
**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*



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Published in Switzerland

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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.

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).

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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 pycnometer; 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.

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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 $\pm 0,000\ 1\ \text{kg/l}$ 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 $\pm 0,05\ ^\circ\text{C}$ of the required temperature.

5.3 Calibrated temperature sensor, capable of measuring the temperature of the cell to an accuracy of at least $0,1\ ^\circ\text{C}$.

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 $0,45\ \mu\text{m}$ 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 $15\ ^\circ\text{C}$ to $65\ ^\circ\text{C}$ 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 $10\ ^\circ\text{C}$ 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 pycnometer 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.