INTERNATIONAL STANDARD

ISO 7971-2

Third edition
2019-02

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

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

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

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

Reference number
ISO 7971-2:2019(E)

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Published in Switzerland

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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.

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.]

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.

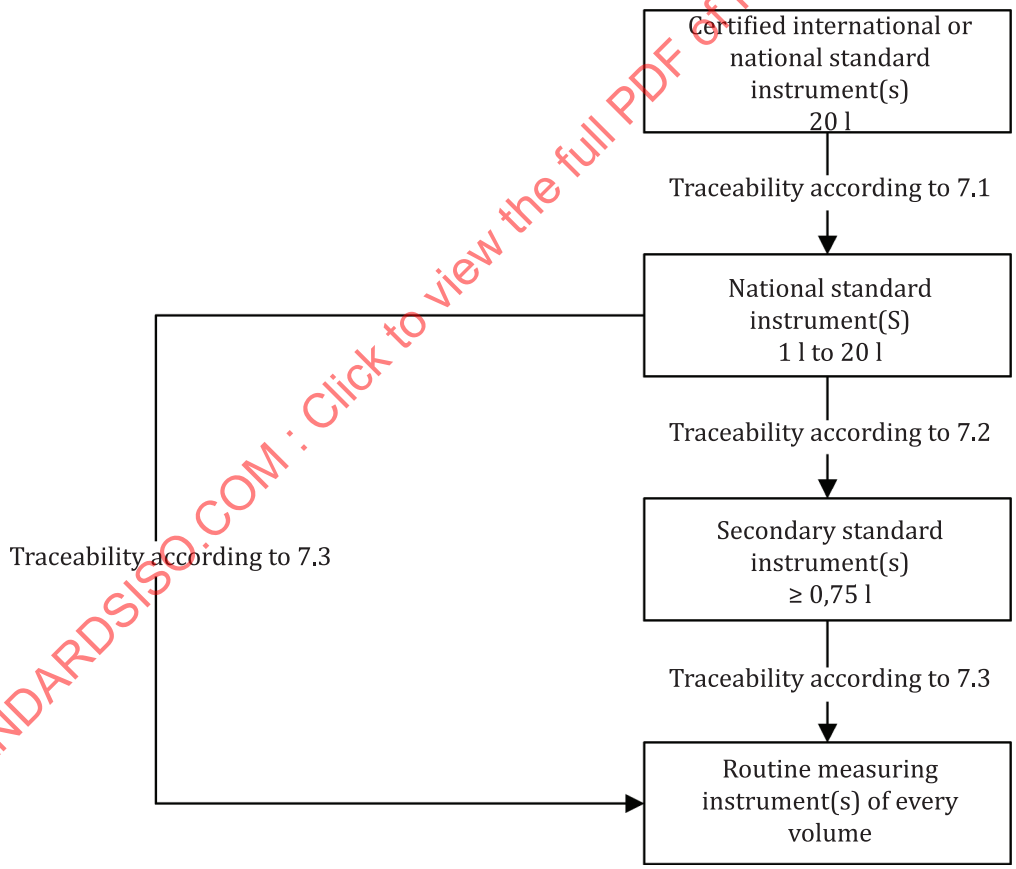

Figure 1 — Principle of traceability of instruments through reference to standard instruments

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 ±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

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 $\rho_{i3}$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.

7.1.4 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.

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 ±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.

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 (common and durum) wheat, one test sample with a mass per hectolitre in the range 72 kg/hl to 78 kg/hl and one test sample with a mass per hectolitre greater than 78 kg/hl;

b) for barley, one test sample with a mass per hectolitre of less than 67 kg/hl and one test sample with a mass per hectolitre in the range 67 kg/hl to 73 kg/hl;

c) a minimum difference between two successive test samples of at least 4 kg/hl;

d) sufficient quantity for measurement on the secondary standard, packed in correctly identified, rigid, airtight containers;

e) 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 ±0,4 g water per 100 g of sample);

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 of the following: broken grain, other cereals, sprouted grain, foreign grain and inert material (such as soil, sand, plastic and glass particles, husk, straw).

Measure or have measured the mass per hectolitre of these four test samples on a secondary standard instrument traced according to the provisions specified in 7.2.

Conserve these test samples 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.

Each test sample may be used a maximum of 50 times. After this number of uses, each test sample shall be re-referenced using the secondary standard instrument (or the national standard instrument). 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.

7.3.3 Use of the referenced test samples

If the referenced test samples are stored in cold conditions, 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 instrument to be controlled, divide each test sample with the appropriate means in order to obtain test portions which are representative of each overall test sample having a size suited to the instrument to be controlled and select one of these test portions. Otherwise, use each test sample in its entirety.

For each test sample or test portion, carry out two measurements using the instrument to be checked. If provided for by the instrument's method of use, calculate the final result of the measurement using a formula or an equivalence table. One thus has, for each of the four referenced test samples, two individual values designated ρ_{i1} to ρ_{i2}, where i represents the serial number of each control test sample.

7.3.4 Interpretation of the results

For each control test sample, calculate the quantities listed.

The amplitude (magnitude of the difference), $\Delta\rho_i$, between the highest and the lowest measurement, is given by

$$\Delta\rho_i = \left|\rho_{i1} - \rho_{i2}\right|$$

The mean of the two measurements, $\bar{\rho}_i$, is given by

$$\bar{\rho}_i = \frac{\rho_{i1} + \rho_{i2}}{2}$$

The difference between the referenced value obtained on the secondary (or national) standard instrument, $\rho_{i,\mathrm{m}}$, and on the measuring instrument, $\bar{\rho}_i$, to undergo traceability, $\Delta\rho_{i,2-\mathrm{m}}$ is given by

$$\Delta\rho_{i,2-\mathrm{m}} = \left|\rho_{i,\mathrm{m}} - \bar{\rho}_i\right|$$

If the specifications given in 6.4 are obtained, run a control-check of the freedom from bias by comparison of means (for bias) as stated in A.2. If equality of means is accepted, produce the conformity certificate (9.2) and mark the instrument as specified in Clause 10. Otherwise, adjust by following 7.3.5.

7.3.5 Adjustment of the instrument

If the instrument is an adjustable model, adjust its bias according to the manufacturer's recommendations. Resume the operations specified in 7.3.3 and 7.3.4 in order to verify the validity of this adjustment.

If specifications given in 6.4 are not obtained and the instrument cannot be adjusted, it is unfit for this purpose and shall not be used.

8 Monitoring of instrument performance

To protect oneself against a possible drift between two traceability operations of national standard instruments and secondary standard instruments, a self-control system should be set up by using a control chart. The control chart measures whether the performances remain within the defined limits at a given point.

Control and surveillance limits are plotted on the graph. These fixed limits are usually estimated at 3 s_{ILR}, where s_{ILR} is an intralaboratory reproducibility standard deviation, corresponding to a risk, α, of 0,2 %, for the control limits and 2 s_{ILR}, corresponding to a risk, α, of 5 %, for the surveillance limits. They are located on either side of a *central line* corresponding to the so-called reference value. The intralaboratory reproducibility standard deviation is calculated on 30 measurements (6 measurements per day for 5 days) on the same test sample.

Each time those standard instruments are used, a test sample referenced with its mass per hectolitre is analysed. The value obtained is plotted on the control chart and decision rules are applied in order to decide whether the system is to be corrected.

The following are generally selected:

a) any value between the surveillance limits and the control limits indicates correct performance;

b) any distribution which makes one suspect a non-random distribution indicates that close surveillance of the instrument is required and correction necessary if a systematic bias persists;

c) three consecutive points all located above or below the surveillance limit indicates close surveillance of the instrument is required in order to launch, if necessary, a new traceability operation;

d) one point outside the control limit indicates the need for the immediate performance of a new traceability operation in order to carry out the adequate setting.

9 Test reports

9.1 Analysis certificate

The test report establishing the mass per hectolitre of a referenced test sample shall include at least the following information:

a) all information required for the complete identification of the test sample;

b) the method used together with reference to this document, i.e. ISO 7971-2;

c) the final result obtained;

d) the uncertainty associated with this result;

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

9.2 Conformity certificate

The certificate establishing the traceability of a standard or the conformity of an instrument in operation shall include at least the following information:

a) all information required for the complete identification of the instrument (manufacturer, model, serial number, etc.);

b) the date of the tests and the identification of the operator who carried them out;

c) the method and details used together with reference to this document, i.e. ISO 7971-2;

d) the mean result of the measurements of each test sample, the corresponding reference value and the associated uncertainty;

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

10 Identification of traced measuring instruments

Measuring instruments used on a routine basis (6.4) for commercial transactions shall, once they have successfully undergone the traceability operations specified in 7.3, receive an identification mark specifying:

a) instrument traced through reference to a certified standard measurement instrument for mass per hectolitre;

b) a reference to this document, i.e. ISO 7971-2;

c) the date of the next traceability operation;

d) the name of the body (public or private) which carried out the traceability operation.

Annex A
(normative)

Method for calculation of performance parameters of standard instruments and of measurement instruments

A.1 Principle

The traceability of a measurement instrument to a standard instrument of superior level is made by a statistical validation of results obtained on this instrument with reference to the results obtained on the standard instrument.

This work consists of validation of an alternate method (the standard instrument to be traced) versus a reference method (the standard instrument of superior level). This annex gives the statistical tools to evaluate different criteria necessary to validate an alternative method. It specifies a check of freedom from bias by comparison of means and a specificity control by testing the slope and the intercept of the straight regression line between the two methods.

The evaluation of capacity of instruments to be traced is done by the same procedure in the case of traceability of a national standard instrument through a certified standard measurement instrument as in the case of traceability of a secondary standard through the national instrument.

A.2 Control of the freedom from bias by comparison of means

To make such calculations easier, the use of a spreadsheet with statistical treatment tools is strongly recommended. For example, it is possible to use the *t*-test on paired two sample means available in the data analysis tool of Microsoft® Excel©[1]. Such an example is given in A.8.

For each referenced test sample, calculate the mean, $\bar{\rho}_i$, of the n measurements carried out on the instrument to undergo traceability according to Formula (A.1):

$$\bar{\rho}_i = \frac{\sum_{j=1}^{n} \rho_{ij}}{n} \tag{A.1}$$

where n is the number of measurements for each test sample.

For each referenced test sample, calculate the difference, $\Delta\rho_{i,\mathrm{m-ref}}$, between $\rho_{i,\mathrm{m}}$ and the reference value $\rho_{i,\mathrm{ref}}$, then calculate the mean, $\overline{\Delta\rho}$, of these m differences as well as their standard deviation, $s_{\Delta\rho}$.

$$\Delta\rho_{i,\mathrm{m-ref}} = \rho_{i,\mathrm{m}} - \rho_{i,\mathrm{ref}} \tag{A.2}$$

$$\overline{\Delta\rho} = \frac{\sum_{i=1}^{m} \Delta\rho_{i,\mathrm{m-ref}}}{m} \tag{A.3}$$

1) Microsoft® Excel© is an example of appropriate software available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

$$s_{\Delta\rho} = \sqrt{\frac{\sum_{i=1}^{m} (\overline{\Delta\rho} - \Delta\rho_{i,\mathrm{m-ref}})^2}{m-1}} \tag{A.4}$$

where m is the number of referenced test samples used for the traceability.

Calculate the ratio t_{obs} according to Formula (A.5):

$$t_{\mathrm{obs}} = \frac{\left|\overline{\Delta\rho}\right|}{s_{\Delta\rho}} \times \sqrt{m} \tag{A.5}$$

Compare the t_{obs} value with a critical value, t_{val}, corresponding to that of a 5 % risk student variable with five degrees of freedom.

If t_{obs} is lower than t_{val}, the means equality presumption is accepted. The instrument is not biased.

A.3 Calculation of coordinates of the straight regression line and signification tests

To make such calculations easier, the use of a spreadsheet with statistical treatment tools is strongly recommended. For example, it is possible to use the regression tool available in the data analysis tool of Microsoft® Excel©[1]). Such an example is given in A.8.

Calculate the coordinates of the straight regression line of slope, a, and intercept, b, between the means $\rho_{i,\mathrm{m}}$ found by the standard instrument to be traced on each referenced test sample with the values given by the standard instrument of superior level, $\rho_{i,\mathrm{ref}}$, according to Formulae (A.6) and (A.7):

$$a = \frac{m\sum_{i=1}^{m} \rho_{i,\mathrm{ref}}\rho_{i,\mathrm{m}} - \left(\sum_{i=1}^{m} \rho_{i,\mathrm{ref}}\right)\left(\sum_{i=1}^{m} \rho_{i,\mathrm{m}}\right)}{m\sum_{i=1}^{m} \rho_{i,\mathrm{m}}^2 - \left(\sum_{i=1}^{m} \rho_{i,\mathrm{m}}\right)^2} \tag{A.6}$$

$$b = \bar{\rho}_{i,\mathrm{ref}} - a\bar{\rho}_{i,\mathrm{m}} \tag{A.7}$$

where

m is the number of referenced test samples used for the traceability (normally m = 14);

$\bar{\rho}_{i,\mathrm{m}}$ are the means of masses per hectolitre based on the instrument to be traced for the m control test samples;

$\bar{\rho}_{i,\mathrm{ref}}$ are the means of masses per hectolitre provided by the standard instrument of superior level for the same m control test samples.

A.4 Test for slope

Calculate the ratio t_{obs} according to Formula (A.8):

$$t_{obs} = \frac{|(1-a)|\sqrt{\sum_{i=1}^{m}(\rho_{i,m} - \bar{\rho}_{i,m})^2}}{s_a} \quad \text{(A.8)}$$

where the slope standard deviation, s_a, is given by Formula (A.9):

$$s_a = \sqrt{\frac{\sum_{i=1}^{m}(\rho_{i,ref} - \bar{\rho}_{i,ref})^2 - a^2\sum_{i=1}^{m}(\rho_{i,m} - \bar{\rho}_{i,m})^2}{m-2}} \quad \text{(A.9)}$$

Compare the t_{obs} value with a critical value t_{val} corresponding to that of a 5 % risk student variable with m – two degrees of freedom.

If t_{obs} is lower than t_{val}, the slope is not significantly different from 1.

A.5 Test for intercept

Calculate the ratio t'_{obs} according to Formula (A.10):

$$t'_{obs} = \frac{\frac{\sum_{i=1}^{m}(\rho_{i,m} - \rho_{i,ref})}{m}}{\sqrt{\frac{\sum_{i=1}^{m}(\rho_{i,m} - \rho_{i,ref})^2 - \frac{\left[\sum_{i=1}^{m}(\rho_{i,m} - \rho_{i,ref})\right]^2}{m}}{m\times(m-1)}}} \quad \text{(A.10)}$$

Compare the t'_{obs} value with a critical value t_{val} corresponding to that of a 5 % risk student variable with m – two degrees of freedom.

If t'_{obs} is lower than t_{val}, the intercept is not significantly different from 0.

A.6 Interpretation and conclusion

A.6.1 Performances of a standard instrument

If tests for freedom from bias by comparison of means or the tests for slope and intercept are not satisfactory, the instrument to be traced shall be adjusted by correcting bias and/or slope according to the values specified in 7.1.5 or 7.2.5.

It is necessary to resume the measurements and calculations after this adjustment to verify the conformity of new results (see Figure A.1).

If the instrument is not adjustable, it cannot claim the status of a standard (national or secondary) and shall be replaced by a more powerful instrument that meets the required specifications.

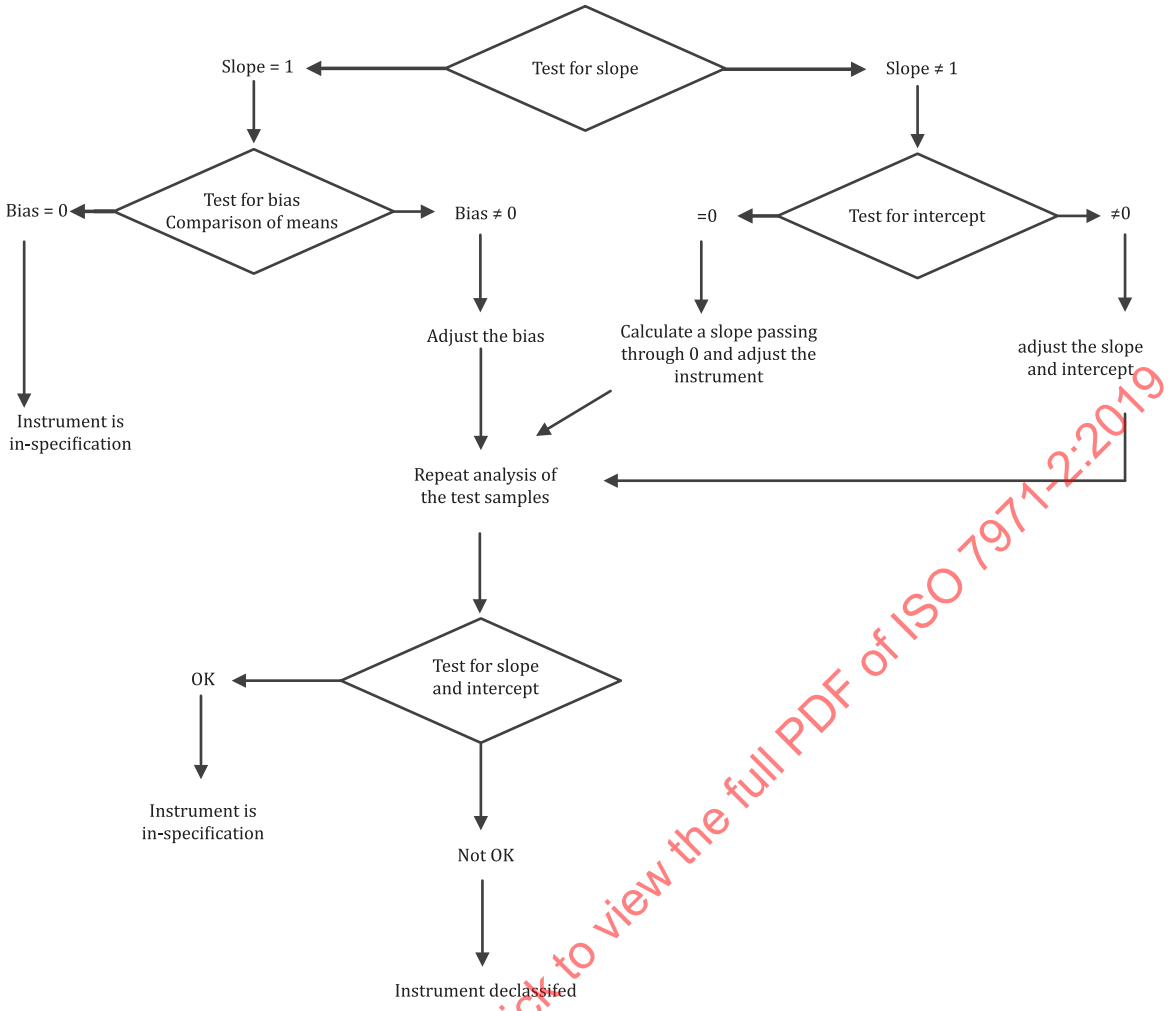

Figure A.1 — Traceability of a standard instrument — Decision flowchart

A.6.2 Performances of a measurement instrument

If the test for freedom from bias is not satisfactory, the instrument shall be adjusted by correcting the bias, as stated in 7.3.5.

It is necessary to resume the measurements and calculations after this adjustment to verify the conformity of new results.

If the instrument is not adjustable, it is unfit for purpose and shall not be used.

A.7 Example of calculations

A.7.1 Traceability of a standard instrument to a superior-level standard instrument

A.7.1.1 General

Each of 14 test samples is measured 3 times by the standard instrument then by the instrument to be traced. Results are given in Tables A.1 and A.2.

A.7.1.2 Calculation and tests for slope and intercept

See Table A.3.

Table A.1 — Reference values, superior-level standard instrument

Measurements on the standard instrument kg/hl		Test sample 1 $\rho_{1j,ref}$	Test sample 2 $\rho_{2j,ref}$	Test sample 3 $\rho_{3j,ref}$	Test sample 4 $\rho_{4j,ref}$	Test sample 5 $\rho_{5j,ref}$	Test sample 6 $\rho_{6j,ref}$	Test sample 7 $\rho_{7j,ref}$	Average of all test samples
Measure 1 kg/hl	$\rho_{i1,ref}$	73,90	67,36	76,66	81,70	69,54	62,32	75,10	
Measure 2 kg/hl	$\rho_{i2,ref}$	73,99	67,46	76,58	81,49	69,68	62,52	74,95	
Measure 3 kg/hl	$\rho_{i3,ref}$	74,01	67,47	76,75	81,35	69,78	62,52	74,95	
Average kg/hl	$\rho_{i,ref}$	73,96	67,43	76,66	81,51	69,66	62,46	75,00	
Measurements on the standard instrument kg/hl		**Test sample 8** $\rho_{8j,ref}$	**Test sample 9** $\rho_{9j,ref}$	**Test sample 10** $\rho_{10j,ref}$	**Test sample 11** $\rho_{11j,ref}$	**Test sample 12** $\rho_{12j,ref}$	**Test sample 13** $\rho_{13j,ref}$	**Test sample 14** $\rho_{14j,ref}$	
Measure 1 kg/hl	$\rho_{i1,ref}$	72,90	76,81	68,79	81,45	83,77	77,43	81,77	
Measure 2 kg/hl	$\rho_{i2,ref}$	72,89	76,88	68,51	81,49	83,95	77,45	81,85	
Measure 3 kg/hl	$\rho_{i3,ref}$	72,91	76,88	68,65	81,41	83,9	77,53	81,80	
Average kg/hl	$\rho_{i,ref}$	72,90	76,86	68,65	81,45	83,87	77,47	81,81	74,98

Table A.2 — Values obtained for the standard instrument to be traced

Measurements on the standard instrument to be traced		Test sample 1 $\rho_{1j,m}$	Test sample 2 $\rho_{2j,m}$	Test sample 3 $\rho_{3j,m}$	Test sample 4 $\rho_{4j,m}$	Test sample 5 $\rho_{5j,m}$	Test sample 6 $\rho_{6j,m}$	Test sample 7 $\rho_{7j,m}$	Average of all test samples
Measure 1 kg/hl	$\rho_{i1,m}$	74,01	67,50	76,78	81,59	69,63	62,50	74,83	
Measure 2 kg/hl	$\rho_{i2,m}$	73,86	67,28	76,86	81,80	69,53	62,58	74,91	
Measure 3 kg/hl	$\rho_{i3,m}$	73,89	67,25	76,80	81,68	69,65	62,75	75,07	
Average kg/hl	$\rho_{i,m}$	73,92	67,34	76,81	81,69	69,60	62,61	74,94	
Measurements on the standard instrument to be traced		**Test sample 8** $\rho_{8j,m}$	**Test sample 9** $\rho_{9j,m}$	**Test sample 10** $\rho_{10j,m}$	**Test sample 11** $\rho_{11j,m}$	**Test sample 12** $\rho_{12j,m}$	**Test sample 13** $\rho_{13j,m}$	**Test sample 14** $\rho_{14j,m}$	
Measure 1 kg/hl	$\rho_{i1,m}$	72,72	76,76	68,80	81,34	83,90	77,37	81,72	
Measure 2 kg/hl	$\rho_{i2,m}$	72,97	76,78	68,77	81,28	83,94	77,34	81,64	
Measure 3 kg/hl	$\rho_{i3,m}$	72,70	76,78	68,71	81,39	83,90	77,52	81,82	
Average kg/hl	$\rho_{i,m}$	72,80	76,78	68,76	81,34	83,91	77,41	81,73	74,97

Table A.3 — Calculation and tests for slope and intercept

Averages of measurements		Test sample 1	Test sample 2	Test sample 3	Test sample 4	Test sample 5	Test sample 6	Test sample 7	Sum	Average
Superior-level standard instrument	$\rho_{i,\mathrm{ref}}$	73,96	67,43	76,66	81,51	69,66	62,46	75,00		
Standard instrument to be traced	$\rho_{i,\mathrm{m}}$	73,92	67,34	76,81	81,69	69,60	62,61	74,94		
Averages of measurements		Test sample 8	Test sample 9	Test sample 10	Test sample 11	Test sample 12	Test sample 13	Test sample 14		
Superior-level standard instrument	$\rho_{i,\mathrm{ref}}$	72,90	76,86	68,65	81,45	83,87	77,47	81,81	1 049,69	74,98
Standard instrument to be traced	$\rho_{i,\mathrm{m}}$	72,80	76,78	68,76	81,34	83,91	77,41	81,73	1 049,64	74,97
		Test sample 1	Test sample 2	Test sample 3	Test sample 4	Test sample 5	Test sample 6	Test sample 7		
$(\rho_{i,\mathrm{ref}} - \bar{\rho}_{i,\mathrm{ref}})^2$		1,04	57,00	2,82	42,64	28,30	156,75	0,00		
		Test sample 8	Test sample 9	Test sample 10	Test sample 11	Test sample 12	Test sample 13	Test sample 14		
$(\rho_{i,\mathrm{ref}} - \bar{\rho}_{i,\mathrm{ref}})^2$		4,33	3,53	40,07	41,86	79,03	6,20	46,65	510,23	
		Test sample 1	Test sample 2	Test sample 3	Test sample 4	Test sample 5	Test sample 6	Test sample 7		
$(\rho_{i,\mathrm{m}} - \bar{\rho}_{i,\mathrm{m}})^2$		1,10	58,22	3,39	45,16	28,84	152,77	0,00		
		Test sample 8	Test sample 9	Test sample 10	Test sample 11	Test sample 12	Test sample 13	Test sample 14		
$(\rho_{i,\mathrm{m}} - \bar{\rho}_{i,\mathrm{m}})^2$		4,71	3,28	38,56	40,58	79,92	5,95	45,70	508,17	
		Test sample 1	Test sample 2	Test sample 3	Test sample 4	Test sample 5	Test sample 6	Test sample 7		
$\rho_{i,\mathrm{m}}^2$		5 464,17	4 534,68	5 899,78	6 673,26	4 844,16	3 920,01	5 616,00		
		Test sample 8	Test sample 9	Test sample 10	Test sample 11	Test sample 12	Test sample 13	Test sample 14		
$\rho_{i,\mathrm{m}}^2$		5 299,84	5 895,17	4 727,94	6 616,20	7 040,89	5 992,31	6 679,79	79 204,18	
		Test sample 1	Test sample 2	Test sample 3	Test sample 4	Test sample 5	Test sample 6	Test sample 7		

Table A.3 *(continued)*

Averages of measurements		Test sample 1	Test sample 2	Test sample 3	Test sample 4	Test sample 5	Test sample 6	Test sample 7	Sum	Average
$\rho_{i,\mathrm{ref}} - \rho_{i,\mathrm{m}}$		5 467,12	4 540,74	5 888,25	6 658,55	4 848,34	3 910,62	5 620,50		
		Test sample 8	Test sample 9	Test sample 10	Test sample 11	Test sample 12	Test sample 13	Test sample 14		
$\rho_{i,\mathrm{ref}} - \rho_{i,\mathrm{m}}$		5 307,12	5 901,31	4 720,37	6 625,14	7 037,53	5 996,95	6 686,33	79 208,89	
		Test sample 1	Test sample 2	Test sample 3	Test sample 4	Test sample 5	Test sample 6	Test sample 7		
$\rho_{i,\mathrm{m}} - \rho_{i,\mathrm{ref}}$		−0,04	−0,09	0,15	0,18	−0,06	0,15	−0,06		
		Test sample 8	Test sample 9	Test sample 10	Test sample 11	Test sample 12	Test sample 13	Test sample 14		
$\rho_{i,\mathrm{m}} - \rho_{i,\mathrm{ref}}$		−0,1	−0,08	0,11	−0,11	0,04	−0,06	−0,08	−0,004	
		Test sample 1	Test sample 2	Test sample 3	Test sample 4	Test sample 5	Test sample 6	Test sample 7		
$\left(\rho_{i,\mathrm{m}} - \rho_{i,\mathrm{ref}}\right)^2$		0,001 6	0,008 1	0,022 5	0,032 4	0,003 6	0,022 5	0,003 6		
		Test sample 8	Test sample 9	Test sample 10	Test sample 11	Test sample 12	Test sample 13	Test sample 14		
$\left(\rho_{i,\mathrm{m}} - \rho_{i,\mathrm{ref}}\right)^2$		0,01	0,006 4	0,012 1	0,012 1	0,001 6	0,003 6	0,006 4	0,15	
Slope	a	1,002								
Intercept	b	0,138								
Slope standard deviation	sa	0,005	Conclusion			Slope is not significantly different from 1. The standard instrument is not skewed.				
Test for slope	t_{obs}	0,387								
Student 5 %	t_{val}	2,179								
Test for intercept	t'_{obs}	0,376	Conclusion			Intercept is not significantly different from 0. The standard instrument is not biased.				
Student 5 %	t_{val}	2,179								

A.7.1.3 Comparison of means

See Table A.4.

Table A.4 — Comparison of means

Comparison of means		**Test sample 1**	**Test sample 2**	**Test sample 3**	**Test sample 4**	**Test sample 5**	**Test sample 6**	**Test sample 7**	**Mean** $\overline{\Delta\rho}$
Difference kg/hl	$\Delta\rho_{i,\mathrm{m\text{-}ref}}$	−0,04	−0,09	0,15	0,18	−0,06	0,15	−0,06	
Comparison of means		**Test sample 8**	**Test sample 9**	**Test sample 10**	**Test sample 11**	**Test sample 12**	**Test sample 13**	**Test sample 14**	**Mean** $\overline{\Delta\rho}$
Difference kg/hl	$\Delta\rho_{i,\mathrm{m\text{-}ref}}$	−0,10	−0,08	0,11	−0,11	0,04	−0,06	−0,08	−0,004

									Sum
Comparison of means	**Test sample 1**	**Test sample 2**	**Test sample 3**	**Test sample 4**	**Test sample 5**	**Test sample 6**	**Test sample 7**		
$(\overline{\Delta\rho})^2$	0,002	0,008	0,023	0,032	0,004	0,023	0,004		
Comparison of means	**Test sample 8**	**Test sample 9**	**Test sample 10**	**Test sample 11**	**Test sample 12**	**Test sample 13**	**Test sample 14**		
$(\overline{\Delta\rho})^2$	0,010	0,006	0,012	0,012	0,002	0,004	0,006	0,15	

	Standard deviation of the mean $s_{\Delta\rho/root(m)}$	0,028

Ratio		t_{obs}	0,126
Student 5 %		t_{val}	2,179

Conclusion $t_{obs} < t_{val}$ The two means are not significantly different.

The two instruments give similar average results.

A.7.2 Traceability of a measurement instrument

A.7.2.1 General

Each of four test samples is measured two times by the measurement instrument to be traced. Results are given in Tables A.5 and A.6.