45

INTERNATIONAL STANDARD ISO 3858

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 — Rapid method

Noir de carbone pour l'industrie du caoutchouc — Détermination de la transmittance lumineuse de l'extrait toluénique — Méthode rapide

First edition — 1977-10-15

UDC 678.046.2 : 620.1

Ref. No. ISO 3858-1977 (E)

Descriptors : carbon black, rubber industry, toluene, physical tests, determination, transmittance, discoloration.

Price based on 2 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3858 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in June 1975.

It has been approved by the member bodies of the following countries :

Australia	India	Spain
Belgium	Ireland	Sweden
Bulgaria	Italy	Thailand
Canada	Mexico	Turkey
France	Netherlands	United Kingdom
Germany	Romania	U.S.S.R.
Hungary	South Africa, Rep. of	Yugoslavia

No member body expressed disapproval of the document.

Printed in Switzerland

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

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a rapid method for the determination of the light transmittance of the toluene extract from carbon black by means of a spectrophotometer.

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

2 REFERENCES

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces*[1].

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, analytical reagent grade.

5 APPARATUS

5.1 Analytical balance, accurate to 0,01 g.

5.2 Oven, preferably gravity convection type, capable of maintaining a temperature of 105 ± 2 °C.

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

The spectrophotometer should 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.

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 ml*.

5.6 Graduated cylinder, capacity 50 ml, with graduations of 1 ml.

5.7 Test tubes, capacity 50 ml.

1) At present at the stage of draft. (Revision of ISO/R 471.)

* The term millilitre (ml) is commonly used for the cubic centimetre (cm^3), particularly to denote the capacity of laboratory glassware. Apparatus with either type of marking is satisfactory to use with this standard.

5.8 Filter funnels, made of chemically resistant glass.

5.9 Filter paper free from matter extractable by toluene, and such that it retains all the carbon black.

6 SAMPLE PREPARATION

Dry approximately 4 g of the 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.

NOTES

1 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.

2 Pellets of carbon black should not be crushed.

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. It is recommended that the reagent and the apparatus be kept in this environment for a time sufficient to reach ambient temperature before being used.

The test room shall be free from 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 50 ml of toluene (4.1) into a test tube (5.7), clean an absorption cell (5.4) 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 spectrophotometer and adjust the instrument to 100 % transmittance using a wavelength of 425 nm.

8.2 Weigh 3,00 ± 0,01 g of the 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 ml of toluene for each additional gram of carbon black.

8.3 Using the graduated cylinder (5.6), pour 30 ± 0,5 ml 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 ± 5 s.

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

8.6 Rinse the absorption cell with approximately 1 ml 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 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.

NOTE — Change the filter paper for each test portion.

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 particulars :

a) a reference to this International Standard;

b) the full identification of the sample;

c) the identification of the spectrophotometer used;

d) the results obtained;

e) the date of the test.