
Cork stoppers — Determination of global migration

Bouchons en liège — Détermination de la migration globale

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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 10106:2018). The main changes compared to the previous edition are that [Clauses 1, 2, 6.1, 6.2, 8 and 10](#) 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 www.iso.org/members.html.

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 definitions

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>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1 global migration

mass of the non-volatile constituents of the cork stopper ceded to the simulant during the test

3.2 simulant

solution intended to simulate the foodstuff

4 Principle

Introduction of the cork stopper in the neck of a bottle containing the appropriate simulant. The contact cork stopper/simulant is carried out in specific conditions of time and temperature. After evaporation of the obtained solution, the global mass transferred to the simulant 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 with a volume fraction minimum of 96 %.

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5.1.3 Ethanol solution, with a volume fraction of either 20 % or 50 %, or 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 the 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 Microfibre glass filter or fritted glass plaque or cellulose filter paper, for qualitative analysis with a retention rate of 98 % for particles and a porosity up to 11 µm.

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 an evaporation neutral glass cup of 500 ml resistant to a temperature of (85 ± 5) °C to be adapted to other equipment (for example hotplate).

5.2.5 Capsule, of neutral glass or any other inert material resistant to temperatures of (103 ± 5) °C.

6 Apparatus

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6.1 Corking machine with four jaws. The compression ratio must be adequate to the kind of cork stopper in use.

6.2 Thermostatic oven.

6.2.1 Thermostatic oven, maintained to (103 ± 5) °C.

6.2.2 Thermostatic oven, maintained to (40 ± 4) °C.

6.3 Rotative evaporator, provided with a vacuum device or any other equipment that may ensure that the simulant is evaporated at a temperature of (85 ± 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 ± 4) °C.

8.2 Contact with the simulant

Fill up each of the nine bottles (5.2.1) with 100 ml of simulant 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 simulant 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 ± 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 (5.2.5) previously weighed (mass m_0). Carefully wash the evaporation flask of neutral glass or evaporation neutral glass (5.2.4) with approximately 5 ml of simulant and repeat the procedure twice.

Dry the content of each capsule in the oven (6.2) thermostatically regulated at (103 ± 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_i).

Repeat the operation of drying and cooling until a constant mass is reached, that is, until two consecutive weightings 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 5.2.3), keeping the three bottles in a vertical position, during the 10 days testing period.

9 Calculation and expression of results

The mass, m , of the extraction residue in the simulant, for each series of three cork stoppers expressed in milligrams per cork stopper and rounded off to 0,5 mg, is given by Formula (1):

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

where

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- m_{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_{r0} is the capsule mass, m_0 , expressed in milligrams and rounded off to 0,1 mg;
- m_{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_{b0} is the capsule mass, m_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:

- a) all the information necessary for the complete identification of the sample;
- b) reference to this International Standard, i.e. ISO 10106:2021;
- c) sampling method;
- d) the type and, if applicable, the alcoholic degree of the used simulant;
- e) the mouth finish used;
- f) the obtained results;
- g) any operational conditions not foreseen in this International Standard;
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- h) any accident that may have affected the results;
- i) type and porosity 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 — Bagues couronnes verre champenoises 26 et 29*
- [3] NF H35-027, *Industries de l'embouteillage — Bague Porto*

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