BS ISO 20752:2014

BSI Standards Publication

Cork stoppers — Determination of releasable 2, 4, 6-trichloroanisol (TCA)

bsi.

...making excellence a habit.™

National foreword

This British Standard is the UK implementation of ISO 20752:2014. It supersedes BS ISO 20752:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/81, Cork.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Published by BSI Standards Limited 2014

ISBN 978 0 580 81959 9
ICS 79.100

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 June 2014.

Amendments/corrigenda issued since publication

Date	Text affected

BS ISO 20752:2014

INTERNATIONAL STANDARD

ISO 20752

Second edition
2014-06-15

Cork stoppers — Determination of releasable 2, 4, 6-trichloroanisol (TCA)

Bouchons en liège — Dosage du 2, 4, 6-trichloroanisol (TCA) relargable

Reference number
ISO 20752:2014(E)

COPYRIGHT PROTECTED DOCUMENT

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

Published in Switzerland

Contents

Page

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 WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 87, *Cork*.

This second edition cancels and replaces the first edition (ISO 20752:2007), which has been technically revised.

Introduction

This International Standard intends to simulate migration phenomena that might occur when cork stoppers are used to close wine bottles.

It consists of determining the content of releasable 2,4,6-trichloroanisol (TCA) based on an equilibrium between the solid (cork) and the liquid (hydro-alcoholic simulant) matrices after submitting a sample of cork stoppers or cork constituents of cork stoppers to a period of maceration in a hydro-alcoholic solution.

INTERNATIONAL STANDARD ISO 20752:2014(E)

Cork stoppers — Determination of releasable 2, 4, 6-trichloroanisol (TCA)

1 Scope

This International Standard specifies a test method to determine releasable 2,4,6-trichloroanisol (TCA) from cork stoppers.

This International Standard is applicable to all types of cork stoppers and all their cork constituents.

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 633, *Cork — Vocabulary*

ISO 17727, *Cork — Cork stoppers for still wine — Sampling plan for the quality control of cork stoppers*

3 Terms and definitions

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

3.1
simulant
solution that intends to simulate the wine

3.2
internal standard
compound of known concentration added to a sample to facilitate the qualitative identification and/or quantitative determination of the sample components

4 Symbols and abbreviated terms

PDMS	polydimethylsiloxane
GC/ECD	gas chromatography/electron capture detector
GC-MS	gas chromatography/mass spectrometry
SPME	solid phase microextraction
TCA	2,4,6-trichloroanisol

5 Principle

Determination of releasable TCA from cork stoppers, previously subjected to maceration in a wine simulant, using solid-phase microextraction followed by the detection and quantification of this compound by GC-MS or GC/ECD.

6 Reagents

Use only reagents of recognized analytical grade.

6.1 Hydro-alcoholic solution, 12 % (v/v) (wine simulant).

6.2 Ethanol, with a minimum purity of 95 %.

6.3 Distilled water.

6.4 Sodium chloride (NaCl) p.a.

6.5 Internal standard, (GC-MS) 2,4,6-trichloroanisol (TCA) d_5, purity ≥98 % or (GC/ECD) 2,3,6–trichloroanisol, purity ≥99 %.

6.6 2,4,6-trichloroanisol (TCA), purity ≥99 %.

7 Apparatus

The usual laboratory apparatus and, in particular, the following:

7.1 Balance, with a resolution of, at least, 0,1 mg.

7.2 Glass maceration flasks, with a stopper made of glass, metal, or any other material not absorbing TCA and an appropriate capacity to the sample size.

7.3 Glass flasks (vial), with 10 ml minimum capacity (solution shall occupy at least 50 % of the vial capacity) with a septum and an appropriate stopper.

7.4 SPME fibre.

EXAMPLE 100 µm PDMS.

7.5 Heating source, for the vial (7.3), set between 30 °C and 50 °C.

7.6 Automatic stirring system, for the SPME.

7.7 Appropriate gas, of chromatographic purity.

7.8 Gas chromatograph, with a mass detector, MS, or an electron capture detector, ECD.

7.9 Low-polarity fused silica capillary column.

EXAMPLE Length of 30 m, internal diameter of 0,25 mm, film thickness of 0,25 µm; stationary phase: copolymer of 5 % diphenyl and 95 % dimethyl polysiloxane.

8 Sampling

The size of the sample to be tested shall be in accordance with the sampling standard (ISO 17727) or agreed between customer and supplier.

9 Procedure

9.1 Calibration

The set of calibration solutions for TCA is obtained by adding known concentrations of analyte to the wine simulant. Standard solutions from 0,5 ng/L to 10 ng/L can be used.

The calibration curve obtained shall be evaluated regularly and whenever any large change is recorded by the GC-MS or GC/ECD.

9.2 Sample preparation

Intact straight cork stoppers are subjected to maceration for 24 h ± 2 h at room temperature, using enough wine simulant, to keep the stoppers entirely immersed, and then analyse the macerate.

EXAMPLE 1 20 corks (45 × 24) mm, in a 1 L flask.

EXAMPLE 2 50 stoppers (45 × 24) mm, in a 2 L flask.

For bar-top stoppers, cut off the flange before maceration.

For granulated cork, 40 g of granulated cork are subjected to maceration for 24 h ± 2 h at room temperature in a 2 L flask, using enough wine simulant to keep the granulated cork entirely immersed, and then analyse the macerate.

For cork discs, 20 cork discs are subjected to maceration for 24 h ± 2 h at room temperature in a 0,5 L flask, using enough wine simulant to keep the granulated cork entirely immersed, and then analyse the macerate.

For cork stoppers for sparkling wines, immerse in the wine simulant only the discs of natural cork and 1 cm of the agglomerate without cutting the stoppers, for 24 h ± 2 h at room temperature, and then analyse the macerate.

9.3 SPME

9.3.1 Test portion

Take a volume of the solution to be tested not greater than 50 % of the vial capacity (7.3).

EXAMPLE 1 For a vial (7.3) of 20 mL, take 10 mL, in such a way that the fibre does not touch the liquid.

The test portion should be saturated with NaCl (approximately 0,30 g/mL).

Add the internal standard solution, 12 %, v/v ethanol/water, to the test portion in the vial (7.3) in such a way that the ethanol content and the total volume of the liquid do not change significantly.

EXAMPLE 2 100 µL internal standard solution/10 mL total liquid.

The concentration of the internal standard solution shall be in the calibration curve range and shall be as close as possible to the specification.

9.3.2 Adsorption

Adjust the time and temperature in the headspace, according to the fibre used.

EXAMPLE Fibre PDMS 100 µm, 15 min in the headspace, stirring at a temperature of 35 °C ± 2 °C.

9.4 GC

9.4.1 Desorption

Splitless injection from 1 min to 2 min at 250 °C. The fibre shall be conditioned/cleaned between two extractions.

9.4.2 Chromatographic analysis

The resolution grade and the time needed for the chromatographic separation shall be optimized by using a temperature program.

An example of a temperature program is shown below.

Temperature	Time	Increment
50 °C	2 min	—
From 50 °C to 150 °C	—	9 °C/min
150 °C	0,5 min	—
From 150 °C to 260 °C	—	20 °C/min
260 °C	5 min	—

9.5 Detection

9.5.1 Determination by MS

Detection either in SIM or in MS/MS mode with detection of 3 ions, and quantification through the most abundant ion.

Ions are the following:

— for 2,4,6 TCA: m/z 195, 210, 212, quantification with m/z 195;

— for 2,4,6 TCA d_5: m/z 199, 215, 217, quantification with m/z 215.

The operating conditions in MS/MS mode are shown in Table 1.

Table 1 — Operating conditions in MS/MS mode

Compound	Precursor ion	Product ion	
2,4,6 TCA d_5	217	199	171
2,4,6 TCA	212	197	169

9.5.2 Determination by ECD

Identify the peaks corresponding to analyte and to the internal standard by comparing, on the chromatogram, the retention time in the sample with the retention time in the standard peak.

9.6 Blank test

Carry out a parallel test, without the stoppers mentioned in 9.2.

10 Expression of results

The results shall be expressed in nanogram per litre and shall be rounded to the nearest to 0,5.

11 Test report

The test report shall include the following information:

a) number of stoppers tested and capacity of the maceration flask used;

b) reference to this International Standard (i.e. ISO 20752);

c) all data needed to identify completely the tested sample;

d) capillary column used, gas chromatography conditions applied, and type of detection used;

e) internal standard used;

f) concentration of releasable 2,4,6 TCA, in nanogram per litre;

g) detection limit and quantification limit;

h) any incident likely to have affected the results, noted during the analysis;

i) all optional operations or those not specified in this International Standard.

Bibliography

[1] ISO 2718, *Standard layout for a method of chemical analysis by gas chromatography*

[2] ISO/IEC 17043, *Conformity assessment — General requirements for proficiency testing*

[3] NF V 03-110*Décembre 1998, Analyse des produits agricoles et alimentaires - Procédure de validation intralaboratoire d'une méthode alternative par rapport à une méthode de référence - Cas de méthodes d'analyse quantitatives*

[4] G-CSQ-02, Guia para los laboratorios que realizan validaciones de métodos de análisis químicos

[5] Relacre Guia 13, Fevereiro 2000, Validação de métodos internos de ensaio em análise química

[6] *IUPAC Compendium of Chemical Terminology*. Second Edition, 1997

ICS 79.100
Price based on 6 pages

This page deliberately left blank

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards -based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email bsmusales@bsigroup.com.

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Copyright

Useful Contacts:

Customer Services
Tel: +44 845 086 9001
Email (orders): orders@bsigroup.com
Email (enquiries): cservices@bsigroup.com

Subscriptions
Tel: +44 845 086 9001
Email: subscriptions@bsigroup.com

Knowledge Centre
Tel: +44 20 8996 7004
Email: knowledgecentre@bsigroup.com

Copyright & Licensing
Tel: +44 20 8996 7070
Email: copyright@bsigroup.com

bsi.

...making excellence a habit.™