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ISO RECOMMENDATION

R 736

OIL SEED RESIDUES

DETERMINATION OF DIETHYL ETHER EXTRACT

1st EDITION

May 1968

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 736, *Oilseed residues – Determination of diethyl ether extract*, was drawn up by Technical Committee ISO/TC 34, *Agricultural food products*, the Secretariat of which is held by the Magyar Szabvanyügyi Hivatal (MSZH).

Work on this question by the Technical Committee began in 1963 and led, in 1965, to the adoption of a Draft ISO Recommendation.

In October 1966, this Draft ISO Recommendation (No. 1043) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

Two Member Bodies opposed the approval of the Draft :

Canada
Ireland

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in May 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 736 May 1968

OIL SEED RESIDUES

DETERMINATION OF DIETHYL ETHER EXTRACT

INTRODUCTION

As 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, it has been considered necessary to establish this method of determination and this is the subject of the present ISO Recommendation.

However, as the determination of the oil content of oilseeds is carried out according to ISO Recommendation R 659, *Oleaginous seeds – Determination of oil content*, by extraction with n-hexane or light petroleum, it has been 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. This method is the subject of ISO Recommendation R 734, *Oilseed residues – Determination of oil content.* The two methods do not always give the same results.

1. SCOPE

This ISO Recommendation describes a method for the determination of diethyl ether extract of residues (excluding compounded products), obtained by the extraction of oil from oilseeds by pressure or solvent.

2. DEFINITION

By *diethyl ether extract* is meant the whole of the substances extracted by this solvent under the operating conditions described below.

3. PRINCIPLE

Extraction of the product with diethyl ether in a suitable apparatus.

4. REAGENTS

4.1 *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 0.0019 %.

4.2 *Sand,* washed with hydrochloric acid and calcined.

4.3 *Pumice stone,* in small particles, previously dried.

4.4 *Sodium sulphate,* anhydrous, analytical quality.

5. APPARATUS

5.1 *Analytical balance.*

5.2 *Mechanical mill,* easy to clean and allowing the residues to be ground, without heating and without appreciable change in the content of water, volatile matter and oil, to particles passing completely through a sieve of aperture diameter 1 mm.

5.3 *Sieve,* with apertures of diameter 1 mm.

5.4 *Extraction thimble,* or filter paper, and cotton wool, free from matter soluble in diethyl ether.

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

5.6 *Electric heating bath,* (sand bath, water bath, etc.).

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

5.8 *Electrically heated vacuum oven,* with temperature control.

5.9 *Desiccator,* containing an efficient desiccant.

6. PROCEDURE

6.1 Preparation of the sample

6.1.1 Use the contract sample obtained as described in ISO Recommendation R . . .,* *Oilseed residues – Sampling.*

6.1.2 Grind the contract sample, if necessary, in the previously well-cleaned mechanical mill (5.2). Use about a twentieth of the sample to complete the cleaning of the mill, and reject these grindings; grind the rest, collect the grindings, mix carefully and carry out the analysis without delay.

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* At present at the stage of a draft proposal.

6.2 Test portion

As soon as grinding is completed, weigh to the nearest 0.01 g about 5 to 10 g of the grindings (6.1.2), according to the assumed content of extractable matter.

6.3 Preliminary drying

Mix the test portion (6.2) in a suitable vessel with 2 to 3 g of anhydrous sodium sulphate (4.4) per 5 g of grindings. Transfer the mixture to a thimble (5.4) and close this with a plug of cotton wool (5.4). If a filter paper (5.4) is used, wrap the mixture in it.

Mixing may be carried out in the thimble itself.

6.4 Determination

Weigh, to the nearest 0.001 g, the flask of the extraction apparatus (5.5) containing one or two particles of pumice stone (4.3) which have been previously dried at a temperature near 100 °C and cooled again for at least 1 hour in the desiccator (5.9) to ambient temperature.

Put the thimble or filter paper containing the test portion into the extractor. Pour into the flask the necessary quantity of solvent (4.1). Fit the flask to the extractor on the electric heating bath (5.6) and so carry out the heating that the extraction rate is at least three drops per second (boiling briskly but not violently).

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

Empty the thimble into a mortar (5.7), add about 10 g of sand (4.2) 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. Continue the extraction for a further 2 hours, using the same flask (see Notes 8.1 and 8.2).

Expel the greater part of the solvent from the flask by distillation on a boiling-water bath. Remove the rest of the solvent by carefully turning the flask, until only traces are left. Expel the last traces of solvent by heating the flask for one and a half hours in the vacuum oven (133 m bar maximum) (see Note 8.3).

Allow the flask to cool again in the desiccator (5.9) for a least 1 hour to ambient temperature, and weigh to the nearest 0.001 g.

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

The difference between these two weighings should be at most 0.01 g. If not heat again for 30-minute periods, until the difference in mass is at most 0.01 g. Record the last weighing of the flask.

Carry out two determinations on the same prepared sample.

7. EXPRESSION OF RESULTS

7.1 Method of calculation and formulae

7.1.1 The diethyl ether extract, as a percentage by mass of the product as received, is equal to

$$M_1 \times \frac{100}{M_0}$$

where

M_0 is the mass, in grammes, of the test portion,

M_1 is the mass, in grammes, of the extract in the extraction flask at the last weighing.

Take as the result the arithmetic mean of the two determinations, if the condition of repeatability is satisfied. If not, repeat the determination on the other test portions. If this time the difference still exceeds 0.2 g, take as the result the arithmetic mean of the four determinations carried out.

Express the results to one decimal place.

7.1.2 If requested, the diethyl ether extract may be expressed in relation to the dry matter. The diethyl ether extract, as a percentage by mass of the dry matter, is equal to

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

where

P is the percentage, by mass, of diethyl ether extract from the product as received,

U is the percentage, by mass, of moisture and volatile matter, determined as described in ISO Recommendation R 771, *Oilseed residues – Determination of moisture and volatile matter.*

7.2 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.