
**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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Contents

	Page
Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Requirements	2
4.1 Certified international or national standard instrument.....	2
4.2 National standard instrument.....	2
4.3 Secondary or internal standard instrument.....	2
4.4 Routine measuring instrument.....	3
5 Principle	3
6 Apparatus	3
7 Procedure	4
7.1 Traceability of national standard instruments through reference to the certified international or national standard instrument.....	4
7.1.1 General description.....	4
7.1.2 Preparation of the test samples.....	5
7.1.3 Use of the referenced test samples.....	5
7.1.4 Interpretation of the results.....	5
7.1.5 Adjustment of the national standard instrument.....	6
7.2 Traceability of the secondary standard instruments through reference to the national standard instrument.....	6
7.2.1 General description.....	6
7.2.2 Preparation of the test samples.....	6
7.2.3 Use of the referenced test samples.....	7
7.2.4 Interpretation of the results.....	7
7.2.5 Adjustment of the secondary standard instrument.....	7
7.3 Verification of instruments in operation.....	7
7.3.1 General description.....	7
7.3.2 Preparation of the control test samples.....	8
7.3.3 Use of the referenced test samples.....	8
7.3.4 Interpretation of the results.....	8
7.3.5 Adjustment of the instrument.....	9
8 Monitoring of instrument performance	9
9 Test reports	10
9.1 Analysis certificate.....	10
9.2 Conformity certificate.....	10
10 Identification of traced measuring instruments	10
Annex A (normative) Method for calculation of performance parameters of standard instruments and of measurement instruments	11
Annex B (informative) Example — Owners of certified standard measurement instruments	22
Bibliography	23

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.

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 documents 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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 7971-2:2009), which has been technically revised. The main changes compared with the previous edition are as follows:

- changes have been made to the test samples used for the traceability of measuring instruments through reference to standard measurement instruments (review of the number, range, distribution and characteristics of the samples);
- further details have been given on statistical tests;
- clarification has been given on the decision rules by adding a decision tree for national standard instruments and secondary or internal standard instruments and by introducing a bias control.

A list of all parts in the ISO 7971 series can be found on the ISO website.

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.

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 document specifies a test method for ensuring the traceability of bulk density, called “mass per hectolitre”, measuring instruments through reference to standard measurement instruments. The mass per hectolitre is of commercial importance for grain cereals. Several types of instruments with varying performances exist for measuring it.

This document 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

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 712, *Cereals and cereal products — Determination of moisture content — Reference method*

ISO 7971-1:2009, *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.

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

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

3.1

mass per hectolitre

bulk density

test weight

<cereals> ratio of the mass of a cereal to the volume it occupies after being poured into a container under defined manufacturer's conditions

Note 1 to entry: Mass per hectolitre is expressed in kilograms per hectolitre of grains as received.

[SOURCE: ISO 7971-1:2009, 2.1, modified — In the definition, “defined manufacturer's conditions” has replaced “well-defined conditions”, and the Note 2 to entry has been deleted.]

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.

Note 1 to entry: The analysis values are plotted on the ordinate against the date (day or hour) of carrying out the measurement on the abscissa.

[SOURCE: ISO 3534-2:2006, 2.3.1, modified — The two notes to entry have been replaced by a new Note 1 to entry.]

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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 example list of suppliers 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 ten 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 two 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) or a national standard instrument (4.2) according to the provisions specified in 7.3.

The frequency of this traceability operation is every one year for hand operated instruments and every two years for automatic devices.

5 Principle

This document 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.

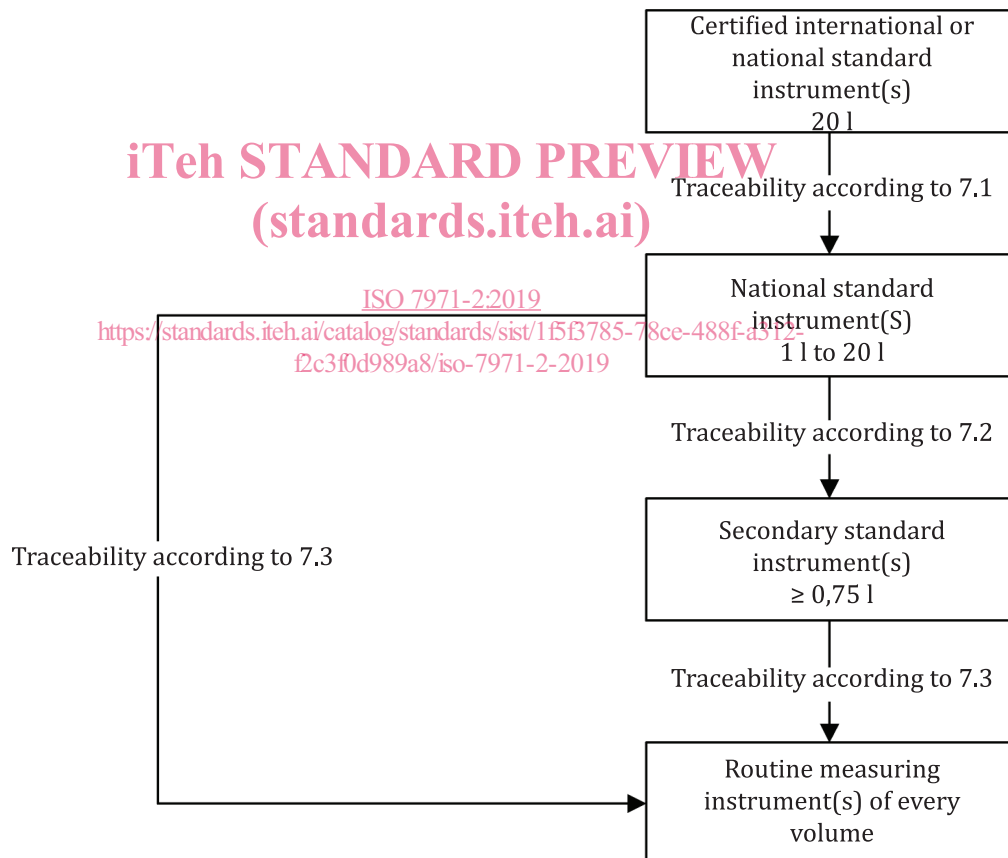


Figure 1 — Principle of traceability of instruments through reference to standard instruments

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 that is not significantly different from 1 and an intercept of bias (difference between the mean of the reference values and the mean of the measurements produced by the instrument) that is not significantly different from 0 at the 5 % threshold [see [Formulae \(A.8\), \(A.9\) and \(A.10\), A.7.1.2 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 that is not significantly different from 1 and an intercept of bias (difference between the mean of the reference values and the mean of the measurements produced by the instrument) that is not significantly different from 0 at the 5 % threshold [see [Formulae \(A.8\), \(A.9\) and \(A.10\), A.7.1.2 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, $\Delta\rho_i$, between the highest and the lowest value, 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, i.e. $\Delta\rho_{i,2-m}$ (see 7.3.4), shall not exceed 0,4 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. 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 seven test samples of common wheat and/or durum wheat and seven test samples of barley having the following characteristics:

- a) the mass per hectolitre of one of the barley test samples shall be less than 64 kg/hl and the mass per hectolitre of one of the (common and/or durum) wheat test samples shall be greater than or equal to 82 kg/hl to ensure that a wide range is covered;
- b) with a difference in mass per hectolitre between two successive test samples that is as regular as possible in the range 1 kg/hl to 3 kg/hl;
- c) an approximate mass of 30 kg, packed in correctly identified, rigid, airtight containers;
- d) a moisture content (mass fraction) between 9 % and 14 % (as determined by ISO 712 or using a rapid method whose measurement does not differ from the reference value by $\pm 0,4$ g water per 100 g of sample);
- e) 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 of the following: broken grain, other cereals, sprouted grain, foreign grain and inert material (such as soil, sand, plastic and glass particles, husk, straw).

Dispatch the fourteen test samples prepared in this manner to an owner of a certified standard measurement instrument for determination of their mass per hectolitre and of the uncertainty associated with this quantity. Check that this uncertainty is no more than 0,30 kg/hl.

When these test samples come back, if they have to be stored prior to use, place them in controlled conditions with temperature capped at 22 °C so as to not affect their properties. Do not store these test samples at negative temperatures.

EXAMPLE

ISO 7971-2:2019
<https://standards.iteh.ai/catalog/standards/sist/1f5f3785-78ce-488f-a312-fc37d989a8/iso-7971-2-2019>

A set of test samples with masses per hectolitre of 71 kg/hl, 73 kg/hl, 75 kg/hl, 78 kg/hl, 81 kg/hl, 82 kg/hl and 84 kg/hl for wheat and 63 kg/hl, 65 kg/hl, 68 kg/hl, 70 kg/hl, 73 kg/hl, 75 kg/hl and 77 kg/hl for barley is suitable.

The wheat and barley distributions overlap, the differences between two successive test samples are in the range 1 kg/hl to 3 kg/hl, and the full range is 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 ρ_{i1} to ρ_{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.

7.1.4 Interpretation of the results

From the unit results obtained on the control test samples, calculate the performance parameters according to the relevant formulae 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 or bias 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.

ISO 7971-2:2019

[https://standards.iteh.ai/catalog/standards/sist/1f5f3785-78ce-488f-a312-](https://standards.iteh.ai/catalog/standards/sist/1f5f3785-78ce-488f-a312-f2c3f0d989a8/iso-7971-2-2019)

7.2.2 Preparation of the test samples

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

- a) the mass per hectolitre of one of the barley test samples shall be less than 64 kg/hl and the mass per hectolitre of one of the (common and/or durum) wheat test samples shall be greater than or equal to 82 kg/hl to ensure that a wide range is covered;
- b) with a difference between two successive test samples that is as regular as possible in the range 1 kg/hl to 3 kg/hl;
- c) sufficient quantity for measurement on the national standard instrument (1,5 l to 25 l), packed in correctly identified, rigid, airtight containers;
- d) a moisture content (mass fraction) between 9 % and 14 % (as determined by ISO 712 or using a rapid method whose measurement does not differ from the reference value by $\pm 0,4$ g water per 100 g of sample);
- e) 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 of the following: broken grain, other cereals, sprouted grain, foreign grain and inert material (such as soil, sand, plastic and glass particles, husk, straw).

Dispatch the fourteen 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. Check that this uncertainty is no more than 0,30 kg/hl.

When these test samples come back, if they have to be stored prior to use, place them in controlled conditions with temperature capped at 22 °C so as to not affect their properties. Do not store these test samples at negative temperatures.

NOTE See [7.1.2](#) for an example 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 ρ_{i1} to ρ_{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 formulae 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 instrument 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 or bias 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.