BS EN ISO 6885:2016

BSI Standards Publication

Animal and vegetable fats and oils — Determination of anisidine value

bsi.

...making excellence a habit.™

National foreword

This British Standard is the UK implementation of EN ISO 6885:2016. It supersedes BS EN ISO 6885:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee AW/307, Oilseeds, animal and vegetable fats and oils and their by-products.

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

Published by BSI Standards Limited 2016

ISBN 978 0 580 91531 4

ICS 67.200.10

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 31 March 2016.

Amendments issued since publication

Date	Text affected

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 6885

March 2016

ICS 67.200.10

Supersedes EN ISO 6885:2007

English Version

Animal and vegetable fats and oils - Determination of anisidine value (ISO 6885:2016)

Corps gras d'origines animale et végétale - Détermination de l'indice d'anisidine (ISO 6885:2016)

Tierische und pflanzliche Fette und Öle - Bestimmung der Anisidinzahl (ISO 6885:2016)

This European Standard was approved by CEN on 30 January 2016.

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 CEN-CENELEC 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 CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN ISO 6885:2016 E

European foreword

This document (EN ISO 6885:2016) has been prepared by Technical Committee ISO/TC 34 “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 2016, and conflicting national standards shall be withdrawn at the latest by September 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN ISO 6885:2007.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Endorsement notice

The text of ISO 6885:2016 has been approved by CEN as EN ISO 6885:2016 without any modification.

Contents

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 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This fourth edition cancels and replaces the third edition (ISO 6885:2006), which has been technically revised by adding a sentence to the Scope and deleting a column in Table A.1.

Animal and vegetable fats and oils — Determination of anisidine value

1 Scope

This International Standard specifies a method for the determination of the anisidine value in animal and vegetable fats and oils. This is a measure of the amount of aldehydes present (principally α, β-unsaturated aldehydes).

Milk and milk products (or fat coming from milk and milk products) are excluded from the scope of this International Standard.

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

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

3.1
anisidine value
one hundred times the increase in absorbance, measured at a wavelength of 350 nm in a 10 mm cell, of a test solution when reacted with *p*-anisidine under the test conditions specified in this International Standard

Note 1 to entry: The anisidine value has no dimensions, and is calculated and quoted on the basis of 1 g of the test sample in 100 ml of a mixture of solvent and reagent.

4 Principle

A test solution is prepared in isooctane (2,2,4-trimethylpentane). It is reacted with an acetic acid solution of *p*-anisidine. The increase in absorbance at 350 nm is measured. The anisidine value is calculated.

5 Reagents

Use only reagents of recognized analytical grade, and water complying with grade 3 of ISO 3696.

5.1 Sodium sulfate (Na_2SO_4), anhydrous.

5.2 Isooctane (2,2,4-trimethylpentane), having an absorbance not exceeding 0,01 against water in the wavelength range 300 nm to 380 nm.

5.3 4-Methoxyaniline (*p*-anisidine), anhydrous cream-coloured crystals.

WARNING — *p*-anisidine is toxic and care shall be taken to avoid contact with the skin.

Store the *p*-anisidine in a dark bottle at 0 °C to 4 °C in the dark.

No coloration (grey or pink) shall be observed. If this is present, purify the *p*-anisidine as follows.

Dissolve 4 g of *p*-anisidine in 100 ml of water at 75 °C. Add 0,5 g of sodium sulfite (Na_2SO_3) and 2 g of charcoal. Stir for 5 min and filter through a medium retention filter paper to give a clear solution. Cool the filtrate to 0 °C and leave at this temperature for at least 4 h. Filter off the crystals, preferably under vacuum, and wash with a small volume of water at about 0 °C. Dry in a vacuum desiccator containing an efficient desiccant.

5.4 **Glacial acetic acid**, of water content not greater than 0,1 % (mass fraction).

5.5 Anisidine reagent.

On the day of use, prepare the minimum quantity of reagent required for the analysis, in view of its toxicity and limited life. Prepare, for example, 50 ml of reagent as follows.

Dissolve 0,125 g of the *p*-anisidine (5.3) in the glacial acetic acid (5.4) in a 50 ml volumetric flask and dilute to the mark with the same solvent, avoiding exposure to strong light.

Check the absorbance against isooctane before use and discard the reagent when the difference is larger than 0,2. In any case, discard any reagent left over on the day of use.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 **Spectrometer**, double- or single-beam, suitable for use at a wavelength of 350 nm, with cells of optical path length 10 mm.

When a double-beam spectrometer is used, it is recommended that a pair of matched 10 mm cells be used.

6.2 **Volumetric flasks**, of 25 ml capacity.

6.3 **Test tubes**, of 10 ml capacity, fitted with ground glass stoppers.

6.4 **Pipettes**, of 1 ml and 5 ml capacities, equipped with a safety suction device.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555.

8 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

If the moisture content of the sample is greater than 0,10 % (mass fraction), it should be dried using the following procedure.

Add sodium sulfate (5.1) in the proportion of 1 g to 2 g per 10 g of the thoroughly mixed sample, at a temperature of not more than 10 °C above the melting point in the case of a solid fat. Stir thoroughly and filter, maintaining the temperature to prevent solidification.

Take care to exclude extraneous moisture during the procedure because it can affect the equilibrium reaction during which water is produced.

9 Procedure

9.1 Test portion and preparation of test solution

Weigh, to the nearest 1 mg, a sufficient mass of the prepared test sample (Clause 8) directly into a 25 ml volumetric flask. Preheat solid samples to 10 °C above their melting point. Dissolve the sample in 5 ml to 10 ml of the isooctane (5.2) and make up to the mark with the same solvent.

The size of the test portion depends on the quality of the sample and the characteristics of the spectrometer used, and should be chosen to avoid readings near the upper and lower ends of the scale. In general, 0,4 g to 4,0 g is used.

9.2 Unreacted test solution

By means of a pipette (6.4), transfer 5 ml of the test solution (9.1) to a test tube (6.3). Add 1 ml of glacial acetic acid (5.4), stopper the tube and shake well. Keep the test tube in the dark at (23 ± 3) °C for 8 min.

Within a further 2 min, transfer the solutions to a clean, dry spectrometer cell. After a total reaction time of 10 min ± 1 min, follow the procedure specified in 9.5.

9.3 Reacted test solution

Transfer, by means of a pipette (6.4), 5 ml of the test solution (9.1) to a test tube (6.3). Add, by means of a pipette (6.4), 1 ml of the anisidine reagent (5.5). Stopper the tube and shake well. Keep the test tube in the dark at (23 ± 3) °C for 8 min.

Within a further 2 min, transfer the solutions to a clean, dry spectrometer cell. After a total reaction time of 10 min ± 1 min from the addition of the anisidine reagent, follow the procedure specified in 9.5.

9.4 Blank

Transfer, by means of a pipette (6.4), 5 ml of isooctane (5.2) to a test tube (6.3). Add, by means of a pipette (6.4), 1 ml of the anisidine reagent (5.5). Stopper the tube and shake well. Keep the test tube in the dark at (23 ± 3) °C for 8 min.

Within a further 2 min, transfer the solutions to a clean, dry spectrometer cell. After a total reaction time of 10 min ± 1 min from the addition of the anisidine reagent, follow the procedure specified in 9.5.

9.5 Spectrometric measurement

Adjust the zero absorption of the spectrometer with isooctane (5.2) at 350 nm.

Measure the following absorbances against isooctane (5.2):

- A_1 of the reacted solution (9.3),
- A_0 of the unreacted test solution (9.2), and
- A_2 of the blank (9.4).

9.6 Absorbance range

If the measured absorbance A_1 of the reacted solution (9.3) is not in the range 0,2 to 0,8, repeat the determination (9.2 to 9.4) with an adjusted amount of test sample.

If the measured absorbance A_2 of the blank exceeds 0,2, purify the anisidine reagent as described in 5.3, and prepare fresh anisidine reagent (5.5). Repeat this test with the fresh anisidine reagent.

10 Expression of results

10.1 The anisidine value (AV) of the sample is equal to Formula (1):

$$\mathrm{AV} = \frac{100QV}{m}[1{,}2(A_1 - A_2 - A_0)] \tag{1}$$

where

V is the volume in which the test sample is dissolved, in millilitres ($V = 25$ ml);

m is the mass of the test portion, in grams;

Q is the sample content of the measured solution based on which the anisidine value is expressed, in grams per millilitre ($Q = 0{,}01$ g/ml);

A_0 is the absorbance of the unreacted test solution (9.2);

A_1 is the absorbance of the reacted solution (9.3);

A_2 is the absorbance of the blank (9.4);

1,2 is the correction factor for the dilution of the test solution with 1 ml of the reagent or glacial acetic acid.

Report the results to 1 decimal place.

10.2 When assessing the oxidative deterioration of an oil, the total oxidation value, or ‘totox value’ (TV), may be helpful. The calculation is shown in Formula (2) [with the peroxide value (PV) expressed in meq O_2/kg]:

$$\mathrm{TV} = (2 \times \mathrm{PV}) + \mathrm{AV} \tag{2}$$

11 Precision

11.1 Interlaboratory tests

Details of two interlaboratory tests on the precision of the method are summarized in Annex A. The values derived from these interlaboratory tests may not be applicable to ranges and matrices other than those given.

11.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases exceed the value of r given in Table 1.

11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in two different laboratories with different operators using different equipment, will in not more than 5 % of cases exceed the value of R given in Table 1.

Table 1 — Repeatability limit (r) and reproducibility limit (R)

Anisidine value	Range of variation	r	R
AV (mean of two determinations)	0 to 100	0,034 AV + 0,31	0,19 AV + 1,41

12 Test report

The test report shall specify:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method, with reference to this International Standard, i.e. ISO 6885;

d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the results;

e) the test results obtained;

f) if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Results of the interlaboratory test

An interlaboratory test carried out at international level in 2004 by ITERG (France), in which 18 laboratories participated (9 countries: Argentina, Canada, France, Germany, Hungary, Netherlands, Portugal, UK, USA), each performing 2 determinations on each sample, gave the statistical results (determined in accordance with ISO 5725-2) given in Table A.1.

Table A.1 — Results of the interlaboratory test

Sample	Soya oil	Crude canola oil	Walnut oil	Fish oil	Fish oil	Poultry fat	Used frying oil
Number of laboratories retained after eliminating outliers	16	17	17	16	17	15	16
Mean	3,46	0,95	6,86	25,46	31,54	4,59	96,80
Standard deviation of repeatability, s_r	0,09	0,08	0,17	0,31	0,72	0,28	1,22
Coefficient of variation of repeatability, %	2,6	8,2	2,5	1,2	2,3	6,0	1,3
Repeatability limit, r ($2{,}8 \times s_r$)	0,25	0,22	0,48	0,86	2,02	0,78	3,43
Standard deviation of reproducibility, s_R	0,27	0,39	0,52	1,75	3,79	1,80	6,74
Coefficient of variation of reproducibility, %	7,9	41,4	7,6	6,9	12,0	39,3	7,0
Reproducibility limit, R ($2{,}8 \times s_R$)	0,77	1,10	1,46	4,89	10,61	5,05	18,86

Bibliography

[1] ISO 5555, *Animal and vegetable fats and oils — Sampling*

[2] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[3] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[4] IUPAC 2.504, *Determination of the p-anisidine value (p-A.V.)*

This page deliberately left blank

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.

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

All the data, software and documentation set out in all British Standards and other BSI publications are the property of and copyrighted by BSI, or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI. Details and advice can be obtained from the Copyright & Licensing Department.

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 Group Headquarters

389 Chiswick High Road London W4 4AL UK