International Standard 3858/2

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Carbon black for use in the rubber industry — Determination of light transmittance of toluene extract — Part 2 : Method for product evaluation

Noir de carbone pour l'industrie des élastomères — Détermination de la transmittance lumineuse de l'extrait toluénique — Partie 2 : Méthode d'évaluation du produit

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Carbon black for use in the rubber industry — Determination of light transmittance of toluene extract — Part 2 : Method for product evaluation

1 Scope and field of application

This part of ISO 3858 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 extracted matter, as is required in product evaluation.

A rapid method for determining the light transmittance of the toluene extract from carbon black is specified in ISO 3858/1[1] and is particularly useful for product control purposes.

The degree of discolouration is quantitatively measured by means of a spectrophotometer.

This method is not applicable to high-extract, thermal-type blacks.

2 References

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

ISO 1126, *Carbon black for use in the rubber industry — 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. Measurement of the light transmittance of the filtrate against pure toluene at a set wavelength using a spectrophotometer.

4 Reagent

4.1 Toluene, spectrography reagent grade.

5 Apparatus

Usual laboratory equipment and

5.1 Analytical balance, accurate to at least 0,01 g.

5.2 Oven, capable of maintaining a temperature of 105 ± 2 °C.

5.3 Spectrophotometer, giving direct readings of light transmittance at 425 nm.

The spectrophotometer shall be of the high resolution prism or grating type eliminating the use of an optical filter. Bandpass shall be within ± 10 nm.

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

NOTE — Current types of colorimeters may differ by the width of a passing band and may therefore give different light transmittance results. The results may be more comparable if such colorimeters are calibrated against the same high resolution spectrophotometer, for example having a passing band which is narrower than 2 nm at 425 nm mean wavelength, and the readings corrected by using the calibration curve for each instrument through the useful range of light transmittance.

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

The internal distance between the parallel faces shall be 10,00 ± 0,05 mm (see notes 1 and 2).

NOTES

1 Cylindrical cells of inner diameter 10,00 ± 0,05 mm may give different results from parallelepipedic cells. If used, it is recommended that they be calibrated against a parallelepipedic cell over the full useful range of light transmittance and that corrections be taken from the calibration curve.

1) At present ISO 3858-1977.

2 If the cell used does not give a 10 mm light path, the following formula may be used to calculate the light transmittance which would be obtained through a cell of 10 mm :

$$\log_{10} T_0 = \frac{10}{L} \times \log_{10} T - \frac{20}{L} + 2$$

where

T_0 is the percentage light transmittance through a 10 mm cell;

T is the percentage light transmittance observed through a cell of path length L mm;

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

3 Absorption cells may differ in their light transmittance. It is recommended that the same absorption cell be used for adjustment of the spectrophotometer.

5.5 Conical flasks, capacity 100 or 125 cm^3,* with ground glass stopper.

5.6 Graduated cylinder, capacity 50 cm^3, with graduations of 1 cm^3.

5.7 Pulverizer : mortar and pestle, high speed blade mixer or equivalent.

5.8 Filter funnels, 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 such that it retains all the carbon black.

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

5.11 Optical lens tissue, lint free.

6 Sample preparation

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

6.2 Dry approximately 4 g of the pulverized carbon black sample for 1 h at a temperature of 105 ± 2 °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.

NOTE — Carbon black should never be dried at a temperature higher than that specified, nor is the use of infra-red lamps permitted for drying, as some of the extract may be driven off and alter the results.

7 Conditions of test

The test shall preferably be carried out under standard laboratory conditions, as given in ISO 471, of 23 ± 2 °C and 50 ± 5 % relative humidity or 27 ± 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.

Toluene should be considered as a hazardous and toxic material; therefore, this test must be carried out in a fume cupboard with suitable fume extraction. Any motor, fan etc. should be spark proof. The cupboard shall also be free from other fumes or vapours which might contaminate the reagent and testing equipment to be used and therefore alter the results.

8 Procedure

8.1 Allow the spectrophotometer (5.3) to warm up for at least 10 min before adjustment (see 5.4, note 3).

Filter approximately 30 cm^3 of toluene (4.1) into a conical flask (5.5) and stopper the flask. Pour a portion of the toluene into a beaker (5.10) and rinse an absorption cell (5.4) three times with filtered toluene, filling to approximately one-third full each time.

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

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

8.2 Weigh 2,0 ± 0,01 g of the pulverized and dried carbon black and transfer this test portion into a conical flask (5.5).

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

8.3 Using the graduated cylinder (5.6), pour 20 ± 0,5 cm^3 of filtered toluene into the conical flask containing the test portion and stopper the flask.

8.4 Within 5 s after adding the toluene, swirl the mixture by hand, using a circular motion, for 60 $^{+5}_{0}$ s. Alternatively, a mechanical shaker, capable of vigorous shaking at a rate of approximately 240 shakes per minute may be used.

* The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm^3), in accordance with a decision of the 12th Conférence Générale des Poids et Mesures. The term millilitre is acceptable, in general, for references in International Standards to capacities of volumetric glassware and to liquid volumes. Glassware with either marking is satisfactory for use with the procedure described in this International Standard.

8.5 Immediately after swirling, filter the mixture through the filter paper (5.9) into a second conical flask (5.5) and stopper the flask. If there is evidence of any trace of carbon black in the filtrate, discard and repeat. Change the filter paper for each test portion.

8.6 Rinse the absorption cell three times, each with approximately 1 cm^3 of the filtrate from 8.5 and empty the cell.

8.7 Fill the absorption cell to the top 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 spectrophotometer (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

Express the light transmittance of the toluene extract as a percentage, through a filtrate thickness 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) the full identification of the sample;

c) the identification of the spectrophotometer used;

d) the results obtained;

e) the date of the test.