INTERNATIONAL STANDARD

ISO 663

Second edition
1992-10-15

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
ISO 663:1992(E)

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.

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 663 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Sub-Committee SC 11, *Animal and vegetable fats and oils*.

This second edition cancels and replaces the first edition (ISO 663:1981), which has been technically revised.

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Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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.

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 to 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 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.

The content is expressed as a percentage by mass.

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.

NOTE 1 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.

4 Principle

Treatment of a test portion with an excess of *n*-hexane or light petroleum, then filtration of the solution obtained. Washing of the filter and residue with the same solvent, drying at 103 °C, and weighing.

5 Reagents

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 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. (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[2].

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).

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, 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) and 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, expressed as a percentage by mass, is equal to

$$(m_2 - m_1) \times \frac{100}{m_0}$$

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.

2) ISO 5555:1991, *Animal and vegetable fats and oils — Sampling.*

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 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, shall not exceed:

— 0,02 % (*m/m*) of insoluble impurities in the case of products containing not more than 0,3 % (*m/m*) of insoluble impurities; or

— 0,05 % (*m/m*) in other cases.

Reject both results if the difference exceeds the indicated repeatability level and carry out two new single determinations (clauses 8 and 9).

12 Test report

The test report shall specify

— the method in accordance with which sampling was carried out, if known,

— the method used,

— the solvent used,

— the test result(s) obtained.

It shall also mention all operating conditions not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the result.

The test report shall include all information necessary for the complete identification of the sample.

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