
**Oilseeds — Determination of oil content
(Reference method)**

Graines oléagineuses — Détermination de la teneur en huile (Méthode de référence)

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ISO 659:1998

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Foreword

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

This second edition cancels and replaces the first edition (ISO 659:1988), which has been technically revised.

Annexes A and B of this International Standard 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

Oilseeds — Determination of oil content (Reference method)

1 Scope

This International Standard specifies a reference method for the determination of the hexane extract (or light petroleum extract), called “oil content”, of oil seeds used as industrial raw materials.

NOTE If required, the following may be analysed separately:

- the pure seeds and the impurities (see 9.3);
- in the case of groundnuts, the pure seeds, the total fines, the non-oleaginous impurities and the oleaginous impurities.

The method has been tested on rapeseed, soya beans and sunflower seeds. This does not, however, preclude its applicability to other commercial seeds. (standards.iteh.ai)

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2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of the publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the International Standard 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 658:1988, *Oilseeds — Determination of impurities*.

ISO 664:1990, *Oilseeds — Reduction of laboratory sample to test sample*.

ISO 665:1977, *Oilseeds — Determination of moisture and volatile matter content*.

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 hexane extract, called “oil content”

all the substances extracted under the operating conditions specified in this International Standard, expressed as a percentage by mass of the product as received [*tale quale*] or on the cleaned seed.

NOTE On request, it may be expressed relative to the dry matter.

4 Principle

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

5 Reagent

5.1 Technical hexane, *n*-hexane or light petroleum, essentially composed of hydrocarbons with 6 carbon atoms, of which less than 5 % distils below 40 °C and more than 95 % distils between 40 °C and 60 °C or between 50 °C and 70 °C. 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 Analytical balance.

6.2 Mechanical mill, easy to clean, appropriate for the nature of the oilseeds, and allowing the oilseeds to be ground to a uniform particle size without heating or appreciable change in moisture, volatile matter or oil content.

NOTE The following mills have been found to give satisfactory results:

- the Christy Norris 8" Laboratory Mill¹⁾ with perforated plate of bar-type screens depending on seed type (e.g. 0,8 mm perforated plate, 3 mm and 6 mm bar screens; see 8.3.2, 8.3.3 and 8.3.4);
- the Ultra Centrifugal Mill (UCM) (Glen Creston)¹⁾ with 1 mm screen for sunflower seeds and other screens according to seed type.

Other mills may be satisfactory, provided they conform to the criteria given above.

6.3 Mechanical micro-grinder, capable of producing a fineness of grinding of the oilseeds of less than 160 µm, with the exception of the "shell", particles of which may reach 400 µm.

NOTE A "Dangoumau" type analytical grinder²⁾ with a 150 ml cylinder and 1 cm, 2 cm and 3 cm diameter steel balls (approximately 7 g, 30 g and 130 g) according to the sample being ground, has been found to be suitable.

The following microgrinders have also been found to be suitable:

- Retsch²⁾, IE Retsch²⁾, Planetary Ball Mill²⁾, S1 and S2 Centrifugal Ball Mill²⁾, Batam Mikro Pulverizer Hammer Mill²⁾, IKA Mill²⁾, Fritch Pulverisette 5 Planetary Ball Mill²⁾ and Steam Mill²⁾.

6.4 Extraction thimble and cotton wool, free from matter soluble in hexane or light petroleum.

6.5 Suitable extraction apparatus, fitted with a flask of capacity 200 ml to 250 ml.

1) The Christy Norris 8" Laboratory Mill and the Ultra Centrifugal Mill (UCM) (Glen Creston) 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.

2) The above-mentioned microgrinders 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.

- 3) NOTE Straight-through extractors, for example the Butt, Smalley or Bolton-Williams type, 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.
- 6.6 Pumice stone**, in small particles, or other anti-bumping granules, previously dried in an oven at $(130 \pm 2) ^\circ\text{C}$ and cooled in a desiccator.
- 6.7 Apparatus for safely removing solvent from extraction thimble** (for example in a current of warm air).
- 6.8 Electric heating bath** (sand bath, water bath, heating mantel, etc.) or **hot-plate**.
- 6.9 Electrically heated oven**, with thermostatic control, capable of being maintained at $(103 \pm 2) ^\circ\text{C}$. The oven should be capable of being used at either atmospheric or reduced pressure (see 9.2.3).
- 6.10 Desiccator**, containing an efficient desiccant (e.g. silica gel with blue indicator, or P_2O_5).
- 6.11 Electrically heated oven**, capable of being maintained at $(130 \pm 2) ^\circ\text{C}$ (for cottonseed, see 8.3.5).
- 6.12 Metal dish**, flat-bottomed, of diameter 100 mm and height approx. 40 mm.

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7 Sampling

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Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 542.

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

8 Preparation of test sample

8.1 Reduction of laboratory sample

Prepare the test sample in accordance with ISO 664. If large non-oleaginous foreign bodies have been separated before reduction of the laboratory sample, make allowance for this in the calculation (see 10.1.3). According to the requirements of the contract, take a sample as received or after separation of the impurities.

8.2 Predrying

8.2.1 The moisture content of the test portion (9.1) shall be less than 10 % (m/m) when extraction of the oil (9.2) is commenced.

NOTE Failure to observe this may result in an incorrect result and invalidate the analysis.

3) The Butt, Smalley or Bolton-Williams straight-through 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.

Use a rapid screening method to assess the moisture content of the analysis sample (8.1). If it is greater than 10 % (*m/m*), reduce it to less than 10 % (*m/m*) by drying the analysis sample (8.1) in a dish in an oven maintained at a temperature not greater than 80 °C. Store the partially dried material in an air-tight jar. Determine the moisture contents of the partially dried analysis sample and the original sample according to the method given in ISO 665 and make allowance for this in the final calculation (see 10.1.6).

8.3 Test sample

8.3.1 Preliminary remarks

It is essential that the oil extractions are carried out within 30 min of grinding, especially if the free fatty acids content of the extracted oil is to be determined.

Care shall be taken to clean all mills thoroughly before and after each sample has been ground. Any material adhering to the mill shall be incorporated with the bulk of the ground material.

NOTE In the following test “seed” or “kernels” implies both whole seeds or kernels and broken fragments of them.

8.3.2 Palm kernels

Shell is considered to be part of the impurities, even when attached to the kernels. Shell and dirt shall be separated quantitatively from the kernels before analysis.

Shell is hard and difficult to mill. It is almost impossible to obtain a homogeneous sample if kernels and impurities are milled together. If the oil content of the whole sample [*tale quale*] is required, analyse the kernels and impurities (shell and dirt) separately and calculate the oil content (see 10.1.2).

Prepare the test samples by grinding separately, 600 g of the well-mixed kernels, and all of the shell and dirt separated during the quantitative determination of impurities according to the method given in ISO 658.

The following milling techniques have been found to be suitable.

- for pure kernels: grind in a Christy Norris Laboratory Mill⁴⁾ (6.2) with a 6 mm bar screen;
- for shell and dirt: grind for 10 min in a microgrinder (6.3) using a 3 cm steel ball.

8.3.3 Copra (and coconut)

The entire sample should be frozen before milling in the mechanical mill (6.2).

The Christy Norris Laboratory Mill⁴⁾ with a 6 mm screen has been found to be satisfactory. The length of particles shall be approximately 2 mm but shall not be greater than 5 mm. Mix the particles carefully and carry out the determination without delay.

Care shall be taken to avoid condensation of moisture onto the seeds during and after grinding.

8.3.4 Large and medium sized seeds (illipe, sheanuts, sunflower, groundnuts, soya, etc.)

Except in the case of cottonseed, grind the test sample in the mechanical mill (6.2), until the major dimension of the particles obtained is not greater than 2 mm. Reject the first particles (about one-twentieth of a sample), collect the rest, mix carefully and carry out the determination without delay.

4) Refer to footnote 1).

The following milling techniques have been found to be satisfactory:

- for soya: Christy Norris Laboratory Mill⁴⁾ with 0,8 mm perforated plate, or Ultra Centrifugal Mill⁴⁾ with 1 mm screen;
- for sunflower: UCM Mill⁴⁾ with 1 mm screen;
- for groundnuts: Christy Norris Laboratory Mill⁴⁾ with 3 mm bar screen; with samples containing more than about 45 % (*m/m*) of oil, care shall be taken to avoid, as far as possible, the formation of a pasty mass;
- other seeds: 6 mm bar screen.

NOTE The grinding of very oily seeds can often be improved if the seeds are first frozen at $-10\text{ }^{\circ}\text{C}$ to $-20\text{ }^{\circ}\text{C}$, but great care should be taken to avoid condensation of moisture onto the seeds during and after grinding.

8.3.5 Cottonseed

Weigh, to the nearest 1 mg in the tared metal dish (6.12), about 15 g from the sample as received. Place the dish and seeds in the oven (6.11) previously heated to $130\text{ }^{\circ}\text{C}$, and leave to dry for 2 h at $(130 \pm 2)\text{ }^{\circ}\text{C}$. Then remove the dish from the oven and allow to cool in air for about 30 min. Transfer the whole test portion to a grinder or mill (6.2) and grind to break up the seeds and linters. Transfer the ground material to an extraction thimble (6.4) and proceed with the determination.

8.3.6 Small seeds (linseed, rapeseed, etc.)

Take a representative portion of approximately 100 g from the sample as received and grind it (6.2) so that no whole seeds remain. Ensure that any fine material remaining in the mill is incorporated into the bulk of the ground material and that the whole is thoroughly mixed. Care shall be taken that moisture is not lost from the ground test sample.

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The grinding time and speed (if variable) shall be predetermined for a particular mill and seed. The grinding shall not cause segregation of the "meat" and "husks" of the seed, the test sample shall not be oily and at least 95 % (*m/m*)

s and prepare
seed such as

9 Procedure

NOTE If it is required to check whether the repeatability limits (11.2) are met, carry out two single determinations in accordance with 9.1 and 9.2.

9.1 Test portion

9.1.1 Weigh, to the nearest 1 mg, $10\text{ g} \pm 0,5\text{ g}$ of the test sample (8.3). For treatment of impurities, see 9.3.

9.1.2 Transfer the test portion to the thimble (6.4), using a small wad of cotton wool (6.4) moistened with solvent (5.1) to transfer the last traces of ground seed from the weighing container to the thimble. Use this cotton wool to plug the thimble.

9.2 Determination

9.2.1 Preparation of flask

Weigh, to the nearest 1 mg, a flask (6.5), containing a few granules of pumice stone (6.6), which has been previously dried in an oven and cooled in a desiccator.

9.2.2 Solvent extraction

The times stipulated for the three extractions (see 9.2.2.1, 9.2.2.2 and 9.2.2.3) may be varied slightly (for example by ± 10 min). On no account should extended extraction times (for example overnight) be employed.

9.2.2.1 First extraction

Place the thimble (6.4) containing the test portion in the extraction apparatus (6.5). 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 (6.8). 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 (6.7).

9.2.2.2 Second extraction

Empty the contents of the thimble into the microgrinder cylinder (6.3) and grind for 7 min. For most seeds, six 1 cm diameter steel balls in a 150 ml cylinder have been found to be satisfactory; for cottonseed with adherent linters, three 2 cm balls are satisfactory.

Put the mixture back into the thimble, using a small piece of cotton wool to remove any residual seed particles from the grinding apparatus. Add this to the thimble. Put the thimble back into the extraction apparatus; re-extract for a further 2 h, using the same flask containing the first extract. Allow to drain and cool, remove the thimble again, eliminate most of the solvent and repeat the grinding as above.

9.2.2.3 Third extraction

Put the mixture back into the thimble, cleaning the grinding apparatus as before (9.2.2.2), and put the thimble back into the extraction apparatus. Extract as before (9.2.2.1) for 2 h, using the same flask.

9.2.3 Removal of solvent and weighing of the extract

Remove the greater part of the solvent from the flask by distillation on the electric heating bath or hot-plate. 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. Remove the last traces of solvent by heating the flask for between 30 min and 60 min in the oven (6.9) at (103 ± 2) °C at atmospheric pressure, or at 80 °C under vacuum.

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

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

In the case of non-lauric oils, when a vacuum oven is available, traces of solvent may be removed by heating at 80 °C under vacuum.

Allow the flask to cool in the desiccator (6.10), for at least 1 h, to ambient temperature and weigh to the nearest 1 mg.

Heat again for between 20 min and 30 min under the same conditions, allow to cool and weigh.

The difference between the two weighings shall not exceed 5 mg. If it does, repeat the operations of heating, cooling and weighing until the difference between two successive weighings does not exceed 5 mg. Note the final mass of the flask.

If there is a significant increase in mass (over 5 mg), oxidation of a drying oil may be taking place and a further analysis should be carried out, taking precautions to exclude oxygen.

9.2.4 Impurities content of the extracted oil

The oil extracted shall be clear; if it is not, determine the impurities content. For this purpose, dissolve the fatty matter in the solvent used for extraction; filter through a filter paper, previously dried at (103 ± 2) °C to constant mass. Wash the filter paper several times with the same solvent to remove the oil completely; dry again at (103 ± 2) °C to constant mass. To cool and weigh the filter paper, use a suitable vessel provided with a lid. Correct the result accordingly.

9.3 “Oil content” of impurities

To determine the “oil content” of the impurities, carry out the analysis in the same manner as for the seeds. However in this case:

- the test portion may be 5 g to 10 g;
- only one extraction, for a period of 4 h, is necessary, the small error thus introduced into the “oil content” of the product as received being negligible.

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10 Expression of results

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10.1 Method of calculation

In all cases, express the results to one decimal place and take as the result the arithmetic mean of the two determinations (see Note in clause 9) provided that the requirement concerning repeatability (see 11.1) is satisfied. Otherwise, repeat the determination on two other test portions (8.3). If the differences still exceed the value stated in 12.1, take as the result the arithmetic mean of the four determinations carried out, provided that the maximum difference between the individual results does not exceed 1,50 % in absolute value.

10.1.1 Determination on product as received

The “oil content”, expressed as a percentage by mass of the product as received [*tale quale*], is equal to w_0 :

$$w_0 = \frac{m_1}{m_0} \times 100 \%$$

where

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

m_1 is the mass, in grams of the dried extract (see 9.2.3).

10.1.2 Palm kernels

The oil (and FFA) contents on a *tale quale* basis are calculated from separate analyses of the seeds and impurities.