
Oilseed meals — Determination of oil content — Rapid extraction method

*Tourteaux de graines oléagineuses — Détermination de la teneur en
huile — Méthode d'extraction rapide*

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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.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 34, *Food products*, Subcommittee SC 2, *Oleaginous seeds and fruits and oilseed meals*.

This first edition cancels and replaces ISO 734-2:2008, which has been renumbered and editorially revised.

Oilseed meals — Determination of oil content — Rapid extraction method

1 Scope

This International Standard specifies an extraction method which may be used to assess the efficiency of a de-oiling process by comparing the oil content of the oilseed with the residual oil content of the corresponding extraction meals, pellets and expeller cakes.

It is not applicable to disputed cases, for which ISO 734 is applicable.

It is applicable to oilseed meals obtained from oilseeds by expelling or by extraction with a solvent, as well as to the pellets made from the residues.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 residues — 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.

3.1

oil content

sum of the mass fractions of the substances extracted under the operating conditions specified in this International Standard on the basis of the product as received

Note 1 to entry: The mass fraction is expressed as a percentage.

Note 2 to entry: On request, the oil content may be expressed relative to dry matter.

4 Principle

The test portion is ground in a micro-ball mill in the presence of a solvent and subsequently extracted with the same solvent in a suitable apparatus. The solvent is removed from the extract by distillation, then the residue is weighed after drying.

5 Reagents and materials

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

5.1 Technical hexane, *n*-hexane or light petroleum ether, essentially composed of hydrocarbons with six 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.

The solvent recovered from the extract by distillation should not be used for further determinations.

5.2 Cotton wool, fat-free.

EXAMPLE Defatted cotton wool for ophthalmic use.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Analytical balance, capable of being read to the nearest 0,001 g.

6.2 Drying oven, capable of being maintained at $103\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

6.3 Mechanical microgrinder, capable of wet grinding in the presence of light petroleum ether and reducing the test portion to an average particle size of less than $10\text{ }\mu\text{m}$. A commercially available ball mill¹⁾ is suitable.

When using grinding beakers made of polytetrafluoroethylene, cooling is obligatory. A suitable cooling jacket is shown in [Figure 1](#).

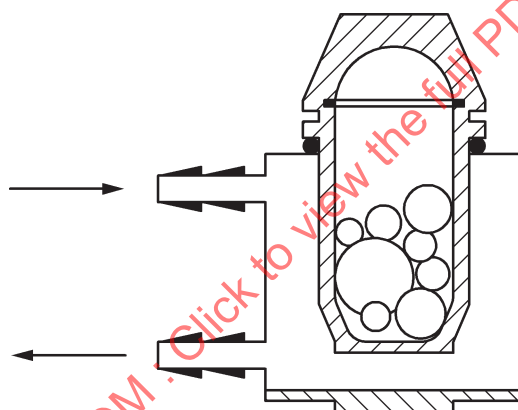


Figure 1 — Grinding beaker with cooling mantle

6.4 Funnel, made of light petroleum ether-resistant plastic, with a diameter of 70 mm, a stem external diameter of 10 mm and a stem length of 100 mm to 150 mm.

The stem shall just reach into the extraction thimble on placing the funnel in the extraction apparatus as described in [9.3.4](#). There is a fixed metal pin in the funnel to retain the stainless steel balls (see [Figure 2](#)).

1) The Dangoumau ball mill is an example of a suitable product available commercially and has been studied in a ring test ([Annex A](#)). This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

Dimensions in millimetres

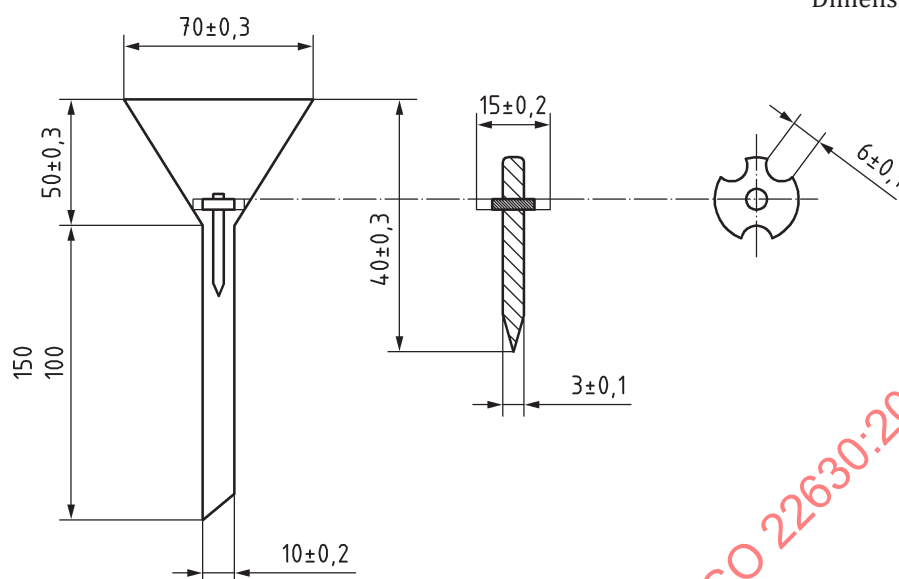


Figure 2 — Funnel with stanced metal pin

6.5 Continuous extraction apparatus²⁾, with ground joints, consisting of a flat bottomed flask of capacity 100 ml to 200 ml, a connecting tube (extractor) to hold the extraction thimble, and a reflux condenser.

The apparatus should include a suitable heating element for boiling technical hexane under reflux conditions. This might be a water bath, steam bath or safety-approved electrical heating unit.

NOTE 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.

6.6 Extraction thimbles, of dimensions 25 mm × 100 mm, of wall thickness 1,5 mm and a reinforced bottom part.

7 Sampling

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

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5500.^[1]

8 Preparation of test sample

Prepare the test sample in accordance with ISO 5502.

2) The Twisselmann and Butt extractors are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products. The Butt-type extractor is described in AOCS Official method Aa 4-38.^[4]

9 Procedure

9.1 General

If it is required to check whether the repeatability limit (11.2) is met, carry out two single determinations in accordance with 9.2 to 9.3.11.

9.2 Test portion

Weigh, to the nearest 0,001 g, 5 g of the well-mixed test sample in an extraction thimble (6.6).

9.3 Determination

9.3.1 Transfer the contents of the thimble to the beaker or tube of the mechanical microgrinder (6.3) containing all stainless-steels balls.

9.3.2 Add about 20 ml of solvent (5.1). Close the grinding beaker or tube and shake in the microgrinder (6.3) for 10 min or shake the stainless steel tubes on a flat bed shaker for 45 min horizontally at a frequency of 240 min⁻¹, with a horizontal displacement of 35 mm.

9.3.3 Place the thimble (6.6) in the connecting tube (extractor) of the extraction apparatus (6.5) and connect this to a dried and weighed flask.

9.3.4 Place the funnel (6.4) in the connecting tube in such a manner that the stem of the funnel reaches into the upper quarter of the thimble.

9.3.5 Then pour the contents of the grinding beaker through the funnel into the extraction thimble in such a manner that the balls remain on the metal pin in the funnel. Carefully rinse the grinding beaker, its lid and the balls with solvent in order to transfer all sample particles quantitatively into the extraction thimble. This requires about 50 ml of solvent, the total volume of which is therefore about 70 ml.

9.3.6 Cover the contents of the extraction thimble with a wad of cotton wool (5.2). Connect the flask and connecting tube (extractor) to the condenser and place the whole apparatus on the heating apparatus.

9.3.7 Extract for 1 h, calculated from when the solvent begins to boil, while maintaining a reflux rate of at least 5 ml/min.

9.3.8 Collect the bulk of the solvent in the collecting vessel of the extraction apparatus using the appropriate actions. Discard recovered solvent.

9.3.9 Remove the flask from the extraction apparatus and leave for about 5 min on the boiling water bath to evaporate any residual solvent.

9.3.10 Dry the flask containing the oil for 1,5 h in the drying oven (6.2) maintained at 103 °C, and weigh after cooling to room temperature.

CAUTION — Drying of highly unsaturated oils (e.g. flaxseed oils) may cause the oils to start oxidizing. This might require special attention if the oils are required for other tests.

9.3.11 Dry the flask for a further 10 min at the same temperature and weigh after cooling. The difference between the two weighings shall not exceed 0,01 g. If this is not the case, repeat the drying and weighing procedures.

10 Expression of results

10.1 Calculate the oil content, w_o , expressed as a percentage mass fraction, in the test sample, from Formula (1):

$$w_o = \frac{m_1}{m_0} \times 100 \quad (1)$$

where

m_0 is the mass, in grams, of the test sample (9.2);

m_1 is the mass, in grams, of the extract after drying (9.3.10).

Express the result to one decimal place.

10.2 On request, the oil content may be expressed as a percentage mass fraction of the dry matter, w_{od} , derived from Formula (2):

$$w_{od} = w_o \times \frac{100}{100 - w_{mv}} \quad (2)$$

where

w_{mv} is the percentage mass fraction 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 exceed 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 with 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 $\bar{w}_o$ % mass fraction	Repeatability limit r % mass fraction	Reproducibility limit R % mass fraction
Rapeseed, soya, and sunflower meals	0 to 5	0,3	0,8

12 Test report

The test report shall contain at least the following information:

- 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 International Standard, i.e. ISO 22630;
- d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);
- e) the test result(s) obtained;
- f) if the repeatability has been checked, the final quoted result obtained.

Annex A (informative)

Results of interlaboratory test

An international collaborative test was organized in 1994 by the German member body (DIN) involving 11 laboratories, each laboratory having performed a duplicate analysis of each sample.

The test was carried out on three samples:

- rapeseed meal;
- soya meal;
- sunflower meal.

The results obtained were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to give the precision data shown in [Table A.1](#).

Table A.1 — Results of the interlaboratory test

Parameter	Sample		
	Rapeseed meal	Soya meal	Sunflower meal
No. participating laboratories	11	11	11
No. laboratories retained after eliminating outliers	10	10	11
No. test results in all laboratories	20	20	22
Mean value of oil content, % mass fraction	3,90	1,33	2,93
Repeatability standard deviation, s_r	0,08	0,04	0,07
Coefficient of variation of repeatability, $CV(r)$, %	1,9	2,6	2,4
Repeatability limit, r	0,21	0,10	0,20
Reproducibility standard deviation, s_R	0,23	0,13	0,29
Coefficient of variation of reproducibility, $CV(R)$, %	5,8	10,0	9,7
Reproducibility limit, R	0,64	0,37	0,80