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

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

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

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potential of sulfidic waste**

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détermination du potentiel de génération d'acide et du
potentiel de neutralisation des déchets sulfurés

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

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Foreword

This document (prEN 15875:2008) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

This document is currently submitted to the CEN Enquiry.

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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 $\text{pH} < 2$ 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 *Characterization of waste – Determination of total organic carbon (TOC) in waste, sludges and sediment*

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

EN 14582 *Characterization of waste – Determination of halogen and sulfur content; oxygen combustion in closed systems and determination methods Method A: bomb combustion*

EN 14899 *Characterization of waste — Sampling of liquid and granular waste materials including paste-like materials and sludges- Part 1: A framework for sampling plan preparation*

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

ISO 351 *Solid mineral fuels - Determination of total sulphur - High temperature combustion method*

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

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

3 Terms and definitions

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**suspension**

mixture of leachant and test portion

3.5**Acid potential (AP)**

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

3.6**Neutralisation potential (NP)**

The capacity of a sample to neutralise the generated acidity

3.7**Carbonate rating (CR)**

Carbonate content of the sample used to specify the volume(s) of acid be added during the procedure (8.2.3)

3.8**Net neutralisation potential (NNP)**

Difference of acid potential and neutralization potential

$$\text{NNP} = \text{NP} - \text{AP}$$

3.9**Neutralisation potential ratio (NPR)**

Ratio of neutralization potential and acid potential

$$\text{NPR} = \text{NP} / \text{AP}$$

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 (check later use of this)

M_s molecular weight of Sulfur

NNP net neutralisation potential

NPR neutralisation potential ratio

t=0 time at the start of the test

t=2h time at two hours from the start of the test

V_{A/B} volume of acid or base added

V_{A, t=0} volume of acid added at *t=0*

V_{A, t=2h} volume of acid added at *t=2h*

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$V_{A, t=22h}$ volume of acid added at $t=22h$

w_{dr} dry residue of the sample

5 Principle

This test method consists of four parts:

- a) Determination of total sulfur by bomb (EN 14582) or high temperature combustion (ISO 351 or ISO 15178) and calculation of acid potential (AP). Instead of total sulfur, sulfides may be determined using techniques described in the informative Annex C.
- b) Determination of carbonate content by dry combustion (ISO 10694 or EN 13137, part A) to give the carbonate rating (CR).
- c) Determination of the neutralisation potential (NP) by hydrochloric acid addition to reach pH=2 to pH=2,5 and back titration with sodium hydroxide to reach pH=8,3 after reaction time of 24 hours.
- d) 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 ($CaCO_3$) 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), 1 mol/l

6.1.3. Sodium hydroxide (analysis grade), 0,1 mol/l

6.2 Laboratory devices

6.2.1 Analytical balance (accurate to 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.

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 suspension 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 $\pm 0,05$ pH units.

6.2.6 Sample dividers (e.g. rotary splitter or riffle divider) or coning and quartering.

6.2.7 Sieves, conforming to the requirements of ISO 3310-1, with openings 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

Crush the laboratory sample to < 2 mm following the procedures given in EN 15002. Take a subsample from the crushed material by e.g. using a suitable divider (6.2.7). 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 to take place.

7.3 Determination of dry residue of the sample

The whole test sample, complying with the size criteria in 7.2 shall not be further dried. 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 as follows:

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

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 (NP)

Prepare a test portion by the use of a suitable divider (6.2.7) 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:

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

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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 Part A: Determination of acid potential (AP)

8.1.1 General

The acid potential (AP) can be either 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 sulfide sulfur directly or by subtraction of sulfate sulfur from total sulfur. There are no CEN or ISO standards for this species analysis (see 8.1.3), methods that are applicable are shown in the (informative) annex C.

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 351 or 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 a similar sulfur binding shall be used to test recovery rates.

When using high temperature combustion techniques operating conditions of the analysis have to be chosen that all sulfur components including temperature stable inorganic sulfates (e.g. calcium sulfate) are detected. This can be achieved by increasing the temperature (e.g. up to 1500 °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 are reacting slower. Calibration has to be made with calibrants having the same range of sulfur concentration as the material to be analysed.

8.1.3 Disulfide (pyrite) content

Methods for the determination of sulfur species (sulfate-, sulfide-, disulfide- sulfur) are not established for the material described in this standard. 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 Calculation of acid potential assumes all sulfur to appear as pyrite

a) expressed as H^+ content in mol/kg as follows:

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

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

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

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 AP and NP values expressed as H^+ content in mol/kg can be converted to carbonate equivalents ($CaCO_3$) in kg/t by multiplying with 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 with 0.02.

8.2 Part B: Determination of Neutralisation Potential (NP)

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 (CR)

Determine the carbonate content following the procedure in EN 13137 part A (clause 10.3.3. of this standard) or ISO 10694.

The carbonate carbon content obtained from EN 13137 (there named "inorganic carbon") or ISO 10694 is expressed as mass fraction in percent, it is used as Carbonate Rating in 8.2.3 (for further explanation see Annex D).

NOTE Calculation of CO_3 -NP assumes all carbonates to be neutralising. However, in reality some carbonates like siderite or manganese carbonate do not contribute.

8.2.3 Neutralisation potential (NP)

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.

The suspension is stirred for 15 ± 5 minutes and the pH of the solution is measured and recorded before the acid addition. If $\text{pH} < 2$ already, this sample cannot be characterised with this test.