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ISO RECOMMENDATION
R 734

OILSEED RESIDUES

DETERMINATION OF OIL CONTENT

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BRIEF HISTORY

The ISO Recommendation R 734, *Oilseed residues – Determination of oil content*, was drawn up by Technical Committee ISO/TC 34, *Agricultural food products*, the Secretariat of which is held by the Magyar Szabvanyugyi 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. 1041) 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 :

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

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 734 Mai 1968

OILSEED RESIDUES

DETERMINATION OF OIL CONTENT

INTRODUCTION

As the determination of the oil content of oleaginous seeds 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 considered necessary that determination of the oil content of oilseed residues should also be carried out in the same way, so as to enable the oil industry to control manufacture. This method is the subject of the present ISO Recommendation.

However, the principal users of oilseed residues, namely, manufacturers of animal feeding stuffs, have always carried out the determination by extraction with diethyl ether and have accumulated a large amount of data on the subject. As the two methods do not always give the same results, it has been considered necessary to establish a method using diethyl ether as well, and this forms the subject of ISO Recommendation R 736, *Oilseed residues – Determination of diethyl ether extract.*

1. SCOPE

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

2. DEFINITION

By *oil* is meant the whole of the substances extracted under the operating conditions described below.

The oil content is expressed as a percentage by mass.

3. PRINCIPLE

Extraction of the oil from the product in a suitable apparatus, with a suitable solvent : n-hexane or, failing this, light petroleum.

4. REAGENTS

4.1 *n-Hexane* or, failing this, light petroleum distilling between 40 and 60 °C and having a bromine value less than 1. For either solvent, the residue on complete evaporation should not exceed 0.002 g/ 100 ml.

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

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

5. APPARATUS

5.1 *Analytical balance.*

5.2 *Mechanical mill*, easy to clean and allowing the residue to be ground, without heating and without appreciable change in the content of moisture, 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 n-hexane or light petroleum.

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

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

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

5.8 *Electrically heated 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.

6.2 Test portion

6.2.1 As soon as grinding is completed, weigh to the nearest 0.01 g, about 10 g of the grindings (6.1.2).

6.2.2 Put the test portion (6.2.1) into a thimble (5.4) and close the latter with a plug of cotton wool (5.4). If a filter paper (5.4) is used, wrap the grindings in it.

* At present at the stage of a draft proposal.

6.3 Preliminary drying

If the residues are very moist (moisture and volatile matter content more than 10 %), put the filled thimble for some time in the oven (5.8) at a temperature not higher than 80 °C, in order to bring the content of moisture and volatile matter below 10 % (see Note 8.1).

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 has been previously dried at a temperature near to 100 °C and cooled again for at least 1 hour in the desiccator (5.9) to ambient temperature.

Put the thimble containing the test portion into the extractor. Pour into the flask the necessary quantity of the solvent (4.1). Fit the flask to the extractor on the electric heating bath or hot plate (5.6), and carry out the heating so 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 expel the greater part of the 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 Note 8.2).

Expel the greater part of the solvent from the flask by distillation on a boiling water bath or a hotplate. 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 approximately 20 minutes at a temperature near 100 °C, without rising above 105 °C (see Note 8.3). Assist the removal either by blowing an inert gas (such as nitrogen or carbon dioxide) for short periods, or by reducing the pressure in the flask (see Note 8.4).

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

Heat again for approximately 10 minutes under the same conditions, cool again and weigh.

The difference between the results at these two weighings should be at most 0.01 g. If not, heat again for periods of approximately 10 minutes, 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 oil content, expressed 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 two other test portions. If this time the difference still exceeds 0.2 g, take as the result the arithmetic mean of the four determination carried out.

Express the result to one decimal place.

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

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

where

H is the percentage by mass of oil in 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 oil per 100 g of product.