INTERNATIONAL STANDARD 749

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Oilseed residues — Determination of total ash

Tourteaux de graines oléagineuses — Détermination des cendres totales

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FOREWORD

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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 749 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 749-1968, which had been approved by the member bodies of the following countries :

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

The member body of the following country had expressed disapproval of the document on technical grounds :

Canada

Printed in Switzerland

Oilseed residues – Determination of total ash

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies 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 REFERENCES

ISO 735, *Oilseed residues – Determination of ash insoluble in hydrochloric acid.*

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

ISO 5500, *Oilseed residues – Sampling.* 1)

3 DEFINITION

total ash : The residue obtained after incineration at 550 ± 15 °C under the operating conditions specified below.

4 PRINCIPLE

Incineration of a test portion at 550 ± 15 °C in an electrically heated muffle furnace, until practically constant mass is reached.

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 moisture, volatile matter and oil content, to particles passing completely through the sieve (5.3).

5.3 Sieve, with apertures of diameter 1 mm.

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

5.5 Electrically heated muffle furnace, with air circulation and capable of being controlled at 550 ± 15 °C.

5.6 Desiccator, containing an efficient desiccant.

6 PROCEDURE

Make all weighings to the nearest 0,001 g.

6.1 Preparation of the test sample

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

6.1.2 Grind the contract sample, if necessary, in the previously well cleaned mechanical mill (5.2). 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.

6.2 Test portion

6.2.1 Weigh the incineration dish (5.4), previously heated for 15 min in the furnace (5.5) at 550 ± 15 °C, and allowed to cool in the desiccator (5.6) to laboratory temperature.

6.2.2 Weigh into the incineration dish about 5 g of the test sample (6.1.2), spread this uniformly over the whole base of the dish and re-weigh.

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

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

6.3 Determination

Place the dish containing the test portion on an electric hot-plate or over a gas flame, and heat progressively until the test portion carbonizes, then place in the furnace (5.5), controlled at 550 ± 15 °C. Continue heating until a white, light grey or reddish ash is obtained, visibly free from carbon particles (generally at least 2 to 3 h).

1) In preparation.

Allow the dish to cool in the desiccator and weigh when it has reached laboratory temperature.

Replace the dish in the furnace and continue heating for 1 h 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 0,002 g, regard the determination as finished. If not, continue with 1 h periods in the 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 or 3 h, it may be moistened with a few drops of a 200 g/l solution of ammonium nitrate (not in excess so as to avoid dispersion and sticking together of the ash). After drying in an oven, resume the calcination. If necessary, repeat the operation until incineration is complete.

Carry out two determinations on the same test sample.

7 EXPRESSION OF RESULTS

7.1 Method of calculation and formulae

7.1.1 The total ash yielded by the sample as received, expressed as a percentage by mass, is equal to

$$\frac{m_2 - m_0}{m_1 - m_0} \times 100$$

where

m_0 is the mass, in grams, of the dish;

m_1 is the mass, in grams, of the dish and test portion;

m_2 is the mass, in grams, of the dish and ash.

Take as the result the arithmetic mean of the two determinations, provided that the requirement concerning repeatability (see 7.2) 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, provided that the maximum difference between the individual results does not exceed 0,5 g per 100 g of sample.

Report the result to one decimal place.

7.1.2 If requested, the total ash may be expressed in relation to the dry matter by multiplying the result obtained in accordance with 7.1.1 by

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

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

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 total ash per 100 g of sample.

8 TEST REPORT

The test report shall show the method used and the result obtained, indicating clearly whether the result 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.