INTERNATIONAL STANDARD

ISO 1666

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Starch — Determination of moisture content — Oven-drying method

Amidon et fécule — Détermination de l'humidité — Méthode par séchage à l'étuve

Reference number
ISO 1666:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 1666 was prepared by Technical Committee ISO/TC 93, *Starch (including derivatives and by-products).*

This second edition cancels and replaces the first edition (ISO 1666:1973), which has been technically revised.

Annex A of this International Standard is for information only.

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Starch — Determination of moisture content — Oven-drying method

1 Scope

This International Standard specifies a method for the determination of the moisture content of starch using oven-drying at 130 °C under atmospheric pressure.

The method is applicable to native or modified starch in the dry form.

In special circumstances, for example if the starch contains substances which are unstable at 130 °C, the method is not applicable.

2 Definition

For the purposes of this International Standard, the following definition applies.

2.1 moisture content of starch: Loss in mass of the material under the test conditions specified in this International Standard, expressed as a percentage by mass.

3 Principle

Dehydration of the test portion in an electrically heated drying oven at 130 °C to 133 °C, under atmospheric pressure, for a period of 1 h 30 min.

4 Apparatus

Usual laboratory apparatus and, in particular, the following.

4.1 Analytical balance, capable of weighing to the nearest 0,001 g.

4.2 Dish, made of a metal unaffected by starch under the test conditions (e.g. aluminium), with a suitable tight-fitting lid, the effective surface being such that the test portion when evenly distributed has a thickness corresponding to not more than 0,3 g/cm^2. Suitable dimensions are 55 mm to 65 mm diameter, 15 mm to 30 mm height and 0,5 mm wall thickness.

4.3 Constant-temperature oven, electrically heated, with suitable air circulation, controlled in such a way that the temperature of the air surrounding the test portions and of the shelves carrying the test portions is within the range 130 °C to 133 °C under normal conditions. The heat capacity shall be such that, when the oven is initially adjusted to 131 °C, it can regain this temperature in less than 30 min after insertion of the maximum number of test portions that can be dried simultaneously.

4.4 Desiccator, provided with a thick perforated metal plate for rapid cooling of the dishes, and containing an effective drying agent.

5 Test sample

The test sample shall be free from any hard and lumpy material. It should be received in an airtight and moisture-tight container. After withdrawing the test portions, the remainder of the sample shall be stored in the same container for further tests if required. It shall be homogenized before use.

6 Procedure

6.1 Test portion

Carry out weighings to the nearest 0,001 g.

After drying at 130 °C and cooling in the desiccator (4.4), weigh the dish (4.2) and its lid (m_0). Using the balance (4.1), weigh 5 g ± 0,25 g of the well-mixed sample and transfer it to the dish with the minimum exposure to the atmosphere. Replace the lid and weigh immediately to determine the mass of the test portion and dish (m_1). Distribute the test portion in a uniform layer over the bottom of the dish.

6.2 Determination

Place the open dish containing the test portion in the oven (4.3) preheated to 130 °C, allowing the lid to lean against the dish, and dry at 130 °C to 133 °C for 1 h 30 min, calculated from the moment when the oven temperature again reaches 130 °C.

After this period, rapidly cover the dish and put it in the desiccator.

Never superimpose dishes in the desiccator.

Allow the test portion to cool to room temperature in the desiccator (4.4) for 30 min to 45 min.

When the dish has cooled to room temperature, weigh it and the lid (m_2) within 2 min of its removal from the desiccator.

Carry out at least two determinations on the same test sample.

7 Calculation

The moisture content, expressed as a percentage by mass, is given by the formula

$$(m_1 - m_2) \times \frac{100\ \%}{m_1 - m_0}$$

where

m_0 is the mass, in grams, of the dried empty dish and lid;

m_1 is the mass, in grams, of the dish with the test portion and lid before drying;

m_2 is the mass, in grams, of the dish with the test portion and lid after drying.

Take as the result the arithmetic mean of the two determinations, if their absolute difference does not exceed the value given for the repeatability limit in 8.1.

Report the result to the first decimal place.

8 Precision

Details of an interlaboratory test on the precision of the method are summarized in annex A. The values derived from the interlaboratory test may not be applicable to analyte concentration ranges and matrices other than those given.

8.1 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the sample equipment within a short interval of time, should not be greater than the repeatability limit r given in table 1, in more than 5 % of cases.

8.2 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, should not be greater than the reproducibility limit R given in table 1, in more than 5 % of cases.

Table 1

Starch type	Repeatability limit, r % (*m/m*)	Reproducibility, R % (*m/m*)
Wheat	0,3	0,4
Maize	0,2	0,4
High amylose content	0,2	0,4
Modified waxy maize	0,2	0,4
Cationic maize	0,1	0,5
Pea	0,3	0,5
Potato	0,1	0,3

9 Test report

The test report shall specify:

— the method used;

— the test result obtained; and

— if the repeatability has been checked, the final quoted result obtained.

It shall also mention all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result.

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

Annex A
(informative)
Results of interlaboratory test

An interlaboratory test was conducted in 1989, with seven laboratories participating on seven samples of starches, including two modified starches. The results are given in table A.1.

Table A.1

Parameter	Type of starch						
	Wheat	Maize	High amylose content	Modified waxy maize	Cationic maize	Pea	Potato
No. of laboratories retained after eliminating outliers	7	7	7	7	7	7	6
No. of outliers (laboratories)	0	0	0	0	0	0	1
No. of accepted results	14	14	14	14	14	14	12
Mean value [% (*m*/*m*)]	11,9	13,3	12,4	13,4	11,9	8,64	18,1
Repeatability standard deviation, s_r [% (*m*/*m*)]	0,97	0,049 6	0,059 4	0,067 8	0,035 2	0,087 9	0,028 9
Repeatability coefficient of variation (%)	0,812	0,373	0,480	0,506	0,296	1,02	0,159
Repeatability limit, $r = 2{,}8 \times s_r$ [% (*m*/*m*)]	0,274	0,140	0,168	0,192	0,099 5	0,249	0,081 7
Reproducibility standard deviation, s_R [% (*m*/*m*)]	0,134	0,133	0,114	0,123	0,157	0,172	0,087 9
Reproducibility coefficient of variation (%)	1,12	1,00	0,928	0,916	1,32	1,99	0,486
Reproducibility limit, $R = 2{,}8 \times s_R$ [% (*m*/*m*)]	0,379	0,376	0,324	0,348	0,443	0,486	0,249

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