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Rubber compounding ingredients — Carbon black — Determination of tinting strength

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination du pouvoir colorant

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document 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*.

This fifth edition cancels and replaces the fourth edition (ISO 5435:2008), which has been technically revised.

The main changes compared to the previous edition are as follows:

— throughout the text, ITRB has been replaced by ITRB or ITRB-2, since both tint references are in use;

— Clause 5 on reagents and its footnotes have been updated to reflect the materials which are currently available;

— a note has been added in Clause 5 to encourage the use of the specified materials only;

— in A.2.1 the type of materials used during the interlaboratory test programme (ITP) in 2005 has been added.

Rubber compounding ingredients — Carbon black — Determination of tinting strength

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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 document specifies a method for the determination of the tinting strength of carbon black relative to an industry tint reference black.

The method is based on the use of five different commercial instruments. Other instruments can be used if the test results for the standard reference blacks are within the control limits given in ASTM D4821.

NOTE The Densichron reflectometer and the Meeco Colormaster are no longer commercially available, but the procedures have been included for the benefit of those who still use these instruments.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1126, *Rubber compounding ingredients — Carbon black — Determination of loss on heating*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

4 Principle

An industry tint reference black (ITRB or ITRB-2) is mixed with zinc oxide and a liquid plasticizer. The paste obtained is mulled and homogenized, then spread as a layer of regular thickness. Its light reflectance is measured with a reflectometer sensitive to different shades of grey.

A test sample is mixed and its light reflectance measured in the same manner and, from the two light reflectance values, the relative tinting strength of the sample is determined.

5 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

NOTE Users of this document are strongly encouraged to use only the materials mentioned below. Different types of oil or zinc oxide can lead to issues during preparation and drawdown of the pastes and can provide erroneous test results.

5.1 Epoxidized soybean oil[1], for use as plasticizer, of relative density 0,92 to 0,99.

It is essential that the epoxidized soybean oil is maintained above 20 °C to prevent turbidity which leads to erroneous results.

5.2 Industry tint reference black (ITRB or ITRB-2)[2].

Minor differences can occur depending on whether ITRB or ITRB-2 is used as a reference black. In this case the parties involved shall agree upon common raw materials or shall determine a correction factor to be applied to the test results.

5.3 Zinc oxide, e.g. industry tint zinc oxide (ITZnO)[3].

5.4 Standard reference blacks[4].

6 Apparatus

Standard laboratory equipment together with the following.

6.1 Analytical balance, accurate to 0,1 mg.

6.2 Automatic muller[5].

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

6.4 Flexible palette knives, preferably tapered, of stainless steel, 100 mm to 150 mm long.

6.5 Syringe, automatic filling, accurate to 0,02 cm^3.

6.6 Light-measuring instrument, sensitive to variations in light reflectance of shades of grey (see the appropriate procedure in Clause 8).

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1) Suitable epoxidized soybean oil is available from Balentine Enterprises, Inc., dba Laboratory Standards and Technologies, 227 Somerset St., Borger, TX 79007, USA, www.carbonstandard.com. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

2) ITRB is depleted. ITRB-2 is available from Balentine Enterprises, Inc., dba Laboratory Standards and Technologies, 227 Somerset St., Borger, TX 79007, USA, www.carbonstandard.com. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

3) Tint zinc oxide is available from from Balentine Enterprises, Inc., dba Laboratory Standards and Technologies, 227 Somerset St., Borger, TX 79007, USA, www.carbonstandard.com. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

4) Standard reference blacks are available from Balentine Enterprises, Inc., dba Laboratory Standards and Technologies, 227 Somerset St., Borger, TX 79007, USA, www.carbonstandard.com. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the products supplied by this company. Equivalent products may be used if they can be shown to lead to the same results.

5) The Hoover Automatic Muller, Model M5, from Hoover Color Corp., PO Box 218, State Highway 693, Hiwassee, VA 24347, USA (e-mail: hoover@hoovercolor.com), and Automatic Pigment Muller, Model JEL 25/53, from J. Engelsmann AG, Frankenthaler Str. 137-141, D-67059 Ludwigshafen, Germany (e-mail: info@engelsmann.de) are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.

6.7 Paste application apparatus (see the appropriate procedure in Clause 8).

7 Test conditions

The test should preferably be carried out under standard temperature and humidity conditions, i.e. 23 °C ± 2 °C and (50 ± 5) % relative humidity or 27 °C ± 2 °C and (65 ± 5) % relative humidity.

8 Procedure

8.1 Preparation of pastes

8.1.1 Dry the zinc oxide (5.3) and carbon black in accordance with ISO 1126. In addition, dry just enough ITRB or ITRB-2 (5.2), as re-drying on a future occasion is not recommended.

8.1.2 Weigh the required amount of carbon black or ITRB or ITRB-2, respectively (see 8.1.10 and 8.1.11) to the nearest 1 mg (crushing of pelleted carbon blacks is beneficial). Weigh 3,750 g ± 1 mg of the ITZnO.

8.1.3 Using the syringe (6.5), place 2,2 cm^3 of the plasticizer (5.1) in the centre of the lower glass plate of the automatic muller (6.2). 2,0 cm^3 of the plasticizer may be used when it is difficult to prepare a paste with 2,2 cm^3 of the plasticizer.

8.1.4 Place the ITZnO and carbon black in the centre of the pool of plasticizer.

8.1.5 Mix the three materials thoroughly with the palette knife (6.4).

8.1.6 Set the muller to a pressure of 0,017 MPa between the plates (445 N force when using 18,4 cm diameter plates) by placing the extra masses, supplied with the instrument, on the arm. Close and mull for 25 revolutions.

8.1.7 Open the plates, scrape the upper plate with a palette knife to remove as much paste as possible and transfer it to the lower plate. Then, with the lower plate rotating, use the palette knife to spread the paste to a flattened circle on the plate and work all the paste to the centre. Repeat this step twice more.

8.1.8 Repeat steps 8.1.6 and 8.1.7 three times, i.e. for a total of 4 × 25 revolutions.

8.1.9 Remove the paste to a clean smooth surface.

Pastes should preferably be tested immediately. In no case shall they be kept for more than 24 h.

8.1.10 For calibration, prepare pastes in duplicate using the masses of carbon black given in Table 1.

Table 1 — Masses of carbon black

ITRB or ITRB-2 g	Calibration tint value %
0,090	90 units
0,100	100 units
0,110	110 units
0,120	120 units
0,130	130 units
0,140	140 units

8.1.11 For determinations of tinting strength of test samples, prepare duplicate pastes with

— 0,100 g of N100 to N400 series carbon blacks;

— 0,200 g of N500 to N700 series carbon blacks.

8.2 Individual procedures

8.2.1 General

The following instructions relate to different types of commercial instrument (6.6).

All instruments shall be operated in accordance with the manufacturer's instructions.

8.2.2 Measurements using the Erichsen Tint Tester

8.2.2.1 Apparatus (in addition to that specified in **6.1** to **6.5**)

Standard laboratory equipment together with the following.

8.2.2.1.1 Erichsen Tint Tester[6], model 517 or 527.

NOTE The model 527 has a specially designed head which keeps the paste off the lens.

8.2.2.1.2 Film applicator, 0,08 mm gauge depth, to give 0,04 mm wet film thickness.

8.2.2.1.3 Glass plate, approximately 750 mm × 500 mm × 10 mm.

8.2.2.2 Calibration

8.2.2.2.1 Switch on the instrument and adjust in accordance with the manufacturer's instructions. Clean the glass plate (8.2.2.1.3) with a tissue to remove any dust particles and film. Using a clean palette knife (6.4), place a portion of one of the mulled pastes with 100 % calibration tint value (see 8.1.10) at the top edge of the glass plate and smear it almost to the bottom edge. Using the film applicator (8.2.2.1.2), draw the paste down to the bottom edge in about 2 s to 3 s.

8.2.2.2.2 Place the reflectometer head on the paste drawdown.

Adjust the meter to read 3,0 for at least three readings, all taken at 75 mm or more from the top of the drawdown. If the results are variable, gather up the paste and make another drawdown.

8.2.2.2.3 Make a duplicate drawdown of the duplicate paste as in 8.2.2.2.1. Without altering the settings, take three readings from the duplicate paste. If the results are variable, gather up the paste and make another drawdown. These pastes are acceptable if a reading of 3,0 ± 0,02 is obtained from the duplicate paste.

NOTE It is beneficial to have the two drawdowns side by side.

8.2.2.2.4 If these pastes are acceptable, blend the two together using a palette knife (6.4).

8.2.2.2.5 If the pastes are not acceptable, prepare a further paste and determine its reflectance using the same procedure.

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6) The Erichsen Tint Tester® is the trade name of an apparatus supplied by Erichsen GmbH u. Co KG, Am Iserbach 14, D-58675 Hemer, Germany (e-mail: info@erichsen.de). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the apparatus named.

8.2.2.2.6 Blend together whichever two of these three pastes do not differ by more than 0,02 units.

8.2.2.2.7 Determine the reflectance values of the remaining calibration pastes (see 8.1.10), taking three readings with each paste.

8.2.2.2.8 Plot a graph of the average readings versus the calibration tint values or determine the equation which gives the best fit.

NOTE The equation is not necessarily linear.

8.2.2.2.9 Periodically, check the proper calibration and standardization of the equipment, reagents, materials and method, as required, using standard reference blacks (5.4).

8.2.2.3 Determination of tinting strength of a test sample

8.2.2.3.1 Prepare duplicate pastes of 100 % calibration tint value as described in 8.1.1 to 8.1.10.

8.2.2.3.2 Prepare the drawdowns and set the instrument to 3,0 as described in 8.2.2.2.1 to 8.2.2.2.6.

8.2.2.3.3 Using the correct mass of test sample (see 8.1.11), prepare duplicate pastes as described in 8.1.1 to 8.1.9.

8.2.2.3.4 Determine the reflectance values of the pastes containing the test sample, taking three readings with each paste.

8.2.2.3.5 Determine the tinting strength of the test sample by using the graph or equation determined in 8.2.2.2.8.

8.2.3 Measurements using the Densichron reflectometer (film drawdown and roller spreader methods)

8.2.3.1 Apparatus (in addition to that specified in **6.1** to **6.5**)

Standard laboratory equipment together with the following.

8.2.3.1.1 Densichron reflectometer, for the film drawdown method.

8.2.3.1.2 Film applicator, 0,08 mm gauge depth, to give 0,04 mm wet film thickness.

8.2.3.1.3 Glass plate, approximately 750 mm × 500 mm × 10 mm.

8.2.3.1.4 Tint roller spreader, for the roller spreader method.

8.2.3.2 Calibration

8.2.3.2.1 Film drawdown method

8.2.3.2.1.1 Clean the glass plate (8.2.3.1.3) with a tissue to remove any dust particles and film. Using a clean palette knife (6.4), place a portion of one of the mulled pastes with 100 % calibration tint value (see 8.1.10) at the top edge of the glass plate and smear it almost to the bottom edge. Using the film applicator (8.2.3.1.2), draw the paste down to the bottom edge in about 2 s to 3 s.

8.2.3.2.1.2 Set the range switch to 2 and operate with no filter in the reflection head.

Use a 4,76 mm aperture if head No. 3882A is used.

Place the reflectometer head on the paste drawdown. Adjust the meter to read a reflectance value of 80 for at least three readings, all taken at 75 mm or more from the top of the drawdown. If the results are variable, gather up the paste and make another drawdown.

8.2.3.2.1.3 Make a duplicate drawdown of the duplicate paste as in 8.2.3.2.1.1. Without altering the settings, take three readings from the duplicate paste. If the results are variable, gather up the paste and make another drawdown. These pastes are acceptable if a reading of 80 ± 0,5 is obtained from the duplicate paste.

NOTE It is beneficial to have the two drawdowns side by side.

8.2.3.2.1.4 If these pastes are acceptable, blend the two together using a palette knife (6.4).

8.2.3.2.1.5 If the pastes are not acceptable, prepare another paste containing 0,100 g of ITRB or ITRB-2 and check using the same procedure.

8.2.3.2.1.6 Blend together whichever two of these three pastes do not differ by more than 0,5 units.

8.2.3.2.1.7 Determine the reflectance values of the remaining calibration pastes (see 8.1.10), taking three readings with each paste.

8.2.3.2.1.8 Plot a graph of the average readings versus the calibration tint values or determine the equation which gives the best fit.

NOTE The equation is not necessarily linear.

8.2.3.2.1.9 Periodically check the proper calibration and standardization of the equipment, reagents, materials and method, as required, using standard reference blacks (5.4).

8.2.3.2.2 Roller spreader method

8.2.3.2.2.1 Using a clean palette knife (6.4), place a portion of one of the mulled pastes of 100 % calibration tint value (see 8.1.10) on the clean turning roller of the tint roller spreader (8.2.3.1.4). Turn the range switch from position 0 to position 2. Using the calibration control, adjust the meter to read a reflectance value of 80. Turn the range switch to position 0. Clean the roller and applicator. Place a portion of the other paste having 100 % calibration tint value on the turning roller and turn the range switch to position 2 without altering the calibration control.

8.2.3.2.2.2 The pastes are acceptable if readings of 80 ± 0,5 are obtained.

8.2.3.2.2.3 If the pastes are not acceptable, prepare another paste containing 0,100 g of ITRB or ITRB-2 and check using the same procedure.

8.2.3.2.2.4 Blend together whichever two of these three pastes do not differ by more than 0,5 units.

8.2.3.2.2.5 Determine the reflectance values of the remaining calibration pastes (see 8.1.10).

8.2.3.2.2.6 Plot a graph of the average readings versus the calibration tint values or determine the best-fit linear equation by the method of least squares.

