

ISO

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 1666

STARCH

DETERMINATION OF MOISTURE CONTENT

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1666, *Starch – Determination of moisture content*, was drawn up by Technical Committee ISO/TC 93, *Starch (including derivatives and by-products)*, the Secretariat of which is held by the Deutscher Normenausschuss (DNA).

Work on this question led to the adoption of Draft ISO Recommendation No. 1666, which was circulated to all the ISO Member Bodies for enquiry in September 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	India	South Africa, Rep. of
Chile	Iran	Spain
Colombia	Ireland	Switzerland
Czechoslovakia	Israel	Thailand
France	Netherlands	Turkey
Germany	New Zealand	U.A.R.
Hungary	Poland	United Kingdom

The following Member Body opposed the approval of the Draft :

U.S.A.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in April 1970, to accept it as an ISO RECOMMENDATION.

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STARCH

DETERMINATION OF MOISTURE CONTENT

1. SCOPE AND FIELD OF APPLICATION

This ISO Recommendation describes two methods for the determination of moisture content in starch :

- the oven-drying method at 130 °C at atmospheric pressure (Method 1);
- the oven-drying method at 100 °C or 73 °C at reduced pressure (Method 2).

These methods are applicable to native starches. For the majority of ordinary transactions concerning native starches the oven-drying method at 130 °C (Method 1) has proved satisfactory.

In special circumstances, for example if the starch contains substances unstable at 130 °C or in an atmosphere of high relative humidity, recourse may be had to the vacuum method (Method 2). Results by the latter method are quite similar, but are usually about 0.1 to 0.3 % higher in absolute value than those obtained by the quick oven-drying method (Method 1).

The vacuum method (Method 2) can be carried out at 100 °C or at 73 °C. The temperature of 73 °C should be chosen if the starch contains substances unstable at 100 °C.

2. DEFINITION

For practical purposes the *moisture content* of starch is taken to mean the loss in mass of the material under specified conditions of test.

3. LABORATORY SAMPLE

The sample for examination should be received in an air-tight and moisture-tight container. After withdrawing the test portions, the remainder of the sample should be stored in the same container for further tests if required.

4. METHOD 1 : OVEN-DRYING AT 130 °C AT ATMOSPHERIC PRESSURE

4.1 Principle

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

4.2 Apparatus

4.2.1 Analytical balance.

- 4.2.2 *Dish*, of metal (unaffected by starch under the conditions of test), for example 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 to 65 mm diameter, 15 to 30 mm height and about 0.5 mm wall thickness.
- 4.2.3 *Constant-temperature oven*, electrically heated, with suitable air circulation, controlled in such a way that the temperature of the air and of the shelves carrying the test portions is within the range 130 to 133 °C, in the neighbourhood of the test portions, in normal working. The heat capacity should be such that, when the oven is initially adjusted to 131 °C, it can regain this temperature in less than 30 minutes after insertion of the maximum number of test portions that can be dried simultaneously.
- 4.2.4 *Desiccator*, containing an effective drying agent, and provided with a thick perforated plate of metal for rapid cooling of the dishes.

4.3 Procedure

Carry out weighings to the nearest 0.001 g.

- 4.3.1 *Test portion*. Weigh the dish (4.2.2) and its lid after drying at 130 °C and cooling in the desiccator (4.2.4). Transfer $5 \pm 0.25 \text{ g}$ of the well mixed sample, which should be free from any hard and lumpy material, to the dish with the minimum exposure to the atmosphere. Replace the lid and weigh immediately to determine the mass of the test portion. Distribute the test portion in a uniform layer over the bottom of the dish.

- 4.3.2 *Determination*. Place the open dish containing the test portion in the drying oven (4.2.3) preheated to 130 °C, allowing the lid to lean against the dish, and dry at 130 to 133 °C for 1 hour 30 minutes reckoned from the moment when the oven temperature again reaches 130 °C.

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

NOTE. — The dishes should never be superimposed in the desiccator.

Allow the test portion to cool to room temperature in the desiccator (4.2.4) for 30 to 45 minutes.

When the dish has cooled to room temperature, weigh it within 2 minutes of its removal from the desiccator.

Carry out at least two determinations on the same well mixed laboratory sample.

4.4 Expression of results

- 4.4.1 *Method of calculation*. The moisture content, expressed as a percentage by mass, is equal to

$$(m_2 - m_3) \times \frac{100}{m_2 - m_1}$$

where

m_1 is the mass, in grammes, of the dried empty dish and lid;

m_2 is the mass, in grammes, of the dish with test portion and lid before drying;

m_3 is the mass, in grammes, of the dish with test portion and lid after drying.

Take as the result the arithmetic mean of the two determinations, if the requirements concerning repeatability are satisfied.

Report the result to the first decimal place.

- 4.4.2 *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 moisture in 100 g of the sample.

If it exceeds 0.2 g, the determination should be repeated in duplicate after ensuring that the original sample is thoroughly mixed*.

For the calculation, use only duplicate results that agree to within 0.2 % of moisture in 100 g of the sample.

* If desired, a duplicate test should be made on another day by another analyst or in another oven.

5. METHOD 2 : DRYING AT 100 °C OR 73 °C AT REDUCED PRESSURE

5.1 Principle

Dehydration of the test portion to constant mass in an electrically heated vacuum drying oven or glass drying tube in the presence of phosphorus pentoxide at 100 °C or 73 °C at a pressure of 20 ± 7 mbar*.

5.2 Apparatus

5.2.1 *Analytical balance.*

5.2.2 *Apparatus for maintaining a pressure of 20 ± 7 mbar*.*

5.2.3 *Mercury manometer, for permanent vacuum control.*

and, for procedure A (see clause 5.3) :

5.2.4 *Dish, of metal (unaffected by starch under the conditions of test), for example 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 to 65 mm diameter, 15 to 30 mm height and about 0.5 mm wall thickness.*

5.2.5 *Vacuum drying oven, electrically heated, thermostatically controlled at 100 ± 2 °C or at 73 ± 2 °C.*

5.2.6 *Air drying train, for drying the air at the end of the vacuum period, and assembled in the order :*

- (a) gas washing bottle containing sulphuric acid ($\rho_{20} = 1.83 \text{ g/ml}$);
- (b) safety flask;
- (c) drying tower filled with silica gel.

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

and, for procedure B (see clause 5.4) :

5.2.8 *Dish**, of metal (unaffected by starch under the conditions of test), 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 .*

5.2.9 *Glass or porcelain boat.*

5.2.10 *Drying tube***, of glass, closed at one end and provided at the other end with a ground stopper which carries a semi-capillary tube, with a stop-cock, for evacuation purposes. The test portion may be cooled in this apparatus after drying, thus making a desiccator unnecessary.*

5.2.11 *Constant-temperature oven, electrically heated, or any other system enabling the part of the drying tube containing the metal dish (5.2.8) to be brought to 100 ± 2 °C or to 73 ± 2 °C.*

5.2.12 *Air-drying train, consisting of a gas washing bottle containing sulphuric acid ($\rho_{20} = 1.83 \text{ g/ml}$) connected to a tube containing pure analytical grade phosphorus pentoxide spread on glass wool.*

5.3 Procedure A (vacuum-drying oven method)

Carry out weighings to the nearest 0.0002 g.

5.3.1 *Test portion.* Weigh the dish (5.2.4) and its lid after drying at 100 °C or 73 °C at a pressure of 20 ± 7 mbar* and cooling in the desiccator (5.2.7). Transfer approximately 5 g of the well mixed sample to the dish with the minimum exposure to the atmosphere. Replace the lid and weigh immediately, to determine the mass of the test portion. Distribute the test portion in a uniform layer over the bottom of the dish.

* About 10 to 20 mmHg.

** See Figure 1 in the Annex.

*** See Figure 2 in the Annex.

5.3.2 Determination. Place the open dish (5.2.4) containing the test portion, together with Petri dishes filled with phosphorus pentoxide, in the drying oven (5.2.5) preheated to 100 °C or to 73 °C. Allow the lid to lean against the dish. Close the door of the oven and reduce the pressure to 20 ± 7 mbar*. Dry for a period of 4 hours at 100 ± 2 °C, reckoned from the moment when the oven temperature again reaches 100 ± 2 °C, or for a period of approximately 24 hours at 73 ± 2 °C, maintaining the specified vacuum. Then shut off the vacuum pump and restore atmospheric pressure inside the drying oven by slowly causing air, which has passed through the drying train (5.2.6), to enter the oven. Rapidly place the lid on the dish and put it in the desiccator (5.2.7).

NOTE. — The dishes should never be superimposed in the desiccator.

Allow the test portion to cool to room temperature in the desiccator for 30 to 45 minutes.

When the dish has cooled to room temperature, weigh it within 2 minutes of its removal from the desiccator.

After weighing, redry the test portion for at least 30 minutes when drying at 100 °C, or for a period of approximately 8 hours when drying at 73 °C, allow to cool to room temperature and weigh again. If the difference in mass does not exceed 0.001 g, drying can be considered complete. If it exceeds 0.001 g the drying should be repeated until the difference between successive weighings is less than 0.001 g.

Carry out at least two determinations on the same well mixed laboratory sample.

5.4 Procedure B (drying tube method)

Carry out weighings to the nearest 0.0002 g.

5.4.1 Test portion. Weigh the dish (5.2.8) and its lid after drying in the drying tube (5.2.10) at 100 °C or 73 °C at a pressure of 20 ± 7 mbar* and cooling to room temperature in the drying tube. Transfer approximately 3 g of the well mixed sample to the dish with the minimum exposure to the atmosphere. Replace the lid and weigh immediately to determine the mass of the test portion. Distribute the test portion in a uniform layer over the bottom of the dish.

5.4.2 Determination. Place the open dish (5.2.8) containing the test portion at the bottom of the drying tube (5.2.10). Introduce, near to it, a boat (5.2.9) containing a layer of phosphorus pentoxide about 1 cm thick. Fit the stopper. Gradually bring the pressure in the enclosure to a value of the order of 20 ± 7 mbar* (for example, using a semi-capillary tube), in order to avoid material being thrown out of the dish. Close the connection to the vacuum apparatus. Put the portion of the drying tube (5.2.10) containing the vessel into the oven preheated to 100 °C or to 73 °C.

When the phosphorus pentoxide has consolidated, renew it after restoring atmospheric pressure inside the drying tube by causing air which has passed through the drying train to enter slowly through a capillary tube. Close the drying tube again and continue drying under vacuum at 100 °C or 73 °C as before.

After 4 hours at 100 °C or about 24 hours at 73 °C take the drying tube out of the oven, allow it to cool to room temperature and restore interior atmospheric pressure as described above. Quickly take out the dish, cover it and weigh it.

Continue the dehydration to constant mass (less than 0.0005 g difference between two weighings made at least 30 minutes apart when dehydrating at 100 °C or about 8 hours apart when dehydrating at 73 °C).

Carry out at least two determinations on the same well mixed laboratory sample.

NOTE. — Renew the phosphorus pentoxide as soon as it consolidates at the surface.

* About 10 to 20 mmHg.

5.5 Expression of results

5.5.1 *Method of calculation.* The moisture content, expressed as a percentage by mass, is equal to

$$(m_2 - m_3) \times \frac{100}{m_2 - m_1}$$

where

m_1 is the mass, in grammes, of the dried empty dish and lid;

m_2 is the mass, in grammes, of the dish with test portion and lid before drying;

m_3 is the mass, in grammes, of the dish with test portion and lid after drying.

Take as the result the arithmetic mean of the results of duplicate determinations if the requirements concerning repeatability are satisfied.

Report the result to the first decimal place.

5.5.2 *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 moisture in 100 g of the sample.

If it exceeds 0.2 g, the determination should be repeated in duplicate after ensuring that the original sample is thoroughly mixed*.

For the calculation, use only duplicate results that agree to within 0.2 % of moisture in 100 g of the sample.

5.6 Note on procedure

If the dehydration (using either procedure A or B) is carried out at 73 °C, the drying period prescribed (24 hours) will usually be long enough. In most cases constant mass will be obtained after a period of 8 hours.

6. TEST REPORT

The test report should show the method used and the result obtained. It should also mention any operating conditions not specified in this ISO Recommendation, or regarded as optional, as well as any circumstances that may have influenced the result.

The report should include all details required for complete identification of the sample.

* If desired, a duplicate test should be made on another day, by another analyst or in another oven.