INTERNATIONAL STANDARD

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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

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 663 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 663:1992). Precision data have been added.

Annex A of this International Standard is for information only.

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

1 Scope

This International Standard 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 the method should be the subject of agreement between the parties concerned.

2 Normative reference

The following normative document contains 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 edition of the normative document 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.*

3 Term and definition

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

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 International Standard

NOTE 1 The content is expressed as a percentage by mass.

NOTE 2 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 Reagent

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 less than 1.

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

5.2 **Kieselguhr,** purified, calcinated, loss in mass at 900 °C (red heat) of 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 **Desiccateur,** 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. (Alternative to 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**. (Alternative to 6.5 for all products including acid oils.)

7 Sampling

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

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

8 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

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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 International Standard and does not constitute an endorsement by ISO of these products.

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).

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) and weigh to the nearest 0,001 g.

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.

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. 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, and complete the evaporation in the oven set at 103 °C. 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 kieselguhr (5.2) as follows. In a 100 ml glass beaker, prepare a slurry consisting of 2 g of kieselguhr 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 kieselguhr 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 cases be greater than 0,01 g of impurities per 100 g of sample containing not more than about 0,10 % (by mass) of insoluble impurities.

11.3 Reproducibility

The absolute difference between two independent 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 0,06 g of impurities per 100 g of sample containing not more than about 0,10 % (by mass) of insoluble impurities.

12 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;

— the solvent used;

— 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

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 Tables A.1 to A.3.

Table A.1 — Interlaboratory test on palm oil

Year of interlaboratory test	1988
Number of laboratories participating	12
Number of nationalities	10
Number of accepted results	11
Mean value, % (by mass)	0,024
Repeatability limit r, % (by mass)	0,007
Reproducibility limit R, % (by mass)	0,038

Table A.2 — Interlaboratory test on crude palm oil

Year of interlaboratory test	1988
Number of laboratories participating	27
Number of nationalities	11
Number of accepted results	25
Mean value, % (by mass)	0,015
Repeatability limit, r, % (by mass)	0,008
Reproducibility limit, R, % (by mass)	0,026

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