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Starches, native or modified — Determination of total fat content

Amidons et fécules, natifs ou transformés — Détermination de la teneur en matières grasses 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.

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It has been approved by the member bodies of the following countries :

Czechoslovakia	Netherlands	Turkey
France	Poland	United Kingdom
Germany	Portugal	Yugoslavia
Iran	Romania	
Mexico	Spain	

No member body expressed disapproval of the document.

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Starches, native or modified — Determination of total fat content

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the total fat content of starches, native or modified, of which the expected total fat content is less than 1,5 % (*m/m*).

2 REFERENCE

ISO 5808, *Starch derivatives and by-products — Determination of "extractable" fat content.*[1)]

3 DEFINITION

total fat content : The residue obtained under the conditions described in the procedure and expressed as a percentage by mass of the product as received.

4 PRINCIPLE

Hydrolysis of the product by boiling hydrochloric acid, and coagulation of the insoluble products, including the total fat, by cooling. Separation by filtration, drying and isolation of the total fat by solvent extraction.

5 REAGENTS

Use distilled water or water of at least equivalent purity.

5.1 Solvent : *n*-hexane or light petroleum (boiling range 40 to 60 °C) or carbon tetrachloride. (These solvents, especially the carbon tetrachloride, must be handled with care owing to their toxicity.)

The residue on complete evaporation shall not exceed 0,001 g/100 ml.

5.2 Hydrochloric acid, ρ_{20} 1,18 g/ml.

5.3 Iodine, 0,001 N solution.

5.4 Methyl orange, 2 g/l aqueous solution.

6 APPARATUS

Glass apparatus should preferably be fitted with ground glass joints.

Ordinary laboratory apparatus and in particular

6.1 Efficient extractor, for example Soxhlet or Twisselmann or other suitable type.

6.2 Extraction flask, suitable for attaching to the lower end of the extractor (6.1).

6.3 Filter paper discs, pore diameter 10 μm, free from matter soluble in the solvent used (5.1).

6.4 Paper extraction thimble, suitable for use in the extractor (6.1), and free from matter soluble in the solvent used (5.1).

6.5 Cotton wool, free from matter soluble in the solvent used (5.1).

6.6 Efficient water-cooled reflux condenser, suitable for attaching to the upper end of the extractor (6.1).

6.7 Electrical heating device, fitted with a variable temperature control.

NOTE — An assembly of multiple extraction units with individual electrical regulation may be used.

6.8 Water bath, at a temperature of 15 to 25 °C.

6.9 Boiling water bath.

6.10 Oven, capable of being controlled at 50 ± 1 °C.

6.11 Vacuum oven, capable of being controlled at 100 ± 1 °C.

6.12 Beaker, of capacity 600 ml.

6.13 Desiccator, containing an efficient desiccant.

6.14 Analytical balance.

1) In preparation.

7 PROCEDURE

7.1 Preparation of the test sample

Mix the laboratory sample well.

7.2 Test portion

Weigh, to the nearest 0,1 g, 25 to 50 g of the test sample (7.1) according to the expected total fat content, transfer to the beaker (6.12) and suspend in 100 ml of water.

7.3 Hydrolysis

Mix 100 ml of the hydrochloric acid (5.2) with 200 ml of water. Bring the solution to the boil and add to the test portion suspension (7.2).

Heat the mixture just to boiling and maintain for 5 min.

Check that the mixture is free from starch, by transferring a few drops of the mixture to a test tube, cooling to ambient temperature and adding one drop of the iodine solution (5.3). If no colour develops, proceed as in 7.4.

If a blue colour develops, continue boiling the mixture, checking frequently to ensure that the solution is free from starch, using the procedure in the previous paragraph. When the mixture is free from starch, proceed as in 7.4.

7.4 Separation of residual products

Place the beaker and its contents in the water bath (6.8) for 30 min. Stir from time to time in order to ensure an even temperature and to precipitate the fatty materials.

Filter the mixture quantitatively through the filter paper (6.3). Remove the fatty materials adhering to the inside of the beaker using pieces of dry filter paper (6.3) and add these to the residue. Wash the residue and the filter paper with water at ambient temperature until the filtrate is neutral to the methyl orange indicator solution (5.4).

Fold the filter paper containing the residue and pieces of filter paper, place on a watch-glass and dry for 3 h in the oven (6.10), controlled at 50 ± 1 °C.

7.5 Extraction of total fat

Place the filter paper and its contents in the extraction thimble (6.4).

Close the neck of the thimble with cotton wool (6.5) and place it in the extractor (6.1).

Place about 50 ml of the solvent (5.1) in the extraction flask (6.2), previously dried and weighed to the nearest 0,001 g. Attach the flask to the extractor (6.1), and place the condenser (6.6) on the top of the extractor. Place the whole assembly on the heating device (6.7), and turn on the cooling water to the condenser (6.6).

NOTE – Ensure that all connections of the extraction assembly are tight in order to avoid loss of solvent during the extraction.

Control the heating so as to produce 150 to 200 drops of condensate per minute or 7 to 10 siphonings per hour, and continue the extraction for 3 h.

Disconnect the flask containing the extracted fatty materials. Immerse the extraction flask and its contents in the boiling water bath (6.9) and distil off almost all the solvent contained in the flask; place the latter for 1 h in the vacuum oven (6.11), controlled at 100 ± 1 °C. Transfer the flask to the desiccator (6.13), allow it to cool to ambient temperature and weigh it to the nearest 0,001 g.

NOTE – Prolonged drying of the extract at high temperature may lead to a high result due to oxidation of the fats.

7.6 Carry out two determinations on the same test sample (7.1).

8 EXPRESSION OF RESULTS

8.1 Method of calculation and formula

The total fat content, expressed as a percentage by mass of the product as received, is given by the formula

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

where

m_0 is the mass, in grams, of the test portion (7.2);

m_1 is the mass, in grams, of the empty extraction flask (see 7.5);

m_2 is the mass, in grams, of the flask and the total fat after drying.

Take as the result the arithmetic mean of two determinations if the conditions of repeatability (see 8.2) are satisfied. Otherwise, repeat the determinations.

8.2 Repeatability

The difference between two determinations carried out simultaneously or in rapid succession by the same analyst on the same test sample shall not exceed 5 % of the mean value.

9 TEST REPORT

The test report shall indicate the method and the solvent used and the result obtained. It shall also mention all details not specified in this International Standard or which are optional, as well as any possible incidents likely to have influenced the results.

The test report shall give all the information necessary for complete identification of the sample.