INTERNATIONAL STANDARD 1666

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Starch — Determination of moisture content — Oven-drying methods

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, International Standard ISO 1666 replaces ISO Recommendation R 1666-1970 drawn up by Technical Committee ISO/TC 93, *Starch (including derivatives and by-products).*

The Member Bodies of the following countries approved the Recommendation :

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

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

U.S.A.

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Starch — Determination of moisture content — Oven-drying methods

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies 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 the purpose of this International Standard, the following definition applies :

moisture content of starch : 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 shall 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 h 30 min.

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 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.

4.2.4 *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 ± 0,25 g of the well-mixed sample, which shall 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 h 30 min 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 min.

When the dish has cooled to room temperature, weigh it within 2 min 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

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

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 test portion and lid before drying;

m_2 is the mass, in grams, 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, shall not exceed 0,2 g in 100 g of the product.

If it exceeds 0,2 g, the determination shall be repeated in duplicate after ensuring that the original sample is thoroughly mixed[1].

For the calculation, use only duplicate results that agree to within 0,2 % in absolute value.

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[2].

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 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/cm². 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 (ρ_{20} 1,83 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 5.4) :

5.2.8 *Dish*[3], 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/cm².

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

2) About 10 to 20 mmHg.

3) See Figure 1 in the Annex.

5.2.9 *Glass or porcelain boat.*

5.2.10 *Drying tube*[1], 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 (ρ_{20} 1,83 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 0,000 2 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.

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 h at 100 ± 2 °C, reckoned from the moment when the oven temperature again reaches 100 ± 2 °C, or for a period of approximately 24 h 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 min.

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

After weighing, redry the test portion for at least 30 min when drying at 100 °C, or for a period of approximately 8 h 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. It it exceeds 0,001 g the drying shall 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 0,000 2 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 h at 100 °C or about 24 h 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 0,000 5 g difference between two weighings made at least 30 min apart when dehydrating at 100 °C or about 8 h 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.

1) See Figure 2 in the Annex.

5.5 Expression of results

5.5.1 *Method of calculation*

The moisture content, expressed as a percentage by mass, is equal to

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

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 test portion and lid before drying;

m_2 is the mass, in grams, 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, shall not exceed 0,2 g in 100 g of the product.

If it exceeds 0,2 g, the determination shall be repeated in duplicate after ensuring that the original sample is thoroughly mixed[1].

For the calculation, use only duplicate results that agree to within 0,2 % in absolute value.

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 h) will usually be long enough. In most cases constant mass will be obtained after a period of 8 h.

6 TEST REPORT

The test report shall show the method used and the result obtained. 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 indentification of the sample.

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

ANNEX

DISH AND DRYING TUBE

A.1 DISH FOR TEST PORTION

The dish shown in Figure 1 has a flat bottom of effective surface 16 cm^2 and an internal height of 14 mm. It may be used with the drying tube shown in Figure 2.

Dimensions in millimetres

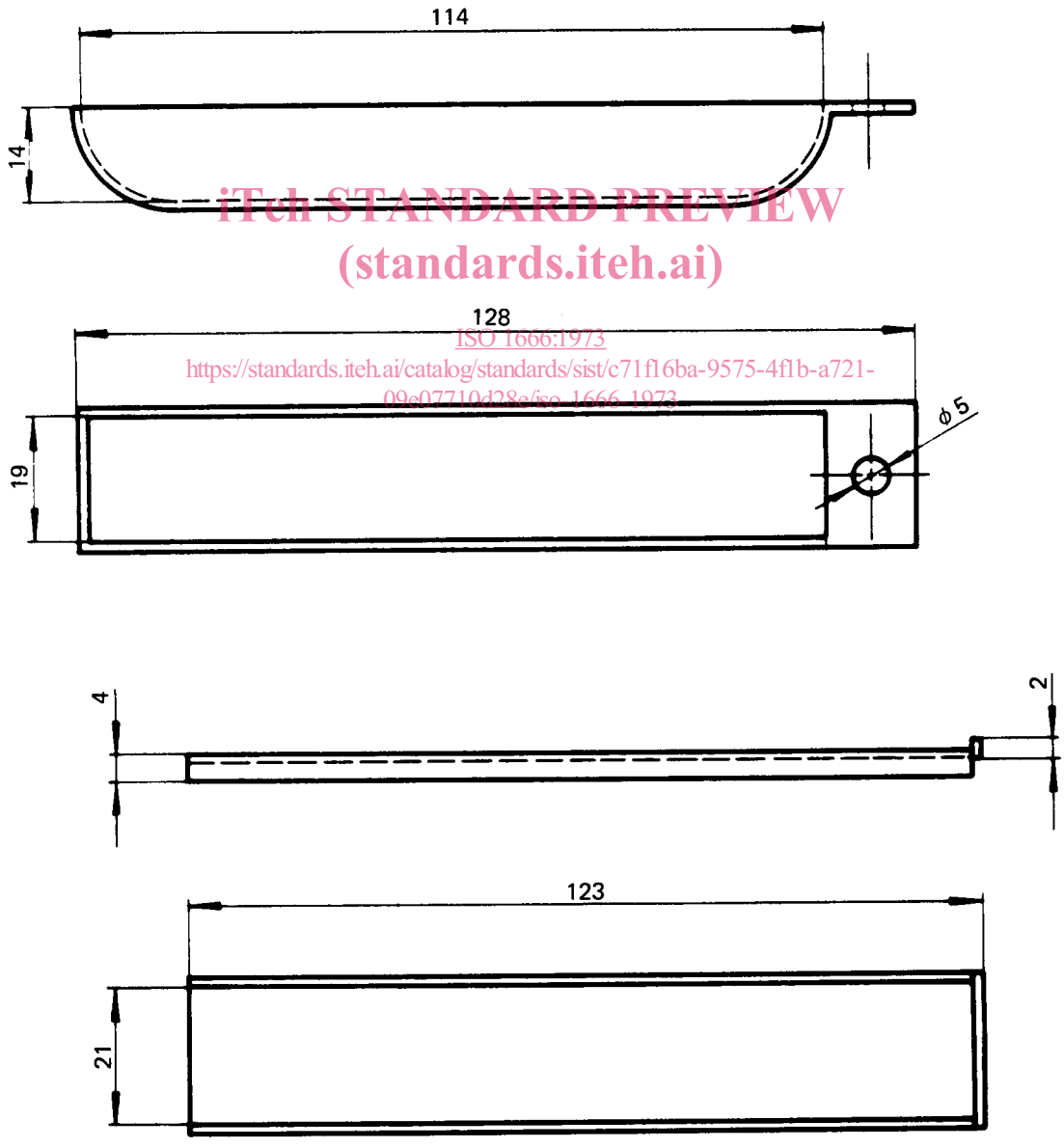

FIGURE 1 – **Diagram of a metal dish** (for guidance only)

A.2 DRYING TUBE

The drying tube shown in Figure 2 has a 40/50 ground joint (40 mm in diameter, 50 mm length of ground portion). It is suitable for use with the dish shown in Figure 1. The olive connection may be replaced by a ground joint.

Dimensions in millimetres

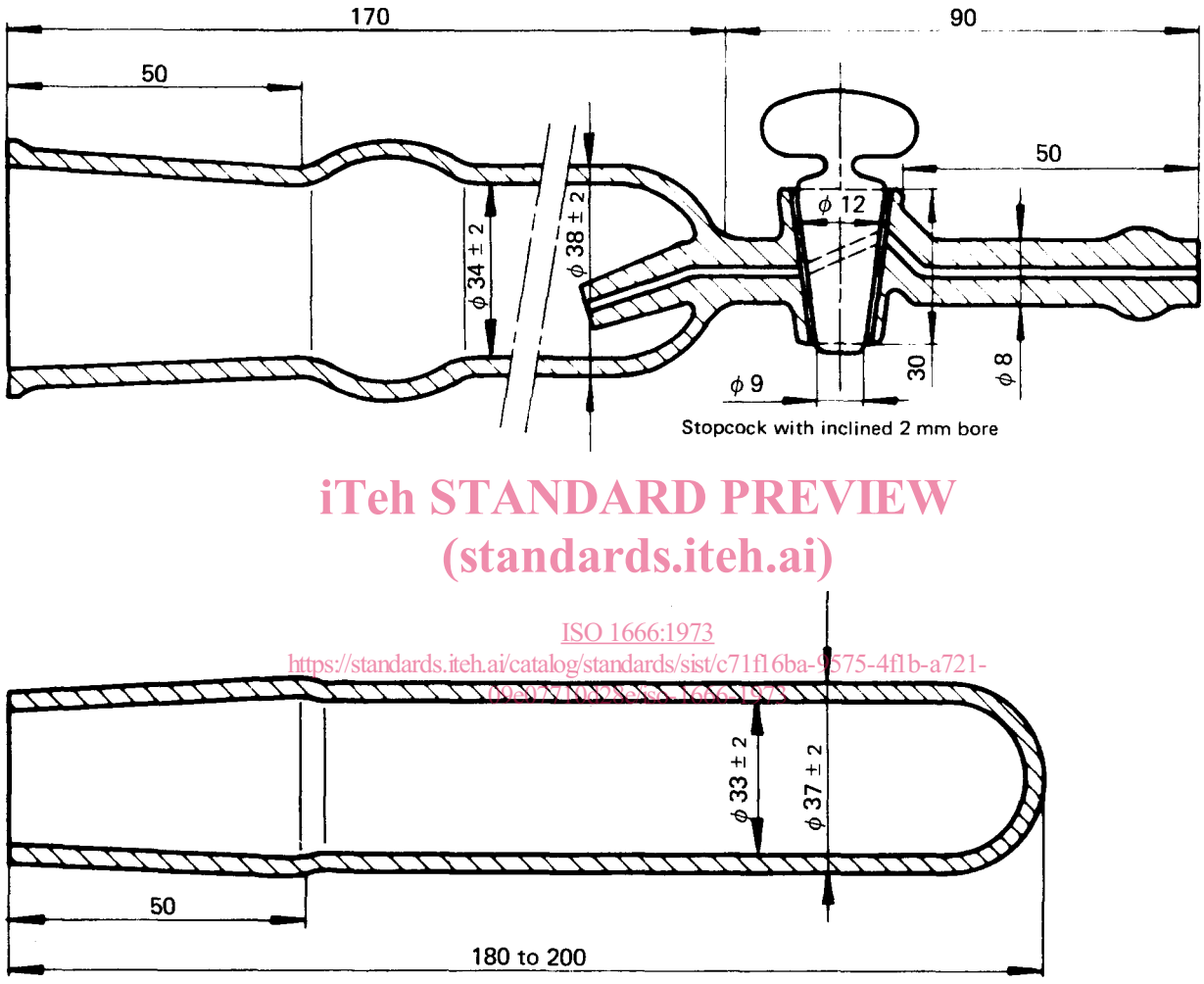

FIGURE 2 – **Diagram of a drying tube** (for guidance only)