Designation: D7723 – 17

Standard Test Method for Rubber Property—Macro-Dispersion of Fillers in Compounds[1]

This standard is issued under the fixed designation D7723; 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 This test method covers a procedure to measure the macro-dispersion of fillers in a rubber matrix by quantifying the surface roughness of a freshly cut specimen using an optical microscope in reflection mode.

1.2 The method provides a procedure to measure the quality of mixing of reinforcing fillers such as silica and carbon black, as well as inert fillers such as chalk, clay and other solids.

1.3 The method includes a sample preparation procedure for filled uncured rubber compounds as well as filled cured rubber compounds.

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

1.5 *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.*

1.6 *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:*[2]

D2663 Test Methods for Carbon Black—Dispersion in Rubber

D3053 Terminology Relating to Carbon Black

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *agglomerates, n*—any number of filler aggregates held together by van der Waals Forces (carbon black) or hydrogen bonding (silica).

3.1.2 *macro dispersion, n*—degree of distribution of filler into a compound, generally on a scale of less than 100 μm but greater than 2 μm; represents micron range agglomeration.

3.1.3 *nodges, n*—bumps in a cut surface caused by filler agglomerates in a rubber matrix.

3.1.4 *surface roughness, n*—the bumps (nodges), or hills and valleys that are on the visible side of a sample.

3.1.5 *white area, n*—the portion of the scan area which contains nodges, or other surface defects; it is described here as white, because the reflected light from these surface defects is white.

4. Summary of Test Method

4.1 This standard uses mathematical algorithms to quantify the surface roughness of freshly cut rubber specimens as measured by a reflected light optical method in two dimensions.

4.2 The reflected light optical method, generally used to determine a comparative dispersion rating, is expanded to give quantitative data as to the size and number of nodges. Nodges do not show the actual size of filler agglomerates. It is assumed that as the sample is cut, large agglomerates are pushed to one side or the other leaving a contoured surface. The diameter and frequency of the surface contours are measured using image processing. These contours are referred to as "nodges" to differentiate them from actual agglomerates. This data is presented in histogram form of count versus nodge diameter, and allows calculating a measure of dispersion.

5. Significance and Use

5.1 The incorporation of fillers into the rubber matrix is characterized by their macrodispersion as an indicator of the quality of mixing. This test method provides a measure of the macro-dispersion of reinforcing fillers, like silica and carbon black, as well as of inert fillers. Based on their polymer nature,

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.12 on Processability Tests.

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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.

different types of rubbers can show a different degree of acceptance for the incorporation of fillers, as indicated by their macro-dispersion.

5.2 Macro-dispersion of carbon black and silica in rubber compounds may be measured by different methods. Carbon black provides a direct physical reinforcement; silica requires a silane coupling agent in order to initiate reinforcement, and therefore, a different technology of mixing. Silica is also a non-conductor, making electrical methods of dispersion measurement impracticable. This test method is specifically appropriate for the characterization of the microdispersion in silica technology.

5.3 This test method also can measure the mixing quality of colored rubbers. It uses variable exposure in order to be able to image a wide range of colors.

5.4 This test method is intended for use in research and development as well as in quality control of filler processability in rubber and may be used for both the evaluation of production processes or referee purposes.

6. Apparatus

6.1 *Razor Blade (recommended) or Sharp Knife*—The specimen may be prepared using a static cut as shown in Fig. 1 or cut while being bi-directionally elongated as shown in Fig. 2.

6.2 *Reflected Light Microscope,* with the following specifications:

6.2.1 Imaging power to resolve to 1 µm, 3 µm, or 10 µm depending on the instrument used.

6.2.2 Dark field illumination as shown in Fig. 3.

6.3 The light microscope is to be equipped with an image sensor. The sensor used to capture the image is a common CCD (Charged Coupled Device) or CMOS (Complementary Metal Oxide Semiconductor) sensor. In the dark field mode, an aperture is lit at a 30° angle for analysis. The sensor picks up the reflection of bumps on the surface, nodges representing undispersed filler. Flat areas of the sample surface are dark.

6.4 A scan is made by taking a digital gray-level image in the dark field mode of the microscope with 1 µm, 3 µm, or 10 µm resolution.

6.5 The image captured by the sensor shall be digitized and analyzed by the image processing software. The area of nodges is represented as "White Area." The percent White Area, URF%, is the percentage of the white area representing the nodges to the total area of the image. Based on this area ratio, percent dispersion can be calculated.

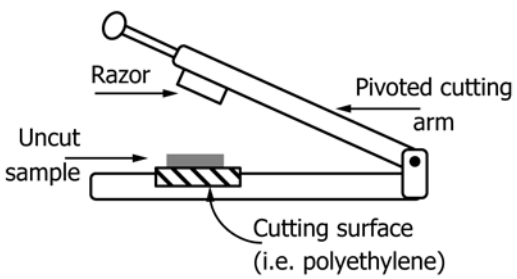

FIG. 1 Static Cut

7. Calculation

7.1 As the specimen is cut, the underlying agglomerates are pushed to one side or another resulting in hills and valleys on the cut surface. They represent agglomerates under the surface of the cut. The nodge diameter that is calculated from these images is larger than the underlying agglomerate. For simplification, nodge size is reported as Agglomerate Size and represented by a histogram (Fig. 4).

7.2 In order to stay within the 2-100 µm range of macro-dispersion defined in Terminology D3053, it is necessary to disregard small nodges that are covering the smaller agglomerates by introducing a threshold. Two different values for the threshold may be used: 5 µm and 23 µm. The threshold value cannot be selected below the resolution of the microscope as stated in 6.2.1. The frequency of occurrence of nodges decreases with a higher level of dispersion. Dispersion, therefore, is calculated from the white area, which is determined from the radius and frequency of all nodges greater than the nodge threshold.

7.3 Calculate the White Area, URF, as a ratio to the total scan area.

7.4 Calculate the percent dispersion of fillers as follows:

$$Dispersion\ \% = (100 - 100 \times URF)/L \quad (1)$$

where:

URF = fraction of total scan area from undispersed filler measured in reflection
L = the filler volume fraction in the compound

7.4.1 For maximum accuracy, the filler volume fraction can be calculated from the following expression, which also appears in Test Methods D2663:

$$L = \frac{compound\ density \times filler\ mass}{filler\ density \times compound\ mass} \quad (2)$$

7.5 If the volume percentage of filler in the rubber compound L is either not given or unknown, calculate the weighted percent dispersion or Z Value as follows:

$$Z\ value = (100 - 100 \times URF)/0.35 \quad (3)$$

NOTE 1—The Z Value assumes a maximum of 35 % white area. Fixing the volume fraction at a maximum value of 0.35 allows the user to skip the time-consuming step of determining the volume fraction, which is not necessary for quality control of a specific compound.

8. Test Specimen

8.1 The test specimen is prepared from a vulcanized or unvulcanized sample of a filled rubber compound. A razor blade cut shall be made, so that two similar surfaces are exposed. The surfaces shall be bigger than 5 by 5 mm.

8.1.1 When preparing the test specimen, it is recommended that a new razor blade be used for each cut. The abrasiveness of the rubber as well as contaminates from the rubber left on the blade may affect successive cuts.

8.1.2 The thickness of the uncut specimen shall be 5 to 10 mm. Thicker specimens cause excessive drag between the razor blade and rubber and may affect the quality of the cut.