BS ISO 18301:2014

BSI Standards Publication

Animal and vegetable fats and oils — Determination of conventional mass per volume (litre weight in air) — Oscillating U-tube method

bsi.

...making excellence a habit.™

National foreword

This British Standard is the UK implementation of ISO 18301:2014.

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.

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

ISBN 978 0 580 80493 9

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 30 September 2014.

Amendments issued since publication

Date	Text affected

BS ISO 18301:2014

INTERNATIONAL STANDARD

ISO 18301

First edition
2014-09-01

Animal and vegetable fats and oils — Determination of conventional mass per volume (litre weight in air) — Oscillating U-tube method

Corps gras d'origines animale et végétale — Détermination de la masse volumique conventionnelle (poids du litre dans l'air) — Méthode du tube en U oscillant

Reference number
ISO 18301:2014(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 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

Introduction

The conventional mass per volume ("litre weight in air", sometimes called "apparent density" or "conventional density") is an important parameter for the shipping of oils and fats. It is used to convert the dipped volume of oil in a tank into the mass of oil in the tank and is thus usually measured at loading and discharge of a ship. The manual method (see ISO 6883) uses a pyknometer; a method which requires a skilled technician to perform it correctly. The automatic method is simpler to carry out and temperature control might also be easier.

INTERNATIONAL STANDARD **ISO 18301:2014(E)**

Animal and vegetable fats and oils — Determination of conventional mass per volume (litre weight in air) — Oscillating U-tube method

1 Scope

This International Standard specifies a method for the determination of the conventional mass per volume of vegetable and animal oils and fats within the range of 0,800 kg/l to 1,000 kg/l which are in a single-phase liquid state at the test temperature.

This method is not intended for use in calibrating online density meters.

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*

3 Terms and definitions

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

3.1
conventional mass per volume
litre weight in air
mass of the substance, divided by its volume, measured in air

Note 1 to entry: Mass is expressed in kilograms per litre while volume is expressed in litres.

3.2
reference temperature
temperature at which the sample "litre weight" shall be reported

3.3
calibration
set of operations that establishes the relationship between the reference "litre weight in air" of standards and the corresponding "litre weight in air" reading of the instrument

4 Principle

A small portion (typically 1 ml) of the test sample is introduced into a temperature-controlled sample cell. The oscillation frequency is noted and the "litre weight" of the test sample is calculated using cell constants previously determined by measuring the oscillation frequencies when the cell is filled with calibration media of known litre weight.

5 Apparatus

5.1 Digital density meter, capable, once calibrated, of determining "litre weight" with a resolution of ±0,000 1 kg/l or better.

A heated injection device is recommended if samples that are solid at room temperature need to be analyzed.

5.2 Circulating constant-temperature bath, if required (see 9.1.1), capable of maintaining the temperature of the circulating liquid to within ±0,05 °C of the required temperature.

5.3 Calibrated temperature sensor, capable of measuring the temperature of the cell to an accuracy of at least 0,1 °C.

6 Reagents

WARNING — The use of this International Standard might involve hazardous materials, operations, and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

Unless otherwise stated, use only reagents of recognized analytical grade.

6.1 Flushing solvent, any solvent may be used provided it is capable of producing a dry cell and thus acetone is preferred, followed by drying with dry air.

6.2 Calibration media, one or two calibration media are needed to calibrate the cell.

They shall be chosen so that their "litre weights" are close to the litre weight of the sample under test. The litre weight of the calibration media shall be traceable to recognized national standards or based on internationally accepted values.

When water is used, the requirements of 6.3 shall be met.

6.3 Water, conforming to ISO 3696 grade 2 or better.

Prior to use, pass the water through a 0,45 µm filter and remove dissolved air by first boiling and then cooling. Once de-aerated, handle the water carefully so as to minimize the amount of air re-dissolved. The "litre weight in air" of water at temperatures from 15 °C to 65 °C is shown in Table 1.

7 Sampling

It is essential that the portion of the sample to be tested is representative of the bulk sample and sample mixing can sometimes be necessary to ensure homogenization prior to subsampling.

8 Preparation of the test sample and test portion

Prepare the sample in accordance with the method given in ISO 661.

Samples shall be heated to at least 10 °C above their melting points before being introduced into the instrument. Oils and fats which are sufficiently mobile should be mixed by gentle agitation, avoiding the incorporation of air.

Table 1 — Conventional mass per volume ("litre weight in air") of water at temperatures from 15 °C to 65 °C

Temperature θ °C	"Litre weight in air" ρ_w g/ml	Temperature θ °C	"Litre weight in air" ρ_w g/ml	Temperature θ °C	"Litre weight in air" ρ_w g/ml
15	0,998 05	35	0,992 98	55	0,984 65
16	0,997 89	36	0,992 64	56	0,984 16
17	0,997 72	37	0,992 28	57	0,983 67
18	0,997 54	38	0,991 92	58	0,983 17
19	0,997 35	39	0,991 55	59	0,982 67
20	0,997 15	40	0,991 17	60	0,982 17
21	0,996 94	41	0,990 79	61	0,981 65
22	0,996 72	42	0,990 39	62	0,981 13
23	0,996 49	43	0,989 99	63	0,980 60
24	0,996 24	44	0,989 58	64	0,980 06
25	0,995 99	45	0,989 17	65	0,979 52
26	0,995 73	46	0,988 74	-	-
27	0,995 46	47	0,988 32	-	-
28	0,995 18	48	0,987 88	-	-
29	0,994 90	49	0,987 44	-	-
30	0,994 60	50	0,986 99	-	-
31	0,994 29	51	0,986 54	-	-
32	0,993 98	52	0,986 07	-	-
33	0,993 65	53	0,985 61	-	-
34	0,993 32	54	0,985 13	-	-

9 Procedure

9.1 Apparatus preparation

9.1.1 Test temperature

The sample "litre weight" shall, whenever possible, be determined at the reference temperature.

If the density meter is fitted with an integral thermostat, set the cell temperature according to the manufacturer's instructions. Otherwise, connect it to the constant-temperature bath. Allow the temperature to stabilize. The manufacturer's specified working temperature and pressure ranges for the density meter cell shall not be exceeded. When temperature controlled baths are used, ensure that the circulating liquid remains clean.

9.1.2 Cell cleaning

Clean and dry the cell using the flushing solvent (6.1) and, if necessary, water (6.3), followed by a water-miscible solvent (6.1) and blow dry with dry air.

9.2 Apparatus calibration

9.2.1 Density meters shall be calibrated when first installed, or when maintenance has been carried out or the system has been disturbed in any other way. The density meter calibration shall be verified within a period of not more than seven days prior to use.

9.2.2 Perform the calibration in accordance with the manufacturer's instructions. Introduce the first calibration medium (6.2) into the cell and allow the cell and its contents to reach temperature equilibrium. Record the oscillation period or the "litre weight in air" reading and the temperature of the cell. Clean the cell in accordance with the procedure given in 9.1.2.

9.2.3 Introduce the second calibration medium (6.2) into the cell and allow the cell and its contents to reach temperature equilibrium. Record the oscillation period or the "litre weight in air" reading and the temperature of the cell.

9.2.4 The cell constants are calculated automatically by the apparatus.

9.2.5 After calibration, clean and dry the cell in accordance with the procedure given in 9.1.2

9.3 Measurement

9.3.1 Check that the density meter reading when the cell is filled with ambient air is within ±1 of the last significant digit compared to the reference value achieved during calibration (9.2). If it is not, re-clean and dry the cell and repeat the check. If the reading still differs, recalibrate the density meter.

9.3.2 Introduce the test portion of the sample into the cell using a suitable syringe or auto sampler, filling the cell according to the manufacturer's instructions.

When testing samples that are solid at room temperature, warm the syringe or auto sampler to a temperature that is 20 °C above the melting point of the sample.

9.3.3 When using an auto sampler, either run samples in duplicate or introduce check samples in order for errors due to bubble formation to be detected and the system performance monitored. When making a manual injection, switch on the cell illumination before injecting. Check the cell for bubbles and fill in accordance with the manufacturer's instructions. If bubbles are detected, empty and refill the cell and recheck for bubbles. With respect to the viewing and illumination of the cell, refer to the manufacturer's recommendations.

9.3.4 When the density meter displays a reading which is steady to within 0,1 g/l for "litre weight in air" or to five significant figures for the oscillation period, note and record the indicated figure and the cell temperature to the nearest 0,1 °C.

A consistent drift in the oscillation period or "litre weight in air" reading normally indicates that the cell has not reached equilibrium temperature. Random variations in readings normally indicate that air or gas bubbles are present in the cell. In this case, the cell should be recharged with a fresh sample.

9.3.5 Clean and dry the cell in accordance with the procedure given in 9.1.2.

10 Calculation

10.1 If the density meter displays an oscillation period, calculate the "litre weight" of the sample in accordance with the manufacturer's instructions.

10.2 If the "litre weight in air" is required at a reference temperature other than that at which it was determined, convert the "litre weight in air" to the litre weight at the reference temperature using the correction factor of 0,000 68 per °C. Not more than 3 °C difference should be converted by this calculation.

Express the "litre weight in air" to the nearest 0,000 1 kg/l at the reference temperature.

11 Precision

11.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in Annex A. It is possible that the values derived from this interlaboratory test are not applicable to concentration ranges and matrices other than those given.

11.2 Repeatability limit, *r*

The repeatability limit, *r*, is the value less than or equal to which the absolute difference between two test results obtained under repeatability conditions can be expected to be with a probability of 95 %. Repeatability conditions are conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time.

11.3 Reproducibility limit, *R*

The reproducibility limit, *R*, is the value less than or equal to which the absolute difference between two test results obtained under reproducibility conditions can be expected to be with a probability of 95 %. Reproducibility conditions are conditions where independent test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment within short intervals of time.

12 Test report

The test report shall specify at least the following information:

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

b) the sampling method used, if known;

c) the test method used, with reference to this International Standard (i.e. ISO 18301:2014);

d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which might have influenced the test result(s);

e) the test result(s) obtained;

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

Annex A
(informative)

Interlaboratory test

The precision of the method has been established by an international interlaboratory test carried out in accordance with ISO 5725. The test was organized by Netherlands Standardization Institute (NEN) in 2013.

The statistical results are given in Table A.1.

Table A.1 — Statistical results for the U-tube method

Type of sample and measuring temperature	Sunflower seed oil (20 °C)	Soya bean oil (20 °C)	Rapeseed oil (20 °C)	Coconut oil (45 °C)	Palm oil (55 °C)	Palm FA distillate (65 °C)
Number of participating laboratories	16	16	15	15	15	14
Number of laboratories retained after eliminating outliers	14	14	12	12	13	12
Number of individual tests in all laboratories	28	28	24	24	26	24
Mean, m (kg/l)	**0,922 31**	**0,919 21**	**0,914 58**	**0,903 52**	**0,886 24**	**0,859 64**
Repeatability standard deviation, s_r	0,000 10	0,000 09	0,000 05	0,000 02	0,000 05	0,000 08
Coefficient of variation of repeatability, $C_{V,r}$ (%)	0,011	0,010	0,005	0,002	0,005	0,010
Repeatability limit, r (2,8 s_r)	**0,000 27**	**0,000 25**	**0,000 13**	**0,000 06**	**0,000 13**	**0,000 24**
Reproducibility standard deviation, s_R	0,000 85	0,000 89	0,000 72	0,000 67	0,000 67	0,000 73
Coefficient of variation of reproducibility, $C_{V,R}$ (%)	0,093	0,097	0,079	0,074	0,076	0,085
Reproducibility limit, R (2,8 s_R)	**0,002 39**	**0,002 50**	**0,002 01**	**0,001 88**	**0,001 88**	**0,002 03**

A pyknometer method (see ISO 6883) was used to measure the “litre weight in air” of the same samples. The results from this test are given in Table A.2 for information and comparison only.

Table A.2 — Statistical results for the pyknometer method (see ISO 6883)

Type of sample and measuring temperature	Sunflower seed oil (20 °C)	Soya bean oil (20 °C)	Rapeseed oil (20 °C)	Coconut oil (45 °C)	Palm oil (55 °C)	Palm FA distillate (65 °C)
Number of participating laboratories	15	15	15	15	15	15
Number of laboratories retained after eliminating outliers	14	14	15	14	15	15
Number of individual tests in all laboratories	28	28	30	28	30	30
Mean, m (kg/l)	**0,922 37**	**0,919 08**	**0,914 19**	**0,903 49**	**0,886 44**	**0,860 12**
Repeatability standard deviation, s_r	0,000 16	0,000 14	0,000 16	0,000 20	0,000 31	0,000 28
Coefficient of variation of repeatability, $C_{V,r}$ (%)	0,017	0,015	0,017	0,022	0,035	0,032
Repeatability limit, r (2,8 s_r)	**0,000 44**	**0,000 40**	**0,000 44**	**0,000 56**	**0,000 88**	**0,000 78**
Reproducibility standard deviation, s_R	0,000 70	0,000 61	0,000 80	0,000 45	0,000 76	0,001 29
Coefficient of variation of reproducibility, $C_{V,R}$ (%)	0,076	0,066	0,087	0,050	0,086	0,150
Reproducibility limit, R (2,8 s_R)	**0,001 96**	**0,001 71**	**0,002 24**	**0,001 26**	**0,002 13**	**0,003 61**

Bibliography

[1] ISO 6883, *Animal and vegetable fats and oils — Determination of conventional mass per volume (litre weight in air)*

ICS 67.200.10
Price based on 8 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.™