# SLOVENSKI STANDARD
# SIST EN 16660:2015

**01-julij-2015**

---

**Karakterizacija odpadkov - Izluževalni preskus - Ugotavljanje redukcijske sposobnosti in zmogljivosti**

Characterisation of waste - Leaching behaviour test - Determination of the reducing character and the reducing capacity

Charakterisierung von Abfällen - Auslaugungsverhalten - Bestimmung der Reduktionseigenschaft und der Reduktionsfähigkeit



Caractérisation des déchets - Essais de comportement à la lixiviation - Détermination des propriétés réductrices et de la capacité de réduction



**Ta slovenski standard je istoveten z:** **CEN/TS 16660:2015**

---

**ICS:**

| | | |
|---|---|---|
| 13.030.01 | Odpadki na splošno | Wastes in general |

**SIST EN 16660:2015** **en,fr,de**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

# CEN/TS 16660

May 2015

ICS 13.030.01

English Version

# Characterization of waste - Leaching behaviour test - Determination of the reducing character and the reducing capacity

Caractérisation des déchets - Essais de comportement à la lixiviation - Détermination des propriétés réductrices et de la capacité de réduction

Charakterisierung von Abfällen - Untersuchung des Elutionsverhaltens - Bestimmung der Reduktionseigenschaft und der Reduktionsfähigkeit

This Technical Specification (CEN/TS) was approved by CEN on 21 March 2015 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.



CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

**CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels**



Ref. No. CEN/TS 16660:2015 E

# Contents

Page

# Foreword

This document (CEN/TS 16660:2015) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

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.

This document has been developed primarily to support the requirements for leaching behaviour testing within EU and EFTA countries.

This document was elaborated on the basis of NEN 7348:2006.

To determine the various aspects of the leaching behaviour (the leaching characteristics) of solid earthy and stony building and waste materials a series of steps should be followed, in particular sampling, sample pretreatment, characterization tests, digestion and chemical analysis of the solid substance or the eluates. An umbrella standard (EN 16457) is developed that gives general instructions. In here the relationship is given between all the standards in each step, each with a specific scope. To determine the leaching characteristics, the general instructions or the specific standards to which reference is made shall be followed with good consistency.

This Technical Specification describes a test that can be used to determine whether or not the material to be tested possesses reducing properties. If this is the case, a next test is used to quantify the reducing capacity of this material or its eluates. Based on the results of this Technical Specification, it can be established whether leaching under practical conditions can differ (considerably) from leaching under standard aerobic laboratory conditions and whether there is justification for testing leaching under low-oxygen conditions (see Annex A, [16]).



The standards that characterize the various aspects of the leaching behaviour are produced and published in phases. This means that upon the publication of this Technical Specification, reference is not yet made in all relevant standards. For the missing aspects, users of this Technical Specification will have to make their own choice of the methods to be used. Annex A gives information on the validation and materials used. Annex B gives a further explanatory note on the reducing capacity. In addition to specifications provided in EN 15002, Annex C gives further guidance on sampling, sample pretreatment and sample storage. For more information, the standards and other publications included in the bibliography that have been published in this respect can be used.

The numbered clauses are normative with the exception of the passages marked with the heading 'NOTE'; the annexes are informative.

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

# Introduction

This document specifies a set of three tests to assess if a material has reducing properties and subsequently to determine the reducing capacity of that material and the reducing capacity of an eluate produced at low liquid to solid ratio. For a proper performance special attention is given to minimize contact with the atmosphere before and during testing.

For the complete characterization of the leaching behaviour of waste under specified conditions the application of other test methods is required (see EN 12920).

Anyone dealing with waste and sludge analysis should be aware of the typical risks of that kind of material irrespective of the parameter to be determined. Waste and sludge samples can contain hazardous (e.g. toxic, reactive, flammable, infectious) substances, which can be liable to biological and/or chemical reaction.

Consequently these samples should be handled with special care. Gases which can be produced by microbiological or chemical activity are potentially flammable and will pressurize sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations should be followed with respect to all hazards associated with this method.

In the different European countries, tests have been developed to characterize and assess the constituents which can be leached from waste materials. The release of soluble constituents upon contact with water is regarded as one of the main mechanism of release which results in a potential risk to the environment during life-cycle of waste materials (disposal or re-use scenario). The intent of these tests is to identify the leaching properties of waste materials. The complexity of the leaching process makes simplifications necessary. Not all of the relevant aspects of leaching behaviour can be addressed in one single standard. This Technical Specification addresses reducing properties of materials and the consequences to the test or test conditions to be applied in performing leaching tests.



Procedures to characterize the behaviour of waste materials can generally be divided into three steps, using different tests in relation to the objective. The following test hierarchy is taken from the Landfill Directive [1)] and the Decision on Annex II of this Directive [2)] for disposal of waste.

a) Basic characterization constitutes a full characterization of the waste by gathering all the necessary information for a safe management of the waste in the short and long term. Basic characterization may provide information on the waste (type and origin, composition, consistency, leachability, etc.), information for understanding the behaviour of waste in the considered management scenario, comparison of waste properties against limit values, and detection of key variables (critical parameters as liquid/solid (L/S) ratios, leachant composition, factors controlling leachability such as pH, redox potential, complexing capacity and physical parameters) for compliance testing and options for simplification of compliance testing. Characterization may deliver ratios between test results from basic characterization and results from simplified test procedures as well as information on a suitable frequency for compliance testing. In addition to the leaching behaviour, the composition of the waste should be known or determined by testing. The tests used for basic characterization should always include those to be used for compliance testing.

b) Compliance testing is used to demonstrate that the sample of today fits the population of samples tested before by basic characterization and through that, is used to carry out compliance with regulatory limit values. The compliance test should therefore always be part of the basic characterization program. The compliance test focuses on key variables and leaching behaviour identified by basic characterization tests. Parts of basic characterization tests can also be used for compliance purposes.

1) Council Directive 1999/31/EC of 26 April 1999 on the landfill of waste.

2) Council Decision 2003/33/EC of 19 December 2002.

c) On-site verification tests are used as a rapid check to confirm that the waste is the same as that which has been subjected to characterization or compliance tests. On-site verification tests are not necessarily leaching tests.

The test procedure described in this document is a basic characterization test and falls in category a).

# 1 Scope

This Technical Specification specifies three laboratory tests to determine the reducing character and the reducing capacity of construction products, waste materials and the eluate resulting from exposure of these solids to a leachant. Reducing species released from the product are titrated to quantify the reducing capacity.

For a specification of the materials with which experience has been acquired with the execution of the tests according to this Technical Specification see Annex A and [16].

NOTE Materials with reducing properties can in practice under both oxidizing and anoxic (isolated) conditions show completely different leaching behaviour than obtained with the leaching tests specified in EN 16457. This may seriously hamper the interpretation of the leaching tests, if this condition is not taken into consideration.

# 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.

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

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

EN ISO 10523, *Water quality - Determination of pH (ISO 10523)*



# 3 Terms and definitions



For the purposes of this document, the following terms and definitions apply. Additional terms and definitions are given in EN 13965-2.

**3.1**
**eluate**
solution obtained by a leaching test

**3.2**
**laboratory sample**
sample or sub-sample(s) sent to or received by the laboratory

[SOURCE: IUPAC, 1990]

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, cutting, sawing, coring, or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test or for analysis. The laboratory sample is the final sample from the point of view of sampling but it is the initial sample from the point of view of the laboratory.

Note 2 to entry: Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

**3.3**
**leaching test**
test during which a material is put into contact with a leachant and some constituents of the material are extracted

[SOURCE: EN 12457-1:2002]

**3.4**
**liquid to solid-ratio**
***L*/*S***
ratio between the amount of liquid (*L*) and of solid (*S*) in the test

Note 1 to entry: *L*/*S* is expressed in l/kg dry matter.

**3.5**
**reducing capacity**
potential for a material to impose reducing conditions

Note 1 to entry: Reducing capacity is expressed in mmol $O_2$/kg.

**3.6**
**redox potential**
value that indicates reducing or oxidized state of a material

Note 1 to entry: Redox potential is expressed in mV.



**3.7**
**sample**
portion of material selected from a larger quantity of material



**3.8**
**test portion**
amount or volume of the test sample taken for analysis, usually of known weight or volume

[SOURCE: IUPAC, 1990]

**3.9**
**test sample**
sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis

[SOURCE: IUPAC, 1990]

## 4 Principle

The purpose of the tests described in this Technical Specification is to:

— establish whether or not a material has reducing properties;

— establish where necessary the reducing capacity of this material or its eluates in quantitative terms.

The reducing character of a material is determined by bringing the material into contact with demineralized water at a low L/S value in an airtight sealed vessel (24 h contact time) and then measuring the redox potential with respect to the redox potential in water with the same pH.

The reducing capacity of a material or its eluates is determined by carrying out a redox titration. In the titration an excess Ce(IV) is used to oxidize the reducing components. Then by back titration the oxidant demand is determined.

NOTE Sulphide is often the major reducing component.

## 5 Preparation of the test portion

The test consists of three parts which in principle can be carried out separately. For the single execution of the test to determine the reducing character according to this Technical Specification (8.2) a test portion $A_1$ of (50 ± 5) g dry matter is necessary, of which the dry matter content $w_{dm}$ is known and of which at least a mass percentage of 95 % (dry matter) of the particles is smaller than 4 mm.

For the single execution of the test to determine the reducing capacity of the solid material according to this Technical Specification (8.3) a test portion $A_2$ of (2 ± 0,002) g dry matter is necessary, of which the dry matter content $w_{dm}$ is known and of which at least a mass percentage of 95 % (dry matter) of the particles is smaller than 125 µm.

For the single execution of the test to determine the reducing capacity of the eluates according to this Technical Specification (8.4) a test portion $A_3$ of (20 ± 0,02) g dry matter is necessary, of which the dry matter content $w_{dm}$ is known and of which at least a mass percentage of 95 % (dry matter) of the particles is smaller than 4 mm.

The dry residue of the sample shall be determined from a subsample which is dried at 105 °C ± 3 °C according to EN 14346.



The dry residue expressed as a percentage of the mass fraction is calculated as follows:



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

where

$w_{dr}$ is the dry residue of the sample, expressed as percentage (%);

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

$m_r$ is the mass before drying, in grams (g).

Calculate the undried mass of the test portion $M$w in grams to be used for the test as follows:

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

where

$M_w$ is the total mass of the test portion, in grams (g);

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

$w_{dr}$ is the dry residue of the sample, expressed as percentage (%).

For the sampling of solid earthy and stony building and waste materials for leaching tests no standards are yet available that are aimed at preserving the reducing capacity of the material to be tested. It is recommended to use the procedures described in EN 14899, taking into account the instructions to limit contact with the outside air (see Annex C).

If the sample from which the test portions are obtained has to undergo pretreatment, use the procedures described in EN 15002, taking into account the instructions to limit contact with the outside air as much as technically feasible (see Annex C).

## 6 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

**6.1 Demineralized water**, with a conductivity of max. 1 mS/cm.

**6.2 Hydrochloric acid**, $c(HCl)$ = (1 ± 0,1) mol/l.

**6.3 Sulphuric acid**, $c(H_2SO_4)$ = (1 ± 0,01) mol/l.

**6.4 Sodium hydroxide solution**, $c(NaOH)$ = (1 ± 0,1) mol/l.

**6.5 Cerium(IV) sulphate solution**, $c(Ce(SO_4)_2)$ = (0,1 ± 0,001) mol/l in (1 ± 0,01) mol/l sulphuric acid.

**6.6 Iron(II) sulphate solution**, $c(FeSO_4)$ = (0,1 ± 0,001) mol/l in (0,1 ± 0,001) mol/l sulphuric acid.



**6.7 Standard redox solutions**, suitable for the calibration of a redox electrode, with an inaccuracy of max. 1 mV.



NOTE A buffer solution with $E_H$ = 439 mV ($E_{meas}$ = 220 mV) and pH 7 will usually suffice.

**6.8 Nitrogen gas**, $N_2$, of a purity of 99,999 %, $O_2$ contamination < 3 ppm.

It shall be possible to supply the nitrogen gas as a constant stream.

NOTE Argon gas can be used instead of nitrogen gas also. The use of nitrogen gas is more cost effective and therefore prescribed in this Technical Specification.

## 7 Equipment

Check the equipment and requisites listed below before use for proper operation and absence of disrupting elements that may affect the result of the test.

Calibrate and/or check the equipment listed under 7.1, 7.2, 7.5, 7.9 and 7.10 before use.

**7.1 $E_H$ electrode** with reference electrode, standard combination of an $E_H$ platinum electrode with a reference electrode of Ag/AgCl or calomel ($Hg_2Cl_2$).

NOTE No standards are available for the calibration and measurement of the redox potential on a laboratory scale. The guidelines of the electrode manufacturer can be followed for this.

**7.2 pH meter**, with a measurement accuracy of at least ± 0,1 pH units and calibrated according to EN ISO 10523.

**7.3 Erlenmeyer**, sealable 100 ml glass Erlenmeyer or glass pot, with a neck that is wide enough to pass a pH electrode or $E_H$ electrode (7.1) through it.