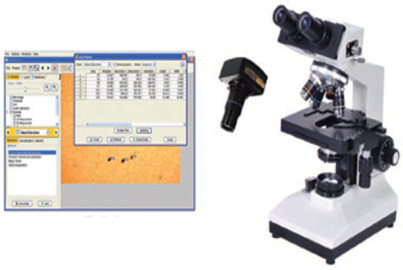
ASTM D5596 – Standard Test Method for Microscopic Evaluation of the Dispersion of Carbon Black in Polyolefin Geosynthetics / Testing Equipment

Description



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 (Rf) of view. From center point of slide make a mark 5 mm to either side. Use a straightedge 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 mm 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 (1 in.). 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 geomembrane specimen (see Note 2). Non-oriented geosynthetics material specimens can be prepared without regard to processing direction. The use of tetrafluoroethane stiffening 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.

Figure – 1 Microscope Cover Slide Overlay Configuration

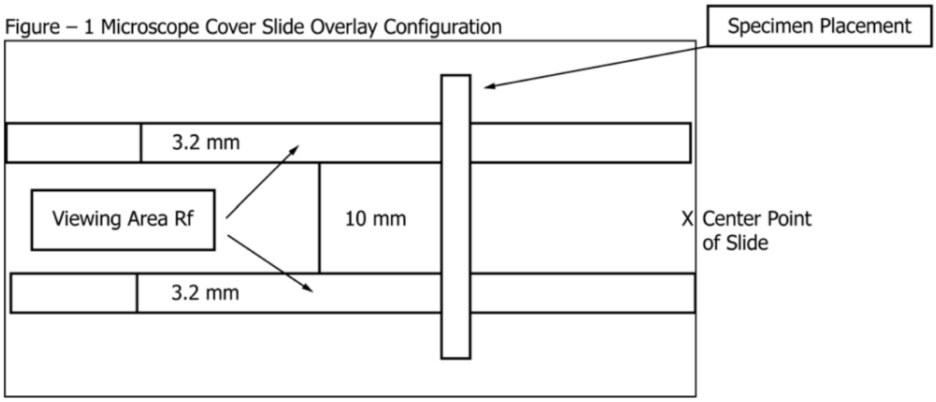


FIG. 1 Microscope Cover Slide Overlay Configuration

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 min 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 min 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, making it easier to slide off the blade and onto the slide glass.

7.3.1 Mount five 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 parallel 3.2 mm viewing area of cover slide is considered the random field of view (Rf). (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 (Rf) 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 fields 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 (Rf).
7.7 Microscopic Evaluation—Examine each (Rf) 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 (Rf)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 (Rf) with the glass overlay in position, additional specimens must be prepared to meet the ten-reading requirement.
7.9 Record all ten 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 ten (Rf) calculations obtained to the nearest whole number

ISO 18553 – Method for the Assessment of the Degree of Pigment or Carbon Black Dispersion in Polyolefin Pipes, Fittings and Compounds / Testing Equipment

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