INTERNATIONAL STANDARD

ISO 24557

First edition
2009-10-01

Pulses — Determination of moisture content — Air-oven method

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

Reference number
ISO 24557:2009(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Foreword

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ISO 24557 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*.

Pulses — Determination of moisture content — Air-oven method

1 Scope

This International Standard specifies a routine reference method for the determination of moisture content of pulses. The procedure is applicable to chickpeas, lentils, peas, and all classes of beans with the exception of soybeans.

NOTE The method is based on AACC approved method 44-17[4].

2 Terms and definitions

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

2.1
moisture content
loss of mass fraction undergone by the product under the conditions specified in this International Standard

NOTE The moisture content is expressed as a percentage mass fraction.

3 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.

4 Apparatus

4.1 Laboratory mill[1)], capable of grinding without undue exposure to atmosphere and without appreciable heating. The mill shall be able to grind large-seeded pulses, such as beans.

Required particle size, d, after grinding:

d < 0,5 mm: more than 20 % mass fraction;

d < 1,0 mm: 70 % mass fraction;

d < 1,7 mm: 100 % mass fraction.

NOTE Grinders operating at speeds higher than 3 600 r/min are unsatisfactory due to excessive moisture loss during grinding, which results in moisture values lower than actual.

1) The Thomas Wiley model ED5 with a 1 mm sieve (1 260 r/min) and the laboratory mill 3303 produced by Perten Instruments, with settings 0 to 4 (3 600 r/min), are examples of suitable devices available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products. Other equipment may be used if it can be shown to give comparable results.

4.2 Oven, either gravity-convection or mechanical-convection, capable of being maintained at 130 °C ± 1 °C uniformly throughout. A thermometer should be situated in the oven so the tip is no more than 100 mm from the top of the moisture dishes.

Determine the effectiveness of the ventilation 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 can accommodate, and drying at a temperature of 130 °C ± 1 °C, the results, after heating the same test portions for 2 h and then for a further 1 h, do not differ by more than 0,15 g of moisture per 100 g of sample.

4.3 Moisture dishes, of heavy gauge aluminium and provided with tightly fitting covers. Identify both dish and cover using the same number and clean them between uses with a vacuum or soft cloth.

4.4 Drying trays, made of a non-absorbent material such as heavy gauge aluminium, of dimensions about 100 mm × 130 mm with tapered 15 mm sides.

4.5 Airtight desiccator, with an effective desiccant.

4.6 Analytical balance, capable of being read to at least the nearest 1 mg.

5 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 24333[3].

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

6 Procedure

6.1 General

If sample moisture content is within the range 9 % mass fraction to 11 % mass fraction, proceed directly to 6.3.

Perform two single determinations per laboratory sample under the conditions specified in 8.2. If the absolute difference between the two results is more than the repeatability limit, r, repeat the determination until the results meet this requirement.

6.2 Preconditioning of laboratory sample

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

6.2.2 Precondition the sample by either of the methods below.

a) *Preferred procedure*: remove the lids from the moisture dishes and dry the laboratory samples (as specified in 6.2.1) in the oven (4.2) at 130 °C ± 1 °C for 15 min; then cool moisture dishes uncovered to room temperature without use of a desiccator, for 2 h.

b) *Optional procedure*: remove the lids from the moisture dishes and 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.

6.2.3 If using moisture dishes, place lids on dishes and reweigh the laboratory sample after preconditioning. Record the mass of 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 11 % mass fraction; therefore, check the value periodically. Checking is even more important for the optional procedure [6.2.2 b)] as different locations vary in humidity, which affects the desired preconditioned moisture levels.

6.3 Grinding and test portion

Grind the remaining preconditioned sample using the laboratory mill (4.1). Rapidly mix the sample with a spoon or spatula and immediately transfer a test portion of 2 g to 3 g into a previously dried and tared moisture dish. Cover the moisture dish and weigh, and 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.

6.4 Drying

Place open dishes containing the test portion (6.3), together with the lid, in the oven (4.2) at 130 °C ± 1 °C and heat for 120 min after the oven regains its temperature.

The oven should regain temperature within 15 min to 20 min after insertion of a full load (24 moisture dishes). Do not use the oven if it requires a longer time to recover.

Remove dishes from the oven, cover rapidly, and transfer to a desiccator as quickly as possible. Weigh 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}.

7 Calculation and expression of results

7.1 Calculation

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

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

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.

7.2 Expression of results

Calculate the arithmetic mean of two determinations satisfying the repeatability conditions (8.2). Round the result to the nearest one decimal place.

8 Precision

8.1 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.

8.2 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 0,3 %, which is the repeatability limit, r, given in Annex A (Tables A.1 and A.2 and Figure A.1).

8.3 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 0,5 %, which is the reproducibility limit, R, given in Annex A (Tables A.1 and A.2 and Figure A.1).

8.4 Critical difference, CD

8.4.1 General

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

8.4.2 Comparison of two groups of measurements in one laboratory

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

$$\mathrm{CD} = 2{,}8 s_r \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2{,}8 s_r \sqrt{\frac{1}{2}} = 1{,}98 s_r = 0{,}22$$

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.

8.4.3 Comparison of two groups of measurements in two laboratories

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

$$\mathrm{CD} = 2{,}8 \sqrt{s_R^2 - s_r^2} \left(1 - \frac{1}{2n_1} + \frac{1}{2n_2}\right) = 2{,}8 \sqrt{s_R^2 - 0{,}55 s_r^2} = 0{,}45$$

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.

8.5 Uncertainty, U

Uncertainty, U, attached to results for laboratories under repeatability conditions is equal to:

$$U = 2s_R = 0{,}36$$

where s_R is the standard deviation of reproducibility.

9 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 sampling method used, if known;

c) the test method used, with reference to this International Standard;

d) 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 results;

e) the test results obtained, clearly mentioning the method of expression used.