

SLOVENSKI STANDARD
oSIST prEN ISO 4531:2017
01-maj-2017

Steklasti in porcelanski emajli - Migracija iz emajliranih predmetov v stiku z živili - Metoda preskušanja in dovoljene meje (ISO/DIS 4531:2017)

Vitreous and porcelain enamels - Migration from enamelled ware in contact with food - Method of test and permissible limits (ISO/DIS 4531:2017)

Emails - Migration aus emaillierten Gegenständen für den Kontakt mit Lebensmitteln - Prüfverfahren und zulässige Grenzwerte (ISO/DIS 4531:2017)

Émaux vitrifiés - Migration dans les articles émaillés en contact avec les aliments - Méthode d'essai et limites admissibles (ISO/DIS 4531:2017)

Ta slovenski standard je istoveten z: prEN ISO 4531

ICS:

25.220.50	Emailne prevleke	Enamels
67.250	Materiali in predmeti v stiku z živili	Materials and articles in contact with foodstuffs
97.040.60	Kuhinjska posoda, jedilni servisi in jedilni pribor	Cookware, cutlery and flatware

oSIST prEN ISO 4531:2017

en

DRAFT INTERNATIONAL STANDARD

ISO/DIS 4531

ISO/TC 107

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Vitreous and porcelain enamels — Migration from enamelled ware in contact with food — Method of test and permissible limits

Émaux vitrifiés — Migration dans les articles émaillés en contact avec les aliments — Méthode d'essai et limites admissibles

ICS: 25.220.50; 67.250; 97.040.60

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ISO/DIS 4531:2017(E)**Foreword**

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The committee responsible for this document is ISO/TC 107.

This international standard cancels and replaces ISO 4531-1:1998 and ISO 4531-2:1998, which have been combined and technically revised.

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Introduction

The migration of metal-ions from enamelled ware requires effective means of control to ensure the protection of the population against possible hazards arising from the use of improperly formulated, applied and fired enamels and/or inorganic decorations on the food contact surfaces of enamelled ware used for the preparation, cooking, serving and storage of foodstuffs.

As a secondary consideration, different requirements from country to country for the control of the release of toxic materials from the surfaces of enamelled ware present non-tarif barriers to international trade in these commodities. Accordingly, there is a need to establish internationally accepted methods of testing enamelled ware for the release of metal-ions.

The amount of metal-ions determined by the method of test specified in this standard will not be higher than the quantities contained in foods and drinks anyway.

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Vitreous and porcelain enamels — Migration from enamelled ware in contact with food — Method of test and permissible limits

1 Scope

This standard specifies a simulating method of test for determination of the release of metal-ions from enamelled ware, which are intended to come into contact with food (including drinks).

This standard also specifies permissible limits for the release of metal-ions from enamelled ware, which are intended to come into contact with food (including drinks).

This standard is applicable to enamelled ware, including tanks and vessels, which are intended to be used for the preparation, cooking, serving and storage of food.

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.

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

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

ISO 3585, *Borosilicate glass 3.3 — Properties*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

ISO 28764, *Vitreous and porcelain enamels — Production of specimens for testing enamels on sheet steel, sheet aluminium and cast iron*

3 Principle

To extract metal-ions, if present, from the surfaces of articles or test specimen a 3 % (V/V) acetic solution shall be used. Three successive migrations shall be performed using the same sample and a fresh test solution per migration. The first two migration solutions (M1, M2) will be discarded, only the third migration solution (M3) shall be used for analysis. A blank test (B1, B2 & B3) is required for each migration, of which only the third migration solution (B3) shall be used for analysis.

4 Reagents

During the determination, use only reagents of recognized analytical grade and only distilled water, or water of equivalent purity (grade 3 water complying with the requirements of ISO 3696). The electrical conductivity of the water shall be in between of 1 and 5 $\mu\text{S}/\text{cm}$.

The usage of ultrapure water having an electrical conductivity $<1 \mu\text{S}/\text{cm}$ is forbidden, because it will cause an osmotically dissolving of the enamel coat leading to wrong results.

It is permissible to prepare proportionately greater quantities of test solution and analytical solutions than specified in 4.2.

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4.1 Acetic acid, (CH_3COOH), concentrated, density $\rho = 1,05 \text{ g/ml}$.

4.2 Test solution, acetic acid, 3 % (V/V) solution.

By means of a graduated measuring cylinder (5.4) add, to 500 ml of water, (30 ± 1) ml of concentrated acetic acid (4.1) and make up to 1 l. Prepare the test solution freshly prior to use and in sufficient quantity to enable the whole of any group of tests and analysis to be completed.

4.3 Analytical stock solutions, the standard stock solutions, or multi-element solutions, used for ICP-Analysis shall be used.

4.4 Dishwashing agent, commercially available non-acidic manual dishwashing detergent in common dilution.

5 Apparatus

Use only laboratory glassware, complying with the requirements of appropriate International Standards, where they exist, and made of borosilicate glass as specified in ISO 3585.

5.1 ICP spectrometer, with a detection limit equal to or smaller than the values in [table 1](#).

Table 1 — minimum detections limits for the ICP spectrometer

Element	Detection limit $\mu\text{g/l}$
Ag	1
Ba	0,15
Cd	0,5
Co	1,2
Cr	1
Cu	1,5
Li	1
Mn	0,15
Mo	2
Ni	2,1
Pb	8
Sb	3
V	2
Zn	0,8

The elements arsenic, thallium, beryllium, and mercury shall not be used in vitreous enamelled products that are intended to be used in contact with food.

5.2 One-mark volumetric flasks, capacities 100 ml and 1 000 ml, complying with the requirements specified for class B or better one-mark volumetric flasks in ISO 1042. Other sizes of one-mark volumetric flasks may also be required.

5.3 One-mark pipettes, capacities 10 ml and 100 ml, complying with the requirements specified for class B or better one-mark pipettes in ISO 648. Other sizes of one-mark pipettes may also be required.

5.4 Graduated measuring cylinders, capacities 50 ml and 500 ml, complying with the requirements specified in ISO 4788. Other sizes of graduated measuring cylinders may also be required.

5.5 Vessels, containers, stoppers and connectors shall consist of a material, such as glass, PTFE or stainless steel, that is inert under the specified test conditions.

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

5.6 Vessels made of borosilicate glass, according to ISO 3585, internal diameter 80 mm, external diameter 90 mm, height 36 mm, 10 mm diameter of the filling hole.

The calculation of the surface/ volume (S/V) ratio is reported below.

The test plate per chamber has a diameter of 80 mm. Therefore the tested area a of one test plate is 5025 mm^2 ($a = \pi r^2$).

The volume of the chamber V with a height h of 36 mm and a gasket thickness t of 2 mm is calculated from [equation \(1\)](#).

$$V = (h + 2 \cdot t) \cdot a \quad (1)$$

$$V = (36 \text{ mm} + 2 \cdot 2 \text{ mm}) \cdot 5025 \text{ mm}^2 = 201000 \text{ mm}^3$$

The surface/ volume ratio can be calculated from [equation \(2\)](#).

$$r = \frac{a}{V} \quad (2)$$

$$r = \frac{5025 \text{ mm}^2}{201000 \text{ mm}^3} = 0,025 \text{ mm}^{-1}$$

NOTE This the surface/ volume-ratio as requested in [clause 3](#): 1 l per 2,5 dm².

5.7 Siliconic gaskets, internal diameter 80 mm, external diameter min. 90 mm, thickness 2mm.

5.8 Silicon stoppers, diameter 10 mm, 20 mm length.

5.9 PTFE-thermo insulating plate, dimension min.: 105 mm × 105 mm, 2 mm thickness.

5.10 Blank samples plate made of borosilicate glass, according to ISO 3585 and dimensions min.: 105 mm × 105 mm, 2 mm thickness.

5.11 Flange frame, corrosion resistant steel.

5.12 Heating apparatus, able to support the migration temperature, e.g. $(95 \pm 2) ^\circ\text{C}$.

5.13 Beaker to preheat the test solution made of borosilicate glass, min. 600 ml volume. The Beaker is also used as a blank sample container when testing articles.

5.14 Boro-silicate glass lid, according to ISO 3585.

6 Samples

The laboratory sample shall consist of two similar single articles, identical in material, shape, dimensions and decoration. The sample may be specifically produced test plates or actual industrially produced articles.