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Fifth edition
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Rubber compounding ingredients — Carbon black — Determination of sieve residue

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination du refus sur tamis

Reference number
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Foreword

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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 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 fifth edition cancels and replaces the fourth edition (ISO 1437:2007), which has been technically revised. The main changes compared to the previous edition are as follows:

— in the list of apparatus (see Clause 5), a rubber hose has been added in 5.1.6;

— Figure 1 has been revised to include a rubber hose;

— precision data have been moved in Annex A.

Rubber compounding ingredients — Carbon black — Determination of sieve residue

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 and to ensure compliance with any national regulatory conditions.

1 Scope

This document specifies a method for determining the water-wash sieve residue from regular, untreated carbon black for the rubber industry. It may not be applicable to oil-treated blacks because the oil could prevent proper wetting of the black by water.

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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

A known mass of carbon black is washed through a test sieve by a controlled flow of water, and the residue dried and weighed. The test sieve aperture is chosen from the range given in the appropriate material specification.

5 Apparatus

5.1 Sieving apparatus (Figure 1), comprising the following main items.

5.1.1 Test sieve, on which the residue is retained. Test sieves shall be of phosphor bronze or stainless steel, according to the characteristics given in ISO 565. Typical nominal apertures are 500 µm, 125 µm and 45 µm.

NOTE By mutual agreement between the interested parties, it is permissible to use sieves with a different aperture size.

5.1.2 Funnel or container, into the bottom of which the test sieve fits.

5.1.3 Nozzle, fed with clean water under controlled pressure by which the carbon black is washed through the sieve.

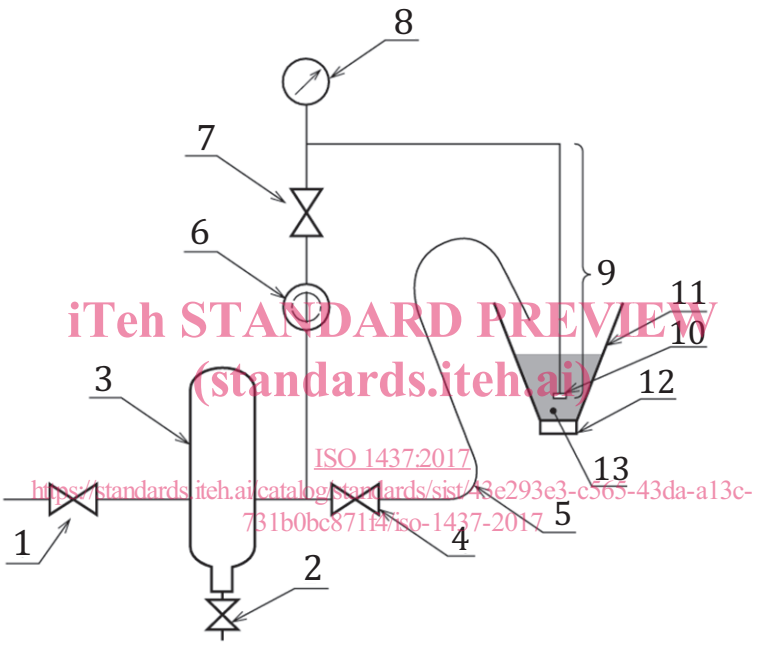
5.1.4 Water pressure regulating device.

5.1.5 Filter, in the water supply line, incorporating a wire screen at least as fine as that in the test sieve.

The tubing downstream of the filter shall not be liable to corrosion by tap water (use copper or stainless steel, for example).

5.1.6 Rubber hose, to supply water to wash down the funnel or container sides.

An example is given in Figure 1.



Key

1	valve A	8	pressure gauge
2	valve B	9	nozzle
3	filter	10	nozzle tip
4	valve C	11	funnel
5	rubber hose	12	sieve
6	water pressure control valve	13	carbon black
7	valve D		

Figure 1 — Schematic diagram of sieving apparatus[1)]

5.2 Balance, with an accuracy of 0,1 g.

5.3 Analytical balance, with an accuracy of 0,1 mg.

1) Suitable apparatus includes the Gallie-Porrit type of apparatus and the one described in ASTM D1514-15. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

5.4 Weighing dishes.

5.5 Oven, gravity-convection type, capable of temperature regulation within ±1 °C at 125 °C and temperature uniformity of ±5 °C.

6 Procedure

6.1 Precautions

6.1.1 Keep the apparatus clean at all times to prevent contamination.

6.1.2 Examine the sieve each time it is used to make sure no cracks or holes develop.

6.1.3 Examine the wire screen in the filter periodically to ascertain if the filter screen is in good condition.

6.2 Determination

6.2.1 Clean the filter used in the water line before starting a test.

6.2.2 Regulate the water pressure to the recommended pressure of (0,2 ± 0,04) MPa. Attach a sieve (5.1.1) of the specified aperture to the funnel or container (5.1.2) and allow water to flow through it for 3 min. Examine the sieve for particles. If none are observed, the apparatus is ready for use.

6.2.3 Weigh, to the nearest 0,1 g, a test portion of carbon black of at least 100 g.

6.2.4 Start the water flow. Carefully add carbon black to the funnel or container, taking care to prevent plugging of the sieve.

A wetting agent may be used before starting the water flow.

6.2.5 Wash down the carbon black from the funnel or container sides with water from the rubber hose (5.1.6). Continue to wash the residue on the sieve until the wash water coming through the sieve is clear.

6.2.6 Remove the sieve and rub the residue lightly with the finger to break up any agglomerates of carbon black which have not been thoroughly wetted by the water. Do not exert so much pressure with the finger that the sieve mesh is distorted.

6.2.7 Replace the sieve and wash for an additional 2 min.

6.2.8 Remove the sieve and dry in the oven (5.5) at 125 °C for 1 h.

6.2.9 Transfer the dried residue to a piece of smooth, white bond paper and rub gently to remove any carbon black remaining in the residue. Rub until the white paper no longer shows any smears.

6.2.10 Transfer the residue to a tared weighing dish (5.4) and weigh to the nearest 0,1 mg.

7 Expression of results

Either calculate the sieve residue, R, expressed in mg/kg (parts per million) using Formula (1):

$$R = \frac{m_1}{m_0} \times 10^6 \quad (1)$$

Or calculate the sieve residue, R, expressed as a percentage (%) using Formula (2):

$$R = \frac{m_1}{m_0} \times 100 \quad (2)$$

where

m_0 is the mass of the test portion, in grams (g);

m_1 is the mass of the residue retained on the sieve, in grams (g).

8 Precision and bias

See Annex A.

9 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 1437;

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

c) the nominal values of the sieve apertures;

d) the type of apparatus used and the water pressure;

e) the results and the units in which the results were expressed;

f) the date of the test.

Annex A
(informative)

Precision and bias

A.1 Precision

A.1.1 The precision of this test method was determined in accordance with ISO/TR 9272:2005, level 2 method. Refer to ISO/TR 9272 for terminology and other statistical details.

A.1.2 The precision results give an estimate of the precision to be expected. The precision parameters are not intended to be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

A.1.3 A type 1 precision interlaboratory-trials programme was conducted. Both the repeatability and the reproducibility determined represent short-term testing conditions. 16 laboratories tested two carbon blacks, twice on each of two different days: one with a considerable level and one with a very high level of sieve residue. Therefore, $p = 16$, $q = 2$ and $n = 4$. A test result is the value obtained from a single determination. Acceptable difference values were not measured.

A.1.4 The results of the precision calculations are given in Table A.1, with the materials arranged in descending order of mean sieve residue. Outliers have been removed. The number of laboratories remaining after outlier deletion is given in Table A.1.

Table A.1 — Precision calculations

Material	Number of laboratories	Mean sieve residue mg/kg (ppm)	Within-laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
A	13	98,6	10,14	28,69	29,09	30,10	89,85	91,11
B	15	362,0	55,88	158,04	43,66	194,37	572,02	158,01
Average	14	230,3						
Pooled values			40,16	113,58	37,10	139,08	409,44	128,97

s_r is the within-laboratory standard deviation;

r is the repeatability (in measurement units);

(r) is the repeatability (in percent);

s_R is the between-laboratory standard deviation;

R is the reproducibility (in measurement units);

(R) is the reproducibility (in percent).

A.1.5 The precision for the pooled values of the sieve residue may be expressed as follows.

a) Repeatability: the repeatability (r) for the sieve residue has been established as 37,1 %. Two single test results (or determinations) that differ by more than 37,1 %, should be considered suspect and dictate that some appropriate investigative action be taken.

b) Reproducibility: the reproducibility (R) for the sieve residue has been established as 129,0 %. Two single test results (or determinations), produced in separate laboratories, that differ by