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# International Standard



# 734

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## Oilseed residues — Determination of hexane extract (or light-petroleum extract), called “oil content”

*Tourteaux de graines oléagineuses — Détermination de l'extrait à l'hexane (ou à l'éther de pétrole), dit «teneur en huile»*

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**Descriptors** : oilseeds, oilseed residues, chemical analysis, determination of content, oils, extraction analysis.

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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.

International Standard ISO 734 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in October 1977.

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

Australia	India	Poland
Canada	Iran	Portugal
Chile	Israel	Romania
Czechoslovakia	Korea, Rep. of	South Africa, Rep. of
Egypt, Arab Rep. of	Malaysia	Thailand
Ethiopia	Mexico	Turkey
France	Netherlands	United Kingdom
Germany, F. R.	New Zealand	USSR
Hungary	Peru	Yugoslavia

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

USA

This International Standard cancels and replaces ISO Recommendation R 734-1968, of which it constitutes a technical revision.

# Oilseed residues — Determination of hexane extract (or light-petroleum extract), called "oil content"

## 0 Introduction

As the determination of the "oil content" of oilseeds is carried out according to ISO 659, *Oilseeds — Determination of hexane extract (or light-petroleum extract), called "oil content"*, it has been considered necessary to provide for control of oil production by establishing a reference method for the determination of "oil content" of oilseed residues in the same way.

However, the principal users of oilseed residues, namely the manufacturers of animal feeding stuffs, carry out the determination by extraction with diethyl ether in some countries, but with *n*-hexane or light petroleum in others. The two methods do not always give the same results, and large amounts of data relating to the formulation of animal feeding stuffs are based on each of the two methods. It was therefore considered necessary to establish a separate International Standard dealing with the method using diethyl ether, and that is the subject of ISO 736, *Oilseed residues — Determination of diethyl ether extract*.

## 1 Scope and field of application

This International Standard specifies a method for the determination of the hexane extract (or light-petroleum extract), called "oil content", of residues (excluding compounded products) obtained by the extraction of oil from oilseeds by pressure or solvent.

## 2 Reference

ISO 771, *Oilseed residues — Determination of moisture and volatile matter content*.

## 3 Definition

**hexane extract, called "oil content"**: The whole of the substances extracted under the operating conditions specified below, and expressed as a percentage by mass of the product as received. On request it may be expressed relative to the dry matter.

## 4 Principle

Extraction of a test portion of the product in a suitable apparatus, with technical hexane or, failing this, light petroleum. Elimination of the solvent and weighing of the extract obtained.

## 5 Reagent

**5.1 Technical *n*-hexane** or, failing this, **light petroleum**, essentially composed of hydrocarbons with 6 carbon atoms, of which less than 5 % distils below 50 °C and more than 95 % distils between 50 and 70 °C, and which has a bromine value less than 1. For either solvent, the residue on complete evaporation shall not exceed 2 mg per 100 ml.

## 6 Apparatus

Usual laboratory apparatus and in particular:

**6.1 Mechanical grinder**, easy to clean and allowing the residues to be ground, without heating and without appreciable change in moisture, volatile matter and oil content, to particles passing completely through a sieve of aperture size 1 mm.

**6.2 Mechanical micro-grinder** (see 10.1), capable of producing a fineness of grinding of oilseed residues of less than 160 µm, with the exception of the "shell", particles of which may reach 400 µm.

**6.3 Extraction thimble and cotton wool, or filter paper**, free from matter soluble in hexane or light petroleum.

**6.4 Suitable extraction apparatus** (fitted with a flask of capacity 200 to 250 ml).

**6.5 Electric heating bath** (sand bath, water bath, etc.) or **hot-plate**.

**6.6 Electrically heated oven**, with thermostatic control, permitting ventilation or obtaining reduced pressure.

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

**6.8 Pumice stone**, in small particles, previously dried in an oven at  $130 \pm 2$  °C and cooled in a desiccator.

**6.9 Analytical balance**.

## 7 Sampling

The sampling of oilseed residues will form the subject of ISO 5500.

## 8 Procedure

### 8.1 Preparation of the test sample

**8.1.1** The preparation of analysis samples will form the subject of ISO 5502.

**8.1.2** Grind the analysis sample (8.1.1), if necessary, in the previously well-cleaned mechanical mill (6.1). First, use about one-twentieth of the sample to complete the cleaning of the mill, and reject these grindings; then grind the rest, collect the grindings, mix carefully and carry out the analysis without delay.

### 8.2 Test portion

**8.2.1** Weigh, to the nearest 0,01 g, about 10 g of the test sample (8.1.2).

**8.2.2** Transfer this test portion to the extraction thimble (6.3) and close the latter with a wad of cotton wool (6.3). If a filter paper is used, wrap the test portion in it.

### 8.3 Pre-drying

If the test portion is very moist [moisture and volatile matter content more than 10 % (*m/m*)], leave the filled thimble for some time in an oven, controlled at a temperature not higher than 80 °C, to reduce the moisture and volatile matter content to less than 10 % (*m/m*). (See 10.2.)

### 8.4 Determination

#### 8.4.1 Preparation of the flask

Weigh, to the nearest 1 mg, the flask of the extraction apparatus (6.4) containing one or two particles of pumice stone (6.8).

#### 8.4.2 First extraction

Place the thimble (6.3) containing the test portion in the extraction apparatus (6.4). Pour into the flask the necessary quantity of solvent (5.1). Fit the flask to the extraction apparatus on the electric heating bath or hot-plate (6.5). Carry out the heating so that the rate of reflux is at least 3 drops per second (boiling moderately, not violently).

After extracting for 4 h, allow to cool. Remove the thimble from the extraction apparatus and place it in a current of air in order to expel the greater part of the residual solvent.

#### 8.4.3 Second extraction

Empty the thimble into the micro-grinder (6.2) and grind as finely as possible. (See 10.1.) Put the mixture back into the thimble and put the latter back into the extraction apparatus; re-extract for a further 2 h, using the same flask containing the first extract.

The solution obtained in the extraction flask shall be clear. If it is not, filter it through a filter paper, collecting the filtrate in another previously dried and tared flask, and washing the first flask and filter paper several times with the same solvent.

#### 8.4.4 Elimination of solvent and weighing of the extract

Expel the greater part of the solvent from the flask by distillation on the electric heating bath or the hot-plate. Expel the last traces of solvent by heating the flask for about 20 min in the electrically heated oven (6.6) at  $103 \pm 2$  °C<sup>1)</sup>. Assist the removal either by blowing air or, preferably, an inert gas (such as nitrogen or carbon dioxide) into the flask for short periods, or by reducing the pressure in the flask.<sup>2)</sup>

Allow the flask to cool in the desiccator (6.7), for at least 1 h, to ambient temperature and weigh to the nearest 1 mg.

Heat again for about 10 min under the same conditions; allow to cool and weigh.

The difference between the two weighings shall not exceed 10 mg. If it does, repeat the operations of heating for 10 min, cooling and weighing until the difference between two successive weighings does not exceed 10 mg. Note the final mass of the flask.

### 8.5 Number of determinations

Carry out two determinations on the same test sample.

1) In the case of residues rich in volatile acids (residues from copra, palm kernel etc.), drying of the extract shall be carried out at atmospheric pressure, and at 80 °C maximum.

2) In the case of drying or semi-drying oilseed residues, it is preferable to remove the residual solvent by drying under reduced pressure.

## 9 Expression of results

### 9.1 Method of calculation and formulae

9.1.1 The "oil content", expressed as a percentage by mass of the product as received, is equal to

$$\frac{m_1}{m_0} \times 100$$

where

$m_0$  is the mass, in grams, of the test portion (8.2.1);

$m_1$  is the mass, in grams, of the extract after drying (see 8.4.4).

Take as the result the arithmetic mean of the two determinations, provided that the requirement concerning repeatability (see 9.2) is satisfied. Otherwise, repeat the determination on two other test portions. If this time the difference still exceeds 0,2 g per 100 g of sample, take as the result the arithmetic mean of the four determinations carried out.

Express the result to one decimal place.

9.1.2 On request, the "oil content" may be expressed as a percentage by mass of the dry matter; it is then equal to

$$H_0 \times \frac{100}{100 - U}$$

where

$H_0$  is the percentage, by mass, of oil in the product as received (calculated according to 9.1.1);

$U$  is the percentage, by mass, of moisture and volatile matter, determined as specified in ISO 771.

### 9.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst should not exceed 0,2 g of oil per 100 g of sample.

## 10 Notes on apparatus and procedure

10.1 In laboratories where a micro-grinder (see 6.2) is not available, micro-grinding of the ground sample (see 8.4.3) may be replaced by trituration with a pestle and mortar, in the presence of about 10 g of sand which has been washed with hydrochloric acid and then calcined. However, grinding in a mortar cannot be applied in the case of multiple analyses because operator fatigue prevents sufficiently efficient grinding of numerous samples, and the extraction of oil from a coarsely ground sample can never be complete.

10.2 As an alternative to the pre-drying procedure described in 8.3, the test portion (8.2.1) may be mixed in a suitable vessel with 2 to 3 g of analytical quality anhydrous sodium sulphate per 5 g of grindings. Continue as indicated in 8.2.2 and 8.4.

### 11 Test report

The test report shall show the method used, the result obtained and the solvent used, indicating clearly whether the result represents the "oil content" of the product as received or the "oil content" in relation to the dry matter. It shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as all circumstances that may have influenced the result.

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

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