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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 749

OILSEED RESIDUES

DETERMINATION OF TOTAL ASH

1st EDITION
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BRIEF HISTORY

The ISO Recommendation R 749, *Oilseed residues – Determination of total ash,* was drawn up by Technical Committee ISO/TC 34, *Agricultural food products,* the Secretariat of which is held by the Magyar Szabványügyi 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. 1017) 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	India	Portugal
Brazil	Iran	Romania
Bulgaria	Ireland	South Africa, Rep. of
Chile	Israel	Thailand
Colombia	Italy	Turkey
Czechoslovakia	Korea, Rep. of	United Kingdom
France	Netherlands	U.S.S.R.
Germany	Paraguay	Yugoslavia
Hungary	Poland	

One Member Body opposed the approval of the Draft :

Canada

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

ISO Recommendation R 749 June 1968

OILSEED RESIDUES

DETERMINATION OF TOTAL ASH

1. SCOPE

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

2. DEFINITION

By *total ash* is meant the residue obtained after incineration at 550 ± 15 °C under the operating conditions described below.

3. PRINCIPLE

Incineration of the product at 550 ± 15 °C in an electrically heated muffle furnace, to practically constant mass.

4. APPARATUS

4.1 *Analytical balance.*

4.2 *Mechanical mill,* easy to clean and allowing the residues 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.

4.3 *Sieve,* with apertures of 1 mm in diameter.

4.4 *Flat-bottomed incineration dish,* of diameter about 60 mm and height not exceeding 25 mm, of platinum, platinum-plated gold, quartz or, if not available, porcelain.

4.5 *Electrically heated muffle furnace,* with air circulation and provided with means for automatic temperature control.

4.6 *Desiccator,* containing an efficient desiccant.

5. PROCEDURE

Carry out all weighings to the nearest 0.001 g.

5.1 Preparation of the sample

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

* At present at the stage of a draft proposal.

5.1.2 Grind the contract sample, if necessary, in the previously well cleaned mechanical mill (4.2). 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.

5.2 Test portion

5.2.1 Weigh the incineration dish (4.4), previously heated for 15 minutes in the electric muffle furnace (4.5) at 550 ± 15 °C and cooled again in the desiccator (4.6) to laboratory temperature

5.2.2 Put into the incineration dish about 5 g of grindings (see clause 5.1.2), spread this uniformly over the whole of the bottom of the dish and re-weigh.

NOTES

1. Carry out these operations as quickly as possible, in order to avoid any appreciable change in moisture content.

2. If the total ash is not used subsequently for the determination of the residue insoluble in hydrochloric acid, the test portion may be reduced to 2 g (see ISO Recommendation R 735, *Oilseed residues – Determination of ash insoluble in hydrochloric acid*).

5.3 Determination

Place the dish containing the test portion on an electric hotplate or over a gas flame, and heat progressively until the substance carbonizes. Put it into the electric muffle furnace (4.5) regulated at 550 ± 15 °C. Continue the heating until a white, light grey or reddish ash is obtained, visibly free from carbon particles (generally at least 2 to 3 hours).

Cool the dish again in the desiccator and weigh when it has reached laboratory temperature.

Put the dish back into the muffle furnace and continue the calcination for another hour at 550 ± 15 °C. Allow the dish to cool again to laboratory temperature and reweigh as before. If the difference between the two weighings is less than or equal to 0.002 g, regard the operation as finished. If not, continue with 1 hour periods in the muffle furnace until the difference between two successive weighings is less than or equal to 0.002 g.

If the ash is rather blackish after the first incineration of 2 to 3 hours, it may be moistened with a few drops of a 20 % solution of ammonium nitrate (not in excess so as to avoid dispersion and sticking together of the ash). After drying in the oven, resume the calcination. If necessary, repeat the operation until incineration is complete.

Carry out two determinations on the same prepared sample.