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Animal and vegetable fats and oils — Determination of insoluble impurities content

Corps gras d'origines animale et végétale — Détermination de la teneur en impuretés insolubles

Reference number
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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 663:2007), of which it constitutes a minor revision to exclude fat coming from milk and milk products.

Animal and vegetable fats and oils — Determination of insoluble impurities content

1 Scope

This document specifies a method for the determination of the insoluble impurities content of animal and vegetable fats and oils.

If it is not desired to include soaps (particularly calcium soaps) or oxidized fatty acids in the insoluble impurities content, it is necessary to use a different solvent and procedure. In this case, an agreement is to be reached between the parties concerned.

Milk and milk products (or fat coming from milk and milk products) are excluded from the scope of this document.

2 Normative reference

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 http://www.iso.org/obp

3.1
insoluble impurities content
quantity of dirt and other foreign matter insoluble in *n*-hexane or light petroleum under the conditions specified in this document

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

Note 2 to entry: These impurities include mechanical impurities, mineral substances, carbohydrates, nitrogenous substances, various resins, calcium soaps, oxidized fatty acids, fatty acid lactones, and (in part) alkali soaps, hydroxy-fatty acids and their glycerides.

4 Principle

A test portion is treated with an excess of *n*-hexane or light petroleum, then the solution obtained is filtered. The filter and residue are washed with the same solvent, then dried at 103 °C and weighed.

5 Reagents

WARNING — Attention is drawn to the regulations which specify the handling of dangerous substances. Technical, organizational and personal safety measures shall be followed.

Use only reagents of recognized analytical grade.

5.1 ***n*-Hexane**, or in the absence of this, **light petroleum**, having a distillation range between 30 °C and 60 °C and having a bromine value of less than 1.

For either solvent, the residue on complete evaporation shall not exceed 0,002 g per 100 ml.

5.2 **Kieselgur**, purified, calcinated, with loss in mass at 900 °C (red heat) of less than 0,2 % by mass.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 **Analytical balance**, with an accuracy of ±0,001 g.

6.2 **Electric drying oven**, capable of operating at 103 °C ± 2 °C.

6.3 **Conical flask**, of 250 ml capacity, with ground glass stopper.

6.4 **Desiccator**, containing an efficient desiccant.

6.5 **Ashless filter paper** (maximum ash content 0,01 %, by mass), retention value of 98 %, by mass, for particles of size greater than 2,5 µm[1)], or an equivalent **glass-fibre filter**, of diameter 120 mm, together with a metal (preferably aluminium) or glass **vessel** with a well-fitting lid.

These are alternatives to the filter (6.6) for all products except acid oils.

6.6 **Filter crucible**, glass, of grade P16 (pore size 10 µm to 16 µm), diameter 40 mm, of capacity 50 ml, together with a **suction bottle**.

This is an alternative to 6.5 for all products including acid oils.

7 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.

8 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

9 Procedure

9.1 Test portion

Weigh, to the nearest 0,01 g, approximately 20 g of the test sample (Clause 8) into a conical flask (6.3).

1) Whatman 42 (2,5 µm) filter paper or Whatman GF/D glass-fibre filter are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

9.2 Determination

9.2.1 Dry the filter paper and the vessel (6.5) with its lid, or the filter crucible (6.6), in the oven (6.2) set at 103 °C. Allow to cool in the desiccator (6.4) then weigh to the nearest 0,001 g. For acid oils, prepare the crucible as described in 9.2.7 and continue as in 9.2.2.

9.2.2 Add 200 ml of *n*-hexane or light petroleum (5.1) to the flask containing the test portion (9.1). Stopper the flask and shake.

For castor oil, the quantity of solvent may be increased to facilitate the operation and this may necessitate the use of a larger flask.

Leave to stand at about 20 °C for about 30 min.

9.2.3 Filter through the filter paper in a suitable funnel or through the filter crucible, using suction if necessary. Rinse the flask to ensure that all impurities are washed into the filter/crucible.

Wash the filter paper or filter crucible by pouring through it small amounts of the same solvent as used in 9.2.2, but no more than is necessary for the final filtrate to be free from fat or oil. Warm the solvent, if necessary, to a maximum temperature of 60 °C in order to dissolve any solidified fats retained on the filter.

9.2.4 If a filter paper is used, remove it from the funnel and place it in the vessel. Allow most of the solvent remaining in the filter paper to evaporate in air and complete the evaporation in the oven set at 103 °C. Then remove from the oven, close the vessel with its lid, allow to cool in the desiccator (6.4) and weigh to the nearest 0,001 g.

9.2.5 If a filter crucible is used, allow most of the solvent remaining in it to evaporate in air under a fume hood and complete the evaporation in the oven set at 103 °C. Then remove from the oven, allow to cool in the desiccator (6.4) and weigh to the nearest 0,001 g.

9.2.6 If it is desired to determine the content of organic impurities, the use of a previously dried and weighed, ashless filter paper is necessary. In this case, the filter paper containing the insoluble impurities shall be ignited and the mass of ash obtained subtracted from the mass of insoluble impurities.

The organic impurities content, expressed as a percentage by mass, is then calculated by multiplying this difference in mass by $100/m_0$, where m_0 is the mass, in grams, of the test portion.

9.2.7 If analysing acid oils, coat the glass filter crucible with kieselgur (5.2) as follows. In a 100 ml glass beaker, prepare a slurry consisting of 2 g of kieselgur and approximately 30 ml of light petroleum (5.1). Pour the mixture into the filter crucible under reduced pressure in order to obtain a layer of kieselgur on the glass filter.

Dry the prepared glass filter crucible for 1 h in the oven (6.2) set at 103 °C. Allow to cool in the desiccator (6.4) and weigh to the nearest 0,001 g.

Carry out two determinations on test portions taken from the same test sample (Clause 8).

10 Expression of results

The insoluble impurities content, w, expressed as a percentage by mass, is equal to

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

where

m_0 is the mass of the test portion (9.1), in grams;

m_1 is the mass of the vessel with its lid and filter paper, or of the filter crucible (see 9.2.1), in grams;

m_2 is the mass of the vessel with its lid and filter paper containing the dry residue (see 9.2.4), or of the filter crucible and dry residue (see 9.2.5), in grams.

Report the result to the second decimal place.

11 Precision

11.1 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 concentration ranges and matrices other than those given.

11.2 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.

11.3 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 exceed the value of the reproducibility limit, R, given in Table A.1.

12 Test report

The test report shall specify the following:

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 document, i.e. ISO 663;

d) the solvent used;

e) 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;

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

Annex A
(informative)

Results of interlaboratory tests

Interlaboratory tests on the determination of insoluble impurities in palm oil, crude palm oil and palm kernel oil were organized by the Federation of Oils, Seeds and Fats Associations (FOSFA) and carried out in accordance with ISO 5725-2.

The results are shown in Table A.1.

Table A.1 — Results of interlaboratory tests on different oil

Sample	RDB palm olein	RDB palm oil	Crude palm kernel oil	Crude palm olein	Crude fish oil	Crude palm oil
Number of participating laboratories	16	35	41	27	41	12
Number of laboratories retained after eliminating outliers	16	31	33	26	35	11
Number of individual test results of all laboratories on each sample	16	93	66	52	70	22
Mean value, %	0,004	0,008	0,012	0,016	0,021	0,025
Repeatability standard deviation, s_r	0,003	0,003	0,003	0,005	0,004	0,004
Repeatability coefficient of variation, %	57,1	41,1	22,4	30,5	20,4	14,8
Repeatability limit, r (s_r × 2,8)	0,007	0,009	0,008	0,013	0,012	0,010
Reproducibility standard deviation, s_R	0,005	0,010	0,010	0,009	0,009	0,013
Reproducibility coefficient of variation, %	116,6	119,6	81,2	58,4	39,8	52,3
Reproducibility limit, R (s_R × 2,8)	0,014	0,027	0,028	0,026	0,024	0,037