
**Rubber compounding ingredients —
Carbon black — Determination of tinting
strength**

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

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 5435 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 fourth edition cancels and replaces the third edition (ISO 5435:1994), which has been technically revised to include two additional types of test equipment and precision data.

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Rubber compounding ingredients — Carbon black — Determination of tinting strength

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 International Standard 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 may 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.

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2 Normative references

[ISO 5435:2008](#)

The following referenced documents are indispensable for the application 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*

ASTM D4821, *Standard Guide for Carbon Black — Validation of Test Method Precision and Bias*

3 Principle

An industry tint reference black (ITRB) 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.

4 Reagents and materials

4.1 **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.

4.2 **Industry tint reference black (ITRB)** ²⁾.

4.3 **Zinc oxide**, e.g. industry tint zinc oxide (ITZnO) ³⁾.

Other zinc oxides may be used, provided that they give the same results.

4.4 **Standard reference blacks** ⁴⁾.

5 Apparatus

5.1 **Analytical balance**, accurate to 0,1 mg.

5.2 **Automatic muller** ⁵⁾.

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

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

5.5 **Syringe**, automatic filling, accurate to 0,02 cm³.

5.6 **Light-measuring instrument**, sensitive to variations in light reflectance of shades of grey (see the appropriate procedure in Clause 7).

5.7 **Paste application apparatus** (see the appropriate procedure in Clause 7).

1) Paraplex® G-62 is the trade name of an epoxidized soybean oil supplied by HallStar, 120 South Riverside Plaza, Suite 1620, Chicago, IL 60606, USA. This information is given for the convenience of users of this International Standard 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 available from Balentine Enterprises, Inc., 227 Somerset St., Borger, TX 79007, USA. This information is given for the convenience of users of this International Standard 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) ITZnO is available from Horsehead Corp., 300 Frankfort Rd., Monaca, PA 15061, USA. This information is given for the convenience of users of this International Standard 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., 227 Somerset St., Borger, TX 79007, USA. This information is given for the convenience of users of this International Standard 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 International Standard 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 Test conditions

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

7 Procedure

7.1 Preparation of pastes

7.1.1 Dry the ITZnO (4.3) and carbon black in accordance with ISO 1126. In addition, dry just enough ITRB (4.2), as re-drying on a future occasion is not recommended.

7.1.2 Weigh the required amount of carbon black or ITRB (see 7.1.10 and 7.1.11) to the nearest 1 mg (crushing of pelleted carbon blacks is beneficial). Weigh $3,750\text{ g} \pm 1\text{ mg}$ of the ITZnO.

7.1.3 Using the syringe (5.5), place $2,2\text{ cm}^3$ of the plasticizer (4.1) in the centre of the lower glass plate of the automatic muller (5.2). $2,0\text{ cm}^3$ of the plasticizer may be used when it is difficult to prepare a paste with $2,2\text{ cm}^3$ of the plasticizer.

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

7.1.5 Mix the three materials thoroughly with the palette knife (5.4).

7.1.6 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.

7.1.7 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.

7.1.8 Repeat steps 7.1.6 and 7.1.7 three times, i.e. for a total of 4×25 revolutions.

7.1.9 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.

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

Table 1 — Masses of carbon black

ITRB 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

7.1.11 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.

7.2 Individual procedures

7.2.1 General

The following instructions relate to different types of commercial instrument (5.6).

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

7.2.2 Measurements using the Erichsen Tint Tester

7.2.2.1 **Apparatus** (in addition to that specified in 5.1 to 5.5)

7.2.2.1.1 **Erichsen Tint Tester**⁶⁾, model 517 or 527.

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

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

7.2.2.1.3 **Glass plate**, approximately 750 mm × 500 mm × 10 mm.

7.2.2.2 Calibration

7.2.2.2.1 Switch on the instrument and adjust in accordance with the manufacturer's instructions. Clean the glass plate (7.2.2.1.3) with a tissue to remove any dust particles and film. Using a clean palette knife (5.4), place a portion of one of the mulled pastes with 100 % calibration tint value (see 7.1.10) at the top edge of the glass plate and smear it almost to the bottom edge. Using the film applicator (7.2.2.1.2), draw the paste down to the bottom edge in about 2 s to 3 s.

7.2.2.2.2 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.

7.2.2.2.3 Make a duplicate drawdown of the duplicate paste as in 7.2.2.2.1. 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 $3,0 \pm 0,02$ is obtained from the duplicate paste.

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

7.2.2.2.4 If these pastes are acceptable, blend the two together using a palette knife (5.4).

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

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

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 International Standard and does not constitute an endorsement by ISO of the apparatus named.

7.2.2.2.7 Determine the reflectance values of the remaining calibration pastes (see 7.1.10), taking three readings with each paste.

7.2.2.2.8 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.

7.2.2.2.9 Periodically, check the proper calibration and standardization of the equipment, reagents, materials and method, as required, using standard reference blacks (4.4).

7.2.2.3 Determination of tinting strength of a test sample

7.2.2.3.1 Prepare duplicate pastes of 100 % calibration tint value as described in 7.1.1 to 7.1.10.

7.2.2.3.2 Prepare the drawdowns and set the instrument to 3,0 as described in 7.2.2.2.1 to 7.2.2.2.6.

7.2.2.3.3 Using the correct mass of test sample (see 7.1.11), prepare duplicate pastes as described in 7.1.1 to 7.1.9.

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

7.2.2.3.5 Determine the tinting strength of the test sample by using the graph or equation determined in 7.2.2.2.8.

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

7.2.3.1 Apparatus (in addition to that specified in 5.1 to 5.5)

7.2.3.1.1 Densichron reflectometer, for the film drawdown method.

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

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

7.2.3.1.4 Tint roller spreader, for the roller spreader method.

7.2.3.2 Calibration

7.2.3.2.1 Film drawdown method

7.2.3.2.1.1 Clean the glass plate (7.2.3.1.3) with a tissue to remove any dust particles and film. Using a clean palette knife (5.4), place a portion of one of the mulled pastes with 100 % calibration tint value (see 7.1.10) at the top edge of the glass plate and smear it almost to the bottom edge. Using the film applicator (7.2.3.1.2), draw the paste down to the bottom edge in about 2 s to 3 s.

7.2.3.2.1.2 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.

7.2.3.2.1.3 Make a duplicate drawdown of the duplicate paste as in 7.2.3.2.1.1. 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 $80 \pm 0,5$ is obtained from the duplicate paste.

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

7.2.3.2.1.4 If these pastes are acceptable, blend the two together using a palette knife (5.4).

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

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

7.2.3.2.1.7 Determine the reflectance values of the remaining calibration pastes (see 7.1.10), taking three readings with each paste.

7.2.3.2.1.8 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.

7.2.3.2.1.9 Periodically check the proper calibration and standardization of the equipment, reagents, materials and method, as required, using standard reference blacks (4.4).

7.2.3.2.2 Roller spreader method

7.2.3.2.2.1 Using a clean palette knife (5.4), place a portion of one of the milled pastes of 100 % calibration tint value (see 7.1.10) on the clean turning roller of the tint roller spreader (7.2.3.1.4). 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.

7.2.3.2.2.2 The pastes are acceptable if readings of $80 \pm 0,5$ are obtained.

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

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

7.2.3.2.2.5 Determine the reflectance values of the remaining calibration pastes (see 7.1.10).

7.2.3.2.2.6 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.

7.2.3.2.2.7 Periodically check the proper calibration and standardization of the equipment, reagents, materials and method, as required, using standard reference blacks (4.4).

7.2.3.3 Determination of tinting strength of a test sample

7.2.3.3.1 Prepare duplicate pastes of 100 % calibration tint value as described in 7.1.1 to 7.1.10.

7.2.3.3.2 Prepare the drawdowns and set the instrument to 80 as described in 7.2.3.2.1.1 to 7.2.3.2.1.6 or 7.2.3.2.2.1 to 7.2.3.2.2.4.

7.2.3.3.3 Using the correct mass of test sample (see 7.1.11), prepare duplicate pastes as described in 7.1.1 to 7.1.9.

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

7.2.3.3.5 Determine the tinting strength of the test sample by using the graph or equation determined in 7.2.3.2.2.6.

7.2.4 Measurements using the Meeco Colormaster

7.2.4.1 Apparatus (in addition to that specified in 5.1 to 5.5)

7.2.4.1.1 Meeco Colormaster.

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

7.2.4.1.3 Glass slides, of dimensions 75 mm × 50 mm × (1,22 mm ± 0,05 mm).

7.2.4.1.4 Glass rod, approximately 6,5 mm × 250 mm.

7.2.4.1.5 Slide preparation apparatus (see Figure 1).

7.2.4.2 Calibration

7.2.4.2.1 Clean a glass slide (7.2.4.1.3) with a tissue to remove any dust particles and film. Using a clean palette knife (5.4), place a small portion of one of the mulled pastes with 100 % calibration tint value (see 7.1.10) along the edge of the slide.

7.2.4.2.2 Place the slide in the slide preparation plate with the paste at the open end (see Figure 1). Draw the glass rod (7.2.4.1.4) across the test sample from the open end towards the closed end three times, uniformly spreading the paste on to the slide.

NOTE It is not necessary to cover the full length of the slide. The glass rod resting on the two slide strips of masking tape gives a film thickness of 0,5 mm (see Figure 1).

7.2.4.2.3 Use the green filter only. Place the prepared slide (see 7.2.4.2.2), with the paste side up, centrally over the left front hole and immediately read the reflectance. Make a duplicate slide with the duplicate paste in the way specified in 7.2.4.2.1 and 7.2.4.2.2 and measure the reflectance from the slide. The pastes are acceptable if their reflectances do not differ by more than ± 0,6 %.

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

7.2.4.2.5 Blend together whichever two of these three pastes do not differ by more than 0,6 %.

7.2.4.2.6 Determine the reflectance values of the remaining calibration pastes (see 7.1.10).

7.2.4.2.7 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.

7.2.4.2.8 Periodically check the proper calibration and standardization of the equipment, reagents, materials and method, as required, using standard reference blacks (4.4).