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Pulses — Determination of moisture content — Air-oven method

Légumineuses — Détermination de la teneur en eau — Méthode par séchage à l'étuve

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*.

This second edition cancels and replaces the first edition (ISO 24557:2009), which has been technically revised.

The main changes are as follows:

— modified description of the apparatus to be used for consistency with ISO 6540 and ISO 712-1, which give moisture determination methods for corn and cereals, respectively;

— minor modifications of the protocol to align with ISO 6540 and ISO 712-1 to simplify the daily routine of laboratories.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Pulses — Determination of moisture content — Air-oven method

1 Scope

This document specifies a routine reference method for the determination of moisture content of pulses.

This document is applicable to chickpeas, lentils, peas, lupinus and all classes of beans with the exception of soybeans.

2 Normative references

There are no normative references in this document.

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 https://www.electropedia.org/

3.1
moisture content
loss of mass fraction

Note 1 to entry: The moisture content is expressed as a percentage mass fraction of loss undergone by the product under the conditions specified in this document.

4 Principle

The method determines moisture content as the loss of mass fraction, expressed as a percentage, of a sample when heated under specified conditions. A preconditioning stage is used to minimize moisture loss during the grinding stage.

5 Apparatus

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

5.1 Laboratory mill, having the following characteristics:

a) made of material that does not absorb moisture;

b) easy to clean and having as little dead space as possible;

c) enabling grinding of 30 g of pulses grains 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 particle size characteristics given in Table 1.

Table 1 — Particle size characteristics of products not requiring grinding

Particle size characteristics mm	Proportion %
≤ 1,7	100
> 1,0	≤ 10
< 0,5	≥ 50

Cutting grinders [1] cooled by a water-circulation systems with a rotation speed in the range of 20 000 r/min to 25 000 r/min are strongly recommended and suitable with these requirements.

5.2 Constant temperature oven, either gravity-convection or mechanical-convection, capable of being maintained within the range of 130 °C to 133 °C.

The oven shall have a heat capacity such that, when initially adjusted to a temperature of 131,5 °C, it can again reach this temperature in less than 45 min (preferably in less than 30 min) after insertion of the maximum number of test portions that can be dried simultaneously.

For the verification of the condition 131,5 °C ± 1,5 °C, a metrological control shall be performed.

An alternative to the metrological control can be done with the determination of the effectiveness of the ventilation using durum wheat semolina, of maximum particle size 1 mm, as the test material. The ventilation may be tested by:

a) inserting the maximum number of test portions that the oven can accommodate;

b) drying them at a temperature of 131,5 °C ± 1,5 °C;

c) heating the same test portions for 2 h and then for a further 1 h.

The results should not differ by more than 0,15 g of moisture per 100 g of sample.

5.3 Moisture metal dish, non-corrodible under the test conditions, or **glass dish**, with a lid and having an effective surface area enabling the test portion to be distributed so as to give a mass per unit area of ≤ 0,3 g/cm^2.

5.4 Drying trays, made of a non-absorbent material (glass or metal), and having an effective surface area that enables a test portion of 50 g to be a single layer.

5.5 Airtight desiccator, with an effective desiccant.

5.6 Analytical balance, able to weigh with an accuracy of ±0,001 g and therefore having a display accuracy of 0,000 1 g capable of being read to at least the nearest 1 mg.

6 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 24333.

Ensure that the laboratory receives a sample that is truly representative and that has not been damaged or changed during transport and storage.

1) The IKA A 10 and Foss Knifetec grinders are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.

7 Procedure

7.1 General

If sample moisture content is within the range 9 % mass fraction to 13 % mass fraction, proceed directly to 7.3.

Perform two single determinations per laboratory sample under the conditions specified in 7.2 to 7.4. If the absolute difference between the two results is more than the repeatability limit, *r*, repeat the determination until the results meet this requirement.

7.2 Preconditioning of laboratory sample

Mix the sample thoroughly and weigh approximately 50 g of a representative portion of unground laboratory sample into a drying tray (preferably to a single layer). Record the mass of sample, m_{L0}, to the nearest 0,001 g.

Precondition the sample by either of the following methods:

a) Preferred procedure: Dry the laboratory samples (as specified in 7.4) in the oven (5.2) between 130 °C and 133 °C for 15 min. Then cool the uncovered sample to room temperature without use of a desiccator, for 2 h at least.

NOTE The drying for pre-conditioning can be done at a lowest temperature (i.e. 80 °C) for a longer time (i.e. 1 h).

b) Optional procedure: Air dry the laboratory samples in a well-ventilated drying cabinet or any location where they are not disturbed. Air dry for 48 h. The room temperature and humidity affect the level to which the sample dries.

If using moisture dishes, place lids on the dishes and reweigh the laboratory sample after preconditioning. Record the mass of the sample, m_{L1}, to the nearest 0,001 g.

Ensure that the moisture content after preconditioning has been reduced to between 9 % mass fraction and 13 % mass fraction. Therefore, check the value periodically. Checking is even more important for the optional procedure [see b) above] as different locations vary in humidity, which affects the desired preconditioned moisture levels.

7.3 Grinding and test portion

Grind the sample (preconditioned or not, depending of the estimated water content) using the laboratory mill (5.1). Rapidly mix the sample with a spoon or spatula and immediately transfer a test portion of 5 g to 8 g into a previously dried and tared moisture dish. Cover the moisture dish and weigh. Record the mass of the test portion, m_{t0}, to the nearest 0,001 g. Clean the mill between samples.

If it is not possible to weigh the test portion immediately after grinding, store the ground laboratory sample in a moisture-proof container until it is weighed.

7.4 Drying

Place open dishes containing the test portion (see 7.3), together with the lid, in the oven (5.2) at 131,5 °C ± 1,5 °C and heat for 90 min after the oven regains its temperature.

Remove the dishes from the oven, cover rapidly and transfer to a desiccator as quickly as possible. Weigh the dishes and contents after they have reached room temperature (normally 45 min to 60 min) and record the mass of the test portion, m_{t1}.

8 Calculation and expression of the results

8.1 Calculation

Use one of the following formulae to determine the total loss of mass fraction, expressed as a percentage, due to moisture removal, w_{H_2O}:

Without pre-conditioning:

$$w_{H_2O} = \left(1 - \frac{m_{t1}}{m_{t0}}\right) \times 100$$

With pre-conditioning:

$$w_{H_2O} = \left(1 - \frac{m_{t1}\, m_{L1}}{m_{t0}\, m_{L0}}\right) \times 100$$

where

m_{L0} is the mass, in grams, of the laboratory sample before preconditioning;

m_{L1} is the mass, in grams, of the laboratory sample after preconditioning;

m_{t0} is the mass, in grams, of the test portion before oven drying;

m_{t1} is the mass, in grams, of the test portion after oven drying.

8.2 Expression of results

Calculate the arithmetic mean of two determinations satisfying the repeatability conditions (see 9.2). Round the result to the nearest two decimal places.

9 Precision

9.1 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 are not necessarily applicable to concentration ranges and matrices other than those given.

9.2 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, shall in not more than 5 % of cases be greater than 0,2 %, which is the repeatability limit, *r*, given in Tables A.1 and A.2 and Figure A.1.

9.3 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, shall in not more than 5 % of cases be greater than 0,44 %, which is the reproducibility limit, *R*, given in Tables A.1 and A.2 and Figure A.1.

9.4 Critical difference

9.4.1 General

Critical difference (D_C) is the difference between two averaged values obtained from two test results under repeatability conditions.

9.4.2 Comparison of two groups of measurements in one laboratory

The critical difference (D_C) between two averaged values obtained from two test results under repeatability conditions is given by:

$$D_C = 2{,}8 s_r \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2{,}8 s_r \sqrt{\frac{1}{2}} = 1{,}98 s_r = 0{,}08$$

where

s_r is the standard deviation of repeatability;

n_1 and n_2 are the number of test results corresponding to each of the averaged values.

9.4.3 Comparison of two groups of measurements in two laboratories

The critical difference (D_C) between two averaged values obtained in two different laboratories from two test results under repeatability conditions is given by:

$$D_C = 2{,}8 \sqrt{s_R^2 - s_r^2} \left(1 - \frac{1}{2n_1} + \frac{1}{2n_2}\right) = 2{,}8 \sqrt{s_R^2 - 0{,}55 s_r^2} = 0{,}44$$

where

s_r is the standard deviation of repeatability;

s_R is the standard deviation of reproducibility;

n_1 and n_2 are the number of test results corresponding to each of the averaged values.

9.5 Uncertainty, U

It is possible to evaluate measurement uncertainties using data obtained from studies carried out in accordance with ISO 5725-2: the reproducibility standard deviation obtained during an interlaboratory test is a valid basis to assess measurement uncertainty because, by definition, uncertainty characterizes the dispersion of values that can be reasonably attributed to the parameter.

The calculated expanded standard uncertainty should be ≤ ±2 reproducibility standard deviations.

Uncertainty, U, attached to results for laboratories under repeatability conditions should be estimated at a maximum of:

$$U = 2 s_R = 0{,}32$$

where s_R is the standard deviation of reproducibility.

10 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) the test method used, with reference to this document, i.e. ISO 24557:2024;

c) the date of the test;

d) the test results obtained, clearly mentioning the method of expression used;

e) all operating details not specified in this document, or regarded as optional, together with details of any incidents which can have influenced the test results.

Annex A
(informative)

Results of an interlaboratory test

An international proficiency test involving five laboratories was carried out on pulse samples including peas, horse beans and fava beans.

The proficiency test was organized by BIPEA during the period from May 2022 to December 2023 and the results obtained were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to give the precision data shown in Tables A.1 and A.2.

There is no statistical link between r or R value and the moisture content (see Figure A.1) and it has been chosen to take constant values for the repeatability limit, r (mean of r values), and the reproducibility limit, R (mean of R values).

Table A.1 — Results in peas (six samples)

Parameter	Pea 1	Pea 2	Pea 3	Pea 4	Pea 5	Pea 6
Number of laboratories participating	5	5	5	5	5	5
Number of test results from remaining laboratories	10	10	10	10	10	10
Mean value, %	12,08	12,38	11,53	10,83	11,40	12,57
Repeatability standard deviation, s_r	0,02	0,05	0,02	0,03	0,04	0,05
Coefficient of variation of repeatability, %	0,17	0,40	0,17	0,28	0,35	0,40
Repeatability limit, r (2,8 s_r)	**0,06**	**0,14**	**0,06**	**0,08**	**0,11**	**0,14**
Reproducibility standard deviation, s_R	0,13	0,21	0,09	0,03	0,15	0,08
Coefficient of variation of reproducibility, %	1,08	1,70	0,78	0,28	1,32	0,64
Reproducibility limit, R (2,8 s_R)	**0,36**	**0,59**	**0,25**	**0,08**	**0,42**	**0,22**

Table A.2 — Continued results in other pulses (six samples)

Parameter	Horse beans	Horse beans	Fava beans	Horse beans	Horse beans	Horse beans
Number of laboratories participating	5	5	5	5	5	5
Number of test results from remaining laboratories	10	10	10	10	10	10
Mean value, %	12,00	13,04	15,46	12,86	12,64	13,48
Repeatability standard deviation, s_r	0,03	0,03	0,04	0,04	0,06	0,05
Coefficient of variation of repeatability, %	0,25	0,23	0,26	0,31	0,47	0,37
Repeatability limit, r (2,8 s_r)	**0,08**	**0,08**	**0,11**	**0,11**	**0,17**	**0,14**
Reproducibility standard deviation, s_R	0,15	0,27	0,33	0,16	0,21	0,09
Coefficient of variation of reproducibility, %	1,25	2,07	2,13	1,24	1,66	0,67
Reproducibility limit, R (2,8 s_R)	**0,42**	**0,76**	**0,92**	**0,45**	**0,59**	**0,25**