INTERNATIONAL STANDARD

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Animal and vegetable fats and oils — Determination of moisture and volatile matter content

Corps gras d'origines animale et végétale — Détermination de la teneur en eau et en matières volatiles

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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 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 third edition cancels and replaces the second edition (ISO 662:1998), of which it constitutes a minor revision. A non-applicability statement for milk and milk products has been added to the Scope.

Annex A of this International Standard is for information only.

Animal and vegetable fats and oils — Determination of moisture and volatile matter content

1 Scope

This International Standard specifies two methods for the determination, by drying, of the moisture and volatile matter content of animal or vegetable fats and oils:

— method A, using a sand bath or hotplate;

— method B, using a drying oven.

Method A is applicable to all fats and oils.

Method B is applicable only to non-drying fats and oils with an acid value less than 4. Under no circumstances are lauric oils be analysed by this method.

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

2 Normative reference

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
moisture and volatile matter content
loss in mass undergone by the product on heating at 103 °C ± 2 °C under the conditions specified in this International Standard

Note 1 to entry: It is expressed as a percentage by mass.

4 Principle

Heating a test portion at 103 °C ± 2 °C until moisture and volatile substances are completely eliminated, and determination of the loss in mass.

5 Sampling

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

It is important that the laboratory receives a sample which is truly representative and has not been damaged or changed during transport or storage.

6 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

7 Method A

7.1 Apparatus

Usual laboratory apparatus and, in particular, the following.

7.1.1 Analytical balance, capable of weighing to the nearest 0,001 g.

7.1.2 Dish, made of porcelain or glass, 80 mm to 90 mm in diameter, about 30 mm deep, with a flat bottom.

7.1.3 Thermometer, graduated from about 80 °C to at least 110 °C, about 100 mm long, with a reinforced tip and with an expansion chamber at its upper end.

7.1.4 Sand bath or **electric hotplate**.

7.1.5 Desiccator, containing an efficient desiccant.

7.2 Procedure

7.2.1 Test portion

Weigh, to the nearest 0,001 g, approximately 20 g of the test sample (Clause 6) into the dish (7.1.2) which has been previously dried and then weighed together with the thermometer (7.1.3).

7.2.2 Determination

Heat the dish containing the test portion (7.2.1) on the sand bath or electric hotplate (7.1.4), allowing the temperature to rise at a rate of about 10 °C/min up to 90 °C, and stirring constantly with the thermometer.

Reduce the rate of heating, observing the rate at which bubbles rise from the bottom of the dish, and allow the temperature to rise to 103 °C ± 2 °C. Do not heat above 105 °C. Continue to stir, scraping the bottom of the dish until all evolution of bubbles has ceased.

To ensure the removal of all moisture, repeat the heating to 103 °C ± 2 °C several times, cooling to 95 °C between the heating periods. Then allow the dish and thermometer to cool in the desiccator (7.1.5) to room temperature and weigh to the nearest 0,001 g. Repeat this operation until the difference between the results of two successive weighings does not exceed 2 mg.

7.2.3 Number of determinations

Carry out two determinations on test portions taken from the same test sample (Clause 6).

8 Method B

8.1 Apparatus

Usual laboratory apparatus and, in particular, the following.

8.1.1 Analytical balance, capable of weighing to the nearest 0,001 g.

8.1.2 Glass vessel, approximately 50 mm in diameter and 30 mm tall, with a flat bottom.

8.1.3 Electric drying oven, capable of being maintained at 103 °C ± 2 °C.

8.1.4 Desiccator, containing an efficient desiccant.

8.2 Procedure

8.2.1 Test portion

Weigh, to the nearest 0,001 g, approximately 5 g or 10 g of the test sample (Clause 6), according to the expected moisture and volatile matter content, into the vessel (8.1.2), which has been previously dried and then weighed.

8.2.2 Determination

Keep the vessel containing the test portion (8.2.1) for 1 h in the drying oven (8.1.3), set at 103 °C. Allow to cool to room temperature in the desiccator (8.1.4) and then weigh to the nearest 0,001 g. Repeat the operations of heating, cooling and weighing, but use successive periods in the oven of 30 min each, until the loss in mass between two successive weighings does not exceed 2 mg or 4 mg, according to the mass of the test portion.

NOTE An increase of mass of the test portion after repeated heating indicates that autoxidation of the fat or oil has occurred. In this case, take for calculation of the result the smallest mass recorded or, preferably, use method A.

8.2.3 Number of determinations

Carry out two determinations on test portions taken from the same test sample (Clause 6).

9 Expression of results

The moisture and volatile matter content, w, expressed as a percentage by mass, is equal to

$$w = \frac{m_1 - m_2}{m_1 - m_0} \times 100\% \quad (1)$$

where

m_0 is the mass, in grams, of the dish and the thermometer (7.2.1), or of the glass vessel (8.2.1);

m_1 is the mass, in grams, of the dish, thermometer and test portion (7.2.1), or of the vessel and test portion (8.2.1) before heating;

m_2 is the mass, in grams, of the dish, thermometer and residue (7.2.2), or of the vessel and residue (8.2.2) after heating.

Take as the result the arithmetic mean of the two determinations, provided that the requirement for repeatability (see 10.2) is satisfied.

Report the result to the second decimal place.

10 Precision

10.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in Annex A. The values derived from this interlaboratory test may not be applicable to concentration 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 0,03 g of moisture and volatile matter per 100 g of sample for a moisture and volatile matter content of about 0,3 %.

10.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than 0,15 g of moisture and volatile matter per 100 g of sample for a moisture and volatile matter content of about 0,3 %.

11 Test report

The test report shall specify the following:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with a reference to this International Standard, i.e. ISO 662;

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

— the test result(s) obtained;

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

Annex A
(informative)

Results of interlaboratory tests

The results given in Tables A.1 and A.2 were obtained in interlaboratory tests on the determination of moisture and volatile matter in fats and oils, organized by the Federation of Oils, Seeds and Fats Association Ltd. (FOSFA) and carried out in accordance with ISO 5725[1)].

Table A.1 — Results obtained using method A (see Clause 7)

Date	1993	1993	1992	1991	1991	1997		1988
Type of oil or fat	Sunflower seed oil		Palm oil	Beef tallow		Coconut oil		Palm oil
	Sample			Sample		Sample		
	a	b		a	b	a	b	
No. of laboratories	27	27	33	17	17	21	21	27
No. of results accepted	27	27	31	17	16	21	21	21
Overall mean value, %	0,13	0,13	0,017	0,260	0,270	0,233	0,231	0,045
Repeatability standard deviation, s_r, %	0,01	0,01	0,003	0,01	0,01	0,009	0,011	0,007
Repeatability coefficient of variation, %	4,68	4,86	15,2	3,99	2,41	3,717	4,593	14,4
Reproducibility standard deviation, s_R, %	0,02	0,02	0,012	0,03	0,03	0,047	0,052	0,024
Reproducibility coefficient of variation, %	15,5	13,3	66,6	12,7	11,7	20,35	22,37	51,4
Repeatability limit r (2,8 s_r), %	0,020	0,020	0,007	0,030	0,020	0,025	0,031	0,020
Reproducibility limit R (2,8 s_R), %	0,060	0,050	0,033	0,090	0,090	0,132	0,145	0,070

1) ISO 5725:1986 (now withdrawn) was used to obtain the precision data.