INTERNATIONAL STANDARD

ISO 28641

Second edition
2018-06

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.

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 on 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 the following URL: www.iso.org/iso/foreword.html

This document 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*.

This second edition cancels and replaces the first edition (ISO 28641:2010), which has been technically revised.

The main changes compared to the previous edition are as follows:

— updating of the normative references and bibliography;

— deletion of Clause 6 giving the procedure for drying samples, and deletion of all references to Clause 6 in the document;

— modification of the procedure regarding sieve residue in 7.3.5 c), 7.3.5 d), 7.3.5 h);

— in the test method for determination of the melting point (7.5), deletion of method B for water-insoluble and waxy samples; addition of a list of apparatus for the method for vaselines;

— in the test method for determination of the density, addition of a method B (constant volume method);

— deletion of Annex A giving examples of sampling apparatus and Annex B giving examples of suitable drying apparatus;

— addition of a new Annex D giving the conversion formulae between density and relative density.

Rubber compounding ingredients — Organic chemicals — General test methods

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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.

1 Scope

This document 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 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 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 976:2013, *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, *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 — Abbreviated terms*

ISO 11235:2016, *Rubber compounding ingredients — Sulfenamide accelerators — Test methods*

ISO 11236:2017, *Rubber compounding ingredients — p-Phenylenediamine antidegradants (PPDs) — 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 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Abbreviated terms

The abbreviated terms for the chemical names of the organic accelerators, antidegradants and vulcanizing agents used in this document are in accordance with ISO 6472.

5 General requirements

5.1 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.

5.2 Desiccator

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

6 Sampling

6.1 Apparatus

The apparatus used for sampling shall be suitable for each test method.

6.2 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.

7 Test methods

7.1 Density and 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 suitable transparent glass, graduated in either density or 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 and the instrumental error shall be known.

NOTE   See Annex D for information about how to correct instrumental errors and how to convert density into relative density or vice versa.

7.1.2.1.2 Thermometer, as specified in 5.1.

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 (as specified in 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.
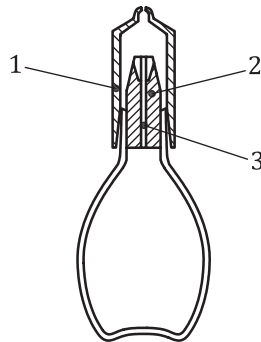
Key
1   cap
2   plug
3   capillary

Figure 1 — Warden pyknometer

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

7.1.3.2.3 Thermometer, as specified in 5.1.

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 $(20 \pm 0,5)$ °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 of 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 $(20 \pm 0,5)$ °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 Formula (1):

$$d = \frac{m_2 - m_0}{m_1 - m_0} \tag{1}$$

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

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

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, brought to a temperature of approximately 20 °C and put it into the pyknometer. Immerse the pyknometer 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.

7.1.3.4.2 Calculation

Calculate the relative density of the powder sample using Formula (2):

$$d = \frac{m_2 - m_0}{m_2 + m_1 - m_0 - m_3} \times D \tag{2}$$

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 or the liquid used, 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 or the liquid used, in grams;

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

7.1.4 Expression of results

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

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 document, i.e. ISO 28641;

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 document 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 8 of ISO 11235:2016 and in Clause 10 of ISO 11236:2017.

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_0).

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_1).

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 and vulcanizing agents) 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_2).

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

Table 1 — Heating temperature and heating time (accelerators and vulcanizing agents)

Accelerator and vulcanizing agent	Temperature °C	Time h	Accelerator	Temperature °C	Time h
BA			CMBT[a]		
DPG			CBS		
DOTG			TBBS		
MBT			DCBS		
MBTS			TETD		
ZMBT	100 ± 2	2	DPTH	55 ± 2	3
ZDMC			DPTT		
ZDEC			ZDBC		
CuDMC			TeDEC		
ZEPC			DETU		
FeDMC[b]			DBTU		
ETU			TMU[c]		
MBSS			DIBS	50 ± 2	3
TMTM					
TMTD	80 ± 2	2			
BQD (vulcanizing agent)					
DBQD (vulcanizing agent)					
[a] Salt of 2-mercaptobenzothiazole.					
[b] Ferric dimethyldithiocarbamate.					
[c] Trimethylthiourea.					

Table 2 — Heating temperature and heating time (antidegradants)

Antidegradant	Temperature °C	Time h	Antidegradant	Temperature °C	Time h
ADPA			ETMQ		
SPH			AANA	75 ± 2	2
MBI			PAN		
ZMBI			ODPA		
o-MBp24	100 ± 2	2	SDPA	70 ± 2	2
p-BBp14			DCD[a]		
p-TBp14			TMQ		
DBHQ			NDBC	50 ± 2	3
DAHQ			MBMTB		
[a] 4,4′-dicumyldiphenylamine.					

7.2.4 Calculation

Calculate the loss on heating using Formula (3):

$$w_v = \frac{m_1 - m_2}{m_1 - m_0} \times 100 \tag{3}$$

where

w_V is the loss on heating, in percent;

m_0 is the mass of the weighing bottle and stopper, in grams;

m_1 is the mass of the weighing bottle, stopper and test portion before heating, in grams;

m_2 is the mass of the weighing bottle, stopper and test portion after heating, in grams.

7.2.5 Expression of results

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

7.2.6 Precision

See C.1 in Annex C, Loss on heating — Method B .

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 document, i.e. ISO 28641;

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 document 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 original condition of 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 with 25 ml water and agitate with a glass rod. When it is difficult to make the portion completely wet, surfactant may be added up to 1 % volume of the water.

d) Add about 50 cm^3 of water and mix thoroughly. Decant the particles floating on the liquid onto the sieve.

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 water.

i) Dry the sieve in the oven maintained at the temperature specified in Table 1 (for accelerators and vulcanizing agents) 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 Formula (4):

$$w_S = \frac{m_1}{m_0} \times 100 \tag{4}$$

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, to one decimal place, in accordance with B.2 of ISO 80000-1:2009.

7.3.8 Precision

See C.2 in Annex C, Sieve residue.

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 document, i.e. ISO 28641;

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 document 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 A.3 and Table A.4, depending on the purpose of the test.

7.4.2.2 Thermometer, as specified in 5.1.

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 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 B, 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 A.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 (as specified in Annex A).

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 document, i.e. ISO 28641;

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 (as specified in Annex A);

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 document 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 vaselines): visually by heating a test sample in a capillary in a heated liquid;

—   Method C: by using a differential scanning calorimeter.

7.5.2   Method A

Method A is the method specified in 7.1 of ISO 11235:2016.

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 5.1.

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)   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 into 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.
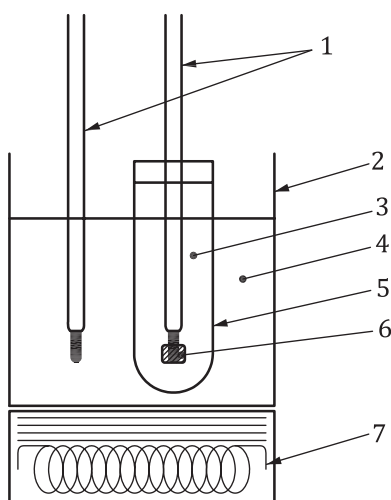
Key

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

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

7.5.4   Method C

Method C is that specified in 7.2 of ISO 11235:2016.

7.5.5   Expression of results

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

7.5.6   Precision

See C.3 in Annex C, Melting point — Method A.

7.5.7   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 document, i.e. ISO 28641;

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

d)   the laboratory temperature and humidity;

e)   the test result;

f)   any operation not included in this document 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 3.

Dimensions in millimetres
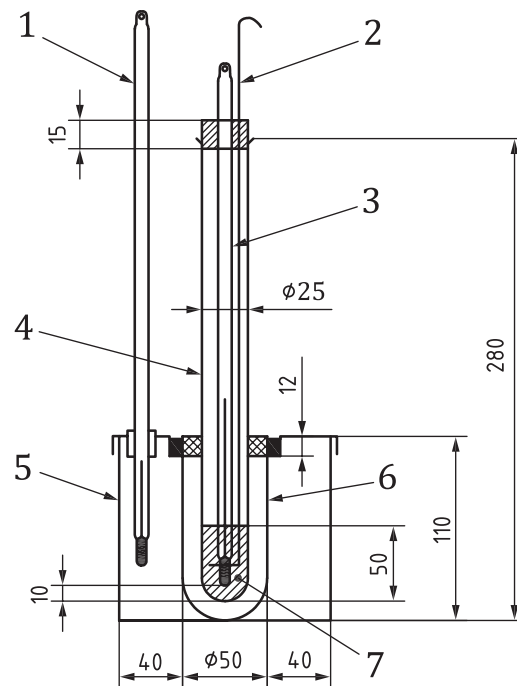
Key

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 3 — 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 into 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 5.1.

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 4 a), but will follow one such as those shown in Figures 4 b), c) and d). In the case of Figures 4 b) and d), extrapolate the lines for the solid and liquid phases and take their point of intersection as the temperature of solidification.

a)

b)

c)

d)
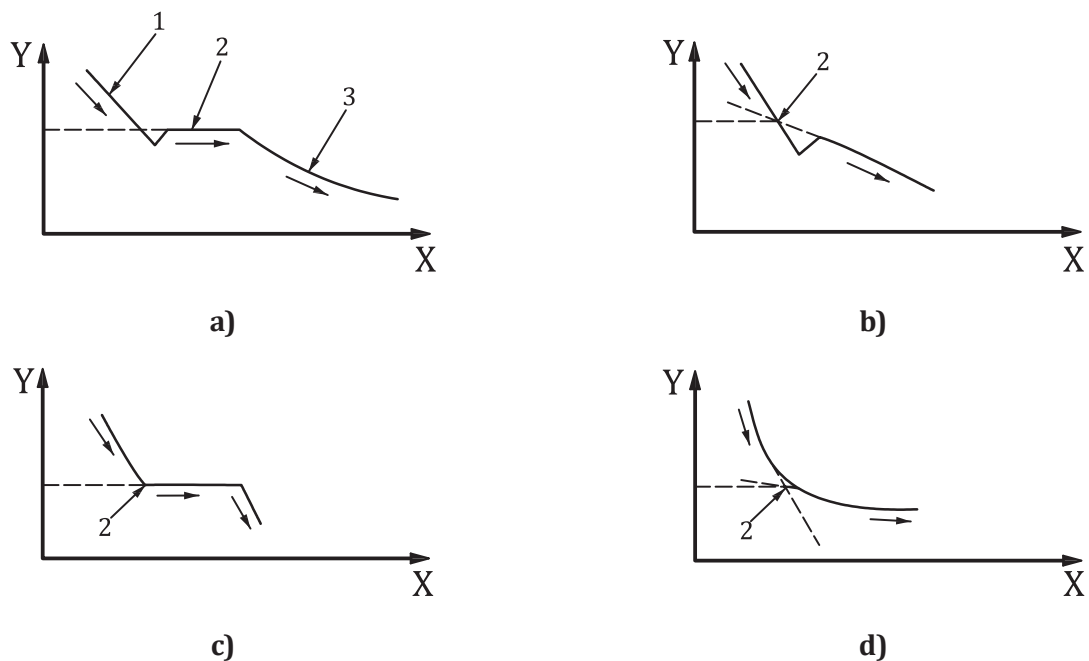
Key

X   time

Y   temperature

1   liquid

2   temperature of solidification

3   solid

Figure 4 — Solidification temperature curves

7.6.4   Expression of results

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

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 document, i.e. ISO 28641;

c)   the laboratory temperature and humidity;

d)   the test result;

e)   any operation not included in this document 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 for which there is 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 5 and specified in ISO 4625-1.

It is recommended that a perforated bottom plate be included as shown in Figure 5 d) in order to assist in homogenizing the temperature of the bath.

7.7.3.2   Thermometer, as specified in 5.1.

Dimensions in millimetres

a) Shouldered ring

b) Ball-centering guide

c) Ring holder

d) Assembled apparatus
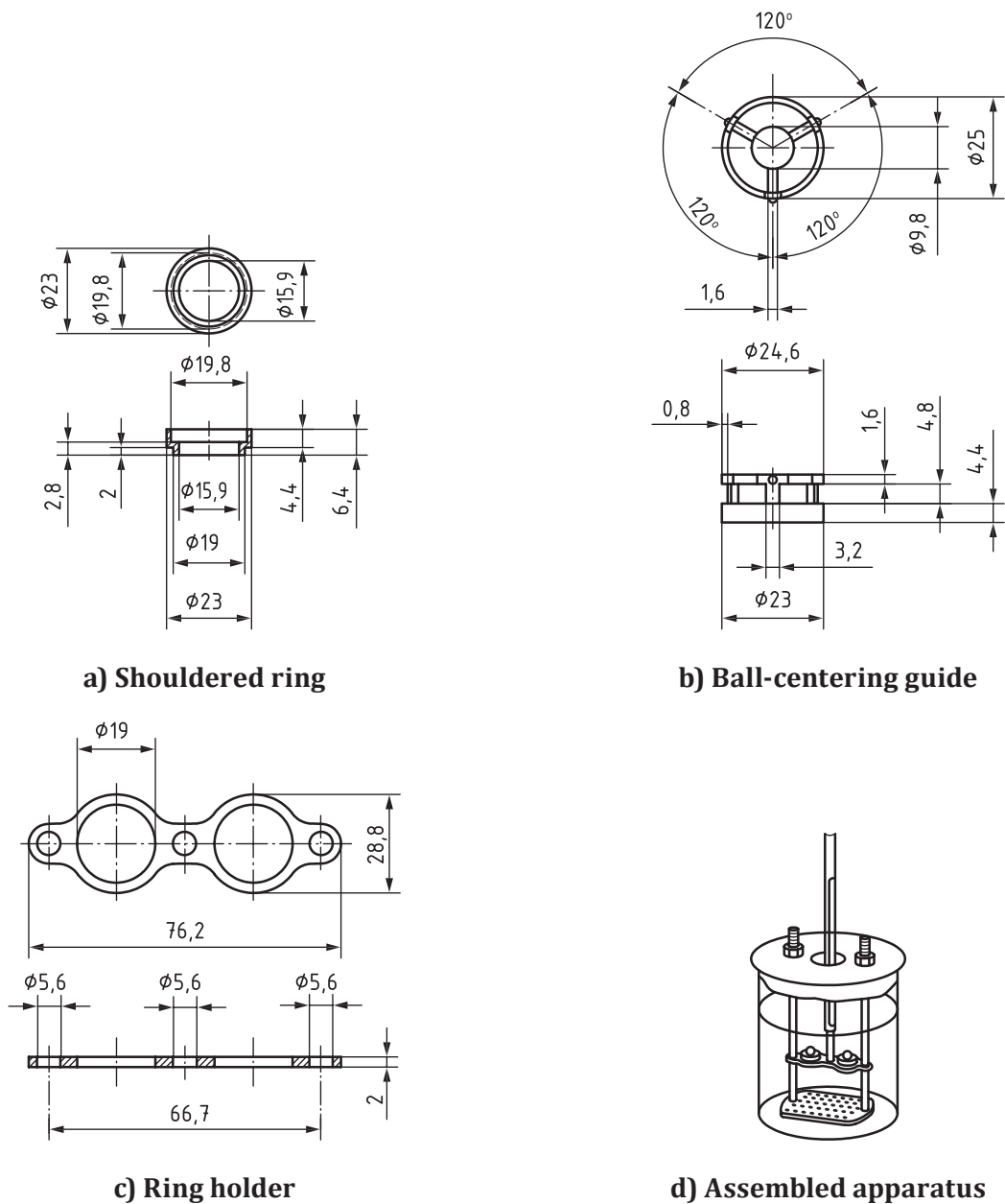
Figure 5 — Softening point measurement apparatus (example)

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

7.7.6   Precision

See C.4 in Annex C, Softening point.

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 document, i.e. ISO 28641;

c)   the laboratory temperature and humidity;

d)   the test result;

e)   any operation not included in this document 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

Bulk density is the mass of powder per unit volume. There are two methods to measure bulk density of rubber compounding ingredient samples.

— Method A (constant mass method): The bulk density is measured with a specified load applied to a test sample.

— Method B (constant volume method): Fill up a test sample to a specified volume without any pressure loaded and calculate the bulk density from the measured mass.

7.8.2   Method A (constant mass method)

7.8.2.1   Apparatus

The apparatus used for method A shall consist of a cylinder and a piston, made of ordinary steel, as shown in Figure 6. 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

$\phi 21,8 \pm 0,05$

115

$\phi 22 \pm 0,05$

100
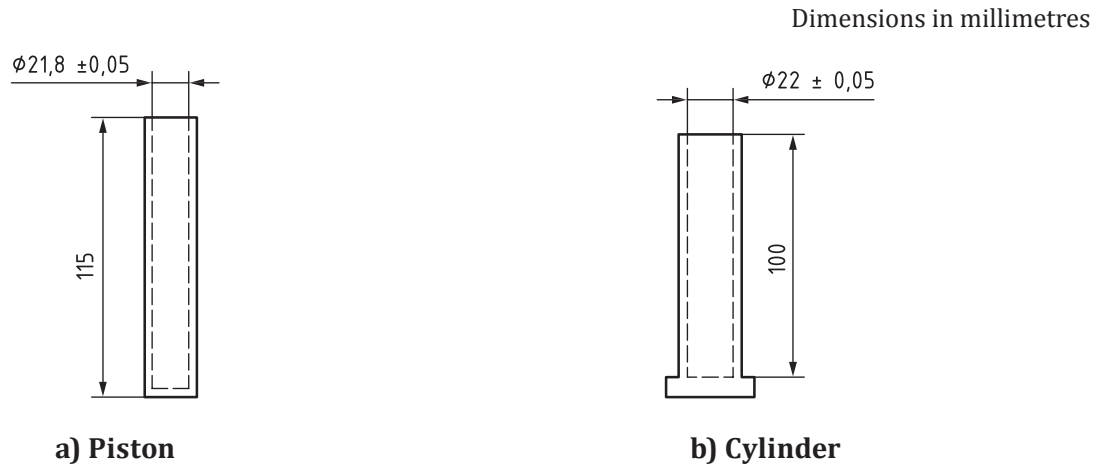
a) Piston   **b) Cylinder**

Figure 6 — Bulk density measurement apparatus

7.8.2.2 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.

7.8.2.3 Calculation

Calculate the density using Formula (5):

$$\rho = \frac{m_0}{0,785\,4\,d^2 \times \left(h_2 - h_1\right)} \tag{5}$$

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.

7.8.2.4   Expression of results

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

7.8.3   Method B (constant volume method)

7.8.3.1   Apparatus

7.8.3.1.1   Container, of 30 cm^3 to 100 cm^3, with a smooth inner surface.

7.8.3.1.2   Sieve, as specified in ISO 3310-1, of an appropriate mesh size which allows all the sample to be passed.

7.8.3.1.3   Levelling tool or **straight scraper.**

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

7.8.3.2   Procedure

a)   Weigh the container (7.8.3.1.1) to the nearest 0,1 mg.

b)   Crush any lumps in the bulk with fingers. Sift it with a sieve (7.8.3.1.2) at a constant height above the container (between 10 cm to 20 cm) to allow the sample to overflow the container. Avoid any extraneous force or pressure to the container or to the sample surface during the procedure.

c)   Remove extra test sample with a levelling tool or a straight scraper (7.8.3.1.3) so that the container is full but there is no sample above the level of the container. Keep a blunt angle while scraping the surface so that no excessive pressure is applied.

d)   Weigh the container full of the test sample to the nearest 0,1 mg.

e)   Repeat the procedure a) to d). Calculate the average of the two determinations and round the value to one decimal place.

7.8.3.3   Calculation

Calculate the bulk density of the sample using Formula (6):

$$G = \frac{m_1 - m_0}{V}$$

(6)

where

G is the bulk density, in g/cm^3;

m_0 is the weight of the container, in g;

m_1 is the rounded average weight of the container with the test sample, in g;

V is the capacity of the container, in cm^3.

7.8.3.4   Expression of results

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

7.8.4   Test report

The test report shall include the following information:

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

b)   a reference to the test method used, i.e. method A or method B, of ISO 28641;

c)   the mass of the test portion;

d)   the laboratory temperature and humidity;

e)   the test result;

f)   any operation not included in this document 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.

7.9.2.3   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 Formula (7):

$$w_A = \frac{m_1}{m_0} \times 100 \tag{7}$$

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, to two decimal places, in accordance with B.2 of ISO 80000-1:2009.

7.9.6 Precision

See C.5 in Annex C, Ash.

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 document, i.e. ISO 28641;

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 document 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 7.

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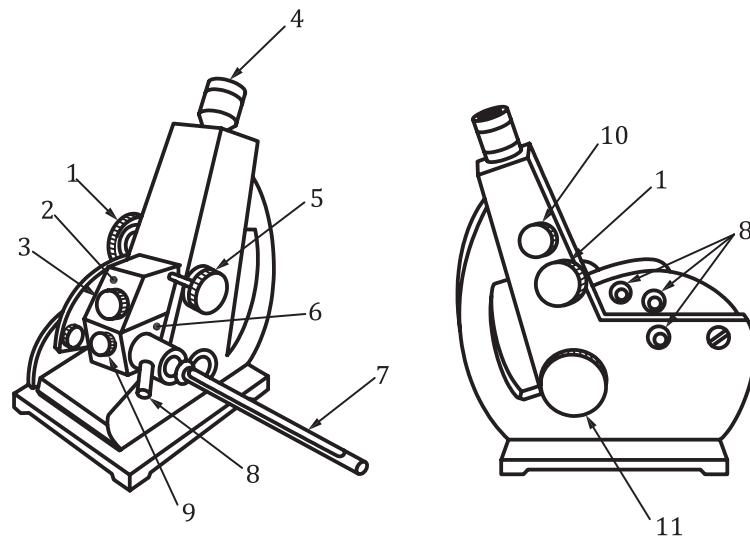
Key

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

Figure 7 — 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 as described in Figure 8.

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 as described in Figure 9.

e) Make three measurements and take the average of the results.
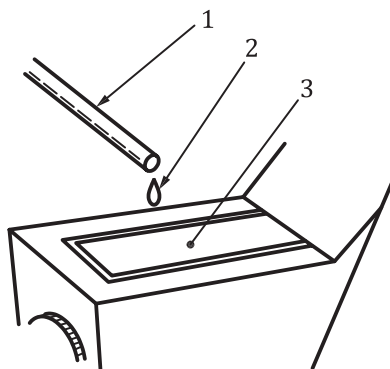
Key
1   glass rod
2   sample
3   surface of main prism

Figure 8 — Method of wetting the surface of the main prism
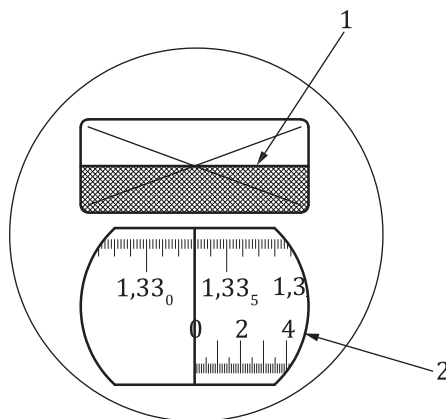
Key
1   dividing line between light and dark areas
2   n_D scale

Figure 9 — Method of reading the n_D scale

7.10.4 Expression of results

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

7.10.5 Precision

See C.6 in Annex C, Refractive index.

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 document, i.e. ISO 28641;

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 document as well as any unusual features noted during the determination;

g) the date of the test.

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Annex A
## (normative)

Verification of accuracy of pH-meter

A.1 General

The accuracy of the pH-meter 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.

A.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 A.3.

A.2.1 Water.

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

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

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

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

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

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

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

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

A.3 Buffer solutions

A.3.1 Preparation

A.3.1.1 Phosphate buffer solution of nominal pH 7

Refer to 3.1 of ISO 976:2013.

A.3.1.2 Phthalate buffer solution of nominal pH 4

Refer to 3.2 of ISO 976:2013.

A.3.1.3 Borate buffer solution of nominal pH 9

Refer to 3.3 of ISO 976:2013.

A.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.

A.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.

A.3.2   pH-value at different temperatures

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

Table A.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.					

A.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. For example, the pH-values of borate buffer solution and carbonate buffer solution are lowered by absorption of carbon dioxide from the air. 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.

A.4   Apparatus

A.4.1   **Thermometer**, as specified in 5.1.

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

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

A.4.4   Funnel.

A.5   Verification

A.5.1   Cleaning the electrode

The electrode of the pH-meter shall be cleaned by washing with water not less than three times. 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.

A.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 A.2.

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

Type of pH-meter (see Table A.4)	Accuracy of measurement of temperature of buffer solution	Stability of temperature of buffer solution during calibration
I		±0,5 °C
II	±0,5 °C	±2 °C
III		

A.5.3   Repeatability check

Dip the electrode of the pH-meter, cleaned as described in A.5.1, into the chosen standard solution and read the pH after 10 min. Then wash the electrode thoroughly with water 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 A.3 for the particular type of pH-meter.

Table A.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

A.5.4   Linearity check

Dip the electrode of the pH-meter into neutral phosphate buffer solution (A.3.1.1), read the pH, and then wash the electrode thoroughly with water. Next dip the electrode of the pH-meter into phthalate buffer solution (A.3.1.2), read the pH and again wash the electrode thoroughly with water. Then dip it into borate buffer solution (A.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 A.4 for the particular type of pH-meter.

Table A.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

Annex B
(normative)

Calibration of the pH-meter

B.1 General

The pH-meter is calibrated, using buffer solutions, both at pH 7 and over the part of the measurement range in which the instrument will be used.

B.2 Buffer solutions

Use the solutions specified in A.3.1.

B.3 Apparatus

Use the apparatus specified in Clause A.4.

B.4 Calibration

B.4.1 General

The pH-meter shall be calibrated, both at pH 7 and over the part of the measurement range in which the instrument will be used, until the pH-values obtained are in accordance with those given in Table A.1, with the repeatability given in Table A.3 and the accuracy given in Table A.4 for the type of pH-meter being used.

B.4.2 Cleaning the electrode

Clean the electrode in the way described in A.5.1.

B.4.3 Temperature

The temperature at which calibration is carried out shall be as specified in A.5.2.

B.4.4 Calibration at pH 7

Dip the electrode into neutral phosphate buffer solution (A.3.1.1) and adjust the zero control to the appropriate value given in Table A.1 for the temperature of the solution. In the case of pH-meters fitted with a temperature-compensation control, set the scale value to that given in Table A.1 for the temperature of the solution.

B.4.5 Calibration over the anticipated measurement range

Carry out the calibration as follows:

a) If the pH of the solution whose pH is to be measured is less than 7, calibrate the pH-meter by dipping the electrode into phthalate buffer solution (A.3.1.2) or tetraoxalate buffer solution (A.3.1.4) and adjusting the control dial to the appropriate value given in Table A.1 for the temperature of the solution.

b) If the pH of the solution whose pH is to be measured is 7 or greater, but less than 11, dip the electrode into phosphate buffer solution (A.3.1.1), borate buffer solution (A.3.1.3) or carbonate buffer solution (A.3.1.5), proceeding in the same way as in B.4.5 a).

c) If the pH of the solution whose pH is to be measured is 11 or higher, 0,1 mol/l sodium hydroxide solution (not containing carbonate) or saturated (at 25 °C) calcium hydroxide solution may be used for the calibration. The pH of these solutions at various temperatures is given in Table B.1.

Table B.1 — pH-values of 0,1 mol/l sodium hydroxide solution and saturated calcium hydroxide solution at temperatures between 0 °C and 60 °C

Temperature °C	pH of 0,1 mol/l sodium hydroxide solution	pH of saturated calcium hydroxide solution
0	13,8	13,43
5	13,6	13,21
10	13,4	13,00
15	13,2	12,81
20	13,1	12,63
25	12,9	12,45
30	12,7	12,30
35	12,6	12,14
40	12,4	11,99
45	12,3	11,84
50	12,2	11,70
55	12,0	11,58
60	11,9	11,45

Annex C
(informative)

Precision

C.1 Loss on heating — Method B

For the determination of precision data, three laboratories (for accelerators and vulcanizing agents) and six laboratories (for antidegradants) participated in an interlaboratory test programme using 10 different accelerators/vulcanizing agents and nine different antidegradants. The loss on heating was measured by method B. The number of within-laboratory replicates was two and the time span for repeatability was seven days. The precision calculations to express the repeatability and reproducibility were performed in accordance with ISO/TR 9272. The precision of the method for accelerators and vulcanizing agents is given in Table C.1 and for antidegradants in Table C.2.

Table C.1 — Precision data for loss on heating of accelerators and vulcanizing agents (by method B)

Accelerator and vulcanizing agent	Test conditions (temperature and time)	Mean value %	Within-laboratory s_r	r	Between-laboratory s_R	R
DPG	100 °C, 2 h	0,12	0,036	0,101	0,077	0,219
ETU	80 °C, 2 h	0,33	0,329	0,932	0,277	0,784
MBT	100 °C, 2 h	0,11	0,015	0,041	0,080	0,227
MBTS	100 °C, 2 h	0,08	0,017	0,048	0,057	0,162
TBBS	55 °C, 3 h	0,07	0,017	0,048	0,040	0,114
DCBS	55 °C, 3 h	0,03	0,022	0,061	0,030	0,086
TMTD	80 °C, 2 h	0,04	0,010	0,029	0,051	0,145
ZDMC	100 °C, 2 h	0,05	0,012	0,035	0,055	0,155
ZDEC	100 °C, 2 h	0,09	0,021	0,059	0,066	0,185
BQD (vulcanizing agent)	80 °C, 2 h	0,12	0,035	0,099	0,076	0,216

$p = 3$, $q = 2$, $n = 2$

s_r: Within-laboratory standard deviation.

r: Repeatability, in measurement units.

s_R: Between-laboratory standard deviation.

R: Reproducibility, in measurement units.

Table C.2 — Precision data for loss on heating of antidegradants (by method B)

Antidegradant	Test conditions (temperature and time)	Mean value %	Within-laboratory		Between-laboratory	
			s_r	r	s_R	R
6PPD	70 °C, 3 h	0,03	0,023	0,066	0,032	0,09
TMQ	50 °C, 3 h	0,03	0,019	0,054	0,000	0,07
PAN	75 °C, 2 h	0,01	0,004	0,012	0,013	0,04
ODPA	70 °C, 2 h	0,00	0,005	0,016	0,007	0,02
SP	100 °C, 2 h	0,30	0,032	0,089	0,212	0,60
MBMTB	50 °C, 3 h	0,01	0,006	0,016	0,016	0,04
DAHQ	100 °C, 2 h	0,06	0,022	0,062	0,059	0,17
ZMBI	100 °C, 2 h	0,17	0,026	0,074	0,064	0,18
NDBC	50 °C, 3 h	0,00	0,003	0,009	0,005	0,01

$p = 6, q = 2, n = 2$

s_r: Within-laboratory standard deviation.

r: Repeatability, in measurement units.

s_R: Between-laboratory standard deviation.

R: Reproducibility, in measurement units.

C.2 Sieve residue

For the determination of precision data, three laboratories participated in an interlaboratory test programme, using 10 different accelerators/vulcanizing agents. The sieve residue was measured by the wet procedure. The number of within-laboratory replicates was two and the time span for repeatability was seven days. The precision calculations to express the repeatability and reproducibility were performed in accordance with ISO/TR 9272. The precision for the residue on sieve is given in Tables C.3 and C.4. The precision shows satisfactory levels when the method is used for non-powder materials such as TBBS and DCBS.

Table C.3 — Precision data for sieve residue by wet procedure (75 µm sieve)

Accelerator and vulcanizing agent	Mean value %	Within-laboratory		Between-laboratory	
		s_r	r	s_R	R
DPG	0,02	0,029	0,081	0,033	0,092
ETU	0,02	0,000	0,000	0,028	0,078
MBT	0,03	0,010	0,029	0,048	0,136
MBTS	0,03	0,022	0,064	0,025	0,070
TBBS[a]	28,85	1,506	4,262	3,471	9,824
DCBS[a]	18,22	0,667	1,887	1,107	3,133
TMTD	0,04	0,004	0,012	0,045	0,126

$p = 3, q = 2, n = 2$

s_r: Within-laboratory standard deviation.

r: Repeatability, in measurement units.

s_R: Between-laboratory standard deviation.

R: Reproducibility, in measurement units.

[a] The TBBS and DCBS samples used were not in powder form.

Table C.3 *(continued)*

Accelerator and vulcanizing agent	Mean value	Within-laboratory		Between-laboratory	
	%	s_r	r	s_R	R
ZDMC	0,02	0,006	0,017	0,006	0,016
ZDEC	0,01	0,002	0,006	0,011	0,031
BQD (vulcanizing agent)	0,01	0,002	0,006	0,008	0,022
$p = 3, q = 2, n = 2$					
s_r: Within-laboratory standard deviation.					
r: Repeatability, in measurement units.					
s_R: Between-laboratory standard deviation.					
R: Reproducibility, in measurement units.					
[a] The TBBS and DCBS samples used were not in powder form.					

Table C.4 — Precision data for sieve residue by wet procedure (150 μm sieve)

Accelerator and vulcanizing agent	Mean value	Within-laboratory		Between-laboratory	
	%	s_r	r	s_R	R
DPG	0,01	0,002	0,006	0,013	0,037
ETU	0,01	0,001	0,003	0,009	0,027
MBT	0,00	0,001	0,003	0,001	0,003
MBTS	0,00	0,001	0,003	0,004	0,013
TBBS[a]	13,61	0,319	0,904	6,172	17,468
DCBS[a]	0,72	0,066	0,188	0,218	0,617
TMTD	0,03	0,005	0,013	0,050	0,142
ZDMC	0,02	0,025	0,070	0,033	0,092
ZDEC	0,02	0,024	0,069	0,036	0,102
BQD (vulcanizing agent)	0,02	0,018	0,052	0,036	0,101
$p = 3, q = 2, n = 2$					
s_r: Within-laboratory standard deviation.					
r: Repeatability, in measurement units.					
s_R: Between-laboratory standard deviation.					
R: Reproducibility, in measurement units.					
[a] The TBBS and DCBS samples used were not in powder form.					

C.3 Melting point — Method A

For the determination of precision data, three laboratories (for organic rubber accelerators and vulcanizing agents) and four laboratories (for antidegradants) participated in an interlaboratory test programme, using 10 different organic rubber accelerators/vulcanizing agents and six different antidegradants. The melting point was measured by method A. The number of within-laboratory replicates was two and the time span for repeatability was seven days. The precision calculations to express the repeatability and reproducibility were performed in accordance with ISO/TR 9272. The precision of the method for organic rubber accelerators and vulcanizing agents is given in Table C.5 and for antidegradants in Table C.6.

Table C.5 — Precision data for melting point of organic rubber accelerators and vulcanizing agents (by method A)

Accelerator and vulcan-izing agent	Mean value	Within-laboratory			Between-laboratory		
	°C	s_r	r	(r)	s_R	R	(R)
DPG	145,6	0,10	0,29	0,2	1,97	5,59	3,8
ETU	196,0	0,22	0,61	0,3	2,90	8,20	4,2
MBT	176,2	0,17	0,47	0,3	2,22	6,27	3,6
MBTS	172,3	0,08	0,23	0,1	0,69	1,96	1,1
TBBS	106,1	0,10	0,28	0,3	1,70	4,81	4,5
DCBS	102,7	0,05	0,14	0,1	0,52	1,46	1,4
TMTD	144,1	2,08	5,89	4,1	3,34	9,44	6,6
ZDMC	251,2	0,62	1,77	0,7	2,08	5,89	2,3
ZDEC	178,2	0,26	0,75	0,4	1,81	5,13	2,9
BQD (vulcan-izing agent)	221,4	0,58	1,63	0,7	9,56	27,05	12,2
$p = 3$, $q = 2$, $n = 2$							
s_r: Within-laboratory standard deviation.							
r: Repeatability, in measurement units.							
(r): Repeatability, in percent (these values represent percent relative, i.e. percent of a percent)							
s_R: Between-laboratory standard deviation.							
R: Reproducibility, in measurement units.							
(R): Reproducibility, in percent (these values represent percent relative, i.e. percent of a percent).							

Table C.6 — Precision data for melting point of antidegradants (by method A)

Antidegradant	Mean value	Within-laboratory			Between-laboratory		
	°C	s_r	r	(r)	s_R	R	(R)
6PPD	48,9	0,30	0,86	1,8	0,37	1,05	2,1
PAN	59,2	0,33	0,92	1,6	0,74	2,09	3,5
ODPA	93,5	0,06	0,17	0,2	1,21	3,43	3,7
MBMTB	130,0	0,06	0,17	0,1	0,39	1,10	0,8
DAHQ	181,8	0,28	0,80	0,4	0,45	1,26	0,7
NDBC	88,0	0,04	0,12	0,1	0,74	2,11	2,4
$p = 4$, $q = 2$, $n = 2$							
s_r: Within-laboratory standard deviation.							
r: Repeatability, in measurement units.							
(r): Repeatability, in percent (these values represent percent relative, i.e. percent of a percent)							
s_R: Between-laboratory standard deviation.							
R: Reproducibility, in measurement units.							
(R): Reproducibility, in percent (these values represent percent relative, i.e. percent of a percent).							

C.4   Softening point

For the determination of precision data, three laboratories participated in an interlaboratory test programme using one antidegradant (TMQ). The number of within-laboratory replicates was two and the time span for repeatability was seven days. The precision calculations to express the repeatability

and reproducibility were performed in accordance with ISO/TR 9272. The precision for the softening point is given in Table C.7.

Table C.7 — Precision data for softening point of antidegradant TMQ

Antidegradant	Mean value	Within-laboratory			Between-laboratory		
	°C	s_r	r	(r)	s_R	R	(R)
TMQ	97,6	0,16	0,47	0,48	1,271	3,60	3,68
$p = 3$, $q = 2$, $n = 2$							
s_r: Within-laboratory standard deviation.							
r: Repeatability, in measurement units.							
(r): Repeatability, in percent (these values represent percent relative, i.e. percent of a percent)							
s_R: Between-laboratory standard deviation.							
R: Reproducibility, in measurement units.							
(R): Reproducibility, in percent (these values represent percent relative, i.e. percent of a percent).							

C.5   Ash

For the determination of precision data, three laboratories (for accelerators and vulcanizing agents) and six laboratories (for antidegradants) participated in an interlaboratory test programme, using 10 different accelerators/vulcanizing agents and nine different antidegradants. The number of within-laboratory replicates was two and the time span for repeatability was seven days. The precision calculations to express the repeatability and reproducibility were performed in accordance with ISO/TR 9272. The precision of the ash determination method is given in Tables C.8 and C.9. The precision shows satisfactory levels when the method is applied to materials containing metals, such as ZDMC, ZDEC, ZMBI and NDBC.

Table C.8 — Precision data for ash of accelerators and vulcanizing agents

Accelerator and vulcanizing agent	Mass of test portion	Mean value	Within-laboratory		Between-laboratory	
	g	%	s_r	r	s_R	R
DPG	3	0,04	0,023	0,065	0,029	0,083
ETU	3	0,01	0,002	0,006	0,005	0,015
MBT	3	0,04	0,025	0,070	0,032	0,089
MBTS	3	0,03	0,018	0,050	0,014	0,040
TBBS	3	0,03	0,012	0,035	0,023	0,065
DCBS	3	0,06	0,029	0,083	0,024	0,069
TMTD	3	0,02	0,016	0,046	0,016	0,046
ZDMC	3	25,16	0,110	0,312	0,364	1,029
ZDEC	3	20,89	0,212	0,599	0,487	1,378
BQD (vulcanizing agent)	3	0,02	0,015	0,042	0,011	0,031
$p = 3$, $q = 2$, $n = 2$.						
s_r: Within-laboratory standard deviation.						
r: Repeatability, in measurement units.						
s_R: Between-laboratory standard deviation.						
R: Reproducibility, in measurement units.						

Table C.9 — Precision data for ash of antidegradants

Antidegradant	Mass of test portion	Mean value	Within-laboratory		Between-laboratory	
	g	%	s_r	r	s_R	R
6PPD	3	0,01	0,002	0,007	0,014	0,039
TMQ	3	0,02	0,007	0,018	0,000	0,058
PAN	3	0,02	0,012	0,035	0,020	0,057
ODPA	3	0,02	0,010	0,028	0,024	0,067
SP	3	0,01	0,016	0,046	0,018	0,051
MBMTB	3	0,01	0,007	0,021	0,012	0,034
DAHQ	3	0,01	0,002	0,007	0,007	0,021
ZMBI	3	22,66	0,064	0,181	0,236	0,667
NDBC	1	14,62	0,120	0,339	1,478	4,183

$p = 6$, $q = 2$, $n = 2$

s_r: Within-laboratory standard deviation.

r: Repeatability, in measurement units.

s_R: Between-laboratory standard deviation.

R: Reproducibility, in measurement units.

C.6   Refractive index

For the determination of precision data, four laboratories participated in an interlaboratory test programme using one antidegradant. The number of within-laboratory replicates was two and the time span for repeatability was seven days. The precision calculations to express the repeatability and reproducibility were performed in accordance with ISO/TR 9272. The precision for refractive index is given in Table C.10.

Table C.10 — Precision data for refractive index of antidegradant SP

Antidegradant	Mean value	Within-laboratory			Between-laboratory		
		s_r	r	(r)	s_R	R	(R)
SP	1,602 0	0,000 2	0,000 6	0,04	0,000 8	0,002 2	0,14

$p = 4$, $q = 2$, $n = 2$

s_r: Within-laboratory standard deviation.

r: Repeatability, in measurement units.

(r): Repeatability, in percent (these values represent percent relative, i.e. percent of a percent)

s_R: Between-laboratory standard deviation.

R: Reproducibility, in measurement units.

(R): Reproducibility, in percent (these values represent percent relative, i.e. percent of a percent).

Annex D
(informative)

Conversion formulae between density and relative density

D.1 Relative-density hydrometer (graduated in 15/4 °C)

When a relative-density hydrometer which is graduated in 15/4 °C is used, the density (20 °C) can be calculated with Formula (D.1):

$$D = 0{,}999\,84\,(S_{15} - E) \tag{D.1}$$

where

D	is density (20 °C) expressed in g/cm^3;
0,999 84	is the coefficient derived from the densities of water[4] and the expansion coefficient of glass;
S_{15}	is reading result using the relative-density hydrometer in (15/4 °C);
E	is instrumental error.

D.2 Density hydrometer (graduated in 20 °C)

When a density hydrometer which is graduated in 20 °C is used, the relative density (20/20 °C) can be calculated with Formula (D.2):

$$S = \frac{D}{0{,}998\,20} \tag{D.2}$$

where

S	is the relative density (20/20 °C);
D	is the density (20 °C) expressed in g/cm^3;
0,998 20	is the density of water at 20 °C[4].

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[1]   ISO 3675, *Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method*

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