

INTERNATIONAL STANDARD

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Third edition
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Carbon black for use in the rubber industry — Determination of light transmittance of toluene extract —

Part 1: Rapid method

*Noir de carbone pour l'industrie du caoutchouc — Détermination de la
transmittance spectrale de l'extrait toluénique —*

Partie 1: Méthode rapide



Reference number
ISO 3858-1:1990(E)

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.

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.

International Standard ISO 3858-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition (ISO 3858-1:1983), of which it constitutes a minor revision.

ISO 3858 consists of the following parts, under the general title *Carbon black for use in the rubber industry — Determination of light transmittance of toluene extract*:

- Part 1: *Rapid method*
- Part 2: *Method for product evaluation*

Carbon black for use in the rubber industry — Determination of light transmittance of toluene extract —

Part 1: Rapid method

1 Scope

This part of ISO 3858 specifies a rapid method for the determination of the light transmittance of the toluene extract from carbon black by means of a spectrometer.

The method provides a qualitative indication of the degree of discoloration of toluene by carbon blacks for use in the rubber industry.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 3858. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 3858 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 471:1983, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces*.

ISO 1126:1985, *Rubber compounding ingredients — Carbon black — Determination of loss on heating*.

3 Principle

Drying of the carbon black and weighing of a test portion. Mixing with a measured volume of toluene at room temperature. Filtration of the mixture and transfer of the filtrate to an absorption cell. Meas-

urement of the light transmittance of the filtrate against pure toluene at a set wavelength using a spectrometer.

4 Reagent

4.1 **Toluene**, analytical reagent grade.

5 Apparatus

Ordinary laboratory equipment and

5.1 **Analytical balance**, accurate to 0,01 g.

5.2 **Oven**, preferably of the gravity-convection type, capable of being maintained at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ or at $125\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

5.3 **Spectrometer**, giving direct readings of light transmittance at 425 nm, and with a selector for continuous variation, for example a high-resolution prism or grating. The passband shall be within $\pm 10\text{ nm}$ (see note 1).

A constant-voltage transformer shall be inserted into the supply circuit if the voltage is known to vary by more than 4 V.

NOTE 1 Current types of colorimeter may differ in passband width and may therefore give different transmittance results. The results may be more comparable if such colorimeters are calibrated over the whole of the useful transmittance range against the same high-resolution spectrometer, for example one having a passband which is narrower than 2 nm at the 425 nm central wavelength, and the readings corrected using the calibration curve thus produced for each colorimeter.

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

The internal distance between the parallel faces shall be $10 \text{ mm} \pm 0,05 \text{ mm}$ (see notes 2 and 3).

NOTES

2 Cylindrical cells of inner diameter $10 \text{ mm} \pm 0,05 \text{ mm}$ may give different results from parallelepipedic cells. If used, it is recommended that they be calibrated against a parallelepipedic cell over the whole of their useful transmittance range and that corrections be taken from the calibration curve thus produced.

3 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

$$\lg \tau_0 = \frac{10}{l} \times \lg \tau - \frac{20}{l} + 2$$

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.

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

5.5 Conical flasks, capacity 100 cm^3 or 125 cm^3 .

5.6 Graduated cylinder, capacity 50 cm^3 , graduated in divisions of 1 cm^3 .

5.7 Test tubes, capacity 50 cm^3 .

5.8 Filter funnels, made of chemically resistant glass.

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

6 Sample preparation

Dry approximately 4 g of the carbon black sample for 1 h at a temperature of either $105^\circ\text{C} \pm 2^\circ\text{C}$ or $125^\circ\text{C} \pm 2^\circ\text{C}$ in accordance with ISO 1126. 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 extract may be driven off, thus affecting the results.

Pellets of carbon black shall not be crushed.

7 Conditions of test

The test shall be carried out under standard conditions, as given in ISO 471, of $23^\circ\text{C} \pm 2^\circ\text{C}$ and $(50 \pm 5) \%$ relative humidity or $27^\circ\text{C} \pm 2^\circ\text{C}$ and $(65 \pm 5) \%$ 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 and toxic material, therefore this test shall be carried out in a fume cupboard with suitable fume extraction. Any motor, fan, etc., shall be spark proof. The cupboard shall also be free from other fumes or vapours which might contaminate the reagent or equipment used and therefore affect the results.

8 Procedure

WARNING — All recognized health and safety precautions shall be taken when using the procedure specified in this part of ISO 3858.

Carbon blacks may contain polynuclear 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 part of ISO 3858. Care should be taken to avoid skin contact with solvent extracts from such carbon blacks.

8.1 Allow the spectrometer (5.3) to warm up for at least 10 min before adjustment (see note 4 to 5.4).

Filter approximately 50 cm^3 of toluene (4.1) into a test tube (5.7), clean an absorption cell with toluene and dry with optical lens tissue.

Fill the cell with the filtered toluene and dry the outside of the cell with optical lens tissue. Place the cell in the spectrometer and adjust the instrument to 100 % transmittance using a wavelength of 425 nm.

8.2 Weigh out $3 \text{ g} \pm 0,01 \text{ g}$ of the dried carbon black and transfer this test portion to a conical flask (5.5).

If the capacity of the absorption cell makes it necessary, use a larger test portion; add 10 cm^3 of toluene for each additional gram of carbon black.

8.3 Using the graduated cylinder (5.6), pour $30 \text{ cm}^3 \pm 0,5 \text{ cm}^3$ of filtered toluene into the conical flask containing the test portion.

8.4 Within 5 s after adding the toluene, swirl the mixture, using a circular motion, for $15 \text{ s} \pm 5 \text{ s}$.

8.5 Within 30 s after swirling, filter the mixture through the filter paper (5.9) into a second test tube (5.7). Change the filter paper for each test portion.

8.6 Rinse the absorption cell with approximately 1 cm³ of the filtrate from 8.5 and empty the cell.

8.7 Within 1 min after filtration, fill the absorption cell with the filtrate from 8.5 and dry the outside of the cell with optical lens tissue.

8.8 Place the cell in the adjusted spectrometer (see 8.1) and read the percentage transmittance at a wavelength of 425 nm.

8.9 Rinse the absorption cell with clean toluene immediately after each determination.

9 Expression of results

The reading obtained in 8.8 is the light transmittance of the toluene extract, expressed as a percentage, for an optical path length through the filtrate of

10 mm at a wavelength of 425 nm, with reference to pure toluene.

Round off the result to the nearest 1 %.

10 Test report

The test report shall include the following information:

- a) a reference to this part of ISO 3858;
- b) all details necessary for full identification of the sample;
- c) all details necessary for identification of the spectrometer used;
- d) the drying temperature used;
- e) the result obtained;
- f) the date of the test.

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