

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 735

OILSEED RESIDUES

DETERMINATION OF ASH INSOLUBLE IN
HYDROCHLORIC ACID

1st EDITION

May 1968

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BRIEF HISTORY

The ISO Recommendation R 735, *Oilseed residues. Determination of ash insoluble in hydrochloric acid*, was drawn up by Technical Committee ISO/TC 34 – *Agricultural food products*, the Secretariat of which is held by the Magyar Szabványügyi (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. 1042) 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,
Brazil	Iran	Rep. of
Bulgaria	Israel	Thailand
Czechoslovakia	Italy	Turkey
Chile	Korea, Rep. of	United Kingdom
Colombia	Netherlands	U.S.S.R.
France	Poland	Yugoslavia
Germany	Portugal	

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 May 1968, to accept it as an ISO RECOMMENDATION.

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OILSEED RESIDUES

DETERMINATION OF ASH INSOLUBLE IN
HYDROCHLORIC ACID

1. SCOPE

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

2. DEFINITION

By *ash insoluble in hydrochloric acid* is meant the fraction of the total ash which remains undissolved after treatment with hydrochloric acid under the operating conditions described below.

3. PRINCIPLE

Treatment of the total ash with hydrochloric acid, to remove the portion soluble in this reagent, then incineration and weighing of the insoluble residue.

4. REAGENTS

4.1 *Hydrochloric acid*, 3 N solution.

4.2 *Silver nitrate solution*, 1 g per 100 ml.

5. APPARATUS

5.1 *Analytical balance*.

5.2 *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.

5.3 *Hardened filter paper*, of medium porosity, ash-free.

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

5.5 *Desiccator*, containing an efficient desiccant.

6. PROCEDURE

Carry out all weighings to the nearest 0.001 g.

6.1 Test portion

Use the total ash obtained in the determination of total ash as described in ISO Recommendation R 749, *Oilseed residues – Determination of total ash*.

6.2 Determination

Moisten the total ash with 10 ml of the hydrochloric acid solution (4.1) covering the incineration dish (5.2) with a watch-glass. Heat gently and, by several washes with hydrochloric acid solution (4.1) using about 50 ml of acid solution in all, transfer the contents of the dish to a beaker of about 250 ml capacity. Heat to boiling and keep gently boiling for about 10 minutes, then filter through hardened filter paper (5.3) and wash with boiling water until chloride ions are removed (test with the silver nitrate solution (4.2)).

Place the filter and the residue in the incineration dish (5.2), previously heated for 15 minutes in the electrically heated muffle furnace (5.4) regulated at 550 ± 15 °C, and cool again in the desiccator (5.5) to laboratory temperature and weigh.

Heat the dish, filter and residue, progressively on an electric hotplate or over a gas flame until the filter paper is carbonized. Put them into the furnace regulated at 550 ± 15 °C. Continue the heating until a residue visibly free from carbon particles is obtained (generally 1 hour).

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

Put the dish back into the furnace and continue the calcination for another 30 minutes at 550 ± 15 °C, allow the dish to cool and re-weigh, as before. If the difference between the two weighings is less than or equal to 0.001 g, consider the operation as finished.

If not, continue with 30-minute periods in the furnace until the difference between two successive weighings is less than or equal to 0.001 g.

Carry out two determinations, starting from the same prepared sample.

7. EXPRESSION OF RESULTS

7.1 Method of calculation and formula

7.1.1 The ash insoluble in hydrochloric acid, expressed as a percentage by mass of the sample as received, is equal to :

$$(M_2 - M_1) \times \frac{100}{M_0}$$

where

M_0 is the mass, in grammes, of the test portion taken for the determination of the total ash,

M_1 is the mass, in grammes, of the tared dish,

M_2 is the mass, in grammes, of the dish containing the residue obtained by calcination.