INTERNATIONAL STANDARD

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Rubber compounding ingredients — Organic chemicals — General test methods

Ingrédients de mélange du caoutchouc — Produits chimiques organiques — Méthodes d'essai générales

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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 28641 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

Rubber compounding ingredients — Organic chemicals — General test methods

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies sampling and test methods for the determination of the general characteristics of organic chemicals such as accelerators, antidegradants (including wax) and vulcanizing agents (excluding peroxides).

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 649-1, *Laboratory glassware — Density hydrometers for general purposes — Part 1: Specification*

ISO 649-2:1981, *Laboratory glassware — Density hydrometers for general purposes — Part 2: Test methods and use*

ISO 760, *Determination of water — Karl Fischer method (General method)*

ISO 976:1996, *Rubber and plastics — Polymer dispersions and rubber latices — Determination of pH*

ISO 1770, *Solid-stem general purpose thermometers*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 3838, *Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods*

ISO 4625-1, *Binders for paints and varnishes — Determination of softening point — Part 1: Ring-and-ball method*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 6353-3, *Reagents for chemical analysis — Part 3: Specifications — Second series*

ISO 6472, *Rubber compounding ingredients — Abbreviations*

ISO 11235:1999, *Rubber compounding ingredients — Sulfenamide accelerators — Test methods*

ISO 11236:2000, *Rubber compounding ingredients — p-Phenylenediamine (PPD) antidegradants — Test methods*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 80000-1:2009, *Quantities and units — Part 1: General*

3 Abbreviations

The abbreviations of the chemical names of the organic accelerators and antidegradants used in this International Standard are in accordance with ISO 6472.

4 General requirements

4.1 Thermometer

Where a thermometer is used, it shall be a solid-stem thermometer meeting the requirements of ISO 1770 and shall be chosen according to the intended purpose. It shall have been calibrated before use with a standard thermometer.

4.2 Dessicator

Where a vacuum dessicator is used, the pressure reduction in the desiccator shall not be more than 2,0 kPa, unless otherwise specified.

5 Sampling

5.1 Apparatus

The apparatus used for sampling (see Annex A) shall be suitable for each test method.

5.2 Sampling method

Carry out sampling in accordance with ISO 15528.

To ensure homogeneity, thoroughly blend at least 250 g of the sample before taking any test portions.

6 Drying the sample

6.1 General

The drying method used will depend on the nature of the sample. Select a suitable method from those specified in 6.2 and 6.3. The method chosen shall not have a deleterious effect on the sample.

6.2 Drying methods for liquid compounding ingredients

6.2.1 Apparatus

6.2.1.1 Vacuum desiccator (see Figure B.1), capable of withstanding the reduced pressure specified.

6.2.1.2 Apparatus for drying liquid samples by passing a dry gas through the boiling liquid (see Figure B.2), consisting of a flask with a reflux condenser, a dry-gas inlet tube and a heating bath. The top of the condenser is connected to a suction pump. A drying tube is connected between the suction pump and the top of the condenser.

6.2.1.3 Apparatus for drying liquid samples by passing a dry gas through the liquid at ambient temperature (see Figure B.3), consisting of a flask and a dry-gas inlet tube. The top of the condenser is connected to a suction pump. A drying tube is connected between the suction pump and the top of the condenser.

6.2.2 Method using a desiccator

If carrying out the drying under ordinary pressure, dry the sample in a desiccator containing a suitable desiccant until the mass of the sample becomes constant, i.e. allow to dry for periods of 30 min until the loss in mass of the sample, unless otherwise specified, between two successive weighings is less than 3 mg.

When carrying out the drying under reduced pressure, dry the sample in a vacuum desiccator (6.2.1.1) containing a suitable desiccant at a pressure reduction of no more than 2,0 kPa until the mass of the sample becomes constant, i.e. allow to dry for periods of 30 min until the loss in mass of the sample, unless otherwise specified, between two successive weighings is less than 3 mg.

NOTE Examples of suitable desiccants are anhydrous calcium chloride, anhydrous sodium sulfate, anhydrous potassium carbonate, calcium oxide, aluminium oxide, potassium hydroxide, sodium hydroxide and silica gel. Phosphoric anhydride, concentrated sulfuric acid and metallic sodium may also be used.

6.2.3 Method using a dry gas

Use the apparatus described in 6.2.1.2 if drying the sample by passing a dry gas through the boiling sample.

Use the apparatus described in 6.2.1.3 if drying the sample at ambient temperature.

Dry the sample until the water content is less than 0,05 % as determined by the most appropriate method in ISO 760 (Karl Fischer).

NOTE Air, nitrogen or carbon dioxide is generally used as the drying gas.

6.2.4 Method using a desiccant added to the sample

Add a suitable desiccant to the sample and disperse it well by shaking. Then, keeping the desiccant suspended, filter the sample through a dried filter paper.

NOTE See the Note to 6.2.2.

6.2.5 Method using a solvent (suitable for viscous liquids)

Dissolve the sample in a suitable solvent and add a suitable desiccant to the solution. Disperse the desiccant by shaking and then filter the suspension through a dried filter paper. Eliminate the solvent remaining in the sample by distillation or simple evaporation.

6.3 Drying methods for solid compounding ingredients

6.3.1 Apparatus

6.3.1.1 Atmospheric-pressure or vacuum drying oven.

6.3.1.2 Apparatus for drying solid samples by passing a dry gas through the molten sample (see Figure B.4), consisting of a cylindrical flask fitted with a dry-gas inlet tube and a gas outlet tube which is connected to a suction pump. A drying tube is connected between the suction pump and the gas outlet tube containing the desiccant. A suitable heating bath is required.

6.3.1.3 Apparatus for drying solid samples by passing a dry gas through the sample at ambient temperature (see Figure B.5), including a cylindrical flask fitted with a perforated plate. Connected to the flask below the plate is a dry-gas inlet tube. Fitted to the top of the flask is a gas outlet tube which is connected to a suction pump. A drying tube containing a suitable desiccant is connected between the suction pump and the gas outlet tube.

6.3.2 Method using a desiccator

Carry out the drying, either at ordinary pressure or at reduced pressure, as described in 6.2.2.

6.3.3 Method using a drying oven

Dry the sample in a drying oven (6.3.1.1), with or without vacuum, to constant mass as defined in 6.2.2.

6.3.4 Method using a dry gas

Use the apparatus described in 6.3.1.2 if drying the sample in the molten state.

Use the apparatus described in 6.3.1.3 if drying the sample at ambient temperature.

Dry the sample until the water content is less than 0,05 % as determined by the most appropriate method in ISO 760 (Karl Fischer).

NOTE Air, nitrogen or carbon dioxide is generally used as the drying gas.

7 Test methods

7.1 Relative density

7.1.1 General

Select one of the following two methods for the determination of relative density, depending the nature of the material under test (hereafter referred to as the “sample”), the quantity available and the accuracy required:

a) hydrometer method (liquid sample);

b) pyknometer method (liquid or solid sample).

NOTE Relative density is generally measured at 20 °C and expressed as relative density (20 °C/20 °C). It represents the ratio of the mass of the sample in air at 20 °C to the mass of an equal volume of water in air at the same temperature.

7.1.2 Hydrometer method

7.1.2.1 Apparatus

7.1.2.1.1 Hydrometer, made of a suitable transparent glass, graduated in relative density at 20 °C, capable of measuring relative density at 20 °C over the range 0,600 to 2,000 and meeting the requirements of ISO 649-1. The hydrometer shall have been calibrated before use with a standard hydrometer.

7.1.2.1.2 Thermometer, as specified in 4.1.

7.1.2.1.3 Hollow cylinder, made of glass, having an inside diameter which is at least 25 mm larger than the maximum diameter of the hydrometer. The height shall be such that, when the hydrometer comes to rest, its base is at least 25 mm above the bottom of the cylinder.

7.1.2.1.4 Constant-temperature water bath, capable of maintaining a temperature of (20 ± 0,5) °C.

7.1.2.2 Procedure

a) Put the sample in the cylinder, avoiding the inclusion of air bubbles. Maintain the cylinder in the constant-temperature water bath. Stir the sample. Monitor the temperature of the sample with the thermometer, immersing it to the designated mark.

b) Condition the hydrometer at 20 °C ± 0,5 °C. When the temperature of the sample has reached 20 °C ± 0,5 °C, slowly put the conditioned hydrometer into the sample and allow it to come to rest. Then push the hydrometer into the sample by about two-scale divisions and release it.

c) When the hydrometer has stopped moving and is not in contact with the cylinder wall, read the scale to half the smallest graduation interval.

   For a translucent sample, read the scale at the point corresponding to the plane of intersection of the sample surface and the stem. Do this by gradually raising the eyes from a level a little below the sample surface and reading the scale when the elliptical sample surface becomes straight.

   For an opaque sample, read the scale at the upper edge of the meniscus of the sample surface and calculate the equivalent lower-edge value by applying a correction in accordance with Clause 4 of ISO 649-2:1981.

d) Record the result.

NOTE It is not necessary to make a correction if a hydrometer with a scale designed to be read at the upper edge of the meniscus is used.

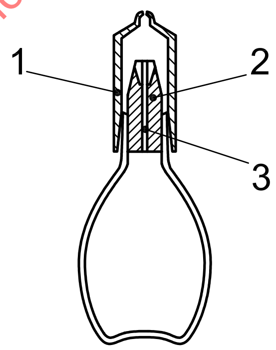
7.1.3 Pyknometer method

7.1.3.1 General

Two procedures are specified: one for liquid samples and one for powder samples.

7.1.3.2 Apparatus

7.1.3.2.1 Warden pyknometer (see ISO 3838), made of glass, with a capacity of about 50 cm^3 and fitted with a plug and a ground-glass cap as shown in Figure 1.



Key

1 cap

2 plug

3 capillary

Figure 1 — Warden pyknometer

7.1.3.2.2 Constant-temperature water bath, capable of maintaining the bath temperature at (20 ± 0,5) °C.

7.1.3.2.3 Thermometer, as specified in 4.1.

7.1.3.2.4 Laboratory balance, capable of weighing to the nearest 0,5 mg.

7.1.3.3 Method for liquid samples

7.1.3.3.1 Procedure

a) Weigh the pyknometer (mass m_0) to the nearest 0,5 mg. Fill it with water at a temperature slightly below 20 °C. Immerse the pyknometer up to its neck in the constant-temperature bath maintained at (20 ± 0,5) °C.

b) When the pyknometer and its contents have reached the bath temperature, insert the stopper, which has also been brought to the bath temperature. Take the pyknometer out the water bath and wipe the top of the stopper so that it is dry and the meniscus of the water in the capillary is flush with the top of the stopper.

c) Thoroughly wipe the external surface with, for instance, a clean dry cloth or tissue paper to remove all moisture and put on the cap.

d) Weigh it (mass m_1) to the nearest 0,5 mg.

e) Empty the pyknometer and dry it thoroughly. Then fill it completely with the sample at a temperature of approximately 20 °C and immerse it up to its neck in the water bath maintained at (20 ± 0,5) °C.

f) When its temperature has become constant, put in the stopper that has been maintained at the same temperature as the bottle. Wipe the top of the stopper so that it is dry and the meniscus of the sample in the capillary is flush with the top of the stopper. Take the pyknometer out of the water bath. Thoroughly wipe the external surface with, for instance, a clean dry cloth or tissue paper to remove all moisture and put on the cap.

g) Weigh it (mass m_2) to the nearest 0,5 mg.

7.1.3.3.2 Calculation

Calculate the relative density of the liquid sample using the following equation:

$$d = \frac{m_2 - m_0}{m_1 - m_0}$$

where

d is the relative density of the sample (at 20 °C/20 °C);

m_0 is the mass of the empty pyknometer, in grams;

m_1 is the mass of the pyknometer filled with water, in grams;

m_2 is the mass of the pyknometer filled with sample, in grams.

7.1.3.4 Method for powder samples

7.1.3.4.1 Procedure

a) Weigh the pyknometer empty (mass m_0) and filled with water (mass m_1) as described in 7.1.3.3.1 a) to d).

b) Empty the pyknometer and dry it thoroughly. Then take a test portion of about 4 cm^3 from the sample that has been dried by one of the drying methods given in Clause 6 and brought to a temperature of approximately 20 °C. Immerse it up to its neck in the water bath maintained at (20 ± 0,5) °C.

c) When its temperature has become constant, put in the stopper that has been maintained at the same temperature as the bottle. Then take the pyknometer out of the water bath, wipe its external surface with, for instance, a clean dry cloth or tissue paper to remove all moisture and put on the cap.

d) Weigh it (mass m_2) to the nearest 0,5 mg.

e) Fill the pyknometer containing the test portion with water at approximately 20 °C. Immerse it up to its neck in the water bath at (20 ± 0,5) °C. When its temperature has become constant, weigh it (mass m_3) to the nearest 0,5 mg, following the same procedure as in c) above.

If the sample is soluble in water, use another liquid, such as ethanol, toluene or *n*-octane.

7.1.3.4.2 Calculation

Calculate the relative density of the powder sample using the following equation:

$$d = \frac{m_2 - m_0}{m_2 + m_1 - m_0 - m_3} \times D$$

where

d is the relative density of the sample (at 20 °C/20 °C);

m_0 is the mass of the empty pyknometer, in grams;

m_1 is the mass of the pyknometer filled with water, in grams;

m_2 is the mass of pyknometer plus test portion, in grams;

m_3 is the mass of pyknometer plus test portion and water, in grams;

D is the relative density of water or the liquid used.

7.1.4 Expression of results

Round the result in accordance with Clause B.2 of ISO 80000-1:2009 to four places of decimals.

7.1.5 Test report

The test report shall include the following information:

a) all details necessary for the identification of the sample;

b) a reference to this International Standard;

c) the test method used (7.1.2 or 7.1.3);

d) the test temperature (20 °C);

e) the size of the test portion;

f) the laboratory temperature and humidity;

g) the test result;

h) any operation not included in this International Standard as well as any unusual features noted during the determination;

i) the date of the test.

7.2 Loss on heating

7.2.1 General

Use one of the following two methods:

— method A, in which the loss in mass of the sample when heated at 70 °C is regarded as the loss on heating;

— method B, in which the heating conditions (temperature, time) are selected from Tables 1 and 2 and the loss in mass of the sample when heated under these conditions is regarded as the loss on heating.

7.2.2 Method A

Method A is that specified in Clause 7 of ISO 11235:1999 and in Clause 10 of ISO 11236:2000.

7.2.3 Method B

7.2.3.1 Apparatus

7.2.3.1.1 Weighing bottle, squat form, 30 mm in height and 60 mm in diameter, fitted with a ground-glass stopper.

7.2.3.1.2 Drying oven, capable of maintaining a temperature selected from the range 35 °C to 110 °C within ± 2 °C.

7.2.3.1.3 Analytical balance, capable of weighing to the nearest 0,1 mg.

7.2.3.1.4 Desiccator.

7.2.3.2 Procedure

a) Dry the clean weighing bottle and the stopper in the drying oven. Allow them to cool to room temperature in the desiccator. Weigh the weighing bottle with the stopper to the nearest 0,1 mg. Record the mass (m_1).

b) Take a test portion of between 3 g and 5 g from the sample and put it into the weighing bottle. Insert the stopper and weigh the bottle to the nearest 0,1 mg. Record the mass (m_2).

c) Place the weighing bottle in the drying oven. Remove the stopper and place it near the bottle. Heat under the conditions specified in Table 1 (for accelerators) or Table 2 (for antidegradants). After heating, transfer the weighing bottle and stopper to the desiccator and leave them to reach equilibrium at room temperature. Weigh the weighing bottle and stopper to the nearest 0,1 mg. Record the mass (m_3).

d) Repeat procedure a) to c) to give a second result.

Table 1 — Heating temperature and heating time (accelerators)

<table>
<tr><th>Accelerator</th><th>Temperature
°C</th><th>Time
h</th><th>Accelerator</th><th>Temperature
°C</th><th>Time
h</th></tr>
<tr><td>BA</td><td rowspan="11">100 ± 2</td><td rowspan="11">2</td><td>CMBT [a]</td><td rowspan="12">55 ± 2</td><td rowspan="12">3</td></tr>
<tr><td>DPG</td><td>CBS</td></tr>
<tr><td>DOTG</td><td>TBBS</td></tr>
<tr><td>MBT</td><td>DCBS</td></tr>
<tr><td>MBTS</td><td>TETD</td></tr>
<tr><td>ZMBT</td><td>DPTH</td></tr>
<tr><td>ZDMC</td><td>DPTT</td></tr>
<tr><td>ZDEC</td><td>ZDBC</td></tr>
<tr><td>CuDMC</td><td>TeDEC</td></tr>
<tr><td>ZEPC</td><td>DETU</td></tr>
<tr><td>FeDMC [b]</td><td>DBTU</td></tr>
<tr><td>ETU</td><td rowspan="6">80 ± 2</td><td rowspan="6">2</td><td>TMU [c]</td></tr>
<tr><td>MBSS</td><td>DIBS</td><td>50 ± 2</td><td>3</td></tr>
<tr><td>TMTM</td><td colspan="3" rowspan="4"></td></tr>
<tr><td>TMTD</td></tr>
<tr><td>BQD</td></tr>
<tr><td>DBQD</td></tr>
<tr><td colspan="6">[a] Salt of 2-mercaptobenzothiazole.
[b] Ferric dimethyldithiocarbamate.
[c] Trimethylthiourea.</td></tr>
</table>

Table 2 — Heating temperature and heating time (antidegradants)

<table>
<tr><th>Antidegradant</th><th>Temperature
°C</th><th>Time
h</th><th>Antidegradant</th><th>Temperature
°C</th><th>Time
h</th></tr>
<tr><td>ADPA</td><td rowspan="9">100 ± 2</td><td rowspan="9">2</td><td>ETMQ</td><td rowspan="3">75 ± 2</td><td rowspan="3">2</td></tr>
<tr><td>SPH</td><td>AANA</td></tr>
<tr><td>MBI</td><td>PAN</td></tr>
<tr><td>ZMBI</td><td>ODPA</td><td rowspan="3">70 ± 2</td><td rowspan="3">2</td></tr>
<tr><td>o-MBp24</td><td>SDPA</td></tr>
<tr><td>p-BBp14</td><td>DCD [a]</td></tr>
<tr><td>p-TBp14</td><td>TMQ</td><td rowspan="3">50 ± 2</td><td rowspan="3">3</td></tr>
<tr><td>DBHQ</td><td>NDBC</td></tr>
<tr><td>DAHQ</td><td>MBMTB</td></tr>
<tr><td colspan="6">[a] 4,4′-dicumyldiphenylamine.</td></tr>
</table>

7.2.4 Calculation

Calculate the loss on heating using the following equation:

$$w_V = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

w_V is the loss on heating, in percent;

m_1 is the mass of the weighing bottle and stopper, in grams;

m_2 is the mass of the weighing bottle, stopper and test portion before heating, in grams;

m_3 is the mass of the weighing bottle, stopper and test portion after heating, in grams.

7.2.5 Expression of results

Round the results in accordance with Clause B.2 of ISO 80000-1:2009 to one place of decimals.

7.2.6 Precision

See Clause E.1.

7.2.7 Test report

The test report shall include the following information:

a) all details necessary for the identification of the sample;

b) a reference to this International Standard;

c) the method used (A or B);

d) the heating conditions (temperature and time);

e) the laboratory temperature and humidity;

f) the mass of the test portion;

g) the test results (individual values and mean value);

h) any operation not included in this International Standard as well as any unusual features noted during the determination;

i) the date of the test.

7.3 Sieve residue

7.3.1 General

The sieve residue shall be determined by a wet method. This method is suitable for powders of particle size up to 150 µm. However, when the sample is soluble in, or swollen by, water (or by ethanol or diethyl ether, which are also used in the determination), the test shall be carried out using other liquids which do not affect the sample.

7.3.2 Principle

The rubber compounding ingredients are passed through a sieve, rinsing with water in a defined manner. The residue left on the sieve is then dried and weighed.

7.3.3 Reagents

7.3.3.1 **Ethanol**, of purity greater than 95 %.

7.3.3.2 **Diethyl ether**, as specified in ISO 6353-3.

7.3.4 Apparatus

7.3.4.1 **Sieve**, as specified in ISO 3310-1.

7.3.4.2 **Flat brush**, 15 mm wide, with soft bristles about 25 mm long.

7.3.4.3 **Drying oven**.

7.3.4.4 **Desiccator**.

7.3.4.5 **Analytical balance**, capable of weighing to the nearest 0,1 mg.

7.3.4.6 **Beaker**, of capacity 100 cm^3.

7.3.4.7 **Evaporating dish**, about 120 mm in diameter.

7.3.5 Procedure

a) Dry the sieve in the oven maintained at a temperature between 105 °C and 110 °C. Allow it to cool in the desiccator and weigh it.

b) Repeat this procedure until the loss in mass on heating for 15 min is 1 mg or less and record the mass.

c) Take a test portion of between 5 g and 10 g from the sample and weigh it (mass m_0), to the nearest 0,1 mg, in the 100 cm^3 beaker. Moisten it by adding ethanol. Press it lightly with a glass rod to crush any lumps.

d) Add about 50 cm^3 of water and mix thoroughly. Decant the particles floating on the liquid onto the sieve the screen of which has previously been thoroughly wetted with ethanol.

e) Pour about 50 cm^3 of water on the remainder of the test portion. Decant it onto the sieve in the same manner as above. Transfer the whole of the test portion to the sieve by repeating this procedure.

f) Wash the test portion through the sieve by pouring successive small amounts of water on to the sieve and shaking the sieve. Then put the sieve in an evaporating dish. Pour water into the dish until it reaches a level 15 mm above the screen and sweep the surface of the screen with the brush at a rate of one stroke per second.

g) After every 20 strokes, lift the sieve from the dish to drain water through the screen and, in addition, renew the water in the dish after every 40 strokes.

h) Repeat this procedure until no more of the test portion passes through the sieve. Wash the solid matter adhering to the brush onto the sieve with water. Wash the sieve thoroughly with ethanol and finally with diethyl ether.

i) Dry the sieve in the oven maintained at the temperature specified in Table 1 (for accelerators) or Table 2 (for antidegradants) for 30 min. Allow it to cool in the desiccator, then weigh it. Repeat this procedure until the loss in mass due to 30 min of heating is 1 mg or less. Calculate the mass of the residue (m_1) by subtracting the mass of the sieve initially recorded from the final mass of the sieve plus dry residue.

7.3.6 Calculation

Calculate the sieve residue using the following equation:

$$w_S = \frac{m_1}{m_0} \times 100$$

where

w_S is the sieve residue, in percent;

m_0 is the mass of the test portion, in grams;

m_1 is the mass of the residue, in grams.

7.3.7 Expression of results

Round the result in accordance with Clause B.2 of ISO 80000-1:2009 to one place of decimals.

7.3.8 Precision

See Clause E.2.

7.3.9 Test report

The test report shall include the following information:

a) all details necessary for the identification of the sample;

b) a reference to this International Standard;

c) the size of opening of the sieve used;

d) the mass of the test portion;

e) the drying temperature used;

f) the laboratory temperature and humidity;

g) the test result;

h) any operation not included in this International Standard as well as any unusual features noted during the determination;

i) the date of the test.

7.4 pH of water extract

7.4.1 Principle

A test solution is prepared and the pH measured using a pH-meter.

7.4.2 Apparatus

7.4.2.1 pH-meter, chosen from the types given in Table C.3 and Table C.4, depending on the purpose of the test.

7.4.2.2 Thermometer, as specified in 4.1.

7.4.2.3 Conical flask, made of hard glass, of capacity 200 cm^3.

7.4.2.4 Analytical balance, capable of weighing to the nearest 1 mg.

7.4.2.5 Funnel.

7.4.2.6 Shaker.

7.4.3 Procedure

7.4.3.1 Preparation of test solution

7.4.3.1.1 General

Use one of the following two methods to prepare the test solution.

7.4.3.1.2 Method A

a) Weigh a test portion of about 5 g from the sample into the conical flask. Add 100 cm^3 of carbon-dioxide-free water and mark the water level on the outside of the flask. Boil for 5 min.

b) Compensate for the loss of water on boiling by making up to the mark on the outside of the flask. Boil the solution gently. Then allow it to cool it to room temperature with the flask lightly stoppered or covered with aluminium foil.

c) Take the solution or slurry, filtered or unfiltered, as the test solution.

7.4.3.1.3 Method B

a) Weigh a test portion of about 5 g from the sample into the conical flask. Add 100 cm^3 of carbon-dioxide-free water. Plug and shake it on the shaker for 5 min.

b) Take the solution or slurry, filtered or unfiltered, as the test solution.

7.4.3.2 Measurement

After calibrating the pH-meter in accordance with Annex D, wash the electrode and immediately measure the pH of the test solution. The amount of test solution shall be sufficient to cover the electrode completely. Furthermore, during the measurement, the variation in the temperature of the test solution shall not exceed the limits given in Table C.2.

NOTE For the measurement of pH-values greater than 11, there is a danger of ordinary glass electrodes generating an alkali error and therefore lowering the measured value. The higher the concentration of alkali metal ions, the larger the error. Therefore, it is preferable to use an electrode with a low alkali error and, in addition, to make a correction to allow for the error.

It is important that the accuracy of the pH-meter be checked at regular intervals (see Annex C).

7.4.4 Test report

The test report shall include the following information:

a) all details necessary for the identification of the sample;

b) a reference to this International Standard;

c) the type of pH-meter used and the name of the manufacturer;

d) the mass of the test portion;

e) the method of preparation of the test solution (method A or B);

f) the buffer solutions used to check the accuracy of the pH-meter (see Annex C);

g) the temperature at which the pH was measured;

h) the laboratory temperature and humidity;

i) the test result;

j) any operation not included in this International Standard as well as any unusual features noted during the determination;

k) the date of the test.

7.5 Melting point

7.5.1 General

Use one of the following methods for determining the melting point:

— method A (for powders): visually by heating a test sample in a capillary tube immersed in a heated liquid;

— method B (for water-insoluble and waxy samples such as fats, fatty acids, paraffins and waxes): visually by heating a test sample in a capillary tube immersed in a heated liquid;

— method C (for vaselines): visually by heating a test sample in a capillary in a heated liquid;

— method D: by using a differential scanning calorimeter.

7.5.2 Method A

Method A is that specified in 6.1 of ISO 11235:1999.

7.5.3 Method B

7.5.3.1 Apparatus

7.5.3.1.1 Melting point measurement apparatus, a suitable example of which is shown in Figure 2.

7.5.3.1.2 Thermometer, as specified in 4.1.

7.5.3.1.3 Water or **silicone oil**, for use as the heating medium. The silicone oil shall not degrade at the measurement temperature and shall have a dynamic viscosity at 25 °C between 50 mm^2/s and 100 mm^2/s.

7.5.3.1.4 Capillary tube, made of hard glass, 70 mm to 100 mm long, 0,8 mm to 1,2 mm in inside diameter, with a wall thickness of 0,2 mm to 0,3 mm.

7.5.3.1.5 Desiccator.

7.5.3.1.6 Beaker.

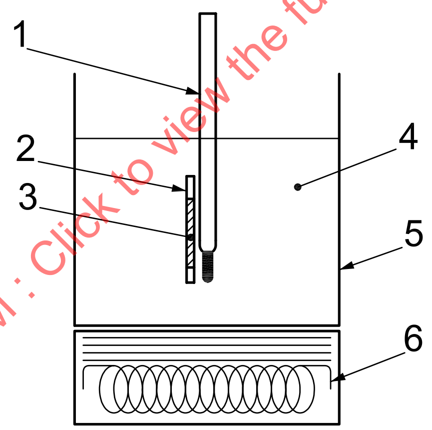
7.5.3.2 Procedure

a) Carefully melt a small portion of the sample at as low a temperature as possible. Suck the molten sample up to a height of about 10 mm in the capillary with both ends open, taking care to avoid the inclusion of bubbles.

b) Mount the capillary in a position such that the sample will not flow out. Condition it at less than 10 °C for at least 2 h. Then attach it to the stem of the thermometer using a rubber band, so that the sample is aligned with the centre of the mercury bulb. Put the capillary and thermometer in a beaker containing water or silicone oil. Keep the upper end of the sample 10 mm below the surface of the liquid.

c) Heat the liquid, while stirring continuously, until the temperature reaches 5 °C below the anticipated melting point. Then continue heating at a rate which will increase the temperature by about 1 °C/min.

d) Take the temperature at which the particles of sample begin to float in the partly melted sample as the melting point.



Key

1 thermometer

2 capillary with both ends open

3 test sample

4 bath liquid

5 beaker

6 heater

Figure 2 — Melting point measurement apparatus (example for method B)

7.5.4 Method C

7.5.4.1 Apparatus

See 7.5.3.1, but refer for an example of a suitable melting point measurement apparatus to Figure 3.

7.5.4.2 Procedure

a) Heat a small portion of the sample gradually up to 90 °C to 92 °C while stirring it. When this temperature, at which the sample will be completely melted, has been reached, stop heating and allow the portion of sample to cool to a temperature 8 °C to 10 °C above the melting point.

b) Cool the thermometer to 5 °C. Wipe and dry it. Put half of the mercury bulb in the molten sample and immediately pull it out and allow it to cool in the vertical position.

c) When the sample adhering to the bulb begins to show signs of turbidity, immerse it in water at a temperature not higher than 16 °C for 5 min.

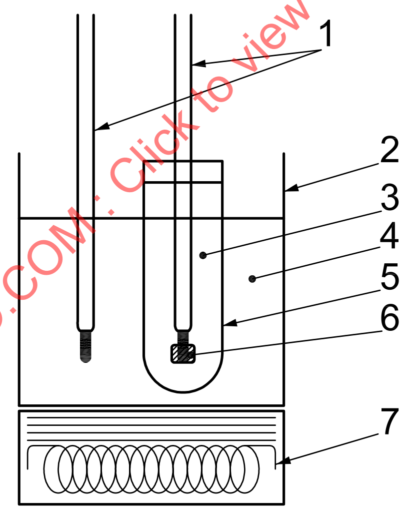
d) Insert the thermometer in a test tube and hold it in place with a cork plug so that the bottom of the bulb is 15 mm above the bottom of the test tube.

e) Suspend the test tube in a beaker containing water at about 16 °C and heat at a rate of about 2 °C/min until the temperature of the bath reaches 30 °C.

f) Record the temperature at which the first drop of sample detaches itself from the thermometer bulb.

g) Repeat this procedure twice (to give three results). If all three measurements lie within 1 °C of each other, take their mean value as the melting point. If the measurements lie over a range which is greater than 1 °C, make another two measurements and take the mean of the five measurements as the melting point.

Though the above procedure is the recommended one, two simultaneous measurements made using two thermometers with sample coated on the bulb of each may also be carried out, as specified in ISO 6244.



Key

1 thermometers
2 beaker
3 air
4 water
5 test tube
6 test sample
7 heater

Figure 3 — Melting point measurement apparatus (example for method C)

7.5.5 Method D

Method D is that specified in 6.2 of ISO 11235:1999.

7.5.6 Expression of results

Round the result in accordance with Clause B.2 of ISO 80000-1:2009 to one place of decimals.

7.5.7 Precision

See Clause E.3.

7.5.8 Test report

The test report shall include the following information:

a) all details necessary for the identification of the sample;

b) a reference to this International Standard;

c) the method used (A, B, C or D);

d) the laboratory temperature and humidity;

e) the test result;

f) any operation not included in this International Standard as well as any unusual features noted during the determination;

g) the date of the test.

7.6 Temperature of solidification

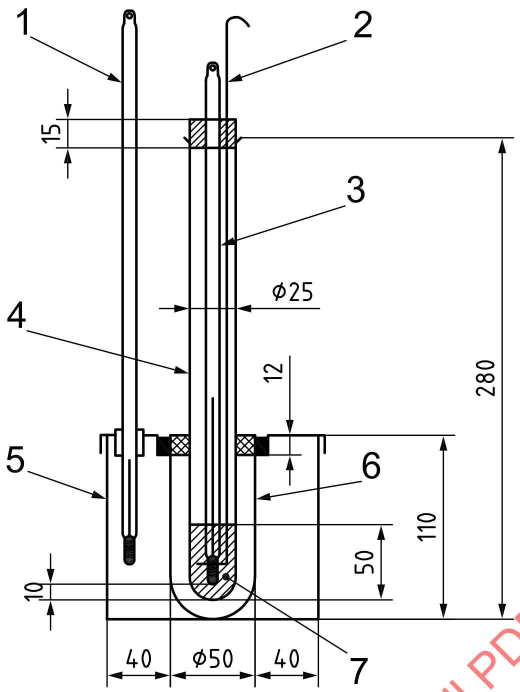
7.6.1 Principle

A test sample is indirectly cooled by cold water or a freezing mixture and the temperature of solidification is determined visually.

7.6.2 Apparatus

7.6.2.1 Solidification temperature measurement apparatus, a suitable example of which is shown in Figure 4.

Dimensions in millimetres



Key

1 thermometer B
2 glass or wire stirrer
3 thermometer A
4 sample holder
5 glass bath
6 glass air-jacket
7 test sample

Figure 4 — Solidification temperature measurement apparatus (example)

7.6.2.2 Glass air-jacket, coated on both the inside and the outside with silicone oil to prevent frosting of the wall.

7.6.2.3 Sample holder, made of hard glass and coated on both the inside and the outside with silicone oil (except the part of the inside surface which will come in contact with the test sample) to prevent frosting of the tube wall. The level to which the tube will be filled with sample is indicated by a circular mark. The test tube is inserted into the glass air-jacket and held in place with a cork stopper.

7.6.2.4 Glass water bath.

7.6.2.5 Glass or wire stirrer, of suitable diameter, with its lower end shaped into a ring of outside diameter 18 mm.

7.6.2.6 Thermometers (A and B), as specified in 4.1.

7.6.3 Procedure

a) If the sample is a solid, melt it by heating carefully so that the anticipated solidification temperature is not exceeded by more than 20 °C. Fill the sample holder up to the mark with molten sample. Pour water at a temperature 5 °C below the anticipated solidification temperature into the water bath.

b) If the sample is a liquid, fill the sample holder up to the mark. Pour water at a temperature 10 °C to 15 °C below the anticipated solidification temperature into the water bath.

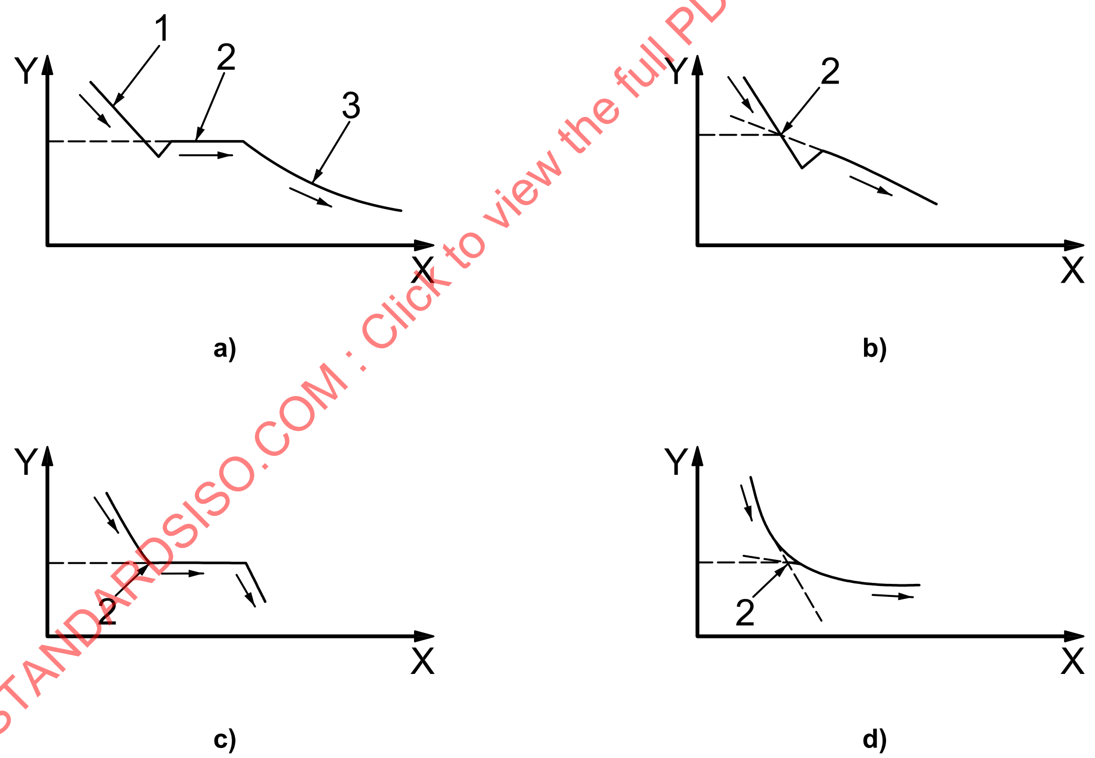
c) Insert the sample holder into the air-jacket. When the sample has cooled to a temperature 5 °C above the anticipated solidification temperature, gently move the stirrer up and down at a rate of 20 cycles/min and read the temperature indicated by thermometer A at 30 s intervals.

d) Stop stirring when the sample begins to solidify and read the temperature.

e) If the sample becomes supercooled, scratch the inside wall of the sample holder with the stirrer to accelerate solidification. When solidification does start, stop stirring and read the temperature at 10 s intervals. When the temperature has remained constant for 1 min, record the thermometer reading.

f) When at least four successive readings do not differ from each other by more than 0,2 °C, take their average as the solidification temperature.

When the sample includes many impurities, the solidification temperature will not follow a curve like that shown in Figure 5 a), but will follow one such as those shown in Figures 5 b), c) and d). In the case of Figures 5 b) and d), extrapolate the lines for the solid and liquid phases and take their point of intersection as the temperature of solidification.



Key

X time

Y temperature

1 liquid

2 temperature of solidification

3 solid

Figure 5 — Solidification temperature curves

7.6.4 Expression of results

Round the result in accordance with Clause B.2 of ISO 80000-1:2009 to one place of decimals.

7.6.5 Test report

The test report shall include the following information:

a) all details necessary for the identification of the sample;

b) a reference to this International Standard;

c) the laboratory temperature and humidity;

d) the test result;

e) any operation not included in this International Standard as well as any unusual features noted during the determination;

f) the date of the test.

7.7 Softening point

7.7.1 General

The softening point shall be determined in accordance with ISO 4625-1. The essential details of the apparatus and procedure are given below.

NOTE The softening point is defined as the softening temperature, measured under specific conditions, for a material, such as petroleum pitch or asphalt, which is opaque and has no definite melting point, and gives a large temperature difference between the softening and melting points.

7.7.2 Principle

A test sample is heated under specified test conditions and the temperature at which a ball falls a specified distance through the softening sample is measured and taken as the softening point.

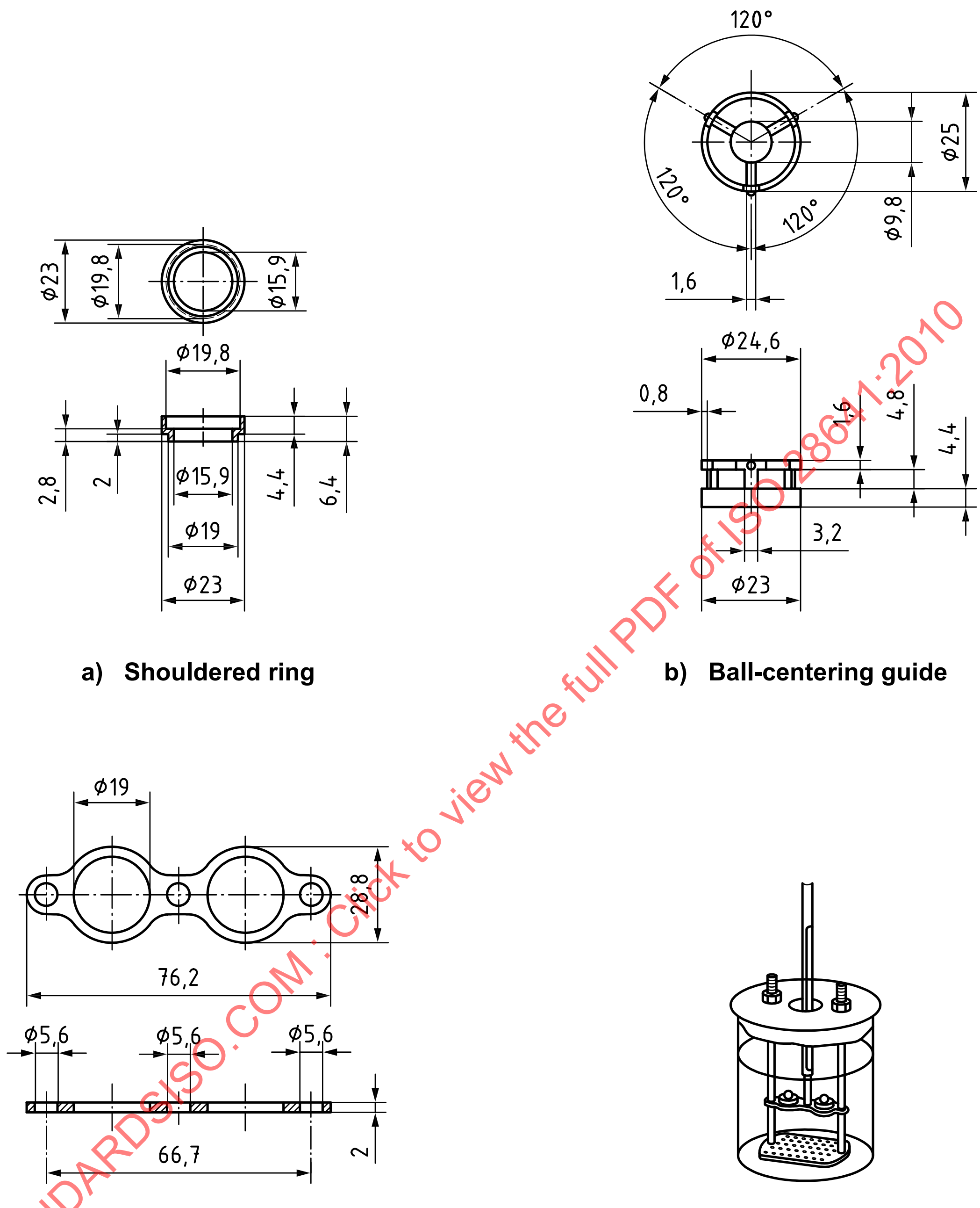
7.7.3 Apparatus

7.7.3.1 Softening point measurement apparatus, of the ring-and-ball type shown in Figure 6 and specified in ISO 4625-1.

It is recommended that a perforated bottom plate be included as shown in Figure 6 d) in order to assist in homogenizing the temperature of the bath.

7.7.3.2 Thermometer, as specified in 4.1.

Dimensions in millimetres



a) **Shouldered ring**

b) **Ball-centering guide**

c) **Ring holder**

d) **Assembled apparatus**

Figure 6 — Softening point measurement apparatus (example)

7.7.4 Procedure

a) Melt a suitable amount of sample at as low a temperature as possible. Place a ring on a sheet of aluminium foil and pour an excess quantity of molten sample into the ring, taking care to avoid the inclusion of bubbles. Leave for about 30 min to cool and solidify.

b) Cut off the excess sample material horizontally with a slightly heated knife. Place the filled ring in the ring holder and place the ball in the centre of the ring.

c) Transfer the ring holder to a beaker filled with water or glycerol (or, if these two liquids are unsuitable, with silicone oil). Mount the thermometer at the specified place and adjust its height so that the lower end of the bulb and the bottom of the ring lie in the same horizontal plane. Heat the water or glycerol to raise its temperature at a rate of approximately 5 °C/min.

d) Read the temperature at the time when the sample has softened to the extent that the ball has fallen onto the bottom plate located 25,4 mm below. Take this temperature as the softening point.

7.7.5 Expression of results

Round the result in accordance with Clause B.2 of ISO 80000-1:2009 to the nearest whole number.

7.7.6 Precision

See Clause E.4.

7.7.7 Test report

The test report shall include the following information:

a) all details necessary for the identification of the sample;

b) a reference to this International Standard;

c) the laboratory temperature and humidity;

d) the test result;

e) any operation not included in this International Standard as well as any unusual features noted during the determination;

f) the date of the test.

7.8 Density of the bulk material

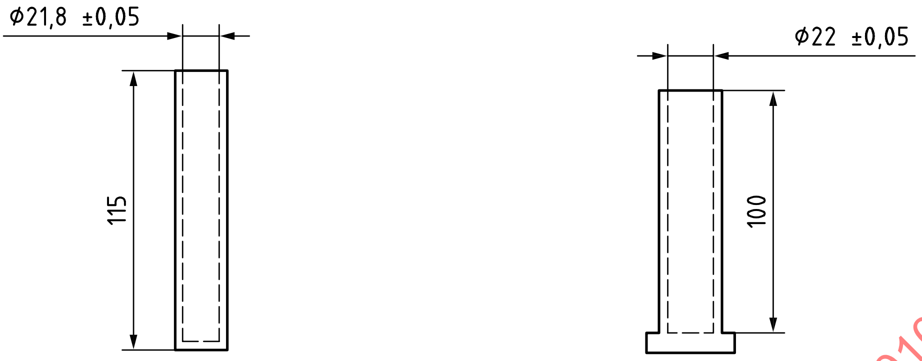
7.8.1 Principle

The bulk density is measured with a specified load applied to a test sample.

7.8.2 Apparatus

The apparatus used for measuring the density shall consist of a cylinder and a piston, made of ordinary steel, as shown in Figure 7. The cylinder shall have an inside diameter of (22,00 ± 0,05) mm and an inside depth of 100 mm. The piston shall have an outside diameter of (21,80 ± 0,05) mm and shall be 115 mm in length. It shall be hollowed out so that its mass is 190 g.

Dimensions in millimetres



a) Piston

b) Cylinder

Figure 7 — Bulk density measurement apparatus

7.8.3 Procedure

a) Put the piston into the cylinder. Allow the piston to sink under its own weight and measure the length of the projecting part to the nearest 0,01 mm.

b) Weigh from the sample, to the nearest 0,01 g, a test portion of 1 g to 5 g. Remove the piston and pour the sample gently into the cylinder. Shake the cylinder lightly or give it a gentle tap to dislodge any test portion adhering to the cylinder wall and to make the upper surface of the test portion flat.

c) Insert the piston into the cylinder and allow it to descend gradually and gently, controlling it by hand. The time taken for the piston to reach the test portion surface shall be 5 s. The arrival of the piston at the test portion surface can be sensed by the hand used to guide it.

d) When the piston has reached the test portion surface, give it one turn lightly with the fingers or tap the cylinder wall lightly with a piece of wood to settle the piston.

e) Measure the length of the projecting part of the piston to the nearest 0,01 mm.

7.8.4 Calculation

Calculate the density using the following equation:

$$\rho = \frac{m_0}{0{,}785\,4\,d^2 \times (h_2 - h_1)}$$

where

ρ is the density;

m_0 is the mass of the test portion, in grams;

h_1 is the length, in centimetres, of the part of the piston which projects from the cylinder when there is no test portion in the cylinder;

h_2 is the length, in centimetres, of the part of the piston which projects from the cylinder with the test portion in the cylinder;

d is the inside diameter of the cylinder, in centimetres.

7.8.5 Expression of results

Round the result in accordance with Clause B.2 of ISO 80000-1:2009 to two places of decimals.

7.8.6 Test report

The test report shall include the following information:

a) all details necessary for the identification of the sample;

b) a reference to this International Standard;

c) the mass of the test portion;

d) the laboratory temperature and humidity;

e) the test result;

f) any operation not included in this International Standard as well as any unusual features noted during the determination;

g) the date of the test.

7.9 Ash

7.9.1 Principle

A test portion is ignited and the mass of the residue determined by weighing.

7.9.2 Apparatus

7.9.2.1 Crucible, made of platinum or porcelain, with a lid.

7.9.2.2 Gas burner.

7.9.2.2 Electric furnace.

7.9.2.4 Desiccator.

7.9.2.5 Analytical balance, capable of weighing to the nearest 0,1 mg.

7.9.3 Procedure

a) Unless otherwise specified, weigh from the sample, to the nearest 0,1 mg, a test portion of 3 g to 5 g (m_0) into a platinum or porcelain crucible. In the case of metal salts, the mass shall be approximately 1 g.

b) Heat the test portion on a gas burner or in an electric furnace, initially at a low temperature, to evaporate the volatile constituents and carbonize the remainder.

c) Remove the lid of the crucible and complete the ashing by igniting the crucible in the electric furnace at 750 °C ± 25 °C for at least 2 h.

d) Determine the mass of the residue after cooling in the desiccator (m_1).

e) Unless otherwise specified, repeat step c) for periods of 30 min, followed each time by step d), until the loss in mass of the test portion between two successive weighings is less than 0,3 mg.

7.9.4 Calculation

Calculate the ash using the following equation:

$$w_A = \frac{m_1}{m_0} \times 100$$

where

w_A is the ash, in percent;

m_0 is the mass of the test portion, in grams;

m_1 is the mass of the residue, in grams.

7.9.5 Expression of results

Round the result in accordance with Clause B.2 of ISO 80000-1:2009 to two places of decimals.

7.9.6 Precision

See Clause E.5.

7.9.7 Test report

The test report shall include the following information:

a) all details necessary for the identification of the sample;

b) a reference to this International Standard;

c) the mass of the test portion;

d) the ignition temperature;

e) the laboratory temperature and humidity;

f) the test result;

g) any operation not included in this International Standard as well as any unusual features noted during the determination;

h) the date of the test.

7.10 Refractive index

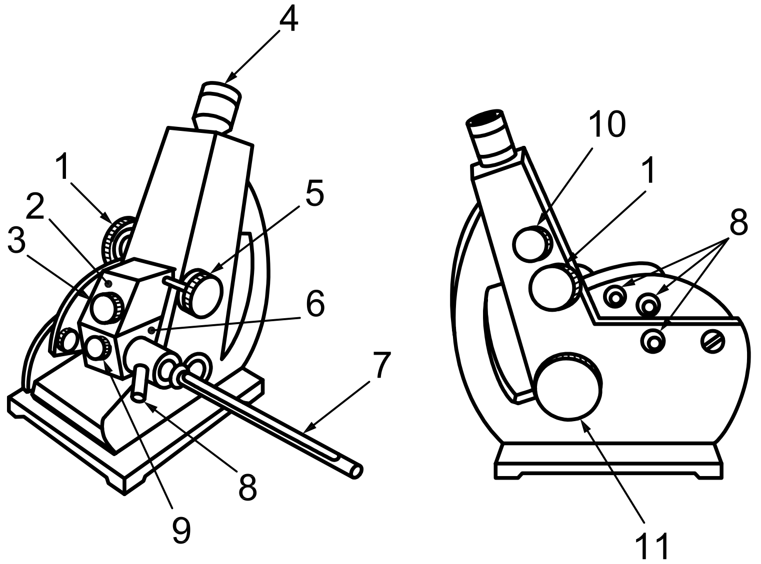
7.10.1 Principle

The refractive index is measured at a constant temperature using an Abbe refractometer.

7.10.2 Apparatus

7.10.2.1 Abbe refractometer, capable of reading to the nearest 0,000 1 units over the range 1,300 to 1,700. An example is shown in Figure 8.

7.10.2.2 Constant-temperature water bath, equipped with a circulating pump and capable of being maintained at 20 °C ± 0,2 °C.



Key

1 dispersion control
2 secondary prism
3 lighting port
4 eyepiece
5 secondary-prism control
6 main prism
7 thermometer
8 constant-temperature water inlet/outlet
9 coloured-liquid observation window
10 graduation adjuster
11 measuring wheel

Figure 8 — Abbe refractometer (example)

7.10.3 Procedure

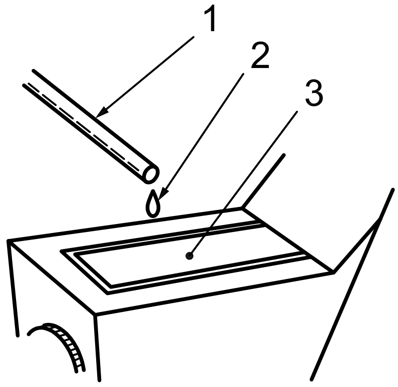
a) Let water at a constant temperature of 20 °C ± 0,2 °C flow through a refractometer whose scale has been adjusted beforehand using a test glass, a standard reference fluid or water (refractive index 1,333 0 at 20 °C and 1,332 0 at 30 °C). Lift the secondary prism and transfer a drop of the sample from the tip of a glass rod on to the main prism, ensuring that the surface of the prism is wetted uniformly (see Figure 9).

b) Immediately close the secondary prism so that the sample covers the surfaces of the main and secondary prisms uniformly. Read the temperature. Allow the refractometer to stand for a few minutes and then confirm that the thermometer indicates the same temperature as before.

c) When a refractometer provided with a reflector for measuring coloured liquids is used, adjust the light intensity beforehand by turning the dispersion control knob on the right-hand side of the instrument, while looking into the eyepiece, to make the dividing line between the light and dark areas distinctive.

d) Turn the measuring wheel so that the dividing line between the light and dark areas just reaches the intersection of the cross-wires in the field of view. Read the refractive index n_D off the scale immediately below the fixed vertical marker line in the field of view. By using the secondary scale at the bottom of the field of view, it should be possible to achieve an accuracy of ±0,000 1 units (see Figure 10).

e) Make three measurements and take the average of the results.



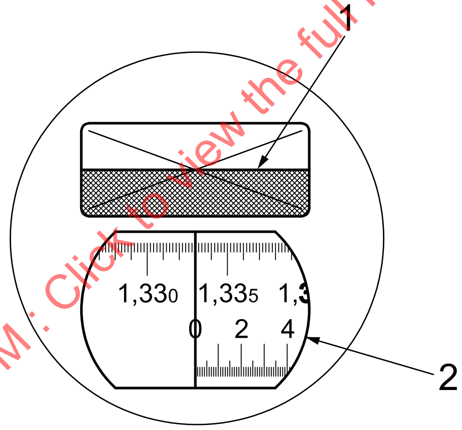
Key

1 glass rod

2 sample

3 surface of main prism

Figure 9 — Method of wetting the surface of the main prism



Key

1 dividing line between light and dark areas

2 n_D scale

Figure 10 — Method of reading the n_D scale

7.10.4 Expression of results

Round the result in accordance with Clause B.2 of ISO 80000-1:2009 to four places of decimals.

7.10.5 Precision

See Clause E.6.

7.10.6 Test report

The test report shall include the following information:

a) all details necessary for the identification of the sample;

b) a reference to this International Standard;

c) the test temperature;

d) the laboratory temperature and humidity;

e) the test results (individual values and mean value);

f) any operation not included in this International Standard as well as any unusual features noted during the determination;

g) the date of the test.

Annex A
(informative)

Examples of sampling apparatus

The following types of apparatus are used for sampling purposes:

a) a small liquid sampler, as shown in Figure A.1;

b) a granule and powder sampling tool, as shown in Figure A.2;

c) a sampling scoop, as shown in Figure A.3;

d) a tool for taking samples from solid materials, as shown in Figure A.4;

e) a spatula;

f) a small shovel.

Dimensions in millimetres

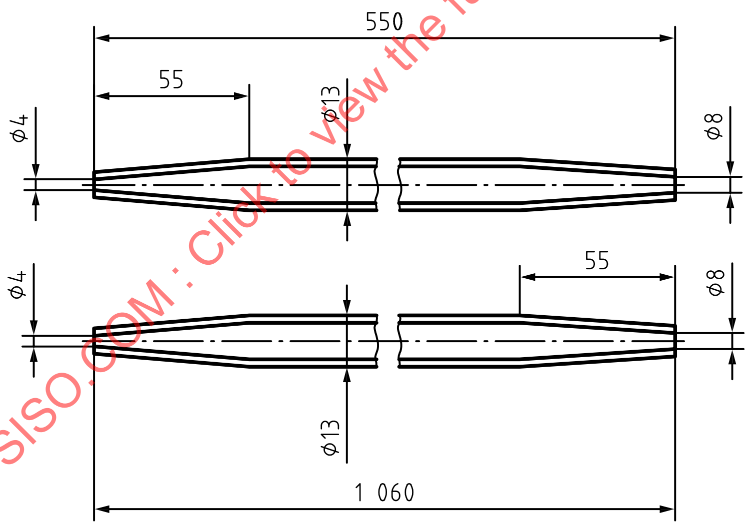


Figure A.1 — Examples of small liquid samplers (made of glass)

Dimensions in millimetres

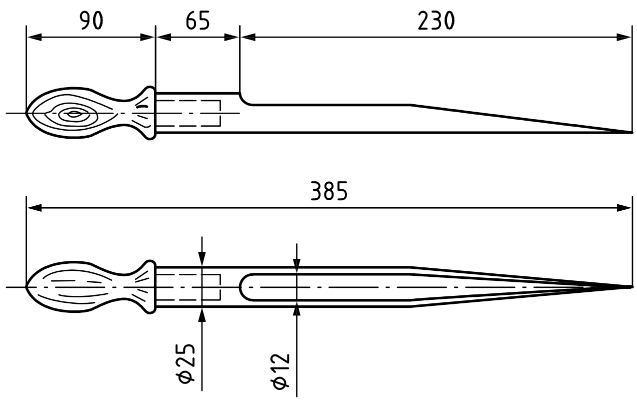


Figure A.2 — Example of a granule and powder sampling tool

Dimensions in millimetres

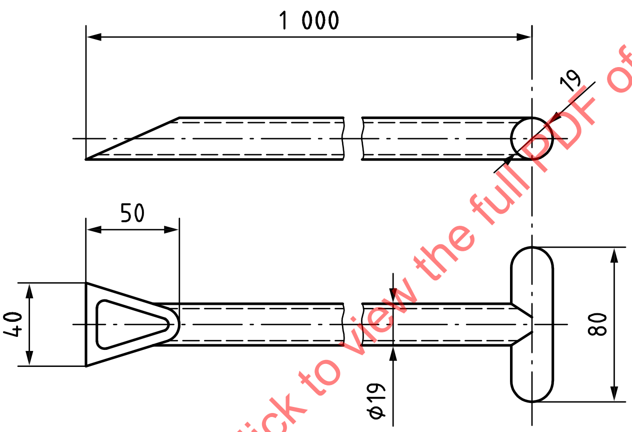
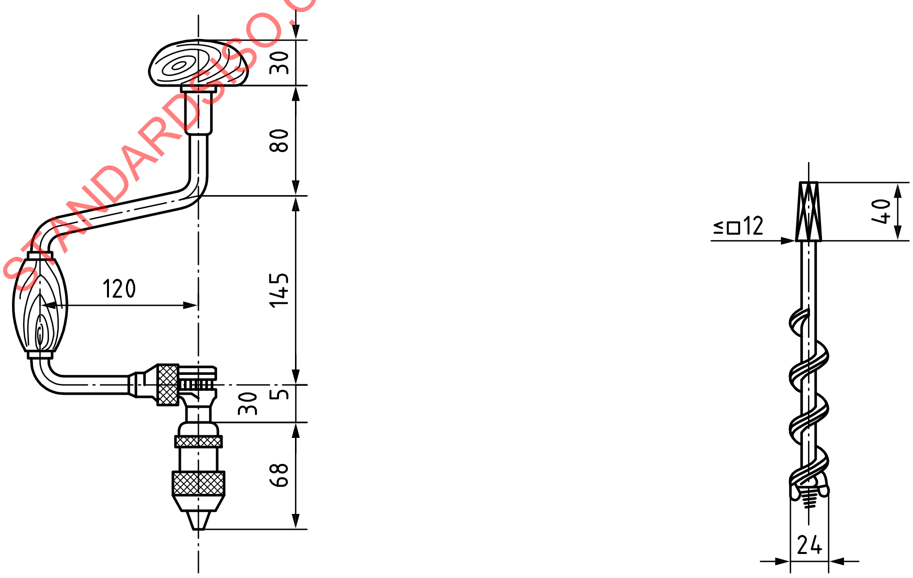


Figure A.3 — Example of a sampling scoop

Dimensions in millimetres



a) Brace with chuck **b) Bit**

Figure A.4 — Example of a tool for taking samples from solid materials

Annex B
(informative)

Examples of suitable drying apparatus

The following types of apparatus are used for drying purposes:

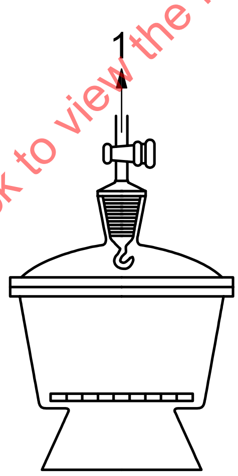
a) a vacuum desiccator, as shown in Figure B.1;

b) an apparatus for drying liquid samples by passing a dry gas through the boiling liquid, as shown in Figure B.2;

c) an apparatus for drying liquid samples by passing a dry gas through the liquid at ambient temperature, as shown in Figure B.3;

d) an apparatus for drying solid samples by passing a dry gas through the molten sample, as shown in Figure B.4;

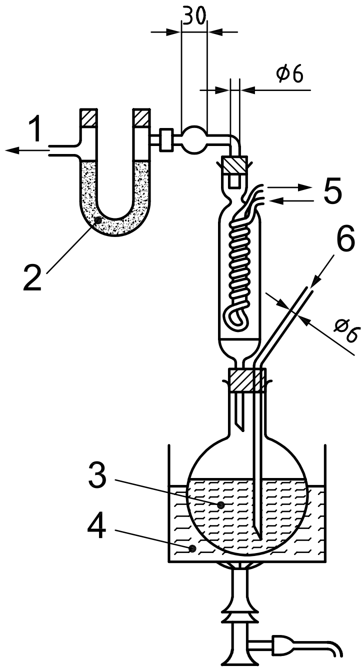
e) an apparatus for drying solid samples by passing a dry gas through the sample at ambient temperature, as shown in Figure B.5.



Key

1 to suction pump

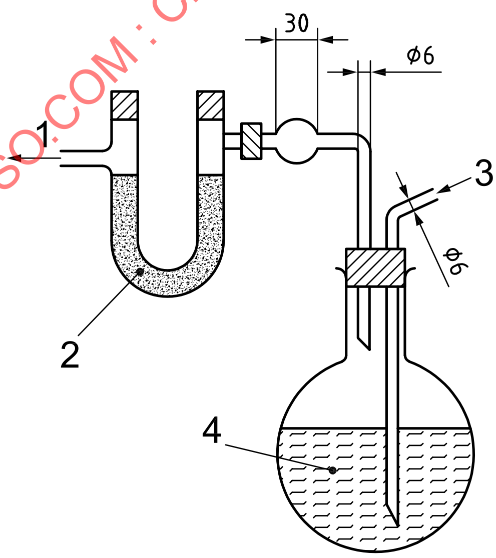
Figure B.1 — Example of a suitable vacuum desiccator



Key

1 to suction pump
2 desiccant
3 sample
4 sulfuric acid or oil bath
5 cooling water
6 dry gas

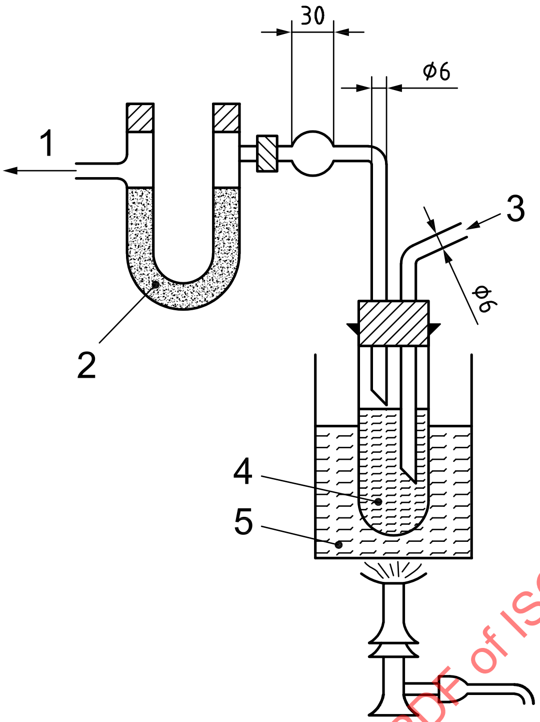
Figure B.2 — Example of a suitable apparatus for drying liquid samples by passing a dry gas through the boiling liquid



Key

1 to suction pump
2 desiccant
3 dry gas
4 sample

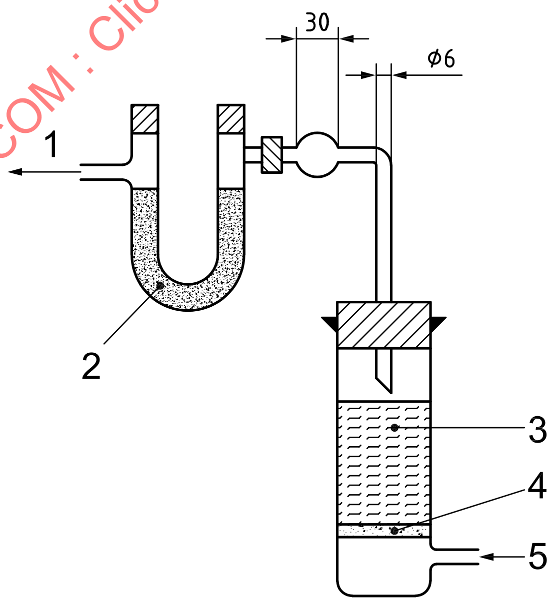
Figure B.3 — Example of a suitable apparatus for drying liquid samples by passing a dry gas through the liquid at ambient temperature



Key

1 to suction pump
2 desiccant
3 dry gas
4 sample
5 bath containing sulfuric acid, glycerol or water

Figure B.4 — Example of an apparatus for drying solid samples by passing a dry gas through the molten sample



Key

1 to suction pump
2 desiccant
3 sample
4 perforated plate
5 dry gas

Figure B.5 — Example of an apparatus for drying solid samples by passing a dry gas through the sample at ambient temperature

Annex C
(normative)

Verification of accuracy of pH-meter

C.1 General

pH-meter accuracy shall be verified by means of two tests, one to check the repeatability of the instrument and the other to check the linearity of the measurement scale. The result of each check shall conform to the requirements for the type of instrument being used.

C.2 Reagents

The analytical-grade buffer-solutions required are commercially available as standard solutions of known pH-value. If not, prepare them in accordance with the procedure specified in Clause C.3.

C.2.1 Water.

Use carbon-dioxide-free distilled water or water of equivalent purity (grade 3 as defined in ISO 3696:1987).

C.2.2 Potassium tetraoxalate, purity more than 99,8 %.

C.2.3 Potassium hydrogen phthalate, meeting the specification given in ISO 6353-3.

C.2.4 Potassium dihydrogen phosphate, meeting the specification given in ISO 6353-3.

C.2.5 Disodium hydrogen phosphate, purity more than 99,5 %.

C.2.6 Sodium tetraborate decahydrate, meeting the specification given in ISO 6353-3.

C.2.7 Sodium hydrogen carbonate, meeting the specification given in ISO 6353-3.

C.2.8 Sodium carbonate (anhydrous), meeting the specification given in ISO 6353-2.

C.3 Buffer solutions

C.3.1 Preparation

C.3.1.1 Phosphate buffer solution of nominal pH 7

Refer to 3.1 of ISO 976:1996.

C.3.1.2 Phthalate buffer solution of nominal pH 4

Refer to 3.2 of ISO 976:1996.

C.3.1.3 Borate buffer solution of nominal pH 9

Refer to 3.3 of ISO 976:1996.

C.3.1.4 Standard solution of pH less than 4

Dissolve 12,71 g of potassium tetraoxalate in water and make up to 1 l. The pH of this solution is 1,68 at 25 °C.

C.3.1.5 Standard solution of pH more than 9

Dissolve 2,10 g of sodium hydrogen carbonate and 2,65 g of sodium carbonate in water and dilute to 1 l. The pH of this solution is 10,02 at 25 °C.

C.3.2 pH-value at different temperatures

The pH-values at various temperatures of the standard pH solutions prepared in C.3.1 are given in Table C.1. pH-values at temperatures not given in Table C.1 shall be obtained by interpolation.

Table C.1 — pH-values of the standard buffer solutions at temperatures between 5 °C and 95 °C

Temperature	pH-value				
°C	Oxalate	Phthalate	Neutral phosphate	Borate	Carbonate [a]
0	1,67	4,01	6,98	9,46	10,32
5	1,67	4,01	6,95	9,39	(10,25)
10	1,67	4,00	6,92	9,33	10,18
15	1,67	4,00	6,90	9,27	(10,12)
20	1,68	4,00	6,88	9,22	(10,07)
25	1,68	4,01	6,86	9,18	10,02
30	1,69	4,01	6,85	9,14	(9,97)
35	1,69	4,02	6,84	9,10	(9,93)
38	—	—	—	—	9,91
40	1,70	4,03	6,84	9,07	—
45	1,70	4,04	6,83	9,04	—
50	1,71	4,06	6,83	9,01	—
55	1,72	4,08	6,84	8,99	—
60	1,73	4,10	6,84	8,96	—
70	1,74	4,12	6,85	8,93	—
80	1,77	4,16	6,86	8,89	—
90	1,80	4,20	6,88	8,85	—
95	1,81	4,23	6,89	8,83	

[a] The values in parentheses are interpolated values.

C.3.3 Storage

After preparation, standard buffer solutions shall be stored in air-tight hard-glass or polyethylene bottles. The pH-values of these solutions can change during a long storage period. After a long period of storage, therefore, a solution shall only be used after its pH has been confirmed to be correct by comparing it with a freshly prepared solution. For example, the pH-values of borate buffer solution and carbonate buffer solution are lowered by absorption of carbon dioxide from the air.

Standard buffer solutions which have been left exposed to the atmosphere shall not be used.

C.4 Apparatus

C.4.1 Thermometer, as specified in 4.1.

C.4.2 Conical flask, of capacity 200 cm^3.

C.4.3 Analytical balance, capable of weighing to the nearest 1 mg.

C.4.4 Funnel.

C.5 Verification

C.5.1 Cleaning the electrode

The electrode of the pH-meter shall be cleaned by washing with water not less than three times and wiping with, for instance, clean filter paper or absorbent cotton. However, when especially dirty, it shall be washed with, for instance, detergent or 0,1 mol/l hydrochloric acid, as required, for a short time and then rinsed well with running water. If a glass electrode has been kept in a dry condition for a long time, it shall be used only after first soaking it in water for 12 h.

C.5.2 Temperature

For repeatability and linearity checks, the temperature of standard solutions shall lie between 10 °C and 40 °C and remain stable within the limits given in Table C.2.

Table C.2 — Accuracy of measurement of temperature of buffer solutions and stability of temperature of solution during checks

<table>
<tr><th>Type of pH-meter
(see Table C.4)</th><th>Accuracy of measurement of temperature of buffer solution</th><th>Stability of temperature of buffer solution during calibration</th></tr>
<tr><td>I</td><td rowspan="3">± 0,5 °C</td><td>± 0,5 °C</td></tr>
<tr><td>II</td><td rowspan="2">± 2 °C</td></tr>
<tr><td>III</td></tr>
</table>

C.5.3 Repeatability check

Dip the electrode of the pH-meter, cleaned as described in C.5.1, into the chosen standard solution and the pH read after 10 min. Then, wash the electrode thoroughly with water, wipe it dry and again dip it into the same standard solution. After 10 min, read the pH indicated by the pH-meter. Measure the pH of the solution three times in this way. All three readings shall conform to the requirement specified in Table C.3 for the particular type of pH-meter.

Table C.3 — Type of pH-meter and required repeatability

Type	Required repeatability	Purpose
I	Repeatability better than ± 0,02 pH-units when used to measure the pH of the standard solution	Precise measurements
II	Repeatability better than ± 0,05 pH-units when used to measure the pH of the standard solution	Ordinary measurements
III	Repeatability better than ± 0,1 pH-units when used to measure the pH of the standard solution	Measurements not requiring any great accuracy

C.5.4 Linearity check

Dip the electrode of the pH-meter into neutral phosphate buffer solution (C.3.1.1), read the pH, and then wash the electrode thoroughly with water and wipe it dry. Next dip the electrode of the pH-meter into phthalate buffer solution (C.3.1.2), read the pH and again wash the electrode thoroughly with water and wipe it dry. Then dip it into borate buffer solution (C.3.1.3), read the pH and wash and dry the electrode as before. Repeat this series of measurements twice more (to give a total of three readings for each solution) and determine the average value for each solution. The difference between the average values and the true pH of each solution shall lie within the limits specified in Table C.4 for the particular type of pH-meter.

Table C.4 — Type of pH-meter and required accuracy in linearity check

Type	Accuracy requirement
I	± 0,03 or better
II	± 0,06 or better
III	± 0,1 or better