SLOVENSKI STANDARD
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Vpliv cementnih proizvodov na pitno vodo - Preskusne metode - 3. del: Prehod snovi iz industrijsko izdelanih cementnih proizvodov

Influence of cementitious products on water intended for human consumption - Test methods - Part 3: Migration of substances from factory-made cementitious products

Einfluss zementgebundener Produkte auf Wasser für den menschlichen Gebrauch - Prüfmethoden - Teil 3: Migration von Substanzen aus fabrikmässig hergestellten zementgebundenen Produkten

Influence des produits à base de ciment sur l'eau destinées à la consommation humaine - Méthodes d'essais - Partie 3 : Migration de substances à partir des produits fabriqués en usine

Ta slovenski standard je istoveten z: **prEN 14944-3 rev**

ICS:

13.060.20	Pitna voda	Drinking water
67.250	Materiali in predmeti v stiku z živili	Materials and articles in contact with foodstuffs
91.100.10	Cement. Mavec. Apno. Malta	Cement. Gypsum. Lime. Mortar

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Influence of cementitious products on water intended for human consumption - Test methods - Part 3: Migration of substances from factory-made cementitious products

Influence des produits à base de ciment sur l'eau destinée à la consommation humaine - Méthodes d'essai - Partie 3 : Migration de substances à partir des produits fabriqués en usine

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This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 164.

If this draft becomes a European Standard, 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.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (prEN 14944-3:2015) has been prepared by Technical Committee CEN/TC 164 "Water supply", the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 14944-3:2007.

In comparison with EN 14944-3:2007, the following changes have been made:

— requirements for disinfection (preconditioning with 50 mg/L chlorine) have been removed;

— procedure for extending the number of migration periods have been included.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This document describes a test method to produce migration waters for the assessment of inorganic and organic substances.

This European Standard will result in one of a series of standards that support appropriate standards.

This European Standard consists of a series dealing with the influence of cementitious and associated non-cementitious products/materials on water intended for human consumption, including:

— *Part 1: Influence on organoleptic parameters and migration of organic substances (TOC) from factory made cementitious products*

— *Part 2: Influence of site-applied cementitious materials and associated non-cementitious products/materials on organoleptic parameters and migration of organic substances (TOC)*

— *Part 3: Migration of substances from factory made cementitious products*

— *Part 4: Migration of substances from site-applied cementitious materials and associated non-cementitious products/materials*

Introduction

With respect to any potential adverse effects of products and materials on the quality of water intended for human consumption, it should be understood that relevant national regulations remain in force until verifiable European acceptance criteria are adopted.

1 Scope

This European Standard specifies a method to determine the migration of substances from factory made cementitious products into test waters after contact with the products.

This European Standard is applicable to factory made cementitious products, e.g. cement mortar linings to metallic pipes, tanks, concrete pipes, etc., intended to be used for the transport and storage of water intended for human consumption, including raw water used for the production of drinking water.

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

EN 1015-2, *Methods of test for mortar for masonry - Part 2: Bulk sampling of mortars and preparation of test mortars*

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

EN 10088-1, *Stainless steels - Part 1: List of stainless steels*

EN 12350-1, *Testing fresh concrete - Part 1: Sampling*

EN 12390-1, *Testing hardened concrete - Part 1: Shape, dimensions and other requirements for specimens and moulds*

EN 12390-2, *Testing hardened concrete - Part 2: Making and curing specimens for strength tests*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

EN ISO 7393-1, *Water quality - Determination of free chlorine and total chlorine - Part 1: Titrimetric method using N, N-diethyl-1,4-phenylenediamine (ISO 7393-1)*

EN ISO 7393-2, *Water quality - Determination of free chlorine and total chlorine - Part 2: Colorimetric method using N, N-diethyl-1, 4-phenylenediamine, for routine control purposes (ISO 7393-2)*

ISO 10523, *Water quality - Determination of pH*

3 Terms and definitions

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

3.1
cementitious product
factory made product containing a cementitious material supplied in the hardened state with a formed surface prior to its incorporation into the construction works

3.2
cementitious material
material that contains a hydraulic cement in sufficient proportion to act as the main binder by forming a hydrate structure which governs the performance of the material

3.3
associated non-cementitious product
product which is applied to the surface of a cementitious product, directly or indirectly, during manufacture (or construction) and which either provides a porous seal to the product or which remains as a residue in contact with water, e.g. porous seal coats, formwork release agents and curing compounds

3.4
porous seal coat
polymeric (usually organic) materials applied in a thin (25 µm – 200 µm thickness) surface layer to a cement mortar lining in order to restrict (but not prevent) interactions between the mortar and conveyed water

Note 1 to entry: See ISO 16132 [1].

3.5
proxy sample
sample of fresh mortar or fresh concrete taken from material to be used for the production of a factory made product, either spray-applied to a laboratory test plate (mortar only) or cast into a mould (mortar or concrete) of appropriate dimensions (e.g. standard cube, cylinder or prism etc.) and compacted (where appropriate), cured and hardened under conditions representative of those intended for the product

3.6
fresh concrete
concrete that is fully mixed and still in a condition capable of being compacted by the chosen method

3.7
fresh mortar
cement mortar that is fully mixed and still in a condition of being applied to a substrate by the chosen method

3.8
test
technical operation that consists of the determination of one or more characteristics of a given product

3.9
test procedure
specified technical method for performing a test

3.10
sample
one or more units, or a specified quantity, drawn from a batch or lot, selected at random for inspection, e.g. at the factory or in a laboratory

3.11
test piece
sample or portion which is to be conditioned, treated or otherwise prepared to be tested to obtain a single test result

3.12
nominal diameter
DN/ID
DN/OD
numerical designation of the size of a component, which is a whole number approximately equal to the actual dimensions in millimetres

Note 1 to entry: This applies to either the internal diameter (DN/ID) or the external diameter (DN/OD).

3.13
preconditioning
succession of contact periods of a test piece with the preconditioning water (3.14) before contact with the test water

3.14
preconditioning water
water used for preconditioning prepared as described in 5.2.1

3.15
test water
water used for testing purposes prepared as described in 5.2.2 and used in accordance with 5.2.3 and 5.2.4

3.16
migration water
test water which has been in contact with a test piece under specified conditions

3.17
blank water
test water which has been kept at the same specified conditions as migration water but without contact with the test piece

3.18
tap water
drinking water distributed by a public supplier.

Note 1 to entry: Tap water is used as a lubricant/coolant for the sawing and coring operations used to obtain test pieces generally from products of large dimensions. See normative Annexes A, B and C.

3.19
demineralized water
water conforming to the requirements in EN ISO 3696 for grade 3

4 Principle

Each test piece is subjected to a specified preconditioning procedure where the surface which is exposed in practice to water intended for human consumption is brought into contact with preconditioning water during five sequential periods: three periods of 24 h, 1 period of 72 h and a final period of 24 h.

The preconditioned test piece is then brought into contact with test water, chlorinated and/or chlorine-free during three sequential migration periods. A migration period is either:

a) 72 h at (23 ± 2) °C for products intended to come into contact with chlorinated or chlorine-free cold water;

b) 24 h at a specified elevated temperature for products intended to come into contact with warm or hot chlorine-free water.

Migration rates are calculated after each contact period by determination of the content of specified substances in the corresponding migration water.

NOTE 1 The test is carried out under conditions that ensure that reliable migration rates are calculated. These conditions are not meant to simulate any service condition. Relating the results obtained from this European Standard to the service condition is carried out using a conversion procedure. This procedure will be specified in regulations.

NOTE 2 The selection of:

c) the appropriate test water, chlorinated and/or chlorine-free, from those made available in this European Standard,

d) the temperature of the test water

is specified in product or system standards or in national or European regulations, as appropriate.

5 Reagents

Use only reagents of analytical quality unless otherwise stated.

5.1 Sodium hypochlorite solution

Prepared from a technical or general purpose reagent grade of sodium hypochlorite (NaOCl), using test water (5.2.2) and having a known concentration of about 0,1 % by mass of free chlorine determined in accordance with either EN ISO 7393-1 or EN ISO 7393-2.

Unless tests have proved otherwise the sodium hypochlorite solution should be considered unstable and be prepared on the day of use.

5.2 Waters to be used for testing

5.2.1 Preconditioning water prepared by dissolving (222 ± 2) mg anhydrous calcium chloride ($CaCl_2$) and (336 ± 2) mg sodium hydrogen-carbonate ($NaHCO_3$) in one litre of demineralized water (3.19). The *pH* is determined in accordance with ISO 10523 and adjusted to 7,4 ± 0,1 by bubbling air and/or CO_2 into the solution.

NOTE The target total hardness is 200 mg/l as $CaCO_3$ and the target alkalinity is 244 mg/l as HCO_3^-.

5.2.2 Test water, prepared by dissolving (110 ± 1) mg anhydrous calcium chloride ($CaCl_2$), (140 ± 1) mg sodium hydrogen carbonate ($NaHCO_3$) and (48 ± 1) mg sodium silicate ($Na_2SiO_3.9H_2O$) in one litre of demineralized water.

The *pH* is determined in accordance with ISO 10523 and adjusted to 7,0 ± 0,1 by bubbling air and/or CO_2 into the solution.

NOTE The target total hardness is 100 mg/l as $CaCO_3$, the target alkalinity is 122 mg/l as HCO_3^- and the silica concentration is 10 mg/l as SiO_2.

5.2.3 Test water without chlorine content (chlorine-free) shall consist of a batch of test water (5.2.2) used for contact with test pieces and preparation of the blank water (3.17).

5.2.4 Test water with chlorine content (chlorinated) shall consist of test water (5.2.2) with a free chlorine content of (1,0 ± 0,2) mg/l as Cl_2, determined in accordance with either EN ISO 7393-1 or EN ISO 7393-2, after addition of sodium hypochlorite solution (5.1).

5.3 Cleaning liquids for apparatus

Use one of the following cleaning liquids:

— non-perfumed biodegradable detergent;

— hydrochloric acid, 2 mol/l;

— nitric acid, 10 % or 1,5 mol/l.

6 Apparatus

6.1 General

For cleaning the glassware, and appropriate apparatus, before use, the following general requirements apply:

a) Clean the glassware to be used, using detergent (5.3). Rinse the glassware in demineralized water.

b) Clean the inner surface of the glassware with hydrochloric acid (5.3) and rinse it with demineralized water. For stainless steel, clean with nitric acid (5.3) and then rinse with demineralized water.

c) Before use, rinse the glassware, and appropriate apparatus, at least three times using preconditioning water before preconditioning (8.3) or test water before the test procedure (Clause 9).

6.2 Apparatus and materials for test piece preparation (see normative Annexes A, B and C)

6.2.1 Stainless steel plates and cylinders

6.2.1.1 Stainless steel

Stainless steel shall be austenitic, super austenitic or duplex grades in accordance with the corresponding numerical designations, 1.4301, 1.4436, 1.4429, 1.4259 or 1.4462 in EN 10088-1 for stainless steels.

NOTE The grades above are specified for the use of stainless steel as reinforcement in concrete. Therefore they are considered to be inert when used in contact with cementitious proxy samples (see normative Annexes A, B and C of this European Standard).

6.2.1.2 Plates

In order to provide a sufficient volume of migration water for assessment, the surface area of one face of a plate should be between 10 000 mm^2 and 90 000 mm^2. The length/width of the plates should be selected to be consistent with the dimensions of the test container and the volume of test water in which they will be immersed.

6.2.1.3 Cylinders

The diameter and length of a cylinder should be consistent with the dimensions of the test piece (see normative Annexes A, B or C and informative Annexes D and E) and the volume of test water appropriate to the specified *S/V* ratio given in 7.3.

6.2.2 Glass cylinders

The diameter and length of a glass cylinder should be consistent with the dimensions of the test piece (see normative Annexes A, B or C and informative Annexes D and E) and the volume of test water appropriate to the specified *S/V* ratio given in 7.3. Glass cylinders should be provided with suitable external (opaque) shielding for use during migration procedures (test pieces and blanks), in order to minimize exposure of migration waters to ambient light.

6.2.3 Moulds for forming test pieces

Moulds for forming prisms of mortar shall conform to the requirements of EN 196-1, as specified for use in EN 1015-11, or to EN 12390-1 for forming cubes/cylinders of concrete, with modifications to materials and dimensional tolerances as specified by the appropriate normative Annex A, B or C of this European Standard.

Clean moulds and any filling frame used with a mould, by thoroughly washing with non-perfumed detergent (5.3) and tap water (3.18), rinsing with copious amounts of tap water, followed by a final rinse with demineralized water (3.19) and dry before use.

Where a factory made cementitious product has been formed in a process where its entire contact surface has been in contact with a release agent then where proxy samples (3.5) are used, the same release agent shall be applied to the internal surfaces of the mould, otherwise the use of release agents is not permitted by this European Standard.

6.3 Apparatus and materials for preconditioning and migration procedure

6.3.1 Vessels, containers, stoppers and connectors shall consist of a material, such as glass, PTFE, steel and stainless steel that is inert under the specified test conditions (Clause 9).

The material PTFE should only be used when there is a small contact area with the test water. Thus PTFE is unsuitable for containers.

6.3.2 **Equipment**, capable of maintaining the test temperature within ± 2 °C for the duration of the test.

6.3.3 Where required, use only **sealants** that do not affect the determinations under the specified test conditions (see Clause 9).

7 Samples and test pieces

7.1 Sampling, transport and storage of samples

Carry out sampling of factory made products in accordance with the relevant product standard, system standard or the national or European regulations, or the relevant normative annex to this European Standard, as appropriate.

Take care that the transport conditions do not influence the test results.

If it is necessary to store samples or test pieces before testing, ensure that they are protected from contamination taking into account any written instructions that are provided.

Where appropriate, clean storage containers using the same procedures as are used for the test containers.

Ensure that the surfaces of the test pieces intended to come into contact with the test water are free from any contamination, e.g. adhesive tape, labels, ink or pencil marks.

7.2 Preparation of test pieces

7.2.1 General

Prepare the test pieces in such a way that only the surface intended to come into contact with drinking water is exposed to the test water except as given in normative Annex C (see C.1.2.3.2.1) where stainless steel plates are coated with cement mortar on one face only prior to complete immersion during testing.

In the preparation of a test piece the following general principles apply:

a) ensure that test pieces are representative of the finished product;

b) during the preparation of test pieces, include any procedures which are performed in practice for curing and cleaning;

c) ensure that the minimum age of the test piece, at test, conforms to that recommended by the manufacturer for the product to be ready for use;

d) ensure that the surface area of the test piece is sufficient to fulfil the appropriate surface area to volume (*S*/*V*) ratio in accordance with the requirements of 7.3.

7.2.2 Factory made pipes, fittings and storage systems

Where possible, use the product or test piece as the test vessel, with dimensions that provide sufficient migration water for assessment. In cases where this is not practicable (e.g. large pipes, storage systems etc.), and where alternatives are specified, use as appropriate, an alternative test piece described in the relevant normative Annex A, B or C to this European Standard and an appropriate test arrangement given in informative Annex E.

NOTE Where it is required to discriminate between porous and non-porous coatings already applied to factory made products, use the test procedure given in informative Annex G of this European Standard.

7.3 Surface area-to-volume ratio (*S*/*V*) for use in the test procedure

7.3.1 General

The following general principles apply for *S*/*V* ratios:

a) the surface area to volume ratio (*S*/*V*) of the test piece exposed to the test water shall not be smaller than the *S*/*V* ratio of the product in the service condition;

b) where no difference in material composition and production process exists in the range of sizes produced, only the largest *S*/*V* ratio is required to be tested;

c) the ratio of the surface area, S, of the test piece intended to come into contact with volume, *V*, of the test water is expressed per decimetre, i.e. dm^{-1}.

NOTE The unit, dm^{-1}, can also be expressed as dm^2/dm^3 or dm^2/l.

7.3.2 Pipes and fittings

The *S*/*V* ratio is calculated, in dm^{-1}, according to the formula:

$$S/V = \frac{400}{[DN/ID]} \quad (1)$$

where

[DN/ID] (3.12) is the nominal internal diameter, in mm.

Test pipes and fittings of DN/ID less than 80, at the actual *S*/*V* ratio of the pipe diameter.

Test pipes and fittings of DN/ID equal to 80 and less than 300, at an *S*/*V* ratio of (5,0 ± 0,2) dm^{-1}.

Test pipes and fittings of DN/ID 300 or greater, at an *S*/*V* ratio of (1,3 ± 0,1) dm^{-1}.

NOTE But see 7.3.1 (b) for the acceptable minimum requirement for testing pipes that are produced in a range of sizes.

7.3.3 Storage systems (cement mortar, cement mortar lined or concrete)

Test storage systems at an *S*/*V* ratio of (1,3 ± 0,1) dm^{-1}.

8 Pre-treatment of samples (curing and preconditioning)

8.1 General

The procedures for curing cementitious products are given in 8.2.

The procedure for preconditioning at (23 ± 2) °C is given in 8.3.

8.2 Curing

Ensure that test pieces have been subject either to the curing conditions used in manufacture of the factory made product or, in the case of test pieces formed from proxy samples (3.5), to curing conditions that are representative of those used in the manufacture of the factory made product (see the relevant normative Annex A, B or C to this European Standard).

8.3 Preconditioning

Precondition test pieces at the appropriate *S*/*V* ratio given in 7.3.

Fill test pieces with, or immerse them in, or otherwise bring them into contact with (see the test arrangements in informative Annex E), preconditioning water (5.2.1) for a succession of five contact periods, without rinsing between contact periods, at a temperature of (23 ± 2) °C as follows:

— three periods of (24 ± 1) h;

— one period of (72 ± 1) h;

— one period of (24 ± 1) h.

After the fifth contact period determine the *pH* of the preconditioning water in accordance with ISO 10523. If the *pH* exceeds 9,5 stop the testing.

Where the *pH* exceeds 9,5 preconditioning may be repeated using new test pieces.

The results of co-normative research [2] obtained using this preconditioning water indicate that where the *pH* exceeds 9,5 after the fifth contact period, then steps should be taken to first investigate and then eliminate the cause(s) before proceeding to the test procedure (see Clause 9).

9 Test procedure

9.1 General

Where testing of products intended to come into contact with chlorinated or chlorine-free cold water is required, carry out the following procedures at 9.2.

Where testing at elevated temperature is required carry out the procedure in accordance with normative Annex F.

9.2 Preparation of migration water for analysis of substances

NOTE The number of tests to be carried out e.g. single tests or duplicates for each water type is specified in product or system standards or in national or European regulations, as appropriate.

9.2.1 Migration procedure

Begin the first contact period immediately after preconditioning the test piece.

Immerse in, or fill with, or otherwise bring the test piece (7.2) into appropriate contact with test water (5.2.3 and/or 5.2.4) and allow to stand for (72 ± 1) h at (23 ± 2) °C. In all cases (immersion, filling or other contact arrangement), ensure that the test piece or vessel/container is completely immersed or filled and minimize headspace in order to minimize contact between the test water and air, using a cover for the vessel/container.

At the end of this period, collect the migration water using appropriate sampling bottles for analysis.

NOTE The choice of the type of test water (chlorinated and/or chlorine-free) is specified by the product standard or system standards or in national or European regulations, as appropriate.

9.2.2 Second and third migration periods

Repeat **9.2.1** two more times using fresh test water each time, ensuring that the test pieces are put into contact with the same type of test water (e.g. chlorine-free) for the three successive periods.

9.2.3 Additional migration periods

Referring standards and/or national or European regulations may specify further sequential migration periods. Refer to Annex H for further guidance on the sequence and number of migration periods that can be specified.

9.3 Control samples (blank test)

For each contact period, carry out a blank test procedure using the same test conditions (test water, test temperature, contact periods, sealants used etc.) as described in 9.2 but omitting the test piece or replacing it by a glass container or plate, as appropriate.

10 Analysis

Carry out the required analysis on the migration waters using the analytical methods specified in referring documents. Determine at the end of each extraction period the concentration of the substance being measured. General guidance on analytical performance requirements such as detection limit and accuracy is contained in Guide to analytical Quality Control for water analysis, ISO/TS 13530 [3].

NOTE 1 If migration waters are not analysed immediately then ensure that the storage time and conditions do not adversely affect the analytical result.

NOTE 2 For some analytical methods and/or specific test procedures, recovery rates for the substances being determined need to be established using positive controls. Annex I gives further guidance.

11 Calculation of test results

11.1 Calculation of the concentration of the substances in the migration water

Calculate for each migration water the concentration of the measured substance as follows:

$$c_n^T = a_n^T - b_n^T \quad (2)$$

where

c^T_n is the concentration of the measured substance in mg/l;

a^T_n is the concentration of the substance in mg/l measured in the migration water;

b^T_n is the concentration of the substance in mg/l measured in the blank water.

For the conditions

T is the test temperature;

n is the sequence number of the migration period.

NOTE The migration of substances into water depends on the type of material and the migration conditions: temperature, contact time, S/V ratio and whether the water is static or flowing. For static test conditions and constant temperature, the increase in the concentration of the substance in the test water is asymptotic. However, for practical purposes the increase with time is assumed to be linear.

11.2 Calculation of the migration rate of the measured substances

Calculate for each migration water the migration rate M^T_n for a migrated substance from the concentration c^T_n as follows:

$$M_n^T = \frac{c_n^T}{(S/V \cdot t)} \quad (3)$$

where

M^T_n is the migration rate for the n'th migration period in $mg.dm^{-2}.day^{-1}$;

t is the duration of the migration period in days i.e. three days (72 ± 1) h for (23 ± 2) °C;

S/V is the surface area-to-volume ratio in dm^{-1}.

11.3 Calculation of the mean migration rate

Calculate the arithmetic mean migration rate $\overline{M_n^T}$ for replicate values of M^T_n for each test water (5.2.3 and 5.2.4).

12 Test report

12.1 General

The test report shall include the following information.