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**Cereals — Determination of bulk density,  
called mass per hectolitre —**

Part 2:

**Method of traceability for measuring  
instruments through reference  
to the international standard instrument**

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*Céréales — Détermination de la masse volumique, dite masse  
à l'hectolitre —*

*Partie 2: Méthode de raccordement des instruments de mesure  
à l'étalon international*

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 7971 (all parts) was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 338, *Cereal and cereal products*, in collaboration with Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This edition is the second to bear the number ISO 7971-2, but the technical content is new. ISO 7971-3:2009 cancels and replaces the first edition (ISO 7971-2:1995).

ISO 7971 consists of the following parts, under the general title *Cereals — Determination of bulk density, called mass per hectolitre*:

- *Part 1: Reference method*
- *Part 2: Method of traceability for measuring instruments through reference to the international standard instrument*
- *Part 3: Routine method*

# Cereals — Determination of bulk density, called mass per hectolitre —

## Part 2: Method of traceability for measuring instruments through reference to the international standard instrument

### 1 Scope

This part of ISO 7971 specifies a test method for ensuring the traceability of mass per hectolitre measuring instruments through reference to standard measurement instruments. The mass per hectolitre or bulk density is of commercial importance for grain cereals. Several types of instruments with varying performances exist for measuring it.

This part of ISO 7971 also specifies the performances required of national standards instruments, secondary standards instruments, and measuring instruments used in laboratories or in collection or storage silos.

### 2 Normative references

ISO 7971-2:2009

The following referenced documents are indispensable for the application 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 712, *Cereals and cereal products — Determination of moisture content — Reference method*

ISO 7971-1, *Cereals — Determination of bulk density, called “mass per hectolitre” — Part 1: Reference method*

### 3 Terms and definitions

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

#### 3.1

##### **bulk density**

##### **“mass per hectolitre”**

⟨cereals⟩ ratio of the mass of a cereal to the volume it occupies after being poured into a container under well-defined conditions

NOTE Bulk density is expressed in kilograms per hectolitre of grains as received.

[ISO 7971-1:2009]

#### 3.2

##### **traceability**

⟨cereals⟩ establishment of a relationship between the usual measuring instrument and the international standard instrument via a chain

**3.3 analysis certificate**  
document supplied by the international standard instrument holder body or national standard instrument holder body containing the individual values of measurements, the mass per hectolitre average value and its uncertainty for the test sample being analysed

**3.4 conformity certificate**  
document, issued by the body in charge of verifying the performances of the instrument, specifying the conformity decision for the requested use

**3.5 control chart**  
chart on which some statistical measure of a series of samples is plotted in a particular order to steer the process with respect to that measure and to control and reduce variation

[ISO 3534-2:2006<sup>[1]</sup>, 2.3.1]

NOTE The analysis values are plotted on the ordinate against the date (day or hour) of carrying out the measurement on the abscissa.

## 4 Requirements

### 4.1 Certified international or national standard instrument

A standard measurement instrument for mass per hectolitre has a 20 l hopper and is certified by a national or international authority. An informative list of such instruments is given in Annex B.

### 4.2 National standard instrument

Each country defines, on the basis of the specifications given in 6.2, the instrument designated as a national standard instrument within the respective country.

This national standard instrument should be an instrument designated by its name (model, manufacturer, serial number). Any other instrument of the same model as that adopted as the national standard instrument shall not be referred to as a "national standard instrument".

Each country defines the public or private organization responsible for the safe keeping, use, and maintenance of this national standard instrument. This same organization ensures its traceability through reference to a certified international or national standard instrument (4.1), according to the provisions specified in 7.1, every 10 years.

Each country shall ensure that no natural or legal person is refused access to the results of this national standard instrument so that any manufacturer, holder, repairer, controller or user can ensure the traceability of his own instruments through reference to the national standard instrument.

### 4.3 Secondary or internal standard instrument

For the purposes of inspection of instruments in operation, a body may possess an appliance specific to this activity. This instrument shall have the required qualities, established using the national standard instrument. It shall then constitute the reference which ensures the traceability of the instruments in operation.

Verify and adjust these secondary standard instruments every 2 years in accordance with 7.2.

### 4.4 Routine measuring instrument

This term designates any mass per hectolitre measuring instrument used in commercial transactions.

The traceability of instruments in operation and of new instruments (prior to commissioning) shall be ensured through reference to a secondary standard instrument (4.3) according to the provisions specified in 7.3.

The frequency of this traceability operation is 1 year.

## 5 Principle

This part of ISO 7971 specifies a procedure for ensuring the traceability of mass per hectolitre measuring instruments through reference to standard instruments.

The traceability principle is illustrated by Figure 1.

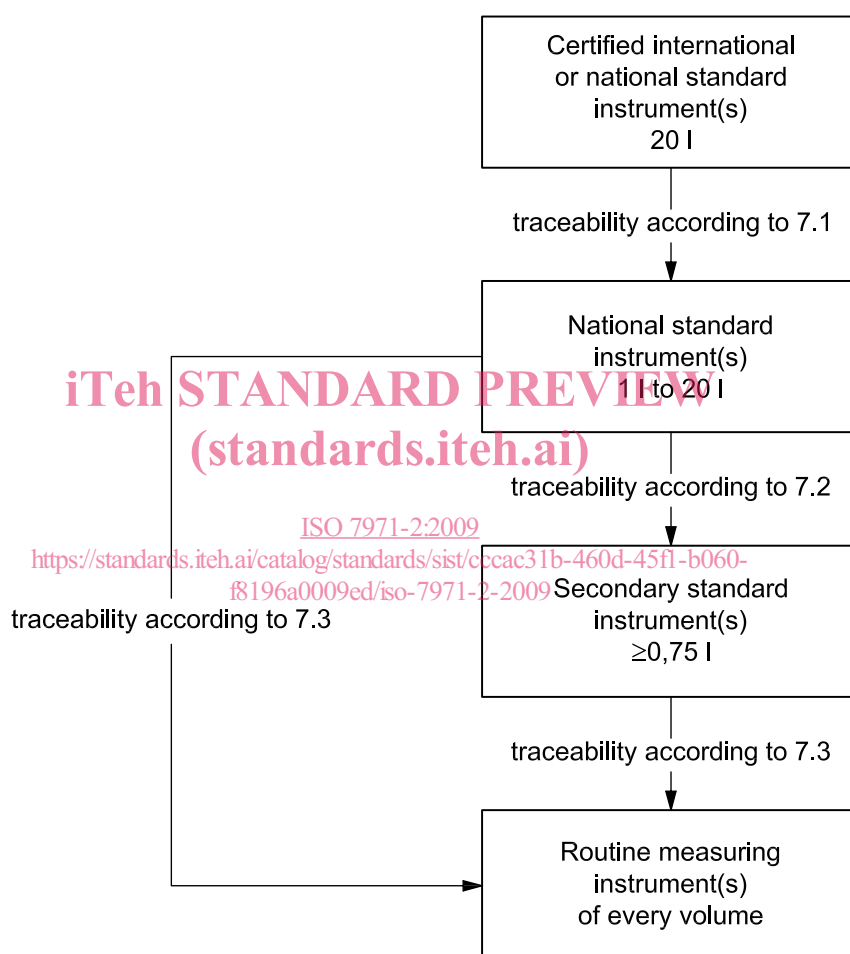


Figure 1 — Principle of traceability of instruments through reference to standard instruments

## 6 Apparatus

**6.1 International standard instrument for mass per hectolitre** as specified in ISO 7971-1.

**6.2 National standard instrument for mass per hectolitre.** Manual or automatic, mechanical, electric or electronic mass per hectolitre measuring instrument, of which the capacity of the measuring container is at least 1 l, accepting a bias correction or a bias and slope correction.

This instrument is used according to the requirements given in 4.2 and traceability is ensured through reference to the international standard instrument according to the procedure specified in 7.1.

The correction made to the instrument ensures a slope and an intercept which are not significantly different from 1 and 0 at the 5 % threshold [see Equations (A.8), (A.9), and (A.10), A.7.3 and A.8.2].

If the national standard instrument is a 20 l apparatus, for any correction refer to ISO 7971-1.

**6.3 Secondary standard instrument for the mass per hectolitre.** Manual or automatic, mechanical, electric or electronic mass per hectolitre measuring instrument, of which the capacity of the measuring container is at least 750 ml, accepting a bias and/or slope correction.

This instrument is used according to the requirements given in 4.3 and traceability is ensured through reference to the national standard instrument according to the procedure specified in 7.2.

The correction made to the instrument ensures a slope and an intercept which are not significantly different from 1 and 0 at the 5 % threshold [see Equations (A.8), (A.9), and (A.10), A.7.3 and A.8.2].

**6.4 Mass per hectolitre measuring instrument.** Manual or automatic, mechanical, electric or electronic, adjustable or fixed mass per hectolitre measuring instrument, associated if necessary with an equivalence or correction table and used according to the requirements given in 4.4.

The verification of these instruments specified in 7.3 concerns both new instruments and instruments in operation.

The amplitude between the highest and the lowest value,  $\Delta\rho_i$ , measured on each control sample (see 7.3.4), shall not exceed 0,3 kg/hl.

For each control sample, the difference between the value given by the national standard instrument or the secondary standard instrument and the mean of the measurements carried out on the instrument,  $\Delta\rho_{i,2-m}$ , (see 7.3.4), shall not exceed 0,3 kg/hl for mass per hectolitre under 70 kg/hl and 0,4 kg/hl for mass per hectolitre greater than or equal to 70 kg/hl.

**6.5 Divider.** Manual or mechanical cone-shaped divider that can representatively reduce a 25 l volume of grains to a volume suited to the instruments to undergo traceability.

## 7 Procedure

### 7.1 Traceability of national standard instruments through reference to the certified international or national standard instrument

#### 7.1.1 General description

The traceability of national standard instruments is conducted using a series of test samples referenced with their mass per hectolitre and accompanied by their analysis certificate (3.3). The use of wheat and barley test samples is recommended because of the regular shape of the grain and the good availability of these species of grains. Other seeds with regular shape of grain, e.g. triticale or rye, can be used if performance requirements are fulfilled. The use of grains with inhomogeneous shape, e.g. oats, is not advisable.

#### 7.1.2 Preparation of the test samples

Select three test samples of common wheat and/or durum wheat and three test samples of barley having the following characteristics:

- a) for wheat (common or durum), one test sample with a mass per hectolitre in each of the following ranges: 72 kg/hl to 76 kg/hl, 75 kg/hl to 79 kg/hl, and 78 kg/hl to 82 kg/hl;
- b) for barley, one test sample with a mass per hectolitre in each of the following ranges: 60 kg/hl to 64 kg/hl, 65 kg/hl to 69 kg/hl, and 70 kg/hl to 74 kg/hl;



- c) a minimum difference between two successive test samples of at least 2 kg/hl;
- d) an approximate mass of 30 kg, packed in correctly identified, rigid, airtight containers;
- e) a moisture content (mass fraction) between 9 % and 14 % (as determined by ISO 712);
- f) freedom from abnormal odours and from live insects, not containing any heterogeneous impurities of a size over 10 mm and guaranteeing a residual impurity content below 2 % mass fraction.

Dispatch the six test samples prepared in this manner to an owner of a certified standard measurement instrument (see list in Annex B) for determination of their mass per hectolitre and of the uncertainty associated with this quantity.

When these test samples come back, if they have to be stored prior to use, place them in ambient conditions which do not affect their properties: temperature below 22 °C and relative humidity below 60 %. Do not store these test samples at negative temperatures.

#### EXAMPLES

A set of test samples with masses per hectolitre of 73 kg/hl, 76 kg/hl, and 80 kg/hl for wheat and 60 kg/hl, 66 kg/hl, and 71 kg/hl for barley is suitable.

A set of test samples with masses per hectolitre of 73 kg/hl, 74 kg/hl, and 80 kg/hl for wheat and 60 kg/hl, 65 kg/hl, and 66 kg/hl for barley is not suitable because there is less than 2 kg/hl between two of the three test samples of each type.

A set of test samples with masses per hectolitre of 70 kg/hl, 73 kg/hl, and 76 kg/hl for wheat and 66 kg/hl, 68 kg/hl, and 70 kg/hl for barley is not suitable because the total range is not covered.

#### 7.1.3 Use of the referenced test samples

If the mass per hectolitre referenced test samples have been stored in a refrigerating chamber, it is necessary to wait until their temperature has reached equilibrium with that of the premises where the tests are being carried out before opening the containers.

If the volume of the referenced test samples is such that they cannot be fully used in the national standard instrument, constitute test portions of appropriate mass, which are representative of the initial test sample, using a divider.

Select three of these test portions and conduct a mass per hectolitre measurement on each of them using the national standard instrument or, if the test samples can be used in their entirety, measure each of them three times. Three individual values marked  $\rho_{i1}$  to  $\rho_{i3}$ , where  $i$  represents the serial number of the referenced sample, are thus obtained.

When equipment using a cutting edge is employed, the test sample shall not be analysed more than 50 times. After that the test sample has to be replaced.

#### 7.1.4 Interpretation of the results

From the unit results obtained on the control test samples, calculate the performance parameters according to the relevant equations in Annex A.

Check that the specifications defined for the national standard instrument in 6.2 are fulfilled. If so, draw up the national standard instrument conformity certificate.

If not, adjust the national standard instrument according to 7.1.5. In the event of it being impossible to obtain the specifications requested in 6.2, the national standard instrument shall be repaired or declassified then replaced by another instrument in order to re-obtain the required performances.

The national standard instrument holder shall keep all of the data (raw and calculated) relative to this traceability operation throughout the instrument's lifetime.

#### 7.1.5 Adjustment of the national standard instrument

If the slope  $a$  and the intercept  $b$  calculated in 7.1.4 are outside the limits specified in 6.2, introduce into the instrument, according to the manufacturer's recommendations, the necessary correction factors required to theoretically bring the slope to 1 and the intercept to 0.

Then resume the operations specified in 7.1.3 and 7.1.4 in order to verify the validity of this correction.

### 7.2 Traceability of the secondary standard instruments through reference to the national standard instrument

#### 7.2.1 General description

The traceability of the secondary standards is conducted using a series of test samples referenced with the mass per hectolitre obtained with the national standard instrument (6.2). The use of wheat and barley test samples is recommended because of the regular shape of the grain and the availability of these species of grains. Other seeds with regular shape of grain, e.g. triticale or rye, can be used if performance requirements are fulfilled. The use of grains with inhomogeneous shape, e.g. oats, is not advisable.

#### 7.2.2 Preparation of the test samples

Select three test samples of common wheat and/or durum wheat and three test samples of barley having the following characteristics:

- a) for wheat (common or durum), one test sample with a mass per hectolitre in each of the following ranges: 72 kg/hl to 76 kg/hl, 75 kg/hl to 79 kg/hl, and 78 kg/hl to 82 kg/hl;
- b) for barley, one test sample with a mass per hectolitre in each of the following ranges: 60 kg/hl to 64 kg/hl, 65 kg/hl to 69 kg/hl, and 70 kg/hl to 74 kg/hl;
- c) a minimum difference between two successive test samples of at least 2 kg/hl;
- d) sufficient quantity for measurement on the national standard instrument (1,5 l to 25 l), packed in correctly identified, rigid, airtight containers;
- e) a moisture content (mass fraction) between 9 % and 14 % (as determined by ISO 712);
- f) freedom from abnormal odours and from live insects, not containing any heterogeneous impurities of a size over 10 mm and guaranteeing a residual impurity content below 2 % mass fraction.

Dispatch the six thus prepared test samples to the national standard instrument holder body for determination of their mass per hectolitre and of the uncertainty associated with this quantity.

When these test samples come back, if they have to be stored prior to use, place them in ambient conditions which do not affect their properties: temperature below 22 °C and relative humidity below 60 %. Do not store these test samples at negative temperatures.

NOTE See 7.1.2 for examples of suitable sets of test samples.

#### 7.2.3 Use of the referenced test samples

If the mass per hectolitre referenced test samples have been stored in a refrigerating chamber, it is necessary to wait until their temperature has reached equilibrium with that of the premises where the tests are being carried out before opening the containers.

If the volume of the referenced test samples is such that they cannot be fully used in the secondary standard instrument, constitute test portions of appropriate mass, which are representative of the initial test sample, using a divider.

Select three of these test portions and conduct a mass per hectolitre measurement on each of them using the secondary standard instrument or, if the test samples can be entirely used, measure each of them three times. Three individual values marked  $\rho_{i1}$  to  $\rho_{i3}$ , where  $i$  represents the serial number of the referenced sample, are thus obtained.

When equipment using a cutting edge is employed, the test sample shall not be analysed more than 50 times. After that the test sample shall be replaced.

#### 7.2.4 Interpretation of the results

From the unit results obtained on the control test samples, calculate the performance parameters according to the relevant equations in Annex A.

Check that the specifications defined for the secondary standard instrument (6.3) are fulfilled. If so, draw up the secondary standard conformity certificate.

If not, adjust the secondary standard instrument according to 7.2.5. In the event of it being impossible to obtain the specifications requested in 6.3, the secondary standard shall be repaired or declassified then replaced by another instrument in order to re-obtain the required performances.

The secondary standard instrument holder shall keep all of the data (raw and calculated) relative to this traceability operation throughout the instrument's lifetime.

#### 7.2.5 Adjustment of the secondary standard instrument

If the slope  $a$  and the intercept  $b$  calculated in 7.2.4 are outside the limits specified in 6.3, introduce into the instrument, according to the manufacturer's recommendations, the necessary correction factors required to theoretically bring the slope to 1 and the intercept to 0.

Then resume the operations specified in 7.2.3 and 7.2.4 in order to verify the validity of this correction.

### 7.3 Verification of instruments in operation

#### 7.3.1 General description

The traceability of instruments in operation is carried out using a series of four control test samples of which the mass per hectolitre has been previously measured using a secondary standard (6.3) or, failing this, using a national standard instrument (6.2). The use of wheat and barley test samples is recommended because of the regular shape of the grain and the availability of these species of grains. Other seeds with regular shape of grain, e.g. triticale or rye, can be used if performance requirements are fulfilled. The use of grains with inhomogeneous shape, e.g. oats, is not advisable.

#### 7.3.2 Preparation of the control test samples

Select two test samples of common wheat and/or durum wheat and two test samples of barley having the following characteristics:

- a) for wheat (common and durum), one test sample with a mass per hectolitre in each of the following ranges: 72 kg/hl to 78 kg/hl and 77 kg/hl to 83 kg/hl;
- b) for barley, one test sample with a respective mass per hectolitre in each of the following ranges: 61 kg/hl to 67 kg/hl and 67 kg/hl to 73 kg/hl;
- c) a minimum difference between two successive test samples of at least 5 kg/hl;