
**Oilseed residues — Determination of oil
content**

Part 1:

Extraction method with hexane (or light
petroleum)

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*Tourteaux de graines oléagineuses — Détermination de la teneur en
huile*

Partie 1: Méthode par extraction à l'hexane (ou à l'éther de pétrole)

ISO 734-1:1998

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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.

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 734-1 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 2, *Oleaginous seeds and fruits*.

This first edition of ISO 734-1, together with ISO 734-2, cancels and replaces ISO 734:1979, which has been technically revised.

ISO 734 consists of the following parts, under the general title *Oilseed residues — Determination of oil content*:

- *Part 1: Extraction method with hexane (or light petroleum)*
- *Part 2: Rapid extraction method*

Annexes A and B of this part of ISO 734 are for information only.

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Introduction

As the determination of the "oil content" of oilseeds is carried out according to ISO 659 [1], it has been considered necessary to provide for the 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 [2].

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Oilseed residues — Determination of oil content —

Part 1:

Extraction method with hexane (or light petroleum)

1 Scope

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 734. At the time of the publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 734 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 771:1977, *Oilseed residues — Determination of moisture and volatile matter content.*

ISO 5502:1992, *Oilseed residues — Preparation of test samples.*

3 Definition

For the purposes of this part of ISO 734, the following definition applies.

3.1

oil content

whole of the substances extracted under the operating conditions specified in this part of ISO 734, and expressed as a percentage by mass of the product as received

NOTE On request, the oil content may be expressed relative to 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 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Technical hexane, *n*-hexane or 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 °C and 70 °C.

For either solvent, the residue on complete evaporation shall not exceed 2 mg per 100 ml.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

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 obtain particles passing completely through a sieve of aperture size 1 mm.

6.2 Mechanical microgrinder, of the Danguomau type¹⁾ capable of producing a fineness of grinding of oilseeds residues of less than 160 µm, with the exception of the "shell", particles of which may reach 400 µm.

NOTE In laboratories where a microgrinder is not available, microgrinding of the ground sample (see 9.3.3) 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.

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 ml to 250 ml.

NOTE Straight-through extractors, for example the Butt, Smalley, Twisselmann and Bolton-Williams²⁾, are suitable.

The use of other extractors is conditional upon the results of a test on a standard material of known oil content to confirm the suitability of the apparatus.

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, capable of being maintained at 103 °C ± 2 °C.

6.7 Desiccator, containing an efficient desiccant.

6.8 Pumice stone, in small particles, previously dried in an oven at 103 °C ± 2 °C then cooled in a desiccator.

6.9 Analytical balance, capable of weighing to an accuracy of ± 0,001 g.

¹⁾ The Danguomau mechanical microgrinder is example of suitable product available commercially. This information is given for the convenience of users of this part of ISO 734 and does not constitute an endorsement by ISO of this product.

²⁾ The Butt, Smalley, Twisselmann or Bolton-Williams straight-through extractors are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 734 and does not constitute an endorsement by ISO of these products.

7 Sampling

Sampling is not part of the method specified in this part of ISO 734. A recommended sampling is given in ISO 5500 [3].

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

8 Preparation of test sample

8.1 Prepare the test sample in accordance with ISO 5502.

8.2 Grind the analysis sample (8.1), 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.

9 Procedure

NOTE If it is required to check whether the repeatability limit (11.2) is met, carry out two single determinations in accordance with 9.1 to 9.3.4.

9.1 Test portion

9.1.1 Weigh, to the nearest 0,001 g, about 10 g of the test sample (8.2).

9.1.2 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.

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9.2 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, maintained at a temperature not higher than 80 °C, to reduce the moisture and volatile matter content to less than 10 % (*m/m*).

As an alternative to the pre-drying procedure described above, the test portion (9.1.1) may be mixed in a suitable vessel with 2 g to 3 g of analytical quality anhydrous sodium sulfate per 5 g of grindings. Continue as indicated in 9.1.2 and 9.3.

9.3 Determination

9.3.1 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).

9.3.2 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.

9.3.3 Second extraction

Empty the thimble into the microgrinder (6.2) and grind as finely as possible. 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.

9.3.4 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 (6.5). Expel the last traces of solvent by heating the flask for about 20 min in the electrically heated oven (6.6) set at 103 °C.

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

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.

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

Allow the flask to cool in the desiccator (6.7), for at least 1 h, to ambient temperature then 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.

10 Expression of results

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

$$H = \frac{m_1}{m_0} \times 100 \%$$

where

m_0 is the mass, in grams, of the test portion (9.1.1);

m_1 is the mass, in grams, of the extract after drying (see 9.3.4).

Express the result to one decimal place.

10.2 On request, the oil content may be expressed as a percentage by mass of the dry matter. It is then equal to

$$H_D = H \times \frac{100 \%}{100 \% - U}$$

where

H is the percentage, by mass, of oil in the product as received (calculated according to 10.1);

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

11 Precision

11.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in annex A. The values derived from this interlaboratory test 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 0,3 g per 100 g.

11.3 Reproducibility

The absolute difference between two 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 0,6 g per 100 g.

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 part of ISO 734;
- all operating details not specified in this part of ISO 734, or regarded as optional, together with details of any incidents which may have influenced the test result(s);
- the test result(s) 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;
- if the repeatability has been checked, the final quoted result obtained.