ASTM E1915) remove sulfates and determine sulfide sulfur in the leached residue. Sulfate sulfur is extracted with a hot aqueous carbonate solution (CARB); some do an additional extraction with carbon disulfide (CS₂) to remove elemental sulfur (CARBCS).

Sulfate sulfur can also be determined directly by extraction with carbonate solution and gravimetric measurement of BaSO₄ after addition of barium chloride (BaCl₂)(CSUL). Sulfide sulfur is calculated by subtraction of sulfate sulfur from sulfur in the untreated sample (= total sulfur).

Another method is described by Sobek et.al.[6]. Sulfates are extracted with HCl at about room temperature and the sulfate sulfur is calculated by subtraction of sulfur in the treated residue from sulfur in the untreated sample (= total sulfur). Pyritic sulfur is removed by treatment with hot HNO₃; pyritic sulfur is sulfur in the HCl-treated residue minus sulfur in the HNO₃-treated residue. Not extractable sulfur is the sulfur in the HNO₃-treated residue. Often a so called "modified Sobek procedure" is applied using hot instead of cold HCl (NITR).

As part of the robustness study for this European standard 9 waste samples were analyzed for sulfur species by 7 laboratories using different methods (coal or metal mining background) described above. The results can be summarized as follows:

When the main constituent of sulfidic minerals is pyrite, all different methods give similar results and may be used.

When a major part of sulfidic minerals is non-pyritic the results differ much more. Higher thermal stabilities are found for minerals that contain other metals as iron, so for pentlandite (Ni,Fe)₉S₈ and even more for mono-sulfides like galena (PbS) and spalerite (ZnS). Therefore, all methods that use low temperature combustion show systematic lower values. If these minerals are present it could be advisable to use carbonate leaching or a direct method. In addition, the modified Sobek procedure can be applied.

The results of the sulfur speciation study are also shown in the following table and two figures:

Table C.1 — Selected waste samples for the sulfur study

Sample number	Type	Country	Information (operation, minerals etc.)	Active mine	Total sulfur (% mass fraction)
1	Tailings	Hungary	Sulfide minerals (Au, Cu)	No	0,23
2	Tailings	Austria	Tungsten	Yes	0,27
3	Tailings	Germany	Hard coal	Yes	0,56
4	Tailings	Sweden	Zn, Pb	Yes	0,68
5	Tailings	Poland	Hard coal	Yes	1,19
6	Tailings	Hungary	Sulfide minerals	No	1,47
7	Waste rock	Finland	Au (arsenopyrite and pyrite)	Yes	1,53
8	Tailings	Finland	Nickel	Yes	1,71
9	Waste rock	Sweden	Zn, Cu, Au, Ag	Yes	3,22

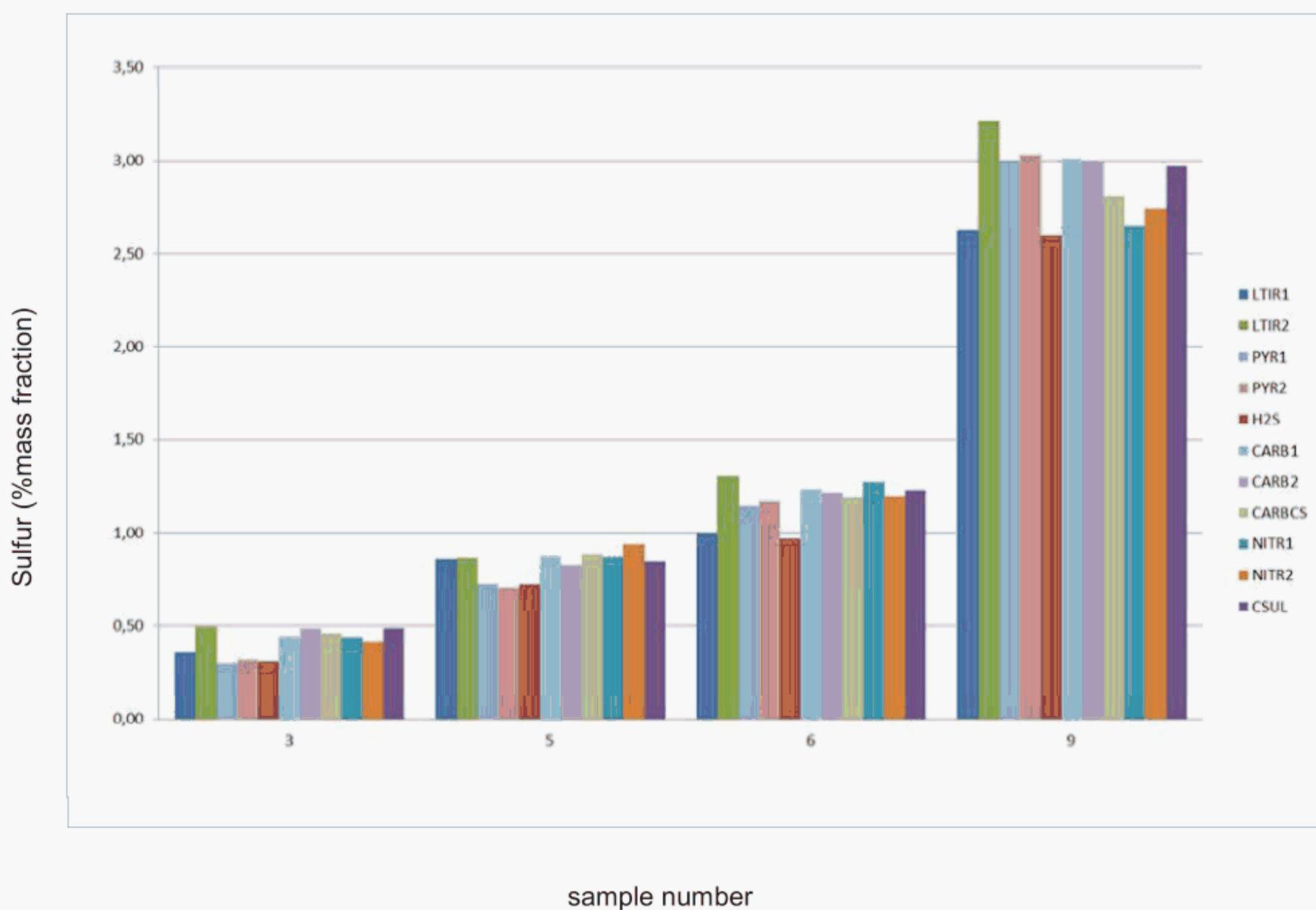


Figure C.1 — Sulfide sulfur (% mass fraction) of 4 pyrite rich waste samples (explanation for abbreviations of methods see text of Annex C)

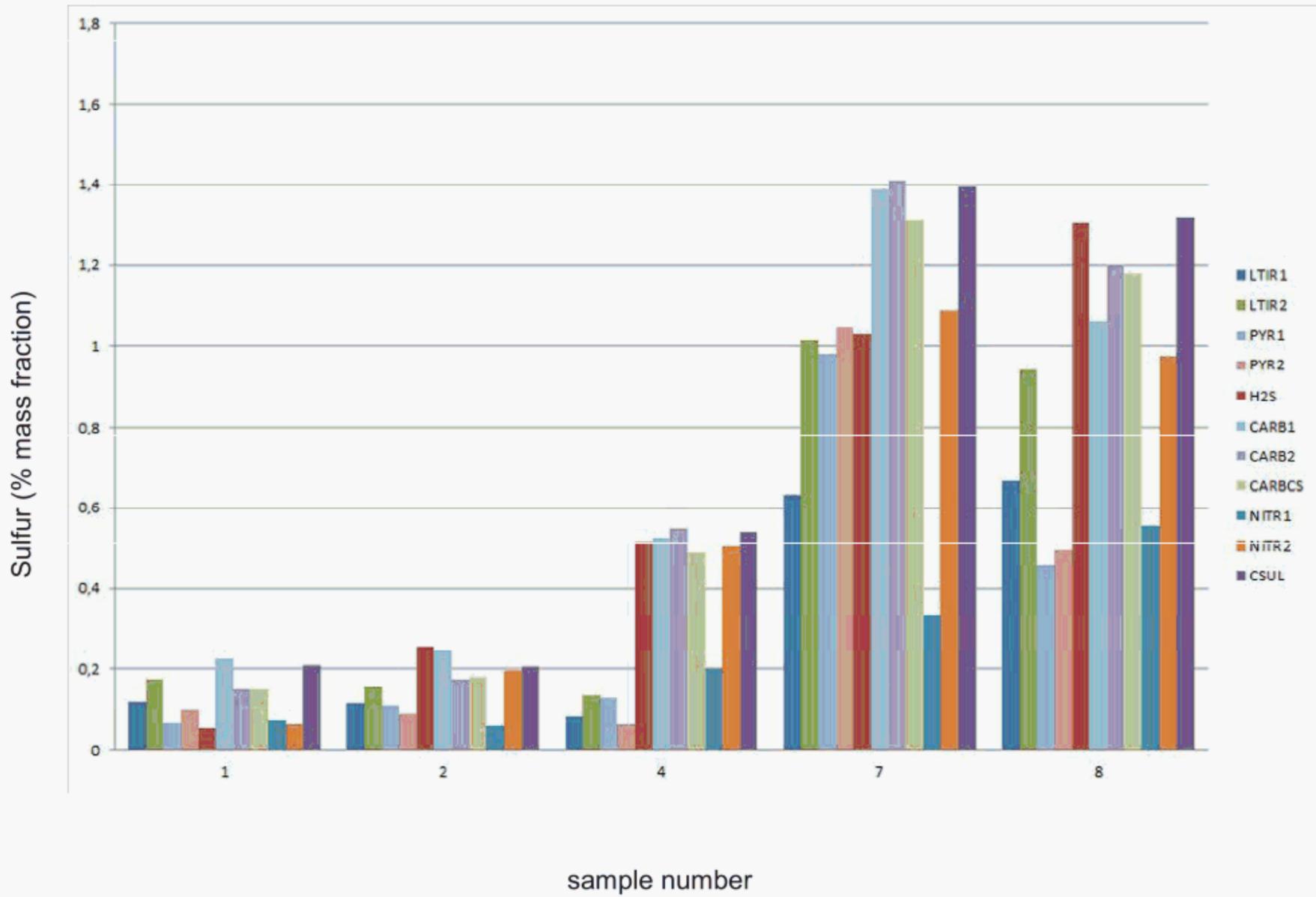


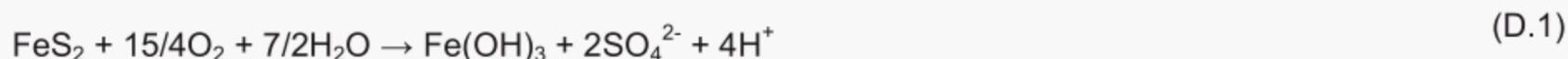
Figure C.2 — Sulfide sulfur (% mass fraction) of 5 waste samples (explanation for abbreviations of methods see text of Annex C)

Annex D (informative)

Explanation of formulas used

D.1 Acid potential

Determination of the acid potential (AP) is based on the following chemical reaction:



According to this one mole of pyrite gives 4 moles of H^+ or – which equivalent – one mole of sulfur give 2 moles of H^+ .

AP expressed as H^+ content in mol/kg is therefore calculated from the sulfur content expressed as mass fraction in % w_s by dividing by the molar mass of sulfur M_s and multiplying with 10 (conversion of mass fraction in % to mol/kg) and again multiplying with 2 (one mole of sulfur gives 2 moles of H^+):

$$\text{AP} = 2 \times 10 \times \frac{w_s}{M_s} \quad (\text{D.2})$$

This can be simplified when $M_s = 32$ g/mol is inserted to be

$$\text{AP} = 0,625 \times w_s$$

AP expressed as carbonate equivalents (CaCO_3) in kg/t is therefore calculated from the sulfur content expressed as mass fraction in % w_s by multiplying with 10 (conversion of mass fraction in % to g/kg) dividing by the molar mass of sulfur M_s and then multiplying with the molar mass of calcium carbonate M_{CaCO_3} .

NOTE No factor of 2 required as one mole of carbonate is equivalent to two moles of H^+ .

$$\text{AP} = 10 \times \frac{w_s}{M_s} \times M_{\text{CaCO}_3} \quad (\text{D.3})$$

This can be simplified when $M_s = 32$ g/mol and $M_{\text{CaCO}_3} = 100$ g/mol are inserted to be

$$\text{AP} = 31,25 \times w_s \quad (\text{D.4})$$

A conversion between the data expressed as H^+ content in mol/kg and data expressed as carbonate equivalents (CaCO_3) in kg/t is possible. It is done by using the conversion factor 50 ($=M_{\text{CaCO}_3}/2$) from H^+ content to carbonate equivalents and the conversion factor of 0,02 ($=2/M_{\text{CaCO}_3}$) from carbonate equivalents to H^+ content.

D.2 Carbonate rating

Carbonate rating (CR) is used for the calculation of acid addition only (see Table 1 in 8.2.3).

However, to show the calculations used to create table 1 some information is given. Therefore, carbonate rating can be converted to be expressed as H^+ content in mol/kg. CR expressed as H^+ content in mol/kg is calculated from the inorganic carbon content $w_{(\text{CO}_3^{2-}\text{-C})}$ expressed as mass fraction in % w_c by dividing by the molar mass of carbon M_c and multiplying with 10 (conversion of mass fraction to mol/kg) and again multiplying with 2 (one mole of carbonate is equivalent to 2 moles of H^+):

$$CR = 2 \times 10 \times \frac{w(\text{CO}_3^{2-} - \text{C})}{M_c} \quad (\text{D.5})$$

This can be simplified when $M_c = 12 \text{ g/mol}$ is inserted to be

$$CR = 1,666 \times w(\text{CO}_3^{2-} - \text{C}) \quad (\text{D.6})$$

EXAMPLE If $w(\text{CO}_3^{2-} - \text{C}) = 1,0$ (mass fraction expressed in %) $CR = 1,666 \cdot 1,0 = 1,666$ (H^+ content in mol/kg). As 2 g sample is used in the test this leads to an absolute H^+ content of 0,00333 mol (multiply with 2 g and divide by 1 000 g) or 3,33 mmol. Therefore a minimum amount of hydrochloric acid of 3,33 mmol has to be added. To insure that the pH is in the desired range of $\text{pH} = 2,0$ to 2,5, slightly more acid is added. In table 1 of 8.3.2 (line 4) initial acid addition of 3,5 ml is given. This is equivalent to 3,5 mmol which is above the theoretical value of 3,33 mmol from the carbonate rating. All other lines are calculated in the same way.

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