INTERNATIONAL STANDARD

ISO 734-2

First edition
1998-08-15

Oilseed residues — Determination of oil content —

Part 2:
Rapid extraction method

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

Partie 2: Méthode rapide par extraction

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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.

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

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This first edition of ISO 734-2, together with ISO 734-1, 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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International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

Oilseed residues — Determination of oil content —

Part 2:
Rapid extraction method

1 Scope

This part of ISO 734 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 applicable to oilseed residues obtained from oilseeds by expelling or by extraction with a solvent, as well as to the pellets made from the residues.

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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

Pulverization of the sample in a micro-ball mill in the presence of a solvent and subsequent extraction with the same solvent in a suitable apparatus. Removal of the solvent from the extract by distillation, then weighing of the residue after drying.

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.

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

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

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

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

6.3 Micro-ball mill, of the Dangoumau type[1].

6.4 Grinding beaker, made of stainless steel or polytetrafluoroethylene, and of capacity 65 ml (see figure 1), with stainless-steel balls.

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The grinding beaker can (as depicted in figure 1) be surrounded by a cooling mantle through which water circulates during grinding. When using grinding beakers made of polytetrafluoroethylene, cooling is obligatory.

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6.5 Funnel, made of light-petroleum-resistant plastic, with a diameter of 70 mm, a stem external diameter of 10 mm and 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 9.2.4. There is a fixed metal pin in the funnel to retain the stainless-steel balls (see figure 2).

6.6 Continuous extraction apparatus, of the Twisselmann type[2] with ground joints, consisting of a flat-bottomed flask of 100 ml to 200 ml capacity, a connecting tube (extractor) to hold the extraction thimble, and a reflux condensor (see figure 3).

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.

6.7 Extraction thimbles, of dimensions 25 mm x 100 mm, with wall strength 1,5 mm and reinforced bottom part.

6.8 Water bath, electrically heated and explosion-proof.

6.9 Steam bath.

6.10 Cotton wool, fat-free.

NOTE For example, defatted cotton wool for ophthalmic use.

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1) The Dangoumau mill is an example of a suitable product available commercially.

2) Twisselmann continuous extraction apparatus is an example of a suitable apparatus 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 method is given in ISO 5500 [1].

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

Prepare the test sample in accordance with ISO 5502.

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 9.2.11.

9.1 Test portion

Weigh, to the nearest 0,001 g, 5 g of the well-mixed test sample in an extraction thimble (6.7).

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9.2 Determination

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9.2.1 Transfer the contents of the thimble to the grinding beaker (6.4) of the micro-ball mill containing all the stainless-steel balls.

9.2.2 Add about 20 ml of solvent (5.1). Close the grinding beaker and shake in the ball mill (6.3) for 10 min. (See 6.4 for warning about cooling.)

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

9.2.4 Place the funnel (6.5) in the connecting tube in such a manner that the stem of the funnel reaches into the upper quarter of the thimble.

9.2.5 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.

9.2.6 Cover the contents of the extraction thimble with a wad of cotton wool (6.10). Connect the flask and connecting tube (extractor) to the condenser and place the whole apparatus in a boiling water bath (6.8) or steam bath (6.9).

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

9.2.8 Then close the cock of the Twisselmann condenser and collect the bulk of the solvent in the collecting vessel of the extraction apparatus. (See note in 5.1.)

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

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

9.2.11 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, H, expressed in grams per 100 g of the test sample, from the following equation

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

where

m_0 is the mass of the test sample, in grams (9.1);

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

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

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

where

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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;

— if the repeatability has been checked, the final quoted result obtained.

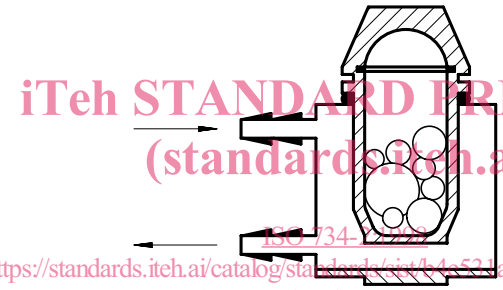

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Figure 1 — Grinding beaker with cooling mantle

Dimensions in millimetres

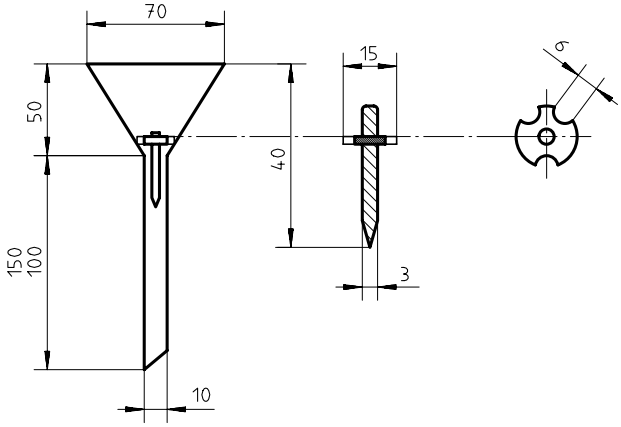

Figure 2 — Funnel with stanced metal pin

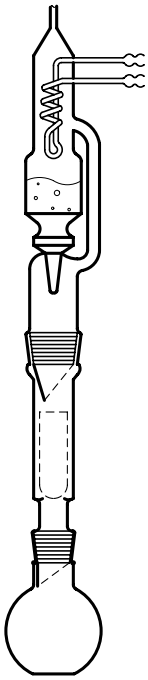

Figure 3 — Extraction apparatus according to Twisselmann

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Annex A
(informative)

Results of an interlaboratory test

An international collaborative test was organized in 1994 by the Institut für Chemie und Physik der Fette (BAGKF – Germany), 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;

and the results obtained were subjected to statistical analysis in accordance with ISO 5725 [2][3] to give the precision data shown in table A.1.

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Table A.1 — Results of interlaboratory test

Parameter	Sample		
	Rapeseed meal	Soya meal	Sunflower meal
Number of laboratories retained after eliminating outliers	11	11	11
Number of accepted laboratories	8	8	7
Mean oil content [% (*m/m*)]	3,9	1,4	3,1
Repeatability standard deviation, s_r [% (*m/m*)]	0,1	0,1	0,1
Repeatability coefficient of variation (%)	6,2	10,1	5,9
Repeatability limit, r (2,8 s_r) [% (*m/m*)]	0,2	0,1	0,2
Reproducibility standard deviation, s_R [% (*m/m*)]	0,1	0,1	0,1
Reproducibility coefficient of variation (%)	10,6	16,6	7,9
Reproducibility limit, R (2,8 s_R) [% (*m/m*)]	0,4	0,2	0,2

3) ISO 5725:1986 (now withdrawn) was used to obtain the precision data.