Designation: D 2663 – 95a (Reapproved 2000)

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

This standard is issued under the fixed designation D 2663; 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 determine the degree of dispersion of carbon black in rubber. Three test methods are described as follows:

	Sections
Test Method A—Visual Inspection	3-11
Test Method B—Agglomerate Count	12-23
Test Method C—Microroughness Measurement	24-34

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

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:*

D 1765 Classification System for Carbon Blacks Used in Rubber Products[2]

D 3051 Practice for Carbon Black—Stating the Precision of ASTM Test Methods[2]

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[2]

D 3396 Practice for Carbon Black—Measuring the Precision of ASTM Test Methods[2]

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

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:

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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] *Annual Book of ASTM Standards*, Vol 09.01.

[3] A separate 8 by 10-in. glossy print of Fig. 1 is available at a nominal charge from ASTM Headquarters, 1916 Race St., Philadelphia, PA 19103. Request Adjunct No. 12-426630-20.

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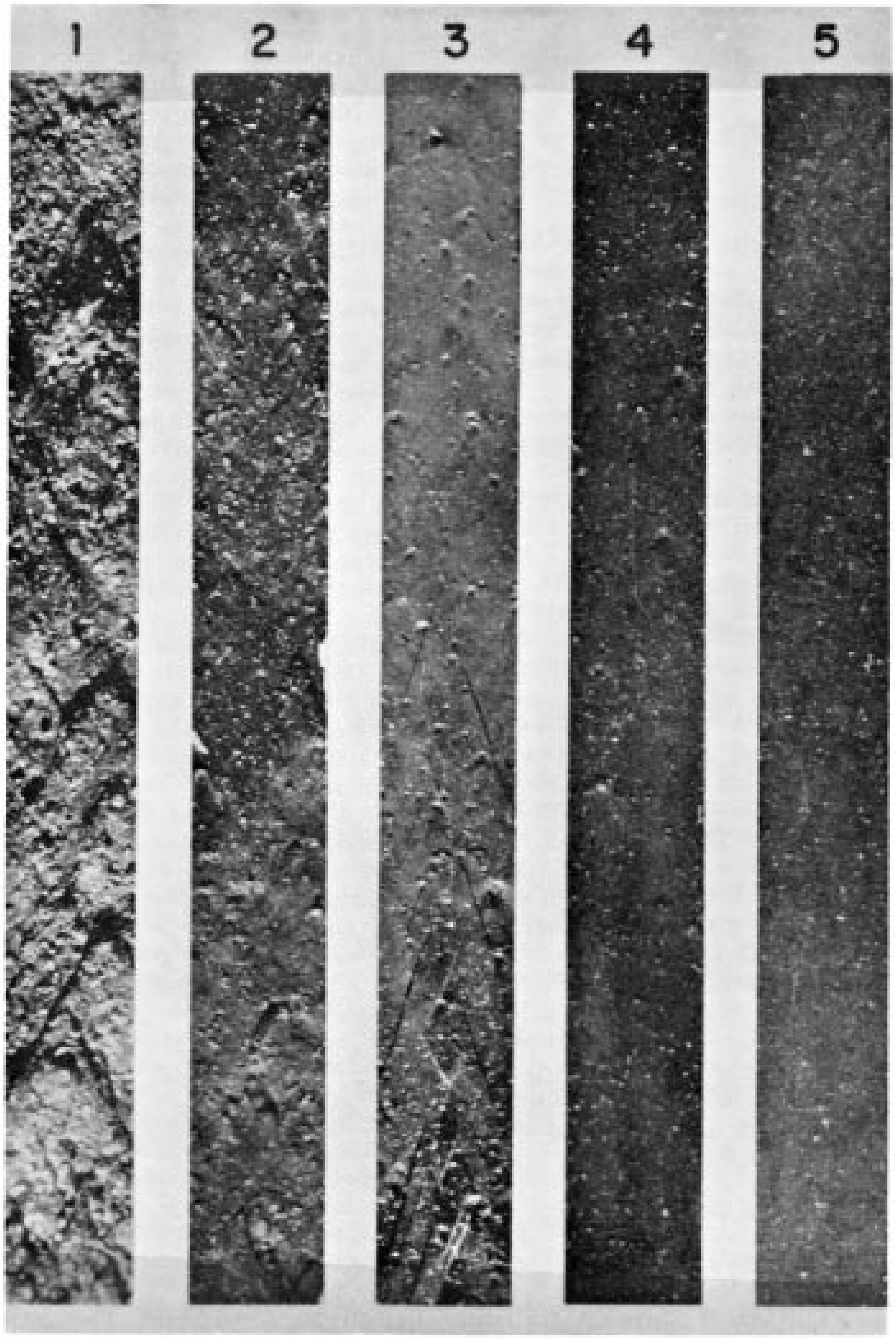

FIG. 1 Carbon Black Dispersion Standards—Visual Analysis of Torn Vulcanizates

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 D 3182. 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

15. Apparatus

15.1 *Microtome*—A sledge-type microtome equipped with specimen clamp and holder for glass knives. Steel knives may also be employed, according to the preference of the operator.

15.2 *Freezing Blocks*, consisting of a brass screw which is threaded into an insulating block of TFE-fluorocarbon (Fig. 2). The TFE-fluorocarbon block shall be 28-mm square in cross

FIG. 2 Brass Sample Mount and TFE-Fluorocarbon Insulator

section and 40-mm high. The threaded part of the brass screw shall be 15 mm in diameter and shall extend into the center of the TFE-fluorocarbon block to a depth of 34 mm. Coarse, expansion-type threads should be used to prevent splitting of the TFE-fluorocarbon when the mount is frozen. The head of the brass screw shall be 18 mm in diameter and shall extend 3 mm above the TFE-fluorocarbon insulator. The top of the screw head shall be cross-hatched with fine grooves to a depth of about 1 mm.

15.3 *Microscope*—An optical microscope with binocular viewing (trinocular type if photomicrographs are desired) 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. (For photomicrographs a 4× plane objective and a 10× periplanatic eyepiece are recommended.)

15.4 *Microscope Accessories*—A 10× wide field eyepiece containing a graticule that is 1 cm^2 in size and divided into 10 000 small squares.

15.5 *Measuring Lens*— A 7× measuring magnifier calibrated down to 0.1 mm for measuring the swelling factor of the microtome sections. A mechanical vernier stage can also be used for this purpose.

15.6 *Glass Pliers and Cutter*—Wide-face glazier pliers and a wheel-type glass cutter are recommended for preparing glass knives.

15.7 *Sample Die*—Tempered steel die for cutting out 3 by 8-mm specimens.

15.8 *Sable Brushes* (00).

15.9 *Dewar Flask*, $2dm^3$(2-L) size (wide-mouth).

15.10 *Glass Knives*— The knives are prepared from 50-mm wide strips of approximately 6-mm thick plate glass.

15.11 *Microscope Slides and Cover Glasses*.

16. Reagents and Materials

16.1 *Liquid Nitrogen*.

16.2 *Water-Soluble Mucilage*.

16.3 *Naphtha*, boiling point range from about 113 to 144°C.

16.4 *Xylene*, boiling point range from 135 to 145°C.

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[4] 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 D 3182.

18. Test Specimen

18.1 Cut out a rectangular specimen 8-mm long, 3-mm wide, and approximately 2-mm deep. Use a cutting die, if available. If a die is not used, the specimen length and width should be recorded using a measuring magnifier.

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

19. Preparation of Glass Knives

19.1 Standard types of plate glass are suitable for making knives. Thickness should be preferably about 6 mm. A large sheet of glass should first be repeatedly broken in half so that it eventually is in 50-mm wide strips. Fracture in this manner is preferred over simply breaking off 50-mm sections one at a time. Uneven strains are encountered in the latter procedure, and irregularities may occur on the side faces on which the knife edge will be formed. Prior to fracture, it is also important that the glass be free of dirt. A wash in a liquid detergent is generally advisable.

19.2 Using a fairly thick straightedge, score a straight line (perpendicular to the side faces) across the glass strip, 50 mm from the end. Take care that the score mark does not actually extend all the way to the side faces or chipping may result. Next, fracture along the score line using the glass pliers. The upper jaw of the pliers should have raised pressure points at both ends of the gripping surface, while the lower jaw should have a raised area in the center. These pressure points can be

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

formed with small strips of plastic tape if they are not already incorporated into the jaw surfaces of the pliers. To induce fracture, grasp the glass with the pliers so that the pressure point on the lower jaw is under the score mark near the center of the strip. The front edges of the plier jaws should be perpendicular to the score line. Firm pressure will readily cause fracture. Then, score the resultant 50-mm square of glass across the diagonal, keeping the mark a bit off center so that the potential knife edge will be formed slightly away from the corner on one of the side faces that was formed during the preparation of the glass strips. Take care again that the score mark does not go all the way out to the edges of the glass. Stay within 1 to 2 mm of each side. Fracture with the glass pliers as before. Each square will generally produce one suitable cutting edge.

19.3 Make several knives at one time and inspect for edge flaws (nicks, curvature, etc.) under a binocular microscope. Straight edges are most desirable but not absolutely essential.

NOTE 1—Glass knives should be stored in a desiccator of absolute ethyl alcohol (chemically pure) to conserve the life of the knive edges. Excessive long periods of storage may cause edge deteriorations.

19.4 During use, each glass knife should be inspected frequently and discarded when the edge becomes badly chipped. The life of a knife depends very much on the number of sections that are cut (particularly in paring down the sample block) and in the nature of the sample itself. Rubbers that contain relatively high amounts of inorganic pigments are particularly prone to cause knife chipping and may require a fresh knife for each different specimen.

19.5 Also to be considered is the clearance angle (the angle formed between the sample and the lower surface of the knife). A clearance angle of about 15° has been found satisfactory for many types of elastomer compounds, but may be varied according to the preference of the individual operators.

20. Procedure

20.1 *Embedding and Freezing Specimens (Liquid Nitrogen)*—Embed the specimen in water-soluble mucilage on the center of the scored brass surface of the specimen mount so that its long axis is parallel to the sides of the TFE-fluorocarbon block (Fig. 3). Take care that the glue comes up to the top edges of the specimen on all four sides. Then, using tongs, immerse the entire sample mount in a Dewar flask of liquid nitrogen. After a few seconds the mucilage will freeze and turn white. At this point, quickly remove the specimen mount and inspect for flaws in embedding, such as air bubbles or tilting of the specimen. If the embedding is satisfactory, it is advisable to use a razor blade to trim off any excess mucilage from the top and sides of the specimen. This will minimize subsequent damage to the knife edge while paring down the specimen. Next, reimmerse the specimen mount in liquid nitrogen for the required amount of time. The immersion time depends, primarily, on the type of polymer and the type and loading of carbon black. The time increases with decreasing black loading, increasing particle diameter, and decreasing second-order transition temperature of the polymer. IIR vulcanizates containing 50 phr of N330 carbon black or oil extended SBR stocks with 50 phr of a N110 black require about a 1-min freeze. BR compounds may require 2-min or longer freezing time. More detailed information regarding freezing times is best compiled by individual operators on their own particular specimens.

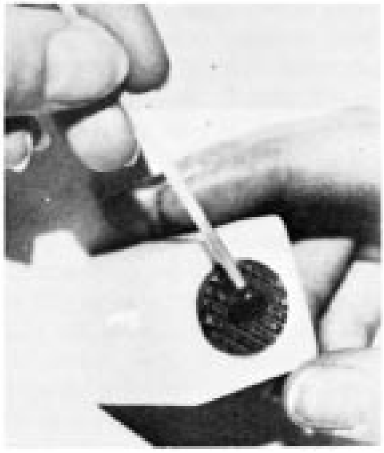

FIG. 3 Embedding Rubber Block in Mucilage

20.2 *Embedding and Freezing Specimens (Liquid CO_2)*—A standard-type CO_2 freezing stage may be used. However, as a safety precaution, it is recommended that the stage be specially reinforced to withstand the high vapor pressure of liquid carbon dioxide.

20.2.1 Connect freezing stage through a flexible hose and filter to a tank of liquid carbon dioxide. The tank should be supplied with a siphon fitting so that the CO_2 comes out as a liquid rather than a gas.

20.2.2 Embed the specimen in mucilage in the center of the freezing stage in the same manner listed in 20.1 and freeze with several short bursts by opening and closing the valve quickly. Refreeze during the sectioning operation whenever necessary. For specimens that are too soft to be microtomed at liquid CO_2 temperatures, pour a little liquid nitrogen onto the specimen block while it is in position on the freezing stage.

20.3 *Microtoming Operation*—Insert the frozen specimen mount in the clamp on the microtome and fasten in place so that the long axis of the specimen is in line with the long axis of the knife (Fig. 4). Then, manually advance the specimen upward so that its top surface almost reaches the level of the

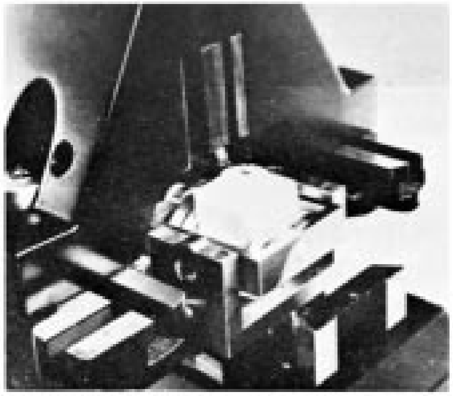

FIG. 4 Frozen Sample Mounted for Sectioning on a Sledge Microtome

knife. At this point, with the advance set in increments of 5 to 10 µm, start microtoming until the specimen is planed level and full-size sections are being cut. All through the entire sectioning operation the knife edge should be continually kept wet with solvent (xylene or naphtha), applied by brush.

20.3.1 After paring down, set the automatic thickness control at 2 µm (a lower setting may be necessary if the black loading is very high or if the rate of thermal expansion is high), and section, using long, even strokes. With a brush dipped in xylene or naphtha, remove the thin sections fairly rapidly from the knife edge after each cut, before drawing the sledge back. Deposit the sections in a petri dish of xylene or naphtha as they are cut. Repeat the operation until twelve or more sections have been cut, to ensure good quality. Avoid excessive time lapses between succeeding cuts, as this will lead to thickening due to thermal expansion. Also, if too much time is taken, the rubber specimen will soften and will have to be refrozen.

20.4 *Mounting Sections on Microscope Slides*—Using a fresh sable brush (not the same one used for removing sections from the knife), transfer a specimen from the solvent to a clean microscope slide. The section will be curled up in a small tight roll. Careful manipulation of the solvent wet brush will unroll and spread the section out flat on the slide (Fig. 5). Continue brushing gently to remove all wrinkles. If the section tends to slip on the slide, hold one corner lightly with a mounted needle.

20.4.1 Brush out five or six sections to ensure having a good one for measurement. Then cover the sections with cover glasses, and seal with tape, or a bit of cement at each corner.

20.5 *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.

20.5.1 Measure the length and width of the section that is selected for counting. The product of these dimensions is the area after swelling. Record this value along with the sample area before swelling.

20.6 *Counting Procedure*—The agglomerate counting is done by means of the ruled graticule located in one of the microscope eye pieces. Magnification should be in the range from 75 to 100×. The exact figure is left to the discretion of the individual operator, based on the specifications of his own particular microscope and lens system. At 100× magnification, each square on the graticule is 10 by 10 µm. 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.

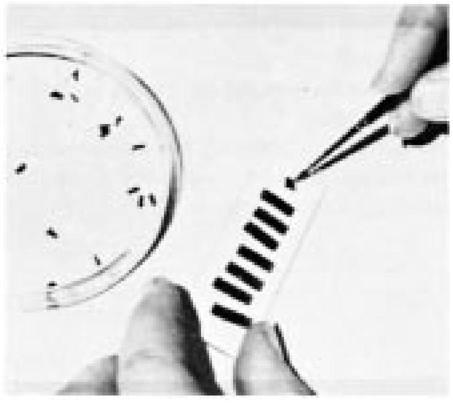

FIG. 5 Brushing Out Sections Swelled in Naphtha

20.6.1 Count five fields on the section, arbitrarily selecting them from the four corners and the center. Each field represents the entire area covered by the 10 000 squares on the graticule. Count all squares that are at least half covered by an agglomerate of carbon black. This will encompass all agglomerates down to about 5 µm in size. The black clumps of carbon are easily distinguished from the background (usually brown in tone) and from the knife marks, which appear as long streaks.

21. Calculation and Interpretation of Results

21.1 *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 = total number of graticule squares that are at least half filled with carbon black. (This represents an average of the five graticule counts on the section. See Note 2.)

NOTE 2—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 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:

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

21.1.1 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.

TABLE 1 Ranges of Dispersion Ratings

Range	A	B	C	D	E	F	G	H
Range boundaries, %	100 to 98.6	98.6 to 96.0	96.0 to 91.8	91.8 to 84.4	84.4 to 71.6	71.6 to 48.4	48.4 to 0	(0)
Average rating, %	99.3	97.3	93.9	88.1	78.0	60.0	30.0	(0)
Range width, %	1.4	2.6	4.2	7.4	12.8	23.2	48.4	(0)

TABLE 2 Categories of Agglomerate Size

Category	1	2	3	4	5	6
Average area, 10-µm squares	2.8	8.4	25.3	76	228	684
Average diameter, µm	19	33	57	98	170	295
Boundaries, 10-µm squares	0.5 to 4.9	4.9 to 14.7	14.7 to 43.9	43.9 to 128.4	128.4 to 395.5	395.5 to ∞

21.2 *Classification of Agglomerate Size and Dispersion Range*—Agglomerate size is also an important aspect of dispersion since, as a rule, larger black agglomerations tend to be most damaging to the end performance of the compound. To facilitate a simple estimation of the average agglomerate size, the Dispersion Classification Chart may be used.[5] This chart also affords a reference scale for estimating the percentage of dispersed black in instances where the more precise measured values are not required. These instances would include in particular the evaluation of compounds at very low dispersion levels which are known to be of poor quality by simple visual inspection of the sections. Such samples are quite tedious to measure.

21.2.1 The Dispersion Classification Chart contains light photomicrographs (magnification 60×) of microtome sections from compounds of varied dispersion quality. Each micrograph represents a square of 1.3 by 1.3 mm on the actual rubber section. In all instances the carbon black volume loading is 18 %. The chart is graduated from A to H in the direction of lower percent dispersion and from 1 to 6 in the direction of increasing agglomerate size. For each letter rating the amount of undispersed black is the same throughout the six agglomerate size categories. However, it will be noted that some of the micrographs are missing from the coarser agglomerate categories of ranges A, B, and C simply because the percentage rating falls out of the range at that size level. The percentage ranges and agglomerate size categories are listed in Table 1 and Table 2. It should be remembered that although each micrograph illustrated in the Dispersion Classification Chart shows a definite amount of undispersed black, the percentage ranges are only valid for an 18 % black volume loading. Compounds containing appreciable lower or higher black volume loadings can also be classified using the chart, but the percentage ratings must then be corrected accordingly.

21.2.2 The Dispersion Classification Chart also serves as a means of describing certain types of unusual black dispersion patterns such as unmilled latex masterbatches or poor masterbatch letdowns. A typical unmilled latex masterbatch (ULM) is shown on the chart. Poor letdowns or varied appearance in masterbatches may often be described in terms of the "H" series. Although this entire series is at a "0" or no dispersion level, the different agglomerate categories do vary considerably in appearance. An accurate description of this appearance is often meaningful in determining the cause of a poor masterbatch letdown.

22. Report

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

22.2 *Agglomerate Size*— Report the average agglomerate size in terms of one of the six categories on the Dispersion Classification Chart. In instances where a bimodal agglomerate distribution exists, two categories may be listed.

22.3 *Estimated Percent Dispersion Values*—Report estimated dispersion ratings in terms of one of the eight ranges (A to H) on the Dispersion Classification Chart.

22.4 *Combined Ratings*— In instances where the percent dispersion has been measured, list the measured value followed by the range and agglomerate size category on the Dispersion Classification Chart, for example, a measured value of 94.3 % with an agglomerate category of "2" would be listed as 94.3 (C2).

22.4.1 Where the dispersion range has been estimated, simply list the range along with the agglomerate size category, for example, the above example would be listed as C2.

22.5 *Compound Identification*—Whenever possible list pertinent information regarding the following:

22.5.1 *Formulation*:

22.5.1.1 Carbon black, type and loading,

22.5.1.2 Other fillers, type and loading,

22.5.1.3 Polymer type, and

22.5.1.4 Extender oil, type and loading.

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

22.5.2.1 A standard mixing procedure,

22.5.2.2 Type of equipment,

22.5.2.3 Masterbatch, and

22.5.2.4 Finished compound.

23. Precision and Bias

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

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[5] Available at a nominal cost from ASTM Headquarters. Order PCN 12-426630-10.

FIG. 6 Components of Dispersion Analyzer System

TEST METHOD C—MICROROUGHNESS MEASUREMENT

24. Scope

24.1 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.

25. Summary of Test Method

25.1 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.

26. Significance and Use

26.1 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 Section 14.1.

27. Apparatus

27.1 *Dispersion Analyzer*[6]—A stylus microroughness measurement device which is also equipped with a specimen holder, sample cutter, and specimen tracking mount (Fig. 6).

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

27.3 *Scissors*.

27.4 *Razor Blades*, [7] single edge (coated) stainless steel type, required for the specimen cutting device.

27.5 *Hand Lens* (10×).

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

27.7 *Logarithmic Graph Paper*, [6] special 2 × 3 cycle.

28. Sampling

28.1 *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.

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

29. Test Specimen

29.1 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.

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

30. Calibration

30.1 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.

30.1.1 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.

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

30.1.3 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.

30.1.4 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.

30.1.5 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.

30.1.6 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.

30.1.7 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.

31. Procedure

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

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

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

31.4 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 32.

31.5 Set the roughness width cutoff at 0.80.

31.6 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.

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

31.8 Insert the rubber specimen into the specimen holder clamp. The longest dimension of the specimen should be

TABLE 3 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)

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[6] Available from the Federal Products Corp., 1144 Eddy St., Providence, RI 02901.

[7] Available from American Safety Razor Company, Industrial Products Div., Razor Blade Lane, Verona, VA 24482.

parallel to the top edge of the clamp with about 8 to 10 mm of the specimen protruding above the clamp.

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

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

31.11 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.

31.12 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.

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

31.14 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.

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

31.16 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.

31.17 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.

31.18 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, $F^2 H$.

31.19 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.

31.20 Repeat 31.8 through 31.19 for additional specimens of the same rubber formulation.

32. Calculation

32.1 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.

32.2 *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.

32.2.1 Measure or estimate the percent dispersion in each standard mix using Test Method B as described in Sections 12 through 23.

32.2.2 Measure the F^2H roughness factors for each standard mix using the procedures described in Sections 26 through 31.

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

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

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^2H.

32.3.1 To determine the values of the A and B constants, plot the measured $F^2 H$ 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.

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

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

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

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.

33. Report

33.1 Report the following information:

33.1.1 Proper identification of the sample as described in 22.5.1.1 through 22.5.1.4,

33.1.2 The A and B values to the nearest 0.001,

33.1.3 The C value,

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

33.1.5 The dispersion index value to the nearest 0.1.

34. Precision and Bias

34.1 *Precision*—The precision results for these test methods originally were derived from an interlaboratory test program (ITP) conducted prior to the adoption of Practice D 4483 as the reference precision standard for D-24 test methods and was not conducted in accordance with Practice D 4483. However, the results of that ITP have been translated into Practice D 4483 precision expression format as much as possible and are given in this section.

34.2 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 34.3. 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.

34.3 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.

34.4 The precision for Method C is given in Table 3 for the average of duplicate tests for each day of testing.

34.5 *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.

35. Keywords

35.1 agglomerate count for carbon black dispersion; carbon black; carbon black dispersion in water; dispersion; microroughness measurement; visual inspection for carbon black dispersion

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