SLOVENSKI STANDARD
SIST EN 1744-1:2010+A1:2013

01-februar-2013

Nadomešča:
SIST EN 1744-1:2010

Preskusi kemičnih lastnosti agregatov - 1. del: Kemijska analiza

Tests for chemical properties of aggregates - Part 1: Chemical analysis

Prüfverfahren für chemische Eigenschaften von Gesteinskörnungen - Teil 1: Chemische Analyse

Essais visant à déterminer les propriétés chimiques des granulats - Partie 1: Analyse chimique

Ta slovenski standard je istoveten z: EN 1744-1:2009+A1:2012

ICS:

71.040.40	Kemijska analiza	Chemical analysis
91.100.15	Mineralni materiali in izdelki	Mineral materials and products

SIST EN 1744-1:2010+A1:2013 en,fr,de

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 1744-1:2009+A1

November 2012

ICS 91.100.15

Supersedes EN 1744-1:2009

English Version

Tests for chemical properties of aggregates - Part 1: Chemical analysis

Essais visant à déterminer les propriétés chimiques des granulats - Partie 1: Analyse chimique

Prüfverfahren für chemische Eigenschaften von Gesteinskörnungen - Teil 1: Chemische Analyse

This European Standard was approved by CEN on 17 October 2009 and includes Amendment 1 approved by CEN on 15 October 2012.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 1744-1:2009+A1:2012: E

Contents

Page

Foreword

This document (EN 1744-1:2009+A1:2012) has been prepared by Technical Committee CEN/TC 154 “Aggregates”, the secretariat of which is held by BSI.

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 May 2012, and conflicting national standards shall be withdrawn at the latest by May 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.

This document supersedes [A1⟩ EN 1744-1:2009 ⟨A1].

This document includes Amendment 1, approved by CEN on 2012-10-15.

The start and finish of text introduced or altered by amendment is indicated in the text by tags [A1⟩ ⟨A1].

This standard forms part of a series of tests for chemical properties of aggregates. Test methods for other properties of aggregates are covered by the following European Standards:

EN 932 (all parts), *Tests for general properties of aggregates*

EN 933 (all parts), *Tests for geometrical properties of aggregates*

EN 1097 (all parts), *Tests for mechanical and physical properties of aggregates*

EN 1367 (all parts), *Tests for thermal and weathering properties of aggregates*

The other parts of EN 1744, *Tests for chemical properties of aggregates*, are:

— *Part 3: Preparation of eluates by leaching of aggregates*

— *Part 4: Determination of water susceptibility of fillers for bituminous mixtures*

— *Part 5: Determination of acid soluble chloride salts*

— *Part 6: Determination of the influence of recycled aggregate extract on the initial setting time of cement*

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: 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.

1 Scope

This European Standard specifies procedures for the chemical analysis of aggregates. It specifies the reference procedures and, in certain cases, an alternative method which can be considered as giving equivalent results.

Unless otherwise stated, the test methods specified in this standard may be used for factory production control, for audit tests or for type tests.

This standard describes the reference methods used for type testing and in cases of dispute (and alternatives methods) for chemical analyses of aggregates. For the purpose of type testing and in cases of dispute only the reference method should be used. For other purposes, in particular factory production control, other methods may be used provided that an appropriate working relationship with the reference method has been established.

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 196-1, *Methods of testing cement — Part 1: Determination of strength*

EN 196-2:2005, *Methods of testing cement — Part 2: Chemical analysis of cement*

EN 459-2, *Building lime — Part 2: Test methods*

EN 932-1, *Tests for general properties of aggregates — Part 1: Methods for sampling*

EN 932-2, *Tests for general properties of aggregates — Part 2: Methods for reducing laboratory samples*

EN 932-5, *Tests for general properties of aggregates — Part 5: Common equipment and calibration*

EN 932-6, *Tests for general properties of aggregates — Part 6: Definitions of repeatability and reproducibility*

EN 933-2, *Tests for geometrical properties of aggregates — Part 2: Determination of particle size distribution — Test sieves, nominal size of apertures*

EN 1015-4, *Methods of test for mortar for masonry — Part 4: Determination of consistence of fresh mortar (by plunger penetration)*

EN 1015-9, *Methods of test for mortar for masonry — Part 9: Determination of workable life and correction time of fresh mortar*

EN 1015-11, *Methods of test for mortar for masonry — Part 11: Determination of flexural and compressive strength of hardened mortar*

EN 1097-6, *Tests for mechanical and physical properties of aggregates — Part 6: Determination of particle density and water absorption*

ISO 384:1978, *Laboratory glassware — Principles of design and construction of volumetric glassware*

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 649-1, *Laboratory glassware — Density hydrometers for general purposes — Part 1: Specification*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

DIN 12242-1:1980, *Laboratory glassware; interchangeable conical ground joints, dimensions, tolerances*

3 Terms and definitions

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

3.1
test portion
subsample used as a whole in a single test

3.2
test specimen
sample used in a single determination when a test method requires more than one determination of a property

3.3
laboratory sample
sample intended for laboratory testing

3.4
constant mass
successive weighings after drying at least 1 h apart not differing by more than 0,1 %

NOTE In many cases constant mass can be achieved after a test portion has been dried for a pre-determined period in a specified oven at (110 ± 5) °C. Test laboratories can determine the time required to achieve constant mass for specific types and sizes of sample dependent upon the drying capacity of the oven used.

4 Reagents

4.1 General

4.1.1 Unless otherwise stated, use only analytical grade reagents and demineralized water, or water of equivalent purity.

NOTE 1 Unless otherwise stated "%" means "% by mass".

NOTE 2 Where no tolerances are given for reagent volumes or masses, the values quoted are approximate. In such cases volumes delivered from measuring cylinders and indicated masses using the ordinary balances specified in 5.2.4 and 5.2.5 are considered sufficiently accurate for the purposes of this European Standard.

NOTE 3 Unless otherwise stated reagent solutions may be assumed to have long-term stability.

NOTE 4 All chemicals should be treated as potential poisons with toxic properties and appropriate precautions taken before their use. Always take time to assess possible hazards before starting any procedures and constant attention should be maintained.

4.1.2 Concentrated liquid reagents shall have the following densities in g/cm³ at 20 °C:

Hydrochloric acid : 1,18 to 1,19

Nitric acid : 1,39 to 1,42

Sulfuric acid : 1,84

Ammonium hydroxide : 0,88 to 0,91

The degree of dilution shall be indicated as a volumetric sum.

NOTE 1 For example in 4.11.4, "hydrochloric acid (1+1)" means that 1 volume of concentrated hydrochloric acid is to be mixed with 1 volume of water.

NOTE 2 Ready for use solutions may be used as an alternative.

4.2 Reagents for determination of water-soluble chloride salts using the Volhard method (Clause 7)

4.2.1 Silver nitrate ($AgNO_3$) solution, 0,100 mol/l, prepared by drying about 20 g of silver nitrate for at least 1 h at a temperature of (110 ± 5) °C, left to cool in a desiccator and then weighing (16,987 ± 0,001) g of the dried silver nitrate, dissolving in water and diluting to 1 l in a volumetric flask (5.3.6). Store the solution in the amber-coloured glass reagent bottle (5.2.14) and protect from prolonged exposure to sunlight.

4.2.2 Thiocyanate (KSCN or NH_4SCN) solution, approximately 0,1 mol/l, prepared by dissolving 9,7 g of potassium thiocyanate or 7,6 g ammonium thiocyanate in water and diluting to 1 l in a volumetric flask.

Pipette 25 ml of silver nitrate solution (4.2.1) into a conical flask (5.3.5) and add 5 ml of nitric acid (4.2.3) and 1 ml of ammonium iron (III) sulfate indicator solution (4.2.5).

Add the thiocyanate solution from a burette (5.2.13) until the first permanent colour change occurs, that is from white opalescence to pale brown. Note the volume of thiocyanate solution added.

Calculate the concentration of the thiocyanate solution c_T, (in mol/l), from the following equation:

$$c_T = 2,5/V_1 \tag{1}$$

where

V_1 is the volume of thiocyanate added (in ml).

Standardize the solution at weekly intervals, or before use if the tests are less frequent.

4.2.3 Nitric acid (HNO_3), approximately 6 mol/l, prepared by adding 100 ml of nitric acid (4.1) to 150 ml water, boiling the diluted acid in a fume cupboard (5.2.17) until it is colourless and leave to cool to room temperature.

4.2.4 Chloride free technical grade 3,5-trimethylhexan-1-ol.

4.2.5 Ammonium iron (III) sulfate $NH_4Fe(SO_4)_2.12H_2O$ indicator solution, prepared by adding 60 g of water to 50 g ammonium iron (III) sulfate, warming to dissolve, and adding 10 ml nitric acid (4.2.3).

Leave the solution to cool to room temperature and store in a glass bottle (5.2.15).

4.3 Reagents for determination of water-soluble chloride salts by potentiometry (Clause 8)

4.3.1 Silver nitrate ($AgNO_3$) solution, 0,01 mol/l, prepared using the same procedure as specified in 4.2.1, but dissolving 1,699 g of dried silver nitrate in a 1 l volumetric flask (5.3.6).

4.3.2 Sodium chloride (NaCl) solution, 0,02 mol/l, prepared by drying about 2 g of sodium chloride at a temperature of (110 ± 5) °C for 1 h to 2 h, leaving to cool and then weighing (1,169 ± 0,001) g of the dried sodium chloride, dissolving in water and diluting to 1 l in a volumetric flask (5.3.6).

4.4 Reagent for factory production control determination of water-soluble chloride salts using the Mohr method (Clause 9)

Potassium chromate (K_2CrO_4) solution, prepared by dissolving 10 g of potassium chromate in 100 ml water.

4.5 Reagents for determination of water-soluble sulfates (Clause 10)

4.5.1 Hydrochloric acid (HCl) solution, made by adding 200 ml concentrated hydrochloric acid (4.1) to 800 ml water.

4.5.2 Barium chloride ($BaCl_2$) solution, made by dissolving 100 g of barium chloride ($BaCl_2.2H_2O$) in 1 l of water, and filtered through a medium grade filter paper before use.

4.6 Reagents for determination of total sulfur content (Clause 11)

4.6.1 Hydrogen peroxide (H_2O_2), 30 % concentration.

4.6.2 Indicator methyl red (dissolve 20 mg methyl red powder in 50 ml of ethanol, then add 50 ml of water).

4.7 Reagents for determination of acid soluble sulfide content (Clause 13)

4.7.1 Lead acetate solution, made by dissolving approximately 0,2 g of lead acetate ($Pb(CH_3COO)_2.3H_2O$) in water and making up to 100 ml.

4.7.2 Ammoniacal zinc sulfate solution, made by dissolving 50 g of zinc sulfate ($ZnSO_4.7H_2O$) in 150 ml water and add 350 ml of concentrated ammonium hydroxide (NH_4OH). Leave to stand for at least 24 h and filter through a medium grade filter paper.

4.7.3 Tin (II) chloride ($SnCl_2.2H_2O$).

4.7.4 Metallic chromium (Cr), in powder form.

4.7.5 Standard potassium iodate solution containing 0,016 7 mol/l, made by dissolving successively in freshly boiled and cooled water in a 1 l volumetric flask, (3,6 ± 0,1) g to the nearest 0,1 mg (6.3) of potassium iodate (KIO_3) dried at (110 ± 5) °C, two pellets (about 0,4 g) of sodium hydroxide (NaOH) and 25 g of potassium iodide (KI). Make up to the mark with freshly boiled and cooled water.

NOTE 1 A trace of sodium hydroxide will help to stabilize this solution. The solution should be discarded when it becomes discoloured.

The factor *F* of this solution is calculated from the following equation:

$$F = \frac{m_1}{3{,}566\,8} \tag{2}$$

where

m_1 is the mass of the portion of potassium iodate in g.

NOTE 2 The value of factor F for the solution should be calculated using the mean value of three determinations, expressed to three decimal places.

NOTE 3 If the sulfide content is less than 0,1 % by mass, solutions ten times less concentrated should be used. They are prepared by pipetting 100 ml of the solutions (4.7.5 and 4.7.6) into 1 l volumetric flasks and making up to the mark with water.

4.7.6 Sodium thiosulfate solution approximately 0,1 mol/l, made by dissolving 24,82 g of sodium thiosulfate ($Na_2S_2O_3.5H_2O$) in water and making up to 1 l.

Before each test series, determine the factor *f* of this solution using one of the two methods that follow.

a) Standardization (preferred method) in relation to the standard potassium iodate solution (4.7.5).

Pipette 20 ml of the standard potassium iodate solution into a 500 ml conical flask and dilute with approximately 150 ml of water. Acidify with 25 ml of hydrochloric acid (1 + 1) and titrate with the approximately 0,1 mol/l sodium thiosulfate solution to a pale yellow colour.

Then add 2 ml of the starch solution (4.7.7) and continue the titration until the colour changes from blue to colourless.

The factor *f* of this solution is calculated from the following equation:

$$f = \frac{20 \times 0{,}016\,67 \times 214{,}01 \times F}{3{,}566\,8 \times V_2} = 20 \times \frac{F}{V_2} \tag{3}$$

where

F is the factor of the standard potassium iodate solution (4.7.5) expressed in mol/l;

V_2 is the volume of the approximately 0,1 mol/l sodium thiosulfate solution used for the titration.

NOTE 1 3,566 8 g/l of potassium iodate corresponds to a solution with exactly 0,016 67 mol/l of potassium iodate and 214,01 is the molecular mass of KIO_3.

b) Standardization in relation to a known quantity of potassium iodate.

Place (70 ± 5) mg of potassium iodate in a 500 ml conical flask and dissolve in approximately 150 ml of water.

Add about 1 g of potassium iodide, acidify with 25 ml of hydrochloric acid (1 + 1) and titrate with the approximately 0,1 mol/l sodium thiosulfate solution until a pale yellow colour is obtained. Then add 2 ml of the starch solution (4.7.7) and titrate until the colour changes from blue to colourless.

The factor *f* of this solution is calculated from the following equation:

$$f = \frac{1\,000 \times m_2}{3{,}566\,8 \times V_3} = 280{,}363\,4 \times \frac{m_2}{V_3} \tag{4}$$

where

m_2 is the mass of the portion of potassium iodate in g;

V_3 is the volume of the approximately 0,1 mol/l sodium thiosulfate solution used for the titration.

NOTE 2 3,566 8 g/l of potassium iodate corresponds to a solution with exactly 0,016 67 mol/l of potassium iodate.

NOTE 3 The value of factor *f* for the solution should be calculated using the mean value of three determinations, expressed to three decimal places.

4.7.7 Starch solution, made taking 1 g of starch (water soluble), adding 1 g of potassium iodide KI, dissolving in water and making up to 100 ml.

4.8 Reagents for determination of lightweight contaminators (see 14.2)

4.8.1 Zinc chloride solution, obtained by dissolving 7 kg of $ZnCl_2$ in 3 l of water to obtain a saturated solution of density of (1,98 ± 0,02) g/cm^3 at (20 ± 3) °C. The relative density of the solution, after cooling to room temperature, shall be checked using a suitable hydrometer (5.9.3).

NOTE Zinc chloride solution is moderately irritating to skin and mucous membranes.

4.8.2 Sodium polytungstate solution (as an alternative to 4.8.1), prepared by dissolving $3Na_2WO_4.9W0_3.H_2O$ crystals in water until the density of well stirred solution containing no undissolved crystals is (1,98 ± 0,02) g/cm^3 at (20 ± 3) °C. The relative density of the solution, after cooling to room temperature, shall be checked using a suitable hydrometer (5.9.3).

4.9 Reagents for determination of potential presence of humus (see 15.1)

4.9.1 A 3 % solution of sodium hydroxide, made by dissolving 30 g of sodium hydroxide pellets in water, cooling to room temperature and diluting to 1 l in a volumetric flask.

4.9.2 Standard colour solution, prepared by dissolving 45,0 g of $FeCl_3.6H_2O$ and 5,50 g of $CoCl_2.6H_2O$ in 279,5 g of water with 1 ml concentrated HCl.

Store the solution in a glass bottle.

NOTE 1 The solution should be stable for at least two weeks.

NOTE 2 The standard colour solution (tangerine-orange) is equivalent to Gardner Colour Standard Number°11 or Organic Plate Number°3[1].

4.10 Reagents for determination of fulvo acid content (see 15.2)

4.10.1 Hydrochloric acid, diluted (1 + 23) (4.1).

4.10.2 Stannous chloride solution, made by dissolving 22,5 g of $SnCl_2.2H_2O$ in 1 l of hydrochloric acid (4.10.1).

NOTE The solution should be stable for at least two weeks.

---

[1] Colour scale discs and equivalent standard solutions can be obtained from:
Tintometer Ltd, Lovibond House, Solar Way, Solstice Park, Amesbury, Wiltshire, SPA 7SZ, UK
Tel: +44.1980.664800; Fax: +44.1980.625412; Email: sales@tintometer.com; website: www.tintometer.com
or
Intertest Benelux v.o.f. P.O. Box 373 NL - 4870 AJ Etten-Leur
Phone: +31 (0)76 501 53 51; Fax: +31 (0)76 503 31 81; Email: intertest@intertest.nl or info@intertest.nl; website: www.intertest.nl.
This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.