Designation: D 5596 – 03

Standard Test Method For Microscopic Evaluation of the Dispersion of Carbon Black in Polyolefin Geosynthetics[1]

This standard is issued under the fixed designation D 5596; 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 equipment, specimen preparation techniques, and procedures for evaluating the dispersion of carbon black in polyolefin geosynthetics containing less than 5 % carbon black by weight.

1.2 This test method allows for a qualitative evaluation of carbon black agglomerates and other inclusions in polyolefin geosynthetics. This evaluation is based on carbon black dispersion size calculated area within microscopic fields of view.

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

1.4 *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.*

NOTE 1—This test method is for the evaluation of carbon black dispersion. This test method does not support or evaluate the distribution of carbon black.

2. Referenced Documents

2.1 *ASTM Standards:*
D 883 Terminology Relating to Plastics[2]
D 3053 Terminology Relating to Carbon Black[3]
D 4439 Terminology for Geotextiles[4]
E 7 Terminology Relating to Metallography[5]
2.2 *ASTM Adjuncts:* ASTM
D 35—Carbon Dispersion Reference Chart[6]

3. Terminology

3.1 *Definitions:*

3.1.1 *carbon black*, *n*—a material consisting essentially of elemental carbon black in the form of near spherical colloidal particles and coalesced particle aggregates of colloidal size, obtained by partial combustion or thermal decomposition of hydrocarbons. **(D 3053)**

3.1.2 *carbon black agglomerate*, *n*—a cluster of physically bound and entangled aggregates. **(D 3053)**

3.1.3 *geosynthetic*, *n*—a planar product manufactured from polymeric material used with soil, rock, earth, or other geotechnical engineering-related material as an integral part of a man-made project, structure, or system. **(D 4439)**

3.1.4 *micrograph*, *n*—a graphic reproduction of an object as seen through the microscope or equivalent optical instrument, at magnifications greater than ten diameters (micrograph). **(E 7)**

3.1.5 *microtome*, *n (that is, sliding microtome)*—an apparatus capable of cutting thin slices (less than 20 µm in thickness) of various geosynthetic samples.

3.1.6 *polyolefin*, *n*—a polymer prepared by the polymerization of an olefin(s) as the sole monomer(s). **(D 883)**

3.1.7 *dispersion*, *n*—a polyolefin product formulated with carbon black.

3.1.8 *distribution*, *n*—a property of a carbon black formulated polyolefin product that refers to the existence of streaks, light or dark, within a microsectioned sample.

4. Summary of Test Method

4.1 This test method consists of two parts: (1) microtome specimen preparation and (2) microscopic evaluation.

4.1.1 *Microtome Specimen Preparation*—A sample is clamped in the sample holder, which can be raised or lowered precisely in increments of approximately 1 µm. A rigid knife is slid manually across the sample so that the specimens range in thickness from 8 to 20 µm.

4.1.2 Microtome specimen examination: These thin sections are evaluated microscopically calculating the largest agglomerate or inclusion in each random field of view (R*f*). The

[1] This test method is under the jurisdiction of ASTM Committee D35 on Geosynthetics and is the direct responsibility of Subcommittee D35.02 on Endurance Properties.

Current edition approved Feb. 10, 2003. Published April 2003. Originally approved in 1994. Last previous edition approved in 1994 as D 5596–94.

[2] *Annual Book of ASTM Standards*, Vol 08.01.
[3] *Annual Book of ASTM Standards*, Vol 09.01.
[4] *Annual Book of ASTM Standards*, Vol 04.09.
[5] *Annual Book of ASTM Standards*, Vol 03.01.
[6] Available from ASTM, 100 Barr Harbor Dr., W. Conshohocken, PA 19428, Request PCN: ADJD5596.

associated carbon dispersion chart can be used to assist to determining shape and area

5. Significance and Use

5.1 Carbon black is added to many polymers to provide long-term resistance to ultraviolet-induced degradation. To achieve this, carbon black should be dispersed uniformly throughout the as-manufactured geosynthetic material. This test method is used to evaluate the uniformity of carbon black dispersion.

5.2 This test method is suitable only for those geosynthetics that can be sampled using a rotary or sledge microtome. The geometry, stiffness (hardness), or elasticity of some geosynthetic products precludes their being sampled with a microtome. The cross-sectional area of the geosynthetic must be composed of a continuous solid polyolefin material to be sampled using a microtome.

5.3 Extruded and oriented geogrids will require that microtome specimens be cut from the nonoriented bars of uniaxial products and the non-oriented nodes of biaxial products.

6. Equipment

6.1 *Microtome*—A rotary or sledge-type microtome equipped with a sample clamp and knife holder is required. Steel knives are recommended; however, glass knives may be suitable.

6.2 *Microtome Accessories*—Lubricant, dust cover, and tweezers are recommended.

6.3 *Microscope*—An optical microscope with binocular viewing (trinocular type, if micrographs are to be taken) is recommended. This should include a movable specimen stage. Lenses should include two 10× wide field eyepieces and objectives in the range of 5 to 20×. Taking into account microscope tube corrections, objectives should be selected so that final magnifications in the range of 50 to 200× are available.

6.4 *Microscope Accessories*—A calibrated reticle (eyepiece micrometer) positioned in one of the eyepieces between the eyepiece-lens and the objective is required.

6.5 *Light Source*—An external white light source with variable intensity is required.

6.6 *Microscope slides and cover slides*, required.

6.7 *Balsam cement or suitable, clear substitute* (for example, clear nail polish), required (Note 2).

NOTE 2—**This clear, adhesive medium should not dissolve or chemically interact otherwise with the thin section.**

6.8 Make a microscope cover slide to obtain random field (R_f) of view. From center point of slide make a mark 5 mm to either side. Use a straight edge and a glass etcher draw two parallel lines the length of the slide at the marks. Measure 3.2 mm from each of the lines toward the outer portion of the slide and make a mark. Etch parallel lines to the original lines. Finished cover should look as Fig. 1.

NOTE 3—Other techniques can to used to make random field of view slide as long as the two (2) 3.2 mmm opening are positioned for the random field of view.

6.9 The Microscope cover slide should be the same size as the slides that the specimens are placed on. The parallel lines should allow viewing of all specimens when placed.

7. Procedure

7.1 *Sampling*—Five samples are selected randomly across the full roll width (where applicable) for each geosynthetic material to be tested. Geomembrane samples should each be approximately 2.54 cm 2 (1 in.2). Geonet samples are selected randomly from five strands across the full roll width. Geogrid samples are selected randomly from five nodes across the full roll width. Pipe and polyolefin components of geocomposite samples are also selected at random.

7.2 *Specimen Preparation*—Using a microtome, prepare one microsection in the cross-machine direction from each

Figure – 1 Microscope Cover Slide Overlay Configuration

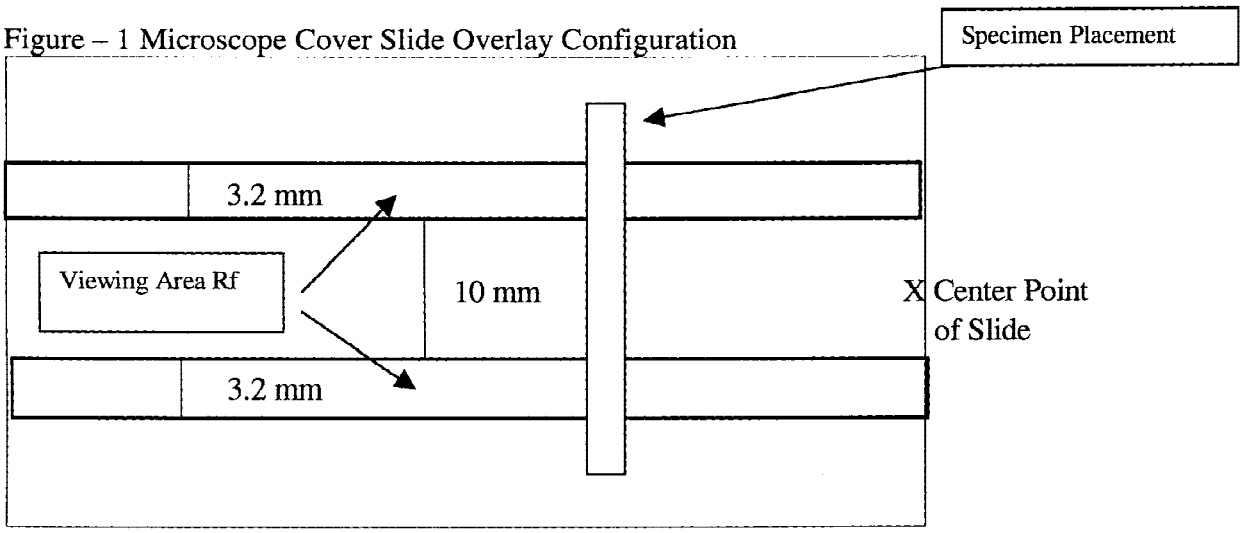

FIG. 1 Microscope Cover Slide Overlay Configuration

geomembrane specimen (See Note 2). Non-oriented geosynthetics material specimens can be prepared without regard to processing direction. The use of tetrafluoroethane stiffen spray will assist microtoming of most materials preventing smearing of carbon black or other constituents in sample. The tetrafluoroethane spray is used to stiffen the sample to −15° C before microtoming the specimens.

NOTE 4—Some extremely flexible or elastomeric materials (e.g., very flexible polyethylene) may require micro-sectioning under low temperature conditions. In these instances, the sample to be micro-sectioned and the microtome knife and sample clamp can be loosely packed in crushed dry ice for approximately 15 minutes or until the specimen, knife, and clamp reach approximately −30° C. The microtome apparatus should be set up so that the specimen can be clamped in place and thin sectioned within 1 to 5 minutes of removal from the dry ice. The sample can be stiffened by spraying with tetrafluoroethane before micro-sectioning. Other means of freezing sample is acceptable if no damage to the plastic occurs.

7.3 Each thin section should be (1) thin enough (8 to 20-µm thick) to allow for adequate light transmission so that carbon agglomerates can be examined easily during microscopy and (2) free from major defects such as gouges caused by a nicked or dull knife, or such as torn or distorted portions of the thin sections caused by over-stressing or rough handling (see Note 5). Mount each excised thin section between a microscope slide and a cover slide, using a suitable clear adhesive medium.

NOTE 5—Because thin sections ≥20 µm thick are usually too thick to permit adequate light transmission through the thin section, thin sections should be 10 to 15 µm thick. These thin sections tend to curl up, making them difficult to handle. The use of a light honing oil on the knife helps the specimen to stick to the blade, make it easier to slide off the blade and onto the slide glass.

7.3.1 Mount five (5) specimens to each slide. Place the microscope cover slide over the five specimens. The cover slide should be placed so that there is a viewing area of each specimen. The part of the specimens that is exposed by the two (2) parallel 3.2 mm viewing area of cover slide is considered the random field of view (R_f). (See Fig. 1)

7.4 *Microscope Setup*—Prepare the microscope for transmitted light microscopy with the calibrated reticle positioned between one eyepiece lens and the objective.

7.5 Place the microscope cover slide (as shown in Fig. 1) on top of the mounted thin-sections.

7.6 *Random Field of View (R f) Selection*—Before attempting any close, microscopic examination of the thin section, place the mounted thin section on the microscope stage positioned between the light source and the objective. Place the microscope cover slide on top of the mounted thin section so that each of the field of view overlaps the thin section fully. The area of the thin section lying within each of the parallel portion of the microscope cover slide is called a random field of view or R*f*.

7.7 *Microscopic Evaluation*—Examine each *R f* microscopically, and locate the largest carbon agglomerate or inclusion. If the microscope is not at 100×, select the objective that allows for viewing at 100×. Calculate the area of the agglomerate or inclusion. Non-spherical agglomerates calculation is made by diametric area of best fit.

7.8 *Iteration*—Repeat the procedures given in 7.5 and 7.6 until ten readings are recorded. No more than two *R f*'s are taken from each of no less than five thin sections (Note 6).

NOTE 6—If specimens from some geosynthetic products are not long enough to provide two full random fields of view (*R f*) with the glass overlay in position, additional specimens must be prepared to meet the ten-reading requirement.

7.9 Record all ten (10) readings (calculation) obtained and express the result rounded to the nearest whole number.

8. Reporting

8.1 Identify the sample(s) for the material or product tested, including sample type, origin, and manufacturer's code or batch number.

8.2 Method of preparation of the specimens (i.e. microtome, frozen specimen, heated specimen, etc).

8.3 Report all 10 R*f* calculations obtained to the nearest whole number

9. Precision and Bias

9.1 *Precision*—The precision of this test method is being established.

9.2 *Bias*—No justifiable statement can be made on the bias of this test method since the true value cannot be established by accepted referee methods.