Designation: D2663 – 14

Standard Test Methods for Carbon Black—Dispersion in Rubber[1]

This standard is issued under the fixed designation D2663; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the degree of dispersion of carbon black in rubber. Four test methods are described as follows:

	Sections
Test Method A—Visual Inspection	3 – 11
Test Method B—Agglomerate Count	12 – 22
Test Method C—Microroughness Measurement with Profilometer	23 – 33
Test Method D—Microroughness Measurement with IFM	34 – 42

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

2.2 *ASTM Adjuncts:*

Carbon Black Dispersion Standards[3]

Carbon Black Dispersion Chart[4]

TEST METHOD A—VISUAL INSPECTION

3. Scope

3.1 Test Method A is a qualitative visual test method. Ratings are made against a set of standard photographs (Fig. 1),[3] and the results are expressed on a numerical scale. This test method cannot be used for compounds that contain fillers other than carbon black.

4. Summary of Test Method

4.1 The compound rubber is torn or cut to expose a fresh surface for examination by the eye, aided preferably by a hand lens or a low-power binocular microscope. The dispersion level of the carbon black is compared against a series of five photographic standards and then rated numerically from 1 (very low) to 5 (high) (see Fig. 1).

5. Significance and Use

5.1 Visual dispersion ratings correlate with certain important physical properties of the compound. A rating of 5 indicates a state of dispersion developing near maximum properties, while a rating of 1 would indicate a state of dispersion developing considerably depressed properties. Normally, the visual dispersion ratings indicate the following levels of compound quality:

Visual Dispersion Rating	Classification
4 to 5	High
3 to 4	Intermediate
2 to 3	Low
1 to 2	Very low

6. Apparatus

6.1 *Sharp Knife or Razor Blade.*

6.2 *Hand Lens* (10×) or binocular microscope (10 to 20×).

6.3 *Illuminator,* microscopical-type.

6.4 *Knife Heater.*

6.5 *Series of Photographic Standards,* rating 1 to 5. These standards give the following percent dispersion ratings by the Agglomerate Count Method:

Visual Rating	Black Dispersed, %
1	70
2	80
3	91
4	96
5	99

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[1] These test methods are under the jurisdiction of ASTM Committee D24 on Carbon Black and are the direct responsibility of Subcommittee D24.71 on Carbon Black Testing in Rubber.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from ASTM International Headquarters. Order Adjunct No. ADJD266302. Original adjunct produced in 1967.

[4] Available from ASTM International Headquarters. Order Adjunct No. ADJD266301. Original adjunct produced in 1967.

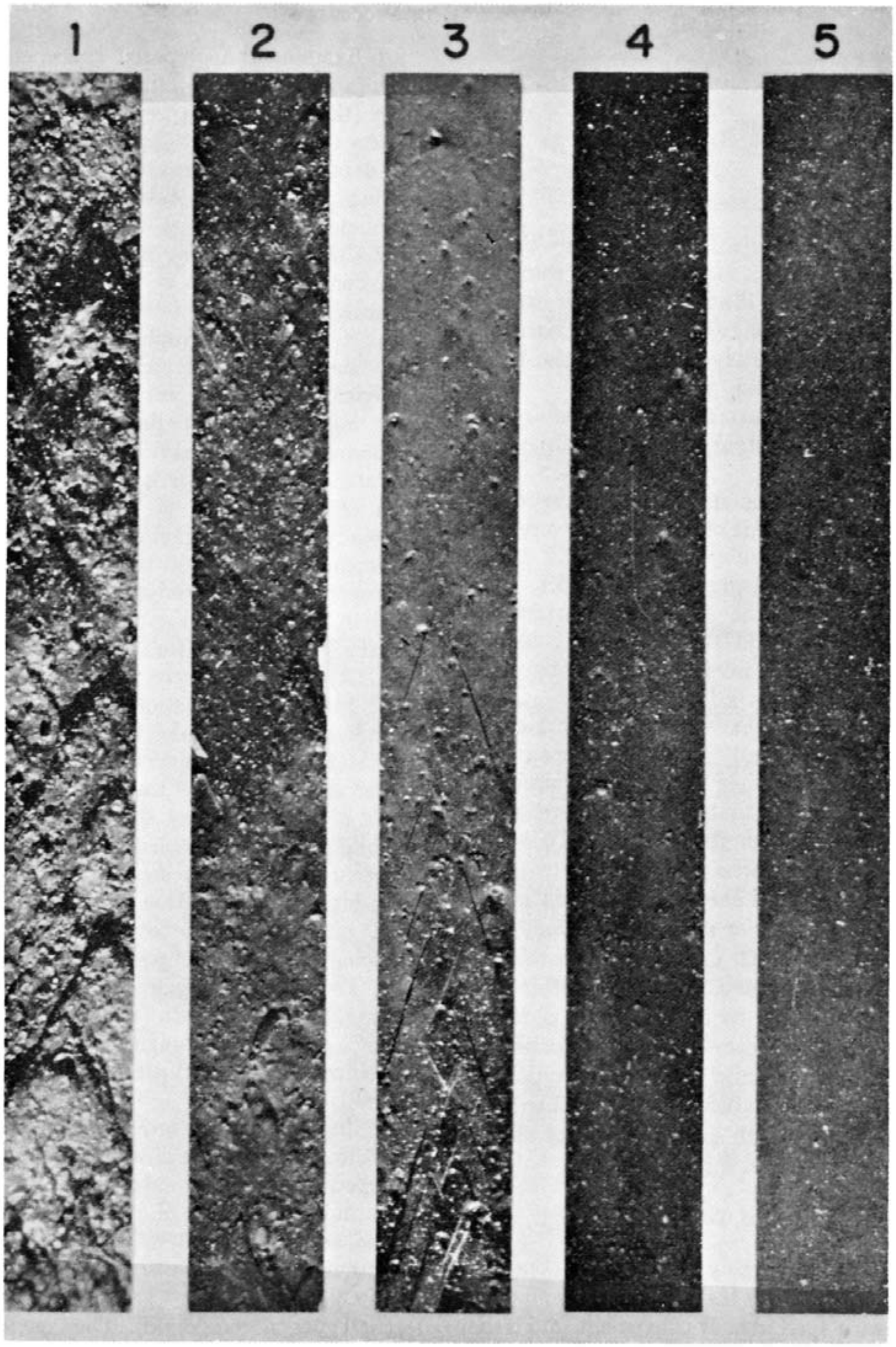

FIG. 1 Carbon Black Dispersion Standards—Visual Analysis of Torn Vulcanizates

7. Test Specimen

7.1 *Vulcanized Compounds*—Use a slab of rubber about 2 mm in thickness. Tear it so that a fresh surface is exposed. The tear may be initiated by a small cut. The most nearly flat part of the tear is used for rating.

7.2 *Unvulcanized Compounds*—Unvulcanized rubber may be examined as follows:

7.2.1 If the specimen contains curing agents, sheet it out and cure in a press to form a vulcanized slab about 2 mm in thickness. Mill and cure in accordance with Practice D3182. Then proceed as in 7.1.

7.2.2 If the specimen contains no curatives, add the appropriate materials with a minimum of mixing. Then cure and proceed as above.

7.2.3 If the specimen contains no curatives and a dispersion evaluation with no further mixing is required, the compound must first be compressed to remove most of the air holes. To accomplish this, press the rubber into a slab between thin sheets of plastic in a mold at a pressure of about 1.03 kPa for 5 min at 105°C. Care should be taken to avoid excessive flow during this step. The surface to be examined is formed with a smooth cutting stroke using a sharp, hot knife (a standard type knife heater may be employed). The most nearly smooth and flat part of the cut surface is used for rating.

8. Number of Tests

8.1 Preferably more than one test (on different tears) should be made for each specimen. If convenient, more than one operator should rate the samples.

9. Procedure

9.1 Examine the prepared specimens under a hand lens or binocular microscope (the latter being preferred), with oblique illumination to accentuate surface detail. Keep the magnification and lighting conditions constant for all specimens.

9.2 Compare the size and frequency of carbon agglomerates in the specimens (showing up as surface bumps or depressions) to the photographic standards. Then assign the most closely matched numerical rating to each compound being rated. In borderline cases, use fractional ratings, for example, 3½ would indicate a rating between 3 and 4. In cases of dissimilarity in the size and frequency of the agglomerates in the specimen and those of the standards, the operator shall assign the rating that in his judgment is most applicable. Certain compounds (for example, NR and IR) are particularly prone to very small black agglomerations which are difficult to resolve by the Visual Inspection Method. In instances of high agglomerate frequency, the surface of stocks of this type may show a general roughness or fine pebbled appearance. Differences are best resolved at somewhat higher magnification (for example, 20×, binocular microscope). If at all possible, examine compounds of this type also by the agglomerate count method, at least until sufficient experience is gained to recognize dispersion differences with the Visual Inspection Method.

9.3 In comparing a series of different compounds, it is also desirable to rate the specimens side by side rather than one at a time. This use of a control compound is also advisable. This is best prepared by individual operators, since dispersion requirements may vary greatly for different types of compounds. The control sample should represent a minimum acceptable dispersion level for the type of compound being rated. Because it can be observed side by side with unknown samples under identical conditions, a control compound is more accurate than the photographic standards in discerning small deviations from what is considered the norm for a specific type of compound. Prepare a fresh surface on the control as often as necessary to ensure cleanliness.

10. Report

10.1 *Ratings:*

10.1.1 List all ratings, including those on any control compound, on the basis of the 1 to 5 scale defined by the standard photographs. Use fractional ratings when necessary.

10.1.2 Average the ratings on different specimens of the same compound as well as the ratings of different operators. Report the final average values.

10.2 *Compound Identification:*

10.2.1 *Formulation*—Whenever possible list the following:

10.2.1.1 Carbon black, type and loading,

10.2.1.2 Other fillers, type and loading,

10.2.1.3 Polymer type, and

10.2.1.4 Extender oil, type and loading.

10.2.2 *Mixing*—Describe the mixing of the compound in terms of one or more of the following:

10.2.2.1 Standard mixing procedure,

10.2.2.2 Type of equipment,

10.2.2.3 Masterbatch,

10.2.2.4 Finished compound (vulcanized), and

10.2.2.5 Finished compound (unvulcanized).

11. Precision and Bias

11.1 No statement is made about either the precision or the bias of Test Method A since the result is qualitative and not applicable to statistical treatment.

TEST METHOD B—AGGLOMERATE COUNT

12. Scope

12.1 Test Method B is a quantitative test method. Dispersion is evaluated by measuring with a light microscope the percentage area covered by black agglomerates in microtomed sections of the compound. Since this test method involves direct measurement, it is quantitative and more accurate than the visual test method. The test is applicable to the analysis of carbon black dispersion in compounds that contain other fillers.

13. Summary of Test Method

13.1 The compounded rubber is microtomed into sections sufficiently thin to permit observation of the carbon agglomerates by transmitted light, with the aid of a light microscope. The total cross-sectional area of all agglomerates 5 µm or larger is counted, and from the known content of carbon black in the stock, the percentage of carbon black below the 5-µm size is calculated and expressed as "Percentage of Carbon Black Dispersed."

14. Significance and Use

14.1 Certain important physical properties of the compound are influenced significantly by the degree of carbon black dispersion within the compound (for example, tensile strength and abrasion resistance). The correlation of these properties with the percentage dispersion determined by the Agglomerate Count Method approximates the following pattern for many types of black loaded rubber compounds:

Dispersion, %	Classification
Above 99	Very high
97 to 99	High
95 to 97	Intermediate
92 to 95	Low
Below 92	Very low

FIG. 2 Rotary Microtome with Cryogenic Attachment for Sectioning Rubber Specimens

15. Apparatus

15.1 *Microtome*—A rotary microtome[5] capable of producing sections from samples up to 3 mm in cross-section and 1 cm in length. Tungsten carbide knives are recommended. (See Fig. 2.)

15.2 *Cryogenic Cooling Unit*—A cryogenic cooling attachment for the above rotary microtome[6] capable of cooling the sample to –160°C. (See Fig. 2.)

15.3 *Microscope*—An optical microscope with binocular viewing and digital image capture is recommended. This should include a movable specimen stage and white light source with variable intensity. Lenses should include two 10× wide field eyepieces and objectives in the range from 6 to 10×. Taking into account microscope tube corrections, objectives should be selected so that magnifications in the range from 75 to 100× are available. (See Fig. 3.)

15.4 *Computer*—A computer should be available and interfaced to the digital camera on the microscope to capture digital photomicrographs of the specimens. (See Fig. 3.)

15.5 *Image Analysis Software*—Suitable image analysis software to allow thresholding of the captured micrographs, conversion of the thresholded image to binary and area fraction determination from the binary images. Examples of this type of software include, but are not limited to, Image J, ImagePro, NIH Image, IDL, and NIST Lispix.

15.6 *Razor Blades.*

15.7 *Sable Brushes* (00).

15.8 *Microscope Slides and Cover Glasses.*

16. Reagents and Materials

16.1 *Liquid Nitrogen.*

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[5] Example, Leica RM2265.

[6] Example, Leica LN22.

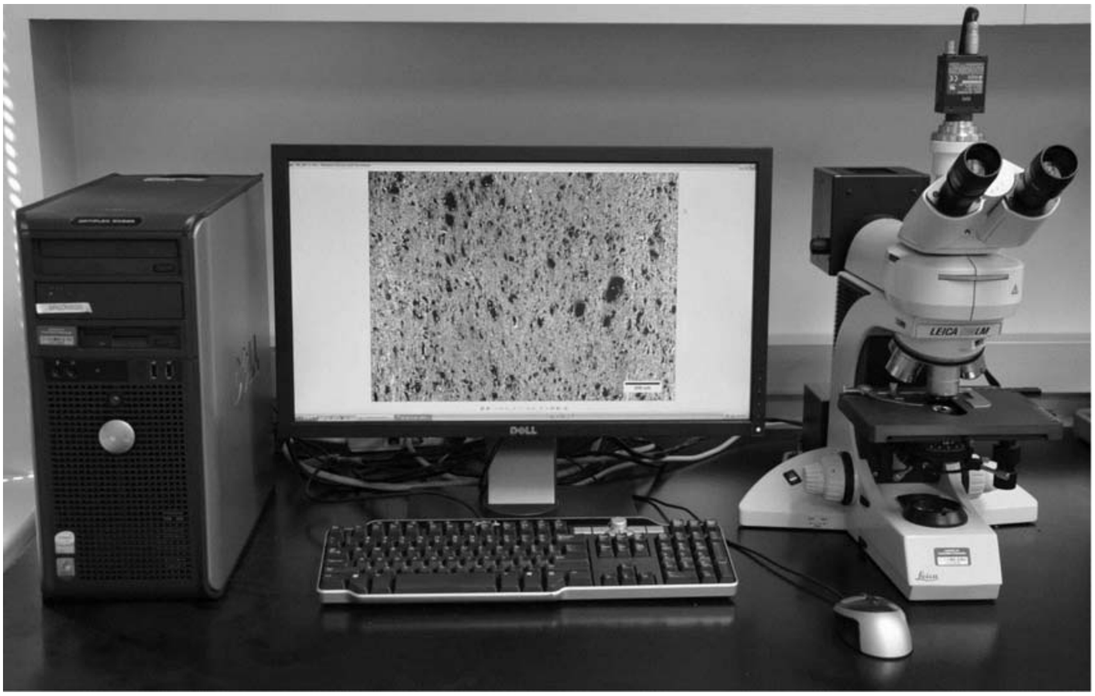

FIG. 3 Light Microscope Equipped with Digital Camera and Computer System

16.2 *Organic Solvents*—Appropriate organic liquid to aid in flattening section onto the glass microscope slides. Examples include xylenes, toluene, and methanol.

17. Sampling

17.1 *Vulcanizates*—Specimens may be cut from standard test sheets (about 2-mm thick) or from pieces of actual cured articles. Vulcanized samples must be employed because of the solvent used to uncurl the thin sections. If pieces other than 2-mm sheets are used, they should first be cut down to a thickness of about 2 to 3 mm.

17.2 *Unvulcanized Compounds*—For rubbers of high unsaturation (for example, OE-SBR, NR, and BR), dust small bits (enough subsequently to form buttons about 10 mm in diameter and about 2 to 3-mm deep) thoroughly with dicumyl peroxide. Cure in a button mold[7] under high pressure at about 155°C. OE-SBR rubbers require about 30 to 60-min cure. BR requires about 10 to 15-min cure. After cure, scrape off the excess peroxide from the sample surface and proceed with sectioning in the standard manner, taking care not to pare down below the cured surface layer.

17.2.1 For IIR, satisfactory surface cures can be obtained with a mixture of 1 part tetramethylthiuram disulfide (TMTD), 1 part mercaptobenzothiazole (MBT), 1 part sulfur, and 5 parts zinc oxide, with a cure of 1 h at 155°C. Other alternative approaches for curing high unsaturation polymers without actually mixing in curatives are (*1*) high-energy radiation and (*2*) chemical treatment with sulfur monochloride. However, before using either of these latter methods, the stock should be pressed out to eliminate most of the air holes. Cure in accordance with Practice D3182.

18. Test Specimen

18.1 Cut out a specimen approximately 1 cm long, 1 cm wide, and approximately 2-mm deep.

18.2 Cut the square block into a trapezoidal shape that will fit the sample chuck on the rotary microtome.

18.3 Prepare one specimen block for each different compound to be examined.

19. Procedure

19.1 *Microtome Preparation*—Turn on the rotary microtome, insert the knife into the microtome and adjust to the correct cutting angle (see microtome manufacturer instructions). Fill the liquid nitrogen dewar and attach to the cryogenic chamber on the microtome. Cool the microtome chamber and knife holder.

19.2 *Sample Preparation*—Insert the prepared specimen block into the microtome chuck and insert the chuck into the microtome such that the long axis of the specimen is parallel to the cutting direction. Cool the sample to approximately 50°C below the elastomer glass transition temperature.

19.3 *Microtome Operation*—Manually advance the specimen so that the cutting face almost reaches the knife. At this point, with the advance set in increments of 5 to 10 µm, start microtoming until the specimen is faced level and full-size sections are being cut.

19.4 *Cutting Thin Sections*—After facing is complete, set the microtome control to the appropriate thickness depending

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[7] A special mold containing several circular cavities that are approximately 10 mm in diameter and 3 mm deep.

on the carbon black loading. For standard elastomer compounds a thickness of 1 to 2 µm is a good starting point. Cut 4 to 5 sections, which will likely roll up, and allow the sections to collect on the back side of the knife and knife holder.

19.5 *Mounting Sections on Microscope Slides*—Using a clean, dry sable brush transfer a section from the knife block to a clean microscope slide placed on the edge of the microtome cryo-chamber. The section will be curled up in a small tight roll and should adhere to the brush with static electricity. Using a second sable brush, add a few drops of the organic liquid to the section. With careful manipulation of the solvent wet brush, unroll and spread the section out flat on the slide. An additional brush or small pointed stick may be helpful to roll out the section. Continue brushing gently to remove all wrinkles. Small amounts of additional solvent may be added as needed.

19.6 Repeat steps 19.4 and 19.5 until a sufficient number of sections have been brushed out. Then cover the sections with cover glasses or another glass microscope slide, and seal with tape, or a bit of cement at each corner.

19.7 *Preparing for Counting*—Inspect the sections for quality under the light microscope, and select one that is relatively free of wrinkles, holes, and knife marks. Also avoid sections that are very thin as some of the clumps of carbon black may be brushed out. If the sections are too thick or have too many wrinkles, holes or knife marks, adjust the microtome accordingly and produce additional sections.

19.8 Once good sections are obtained, remove the specimen from the microtome and measure the length and width of the faced block where the sections were obtained. The product of these dimensions is the area before swelling. Also, measure the length and width of a few of the sections mounted on the glass slides. Average these dimensions and their product is the section area after swelling. Record this value along with the sample area before swelling.

19.9 *Micrograph Acquisition*—Place the slides in the light microscope in transmission mode and select the magnification. Magnification should be in the range from 75 to 100× but the exact figure is left to the discretion of the individual operator, based on the specifications of his own particular microscope and lens system. Within the limits of 75 to 100×, the percent dispersion rating on a given section will not change significantly, provided that sampling is adequate. However, magnification should be kept constant in comparing and classifying agglomerate size within different samples. Adjust the lighting and exposure conditions to obtain good images and acquire ten non-overlapping images showing the carbon black agglomerates in the elastomer matrix (Fig. 4). Save the micrographs in a non-lossy (uncompressed image in order not to lose micrograph information) file format.

19.10 *Micrograph Analysis*—In an appropriate image analysis software package, open the first micrograph. To analyze the images, the first step is to threshold the image such that the carbon black aggregates are isolated from the background (usually brown in color). Care should be taken to minimize the number of defects (knife marks, folds, etc.) that are included in the area selected by the threshold operation. Once the threshold is complete, a binary image will be generated (Fig. 4). Using the appropriate software tool, the agglomerates greater than 5 µm in size should be counted and a total area fraction of these agglomerates calculated. Repeat this analysis for each image and average the ten area fraction values together to obtain the overall agglomerate area fraction.

20. Calculation and Interpretation of Results

20.1 *Percent Dispersion*—Calculate the percent dispersion, representing the percentage of carbon black that has been dispersed below the 5-µm agglomerate size, as follows:

$$\text{Dispersion, \%} = 100 - SU/L$$

where:

U = agglomerate area fraction. (This represents an average of the ten area fraction measurements on the sections. See Note 1.)

NOTE 1—Most agglomerates are not composed entirely of carbon black. They may contain substantial amounts of polymer or extender oil. In extreme cases, where U is very large, negative dispersion ratings are therefore possible. Such stocks are extremely poor and may simply be classified at a "0" or "no dispersion" rating. It must also be assumed that

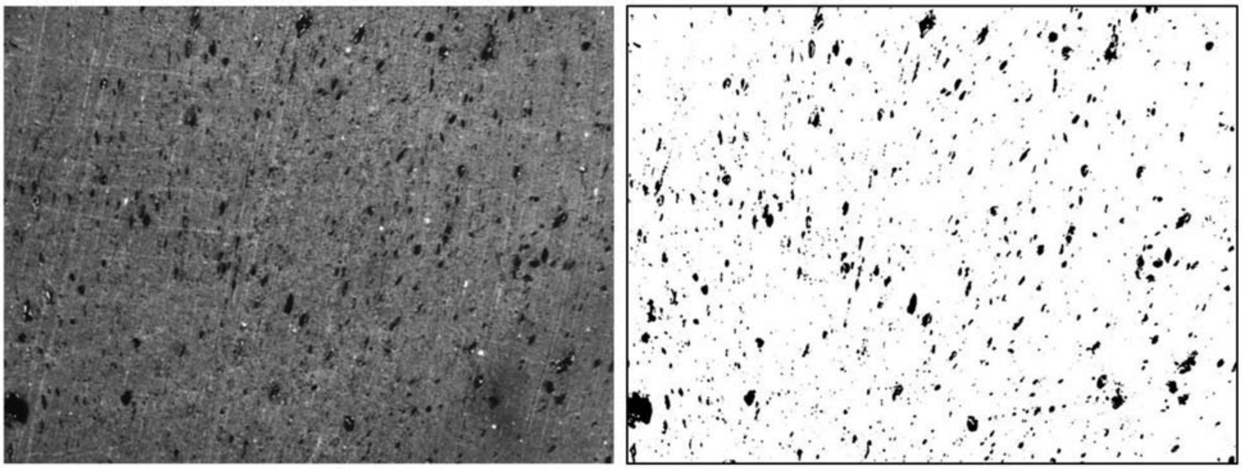

FIG. 4 Left: Light micrograph showing the carbon black agglomerates (dark regions) in a rubber sample. Right: The binary image produced from the micrograph after thresholding to isolate the carbon black agglomerates

the absolute level of all the percent dispersion values is probably higher than reported. There is no satisfactory test method presently available for determining the precise amount of carbon black in each agglomerate.

S = area swelling factor from the action of the solvent used to uncurl the sections (a ratio of the section area after swelling to the area before swelling), and
L = volume percentage of black in the compound.

For maximum accuracy, the black volume percentage can be calculated from the following expression:

$$L_1 = \frac{\text{density of compound} \times \text{mass of black}}{\text{density of black} \times \text{total mass of compound}} \times 100$$

However, when dealing with hydrocarbon rubbers, for practical purposes the density of the carbon black can simply be considered as being twice that of the polymer and oil, and the weight contribution of the curing agents can be disregarded. Then, the volume percentage of black can be calculated from the following simplified expression where:

$$L_2 = \frac{\text{mass of black}}{\text{mass of black} + 2 \times (\text{total mass of polymer} + \text{oil})} \times 100$$

20.1.1 In dealing with rubbers such as SBR, NR, BR, IIR, and EPDM, the two different test methods for calculating percent black volume produce negligible differences in the final values for percent dispersion. However, for halogenated hydrocarbons such as CR or nonhydrocarbons such as silicone rubber, the actual density of the polymer should be taken into consideration.

21. Report

21.1 *Measured Percent Dispersion Values*—Express measured dispersion ratings to the nearest 0.1 %.

21.2 *Measured Area Fraction Values*—Report the average agglomerate area fraction to the nearest 0.1 %.

21.3 *Compound Identification*—Whenever possible list pertinent information regarding the following:
21.3.1 *Formulation:*
21.3.1.1 Carbon black, type and loading,
21.3.1.2 Other fillers, type and loading,
21.3.1.3 Polymer type, and
21.3.1.4 Extender oil, type and loading.
21.3.2 *Mixing*—Describe the mixing of the compound in terms of one or more of the following:
21.3.2.1 A standard mixing procedure,
21.3.2.2 Type of equipment,
21.3.2.3 Masterbatch, and
21.3.2.4 Finished compound.

22. Precision and Bias

22.1 Due to limited use, a precision and bias statement for Test Method B cannot be determined.

TEST METHOD C—MICROROUGHNESS MEASUREMENT WITH PROFILOMETER

23. Scope

23.1 Test Method C is a quantitative test method. The cut surface of a rubber specimen is traced with a stylus which measures the amount of roughness caused by carbon black agglomerates. This test method is applicable to rubber compounds containing all types of carbon blacks over a wide range of loadings.

24. Summary of Test Method

24.1 The compounded rubber is cut to expose a fresh internal surface. This surface is traced with a fine stylus (2.5-μm radius tip, 200-mg force) which measures a roughness factor based on the number and average height of the surface irregularities (protrusions or depressions) caused by carbon black agglomerates. The measured roughness factor is used to derive a dispersion index which is expressed on the same scale (0 to 100) as Test Method B. The percent dispersion values obtained by Test Method B are used to establish the dispersion index scale for different rubber formulations.

25. Significance and Use

25.1 Certain important physical properties of the compound are influenced significantly by the level of carbon black dispersion (for example, tensile strength, abrasion resistance, and fatigue life). The correlations of these properties with the dispersion index determined by the microroughness measurement method exhibit the same pattern described for the agglomerate count method in 14.1.

26. Apparatus

26.1 *Dispersion Analyzer*[8]—A stylus microroughness measurement device which is also equipped with a specimen holder, sample cutter, and specimen tracking mount (Fig. 5).

26.2 *Vibration Isolation Slab,* about 66 by 51 cm and 4 cm deep is recommended for mounting the drive unit and the specimen tracking mount.

26.3 *Scissors.*

26.4 *Razor Blades,*[9]single edge (coated) stainless steel type, required for the specimen cutting device.

26.5 *Hand Lens* (10×).

26.6 *Freezer*—A standard refrigerator freezer unit (−5°C) is required for unvulcanized compounds.

26.7 *Logarithmic Graph Paper,*[8]special 2 × 3 cycle.

27. Sampling

27.1 *Vulcanizates*—Specimens may be cut from standard test sheets (about 2-mm thick) or from actual rubber products which can be cross-sectioned to a uniform thickness of about 2 to 3 mm.

27.2 *Unvulcanized Compounds*—Specimens may be prepared from rubber slabs sheeted out to a uniform thickness of 2 to 3 mm.

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[8] Formerly available from Mohr Federal, Inc., 1144 Eddy St., Providence, RI 02905. This equipment is no longer manufactured or supported.

[9] The sole source of supply of the apparatus known to the committee at this time is American Safety Razor Company, Industrial Products Div., Razor Blade Lane, Verona, VA 24482. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

FIG. 5 Components of Profilometer Dispersion Analyzer System

28. Test Specimen

28.1 Using a scissors, cut out a rectangular specimen that is approximately 3.5-cm long, 2-cm wide, and 0.2-cm deep. The longest dimension of the specimen should be cut along the direction in which the rubber slab was sheeted out.

28.2 Store unvulcanized specimens at about −5°C for a minimum of 30 min prior to testing.

29. Calibration

29.1 The dispersion analyzer drive unit must be leveled so that the stylus moves in a horizontal plane. Position the drive unit on the vibration isolator slab prior to this procedure.

29.1.1 Position the stylus to trace over a known flat surface which provides a suitable horizontal reference plane. A sheet of plate glass on the surface of the vibration isolator is suitable for this purpose.

29.1.2 Set the length of the trace at about 10 cm by positioning the steps on the side of the drive unit.

29.1.3 Press the high-speed (2.5 mm/s) switch and then activate the RUN switch on the control console. This will start the stylus tracking in an alternating in and out direction above the horizontal reference surface.

29.1.4 Lower the stylus by turning the control knob on top of the probe in a clockwise direction. Continue until the stylus makes contact with the reference surface. The position of the stylus is indicated as HIGH or LOW by an indicator LED on the right side of the vertical display on the control console. The HIGH and LOW designations refer to the pressure of the stylus on the surface.

29.1.5 Observe whether the stylus is HIGH or LOW during the trace and stop the drive unit at the extreme point by activating the HALT switch on the control console.

29.1.6 Correct the height of the stylus using the leveling knob at the top rear of the drive unit. Turn the leveling knob clockwise to move the stylus in the LOW direction and counterclockwise for HIGH. This adjustment must be coordinated with a correction in the opposite direction for the overall height of the stylus probe.

29.1.7 Activate the RUN switch on the control console and again observe the variations in stylus height across the reference surface. Repeat the leveling operation and complementing height correction until the indicator bar remains close to the center point between HIGH and LOW across the entire 10-cm trace.

30. Procedure

30.1 Turn on the power to the control unit and recorder.

30.2 Clear the profile switch (red indicator lamp should be off).

30.3 Stabilize the drive unit by operating in the RUN mode (no specimen) for 15 min prior to making the first roughness trace.

30.4 Enter the regression constants, *A* (slope) and *B* (intercept), for the dispersion index calculation. These constants are specific to individual formulations. If the constants are not available for the rubber formulation that is to be analyzed, see Section 31.

30.5 Set the roughness width cutoff at 0.80.

30.6 Enter the constant, *C*, for minimum roughness peak height. This constant eliminates high frequency electronic or vibrational noise which may be dependent on the location of the instrument. A value of $C = 0.7$ µm is typically used when the drive unit is mounted on a vibration isolation slab. Lower or higher values for C may be used at the discretion of the operator. This selection may depend on the type of rubber formulation or the size range of agglomerates that are pertinent to specific aspects of product performance.

30.7 Set the drive unit for a trace length of 2.0 cm.

30.8 Insert the rubber specimen into the specimen holder clamp. The longest dimension of the specimen should be parallel to the top edge of the clamp with about 8 to 10 mm of the specimen protruding above the clamp.

30.9 Mount the specimen holder over the alignment pins on the specimen cutting device. In the cutting position the clamp handle should be facing upright.

TABLE 1 Type 1—Method C Precision Results (Measured Dispersion Index)

Dispersion Index (average)	S_r	*r*	(*r*)	S_R	*R*	(*R*)
35.4	3.52	9.96	28.1	7.59	21.5	60.7
85.3	1.09	3.08	3.61	2.03	5.74	6.73
92.0	1.31	3.70	4.02	1.35	3.82	4.15
98.5	0.88	2.49	2.53	0.77	2.18	2.21

S_r = repeatability standard deviation (in measurement units),
r = repeatability (in measurement units),
(r) = repeatability (in relative percent),
S_R = reproducibility standard deviation (in measurement units),
R = reproducibility (in measurement units), and
(R) = reproducibility (in relative percent)

30.10 Insert a new razor blade into the specimen cutter with the cutting lever in the upright position.

30.11 Lower the cutting lever in a slow, smooth stroke until the razor blade has passed through the specimen. Remove the specimen holder from the cutter, and discard the used blade and the piece of rubber cut from the specimen.

30.12 Inspect the cut rubber surface on the specimen in the holder using a 10× hand lens. If the surface is uneven or contains any severe cutting artifacts, repeat the cutting operation with a new razor blade. The same specimen may be recut by readjusting the position of its exposed edge to a distance of about 8 to 10 mm above the top of the holder. This applies only to vulcanized specimens. Unvulcanized specimens should be recooled to −5°C prior to cutting.

30.13 Insert the specimen holder over the alignment pins in the tracking mount so that the cut surface of the specimen is on top.

30.14 Position and align the specimen holder so that the stylus will move lengthwise along the specimen in a path that is near the center (edge to edge) of the cut and which starts about 0.5 cm in from the end.

30.15 Set the tracking speed of the stylus for normal operation (0.25 mm/s).

30.16 Bring the stylus into contact with the surface of the specimen by adjusting the height control switch until the indicator bar is midway between the HIGH and LOW extremes. This setting will remain constant for subsequent specimens which can simply be mounted in place by gently lifting the stylus with a finger.

30.17 Activate the single cycle switch on the control console. The stylus will move outward 2.0 cm at a speed of 2.5 mm/s, pause briefly, and then start the trace of the specimen in an inward direction.

30.18 When the trace has been completed (80 s), record the measured values for dispersion index (DI), number of roughness peaks/cm, *F*, average roughness peak height, *H*, and roughness factor, F^2H.

30.19 Displace the mounted specimen laterally by about 0.2 mm and make a second roughness trace. Record the measurements and average the values for the first and second traces. These average values represent a single test result.

30.20 Repeat 30.8 through 30.19 for additional specimens of the same rubber formulation.

31. Calculation

31.1 The dispersion index (0 to 100 scale) and roughness measurements for each sample are printed on the recorder chart, and DI, *F*, and *H* may also be viewed directly on the control console. If the *A* and *B* constants for the DI calculation are unknown, however, they must be derived using a series of standard mixes which have been analyzed by Test Method B.

31.2 *Preparation of Standards*—Prepare a series of four different carbon black dispersion levels for the rubber formulation of interest by varying the total mixing energy or time.

The overall range of dispersion levels should be similar to the range of values listed in 6.5.

31.2.1 Measure or estimate the percent dispersion in each standard mix using Test Method B as described in Sections 12 through 22.

31.2.2 Measure the F^2H roughness factors for each standard mix using the procedures described in Sections 25 through 30.

31.3 *Derivation of Dispersion Index*—The dispersion index is calculated as follows:

$$\mathrm{DI} = 100 - 10\exp\left[\mathrm{A}\log F^2H + \mathrm{B}\right]$$

where:

F = the number of roughness peaks per cm, and
H = the number average peak height, µm.

A and B are constants for each specific rubber formulation and may vary with polymer type, carbon black type, black-oil loading, and state of cure. The values for dispersion index are inversely proportional to F^2 H.

31.3.1 To determine the values of the A and B constants, plot the measured F^2H values for the standard mixes against the respective percent dispersion, *d*, values from Test Method B using the special log paper. Draw the best regression line and select two different points along the line where the respective percent dispersion and F^2H values can be seen clearly. Record the values for these two points.

31.3.2 Calculate A (slope) and B (intercept) as follows:

$$A = \frac{\mathrm{Log}_{10}(100 - d)_2 - \mathrm{Log}_{10}(100 - d)_1}{\mathrm{Log}_{10}\left(F^2H\right)_2 - \mathrm{Log}_{10}\left(F^2H\right)_1}$$

$$B = \mathrm{Log}_{10}\left(100 - F^2H\right)_2 - \mathrm{A}\ \mathrm{Log}_{10}\left(F^2H\right)_1$$

As listed above, Point 2 represents a higher dispersion level than Point 1. The values for A are always positive, and those for B are negative because the intercept is a fraction.

32. Report

32.1 Report the following information:

32.1.1 Proper identification of the sample as described in 21.3.1.1 – 21.3.1.4,

32.1.2 The A and B values to the nearest 0.001,

32.1.3 The C value,

32.1.4 The F^2H roughness factor to the nearest 1.0, and

32.1.5 The dispersion index value to the nearest 0.1.

33. Precision and Bias

33.1 *Precision*—The precision results for these test methods originally were derived from an interlaboratory test program (ITP) conducted prior to the adoption of Practice D4483 as the reference precision standard for D24 test methods and was not conducted in accordance with Practice D4483. However, the results of that ITP have been translated into Practice D4483 precision expression format as much as possible and are given in this section.

33.2 The precision results in this precision section give an estimate of the precision of the test method with the materials used in the particular ITP as described in 33.3. The precison parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those materials and the specific testing protocols of the test method.

33.3 The Type 1 precision is based on a program that employed four materials (carbon black compounds) measured or tested in duplicate on each of two days by six laboratories. Each measurement was made as a 2.0 cm roughness trace. The test result range (measured dispersion index) was from approximately 35 to 98.

33.4 The precision for Method C is given in Table 1 for the average of duplicate tests for each day of testing.

33.5 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method, since the value or level of the test property is defined exclusively by the test method. Bias, therefore, cannot be determined.

TEST METHOD D—MICROROUGHNESS MEASUREMENT WITH IFM

34. Scope

34.1 Test Method D is a quantitative test method. The cut surface of a rubber specimen is characterized with an interference microscope which measures the amount of roughness caused by carbon black agglomerates. This test method is applicable to rubber compounds containing all types of carbon blacks over a wide range of loadings.

35. Summary of Test Method

35.1 The compounded rubber is cut to expose a fresh internal surface. This surface is measured with an interference microscope (512 × 512 µm field of view, 1 μm^2 resolution) which measures the RMS roughness and surface kurtosis of the surface irregularities (protrusions or depressions) caused by carbon black agglomerates. These measured roughness parameters are used to derive a dispersion index which is expressed on the same scale (0 to 100) as Test Methods B and C. The dispersion index values are universal and apply to different rubber formulations and filler loadings.

36. Significance and Use

36.1 Certain important physical properties of the compound are influenced significantly by the level of carbon black dispersion (for example, tensile strength, abrasion resistance, and fatigue life). The correlations of these properties with the dispersion index determined by the microroughness measurement method exhibit the same pattern described for the agglomerate count method in 14.1.

37. Apparatus

37.1 *Dispersion Analyzer*[10]—An interference microscopy based microroughness measurement device which is also equipped with a specimen holder and sample cutter (Fig. 6).

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[10] The sole source of supply of the apparatus known to the committee at this time is Ambios Technology Inc., 100 Pioneer Street, Santa Cruz, CA 95060. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

FIG. 6 Components of IFM Dispersion Analyzer System

37.2 *Vibration Isolation Table,* about 43 by 53 cm and 2 cm deep is recommended for mounting the microscope head.

37.3 *Scissors.*

37.4 *Razor Blades,*[9] single edge (coated) stainless steel type, required for the specimen cutting device.

37.5 *Hand Lens (10×).*

37.6 *Freezer*—A standard refrigerator freezer unit (–5°C) is required for unvulcanized compounds.

38. Sampling

38.1 *Vulcanizates*—Specimens may be cut from standard test sheets (about 2 mm thick) or from actual rubber products which can be cross-sectioned to a uniform thickness of about 2 to 3 mm.

38.2 *Unvulcanized Compounds*—Specimens may be prepared from rubber slabs sheeted out to a uniform thickness of 2 to 3 mm.

39. Test Specimen

39.1 Using scissors, cut out a rectangular specimen that is approximately 3-cm long, 1-cm wide, and 0.2-cm deep. The longest dimension of the specimen should be cut along the direction in which the rubber slab was sheeted out.

39.2 Store unvulcanized specimens at about 5°C for a minimum of 30 min prior to cutting.

40. Procedure

40.1 Turn on the power to the computer and motor controller.

40.2 Start the Scanned Probe Image Processing (SPIP) software.

40.3 Start the batch processor module within the SPIP software.

40.4 Open a file saved during a previous run and allow the batch processor software to process this file.

40.5 Leave the SPIP software running and start the ImageStudio Software.

40.6 Within ImageStudio, start the Dispersion index module.

40.7 Within the “Microscope Control” window verify the following parameter values:
40.7.1 “Mode” is set to “Texture”.
40.7.2 “Pixels” is set to “480 × 480”.
40.7.3 “Magnification” is set to “×10.0”.

40.8 Insert the rubber specimen into the specimen holder clamp. The longest dimension of the specimen should be parallel to the top edge of the clamp with about 5 to 7 mm of the specimen protruding above the clamp.

40.9 Position the specimen holder into the specimen cutting device. In the cutting position the clamp screw should be facing upright.

40.10 Insert a new razor blade into the specimen cutter with the cutting lever in the upright position.

40.11 Lower the cutting lever in a slow, smooth stroke until the razor blade has passed through the specimen. Remove the specimen holder from the cutter, and discard the used blade and the excess piece of rubber cut from the specimen.

40.12 Inspect the cut rubber surface on the specimen in the holder using a 10× hand lens. If the surface is uneven or contains any severe cutting artifacts, repeat the cutting operation with a new piece of sample and a new razor blade.

40.13 Insert the specimen holder into the mounting bracket on the microscope stage such that the cut surface is on top and toward the back of the stage.

40.14 Click on the “Move Sample In” button to move the sample under the microscope objective. If necessary, adjust the position of the specimen holder and stage to bring the cut surface beneath the light spot from the microscope. The light spot should be centered front to back and approximately 5 mm from the right hand side of the specimen.

40.15 Adjust the microscope lamp intensity to obtain sufficient illumination for an image of the sample surface.

40.16 Adjust the Z height with the focus knob to obtain interference fringes in the surface image.

40.17 Using the roll and pitch adjustments maximize the coverage of the fringes across the image. For samples with significant amounts of tilt, this may cause the light spot to move off of the specimen. If so, adjust the position of the specimen to bring it back under the microscope objective.

40.18 Click on "Auto Light" button in the ImageStudio software.

40.19 Using the focus knob, determine the location of the uppermost and lowermost points within the field of view. Based on these points, select the appropriate scan length and set the "Z range (um)" accordingly. The scan length should be sufficiently long to allow for complete data acquisition, but as short as possible for efficient data acquisition.

40.20 If necessary, adjust the focus knob to center the fringes in the image window. Now move the head of the microscope UP approximately half of the scan length. Marks on the focus knob delineate 1 µm.

40.21 Click on the "Start" button and wait while the microscope scans across the sample.

40.22 After acquisition, the data will be shown and a prompt to accept the image will display:

40.22.1 If the data is acceptable, select "Yes."

40.22.2 If the data contains significant errors, select "No" adjust the microscope and repeat the step in 40.21.

40.23 Once the data is accepted the stage moves forward one step.

40.24 Repeat the steps in 40.16 – 40.23 until 10 images are obtained.

40.25 Click on "Move Sample Out" button.

40.26 Click on the "Calculate DI" button. The SPIP software will appear and perform the calculations.

40.27 Upon completion of the calculations, the DI report will open and display. When finished with the report, close the report and MS Word.

40.28 Repeat the steps in 40.8 – 40.27 for any additional samples.

41. Calculation

41.1 During analysis of the data, SPIP calculates the following parameters from each data files:

41.1.1 FRq: RMS surface roughness of the data after application of the fast Fourier transform (FFT) filter.

41.1.2 ISku: Surface kurtosis of the data before application of the FFT filter.

41.1.3 FSku: surface kurtosis of the data after application of the FFT filter.

41.2 The dispersion index is calculated as follows:

$$DI = 100.86 - 34.57*FRq + 111.55*ISku^{-1} - 3510.80*FSku^{-2}$$

where the calculated parameters from each of the ten data files are averaged together for use in this equation.

42. Report

42.1 Report the following information:

42.1.1 Proper identification of the sample as described in 21.3.1.1 – 21.3.1.4,

42.1.2 The dispersion index value to the nearest 0.1, and

42.1.3 The FRq, ISku, and FSku values to the nearest 0.1.

43. Keywords

43.1 agglomerate count for carbon black dispersion; carbon black; carbon black dispersion in water; dispersion; interference microscope ; micro-roughness measurement; profilometer; visual inspection for carbon black dispersion