TC 45

International Standard ISO 3858/2

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

Rubber compounding ingredients — Carbon black — Determination of light transmittance of toluene extract — Part 2 : Method for product evaluation

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de la transmittance spectrale de l'extrait toluénique — Partie 2 : Méthode d'évaluation du produit

Second edition — 1983-12-15

UDC 678.046.2 : 535.243 **Ref. No. ISO 3858/2-1983 (E)**

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

Price based on 3 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (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 authorized 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 3858/2 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition was submitted directly to the ISO Council, in accordance with clause 6.11.2 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 3858/2-1982), which had been approved by the member bodies of the following countries :

Australia	India	Romania
Austria	Ireland	South Africa, Rep. of
Belgium	Italy	Spain
Brazil	Korea, Dem. P. Rep. of	Sri Lanka
Canada	Korea, Rep. of	Sweden
China	Malaysia	Thailand
Czechoslovakia	Mexico	Turkey
Denmark	Netherlands	United Kingdom
France	New Zealand	USSR
Germany, F. R.	Poland	
Hungary	Portugal	

The member body of the following country had expressed disapproval of the document on technical grounds :

USA

Printed in Switzerland

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0 Introduction

ISO 3858 consists of the following parts :

— Part 1 : Rapid method.

— 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 discoloration caused by the extracted matter, as is required in product evaluation.

The degree of discoloration is quantitatively measured by means of a spectrometer.

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 spectrometer.

4 Reagent

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 being maintained at 105 ± 2 °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. Bandpass shall be within ± 10 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.

The spectrometer shall be equipped with absorption cells, of thickness 10,00 ± 0,05 mm (see notes 2 and 3), with parallel sides polished flat to within 10 nm.

NOTES

1 Current types of colorimeters may differ by the width of a passing band and may therefore give different transmittance results. The results may be more comparable if such colorimeters are calibrated against the same high resolution spectrometer, 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 transmittance.

2 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 transmittance and that corrections be taken from the calibration curve.

3 If the cell used does not give a 10 mm optical path length, the transmittance which would be obtained through a cell of 10 mm is given by the equation

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

where

τ_0 is the percentage transmittance through a 10 mm cell;

τ is the percentage transmittance observed through a cell of thickness l mm;

l is the thickness, 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.

5.4 Conical flasks, capacity 100 or 125 cm^3, * with ground glass stoppers.

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

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

5.7 Filter-funnels, 75 mm inside diameter at top, made of chemically resistant glass.

5.8 Filter-paper, 150 mm diameter, free from matter extractable by toluene, and such that it retains all the carbon black.

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

5.10 Optical lens tissue, lint free.

6 Sample preparation

6.1 Pulverize pelletized samples using the mortar and pestle (5.6) 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 must not 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 spectrometer (5.3) to warm up for at least 10 min before adjustment (see note 4 to 5.3).

Filter approximately 30 cm^3 of toluene (clause 4) into a conical flask (5.4) and stopper the flask. Pour a portion of the toluene into a beaker (5.9) and rinse an absorption cell three times with filtered toluene, filling 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 (5.10). Place the cell in the spectrometer 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 (5.4).

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 (5.5), 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.

8.5 Immediately after swirling, filter the mixture through the filter-paper (5.8) into a second conical flask (5.4) 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 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.

* 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.

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 spectrometer used;

d) the results obtained;

e) the date of the test.