International Standard 659

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Oilseeds — Determination of hexane extract (or light petroleum extract), called "oil content"

Graines oléagineuses — Détermination de l'extrait à l'hexane (ou à l'éther de pétrole), dit «teneur en huile»

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Foreword

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It has been approved by the member bodies of the following countries :

Australia	India	Portugal
Canada	Iran	Romania
Chile	Israel	South Africa, Rep. of
Czechoslovakia	Korea, Rep. of	Thailand
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The member body of the following country expressed disapproval of the document on technical grounds :

USA

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Oilseeds — Determination of hexane extract (or light petroleum extract), called "oil content"

1 Scope and field of application

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

NOTE — If required, the following may be analysed separately

— the pure seeds and the impurities (see 10.2);

— in the case of groundnuts, the pure seeds, the total fines, the non-oleaginous impurities and the oleaginous impurities.

2 References

ISO/R 542, *Oilseeds — Sampling.*

ISO 664, *Oilseeds — Reduction of contract samples to analysis samples.*

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

3 Definition

hexane extract, called "oil content" : The whole of the substances extracted under the operating conditions specified below, and expressed as a percentage by mass of the product as received. 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, failing this, light petroleum. Elimination of the solvent and weighing of the extract obtained.

5 Reagents

5.1 Technical *n*-hexane, or, failing this, **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 and 70 °C and which has a bromine value less than 1. For either solvent, the residue on complete evaporation shall not exceed 2 mg per 100 ml.

5.2 Hydrochloric acid, concentrated, ϱ_{20} 1,19 g/ml (only in the case of cottonseed with adherent linters — see 8.1.4).

6 Apparatus

Usual laboratory apparatus and in particular :

6.1 Analytical balance.

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

6.3 Mechanical grater or, failing this, **hand grater** (only in the case of copra — see 8.1.2).

6.4 Mechanical micro-grinder (see 10.1), capable of producing a fineness of grinding of oilseeds of less than 160 µm, with the exception of the "shell", particles of which may reach 400 µm.

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

6.6 Suitable extraction apparatus (fitted with a flask of capacity 200 to 250 ml).

NOTE — Several flasks are necessary (see 8.4).

6.7 Pumice stone, in small particles, previously dried in an oven at 130 ± 2 °C and cooled in a desiccator.

6.8 Electric heating bath (sand bath, water bath, etc.) or **hot-plate.**

6.9 Electrically heated oven, with thermostatic control, permitting ventilation or obtaining reduced pressure.

6.10 Desiccator, containing an efficient desiccant.

6.11 In the case of cottonseed with adherent linters, the following are also required.

6.11.1 Electrically heated oven, capable of being maintained at 130 ± 2 °C.

6.11.2 Fumigation oven, with thermostatic control capable of heating a sample to 115 °C in 30 min.

6.11.3 Metal dish, flat-bottomed, diameter 100 mm, height about 40 mm.

6.11.4 Porous vessel, of ceramic material, cylindrical, internal diameter 68 mm, external diameter 80 mm, height 85 mm, thickness of walls and base 6 mm.

6.11.5 Watch glass, diameter 80 to 90 mm.

6.11.6 Pipette, 2 ml, graduated in 0,1 ml.

7 Sampling

See ISO/R 542.

8 Procedure

8.1 Preparation of the test sample

8.1.1 Reduction of sample

Take an analysis sample obtained in accordance with ISO 664. If large non-oleaginous foreign bodies have been separated before the reduction of the laboratory sample, make allowance for this in the calculation (see 9.3.3). According to the requirements of the contract, use an analysis sample as received or after separation of the impurities.

8.1.2 Copra

Grate the product by hand or, preferably, using a mechanical grater (6.3) which allows the whole sample to be treated. When grating by hand (a process which does not allow all the analysis sample to be grated), endeavour to obtain a test sample which is as representative as possible and, to this end, take account of the size and colour of different fragments.

The length of the particles shall be close to 2 mm but shall not be greater than 5 mm. Mix the particles carefully and carry out the determination without delay.

8.1.3 Seeds of medium-size (sunflower, groundnut, soya, etc.)

Except in the case of cottonseed with adherent linters, grind the analysis sample in the mechanical mill (6.2), which has previously been well cleaned, until the major dimension of the particles obtained is not greater than 2 mm. Reject the first particles (about one-twentieth of the sample), collect the rest, mix carefully and carry out the determination without delay.

8.1.4 Cottonseed with adherent linters

Weigh, to the nearest 1 mg in the tared metal dish (6.11.3), about 60 g of the analysis sample as received. Place the dish and seeds in the oven (6.11.1), previously heated to 130 °C, and leave to dry for 2 h at 130 ± 2 °C; then remove the dish from the oven and allow to cool in air for about 30 min. Transfer the dried seeds to the porous ceramic vessel (6.11.4), the inside walls and the base of which have been previously moistened with 1,5 ml of the hydrochloric acid (5.2) by means of the pipette (6.11.6), taking care that the acid is completely absorbed without forming adherent drops. Cover the vessel with the watch glass (6.11.5) and place it in the fumigation oven (6.11.2). Heat so as to reach 115 °C in 30 min; do not heat beyond this temperature and maintain it for another 30 min.

Remove the vessel from the oven, allow to cool for 1 h in air, reweigh the treated seeds to the nearest 1 mg, then grind the seeds in the mechanical mill (6.2) and proceed as specified in 8.1.3.

8.1.5 Small seeds (linseed, colza, etc.)

Carefully mix the analysis sample without previous mechanical grinding.

8.2 Test portion

8.2.1 The test portion shall be representative of the analysis sample.

8.2.2 Weigh, to the nearest 1 mg, about 10 g of the test sample (see 8.1.2, 8.1.3, 8.1.4 or 8.1.5, as appropriate).

NOTE — In the case of groundnuts, the test portion may comprise the separated fractions of pure seeds, non-oleaginous and oleaginous impurities, and the total fines in quantities proportional to those of the different constituents in the analysis sample itself.

8.2.3 In the case of copra and medium-sized seeds, including cottonseed with adherent linters, transfer the test portion to the thimble (6.5) and plug the latter with a wad of cotton wool (6.5).

8.2.4 In the case of small seeds, grind the test portion in the micro-grinder (6.4) or in the mill (6.2), taking care not to leave any seeds intact. Transfer the ground seeds, without loss, to the thimble (6.5), using a spatula. Wipe the bowl of the micro-grinder or mill and the spatula with a wad of cotton wool (6.5) soaked with solvent (5.1), and plug the thimble with this wad.

8.3 Predrying

If the test portion is very moist [moisture and volatile matter content above 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).

8.4 Determination

8.4.1 Preparation of flasks

Weigh, to the nearest 1 mg, two flasks A and B (6.6) each containing one or two particles of pumice stone (6.7).

8.4.2 First extraction

Place the thimble (6.5) containing the test portion in the extraction apparatus (6.6). Pour into flask A 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.

8.4.3 Second extraction

Empty the thimble into the micro-grinder (6.4) and grind as finely as possible. (See 10.1.) 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 A containing the first extract. Allow to cool, remove the thimble again, eliminate most of the solvent and repeat the grinding as above.

8.4.4 Third extraction

Put the mixture back into the thimble and put the latter back into the extraction apparatus. Pour into flask B the necessary quantity of solvent. Fit the flask as above and proceed with a third extraction for 2 h.

8.4.5 Elimination of solvent and weighing of the extract

Expel the greater part of the solvent from the flasks A and B by distillation on the electric heating bath or the hot-plate. Expel the last traces of solvent by heating the flasks for about 20 min in the oven (6.9) at 103 ± 2 °C[1]. Assist the removal either by blowing air or, preferably, an inert gas (such as nitrogen or carbon dioxide) into the flasks for short periods, or by reducing the pressure in the flasks[2].

Allow the flasks 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 10 min under the same conditions; allow to cool and weigh.

For each flask, 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 masses of flasks A and B.

If the mass of extract in flask B does not exceed 10 mg, the extraction is completed. Otherwise, carry out a fresh extraction for 2 h under the same conditions (see 8.4.4), using a new flask, until the mass of extract from the last extraction is not more than 10 mg.

8.4.6 Impurities content

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.

8.5 Number of determinations

Carry out two determinations on the same test sample.

9 Expression of results

9.1 Method of calculation and formulae

9.1.1 Determination on product as received

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

$$\frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (8.2.2);

m_1 is the sum of the masses, in grams, of the extracts found in the flasks after drying (see 8.4.5).

Take as the result the arithmetic mean of the two determinations (see 8.5), provided that the requirement concerning repeatability (see 9.2) is satisfied. Otherwise, repeat the determination on two other test portions. If this time the difference still exceeds 0,4 g per 100 g of sample, take as the result the arithmetic mean of the four determinations carried out.

Express the result to one decimal place.

9.1.2 Separate analyses of pure seeds and impurities

The formula given in 9.1.1 also serves for calculating the "oil content" of both pure seeds and the impurities when the pure seeds and the impurities are analysed separately (see 10.2).

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

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

In this case, the "oil content" expressed as a percentage by mass of the product as received (pure seeds and impurities), is equal to

$$H_1 - \frac{P}{100}(H_1 - H_2)$$

where

H_1 is the percentage, by mass, of oil in the pure seeds;

H_2 is the percentage, by mass, of oil in the impurities;

P is the percentage, by mass, of impurities in the product as received.

9.1.3 Cottonseed with adherent linters

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

$$\frac{m_1}{m_0} \times \frac{m_0''}{m_0'} \times 100$$

where

m_0 and m_1 have the same meanings as in 9.1;

m_0' is the mass, in grams, of the test portion (about 60 g) before pre-treatment (see 8.1.4);

m_0'' is the mass, in grams, of the same test portion after pre-treatment (see 8.1.4) and before grinding.

9.1.4 Case where large non-oleaginous foreign bodies have been separated before the analysis (see 8.1.1)

The result obtained in accordance with 9.1.1, 9.1.2, or 9.1.3 for the "oil content" of the product as received shall be corrected according to the formula

$$H_0 \times \frac{100 - X}{100}$$

where

H_0 is the percentage, by mass, of oil in the material analysed (calculated according to 9.1.1, 9.1.2, or 9.1.3, as appropriate);

X is the percentage, by mass, of large non-oleaginous foreign bodies previously separated in the original product as received.

9.1.5 Groundnuts

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

$$H_1 - \frac{P + I_o + I_n}{100}(H_1 - H_2)$$

where

P is the percentage, by mass, of total fines;

I_o is the percentage, by mass, of oleaginous impurities;

I_n is the percentage, by mass, of non-oleaginous impurities;

H_1 is the percentage, by mass, of oil in the pure seeds;

H_2 is the percentage, by mass, of oil in the impurities.

If the extraction has been carried out in a single thimble, calculate the "oil content" according to 9.1.1.

9.1.6 "Oil content" expressed in relation to the dry matter

On request, the "oil content" may be expressed as a percentage by mass of the dry matter; it is then equal to

$$H_0 \times \frac{100}{100 - U}$$

where

H_0 is the percentage, by mass, of oil in the product as received;

U is the percentage, by mass, of water and volatile matter, determined according to ISO 665.

9.2 Repeatability

The difference between two determinations carried out simultaneously or in rapid succession by the same analyst should not exceed 0,4 g of oil per 100 g of sample.

10 Notes on apparatus and procedure

10.1 In laboratories which do not regularly carry out oilseed analyses, and where a micro-grinder (see 6.2) is not available, micro-grinding of the ground sample (see 8.4.3) may be replaced by trituration with a pestle and mortar. After extracting for 4 h, according to 8.4.2, empty the thimble into the mortar add about 10 g of sand which has been washed with hydrochloric acid and then calcined, and triturate as finely as possible. Then proceed with the analysis as specified in 8.4.2. Since hand grinding is less efficient than mechanical grinding in a micro-grinder, the quantity of oil obtained in flask B containing the result of the third extraction is, in the case of seeds difficult to extract, such as colza, nearly always greater than 10 mg. Therefore, it is essential to carry out successive extractions as specified in this International Standard until the mass of the extracted oil during the last extraction is not more than 10 mg; more than five extractions may be required.

Grinding in a mortar cannot, usually, 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.