# International Standard 663

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

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

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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International Standard ISO 663 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in January 1979.

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It has been approved by the member bodies of the following countries :

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The member body of the following country expressed disapproval of the document on technical grounds :

Malaysia

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

It cancels and replaces ISO Recommendations ISO/R 663-1968 and ISO/R 932-1969, of which it constitutes a technical revision.

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

## 1 Scope and field of application

This International Standard specifies a method for the determination of the insoluble impurities content of animal or vegetable fats or oils.

The method is not applicable to cottonseed black grease or to sulphur olive oil.

## 2 Reference

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

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## 3 Definition

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**insoluble impurities content:** The dirt and other foreign matter insoluble in *n*-hexane or light petroleum under the conditions specified in this International Standard, and 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 — If it is not desired to include soaps (particularly calcium soaps) or oxidized fatty acids in the insoluble impurities, 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 ± 2 °C, and weighing.

## 5 Reagent

***n*-Hexane,** or, failing this, **light petroleum** having a distillation range between 30 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.

## 6 Apparatus

Usual laboratory apparatus, and in particular :

**6.1 Analytical balance.**

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

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

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

**6.5 Ashless filter paper** or **glass fibre filter,** diameter 120 mm, together with a metal (preferably aluminium) or glass **vessel** with a well-fitting lid; or

**6.6 Filter crucible,** pore diameter 10 to 16 µm, together with a **suction bottle.**

## 7 Procedure

### 7.1 Preparation of the test sample

Prepare the test sample in accordance with ISO 661.

### 7.2 Test portion

Weigh, to the nearest 0,01 g, approximately 20 g of the test sample (7.1) into the conical flask (6.3).

### 7.3 Determination

**7.3.1** Dry either the filter paper and the vessel (6.5) with its lid, or the filter crucible (6.6), in the oven (6.2), controlled at 103 ± 2 °C. Allow to cool in the desiccator (6.4) and weigh to the nearest 0,001 g.

**7.3.2** Add 200 ml of *n*-hexane or light petroleum (clause 5) to the flask containing the test portion (7.2), 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.

**7.3.3** 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 7.3.2, but not more than is necessary for the final filtrate to be free of fat or oil.

**7.3.4** 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 the air, and complete the evaporation in the oven at 103 ± 2 °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.

**7.3.5** If a filter crucible is used, allow most of the solvent remaining in it to evaporate in the air, and complete the evaporation in the oven at 103 ± 2 °C. Allow to cool in the desiccator (6.4) and weigh to the nearest 0,001 g.

NOTE — 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 should 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, should then be calculated by multiplying this difference in mass by $100/m_0$, where $m_0$ is the mass, in grams, of the test portion.

### 7.4 Number of determinations

Carry out two determinations on test portions taken from the same test sample (7.1).

## 8 Expression of results

### 8.1 Method of calculation and formula

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

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

where

$m_0$ is the mass, in grams, of the test portion (7.2);

$m_1$ is the mass, in grams, of the vessel with its lid and filter paper, or of the filter crucible (see 7.3.1);

$m_2$ is the mass, in grams, of the vessel with its lid and filter paper containing the dry residue (see 7.3.4), or of the filter crucible and dry residue (see 7.3.5).

Take as the result the arithmetic mean of the two determinations, provided that the requirement for repeatability (see 8.2) is satisfied.

Report the result to the second decimal place.

### 8.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,02 g of insoluble impurities per 100 g of sample in the case of products containing not more than 0,3 g of insoluble impurities per 100 g of sample, or 0,05 g per 100 g of sample in other cases.

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## 9 Test report

The test report shall show the method and solvent used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details required for the complete identification of the sample.