
**Rubber compounding ingredients —
Organic chemicals — General test
methods**

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 28641:2010

<https://standards.iteh.ai/catalog/standards/sist/0059af73-fe6d-49c2-9e99-3da9734b2ea6/iso-28641-2010>



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 28641:2010

<https://standards.iteh.ai/catalog/standards/sist/0059af73-fe6d-49c2-9e99-3da9734b2ea6/iso-28641-2010>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2010

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Abbreviations	2
4 General requirements	2
4.1 Thermometer	2
4.2 Dessicator	2
5 Sampling	2
5.1 Apparatus	2
5.2 Sampling method	2
6 Drying the sample	2
6.1 General	2
6.2 Drying methods for liquid compounding ingredients	2
6.3 Drying methods for solid compounding ingredients	3
7 Test methods	4
7.1 Relative density	4
7.2 Loss on heating	8
7.3 Sieve residue	10
7.4 pH of water extract	12
7.5 Melting point	14
7.6 Temperature of solidification	17
7.7 Softening point	20
7.8 Density of the bulk material	22
7.9 Ash	24
7.10 Refractive index	25
Annex A (informative) Examples of sampling apparatus	29
Annex B (informative) Examples of suitable drying apparatus	31
Annex C (normative) Verification of accuracy of pH-meter	34
Annex D (normative) Calibration of the pH-meter	38
Annex E (informative) Precision	40
Bibliography	47

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*.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 28641:2010](https://standards.iteh.ai/catalog/standards/sist/0059af73-fe6d-49c2-9e99-3da9734b2ea6/iso-28641-2010)

<https://standards.iteh.ai/catalog/standards/sist/0059af73-fe6d-49c2-9e99-3da9734b2ea6/iso-28641-2010>

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 pycnometer and graduated bicapillary pycnometer 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.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

5 Sampling

5.1 Apparatus

ISO 28641:2010
<https://standards.iteh.ai/catalog/standards/sist/0059af73-fe6d-49c2-9e99-3da9734b2ea6/iso-28641-2010>

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

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:

[ISO 28641:2010](#)

- a) hydrometer method (liquid sample); [ds.iteh.ai/catalog/standards/sist/0059af73-fe6d-49c2-9e99-3da9734b2ea6/iso-28641-2010](#)

- b) pycnometer 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 $20\text{ °C} \pm 0,5\text{ °C}$. When the temperature of the sample has reached $20\text{ °C} \pm 0,5\text{ °C}$, 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

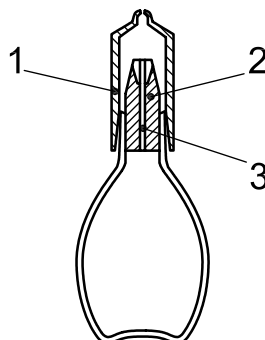
7.1.3.1 General

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.

<https://standards.iteh.ai/catalog/standards/sist/0059af73-fe6d-49c2-9e99-3da9734b2ea6/iso-28641-2010>



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 $(20 \pm 0,5)\text{ °C}$.
- 7.1.3.2.3 **Thermometer**, as specified in 4.1.
- 7.1.3.2.4 **Laboratory balance**, capable of weighing to the nearest $0,5\text{ mg}$.

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 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.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

7.1.3.3.2 Calculation

ISO 28641:2010

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

<http://standards.iteh.ai/catalog/standards/sist/0059af73-f66d-49c2-9e99-3da9734b2ea6/iso-28641-2010>

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

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 cm³ 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 $(20 \pm 0,5)$ °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 pycnometer containing the test portion with water at approximately 20 °C. Immerse it up to its neck in the water bath at $(20 \pm 0,5)$ °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 pycnometer, in grams;
- m_1 is the mass of the pycnometer filled with water, in grams;
- m_2 is the mass of pycnometer plus test portion, in grams;
- m_3 is the mass of pycnometer 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.

<https://standards.iteh.ai/catalog/standards/sist/0059af73-fe6d-49c2-9e99-3da9734b2ea6/iso-28641-2010>

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)

Accelerator	Temperature °C	Time h	Accelerator	Temperature °C	Time h
BA	100 ± 2	2	CMBT ^a	55 ± 2	3
DPG			CBS		
DOTG			TBBS		
MBT			DCBS		
MBTS			TETD		
ZMBT			DPTH		
ZDMC			DPTT		
ZDEC			ZDBC		
CuDMC			TeDEC		
ZEPC			DETU		
FeDMC ^b			DBTU		
ETU			TMU ^c		
MBSS			DIBS		
TMTM	80 ± 2	2	<p style="text-align: center;">iTeH STANDARD PREVIEW (standards.iteh.ai)</p> <p style="text-align: center;">ISO 28641:2010 https://standards.iteh.ai/catalog/standards/sist/0059af73-fe6d-49c2-9e99-5ab974e2ea6/iso-28641-2010</p>		
TMTD					
BQD					
DBQD					
^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	100 ± 2	2	ETMQ	75 ± 2	2
SPH			AANA		
MBI			PAN		
ZMBI			ODPA	70 ± 2	2
<i>o</i> -MBp24			SDPA		
<i>p</i> -BBp14			DCD ^a		
<i>p</i> -TBp14			TMQ	50 ± 2	3
DBHQ			NDBC		
DAHQ	MBMTB				
^a 4,4'-dicumyldiphenylamine.					

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 cm³.

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

7.3.5 Procedure

- (standards.iteh.ai)
- 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. <https://standards.iteh.ai/catalog/standards/sist/0059af73-fe6d-49c2-9e99-3de0734b2ca6/iso-28641-2010>
 - 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 cm³ beaker. Moisten it by adding ethanol. Press it lightly with a glass rod to crush any lumps.
 - d) Add about 50 cm³ 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 cm³ 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.