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Animal and vegetable fats and oils — Determination of conventional mass per volume (litre weight in air)

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

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Foreword

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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 World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso. org/iso/foreword. html.

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

This fifth edition cancels and replaces the fourth edition (ISO 6883:2007), of which it constitutes a minor revision to exclude its applicability for fat coming from milk and milk products and to include further precision data.

Animal and vegetable fats and oils — Determination of conventional mass per volume (litre weight in air)

1 Scope

This document specifies a method for the determination of the conventional mass per volume ("litre weight in air") of animal and vegetable fats and oils (hereinafter referred to as fats) in order to convert volume to mass or mass to volume.

The procedure is applicable to fats only when they are in a liquid state. Milk and milk products (or fat coming from milk and milk products) are excluded from the scope of this document.

NOTE The determination of conventional mass per volume (litre weight in air) using the oscillating U-tube method can be found in ISO 18301.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp/

3.1
conventional mass per volume
litre weight in air
quotient of the mass in air of fat to its volume at a given temperature

Note 1 to entry: It is expressed in kilograms per litre (numerically equal to grams per millilitre).

4 Principle

The mass of a volume of liquid fat in a calibrated pyknometer is measured at a specified temperature.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Water bath, capable of being maintained to within 0,1 °C of the temperatures chosen for the calibration and determination.

It should be fitted with a calibrated thermometer, graduated in divisions of 0,1 °C covering the relevant temperature range.

5.2 Pyknometer (Jaulmes), of capacity 50 ml, with side-arm.

It should be fitted by means of conical joints with a calibrated thermometer graduated in divisions of 0,1 °C and with a cap perforated at the top for the side-arm (see Figure 1).

The pyknometer should preferably be made of borosilicate glass, but if this is not available, then one made of soda glass may be used.

NOTE The cap is only essential if the determination is carried out at a temperature below ambient.

Alternatively, the Type 3 (Gay-Lussac) pyknometer (see Figure 2) specified in ISO 3507 may be used; however, the use of a pyknometer with thermometer is preferred.

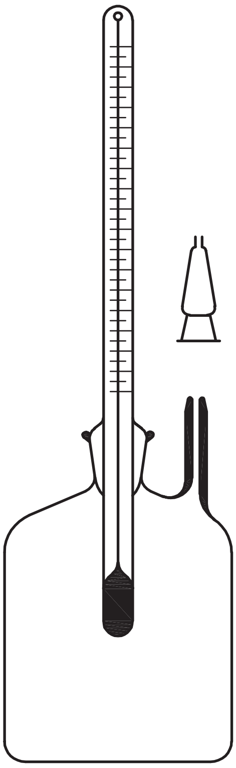

Figure 1 — Jaulmes pyknometer

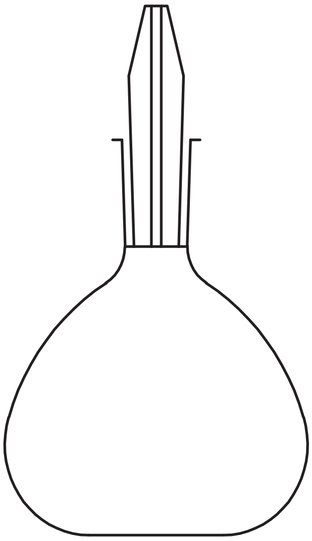

Figure 2 — Gay-Lussac pyknometer

6 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 document. A recommended sampling method is given in ISO 5555.

7 Preparation of test sample

Prepare the test sample in accordance with ISO 661, but do not filter or dry it.

Take care not to include air bubbles in the fat.

8 Procedure

8.1 Calibration of pyknometer

8.1.1 Calibrate the pyknometer (5.2) at least once a year, and at least in duplicate, by the procedure described in 8.1.2. Calibrate a pyknometer made of soda glass at least once every 3 months, at least in duplicate.

NOTE The calibration procedure described is used to determine the volume of the pyknometer when filled with water at the temperature θ_c.

8.1.2 Calibrate the pyknometer at the following temperatures:

a) at 40 °C if the mean coefficient of cubic expansion (γ) of the pyknometer glass is known;

b) at 20 °C and 60 °C if γ is not known.

8.1.3 Clean and thoroughly dry the pyknometer. Weigh, to the nearest 0,1 mg, the empty pyknometer with the thermometer and cap or with the stopper (m_1).

Bring recently distilled water or water of equivalent purity, free from air, to a temperature approximately 5 °C below the temperature of the water bath. Remove the thermometer and cap (or the stopper) and fill the pyknometer with the prepared water. Replace the thermometer or stopper. Take care not to include air bubbles during these operations. Place the filled pyknometer in the water bath, so that it is immersed up to the middle of its conical socket, until the contents have reached a stable temperature (which takes about 1 h). Allow the water to overflow from the side-arm or stopper outlet. Record the temperature, θ_c, of the pyknometer contents to the nearest 0,1 °C. Carefully remove any water that has overflowed from the top and side of the side-arm or stopper. Place the cap on the side-arm. Remove the pyknometer from the water bath, wiping it thoroughly with fluff-free material until dry. Allow its temperature to reach ambient.

Weigh the full pyknometer with the thermometer and cap, or with the stopper, to the nearest 0,1 mg (m_2).

If the value of γ for the pyknometer glass is not known, adjust the water bath to the desired second calibration temperature and repeat the calibration procedure.

8.2 Determination

8.2.1 General

The temperature of determination applied for any fat should be such that the fat does not deposit crystals at that temperature.

For a temperature of determination below ambient temperature, use a Jaulmes pyknometer.

Clean and thoroughly dry the pyknometer. Weigh, to the nearest 0,1 mg, the empty pyknometer with the thermometer and cap or with the stopper.

Adjust the water bath (5.1) to a temperature that does not vary by more than 1 °C from the temperature required for the determination, i.e. the temperature at the time of measurement of the fat in the bulk tank.

Bring the prepared test sample (Clause 7) to a temperature of 3 °C to 5 °C below the temperature of the water bath. Mix carefully.

8.2.2 Fats which are solid at ambient temperatures

Heat the test sample (Clause 7) to approximately 5 °C to 10 °C above its melting point. Stir until all the crystals are seen to be dissolved completely. Follow the procedure given in 8.2.1, allowing the full pyknometer to cool before weighing it.

8.2.3 Using the Jaulmes pyknometer

Weigh, to the nearest 0,1 mg, the empty pyknometer with the thermometer and cap.

Remove the cap from the side-arm and replace it by a short piece of flexible plastic tubing (3 cm to 5 cm) to form a watertight joint. Fill the pyknometer with the test sample and replace the thermometer, taking care not to include air bubbles.

NOTE Some of the sample rises into the plastic tube and is then able to expand or contract, as appropriate.

Immerse the filled pyknometer, up to the middle of its conical socket, for 2 h in the water bath (5.1) maintained at the temperature chosen for the determination, to allow the contents to reach this temperature. Remove the filled plastic tube with thumb and forefinger and wipe dry the surplus sample from the outlet. Replace the cap. Record the temperature, θ_d, of the pyknometer to the nearest 0,1 °C.

Remove the pyknometer from the water bath, wiping it carefully with fluff-free material until dry. Allow its temperature to reach ambient, then weigh to the nearest 0,1 mg, the full pyknometer with the thermometer and cap (m_3).

8.2.4 Using the Gay-Lussac pyknometer

Weigh, to the nearest 0,1 mg, the empty pyknometer with the stopper.

Fill the pyknometer with the test sample (Clause 7) and replace the stopper taking care not to include air bubbles. Immerse the filled pyknometer, up to the middle of its conical socket, for 2 h in the water bath (5.1) maintained at the temperature chosen for the determination, to allow the contents to reach this temperature.

Allow the sample to overflow and wipe the surplus from the outlet. Record the temperature, θ_d, of the water bath to the nearest 0,1 °C. Wipe dry the surplus from the outlet.

Remove the pyknometer from the water bath, wiping it carefully with fluff-free material until dry. Allow its temperature to reach ambient, then weigh to the nearest 0,1 mg, the full pyknometer with stopper (m_3).

9 Expression of results

9.1 Calculation of the volume of the pyknometer

Calculate the volume of the pyknometer at the calibration temperature θ_c by Formula (1):

$$V_c = \frac{m_2 - m_1}{\rho_w} \tag{1}$$

where

V_c is the volume, in millilitres, of the pyknometer at calibration temperature θ_c;

m_2 is the mass, in grams, of the pyknometer filled with water, including thermometer and cap or stopper;

m_1 is the mass, in grams, of the empty pyknometer with thermometer and cap or with stopper;

ρ_w is the conventional mass per volume of water at calibration temperature θ_c, in grams per millilitre (deduce ρ_w from Table 1, if necessary by interpolation).

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		

If the mean coefficient of cubic expansion (γ) of the pyknometer glass is not known, calculate γ from the calibration results at 20 °C and 60 °C by Formula (2):

$$\gamma = \frac{V_{c2} - V_{c1}}{V_{c1}(\theta_2 - \theta_1)} \tag{2}$$

where

γ is the mean coefficient of cubic expansion of the pyknometer glass, per degree Celsius;

V_{c2} is the volume, in millilitres, of the pyknometer at calibration temperature θ_2;

V_{c1} is the volume, in millilitres, of the pyknometer at calibration temperature θ_1;

θ_1 is the temperature, in degrees Celsius, close to 60 °C, at which the pyknometer was calibrated;

θ_2 is the temperature, in degrees Celsius, close to 20 °C, at which the pyknometer was calibrated.

NOTE The mean coefficient of cubic expansion of glass depends on the composition of the glass, for example:

— borosilicate glass D 50: $\gamma \approx 0{,}000\,01$ per degree Celsius;

— borosilicate glass G 20: $\gamma \approx 0{,}000\,015$ per degree Celsius;

— soda glass: $\gamma \approx 0{,}000\,025$ to $0{,}000\,030$ per degree Celsius.

Calculate the volume of the pyknometer at a temperature θ_d by Formula (3):

$$V_d = V_c[1 + \gamma(\theta_d - \theta_c)] \tag{3}$$

where

V_d is the volume, in millilitres, of the pyknometer at a temperature θ_d;

V_c is the volume, in millilitres, of the pyknometer at calibration temperature θ_c;

γ is the mean coefficient of cubic expansion of the pyknometer glass, per degree Celsius;

θ_d is the temperature, in degrees Celsius, at which one wants to know the volume of the pyknometer;

θ_c is the temperature (or one of the temperatures), in degrees Celsius, at which the pyknometer was calibrated.

9.2 Calculation of the conventional mass per volume

Calculate the conventional mass per volume of the test sample, p_θ, in grams per millilitre, at the specified or required temperature by Formula (4):

$$p_\theta = \frac{m_3 - m_1}{V_d} + k(\theta_d - \theta) \tag{4}$$

where

m_1 is the mass, in grams, of the empty pyknometer with the thermometer and cap or with the stopper;

m_3 is the mass, in grams, of the pyknometer filled with test sample, including the thermometer and cap or stopper;

V_d is the volume, in millilitres, of the pyknometer at a temperature θ_d;

θ_d is the temperature, in degrees Celsius, at which the determination was performed;

θ is the temperature, in degrees Celsius, at which the conventional mass per volume is to be established;

k is the mean change in the conventional mass per volume of fat due to the temperature change, in grams per millilitre per degree Celsius (k = 0,000 68 g/ml per degree Celsius).

The value for k of 0,000 68 g/ml per degree Celsius is an approximate mean value for fats. If the actual value for k is known, this value should be used in the interest of greater accuracy.

The corrections in grams per millilitre per degree Celsius may also be used to convert litre weight in air at one temperature to another, provided that the differences in temperature are not more than 5 °C.

Express the result to the nearest 0,000 1 g/ml.

10 Precision

10.1 Interlaboratory tests

Details of 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.

10.2 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 the cases exceed the value of the repeatability limit, *r*, given in Table A.1.

10.3 Reproducibility

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

11 Test report

The test report shall specify the following:

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

b) the method of sampling used, if known;

c) the test method used, with reference to this document, i.e. ISO 6883;

d) the type of pyknometer used;

e) the temperature of determination and the specified or required temperature;

f) any operating details not specified in this document, or regarded as optional, together with details of any incidents which may have influenced the test results;

g) the test result obtained or, if the repeatability has been checked, the final result obtained.

Annex A
(informative)

Results of interlaboratory tests

International collaborative tests have been carried out on the method given in this document in accordance with ISO 5725-1 and ISO 5725-2.

Tests on the following were organized by FOSFA International:

— refined bleached and deodorized (RBD) palm olein (A + B);

— crude coconut oil (C + D);

— crude rapeseed oil (E + F);

— crude degummed rapeseed oil (G).

The results are given in Table A.1.

Table A.1 — Precision data

Sample	A	B	C	D	E	F	G
Number of participating laboratories (*N*)	53	52	35	35	54	54	87
Number of laboratories retained after eliminating outliers (*n*)	43	44	29	29	42	42	80
Number of individual test results of all laboratories on each sample (*z*)	86	88	62	62	84	84	160
Mean value (*m*), g/ml	0,890 58	0,890 64	0,907 32	0,907 47	0,904 55	0,904 53	0,916 86
Repeatability standard deviation (s_r), g/ml	0,000 08	0,000 07	0,000 05	0,000 07	0,000 09	0,000 07	0,000 10
Repeatability coefficient of variation, %	0,009 46	0,008 03	0,005 66	0,007 72	0,009 66	0,007 31	0,010 42
Repeatability limit, *r*, g/ml ($s_r \times 2,8$)	0,000 24	0,000 20	0,000 14	0,000 20	0,000 24	0,000 19	0,000 27
Reproducibility standard deviation (s_R), g/ml	0,000 47	0,000 71	0,000 75	0,000 83	0,000 49	0,000 47	0,000 67
Reproducibility coefficient of variation, %	0,052 57	0,079 57	0,082 54	0,091 03	0,054 36	0,051 45	0,072 92
Reproducibility limit, *R*, g/ml ($s_R \times 2,8$)	0,001 31	0,001 98	0,002 10	0,002 31	0,001 38	0,001 30	0,001 87

In 2013, a further international interlaboratory test was carried out in accordance with ISO 5725 and organized by Netherlands Standardization Institute (NEN) to compare this document with the method using a U-tube (ISO 18301). The precision data from this test are shown in Tables A.2 and A.3.

Table A.2 — Statistical results for the pyknometer method (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**

Table A.3 — Statistical results for the U-tube method (ISO 18301)

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**

Bibliography

[1] ISO 3507, *Laboratory glassware — Pyknometers*

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

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

[4] 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*

[5] ISO 18301, *Animal and vegetable fats and oils — Determination of conventional mass per volume (litre weight in air) — Oscillating U-tube method*

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