



## Designation: D2663 – 14 (Reapproved 2019)<sup>ε1</sup>

# Standard Test Methods for Carbon Black—Dispersion in Rubber<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

<sup>ε1</sup> NOTE—Corrected 2.2 editorially in May 2021.

## 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

[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](#)

<sup>1</sup> 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.

Current edition approved May 1, 2019. Published June 2019. Originally approved in 1967. Last previous edition approved in 2014 as D2663 – 14. DOI: 10.1520/D2663-14R19E01.

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

### 2.2 ASTM Adjuncts:

[Carbon Black Dispersion Standards<sup>3</sup>](#)

[Carbon Black Dispersion Chart<sup>4</sup>](#)

## 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](#)),<sup>3</sup> 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×).*

<sup>3</sup> Available from ASTM International Headquarters. Order Adjunct No. [ADJD266302-E-PDF](#). Original adjunct produced in 1967.

<sup>4</sup> Available from ASTM International Headquarters. Order Adjunct No. [ADJD266301-E-PDF](#). Original adjunct produced in 1967.

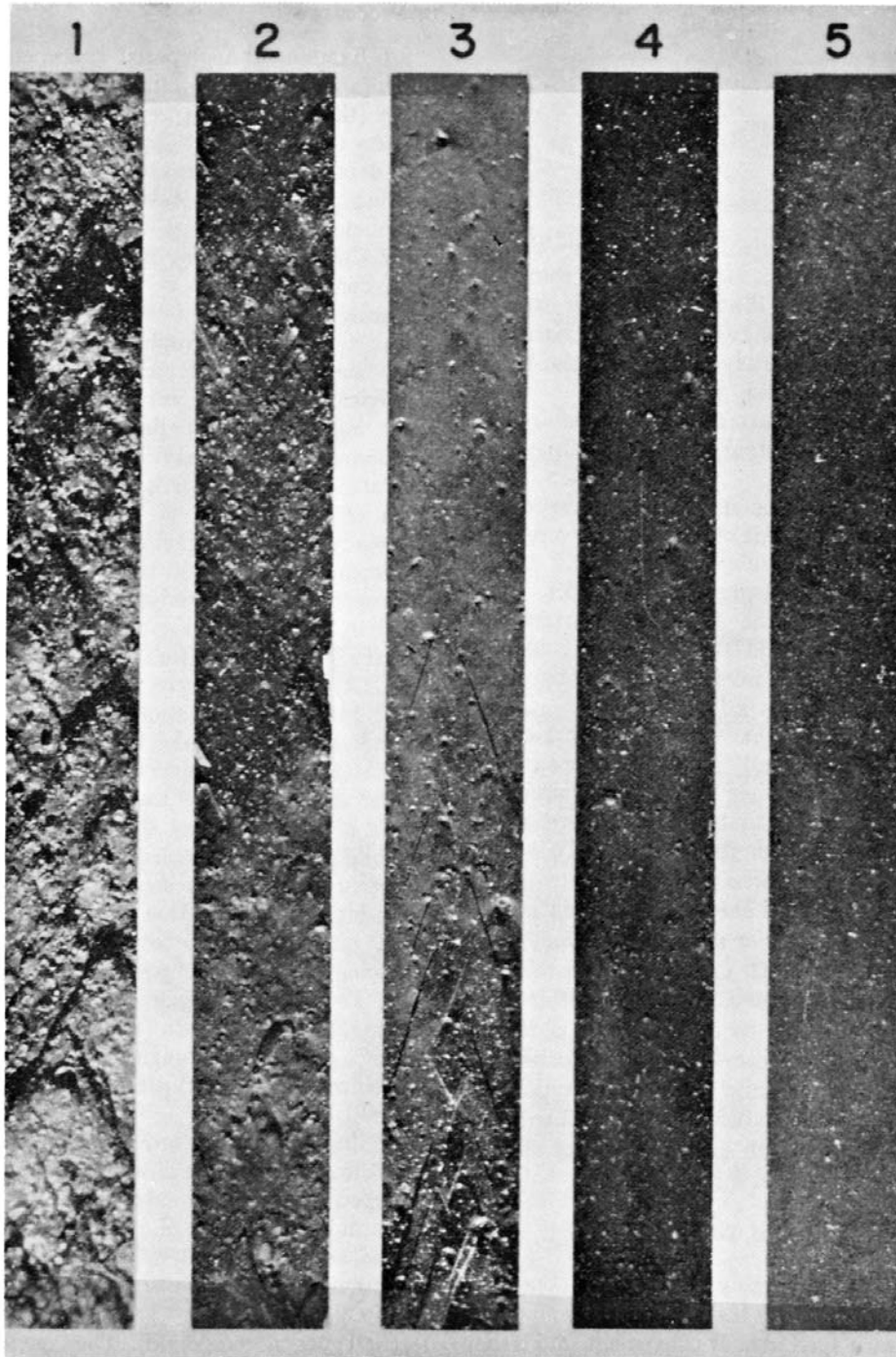


FIG. 1 Carbon Black Dispersion Standards—Visual Analysis of Torn Vulcanizates

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:

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.”