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Cork stoppers — Determination of oxidizing residues — Iodometric titration method

Bouchons en liège — Dosage des résidus oxydants — Méthode par titrage iodométrique

Reference number
ISO 21128:2006(E)

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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 21128 was prepared by Technical Committee ISO/TC 87, *Cork*.

Cork stoppers — Determination of oxidizing residues — Iodometric titration method

1 Scope

This International Standard specifies an iodometric titration method for determining the oxidizing residues released by cork stoppers ready to use.

NOTE This Standard concerns cork stoppers previously submitted to treatments with oxidizing products. It this is not the case, the result of the test method is probably negative.

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:—[1)], *Cork — Vocabulary*

3 Terms and definitions

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

3.1
oxidizing residues
residues of bleaching products on the cork stoppers

4 Principe

Titrimetric determination of iodine, formed by oxidation of the peroxide residues on the cork stoppers, by using thiosulfate in an acidic environment.

The chemical reaction is:

$$H_2O_2 + 2H^+ + 2I^- \Leftrightarrow I_2 + 2H_2O \quad (1)$$

$$2\ S_2O_3^{2-} + I_2 \Leftrightarrow S_4O_6^{2-} + 2I^- \quad (2)$$

5 Sampling

Take a gross sample, statistically representative of the cork stoppers to be characterized, by following a chosen sampling plan.

1) To be published. (Revision of ISO 633:1986)

6 Conditioning

Tests shall be carried out at a temperature of (21 ± 4) °C and a relative humidity of (60 ± 20) %.

7 Reagents and materials

Use only reagents of recognized analytical grade and distilled or demineralised water or water of equivalent purity.

7.1 Sulfuric acid (H_2SO_4), dilute, 25 %.

7.2 Potassium iodide solution (KI), 50 g/l.

7.3 Ammonium heptamolybdate solution, 3 %.

7.4 Sodium thiosulfate, standard solution, 0,02 N.

7.5 Iodine indicator or **fresh starch solution**, 0,1 % (m/V) (see Annex A).

7.6 Acetic acid solution, 50 %.

7.7 Nitrogen.

8 Apparatus

Usual laboratory apparatus and in particular, the following.

8.1 Volumetric pipettes, of capacity 1 ml, 5 ml and 25 ml.

8.2 Graduated burette, in 0,1 ml, class A.

8.3 Flasks with screw caps, of nominal capacity 250 ml and 500 ml.

8.4 Orbital stirrer with plate, adjustable to (150 ± 10) rev/min.

9 Procedure

Perform the test twice, on two groups, each one with 4 cork stoppers.

Add to a screwed flask (8.3), in order, 25 ml of potassium iodide solution (7.2), 5 ml of dilute sulfuric acid (7.1), 5 ml of acetic acid solution (7.6), 1 ml of ammonium heptamolybdate solution (7.3), and iodine indicator (7.5).

Introduce 4 cork stoppers into the flask.

Add 200 ml of demineralised water and allow nitrogen (7.7) to flow through the obtained solution, or remove O_2 by ultrasounds.

Close the flask and place it, for 2 h, on the plate of the orbital stirrer (8.4), protected from light.

If the solution colour changes into blue, then oxidizing residues are present. Remove cork stoppers and titrate with the sodium thiosulfate solution 0,02 N (7.4), stirring frequently, until a persistant turning point is obtained for at least 10 s. Near the turning point, add iodine indicator (7.5).

Report the volume of sodium thiosulfate 0,02 N used, expressed in millilitres.

If the solution colour does not change to blue, stop the test; that means that the solution does not contain any oxidizing residues.

At the same time a blank test shall be carried out, where cork stoppers are absent.

10 Calculation

The quantity of oxidizing residues for each group, expressed in milligrams of peroxide (H_2O_2) per stopper, shall be given by the formula:

$$\frac{V \times 0,02 \times 17}{4}$$

where V is the volume, in millilitres, of thiosulfate solution, rounded off to the nearest tenth, after subtracting the volume of thiosulfate required for the blank test.

If the concentration of each solution used is not the one mentioned in Clause 7, corrections should be introduced.

The test result is the greatest value obtained from the groups, expressed in milligrams of peroxide per stopper, rounded off to the nearest tenth.

11 Test report

The test report shall include at least the following information:

a) a reference to this International Standard, i.e. ISO 21128;

b) the complete identification of the product to be tested, including type, origin and manufacturer's reference;

c) the result obtained;

d) any operation not included in this International Standard, or regarded as optional;

e) any deviation from this International Standard, which may have changed the results.

Annex A
(informative)

Preparation of a starch solution, 0,1 % (m/V)

A.1 Reagents

A.1.1 Soluble starch, analytical grade.

A.1.2 Demineralised water, or any equivalent quality.

A.1.3 Sodium chloride, analytical grade.

A.2 Apparatus

A.2.1 Balance, with a resolution of 0,1 g.

A.2.2 Watch glass.

A.2.3 Graduated cylinder, of capacity 100 ml.

A.2.4 Mortar.

A.2.5 Laboratory flask, of capacity 250 ml.

A.2.6 Heating plate.

A.2.7 Funnel.

A.2.8 Whatman paper No.4 [2)], or similar filter paper.

A.2.9 Glass flasks.

A.3 Procedure

Weigh 0,1 g of soluble starch (A.1.1).

Carefully triturate with a few millilitres of cold demineralised water (A.1.2).

Pour the paste obtained into 100 ml of boiling water containing 5 g of sodium chloride (A.1.3).

Keep boiling for about 2 min until a persistent transparent solution is obtained.

Filter the hot solution using Whatman paper No. 4 (A.2.8), or let it cool, to allow excess starch to precipitate at the bottom of the flask and pour it off.

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2) Whatman is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

A.4 Stock

Keep the starch solution in an identified glass flask (A.2.9).

Starch solutions are an excellent environment to feed micro-organisms and therefore they quickly deteriorate. If the colour obtained during titration is brownish, instead of bluish, that means that the starch is spoiled and shall not be used as an indicator any longer.

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