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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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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.

ISO 10106 was prepared by Technical Committee ISO/TC 87, *Cork*.

Cork stoppers — Determination of global migration

1 Scope

This International Standard 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.

To carry out the test, a bottle with an adequate bottle finish should be available.

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.

ISO 633, *Cork — Vocabulary*[1)]

3 Terms and definitions

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

3.1
global migration
mass of the non-volatile constituents of the cork stopper ceded 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 the bottle containing the appropriate simulator. The contact cork stopper/simulator is carried out in specific conditions of durability 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 Deionized water

1) Under revision.

5.1.2 Ethanol solution, at 10 %, or with an alcoholic content corresponding to the alcoholic beverage bottled with the cork stoppers under test, prepared by mixing ethanol and deionized water.

5.2 Materials

5.2.1 Bottles, of capacity 37,5 cl, for cork stoppers, with a standardized mouth finish (e.g. mouth finish as given in EN 12726[2)] for still wines, mouth finish CHAMPENOISE as given in NF H35-029[3)] for effervescent wines).

5.2.2 Microfibre glass filter or fritted glass plaque, with porosity 10 µm to 15 µm.

5.2.3 Aluminium film, coated with an extensible and auto-adherent film of plastic material, appropriate for contact with foodstuffs.

5.2.4 Round flask, of capacity 500 ml, of neutral glass for evaporation.

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

6 Apparatus

6.1 Corking machine, convenient for the cork stoppers under test.

6.2 Thermostatic oven, controlled to (103 ± 5) °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 ± 5) °C (for example, a hotplate or a water or oil bath).

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 simulator

Fill up each of the nine bottles (5.2.1) with 100 ml of simulator and cork them with a corking machine (6.1.1). Leave to settle for 60 min.

Dispose the bottles turned downwards, for 10 days, at the temperature specified in 8.1.

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2) EN 12726, Packaging — Cork mouth finish with a bore diameter of 18,5 mm for corks and tamper evident capsules.

3) NF H35-029, Industrie de l'embouteillage — Bouteilles en verre — Bagues couronnes verre champenoises 26 et 29.

8.3 Determination

Open each bottle and filter its content. Collect the filtrate of each three-bottle test piece in a 500 ml round flask (5.2.4).

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

Pour each solution into a glass capsule (5.2.5) previously weighed (mass m_0). Rinse each flask with approximately 5 ml of simulator and repeat two times.

Dry the content of each capsule in the oven (6.2) regulated at (103 ± 5) °C for 2 h and, after this period, place them in a desiccator (6.5) to cool for 30 min. Weigh each capsule and its content and register its mass (m_1).

Repeat the drying operation for 1 h, and the cooling operation for 30 min, until constant mass, that is, until two consecutive weighings do not differ by more than 0,5 mg.

8.4 Blank test

At the same time, carry out a blank determination, that is, the couple “cork stopper/bottle” is exchanged by a couple “aluminium film/bottle” (see 5.2.3).

The test conditions are those described in 8.1 to 8.3, except for the simulator volume used, which is 300 ml, and the bottle position, that is maintained standing.

9 Calculation and expression of results

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

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

where

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 result of the test 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. The individual results are also reported.

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;

c) sampling method;

d) the alcoholic degree of the used simulator;

e) the mouth finish used;

f) the obtained results;

g) any operational conditions not foreseen in this International Standard;

h) any accident that may have affected the results.

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