INTERNATIONAL STANDARD

ISO 662

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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

Foreword

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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.

International Standard ISO 662 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This second edition cancels and replaces the first edition (ISO 662:1980), which has been technically revised.

Annexes A and B of this International Standard are for information only.

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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 should lauric oils be analysed by this method.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject ot revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

3 Definition

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

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 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 laboratory receive 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 mercury bulb 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 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

9.1 Method of calculation

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

$$w = \frac{m_1 - m_2}{m_1 - m_0} \times 100\ \%$$

where

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

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

m_2 is the mass, in grams, of the dish, thermometer and residue (see 7.2.2), or of the vessel and residue (see 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:

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

— the sampling method used, if known;

— the test method used, with reference to this International Standard;

— 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; or

— 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.

Table A.2 — Results obtained using Method B (see clause 8)

Date	1995	1995	1993	1993	1991	1991	1989
Type of oil or fat	Soyabean oil		Sunflower seed oil		Beef tallow		Fish oil
	Sample		Sample		Sample		
	a	b	a	b	a	b	
No. of laboratories	51	51	25	25	25	25	43
No. of results accepted	51	51	25	25	24	25	39
Overall mean value, %	0,040	0,094	0,130	0,130	0,250	0,250	0,090
Repeatability standard deviation, s_r, %	0,004	0,005	0,01	0,01	0,01	0,01	0,006
Repeatability coefficient of variation, %	10,00	5,32	5,24	4,06	3,97	3,25	6,43
Reproducibility standard deviation, s_R, %	0,16	0,02	0,03	0,03	0,04	0,04	0,03
Reproducibility coefficient of variation, %	40,0	21,28	18,90	19,70	18,20	14,40	34,63
Repeatability limit, r (2,8 s_r), %	0,012	0,013	0,020	0,010	0,030	0,020	0,020
Reproducibility limit, R (2,8 s_R), %	0,046	0,056	0,070	0,070	0,110	0,100	0,090

Annex B
(informative)

Bibliography

[1] ISO 555:1991, *Animal and vegetable fats and oils — Sampling*.

[2] ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*.

[3] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

[4] 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.*