INTERNATIONAL STANDARD 736

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION · МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ · ORGANISATION INTERNATIONALE DE NORMALISATION

Oilseed residues — Determination of diethyl ether extract

Tourteaux de graines oléagineuses — Détermination de l'extrait à l'oxyde diéthylique

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 736 was developed by Technical Committee ISO/TC 34, *Agricultural food products*.

It was submitted directly to the ISO Council, in accordance with clause 6.12.1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 735-1968, which had been approved by the member bodies of the following countries :

Australia	Hungary	Portugal
Bulgaria	India	Romania
Czechoslovakia	Iran	South Africa, Rep. of
Chile	Israel	Thailand
Colombia	Italy	Turkey
Egypt, Arab Rep. of	Korea, Rep. of	United Kingdom
France	Netherlands	Yugoslavia
Germany	Poland	

The member bodies of the following countries had expressed disapproval of the document on technical grounds :

Canada
Ireland

Printed in Switzerland

Oilseed residues — Determination of diethyl ether extract

0 INTRODUCTION

This International Standard was established because the principal users of oilseed residues, namely the manufacturers of animal feeding stuffs, have always determined the oil content of oilseed residues by extraction with diethyl ether and have accumulated a large amount of data on the subject.

However, as the determination of the oil content of oilseeds is carried out according to ISO 659, *Oilseeds — Determination of* n-*hexane extract (or light petroleum extract), called "oil content"*[1], it was thought necessary that the oil content of oilseed residues should be determined in the same way in order to provide for control of oil production. That method is therefore the subject of ISO 734, *Oilseed residues — Determination of* n-*hexane extract (or light petroleum extract), called "oil content"*[2]. The two methods do not always give the same results.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the diethyl ether extract of residues (excluding compounded products) obtained by the extraction of oil from oilseeds by pressure or solvent.

2 REFERENCES

ISO 771, *Oilseed residues — Determination of moisture and volatile matter content.*

ISO 5500, *Oilseed residues — Sampling.*[3]

3 DEFINITION

diethyl ether extract : The whole of the substances extracted by diethyl ether under the operating conditions specified below.

4 PRINCIPLE

Extraction of a test portion with diethyl ether in a suitable apparatus.

5 REAGENTS AND MATERIALS

5.1 Diethyl ether, anhydrous, practically free from peroxides (ρ_{20} 0,712 to 0,716 g/ml, boiling point 34,5 °C), analytical quality, the non-volatile residue of which at 80 °C is not greater than 0,001 % (*m/m*).

5.2 Sand, washed with hydrochloric acid and calcined.

5.3 Pumice stone, in small particles, previously dried.

5.4 Sodium sulphate, anhydrous, analytical quality.

6 APPARATUS

6.1 Analytical balance.

6.2 Mechanical mill, easy to clean and allowing the residues to be ground, without heating and without appreciable change in moisture, volatile matter and oil content, to particles passing completely through the sieve (6.3).

6.3 Sieve, with apertures of diameter 1 mm.

6.4 Extraction thimble and **cotton wool,** or filter paper.

Materials shall be free from matter soluble in diethyl ether.

6.5 Suitable extraction apparatus (capacity of flask 200 to 250 ml, for example).

6.6 Electric heating bath (sand bath, water bath, etc.).

6.7 Pestle and mortar, of porcelain, iron or bronze, or, preferably, a suitable **mechanical micro-grinder.**

6.8 Electrically heated vacuum oven, with thermostatic control.

6.9 Desiccator, containing an efficient desiccant.

1) At present at the stage of draft. (Revision of ISO/R 659-1968.)

2) At present at the stage of draft. (Revision of ISO/R 734-1968.)

3) In preparation.

7 PROCEDURE

7.1 Preparation of the test sample

7.1.1 Take the contract sample obtained in accordance with ISO 5500.

7.1.2 Grind the contract sample, if necessary, in the previously well-cleaned mechanical mill (6.2). 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.

7.2 Test portion

As soon as grinding is completed, weigh, to the nearest 0,01 g, about 5 to 10 g of the test sample (7.1.2), according to the expected content of extractable matter.

7.3 Preliminary drying

Mix the test portion (7.2) in a suitable vessel with 2 to 3 g of the anhydrous sodium sulphate (5.4) per 5 g of grindings. Transfer the mixture to a thimble (6.4) and close this with a plug of cotton wool (6.4). If a filter paper is used, wrap the mixture in it.

Mixing may be carried out in the thimble itself.

7.4 Determination

Weigh, to the nearest 0,001 g, the flask of the extraction apparatus (6.5) containing one or two particles of pumice stone (5.3), which have been previously dried at a temperature near 100 °C and then allowed to cool for at least 1 h in the desiccator (6.9) to ambient temperature.

Put the thimble or filter paper containing the test portion into the extractor. Pour into the flask the necessary quantity of the diethyl ether (5.1). Fit the flask to the extractor on the electric heating bath (6.6) and carry out the heating so that the extraction rate is at least 3 drops per second (boiling briskly but not violently).

After extraction for 4 h, allow to cool. Remove the thimble from the extractor, and place it in a current of air in order to remove the greater part of the solvent impregnating it.

Empty the thimble into a mortar (6.7), add about 10 g of the sand (5.2) and triturate as finely as possible (if a micro-grinder is used, grind without adding sand). Put the mixture back into the thimble and put the latter back into the extractor. Extract again for 2 h, using the same flask (see 9.1 and 9.2).

Expel the greater part of the solvent from the flask by distillation on a boiling-water bath. Continue to remove solvent by carefully turning the flask, until only traces are left. Expel the last traces of solvent by heating the flask at 75 °C for 1,5 h in the vacuum oven (6.8) (pressure 133 mbar maximum) (except as provided for in 9.3).

Allow the flask to cool in the desiccator (6.9) for a least 1 h to ambient temperature, and weigh to the nearest 0,001 g.

Carry out a second heating for 30 min under the same conditions, cool again and weigh.

The difference between these two weighings should not exceed 0,01 g. If it does, heat again for periods of 30 min until the difference in mass does not exceed 0,01 g. Record the last weighing of the flask.

Carry out two determinations on the same test sample.

8 EXPRESSION OF RESULTS

8.1 Method of calculation and formulae

8.1.1 The diethyl ether extract, expressed as a percentage by mass of the sample as received, is equal to

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

where

m_0 is the mass, in grams, of the test portion (7.2);

m_1 is the mass, in grams, of the extract in the extraction flask at the last weighing.

Take as the result the arithmetic mean of the two determinations, provided that the requirement concerning repeatability (see 8.2) is satisfied. Otherwise, repeat the determination on two other test portions. If this time the difference again exceeds 0,2 g per 100 g of sample, take as the result the arithmetic mean of the four determinations carried out.

Report the result to one decimal place.

8.1.2 If requested, the diethyl ether extract may be expressed in relation to the dry matter by multiplying the result obtained in accordance with 8.1.1 by

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

where U is the percentage by mass of moisture and volatile matter determined as specified in ISO 771.

8.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst should not exceed 0,2 g of diethyl ether extract per 100 g of sample.

9 NOTES ON PROCEDURE

9.1 For most oilseed residues, a single extraction for 6 h, without an additional trituration, gives equivalent results, but it is the responsibility of the analyst to confirm this in each particular case.

9.2 The solution obtained in the extraction flask should be clear. If it is not, filter it through a filter paper, collecting the filtrate in another flask which has been previously dried and weighed, and wash the first flask and the filter paper several times with diethyl ether. Expel the solvent and dry the residue as described in 7.4.

9.3 In the case of residues rich in volatile acids (residues of copra, palm-kernel, etc.), the extract should be dried at 60 °C and at atmospheric pressure.

10 TEST REPORT

The test report shall show the method used and the result obtained, indicating clearly whether this is expressed in relation to the product as received or in relation to the dry matter. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details required for complete identification of the sample.

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ERRATUM

Inside front cover

In the third line of the fourth paragraph of the foreword, correct "R 735-1968" to "R 736-1968".

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