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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 663

CRUDE VEGETABLE OILS AND FATS

DETERMINATION OF INSOLUBLE IMPURITIES

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BRIEF HISTORY

The ISO Recommendation R 663, *Crude vegetable oils and fats – Determination of insoluble impurities,* was drawn up by Technical Committee ISO/TC 34, *Agricultural food products,* the Secretariat of which is held by the Magyar Szabványügyi Hivatal (MSZH).

Work on this question by the Technical Committee led in 1963, to the adoption of a Draft ISO Recommendation.

In March 1966, this Draft ISO Recommendation (No. 905) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Hungary	Romaina
Australia	India	South Africa, Rep. of
Belgium	Iran	Turkey
Bulgaria	Ireland	U.A.R.
Chile	Israel	United Kingdom
Colombia	Italy	U.S.S.R.
Czechoslovakia	Netherlands	Yugoslavia
Finland	New Zealand	
France	Norway	
Germany	Poland	

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in February 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 663 February 1968

CRUDE VEGETABLE OILS AND FATS

DETERMINATION OF INSOLUBLE IMPURITIES

1. SCOPE

This ISO Recommendation describes a method for the determination, in crude vegetable oils and fats, of insoluble impurities.

2. DEFINITION

By *insoluble impurities* is meant the dirt and other foreign matter, expressed as a percentage by mass, which are insoluble in *n*-hexane or light petroleum under the conditions specified.

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

3. PRINCIPLE

Treatment of the material with an excess of *n*-hexane or light petroleum, filtration of the solution, washing of the filtering system with the same solvent, drying at 103 ± 2 °C and weighing of the filtering system and dry residue.

4. REAGENT

n-Hexane; failing this, *light petroleum* distilling between 40 and 60 °C and having a bromine value below 1. For either solvent the residue on complete evaporation should not exceed 0.002 g/100 ml.*

5. APPARATUS

5.1 *Electric oven,* with temperature regulation.

5.2 *Analytical balance.*

5.3 *Metal vessel,* (preferably of aluminium), or *glass vessel* with a well fitting cover.

5.4 *Desiccator,* containing an efficient desiccant, such as phosphorus pentoxide, silica gel, activated alumina, etc.

* This ISO Recommendation has been prepared in the context of food products, and the choice of solvent has been limited for this reason.

6. PROCEDURE

6.1 Preparation of sample

The contract sample should be prepared in accordance with ISO Recommendation R 661, *Crude vegetable oils and fats – Preparation of contract sample for analysis.*

6.2 Test portion

Weigh, to the nearest 0.01 g, about 20 g of the prepared sample (see clause 6.1) into a conical flask with ground neck.

6.3 Determination

Add 200 ml of *n*-hexane or light petroleum (see section 4), stopper the flask and shake. For castor oil the quantity of solvent may be increased.

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

Filter through an ashless filter paper of 12 cm diameter, previously dried at 103 ± 2 °C and weighed in the vessel (5.3), or through a previously dried and tared filter crucible.

Wash the filter paper or filter crucible by pouring through it small amounts of solvent, but not more than is necessary for the final filtrate to be oil-free.

If ashless filter paper is used, remove the filter paper from the funnel, introduce it into the vessel (5.3), allow the solvent remaining in the filter paper to evaporate in the open air and complete the evaporation in the oven at 103 ± 2 °C. Remove from the oven, close the vessel with its cover, allow to cool in the desiccator (5.4) and weigh the filter paper and vessel.

If a filter crucible is used, evaporate the solvent as above and weigh the crucible without the vessel (5.3).

Carry out two determinations on the same prepared sample.

7. EXPRESSION OF RESULTS

7.1 Method of calculation and formula

Calculate the content of impurities insoluble in *n*-hexane or light petroleum, as a percentage by mass of the material as received, by means of the following formula :

$$\text{insoluble impurities, per cent by mass} = \frac{M_1 - M_2}{M_0} \times 100$$

where

M_0 is the mass, in grammes, of the test portion,

M_1 is the mass, in grammes, of the tared vessel and filter paper containing the dry residue, or of the filter crucible and dry residue,

M_2 is the mass, in grammes, of the tared vessel and filter paper or of the filter crucible.

Take as the result the arithmetic mean of two parallel determinations if the condition of repeatability is satisfied. Otherwise, take two other test portions, analyse one as above and keep the other for a fourth determination if necessary. In this case, take as the result the arithmetic mean of the result obtained by the third analysis and the nearer result obtained in the previous analyses, provided that the difference does not exceed the permitted limit. If it does, analyse also the fourth test sample and take as the result the arithmetic mean of the four determinations.

Report the result to one decimal place.