

Animal and vegetable fats and oils - Determination of the content of trans fatty acid isomers of vegetable fats and oils - Gas chromatographic method (ISO 15304:2002)

Tierische und pflanzliche Fette und Öle - Bestimmung des Gehaltes an trans-Fettsäure-Isomeren in pflanzlichen Fetten und Ölen - Gaschromatographisches Verfahren (ISO 15304:2002)

Corps gras d'origines animale et végétale - Détermination de la teneur en isomeres trans d'acides gras de corps gras d'origine vegetale - Methode par chromatographie en phase gazeuse (ISO 15304:2002)

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Ta slovenski standard je istoveten z: EN ISO 15304:2002

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EUROPEAN STANDARD
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English version

**Animal and vegetable fats and oils - Determination of the
content of trans fatty acid isomers of vegetable fats and oils -
Gas chromatographic method (ISO 15304:2002)**

Corps gras d'origines animale et végétale - Détermination
de la teneur en isomères trans d'acides gras de corps gras
d'origine végétale - Méthode par chromatographie en phase
gazeuse (ISO 15304:2002)

Tierische und pflanzliche Fette und Öle - Bestimmung des
Gehaltes an trans-Fettsäure-Isomeren in pflanzlichen
Fetten und Ölen - Gaschromatographisches Verfahren
(ISO 15304:2002)

This European Standard was approved by CEN on 8 March 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

EN ISO 15304:2002 (E)

CORRECTED 2002-04-17

Foreword

This document (ISO 15304:2002) has been prepared by Technical Committee ISO/TC 34 "Agricultural food products" in collaboration with Technical Committee CEN/TC 307 "Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2002, and conflicting national standards shall be withdrawn at the latest by September 2002.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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Endorsement notice

The text of the International Standard ISO 15304:2002 has been approved by CEN as a European Standard without any modifications.

NOTE Normative references to International Standards are listed in annex ZA (normative).

Annex ZA
(normative)

**Normative references to international publications
with their relevant European publications**

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

NOTE Where an International Publication has been modified by common modifications, indicated by (mod.), the relevant EN/HD applies.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN</u>	<u>Year</u>
ISO 661	1989	Animal and vegetable fats and oils — Preparation of test sample	EN ISO 661	1995
ISO 5509	2000	Animal and vegetable fats and oils — Preparation of methyl esters of fatty acids.	EN ISO 5509	2000

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INTERNATIONAL STANDARD

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2002-03-15

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2003-05-15

Animal and vegetable fats and oils — Determination of the content of *trans* fatty acid isomers of vegetable fats and oils — Gas chromatographic method

*Corps gras d'origines animale et végétale — Détermination de la teneur en
isomères trans d'acides gras de corps gras d'origine végétale — Méthode
par chromatographie en phase gazeuse*
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Contents

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Reagents and materials	2
6 Apparatus	2
7 Sampling	3
8 Preparation of test sample	3
9 Preparation of methyl esters	3
10 Procedure	3
11 Calculations	5
12 Precision	7
13 Test report	7
Annex A (informative) Optimum conditions	8
Annex B (informative) Examples of typical chromatograms obtained under the recommended conditions	11
Annex C (informative) Equivalent chain length (ECL) values	16
Annex D (informative) FID response factor and FID correction factor	17
Annex E (informative) Results of interlaboratory test	18
Bibliography	20

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ISO 15304:2002(E)

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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15304 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

Annexes A to E of this International Standard are for information only.

In this corrected version, the identification of the main C18:2 *cis* isomer peak in Figure B.2 (the central peak in the figure) has been corrected from

C18:1 12*cis*

to

C18:2 9*cis*, 12*cis*

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Animal and vegetable fats and oils — Determination of the content of *trans* fatty acid isomers of vegetable fats and oils — Gas chromatographic method

1 Scope

This International Standard specifies a gas chromatographic method using capillary columns for the determination of the content of *trans* fatty acid isomers of vegetable oils and fats.

The method is specially designed to evaluate, by a single capillary gas chromatographic (GC) procedure, the level of *trans* isomers as formed during (high temperature) refining, or during hydrogenation of vegetable oils or fats.

The method may also be used to report all other fatty acids (e.g. to obtain the full fatty acid composition and total amounts of saturated fatty acids, mono-unsaturated fatty acids and poly-unsaturated fatty acids) from the same sample and same analysis.

NOTE 1 The *trans*-isomer content as obtained with this method may not agree with the *trans*-isomer content as obtained using other methods.

NOTE 2 During (high temperature) refining (deacidification and deodorization), only geometrical isomers are formed of the mono- and poly-unsaturated fatty acids; i.e. the double bond(s) remain(s) at the same natural position. During hydrogenation, both positional and geometrical isomers are formed.

NOTE 3 For some specific *cis*- and *trans*-isomers formed during hydrogenation, co-elution is possible. This could influence the accuracy of the result. The level of these isomers is usually negligible in normal partially hydrogenated oils and fats.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 661, *Animal and vegetable fats and oils — Preparation of test sample*

ISO 5509, *Animal and vegetable fats and oils — Preparation of methyl esters of fatty acids*

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

3.1

content of *trans* fatty acid isomers of (high temperature) refined oils and fats

sum of the C18:1 *trans*, C18:2 *trans* and C18:3 *trans* fatty acid methyl esters, expressed as a mass fraction of all fatty acid methyl esters

ISO 15304:2002(E)

3.2

content of *trans* fatty acid isomers of partially hydrogenated oils and fats

sum of all *trans* double-bond-containing fatty acid methyl esters, expressed as a mass fraction of all fatty acid methyl esters

NOTE The content of *trans* fatty acid isomers is expressed in percent.

4 Principle

The methylated fatty acids of the sample are separated on a capillary gas chromatography column with a high polar stationary phase, with respect to their chain length, degree of (un)saturation and geometry and position of the double bonds.

5 Reagents and materials

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Carrier gas, preferentially helium or hydrogen, or otherwise nitrogen, of gas chromatographic quality, dried and with oxygen removed by suitable filters.

WARNING — Hydrogen, which is used only with capillary columns, can double the speed of the analysis (in comparison with helium) but is hazardous. Safety devices are available and it is essential that a suitable device be incorporated into the apparatus.

5.2 Certified Reference Material (CRM), BCR 162 (soya/maize blend), European Commission, Community Bureau of Reference.¹⁾

NOTE In addition to the use of CRM from the EC, the use of other calibration standards from reputable suppliers such as Supelco, Larodan, Nuchek and Sigma may be accepted. Perhaps different standards will be necessary for different hydrogenated oils (e.g. lauric, non-lauric oils and palm oil).

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Gas chromatograph, equipped with a capillary injection system (preferred split mode, operated at a split ratio of approximately 1:100) and flame ionization detector (FID).

6.2 Capillary column, with a high polar stationary phase (e.g. CPTM-Sil 88²⁾, SP-2340³⁾, BPX-70⁴⁾ or similar highly polar cyanopropyl phases such as SP-2380 and SP-2560 which can give similar resolution of the various geometrical isomers).

NOTE For improved separations, a 100 m SP-2560 or CPTM-Sil 88 column and hydrogen as carrier gas are recommended.

1) European Commission, Joint Research Centre, Institute for Reference Materials and Measurements (IRMM), Geel, Belgium.

2) Available from Chrompack, Middelburg, The Netherlands.

3) Available from Supelco, Bellefonte, PA, USA.

4) Available from SGE Inc., Austin, Texas, USA.

These types of columns are examples of suitable products which are available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.