SLOVENSKI STANDARD
SIST EN 15875:2012

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Karakterizacija odpadkov - Statični preskus za določevanje kislinske in nevtralizacijske kapacitete odpadkov, ki vsebujejo sulfid

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

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

Ta slovenski standard je istoveten z: **EN 15875:2011**

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13.030.10	Trdni odpadki	Solid wastes

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English Version

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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of an identical text or by endorsement, at the latest by April 2012, and conflicting national standards shall be withdrawn at the latest by April 2012.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
$\mathrm{M_s}$	molecular weight of sulfur
NNP	net neutralisation potential
NPR	neutralisation potential ratio
$t = 0$	time at the start of the test (after 15 min ± 5 min stirring)
$V_{\mathrm{A/B}}$	volume of acid or base added
$V_{\mathrm{A,\ t=0}}$	volume of acid added at $t = 0$
$V_{\mathrm{A,\ t=22h}}$	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 ($CaCO_3$) 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), $c(\text{HCl})$ = 1 mol/l

6.1.3 Sodium hydroxide (analysis grade), $c(\text{NaOH})$ = 0,1 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 ± 0,05 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):

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

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):

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

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 automatisation 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))

$$\mathrm{AP} = 0{,}625 \times w_\mathrm{S} \qquad (3)$$