INTERNATIONAL STANDARD

ISO 5435

Fourth edition
2008-11-15

Rubber compounding ingredients — Carbon black — Determination of tinting strength

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

Reference number
ISO 5435:2008(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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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.

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.

2 Normative references

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 cm^3.

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).

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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 °C ± 2 °C and (50 ± 5) % relative humidity or 27 °C ± 2 °C and (65 ± 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 g ± 1 mg of the ITZnO.

7.1.3 Using the syringe (5.5), place 2,2 cm^3 of the plasticizer (4.1) in the centre of the lower glass plate of the automatic muller (5.2). 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.

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 ± 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.

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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 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.

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 ± 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 mulled 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 ± 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 %.

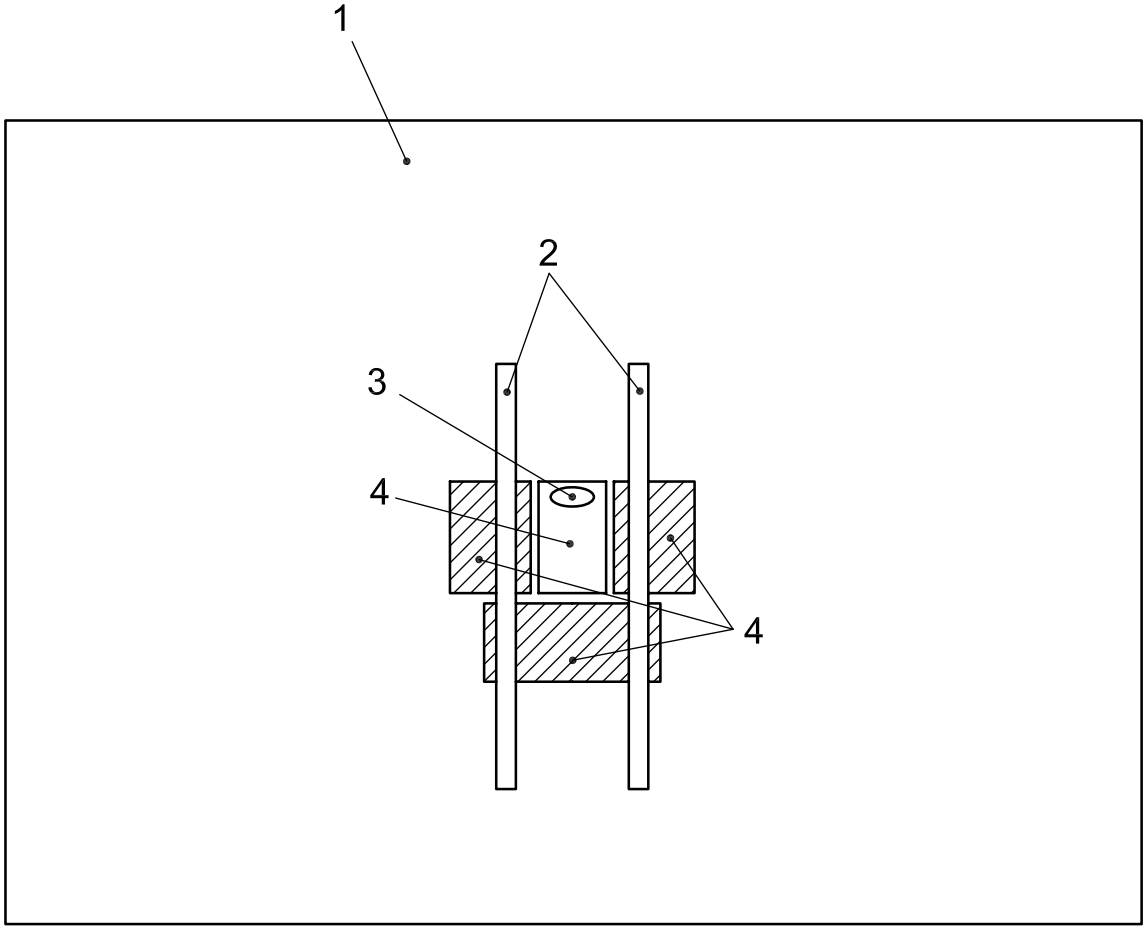
7.2.4.2.4 If the pastes are not acceptable, prepare another paste containing 0,100 g of ITRB and check using the same procedure.

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).



Key

1 glass plate (750 mm × 500 mm × 10 mm)
2 double layer of adhesive tape (thickness 0,5 mm)
3 paste
4 glass slides (thickness 1,22 mm)

Figure 1 — Slide preparation apparatus for Meeco Colormaster

7.2.4.3 Determination of tinting strength of a test sample

7.2.4.3.1 Prepare duplicate pastes of 100 % calibration tint value as described in 7.1.1 to 7.1.10 and, using the method in 7.2.4.2.3, take a reading for each.

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

7.2.4.3.3 Take readings from these pastes as described in 7.2.4.2.3 and calculate the tinting strength using the graph or equation determined as in 7.2.4.2.7.

7.2.5 Measurements using the Hunter Miniscan

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

7.2.5.1.1 Hunter Miniscan [7], model XE or XE Plus.

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

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

7.2.5.2 Calibration

7.2.5.2.1 Switch on the instrument and adjust in accordance with the manufacturer's instructions.

7.2.5.2.2 Calibrate the instrument using the black and white tiles.

7.2.5.2.3 Through the setup mode, select the following:

— CIE XYZ colour scale;

— D65 illuminant;

— 10° observer;

— average of three readings.

7.2.5.2.4 Clean the glass plate (7.2.5.1.3) 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 (7.2.5.1.2), draw the paste down to the bottom edge in about 2 s to 3 s.

7.2.5.2.5 Read the standard paste three times, taking all the readings at 75 mm or more from the top of the drawdown. The Y-value is the one of interest as it refers directly to lightness and darkness. The average Y-value for the 0,100 g paste should be approximately 2,60. If the results are variable, gather up the paste and make another drawdown.

7.2.5.2.6 Make a duplicate drawdown of the duplicate paste as in 7.2.5.2.4. 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 the average Y-value does not differ by more than ± 0,02 units from the average Y-value of the first paste (7.2.5.2.5).

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

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

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

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

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

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7) The Hunter Miniscan® is the trade name of an apparatus supplied by Hunter Associates Laboratory, 11491 Sunset Hills Road, Reston, VA 20190-5280, USA. 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.

7.2.5.2.11 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.

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

7.2.5.3 Determination of tinting strength of a test sample

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

7.2.5.3.2 Prepare the drawdowns and measure the reflectance values as described in 7.2.5.2.4 to 7.2.5.2.9.

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

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

7.2.5.3.5 Determine the tinting strength of the test sample by using the graph or equation determined in 7.2.5.2.11.

7.2.6 Measurements using the Photochron

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

7.2.6.1.1 Photochron [8], model TR-200P.

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

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

7.2.6.2 Calibration

7.2.6.2.1 Switch on the instrument and adjust in accordance with the manufacturer's instructions. Clean the glass plate (7.2.6.1.3) 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 (7.2.6.1.2), draw the paste down to the bottom edge in about 2 s to 3 s.

7.2.6.2.2 Place the reflectometer head on the drawdown of the standard paste. Adjust the meter reading to read 100 % for at least three readings, all taken at 75 mm or more from the top of the drawdown.

7.2.6.2.3 Make a duplicate drawdown of the duplicate paste as in 7.2.6.2.1. 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 100 ± 0,6 is obtained from the duplicate paste.

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

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

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8) The Photochron® is the trade name of an apparatus supplied by Tokyo Denshoku Co., Ltd., 3, Katamachi, Shinjuku-ku, Tokyo 160-0001, Japan. 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.

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

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

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

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

NOTE The equation is not necessarily linear.

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

7.2.6.3 Determination of tinting strength of a test sample

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

7.2.6.3.2 Prepare the drawdowns and set the instrument to 100 % as described in 7.2.6.2.1 to 7.2.6.2.6.

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

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

7.2.6.3.5 Determine the tinting strength of the test sample by using the graph or equation determined in 7.2.6.2.8.

8 Expression of results

Express the percentage tint value to the nearest 0,1 tint units.

If the calibration graph is a straight line, or if a linear equation determined by the method of least squares gives results within 0,8 units of the calibration values, then the following equation can be used:

$$T = \frac{R}{S} \times 100$$

where

T is the percentage tint value;

R is the reflectance of the paste with 100 % calibration tint value;

S is the reflectance of the test sample.

For N500, N600 and N700 series carbon blacks, where 0,2 g of test sample has been used, divide the result by 2. However, if a statistical equation is used to correct measured tint values, the values for N500, N600 and N700 series carbon blacks shall be corrected before dividing the result by 2.

9 Precision

See Annex A.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the identification of the sample;

c) the temperature used for drying the zinc oxide and the carbon black;

d) the reflectance instrument used;

e) the method of preparing the paste for measurement;

f) the results of replicate tests;

g) any deviation from the procedures specified in this International Standard;

h) the date of testing.

Annex A
(informative)

Precision statement

A.1 Background

An interlaboratory test programme (ITP) to determine the precision of the method specified in this International Standard was conducted in 2005, using the procedures and guidelines described in ISO/TR 9272:2005.

The precision of the tinting strength measurement method was determined using three different carbon black grades, with a range of values, supplied to each of the 17 laboratories participating in the ITP. Each carbon black sample was tested four times in total (twice on two different days). Four different light-measuring instruments (Miniscan, Erichsen, Densichron and Photochron) were used for the ITP and the results obtained showed that no significant differences could be attributed to the instrument used.

The precision results obtained by this ITP should not be applied to acceptance or rejection testing of any group of materials without documentation that the results obtained from the ITP actually apply to the materials tested.

A.2 Precision results

A.2.1 General

The precision results are given in Table A.1, with the carbon black grades listed in order of increasing tinting strength. These results were obtained after removal of statistical outliers when calculating the mean and the standard deviation. General statements for the use of the precision results are given in A.2.2 and A.2.3. They are given in terms of both the absolute precision, r and R, and the relative precision, (r) and (R).

Table A.1 — Precision for determination of tinting strength

Material	Mean tinting strength	Within laboratory			Between laboratories			Number of laboratories retained (out of 17)
		s_r	r	(r)	s_R	R	(R)	
N772	58,7	0,29	0,81	1,38	0,93	2,62	4,46	15
N326	108,9	0,36	1,02	0,94	0,96	2,73	2,50	15
N134	129,5	0,41	1,15	0,89	1,49	4,22	3,26	13
	Grand mean	—	1,00	1,07	—	3,19	3,41	—

s_r is the within-laboratory standard deviation (in measurement units);

r is the repeatability (in measurement units);

(r) is the relative repeatability, as a percentage of the mean value;

s_R is the between-laboratory standard deviation (in measurement units);

R is the reproducibility (in measurement units);

(R) is the relative reproducibility, as a percentage of the mean value.

A.2.2 Repeatability

The repeatability, or local domain precision, for each material is given in Table A.1. Two individual test results obtained in the same laboratory (by the proper use of this International Standard) that differ by more than the tabulated values for r, in measurement units, and (r), as a percentage, should be considered suspect, i.e. to have come from different populations, and should suggest that some appropriate investigative action be taken.

A.2.3 Reproducibility

The reproducibility, or global domain precision, for each material is given in Table A.1. Two individual test results obtained in different laboratories (by the proper use of this International Standard) that differ by more than the tabulated values for R, in measurement units, and (R), as a percentage, should be considered suspect, i.e. to have come from different populations, and should suggest that some appropriate investigative action be taken.

A.2.4 Bias

Bias is the difference between a measured average test result and a reference, or true, value for the measurement in question. Reference values do not exist for this test method and therefore bias cannot be determined.

Bibliography

[1] ISO/TR 9272:2005, *Rubber and rubber products — Determination of precision for test method standards*

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