INTERNATIONAL STANDARD

ISO 3858

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Rubber compounding ingredients — Carbon black — Determination of light transmittance of toluene extract

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de la transmittance spectrale de l'extrait toluénique

Reference number
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Foreword

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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 3858 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*.

It cancels and replaces ISO 3858-1:1990 and ISO 3858-2:1990, of which it constitutes a technical revision.

Rubber compounding ingredients — Carbon black — Determination of light transmittance of toluene extract

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 a method for the determination of the light transmittance of the toluene extract from carbon black for use in the rubber industry, as a means of measuring the discolouration caused by the extractable matter.

The light transmittance value provides an estimate of the degree of discolouration caused by the toluene-extractable matter present on the surface of the carbon black.

This method may not be applicable to carbon blacks with a high extractable-matter content.

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 1124, *Rubber compounding ingredients — Carbon black shipment sampling procedures*

ISO 1126:—[1)], *Rubber compounding ingredients — Carbon black — Determination of loss on heating*

ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

3 Principle

A sample of the carbon black is dried and a test portion weighed out and mixed with a measured volume of toluene at room temperature. The mixture is filtered and a portion of the filtrate transferred to an absorption cell. The light transmittance of the filtrate is measured against pure toluene at a set wavelength using a spectrophotometer.

4 Reagent

4.1 Toluene, analytical reagent grade, C.A.S. No. 108-88-3.

1) To be published. (Revision of ISO 1126:1992)

5 Apparatus

Ordinary laboratory equipment, plus the following:

5.1 Analytical balance, accurate to 0,1 mg.

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

5.3 Spectrophotometer, with a tungsten filament lamp, 20 nm maximum spectral passband width, capable of measuring percent transmittance at a wavelength of 425 nm. The instrument shall be of the high-resolution prism or grating type, eliminating the use of an optical filter. The instrument shall be operated in accordance with the manufacturer's operating manual for optimum performance. Some instruments may require the use of a constant-voltage transformer in the electricity circuit in order to compensate for voltage variations of more than 4 V.

NOTE Current types of photometer could be used. However, they usually differ in passband width from the specified one, and they may give different transmittance results. Proper calibration of such instruments over the whole transmittance range against a high-resolution spectrophotometer (of a passband width of 2 nm at 425 nm, for example) is recommended for possible corrections of the readings.

5.4 Absorption cells, with parallel sides polished flat to within 10 nm.

The internal distance between the parallel faces shall be 10 mm ± 0,05 mm.

NOTE 1 If the cell used does not have an optical path length of 10 mm, the transmittance which would be obtained with a 10 mm cell is given by the equation

$$\log_{10} \tau_0 = \frac{10}{L} \times \log_{10} \tau - \frac{20}{L} + 2 \qquad (1)$$

where

τ_0 is the percentage transmittance through a 10 mm cell;

τ is the percentage transmittance observed through the cell used;

L is the optical path length, in millimetres, of the cell used.

NOTE 2 Absorption cells may differ in their transmittance. It is recommended that the same absorption cell be used for adjustment of the spectrophotometer as for the actual measurements.

5.5 Conical flasks, capacity 100 cm^3 or 125 cm^3, with ground-glass stoppers.

5.6 Graduated cylinder, capacity 50 cm^3, graduated in divisions of 1 cm^3, or **automatic dispenser**, bottle type.

5.7 Pulverizer, i.e. mortar and pestle, high-speed blade mixer or equivalent.

5.8 Filter funnel, 75 mm inside diameter at top, made of chemically resistant glass.

5.9 Filter paper, 150 mm diameter, free from matter extractable by toluene and capable of retaining all the carbon black.

5.10 Beakers, capacity 50 cm^3 or 100 cm^3, with pouring lip.

5.11 Wiping paper, lint free, or **optical lens tissue**.

5.12 Cotton swabs.

5.13 Fume hood, fully enclosed on three sides, with suitable fume extraction and spark-proof fan and motor.

5.14 Safety container, to discard the used toluene and carbon black extracts.

6 Sample preparation

6.1 Samples shall be taken in accordance with ISO 1124.

6.2 Pulverize pelletized samples using the mortar and pestle or equivalent (5.7).

6.3 Dry approximately 4 g of the pulverized carbon black sample for 1 h at a temperature of 125 °C in the oven (5.2) as specified in ISO 1126:—, method 1. Allow to cool to ambient temperature in a desiccator. Keep the dried sample in the desiccator until ready for testing.

Carbon black shall not be dried at a temperature higher than that specified, nor dried using infra-red lamps, as some of the extractable matter may be driven off, thus affecting the results.

NOTE Drying is optional for blacks, such as thermal blacks, which are produced by a “dry” process.

7 Conditions of test

The test shall be carried out under standard conditions of 23 °C ± 2 °C and (50 ± 5) % or 27 °C ± 2 °C and (65 ± 5) % relative humidity. The reagent and apparatus shall be kept in the test environment for a time sufficient to reach ambient temperature before being used.

IMPORTANT — Toluene is a hazardous material, therefore this test shall be carried out in a fume hood with suitable fume extraction. Any motor, fan, etc., shall be spark-proof. The hood shall also be free from other fumes or vapours which might contaminate the reagent or equipment used and therefore affect the results.

8 Procedure

8.1 Standardization of spectrophotometer

8.1.1 Allow the spectrophotometer (5.3) to warm up for the length of time specified in the instrument operating manual.

8.1.2 Set the wavelength of the instrument at 425 nm. Check the zero reading of the instrument and adjust if necessary.

8.1.3 Place a filter paper (5.9) in a funnel (5.8) and filter approximately 30 cm^3 of toluene (4.1) into a conical flask (5.5), and stopper the flask.

8.1.4 Pour a portion of the filtered toluene into a beaker (5.10).

8.1.5 With the help of the beaker pouring lip, rinse an absorption cell (5.4) three times with the filtered toluene, filling to approximately one-third full each time.

Handle the absorption cell on the ground-glass sides only. Do not touch the smooth, clear sides with the fingers.

8.1.6 Fill the cell with filtered toluene and wipe the outside surfaces thoroughly with wiping paper or optical lens tissue (5.11), while holding the cell in front of a suitable light source for proper inspection.

The contents of the cell shall be free of any contaminant, such as lint particles, which might cause light scattering and influence the results. If necessary, clean the inside surface with a cotton swab (5.12), or wipe again the outside until perfectly clean. If cleaning of the internal cell surface is necessary, restart the procedure as in 8.1.5.

8.1.7 Place the absorption cell in the spectrophotometer, and adjust the instrument to read 100 % transmittance at a wavelength of 425 nm.

8.2 Sample testing

8.2.1 Except for N990, N991, N907 and N908 carbon blacks, weigh out 2 g ± 0,01 g of the pulverized and dried carbon black (see 6.2) and transfer this test portion to a conical flask (5.5).

For N990 and N991 carbon blacks, weigh out 5,0 g ± 0,01 g of black; for N907 and N908 carbon blacks, weigh out 3,0 g ± 0,01 g of black.

8.2.2 Using the graduated cylinder or automatic dispenser (5.6), add 20 cm^3 ± 0,5 cm^3 of toluene to the conical flask containing the test portion and stopper the flask (for N990 and N991 add 50 cm^3 ± 0,5 cm^3 of toluene, for N907 and N908 add 30 cm^3 ± 0,5 cm^3 of toluene).

If necessary, larger quantities of test portion and toluene may be used, but they shall remain in the ratio of 10 cm^3 of toluene for every 1 g of carbon black.

8.2.3 Within 5 s of adding the toluene, shake the mixture vigorously by hand for 60 s to 65 s. Alternatively, a mechanical shaker, capable of vigorous shaking at a rate of about 240 shakes per minute, may be used.

8.2.4 Immediately after shaking, filter the mixture with a funnel (5.8) and a filter paper (5.9) into a second conical flask (5.5), and stopper the flask.

If there is evidence of any trace of carbon in the filtrate, discard it, and repeat.

Change the filter paper for each test portion.

WARNING — Carbon blacks may contain polycyclic aromatic compounds, some of which are known carcinogens. These compounds, when present, are so strongly bound to the carbon black that they are biologically inactive, but they may be removed by the procedure specified in this standard. Care should be taken to avoid skin contact with solvent extracts from carbon blacks.

8.2.5 Using an absorption cell matching in transmittance the one used in 8.1.5, or if possible the same cell, repeat with the filtrate (see 8.2.4) the operations described in 8.1.4 to 8.1.6.

8.2.6 Place the absorption cell in the standardized (see 8.1) spectrophotometer, and record the percentage transmittance at the 425 nm wavelength to the nearest 1 %.

8.2.7 Rinse the absorption cell with clean toluene (4.1) immediately after each determination.

8.2.8 If possible, correct the transmittance values given by the spectrophotometer in accordance with the Note to 5.3 and Note 1 to 5.4 and record the result to the nearest 1 %.

9 Precision and bias

9.1 Precision and bias information have been obtained from an interlaboratory test programme carried out in accordance with ISO/TR 9272.

9.2 The results give an estimate of the precision as described below. The precision parameters shall not 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.

9.3 A Type 1 interlaboratory precision programme was conducted as detailed in Table 1. Both repeatability and reproducibility represent short-term (daily) test conditions. Testing was carried out by two operators in each laboratory performing the test once on each of two days. A test result was the value obtained from a single determination. Acceptable difference values were not measured. The between-operator component of variation is included in the values calculated for r and R.

Table 1 — Interlaboratory test programme

Nominal test period	Material	Number of laboratories
March 1996	N650	48
October 1996	IRB 6 (N330)	40
March 1997	SRB N762	44
September 1997	SRB A5 (N135)	39
March 1998	N550	45

9.4 The results of the precision calculations are given in Table 2, expressed in absolute values (percent transmittance).

Table 2 — Precision parameters

All values in percent

Material	Mean value	s_r	(r)	s_R	(R)
N550	96,91	0,68	1,93	1,87	5,28
N660	97,41	0,65	1,83	1,34	3,79
SRB N762	98,01	0,65	1,84	1,23	3,49
SRB A5 (N135)	98,99	0,39	1,09	0,64	1,81
IRB 6 (N330)	98,99	0,38	1,08	0,81	2,30
Average	98,06				
Pooled values		0,56	1,60	1,25	3,55

9.5 The precision for the pooled values of the light transmittance of the toluene extract may be expressed as follows:

9.5.1 Repeatability: The pooled relative repeatability, (r), of this test has been established as 1,60 %. Any other value in Table 2 may be used as an estimate of repeatability, as appropriate. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the repeatability on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results that differ by more than the appropriate value from Table 2 must be suspected of being from different populations and some appropriate action taken.

9.5.2 Reproducibility: The pooled relative reproducibility, (R), of this test has been established as 3,55 %. Any other value in Table 2 may be used as an estimate of reproducibility, as appropriate. The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results produced in different laboratories that differ by more than the appropriate value from Table 2 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.