INTERNATIONAL STANDARD

ISO 7971-3

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

Part 3: Routine method

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

Partie 3: Méthode pratique

Reference number
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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).

The first edition of ISO 7971-3 cancels and replaces the first edition of ISO 7971-2:1995, which has been technically revised.

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 3: Routine method

1 Scope

This part of ISO 7971 specifies a routine method for the determination of bulk density, called "mass per hectolitre" of cereals as grain using manual or automatic, mechanical, electric or electronic mass per hectolitre measuring instruments.

NOTE Further details of the measuring instruments are specified in ISO 7971-2:2009, 6.4.

2 Normative references

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 7971-2, *Cereals — Determination of bulk density, called mass per hectolitre — Part 2: Method of traceability for measuring instruments through reference to the international standard instrument*

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

NOTE 2 The bulk density, as defined in this part of ISO 7971, is different from "packing density" or "intrinsic density" of cereals.

[ISO 7971-1:2009]

4 Principle

The mass per hectolitre of a cereal is obtained from the mass of a volume of cereal determined under controlled sample filling and flow conditions.

The mass per hectolitre can be affected by:

a) space between the grains, which depends on the grain size and shape;

b) density of the grains.

5 Apparatus

5.1 General requirement for bulk density apparatus. Any apparatus (5.2 and 5.3) shall be verified according to ISO 7971-2 and shall fulfil the performance demands specified therein.

5.2 Hand-operated measuring instrument. Apparatus consisting of a filling hopper, a measuring container and the accessories necessary for their use.

The manner in which the grain is poured into the measuring container and the way in which it packs into the container can cause the measurements taken by the various instruments to vary and lead to measurement errors.

To minimize such variations, special attention should be given to ensuring that the design of the instruments and their size, material and shape are appropriate.

NOTE Annexes A and B of this part of ISO 7971 contain examples of technical specifications of two hand-operated instruments with a capacity of 1 l.

5.3 Automatic measuring instrument. This category includes various types of devices, some of which can be used on their own or combined with an infrared analyser.

The measurement is based on the application of equations to allow the correcting of the bias and/or the drifts monitored. It does not include manual weighing. The numeric value of the hectolitre mass is directly displayed.

5.4 Analytical balance, capable of being read to the nearest 0,1 g or 0,01 g depending on the volume of the container (see 6.2).

6 Procedure

6.1 General

The measurements shall be taken using grain from which large impurities (straw, stones, husks, etc.) have been discarded, taking environmental conditions into consideration to ensure that there is no difference in temperature between the grain and the room in which the test is performed.

Determine the bulk density in duplicate. For all the devices and for every sample, it is advisable to perform the two measurements on two different grain test portions.

NOTE Repeating the measurement on the same grain test portion changes the friction coefficient which therefore makes it easier for the grains to slide; they are then more tightly packed, which increases the value of the bulk density.

6.2 Hand-operated instruments

Check that the various components of the instrument are clean and that they are working properly.

Make sure that equipment is placed on a firm, flat base, after using a spirit level to check that the base is horizontal.

Take great care to avoid any impact during filling. If the apparatus is jolted, cancel the test and start again.

Each type of apparatus is different; use each according to the manufacturer's instructions.

When using the analytical balance (5.4), weigh to the nearest 1 g for a 1 l container or the nearest 0,1 g for apparatus with a container of smaller volume.

6.3 Automatic instruments

As the operations to be performed prior to the actual measurement differ according to the type of equipment used, reference to the manufacturer's instructions is recommended.

Ensure that the instrument is placed on a horizontal surface in a room protected from extreme temperatures, humidity, dust and vibrations.

Take particular care to:

a) select the correct cereal to be measured to ensure that the right calibration is used;

b) use the volume of cereals recommended for the device in question;

c) empty the collector drawer between samples.

6.4 Expression of results

Take the arithmetic mean of the two determinations as the result if the repeatability conditions are met.

Express the result to the nearest 0,1 kg/hl.

7 Precision

7.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in Annex C. The values derived from this interlaboratory test cannot be applied to other bulk density ranges and matrices than those given.

7.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, shall not in more that 5 % of cases be greater than the repeatability limit

$$r = 0{,}4$$

for products whose mass per hectolitre is between 67,5 kg/hl and 84,5 kg/hl (see Tables C.1, C.2, and Figure C.1).

7.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, shall not in more that 5 % of cases be greater than the reproducibility limit

$$R = 1{,}2$$

for products whose mass per hectolitre is between 67,5 kg/hl and 84,5 kg/hl (see Tables C.1, C.2, and Figure C.1).

7.4 Comparison of two groups of measurements in one laboratory

Critical difference, CD_r, is the difference between two averaged values obtained from two test results under repeatability conditions. As the result is a mean of two values (see 6.1), the comparison of two bulk densities shall be made with critical difference.

The critical difference, CD_r, between two averaged values obtained from two test results under repeatability conditions is given by:

$$CD_r = 2{,}8\, s_r \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2{,}8\, s_r \sqrt{\frac{1}{2}} = 1{,}98\, s_r = 0{,}23$$

i.e. 0,2 kg/hl, after rounding, where

s_r is the standard deviation of repeatability;

n_1 and n_2 are the number of test results corresponding to each averaged value (here, $n_1 = n_2 = 2$).

7.5 Comparison of two groups of measurements in two laboratories

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

$$CD_R = 2{,}8 \sqrt{s_R^2 - s_r^2 \left(1 - \frac{1}{2n_1} - \frac{1}{2n_2}\right)} = 2{,}8 \sqrt{s_R^2 - 0{,}5\, s_r^2} = 1{,}18$$

i.e. 1,2 kg/hl, after rounding, where

s_r is the standard deviation of repeatability;

s_R is the standard deviation of reproducibility;

n_1 and n_2 are the number of test results corresponding to each of the averaged values (here $n_1 = n_2 = 2$).

7.6 Uncertainty

Uncertainty, U_e, is a parameter representing the distribution of the values which may reasonably be attributed to the result. This uncertainty is given by a statistical distribution of the results from the interlaboratory test and is characterized by the experimental standard deviation.

For mass per hectolitre, the uncertainty is given by

$$U_e = \pm 2 s_R$$

$$U_e = \pm 0{,}43 \times 2 = \pm 0{,}86$$

i.e. 0,9 kg/hl, after rounding.

8 Test report

The test report shall contain at least the following information:

a) an indication of the method used, including a reference to this part of ISO 7971;

b) the result obtained;

c) all the operating details not specified in this part of ISO 7971 or those regarded as optional, in addition to any possible incidents that might have affected the result;

d) all the information necessary for a full identification of the sample.

Annex A
(informative)

Description of dimensions and use of KERN[1)] apparatus

A.1 Dimensions of the apparatus

A.1.1 General

The dimensions of the various components of the apparatus should be as specified in A.1.2 to A.1.7. See Figure A.1 for a depiction of the components.

A.1.2 Pre-filling measure

Volume to level mark	1 350 ml ± 10 ml
Internal diameter	86 mm ± 0,2 mm

A.1.3 Filling hopper

Internal diameter	79 mm ± 0,1 mm
Wall thickness	1 mm ± 0,2 mm
Height above piston	280 mm ± 2 mm

A.1.4 Piston

Diameter	87,5 mm ± 0,1 mm
Height	40 mm ± 0,2 mm
Mass	450 g ± 2 g

A.1.5 Measuring container

Internal diameter	88,2 mm ± 0,1 mm
Internal height above piston	163,7 mm ± 0,1 mm
Wall thickness	1,2 mm ± 0,5 mm

External reinforcement of upper edge:

— Thickness	2,5 mm ± 0,5 mm
— Height	6,0 mm ± 1,0 mm

1) Example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 7971 and does not constitute an endorsement by ISO of this product.

Base thickness	4,5 mm ± 0,1 mm
Diameter of base perforations	3,0 mm ± 0,1 mm
Foot height	9,0 mm ± 0,1 mm
Foot diameter	6,0 mm ± 0,1 mm
Gap between base and base plate	6,0 mm ± 0,1 mm
Number of perforations in base	1 + 4 + 8 + 12 + 16 + 20 + 24 = 85
Measuring ring:	
— internal diameter	88,2 mm ± 0,1 mm
— height	40,5 mm ± 0,1 mm

A.1.6 Base plate

Locating circle diameter	80,0 mm ± 0,1 mm

A.1.7 Straightedge (levelling blade)

Thickness	1 mm ± 0,05 mm
Cut-out angle	90° ± 2°
Width of cutting edge bevel	3 mm ± 0,5 mm

A.2 Specifications of the apparatus

A.2.1 Pre-filling measure. The pre-filling measure should be made of metal in the shape of a straight-sided cylinder closed at the bottom end, with a flat base plate. It should have a circular level mark on the inside wall situated no less than 10 mm and no more than 30 mm from the open end.

NOTE The pre-filling measure controls the way in which the filling hopper (A.2.2) is filled with grain and consequently reduces or eliminates possible operator errors.

A.2.2 Filling hopper. The hopper should be made of metal in the shape of a straight-sided cylinder which is open at both ends. At the bottom of the cylinder, an extended projection around the circumference of the cylinder enables the filling hopper to be pushed on to the ring at the top of the measuring container (A.2.3). The hopper receives a quantity of grain of more than 1 l from the pre-filling measure (A.2.1).

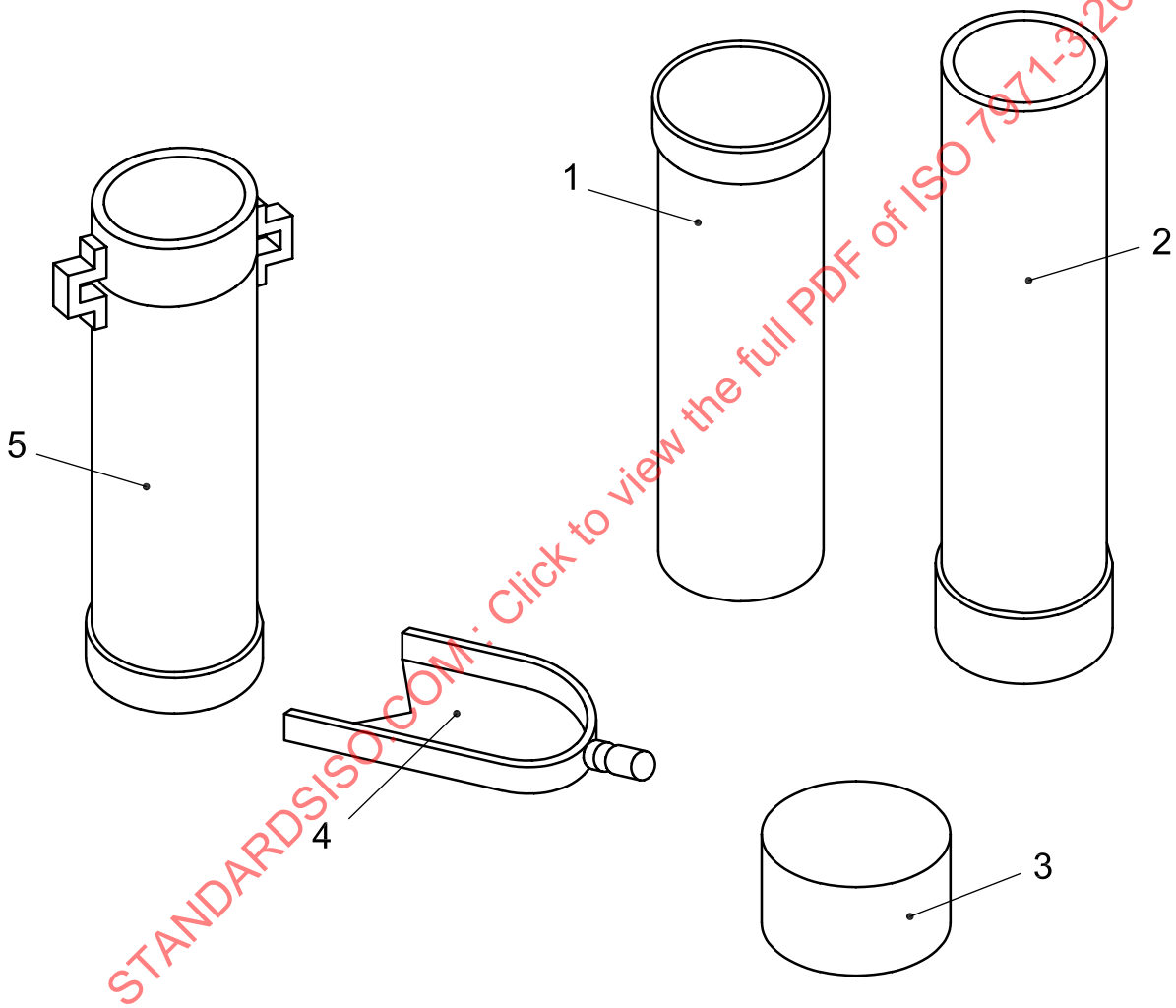
A.2.3 Measuring container with measuring ring. The 1 l volume of the measuring container is delimited by the inside surface of the container wall, the upper side of the piston (A.2.4) and the underside of the straightedge (A.2.5), once it is in position. The maximum permissible relative error in the capacity of the container is ± 3/1 000. The wall of the measuring container consists of a seamless drawn brass tube or a stainless steel tube in the shape of a straight-sided cylinder that is open at the top and closed at its base, with a reinforced outer edge. The top edge should be ground flat.

A measuring ring with the same internal diameter as the measuring container should be attached to the edge of the measuring container. The gap between the edge and the measuring ring should be sufficient to allow the straightedge (A.2.5) to pass through easily, but the clearance should not be too great.

The base of the measuring container should be flat and perforated to allow air to pass through when the instrument is in use. The external reinforcement around the base of the measuring container and its three feet should be in one piece. It should be firmly welded to the wall of the measuring container.

A.2.4 Piston. The piston should be made of brass in the shape of a straight-sided cylinder with flat ends. Its inner wall should be strengthened to enable it to withstand stamping without its surface being distorted. In the event of distortion or any other damage, the piston should be replaced to ensure that the volume of grain measured is not affected.

When the straightedge (A.2.5) is withdrawn, the piston moves slowly down the measuring container (A.2.3), forcing air through the vent holes drilled in the base of the measuring container. This movement controls the speed of the falling grain and ensures an even flow into the measuring container (A.2.3) from the filling hopper (A.2.2).



Key

1 pre-filling measure
2 filling hopper
3 piston
4 straightedge
5 measuring container

Figure A.1 — Apparatus for the determination of bulk density of cereals using a 1 l measuring container

A.2.5 Straightedge. The straightedge should consist of a thin but rigid, flat hardened steel blade with a handle. Its surfaces should be flat and parallel. It should be sufficiently wide to completely cover the cross-section of the measuring container at the end of its travel. The blade should be cut into a V shape which is open at the front and bevelled to ensure that the cutting line is in the middle of the thickness of the blade.

The blade slides horizontally into the slot in the measuring container (A.2.3) and, guided by this slot, it is thrust manually into the grain in a continuous, but smooth, movement. This movement separates exactly 1 l of grain (below the blade) from the excess grain (above the blade).

A.2.6 Base plate. The base plate should be made of metal and mounted in such a way that the measuring container (A.2.3) can be fitted securely to the base plate simply by rotating it. The base plate should not be perforated. It should be secured to a hardwood mounting plate or to the hardwood lid of the instrument carrying case. The mounting plate or case should be provided with vertical adjustment screws and a spirit level to ensure that the instrument remains vertical and in position after being placed on a flat, horizontal surface. Otherwise, errors are inevitable.

A.3 Determination

Fill the pre-filling measure with the sample of grain up to the level mark. Then empty it to within 30 mm or 40 mm from the upper edge of the filling hopper in such a way that the grain sample flows evenly into the middle of the filling hopper in 11 s to 13 s. After filling, quickly pull out the straightedge, but without shaking the apparatus.

When the piston and the grain have fallen into the measuring container, place the straightedge back in the slit and push it through the grain in a single stroke. If a particle becomes jammed between the slit edge and the straightedge in the process, the pouring should be repeated. Throw out excess grain lying on the straightedge. Then remove the filling hopper and straightedge.

Throughout the procedure, the apparatus should not be tapped, knocked or shaken, otherwise a falsely high result is obtained. However, once the 1 l volume has been isolated, this restriction need not be observed.

Use the balance (5.4) to weigh the content of the measuring container to the nearest 1 g. Alternatively, the grain may be poured into a separate previously tared receptacle and weighed to the nearest 1 g.

A.4 Expression of results

To calculate the bulk density, ρ, expressed in kilograms per hectolitre, with this type of device, the following equations are applied:

for wheat

$$\rho = 0{,}100\,2\,m + 0{,}53 \qquad \text{(A.1)}$$

for barley

$$\rho = 0{,}103\,6\,m - 2{,}22 \qquad \text{(A.2)}$$

for rye

$$\rho = 0{,}101\,7\,m - 0{,}08 \qquad \text{(A.3)}$$

for oats

$$\rho = 0{,}101\,3\,m - 0{,}61 \qquad \text{(A.4)}$$

where m is the mass, in grams, of the cereal.

NOTE Equations (A.1) to (A.4) provide linear mathematical conversions from grams per litre to kilograms per hectolitre. The factors are taken from data published in Reference [6].

Annex B
(informative)

Description of dimensions and use of NILEMA LITRE[2)] apparatus

B.1 Dimensions of the apparatus

See Figure B.1.

B.2 Specifications of the apparatus

B.2.1 Filling hopper with a gate valve, designed to allow an even flow of grain. The hopper fits on to the 1 l measuring container by means of a riser sleeve with a horizontal slot at the bottom allowing the straightedge to slide horizontally.

B.2.2 Straight-sided cylindrical measuring container with a capacity of 1 l.

B.2.3 Flat straightedge ending in a sharp V-shaped cutting edge.

B.3 Determination

Use the balance (5.4) to weigh the empty measuring container to the nearest 1 g.

Pour the grain sample into the filling hopper, filling it to the rim or to the reference mark, depending on the type of equipment, without compacting the grain.

Open the shutter and allow all the grain to flow into the measuring container.

Insert the straightedge gently into the slot as far as it can go, holding the container firmly in place to minimize vibrations and compaction.

With the container full of levelled grain, use the balance (5.4) to weigh to the nearest 1 g.

B.4 Expression of results

The bulk density, ρ, expressed in kilograms per hectolitre, of the cereal is given by:

$$\rho = \frac{m}{1000} \times \frac{100}{V} = \frac{m}{10V} \qquad \text{(B.1)}$$

2) Example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 7971 and does not constitute an endorsement by ISO of this product.

where

m is the mass, in grams, of the cereal in the measuring container obtained from $(m_1 - m_0)$

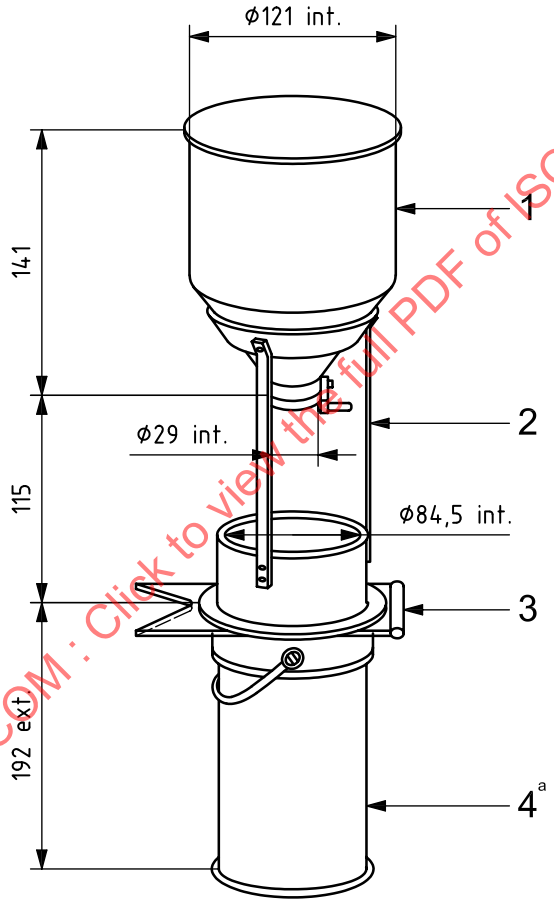
in which

m_0 is the mass, in grams, of the empty container,

m_1 is the mass, in grams, of the container filled with grain;

V is the volume, in litres, of the measuring container.

Dimensions in millimetres



Key

1 filling hopper
2 riser
3 straightedge
4 measuring container

[a] Capacity 1 l.

Figure B.1 — Nilema litre

Annex C
(informative)

Results of interlaboratory tests

The repeatability, reproducibility and critical difference of the method were established by statistical treatment of data obtained during two interlaboratory tests organized by Foss AB (SE) in 2006 and 2007. This treatment was done in accordance with the requirements of ISO 5725-2 [1], ISO 5725-3 [2], and ISO 5725-6 [3].

Between 13 and 16 laboratories took part in these tests on common wheat and barley. A total of 12 samples of wheat and four samples of barley were analysed.

The statistical results of the study are presented in Tables C.1, C.2, and Figure C.1.

Table C.1 — Statistical results of the interlaboratory test on wheat

Parameter	Test sample											
	1	2	3	4	5	6	7	8	9	10	11	12
Cereal	Wheat	Wheat	Wheat	Wheat	Wheat	Wheat	Wheat	Wheat	Wheat	Wheat	Wheat	Wheat
Year	2007	2006	2007	2006	2007	2007	2007	2007	2006	2006	2006	2006
Number of laboratories	16	13	16	13	16	16	16	16	13	13	13	13
Number of laboratories retained after elimination of outliers	16	12	16	12	16	16	16	16	13	13	13	13
Test bulk density, mean values, kg/hl	72,30	74,13	74,40	78,42	78,42	79,20	79,90	80,20	80,25	80,29	80,69	84,44
Repeatability standard deviation, s_r	0,10	0,08	0,08	0,13	0,11	0,13	0,17	0,13	0,09	0,16	0,11	0,11
Coefficient of variation of repeatability, CV(r), %	0,14	0,11	0,11	0,17	0,14	0,17	0,21	0,16	0,11	0,19	0,13	0,14
Repeatability limit ($r = 2{,}8\ s_r$)	0,28	0,22	0,22	0,36	0,31	0,36	0,48	0,36	0,25	0,45	0,31	0,31
Reproducibility standard deviation, s_R	0,29	0,35	0,31	0,31	0,32	0,36	0,35	0,64	0,44	0,38	0,37	0,89
Coefficient of variation of reproducibility, CV(R), %	0,40	0,48	0,42	0,4	0,41	0,46	0,43	0,80	0,55	0,48	0,46	1,06
Reproducibility limit ($R = 2{,}8\ s_R$)	0,81	0,98	0,87	0,87	0,90	1,01	0,98	1,79	1,23	1,06	1,04	2,49