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भाग 5 नमी की मात्रा का निर्धारण (संदर्भ पद्धति)

(दूसरा पुनरीक्षण)

Meat and Meat Products — Methods of Test

Part 5 Determination of Moisture Content (Reference Method)

(Second Revision)

ICS 67.120.10

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Price Group 7

Slaughter House and Meat Industry Sectional Committee, FAD 18

NATIONAL FOREWORD

This Indian Standard (Part 5) (Second Revision) which is identical to ISO 1442 : 2023 'Meat and meat products — Determination of moisture content — Reference method' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on recommendation of the Slaughter House and Meat Industry Sectional Committee and approval of the Food and Agriculture Division Council.

This Indian Standard is published in several parts. The other parts in this series are:

- (Part 1) Determination of nitrogen content
- (Part 2) Determination of total ash
- (Part 3) Determination of total fat content
- (Part 4) Determination of free fat content
- (Part 6) Determination of chloride content
 - (Sec 1) Volhard method
 - (Sec 2) Potentiometric method
- (Part 7) Determination of nitrite content
- (Part 8) Determination of nitrate content
- (Part 10) Measurement of *p*H Reference method
- (Part 11) Determination of glucono-delta-lactone content
- (Part 12) Determination of L-(+)-glutamic acid content Reference method
- (Part 13) Detection of condensed phosphates
- (Part 14) Determination of starch content
- (Part 15) Determination of L (-) hydroxyproline content
- (Part 17) Determination of starch and glucose content Enzymatic method

This standard (Part 5) was originally published in 1971 and was subsequently revised in 2001 as identical adoption of ISO 1442 : 1997 'Meat and meat products — Determination of moisture content (reference method)' under dual numbering. Subsequently, ISO 1442 was revised in 2023. Accordingly, the second revision of the standard has been undertaken to harmonize with the latest version of ISO 1442 and is an identical adoption of the ISO document under dual numbering.

The text of ISO standard has been approved as suitable for publication as an Indian Standard without deviations. Certain terminologies and conventions are, however, 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.

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Indian Standard

MEAT AND MEAT PRODUCTS — METHODS OF TEST

PART 5 DETERMINATION OF MOISTURE CONTENT (REFERENCE METHOD)

(Second Revision)

1 Scope

This document specifies two reference methods for the determination of the moisture content of meat and meat products: a direct drying method and a distillation method.

The direct drying method is applicable to meat and meat products with low volatile substances in addition to moisture.

The distillation method is applicable to meat and meat products with high volatile substances in addition to moisture.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, Water for analytical laboratory use — Specification and test methods

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at <u>https://www.electropedia.org/</u>

3.1

moisture content

loss in mass obtained under a direct drying method or content of water extracted or collected under a distillation method, divided by the mass of the test portion

Note 1 to entry: As determined using the conditions specified in this document.

Note 2 to entry: Moisture content is expressed as a percentage by mass.

3.2

test result

value of a characteristic obtained by carrying out a specified test method

[SOURCE: ISO 5725-1:2023, 3.1, modified — Notes to entry deleted.]

4 Principle

4.1 Direct drying method

Thorough mixing of the test portion with sand and drying to constant mass at (103 ± 2) °C.

4.2 Distillation method

Using the physicochemical properties of the moisture in the meat and meat products, the moisture in the meat and meat products is co-distilled with toluene or xylene using a moisture analyser, then the moisture content in the sample is calculated from the volume of the received water.

5 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in CAC/GL 50-2004.

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

Start from a representative sample of at least 200 g.

Store the sample in such a way that deterioration and change in composition are prevented.

6 Preparation of test sample

Homogenize the laboratory sample with the appropriate equipment (7.2.1). For frozen samples, samples are first thawed at 15 °C to 25 °C. Take care that the temperature of the sample material does not rise above 25 °C. If a mincer is used, pass the sample at least twice through the equipment.

Fill a suitable airtight container with the prepared test sample, close the container and store in such a way that deterioration and change in composition are prevented. Analyse the test sample as soon as practicable. If the homogenized sample cannot be tested in time, it should be sealed and refrigerated at 4 °C to 7 °C, but always within 24 h after homogenization.

7 Direct drying method

7.1 Reagents

Use only reagents of recognized analytical grade.

7.1.1 Sand, clean, acid-washed, of a size such that it passes through a sieve of aperture size 1,4 mm and stays on a sieve of aperture size $250 \mu m$.

Dry the sand before use at 150 °C to 160 °C and store in an airtight closed bottle.

If clean (acid-washed) sand is not available, the sand shall be cleaned by the following procedure.

Wash the sand with running water. Boil the sand with dilute hydrochloric acid, $\rho_{20} = 1,19$ g/ml, diluted (1+1), for 30 min while stirring continuously. Repeat the boiling operation with another portion of the acid until the acid no longer turns yellow after boiling. Wash the sand with distilled water until the test for chloride is negative. For storage, dry the sand at 150 °C to 160 °C.

7.2 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

7.2.1 Mechanical or **electrical equipment**, capable of homogenizing the laboratory sample. This includes a high-speed rotational cutter, or a mincer fitted with a plate with holes not exceeding 4,0 mm in diameter.

7.2.2 Flat dish, made of glass, porcelain or metal (e.g. nickel, aluminium, stainless steel), of diameter at least 60 mm and height about 25 mm.

- **7.2.3** Thin glass rod, flattened at one end, slightly longer than the diameter of the dish (7.2.2).
- **7.2.4 Drying oven**, electrically heated, capable of operating at (103 ± 2) °C.
- **7.2.5 Desiccator**, containing an efficient desiccant, such as silica gel.
- **7.2.6** Analytical balance, capable of weighing to the nearest 0,001 g.

7.3 Procedure

7.3.1 General

If it is required to check whether the repeatability limit (see <u>7.5.2</u>) is met, take two single determinations in accordance with <u>7.3.4</u> under repeatability conditions.

7.3.2 Preparation of the dish and sand

Transfer to the dish (7.2.2) a quantity of sand (7.1.1) equal to three to four times the mass of the test portion (see 7.3.3) and dry the dish, sand and glass rod (7.2.3) for 30 min in the oven (7.2.4) set at 103 °C.

Allow the dish with its contents and the glass rod to cool in the desiccator (7.2.5) to room temperature and weigh to the nearest 0,001 g (m_0).

7.3.3 Test portion

Transfer between 5 g to 8 g of the prepared test sample (see <u>Clause 6</u>) to the prepared dish (see <u>7.3.2</u>) and weigh the dish with its contents and the glass rod to the nearest 0,001 g (m_1).

7.3.4 Determination

7.3.4.1 Mix the contents of the dish by means of the glass rod (7.2.3).

In cases of difficulty in mixing the test portion with sand, ethanol shall be added as necessary. In this case, the ethanol shall be evaporated gently before drying the sample in the oven.

Heat the dish with its contents and the glass rod for 2 h in the oven (7.2.4) set at 103 °C. Remove the dish with its contents and the glass rod from the oven and place them in the desiccator (7.2.5).

Allow the dish, its contents and the glass rod to cool to room temperature, then weigh to the nearest 0,001 g.

7.3.4.2 Repeat the heating, cooling and weighing operations specified in $\underline{7.3.4.1}$ until the results of two successive weighings (m_2), separated by 1 h of heating, do not differ by more than 0,1 % of the mass of the test portion.

7.4 Expression of results

Calculate the moisture content, *w*, as a percentage by mass, using <u>Formula (1)</u>:

$$w = \frac{m_1 - m_2}{m_1 - m_0} \times 100 \tag{1}$$

where

- m_0 is the mass, in grams, of the dish, rod and sand (see <u>7.3.2</u>);
- m_1 is the mass, in grams, of the dish containing the test portion, rod and sand, before drying (see <u>7.3.3</u>);
- m_2 is the mass, in grams, of the dish containing the test portion, rod and sand, after drying (see <u>7.3.4.2</u>).

Report the result round to one decimal place.

7.5 Precision

7.5.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are given in <u>Annex A</u>. The values derived from this interlaboratory test are not necessarily applicable to concentration ranges and matrices other than those given.

7.5.2 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 same equipment within a short interval of time, should not be greater than the value of *r* given by Formula (2):

$$r = 0,593 \% + 0,001 7 w \tag{2}$$

where is the *w* mean moisture content of both results, expressed as a percentage by mass.

7.5.3 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 value of R given by Formula (3):

$$R = 0,797 \ \% + 0,004 \ 71 \ w$$
(3)

where \overline{w} is the mean moisture content of both results, expressed as a percentage by mass.

8 Distillation method

8.1 Reagents

Use only reagents of recognized analytical grade, and water conforming to at least grade 3 in accordance with ISO 3696.

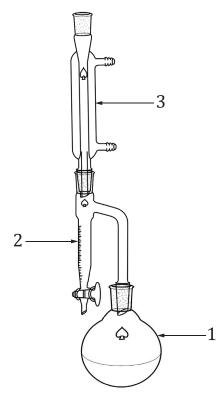
8.1.1 Toluene or xylene, analytical purity.

8.2 Preparation of reagents

Take toluene or xylene (8.1.1) and saturate with water. Then separate the water layer, distillate and collect the distillate for use.

8.3 Apparatus

8.3.1 Moisture analyser, as shown in Figure 1 (with adjustable heating sleeve), water receiving tube capacity (5 ml), minimum scale value (0,1 ml), capacity error less than 0,1 ml.



Кеу

- 1 distillation flask (250 ml)
- 2 moisture receiving tube, with scale
- 3 condenser tube

Figure 1 — Moisture analyser

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

8.4 Procedure

Weigh, to the nearest 0,001 g, an appropriate amount of the sample (final distilled water shall be between 2 ml and 5 ml, but the maximum sample amount shall not exceed 2/3 volume of the distillation flask) and transfer to a 250 ml distillation flask. Add 75 ml of newly distilled toluene (or xylene), connect the condenser tube with the moisture receiving tube, inject toluene (or xylene) from the top of the condenser tube and fill the moisture receiving tube. At the same time, make a blank test of toluene (or xylene) (V_0).

Bring to boil and distil slowly, about two drops per second, until most of water distils over. Then increase the rate of distillation to about four drops per second. When the water volume in the moisture-receiving tube no longer increases, rinse carefully with toluene (or xylene) from the top of the condenser tube. If water droplets are attached to the condensation tube, they can be wiped off by a copper wire with a small rubber. Continue distillation until there are no water droplets at the upper part of the moisture-receiving tube and the inner wall of the condensation tube. When the horizontal surface of the receiving tube remains unchanged for 10 min, read the volume (*V*) of the moisture from the scale.

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8.5 Expression of results

Calculate the moisture content, *w*, as a percentage by mass, using Formula (4):

$$w = \frac{V - V_0}{m} \times \rho \times 100 \tag{4}$$

where

V is the volume of moisture in the receiving tube (ml);

 V_0 is the volume of moisture in the receiving tube of blank test (ml);

m is the sample mass (g);

 ρ is the density of water (g/ml).

Three-bit valid numbers are reserved for the results of the calculation.

8.6 Precision

8.6.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are given in <u>Annex A</u>. The values derived from this interlaboratory test are not necessarily applicable to concentration ranges and matrices other than those given.

8.6.2 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 same equipment within a short interval of time (for the results, see <u>Annex A</u>).

8.6.3 Reproducibility

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment (for the results, see <u>Annex A</u>).

9 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 document, i.e. ISO 1442;
- all operating details not specified in this document, or regarded as optional, together with details of any incidents which can influence the test result(s);
- the test result obtained, or the two test results obtained if the repeatability has been checked;
- the date of the test.

Annex A

(informative)

Precision of the method

The interlaboratory collaborative trial test was organized in 2022 with 10 laboratories participating in the determination of moisture content in a Fapas quality control sample with the concentration range of 61,2% to 63,0% as well as with an assigned value of 62,1%.

All the precision data are shown in <u>Table A.1</u> for reference.

Table A.1 — Summary of the results of the interlaboratory trial test on Fapas canned meat

Index	Method	m	0 %	M %	s _r %	C _{V,r} %	r %	s _R %	C _{V,R} %	R %
Moisture	Distillation method	10	0	62,0	0,214	0,35	0,599	0,252	0,41	0,705
Key:										
<i>m</i> is the number of laboratories after outlier rejection;										
<i>o</i> is the percentage of outliers;										
<i>M</i> is the general mean value of the results;										
s_r is the standard deviation of repeatability;										
C_{Vr} is the coefficient of variation of repeatability;										
<i>r</i> is the repeatability, $r = 2.8 \times s_r$;										
s_R is the standard deviation of reproducibility;										
C_{VR} is the coefficient of variation of reproducibility;										
R is the reproducibility, $R = 2.8 \times s_R$.										

Bibliography

- [1] ISO 5725-1:2023, Accuracy (trueness and precision) of measurement methods and results Part 1: General principles and definitions
- [2] CAC/GL 50-2004, General guidelines on sampling

(Continued from second cover)

The Committee has reviewed the provision of the following International Standard referred in this adopted standard and has decided that it is acceptable for use in conjunction with this standard:

International Standard

Title

ISO 3696 Water for analytical laboratory use — Specification and test methods

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 : 2022 'Rules for rounding off numerical values (*second revision*)'.

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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Amendments Issued Since Publication

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