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EAS 316 (2002) (English): Animal and vegetable fats and oils - Determination of conventional mass per volume (litre weight in air)

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EAST AFRICAN STANDARD

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

EAST AFRICAN COMMUNITY

Foreword

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INTERNATIONAL STANDARD

Third edition 2000-07-01

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 dans l'air («poids du litre dans l'air»)



Reference number ISO 6883:2000(E)

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

International Standard ISO 6883 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This third edition cancels and replaces the second edition (ISO 6883:1995), of which it constitutes a minor revision.

Annex A of this International Standard is for information only.

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

1 Scope

This International Standard specifies a method for the determination of 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 only to fats in a liquid state.

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

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 661:1989, Animal and vegetable fats and oils — Preparation of test sample.

ISO 3507:1976, Pyknometers.

3 Term and definition

For the purposes of this International Standard, the following term and definition applies.

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 It is expressed in kilograms per litre (numerically equal to grams per millilitre).

4 Principle

Measurement of the mass, at a specified temperature, of a volume of liquid fat in a calibrated pyknometer.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Water bath, capable of being maintained 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

6 Sampling

It is important that the laboratory receive a sample which is truly representative and has not 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.

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

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 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, $\theta_{\rm C}$, by the equation:

$$V_{\rm C} = \frac{m_2 - m_1}{\rho_{\rm W}}$$

where

- $V_{\rm c}$ is the volume, in millilitres, of the pyknometer at calibration temperature $\theta_{\rm c}$;
- 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;
- $\rho_{\rm W}$ is the conventional mass per volume of water at calibration temperature $\theta_{\rm c}$, in grams per millilitre (deduce $\rho_{\rm W}$ from Table 1, if necessary by interpolation).

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 the equation:

$$\gamma = \frac{V_{c2} - V_{c1}}{V_{c1} \left(\theta_2 - \theta_1\right)}$$

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:	$\gamma~pprox~$ 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 the equation:

$$V_{\rm d} = V_{\rm c} \big[1 + \gamma \big(\theta_{\rm d} - \theta_{\rm c} \big) \big]$$

where

- $V_{\rm d}$ is the volume, in millilitres, of the pyknometer at a temperature $\theta_{\rm d}$;
- $V_{\rm c}$ is the volume, in millilitres, of the pyknometer at calibration temperature $\theta_{\rm c}$;

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- 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;
- $\theta_{\rm C}$ is the temperature (or one of the temperatures), in degrees Celsius, at which the pyknometer was calibrated.

Temperature	"Litre weight in air"	Temperature	"Litre weight in air"	Temperature	"Litre weight in air"	
θ	$ ho_{W}$	θ	$ ho_{W}$	θ	$ ho_{W}$	
°C	g/ml	٥C	g/ml	٥C	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			

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

9.2 Calculation of the conventional mass per volume

Calculate the conventional mass per volume of the test sample, ρ_{θ} in grams per millilitre, at the specified or required temperature by the equation:

$$\rho_{\theta} = \frac{m_3 - m_1}{V_{d}} + k(\theta_{d} - \theta)$$

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;
- $V_{\rm d}$ is the volume, in millilitres, of the pyknometer at a temperature $\theta_{\rm d}$;
- 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).

NOTE 1 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.

NOTE 2 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 cases be greater than 0,000 2 g/ml.

If the difference, as described above, exceeds 0,000 2 g/ml, repeat the determination using a further test sample.

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 cases be greater than 0,007 g/ml.

11 Test report

The test report shall specify:

- all information necessary for the complete identification of the sample;
- the method of sampling used, if known;
- the test method used, with reference to this International Standard;
- the type of pyknometer used;
- the temperature of determination and the specified or required temperature;
- any operating details not specified in this International Standard, or regarded as optional, together with details
 of any incidents which may have influenced the test results;
- 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 International Standard in accordance with ISO 5725-1 and ISO 5725-2.

The tests on refined bleached and deodorized (RBD) palm olein, crude coconut oil and crude rapeseed oil were organized by FOSFA International. The results are given in Table A.1.

	RBD palm olein		Crude coconut oil		Crude rapeseed oil	
	1995	1995	1997	1997	1999	1999
No. of laboratories returning results	53	53	36	36	62	62
No. of laboratories with acceptable results	40	40	33	33	51	51
Mean value, g/ml	0,8906	0,8906	0,9078	0,9079	0,9054	0,9050
Repeatability limit, r, g/ml	0,0002	0,0002	0,0001	0,0002	0,0003	0,0002
Reproducibility limit, R, g/ml	0,0011	0,0012	0,0065	0,0065	0,0082	0,0062

Table A.1 — Precision data

Bibliography

- [1] ISO 5555:1991, Animal and vegetable fats and oils Sampling.
- [2] ISO 5725-1:1994, Accuracy (trueness and precision) of measurement methods and results Part 1: General principles and definitions.
- [3] ISO 5725-2:1994, 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.

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