# INTERNATIONAL STANDARD

ISO 10106

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### **Cork stoppers** — **Determination of global migration**

Bouchons en liège — Détermination de la migration globale

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#### **Foreword**

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see <a href="www.iso.org/directives">www.iso.org/directives</a>).

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This document was prepared by Technical Committee ISO/TC 87, Cork.

This second edition cancels and replaces the first edition (ISO/10106/2003). The main changes compared to the previous edition are that <u>Clauses 1</u>, <u>2</u>§ <u>Subclauses 6.1</u>9 <u>602</u>, <u>70lauses 8</u> and <u>10</u> have been technically revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at <a href="https://www.iso.org/members.html">www.iso.org/members.html</a>.

### **Cork stoppers** — **Determination of global migration**

#### 1 Scope

This document specifies a test method to measure the global migration of cork stoppers.

It is applicable to all types of cork stoppers that are ready to use, simulating the real conditions of use. This includes all kind of cork stoppers (completely or partially inserted in the bottle neck).

A bottle with an adequate finish is used to carry out the test.

#### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 633, Cork — Vocabulary

### 3 Terms and definitionSTANDARD PREVIEW

For the purposes of this document, the terms and definitions given in ISO 633 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform, available at https://www.iso.org/obp<sub>645-</sub>
- IEC Electropedia: available at <a href="http://www.electropedia.org/">http://www.electropedia.org/</a>

#### 3.1

#### global migration

mass of the non-volatile constituents of the cork stopper transferred to the simulator during the test

#### 3.2

#### simulator

solution intended to simulate the foodstuff

#### 4 Principle

Introduction of the cork stopper in the neck of a bottle containing the appropriate simulator. The contact cork stopper/simulator is carried out in specific conditions of time and temperature. After evaporation of the obtained solution, the global mass transferred to the simulator from the cork stopper is determined, by weighing.

#### 5 Reagents and materials

#### 5.1 Reagents

#### 5.1.1 Demineralized water.

#### **5.1.2 Ethanol grade**, minimum 96 %.

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- **5.1.3 Ethanol solution**, with a volume fraction of either 20 % or 50 % and with an alcoholic content corresponding to the alcoholic beverage bottled with the cork stoppers under test.
- **5.1.4 Acetic acid** p.a.
- **5.1.5 Acetic solution**, with a volume fraction of 3 %.
- **5.1.6 Other reagents to prepare simulant solution**, depending on the use of the cork stopper.
- 5.2 Materials
- **5.2.1 Bottles**, with adequate capacity, provided that the mouth finish is known and adequate with the use. When no reference is given, use a standardized bottle neck.
- **5.2.2** Cellulose filter paper, for qualitative analysis with a retention rate of 98 % for particles up to 11  $\mu$ m.
- **5.2.3 Watch glass** appropriate for contact with foodstuffs.
- **5.2.4 Evaporation flask of neutral glass**, with capacity 500 ml fitted to be adapted on a rotary evaporator without any kind of lubricant/grease, or a evaporation neutral glass cup with a capacity of 500 ml, resistant to a temperature of  $(85 \pm 5)$  °C to be adapted to other equipment (for example, a hotplate).
- **(standards.iteh.ai) 5.2.5 Capsule**, of neutral glass or any other inert material, resistant to temperatures of (103 ± 5) °C.

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6 Apparatus

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- **6.1 Corking machine with four jaws**. The compression ratio shall be adequate for the kind of cork stopper in use.
- 6.2 Thermostatic oven.
- **6.2.1** Thermostatic oven, maintained to  $(103 \pm 2)$  °C.
- **6.2.2** Thermostatic oven, maintained to  $(40 \pm 4)$  °C.
- **6.3 Rotative evaporator**, provided with a vacuum device or any other equipment that may assure that the simulator is evaporated at a temperature of  $(85 \pm 5)$  °C (for example, a hotplate or a water bath, or oven).
- **6.4 Balance**, with a resolution of 0,1 mg.
- **6.5 Desiccator**, with an appropriate desiccating agent.

#### 7 Preparation of test pieces

Take a laboratory sample of at least nine cork stoppers and take three test pieces, each one with three cork stoppers.

#### 8 Procedure

#### 8.1 Test conditions

The test shall be carried out at a temperature of  $(40 \pm 4)$  °C.

#### 8.2 Contact with the simulator

Fill up nine bottles (5.2.1), each with 100 ml of simulator and cork them with a corking machine (6.1).

Assure that the corking process is done correctly, accordingly to the type of cork stopper and normal use conditions.

Leave the simulator to settle for 60 min in the upright bottles, assuring that nothing modifies the contact surface between the cork stopper and the simulant.

Then, turn the bottles upside down and leave them in this position for 10 days, at the temperature specified in 8.1.

#### 8.3 Determination

Filter the content of three bottles and collect all the three filtrates in a 500 ml evaporation flask of neutral glass or evaporation neutral glass (5.2.4).

Using a rotative evaporator or a hotplate (6.3), evaporate these filtrates at a temperature of  $(85 \pm 5)$  °C (6.3), in such a way that the volume of the solution is reduced to approximately 10 ml.

Pour the volume of the solution into a capsule ( $\underline{5.2.5}$ ) previously weighed (mass  $m_0$ ). Carefully wash the evaporation flask of neutral glass or evaporation neutral glass ( $\underline{5.2.4}$ ) with approximately 5 ml of simulator and repeat the procedure twice  $\underline{SO}$  10106:2018 https://standards.iteh.ai/catalog/standards/sist/9700f8dc-44a3-4e90-a645-

Dry the content of each capsule in the oven (6.2) thermostatically regulated at (103  $\pm$  5) °C until complete evaporation. Afterwards, place the capsules in a desiccator (6.5) to cool for 30 min. Weigh each capsule and its content and register its mass ( $m_1$ ).

Repeat the operation of drying and cooling until a constant mass is reached, that is, until two consecutive weighings do not differ by more than 0,5 mg.

Repeat all the procedures mentioned in this subclause for the remaining series of bottles.

#### 8.4 Blank test

At the same time, carry out a blank determination, by replacing the "cork stopper/bottle" pair with a "watch glass /bottle" pair (see <u>5.2.3</u>), keeping the three bottles in a vertical position, head down, during the 10 days testing period.

#### 9 Calculation and expression of results

The mass, m, of the extraction residue in the ethanol solution, for each series of three cork stoppers expressed in milligrams per cork stopper and rounded off to 0,5 mg, is given by the expression:

$$m = \frac{(m_{\rm r1} - m_{\rm r0}) - (m_{\rm b1} - m_{\rm b0})}{3} \tag{1}$$

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#### where

- $m_{\rm r1}$  is the capsule and residue mass, after drying  $(m_1)$ , obtained from the determination with the cork stoppers, expressed in milligrams and rounded off to 0,1 mg;
- $m_{\rm r0}$  is the capsule mass  $(m_0)$ , expressed in milligrams and rounded off to 0.1 mg;
- $m_{\rm b1}$  is the mass  $(m_1)$  of the capsule and of the residue, after drying, obtained in the blank test, expressed in milligrams and rounded off to 0,1 mg;
- $m_{\rm h0}$  is the capsule mass  $(m_{\rm 0})$ , obtained in the blank test, expressed in milligrams and rounded off to 0.1 mg.

The final result of the test, m, is the arithmetic average of the results of each series of three cork stoppers, expressed in milligrams per cork stopper and rounded off to the unit.

#### 10 Test report

The test report shall contain the following indications:

- all the information necessary for the complete identification of the sample;
- b)

a reference to this document, i.e. ISO 10106:2018:

the sampling method; c)

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- the type and, if applicable, the alcoholic degree of the used simulator;
- e)

the mouth finish used; https://standards.iteh.ai/catalog/standards/sist/9700f8dc-44a3-4e90-a645-

- the obtained results: f)
- d867b33c21a1/iso-10106-2018
- any operational conditions not foreseen in this document; g)
- any accident that may have affected the results: h)
- type of the filter used for the test (5.2.2).

### **Bibliography**

- [1] EN 12726, Packaging Cork mouth finish with a bore diameter of 18,5 mm for corks and tamper evident capsules
- [2] NF H35-029, Industries de l'embouteillage Bouteilles en verre Bague couronnes verre champenoises 26 et 29
- [3] NF H35-027, Industries de l'embouteillage Bague Porto

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