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Check of the calibration of moisture meters —

Part 2 :
Moisture meters for oilseeds

Contrôle d'étalonnage des humidimètres —

Partie 2 : Humidimètres pour graines oléagineuses

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Foreword

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Check of the calibration of moisture meters — Part 2 : Moisture meters for oilseeds

0 Introduction

The use of calibrated moisture meters may, for stable samples and ideal measuring conditions, prove entirely satisfactory. However, the results obtained with the same moisture meter can be affected by many variables of cultivation, ripeness, humidity, temperature, harvesting, transport and level of impurities, particularly for oilseeds received with high moisture content.

Information concerning the maximum permitted errors is given in the annex, which does not form an integral part of this International Standard.

1 Scope and field of application

This part of ISO 7700 specifies a method of checking the calibration of moisture meters in service for measuring the moisture content of previously cleaned oilseeds, by checking several values or a range covering all the values for which the moisture meter is used.

The checking of several values allows the verification that the moisture meter, at the values for which it is normally used, does not give an error, while the checking of a range covering all the values provides the information necessary for calibration of the moisture meter with a view to carrying out appropriate adjustments.

Its application to colza (rape) seed, sunflower seed and soya beans has been verified.

2 References

ISO 665, *Oilseeds — Determination of moisture and volatile matter content.*

ISO 729, *Oilseeds — Determination of acidity of oils.*

3 Principle

For each species of oilseed to be tested using the moisture meter, preparation of several test samples, or a range of test samples, with different moisture contents, under specified conditions, determination of their moisture contents by a reference method and measurement with the moisture meter to be checked.

4 Reagent

Use only distilled water or water of equivalent purity.

Sodium hypochlorite (bleach), solution of approximately 5,7 % (m/m) active chlorine (18 chlorometric degrees).

5 Apparatus

Usual laboratory apparatus, and in particular

5.1 Bottles, with airtight seals, of capacity approximately 2 l, cleaned with a bactericide and fungicide, such as the bleach (clause 4), rinsed three times with distilled water and dried.

5.2 Apparatus required for the determination of moisture content (see ISO 665).

5.3 Sieves, with circular holes of diameter 1,50 and 2,80 mm and mesh of openings 3,55 mm, or a **mechanical separator**.

6 Procedure

6.1 Selection and cleaning of samples

Select a variety or varieties or a mixture of varieties of oilseeds from those which are the most prevalent in the region where the moisture meter is used.

It is strongly recommended that samples which in their natural state have the moisture contents necessary for the test are chosen.

Clean the samples using one of the following methods :

a) manual sieving using the sieves (5.3) as follows :

— sieve with circular holes of diameter 1,50 and 2,80 mm for colza (rape) seed;

— sieve of 3,55 mm mesh openings, removing large impurities by hand, for sunflower seed and soya beans;

b) using the mechanical separator (5.3).

The acid value, determined in accordance with ISO 729, shall be less than 2 (or 1 % expressed in terms of oleic acid).

6.2 Preparation of test samples

6.2.1 Procedure when checking several values

6.2.1.1 According to the number of values to be checked, prepare a greater or lesser number of test samples, each having a mass of approximately 900 g for colza (rape) seed and soya beans, and 600 g for sunflower seed, and various moisture contents between 6 and 20 % (m/m) for colza (rape) seed and between 6 and 25 % (m/m) for sunflower seed and soya beans.

Preferably choose moisture contents close to those which are most frequently observed in the region where the moisture meter is used.

6.2.1.2 To prepare the test samples, use the samples selected in 6.1 which, by preference, in their natural state have the moisture contents necessary for the test, or if necessary samples specially conditioned by the procedure specified in 6.2.3.

Place the samples in the bottles (5.1) and seal them.

If the calibration check on the moisture meter is not carried out within 24 h, keep the bottles at a temperature of approximately 5 °C, for example in a refrigerator.

6.2.2 Procedure when checking a range

6.2.2.1 Prepare a calibration range of 10 test samples each having a mass of approximately 1 kg and various moisture contents, at intervals as regular as possible, between 6 and 20 % (m/m) for colza (rape) seed, and between 6 and 25 % (m/m) for sunflower seed and soya beans.

6.2.2.2 To prepare this range of test samples, proceed as specified in 6.2.1.2.

6.2.3 Conditioning of samples

6.2.3.1 If conditioning is necessary, take a sample (see 6.1) having a moisture content at the time of harvesting equal to or less than the lowest moisture content selected for the calibration range, or, failing that, bring the required quantity of sample selected to this minimum moisture content by drying very gradually at a temperature not exceeding 30 °C, using ventilation if necessary.

6.2.3.2 For each desired moisture content, place in a bottle (5.1) a quantity of the sample such that the bottle is between half and two-thirds full.

6.2.3.3 Calculate the quantity of distilled water necessary to bring each sample to the selected moisture content, using the formula

$$m \times \frac{w_2 - w_1}{100 - w_2}$$

where

m is the mass, in grams, of the sample;

w_1 is the moisture content, expressed as a percentage by mass, of the sample;

w_2 is the moisture content, expressed as a percentage by mass, selected for the calibration.

Using a burette or graduated pipette, add in rapid drops to each sample, in one or two portions, as the case may be (see 6.2.3.4), the volume of distilled water thus calculated, while shaking the bottle. Seal the bottle.

6.2.3.4 In order to ensure that the water is evenly distributed, shake the bottles by inverting them by hand, under the following conditions :

— If the difference between the desired moisture content and the moisture content of the sample is less than 10 % (m/m) (absolute), add the quantity of the water calculated above in one portion, and shake energetically over a minimum of 4 days as described in table 1, taking great care to turn the bottle upright again after shaking.

— If the difference between the desired moisture content and the moisture content of the sample is more than 10 % (m/m) (absolute), add the quantity of the water calculated above in two equal portions at an interval of 24 h and shake energetically at regular intervals over a minimum of 5 days as described in table 2, taking great care to turn the bottle upright again after shaking.

In all cases, the bottles shall be kept at a temperature of approximately 5 °C, for example in a refrigerator.

NOTE — Instead of shaking by hand, it is possible to use an apparatus allowing either very slow, continuous shaking to be carried out over 5 days at 5 °C, or vigorous shaking in accordance with the requirements given in tables 1 and 2 for manual shaking.

6.3 Checking the moisture meter

6.3.1 Procedure when checking several values

6.3.1.1 If the test samples have not been conditioned, ensure that they have been kept under the same temperature conditions as the moisture meter before the test; if not, keep them under these conditions to allow them to reach thermal equilibrium with the moisture meter. If the test samples have been conditioned, remove the bottles from the refrigerator at least 16 h (usually overnight) before the test to allow them to reach thermal equilibrium with the moisture meter. In all cases, note the temperature of thermal equilibrium.

Reject the test samples if they emit an odour of fermentation or are mouldy (in the case of a range, it is necessary to start again).

6.3.1.2 On each previously mixed test sample, carry out the following operations :

a) Determine the moisture content by the reference method specified in ISO 665.

Take as the result the arithmetic mean of two determinations. It is imperative to comply with this condition.

Table 1 — Requirements for manual shaking when one portion of water has been added

Period		Approximate duration of shaking s
First day	As soon as water has been added	60
	First hour	15
	Second hour	15
	Third hour	15
	Between the third hour and the end of the first day	15
Second day		15
Third day		15
Fourth day and subsequent days		15

Table 2 — Requirements for manual shaking when two portions of water have been added

Period		Approximate duration of shaking s
First day	As soon as the first portion of water has been added	60
	First hour	15
	Second hour	15
	Third hour	15
	Between the third hour and the end of the first day	15
Second day	As soon as the second portion of water has been added	60
	First hour	15
	Second hour	15
	Third hour	15
	Between the third hour and the end of the second day	15
Third day		15
Fourth day		15
Fifth day and subsequent days		15

b) Using the moisture meter, carry out four successive measurements using four test portions taken from the test sample.

In the case of moisture meters designed to take readings on whole seeds, which in general require large test portions, transfer each test portion back into the bottle containing the test sample after each measurement, and mix by shaking the bottle before taking a new test portion.

In the case of moisture meters for taking readings on crushed seeds, which, in general, require small test portions (less than 50 g), carry out the crushing and measurement strictly according to the manufacturer's instructions. Discard all test portions after use.

c) After four measurements have been taken, again determine the moisture content by the reference method, proceeding as described in a).

6.3.2 Procedure when checking a range

On each test sample, carry out the same operations specified in 6.3.1 and repeat the operations specified in 6.3.1.2 at an interval of 24 h using the same test samples.

7 Expression of results

7.1 Procedure when checking several values

7.1.1 For each test sample, the following values are available :

— two results obtained by the reference method, x.

The difference between these two results shall not exceed 0,20 g of moisture per 100 g of sample. Otherwise, repeat the test;

— four measurements carried out with the moisture meter, y.

7.1.2 For each test sample, calculate the difference between the result of each measurement carried out with the moisture meter, y, and the mean of the two results obtained by the reference method, $\bar{x}$, i.e. $y - \bar{x}$.

The values $y - \bar{x}$ shall be less than the maximum permitted errors such as those specified in the annex.

7.2 Procedure when checking a range

7.2.1 Deal separately with the two series of measurements carried out at an interval of 24 h and compare them to ensure that there has been no development of the seed and/or variation in the response of the moisture meter over this 24 h period.

7.2.2 For each test sample and for each series of measurements, the following values are available :

— two results obtained by the reference method, x.

The difference between these two results shall not exceed 0,20 g of moisture per 100 g of sample. Otherwise, repeat the test;

— four measurements carried out with the moisture meter, y.

7.2.3 For each test sample and for each series of measurements, calculate the difference between the mean of the four measurements carried out with the moisture meter, $\bar{y}$, and the mean of the two results determined by the reference method, $\bar{x}$, i.e. $\bar{y} - \bar{x}$.

For each series of measurements, the values of $\bar{y} - \bar{x}$ shall be less than the maximum permitted errors, such as those specified in the annex. If a value of $\bar{y} - \bar{x}$ is greater than the maximum permitted error, repeat the measurements on the corresponding test sample.

8 Test report

The test report shall show the method used, the test temperature, the type of moisture meter used and its precision class, and the results obtained. In addition, it shall mention any operating details not specified in this part of ISO 7700, or regarded as optional, as well as any incidents likely to have influenced the results.

Annex

Maximum permitted errors

(This annex does not form an integral part of the standard.)

The maximum permitted errors for moisture meters in service, in accordance with the International Organization of Legal Metrology OIML International Recommendation No. 59 concerning moisture meters for cereals and oilseeds, are as follows.[1)]

A.1 Class I moisture meters (see also the figure)

— For oilseeds other than sunflower seeds :

0,7 (absolute) for a mean moisture content, $\bar{x}$, less than 10 % (m/m);

0,4 (absolute) plus 3 % (relative) for a mean moisture content, $\bar{x}$, greater than 10 % (m/m).

— For sunflower seeds :

0,8 (absolute) for a mean moisture content, $\bar{x}$, less than 10 % (m/m);

0,4 (absolute) plus 4 % (relative) for a mean moisture content, $\bar{x}$, greater than 10 % (m/m).

A.2 Class II moisture meters

— For oilseeds other than sunflower seeds :

0,8 (absolute) for a mean moisture content, $\bar{x}$, less than 10 % (m/m);

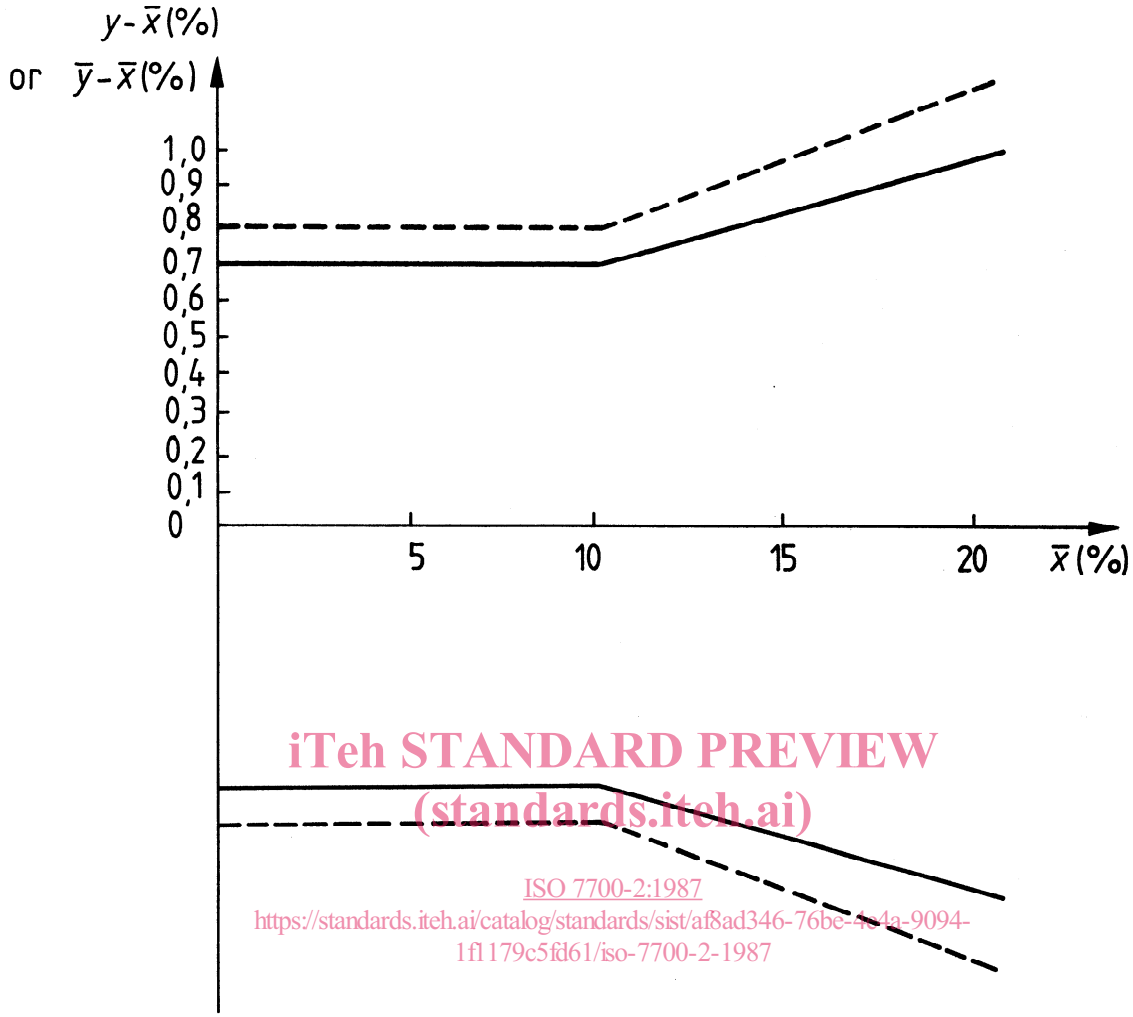
0,4 (absolute) plus 4 % (relative) for a mean moisture content, $\bar{x}$, greater than 10 % (m/m).

— For sunflower seeds :

0,9 (absolute) for a mean moisture content, $\bar{x}$, less than 10 % (m/m);

0,4 (absolute) plus 5 % (relative) for a mean moisture content, $\bar{x}$, greater than 10 % (m/m).

1) The precision classes of moisture meters are also defined in OIML International Recommendation No. 59.



The values of $y - \bar{x}$ (see 7.1) or $\bar{y} - \bar{x}$ (see 7.2) shall be situated within the limits defined by this graph.

Figure — Graph showing the maximum permitted errors for class I moisture meters

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