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Indian Standard

METHOD OF ANALYSIS FOR FOODGRAINS

PART 2 DETERMINATION OF MOISTURE CONTENT

( First Revision )

ICS 67.060

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 4
NATIONAL FOREWORD

This Indian Standard ( Part 2 ) ( First Revision ) which is identical with ISO 712:1998 'Cereals and cereal products — Determination of moisture content — Routine reference method' issued by the International Organization for Standardization ( ISO ) was adopted by the Bureau of Indian Standards on the recommendation of the Foodgrains, Foodgrain Industries and Starches Sectional Committee and approval of the Food and Agriculture Division Council.

This standard was first published in 1967. In this revision, it is being aligned with the corresponding ISO Standard under dual numbering.

In the adopted standard, certain terminology and conventions are not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'; and

b) Comma ( , ) has been used as a decimal marker while in Indian Standards, the current practice is to use a point ( . ) as the decimal marker.

CROSS REFERENCES

In this adopted standard, the following International Standards are referred to. Read in their respective places, the following:

<table>
<thead>
<tr>
<th>International Standard</th>
<th>Corresponding Indian Standard</th>
<th>Degree of Equivalence</th>
</tr>
</thead>
<tbody>
<tr>
<td>ISO 13690:1999 Cereals, pulses and milled products — Sampling of static batches</td>
<td>IS 14818 : 2000 Cereals, pulses and milled products — Sampling of static batches</td>
<td>Identical</td>
</tr>
</tbody>
</table>

The technical committee responsible for the preparation of this standard has reviewed the provisions of the following ISO Standards and decided that they are acceptable for use in conjunction with this standard:

ISO 711 : 1985 Cereals and cereal products — Determination of moisture content — Basic reference method

ISO 6540 : 1980 Maize — Determination of moisture content ( on milled grains and on whole grains )

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( revised )'.
Indian Standard

METHOD OF ANALYSIS FOR FOODGRAINS

PART 2 DETERMINATION OF MOISTURE CONTENT

( First Revision )

1 Scope

This International Standard specifies a routine reference method for the determination of the moisture content of cereals and cereal products.

It is applicable to the following products: wheat, durum wheat, rice (paddy, husked and milled rice), barley, millet (Panicum miliaceum), rye, oats, triticale, sorghum and kaffir (Sorghum vulgare caffrorum), in the form of grains, milled grains, semolina or flour.

The method is not applicable to maize, for which a method is specified in ISO 6540.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 711, Cereals and cereal products — Determination of moisture content — Basic reference method.

3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

3.1 moisture content
loss in mass, expressed as a percentage, undergone by the product under the conditions specified in this International Standard

4 Principle

If necessary, the sample is ground, after pre-conditioning, when required. A test portion is dried at a temperature of 130 °C ± 3 °C, under conditions which enable a result to be obtained which is in agreement with that obtained by the basic reference method (see ISO 711).

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Analytical balance, capable of weighing to an accuracy of ± 0,001 g.
5.2 Grinding mill, having the following characteristics:
   a) made of material which does not absorb moisture;
   b) easy to clean and having as little dead space as possible;
   c) enabling grinding to be carried out rapidly and uniformly, without appreciable development of heat and, as far as possible, without contact with the outside air;
   d) adjustable so as to obtain particles of the dimensions indicated in 7.1.

5.3 Metal dish, non-corrodible under the test conditions, or, failing this, a glass dish, with a sufficiently tight-fitting lid, and having an effective surface area enabling the test portion to be distributed so as to give a mass per unit area of not more than 0.3 g/cm².

5.4 Constant-temperature oven, electrically heated, capable of being controlled in such a way that, during normal working, the temperature of the air and of the shelves carrying the test portions is 130 °C ± 3 °C in the neighbourhood of the test portions.

The oven shall have a heat capacity such that, when initially adjusted to a temperature of 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.

The effectiveness of the ability of the oven to regain its temperature and the ventilation shall be determined using durum wheat semolina, of maximum particle size 1 mm, as the test material. The ventilation shall be such that after insertion of the maximum number of test portions that the oven will accommodate and drying at a temperature of 130 °C ± 3 °C, then heating the same test portions for 2 h and then for a further 1 h, the results do not differ by more than 0.15 g of moisture per 100 g of sample.

5.5 Desiccator, containing an effective desiccant.

6 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 13690.

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

7 Preparation of test sample

7.1 Products not requiring grinding

Products having the particle size distribution given in Table 1 do not need to be ground before the determination.

Mix the laboratory sample thoroughly before taking the test portion (8.2).

7.2 Products requiring grinding

7.2.1 General

If the sample does not have the particle size characteristics specified in Table 1, it shall be ground either without pre-conditioning (7.2.2) or with pre-conditioning (7.2.3).
Table 1 — Particle size distribution of products not requiring grinding

<table>
<thead>
<tr>
<th>Particle size</th>
<th>Proportion</th>
</tr>
</thead>
<tbody>
<tr>
<td>mm</td>
<td>%</td>
</tr>
<tr>
<td>\leq 1,7 (1,8)^a</td>
<td>100</td>
</tr>
<tr>
<td>&gt; 1,0 (1,0)^b</td>
<td>10</td>
</tr>
<tr>
<td>&lt; 0,5 (0,56)^a</td>
<td>50</td>
</tr>
</tbody>
</table>

^a Nominal aperture size of sieve (in millimetres) in accordance with ISO 3310-1 through which this particle size passes.

^b Nominal aperture size of sieve in accordance with ISO 3310-1 through which this particle size does not pass.

7.2.2 Grinding without pre-conditioning

For products which are not likely to undergo variations in moisture content in the course of grinding (in general, products with a moisture content between 7 % and 17 %\(^1\)), carry out grinding without pre-conditioning.

NOTE The range of moisture contents given for conditioning products before grinding corresponds approximately to a laboratory atmosphere of temperature 20 °C and relative humidity 40 % to 70 %.

Adjust the grinding mill (5.2) to obtain particles of the dimensions indicated in Table 1. Grind a small quantity of the laboratory sample and discard it.

Then quickly grind a quantity of the laboratory sample slightly greater than that required for the test portion (about 5 g), and immediately proceed in accordance with 8.2.2.

7.2.3 Grinding with pre-conditioning

Products which are likely to undergo changes in moisture content in the course of grinding (in general, products with a moisture content of more than 17 %\(^1\) or less than 7 %) shall be pre-conditioned so as to bring their moisture content to between 7 % and 17 %\(^1\) (if possible between 9 % and 15 % (see note in 7.2.2)) before grinding.

If the moisture content is more than 17 %\(^1\) (the more frequent case), weigh, to the nearest 0,001 g, a sufficient quantity of the laboratory sample to provide a test sample slightly greater than 5 g (see 8.2.2) and carry out a predrying operation according to the instructions in 8.3, except that the time of heating in the oven (5.4) shall be 7 min to 10 min and the cooling of the product to laboratory temperature shall be carried out with the dish (5.3) uncovered and without a desiccator, for at least 2 h.

NOTE These times/conditions may not be suitable for all commodities, for example paddy.

If the moisture content is less than 7 %, weigh, to the nearest 0,001 g, a sufficient quantity of the laboratory sample to provide a test sample slightly greater than 5 g (see 8.2.2). Place it in a suitable atmosphere (generally that of the laboratory) and leave it until a moisture content within the limits indicated above is obtained.

After conditioning, weigh the sample to the nearest 0,001 g. Immediately grind it, controlling the grinding so as to obtain particles of the dimensions indicated in Table 1, and immediately proceed in accordance with 8.2.2.

\(^1\) 15 % in the case of oats and rice (paddy, husked and milled rice).
8 Procedure

8.1 Number of determinations

It is required to carry out two single determinations in accordance with 8.2 to 8.3 under the conditions specified in 10.2. If the absolute difference between the two results is more than the repeatability limit \( r \), repeat the determination until the result meets this requirement.

8.2 Test portion

8.2.1 For products not requiring grinding, rapidly weigh, to the nearest 0.001 g, 5 g ± 1 g of the laboratory sample (7.1) in the dish (5.3), previously dried and tared, together with its lid, to the nearest 0.001 g.

8.2.2 In the case of products which have had to be ground, rapidly weigh all the grindings obtained (7.2.2 or 7.2.3) to the nearest 0.001 g, in the dish (5.3), previously dried and tared, together with its lid, to the nearest 0.001 g.

8.3 Drying

Do not open the oven door during drying. At the end of the drying period, remove the dried test portions before placing moist products in the oven, otherwise partial rehydration of the dried test portions may result.

Place the open dish containing the test portion (8.2) together with the lid, in the oven (5.4) and leave for 120 min ± 5 min (90 min for flours) from the moment when the oven temperature is again 130 °C ± 3 °C.

**NOTE** In certain cases, mainly in hot and dry climatic countries, the drying time could be reduced to 60 min ± 5 min, as samples could reach the constant weigh within this period. However this should be checked periodically.

Rapidly take the dish out of the oven, cover it and place it in the desiccator (5.5). When several tests are carried out simultaneously, never stack dishes directly on top of one another in the desiccator, always displace them sideways.

8.4 Weighing

When the dish has cooled to laboratory temperature (generally between 30 min and 45 min after it has been placed in the desiccator), weigh it to the nearest 0.001 g.

9 Calculation and expression of results

The moisture content, \( w \), expressed as a percentage by mass of the product as received, is given by the following equations.

a) Without pre-conditioning:

\[
    w = \left(1 - \frac{m_1}{m_0}\right) \times 100 \%
\]

where

\( m_0 \) is the mass, in grams, of the test portion (8.2.1 or 8.2.2);

\( m_1 \) is the mass, in grams, of the test portion after drying (8.4).
b) With pre-conditioning:

\[
\frac{m_0}{m_1} + m_3 - m_2 \right) \times 100 \%
\]

where

- \( m_2 \) is the mass, in grams, of sample taken before pre-conditioning (7.2.3);
- \( m_3 \) is the mass, in grams, of the preconditioned sample (7.2.3).

The result is the arithmetic mean of two single determinations which meet the repeatability requirement (10.2). It is expressed to two decimal places.

10 Precision

10.1 Interlaboratory test

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

10.2 Repeatability

In the case of wheat samples, 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 same equipment carried out within a short interval of time, will in not more than 5% of cases exceed the repeatability limit \( r \) calculated from the following equation:

\[
r = 0.013 m - 0.06
\]

where \( m \) is the mean of the two test results, expressed in grams per 100 g.

NOTE The results compared with those obtained by the basic reference method (see ISO 711) generally differ by less than 0.15%.

10.3 Reproducibility

In the case of wheat samples, 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 0.59%.

11 Test report

The test report shall specify:

- all information necessary for the complete identification of the sample;
- the sampling method used, if known;
- the test method used, with reference to this International Standard;
- 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(s);
- the test result(s) obtained; or
- if the repeatability has been checked, the final quoted result obtained.
Results of interlaboratory test

An interlaboratory test carried out on wheat in 1994 by the Grain Research Laboratory, Winnipeg, Canada, in which 17 laboratories participated, each of which carried out two determinations on each sample, gave the statistical results shown in Table A.1.

Table A.1 — Statistic results for wheat samples

<table>
<thead>
<tr>
<th></th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
<th>Sample 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of participating laboratories after eliminating outliers</td>
<td>16</td>
<td>17</td>
<td>17</td>
<td>15</td>
</tr>
<tr>
<td>Mean moisture content, g/100 g</td>
<td>11.63</td>
<td>13.22</td>
<td>15.66</td>
<td>17.87</td>
</tr>
<tr>
<td>Repeatability standard deviation, ( s_r ), g/100 g</td>
<td>0.0292</td>
<td>0.0460</td>
<td>0.0367</td>
<td>0.0674</td>
</tr>
<tr>
<td>Coefficient of variation of repeatability, %</td>
<td>0.251</td>
<td>0.348</td>
<td>0.234</td>
<td>0.377</td>
</tr>
<tr>
<td>Repeatability limit ( r (2.83 s_r) ), g/100 g</td>
<td>0.08</td>
<td>0.13</td>
<td>0.10</td>
<td>0.19</td>
</tr>
<tr>
<td>Reproducibility standard deviation, ( s_R ), g/100 g</td>
<td>0.1740</td>
<td>0.2188</td>
<td>0.2417</td>
<td>0.1968</td>
</tr>
<tr>
<td>Coefficient of variation of reproducibility, %</td>
<td>1.497</td>
<td>1.653</td>
<td>1.544</td>
<td>1.101</td>
</tr>
<tr>
<td>Reproducibility limit ( R (2.83 s_R) ), g/100 g</td>
<td>0.49</td>
<td>0.62</td>
<td>0.68</td>
<td>0.56</td>
</tr>
</tbody>
</table>
Bibliography

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Amendments Issued Since Publication

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