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Third edition
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Oilseed meals — Determination of oil content — Extraction method with hexane (or light petroleum)

Tourteaux de graines oléagineuses — Détermination de la teneur en huile — Méthode par extraction à l'hexane (ou à l'éther de pétrole)

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 2, *Oleaginous seeds and fruits and oilseed meals*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 307, *Oilseeds, vegetable and animal fats and oils and their by-products — Methods of sampling and analysis,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 734:2015), which has been technically revised in order to include some safety warnings.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

A method for the determination of the oil content of oilseeds is specified in ISO 659. To provide for the control of oil production, this document specifies a reference method for the determination of the oil content of oilseed meals in the same way.

Oilseed meals — Determination of oil content — Extraction method with hexane (or light petroleum)

1 Scope

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 5502, *Oilseed residues — Preparation of test samples*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
oil content
all of the substances extracted under certain operating conditions of the product as received

Note 1 to entry: For the purposes of this document, the operating conditions are those specified in this document.

Note 2 to entry: It is expressed as a mass fraction, in per cent.

Note 3 to entry: The oil content may also be expressed relative to dry matter.

4 Principle

A test portion of the product is extracted in a suitable apparatus, with technical hexane or, failing this, light petroleum. The solvent is eliminated and the extract obtained is weighed.

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 six carbon atoms, of which less than 5 % (volume/volume) distils below 40 °C and more than 95 % (volume/volume) distils between 40 °C and 60 °C or between 50 °C and 70 °C, and which has a bromine value of less than 1. The residue on complete evaporation shall not exceed 2 mg per 100 ml.

6 Apparatus

WARNING — All reflux or boiling of solvent shall be carried out in a chemical fume hood to minimize employee exposure to solvent fumes.

The usual laboratory apparatus and, in particular, the following shall be used.

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

6.2 Mechanical microgrinder, of the Dangoumau type[1] capable of producing a fineness of grinding of oilseed meals of less than 160 µm, with the exception of the "shell" whose particles may reach 400 µm.

In laboratories where a microgrinder is not available, microgrinding of the ground sample (see 9.4.3) may be replaced by trituration with a pestle and mortar, in the presence of about 10 g of sand that 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**, or **glass wool** 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, e.g. the Butt, Smalley, Twisselmann and Bolton-Williams[2] 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 Rotary evaporator, electric heating bath (e.g. sand bath, water bath) or **hot plate**.

The apparatus used shall be suitable for working with potentially flammable solvents.

6.6 Electrically heated oven, with thermostatic control, permitting ventilation or obtaining reduced pressure, capable of being maintained at 103 °C ± 2 °C.

The apparatus used shall be suitable for working with potentially flammable solvents.

6.7 Desiccator, containing an efficient desiccant.

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

6.9 Analytical balance, capable of weighing to the nearest 0,001 g.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

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[1] The Dangoumau mechanical microgrinder is an example of a suitable product available commercially. This information is given for the convenience or users of this document and does not constitute an endorsement by ISO of the product named.

[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 document and does not constitute an endorsement by ISO of the products named.

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 5500.

8 Preparation of test sample

8.1 Prepare the test sample in accordance with ISO 5502.

8.2 If necessary, grind the test sample 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

9.1 Number of determinations

If it is required to check whether the repeatability (see 11.2) is met, carry out two single determinations in accordance with 9.2 to 9.4.4.

9.2 Test portion

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

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

9.3 Pre-drying

If the test portion is very moist [moisture and volatile matter content more than 10 % (mass fraction)], 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 % (mass fraction).

As an alternative to the pre-drying procedure described above, the test portion (see 9.2.1) 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 9.2.2 and 9.4.

9.4 Determination

9.4.1 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).

9.4.2 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 three 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.

9.4.3 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, then wash the first flask and filter paper several times with the same solvent.

9.4.4 Elimination of solvent and weighing of the extract

Expel the greater part of the solvent from the flask using a rotary evaporator or by distillation on the electric heating bath or the hot plate (6.5).

NOTE The rotary evaporator permits to minimize employee exposure to solvent fumes and allows those solvents to be properly discarded rather than vented to the atmosphere.

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.

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

Assist the removal of solvent 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.

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

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

Heat again for about 10 min under the same conditions. Allow to cool and reweigh.

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 of the product as received, w, expressed as a mass fraction, in per cent, is equal to Formula (1):

$$w = \frac{m_1}{m_0} \times 100 \qquad (1)$$

where

m_0 is the mass, in grams, of the test portion (see 9.2.1);

m_1 is the mass, in grams, of the extract after drying (see 9.4.4).

Express the result to one decimal place.

10.2 On request, the oil content may be expressed as a mass fraction, in per cent, of the dry matter, w_D. It is then equal to Formula (2):

$$w_D = w \times \frac{100}{100 - w_M} \tag{2}$$

where

w is the mass fraction, in per cent, of oil in the product as received (calculated according to 10.1);

w_M is the mass fraction, in per cent, 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 are not necessarily 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 the value of the repeatability limit *r* given in Table 1.

11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will in not more than 5 % of cases exceed the value of the reproducibility limit *R* given in Table 1.

Table 1 — Repeatability and reproducibility limits

Sample	Mean value oil content % (mass fraction)	*r* % (mass fraction)	*R* % (mass fraction)
Rapeseed meal	0 to 5	0,3	1,1
Soya and sunflower meals	0 to 5	0,2	0,7

12 Test report

The test report shall specify at least the following:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this document, i.e. ISO 734:2023;

d) all operating details not specified in this document, or regarded as optional, together with details of any incidents which can have influenced the test result(s);

e) 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;

f) if the repeatability has been checked, the final quoted result obtained;

g) the date of the test.