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

This second edition cancels and replaces ISO 734-1:2006, which has been renumbered and editorially revised.

Introduction

A method for the determination of the oil content of oilseeds has been specified in ISO 659. It is therefore necessary to provide for control of oil production by establishing 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 International Standard 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, 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
all of the substances extracted under the operating conditions specified in this International Standard, and expressed as a mass fraction, in percent, of the product as received

Note 1 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.

Less than 5 % shall distil below 50 °C and more than 95 % between 50 °C and 70 °C.

For any of these solvents, 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 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**, 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, for example 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 Electric heating bath (e.g. sand bath, water bath) or **hot plate**.

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

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 an accuracy of ±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.

Sampling is not part of the method specified in this International Standard. 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.

1) The Dangoumau mechanical microgrinder is an example of suitable apparatus available commercially. This information is given for the convenience or users of this International Standard and does not constitute an endorsement by ISO of this apparatus.

2) The Butt, Smalley, Twisselmann or Bolton-Williams straight-through extractors are examples of suitable apparatus available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this apparatus.

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 (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 (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 (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 (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 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.

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.