



Standard Practice for Microscopical Examination of Pigment Dispersion in Plastic Compounds¹

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1. Scope

1.1 This practice describes microscopical examination of plastic compounds to check quality of pigment dispersion. Thin sections of polymers are prepared for observation with transmitted light. Microscopical examination is normally made for grading or classification by comparison against observational standards.

1.2 Operations are listed for practice generally applicable to many thermoplastic compounds. Details specific to wire- and cable-grade Type I polyethylene is given in Appendix X1.

NOTE 1—There is no similar or equivalent ISO 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 883 Terminology Relating to Plastics²

D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer²

F 412 Terminology Relating to Plastic Piping Systems³

3. Terminology

3.1 **Definitions**—Terms applicable to this practice are defined in Terminologies D 883 and F 412.

4. Significance and Use

4.1 Examination is made because of the general relationship between observed quality on the microscopical scale and overall serviceability, and to some extent the appearance, of the plastic compound. The method may be employed to measure degree of dispersion of pigments, presence of foreign matter, spots of unpigmented resin, and resin degradation. Serviceability of a compounded plastic can be quite sensitive to the quality

of pigment dispersion. Black, weather-resistant polyethylene serves as a good example where quality of the dispersion is indicative of weatherability which is achieved by effective screening of ultraviolet light by the carbon black.

4.2 The test is directly applicable to plastic compounds that are translucent in thin (microscopical) sections. Up to 5 % carbon black-filled polyethylene, for example, could be so examined for dispersion. The test may also be employed indirectly with opaque samples or pigment concentrates. Concentrates (which are extended in use) may be diluted with base resin to their final pigment level, as for example, by roll milling, such that examination of test specimens may be made with transmitted light. As such processing can effect improved dispersion, dilution must not be employed without regard to end use of the material being sampled.

4.3 Examination and report are normally made with respect to observational standards which are not included in this practice (see Section 7).

5. Apparatus

5.1 **Microscope**, 100-power (10× eyepiece, 10× objective), unless otherwise agreed.

5.2 **Eyepiece Micrometer** (optional), calibrated for magnification employed. A stage micrometer (standard calibrating slide), such as one having a 2-mm scale divided into units of 0.01 mm (10 μm), may be employed to calibrate the eyepiece scale.

5.3 **Light Source**, suitable for the microscope.

5.4 **Microscope Slides**, nominal 25 by 75 by 1 mm (1 by 3 by 0.040 in.)

NOTE 2—A liquid mixture of approximately the composition by volume given as follows has been found suitable for cleaning microscope slides. After being cleaned, the slides shall be wiped dry with lint-free cleansing tissue.

Ethyl alcohol (any grade)	85 %
Methyl alcohol (reagent grade)	10 %
Concentrated ammonium hydroxide (stock solution)	5 %

5.5 **Hot Plate**, capable of maintaining temperature appropriate for press-out of samples.

5.6 **Lens-Cleaning Paper**.

5.7 **Pyrometer**, or other suitable device capable of measuring surface temperatures of hot plate.

5.8 **Metal Shimstock, Brass, Aluminum, or Other Metal**—Two pieces 3.8 cm by 7.9 mm (1.5 by 5/16 in.), to control

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ *Annual Book of ASTM Standards*, Vol 08.04.

thickness in pressing test specimens. Shimstock of 25- μ m (0.001-in.) thickness has been found useful, but may be varied to accommodate samples of different opacity.

5.9 *Wood Block*, with flat surface to be placed on the top microscope slide of test specimen assembly to distribute force in pressing.

6. Test Specimens

6.1 Samples of concentrate may be diluted with unpigmented base resin to the intended pigment content of the final compound. The base resin used must be the same as that to be used in the final compound. Also as dilution can be performed in a variety of ways such as roll milling, passing through an extruder, etc., the operation must be identified and the technique performed in accordance with specific purchaser-seller agreements.

6.2 Prepare specimens on microscope slides for viewing.

6.2.1 Wash slides, knife blade, and all other items which come in contact with samples. These items must be dried with cleansing tissue.

6.2.2 Cover the top surface of the hot plate with clean aluminum foil, and set controls for a temperature suitable to press-out the polymer under test. Change the aluminum foil of any polymer contamination, and clean prior to placing slides on surface.

NOTE 3—The hot plate should be controlled at a temperature chosen according to polymer type, its melt viscosity, and any other factor such as thermal sensitivity of the pigment system. Thermoplastics with melt flow rates at condition 190/2.16 of Test Method D 1238, greater than 1.0 g/10 min might well be pressed at 175 to 225°C, while those with melt flow rates less than 1.0 g/10 min might be pressed at 200 to 250°C. Poly(vinyl chloride) should be pressed at a temperature below 200°C while nylon would require temperatures as high as 300°C.

6.2.3 Cut six small specimens approximately 1.6 mm ($\frac{1}{16}$ in.) or less in diameter from the interior of six separate granules or pellets of the compound sample or diluted concentrate. Center specimens at about 10-mm ($\frac{3}{8}$ -in.) intervals on a clean slide, and immediately place a second clean slide squarely on the one containing the specimens forming an assembly of two slides with the specimens between the slides.

NOTE 4—Use of a slide in place of a cover glass over the specimens presents no problems in viewing at magnification of 100 power. Higher magnification may require use of a cover glass.

6.2.4 Insert a metal shim of an appropriate thickness at each end of the slide assembly, place the assembly on the hot plate, and press down fully with the block of wood. All six specimens will then be pressed to the same thickness, and between 4.7 and 6.4 mm ($\frac{3}{16}$ and $\frac{1}{4}$ in.) in diameter. If some specimens are not within this diameter a new slide is prepared.

NOTE 5—Some prefer to space the specimens further apart and insert a shim between adjacent specimens. Then three specimens at least 10 mm apart are conveniently accommodated in each slide assembly.

6.2.5 Remove slides from the hot plate and place on a heat-resistant surface to cool. Do not heat slides any longer than necessary because excessive heating may cause oxidation of resin and give misleading results.

6.2.6 Metal shims may be removed.

7. Standardization

NOTE 6—Observational standards will normally be employed for comparison, rating, and report of the microscopical examination. Such standards are not, however, included in this practice, but typically would be developed and employed according to purchaser-seller agreements, or preferably as specified in relevant material specifications.

7.1 Observational standards should be based on standard resins, and take the form of specially prepared microscope slides or photomicrographs of such slides. Color photomicrographs may be necessary, but black and white should suffice where only size and shape are important. Use of color photomicrographs would require definition of the illumination used in viewing.

7.2 Standards should be numbered in an ascending order with quality from the best (one) to the worst (ten or other maximum number). Direct utilization and full effectiveness of this practice is achieved only when the point of minimum acceptability is defined on this scale.

7.3 Dispersion standards must be established for each polymer-pigment system under evaluation.

7.4 Additional standards may also be developed for agglomerate size, spots of unpigmented resin, impurities, background, etc.

NOTE 7—Color and texture of background are important clues to ultimate dispersion of pigments having particle size well below that discernible at magnification of 100 power.

8. Procedure

8.1 Place the specimen assembly on the stage of a microscope and compare at a magnification of 100 with the proper observational standards using the suitable substage source of illumination. Rate each specimen by reporting the value of dispersion standard nearest in appearance.

8.2 Measure the diameter of the largest particle within each specimen by using a grid or scale. If the particle is not spherical, the size is described by the average of the length and width of the particle.

8.3 Compare each specimen to standards on the presence of unpigmented resin and determine the average rating for each slide. Note identifiable oxidized material in this rating; also smears, and light and dark areas.

8.4 Retests are normally performed if ratings on the six specimens of a test slide assembly differ from each other by more than one rating value, except for materials expected to be nonuniform.

9. Report

9.1 Report the following information:

9.1.1 Complete identification of the material tested including a description of any extension or letdown of concentrate,

9.1.2 Identification of observational standards,

9.1.3 Numerical rating of each specimen in each test assembly, against the pertinent series of observational standards (dispersion, foreign matter, etc.),

9.1.4 Average rating and lowest quality specimen observed, and

9.1.5 Any unusual observation.

