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Second edition
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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.

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).

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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.

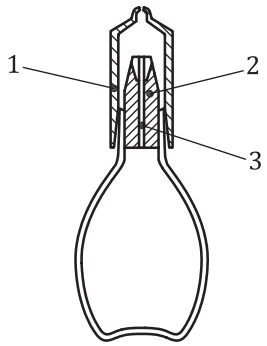
7.1.3 Pyknometer method

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 ± 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 ± 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 ± 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.