Designation: D 4797 – 88 (Reapproved 2004)

Standard Test Methods for Chemical and Gravimetric Analysis of White and Yellow Thermoplastic Traffic Marking Containing Lead Chromate and Titanium Dioxide[1]

This standard is issued under the fixed designation D 4797; 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 These test methods cover procedures for the chemical and gravimetric analysis of white and yellow thermoplastic traffic marking containing lead chromate and titanium dioxide pigment.

1.2 The analytical procedures appear in the following order:

	Sections
Percent Binder	10
Percent Glass Beads (Note 1)	11
Percent Titanium Dioxide	12
Percent Lead Chromate and Analysis of Chrome Yellow and Chrome Orange Pigments (Note 2)	13

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—Test for 1.50 glass spheres only.

NOTE 2—This modified analysis of chrome yellow and chrome orange pigments must be used because the heat resistant chrome yellows in the thermoplastic cannot be analyzed by Test Methods D 126.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 126 Test Methods for Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate and Chromium Oxide Green

D 883 Terminology Relating to Plastics

D 1193 Specification for Reagent Water

D 1394 Test Methods for Chemical Analysis of White Titanium Pigments

F 412 Terminology Relating to Plastic Piping Systems

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminology D 883 and F 412, unless otherwise indicated.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *ash*, *n*—the inorganic components of thermoplastic traffic marking including the pigment, glass spheres, and filler.

3.2.2 *binder*, *n*—the organic components of thermoplastic traffic marking that bind the pigments, glass spheres, and filler together as a unit.

3.2.3 *filler*, *n*—the inorganic components of thermoplastic traffic marking not including the pigments or glass spheres.

3.2.4 *pigment*, *n*—titanium dioxide and lead chromate colorants.

3.2.5 *thermoplastic*, *n*—See *thermoplastic traffic marking*.

3.2.6 *thermoplastic traffic marking*, *n*—a highly filled 100 % total solids highway marking system that when heated to a molten state can be extruded or sprayed onto a road surface and when cooled forms a solid durable delineator.

4. Summary of Test Method

4.1 The thermoplastic material is prepared for the described test methods by melting a sample to its application temperature under continuous agitation. The specimen is then poured into round patties on a clean tin plate or baking pan. The patties are then broken into pieces for ignition in a muffle furnace. The percent binder is calculated from the ashed specimen and the various tests for glass spheres, titanium dioxide, and lead chromate pigment are performed on the ashed residue. The tests for pigment type or glass spheres may be run on the same ashed specimen. Specimen selection and preparation are the same for each sample tested.

5. Significance and Use

5.1 The function of these test methods is to define the percent of binder, glass, titanium dioxide, and lead chromate present in the composition of the thermoplastic traffic marking

[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

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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.

as defined by the applicable specification for the manufacture of a specific thermoplastic traffic marking.

6. Apparatus

6.1 *Balance*, analytical, accurate to 0.1 mg.

6.2 *Buret*, 10 mL, 0.1-mL divisions.

6.3 *Buret*, 50 mL, 0.1-mL divisions.

6.4 *Crucibles*, 30 mL, porcelain.

6.5 *Desiccator*.

6.6 *Erlenmeyer flask*, 500 mL.

6.7 *Furnace (Muffle)*, capable of maintaining 1100°C.

6.8 *Hot Plate*, capable of maintaining 537°C.

6.9 *Jones Reductor*.

6.10 *Mortar and Pestle*, glazed ceramic or other impervious type.

6.11 *Oven*, capable of maintaining 260°C.

6.12 *Sieve*, 3 in., 45-μm (No. 325) (metal).

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

7.3 *Alcohol*—Ethyl alcohol 95 %.

7.4 *Potassium Chloride* (KCl).

7.5 *Potassium Hydroxide Solution*—Dissolve 50 g of potassium hydroxide (KOH) in 100 mL of freshly distilled water.

7.6 *Potassium Iodide* (KI).

7.7 *Potassium Permanganate Solution* ($KMnO_4$) (0.1*N*).

7.8 *Sodium Thiosulfate, Standard Solution*—($Na_2S_2O_3 \cdot 5H_2O$) (0.1 *N*).

7.9 *Starch (Soluble) Indicator Solution*—Dissolve 10 g of soluble starch in 1 L of deionized or distilled water.

7.10 *Hydrochloric Acid Solution* (HCl) (1 + 2).

7.11 *Hydrochloric Acid, Concentrated* (HCl).

8. Sampling

8.1 Samples may be obtained by an appropriate quartering or riffle sampling method where deemed necessary considering the physical form of the material.

9. Preparation of Specimens

9.1 Melt a sample of approximately 1000 g of thermoplastic traffic marking to 210 to 218°C under continuous agitation on a hot plate set at 537°C or stir every 15 min in an oven set at 260°C.

9.2 Flow the sample out on a smooth clean surface and allow it to cool to room temperature. Patties 3 mm (⅛ in.) thick will facilitate breaking up specimens for the described analysis.

9.3 Break the specimen into small pieces and weigh 10 g to the nearest 0.1 mg into a 30-mL weighed crucible.

9.4 Cover the crucible and place into a muffle furnace preheated to 540°C and ash for 1 h or until no carbonaceous materials remain.

9.5 Remove the crucible with the ashed remains of the specimen and place into a desiccator and cool to room temperature.

10. Percent Binder

10.1 *Procedure*:

10.1.1 Weigh the crucible and ash (see Section 9) to the nearest 0.1 mg and calculate the percent of organic binder *D* as follows:

$$D = [1 - (S/W)] \times 100 \quad (1)$$

where:

S = ashed weight of thermoplastic specimen, g, and
W = weight of thermoplastic specimen, g.

11. Percent Glass Beads

11.1 *Interferences*—Acid-insoluble fillers will affect the glass-sphere analysis and must be removed by some physical separation method or accounted for quantitatively, or both.

11.2 *Procedure*:

11.2.1 Weigh the crucible and ash (see Section 9) to 0.1 mg and calculate the percent ash.

11.2.2 After the ashed material has been weighed, transfer the ash to a mortar and pestle and grind with minimal pressure to reduce the ash to a fine grained consistency without crushing the beads. Carefully transfer the ashed contents into a 400-mL beaker.

11.2.3 Add to the ash approximately 150 mL of cold 1 + 2 HCl and stir occasionally until the effervescence has ceased completely.

11.2.4 Place the beaker on a hot plate preheated to approximately 260°C and boil for 20 min under continuous agitation to dissolve all acid-soluble filler pigments.

11.2.5 Remove the beaker from the hot plate, and while hot, immediately dilute the contents with 150 mL of cold water. Allow the beads to settle. Decant the water carefully so as not to lose any glass beads.

11.2.6 Continue diluting with 150-mL aliquots of water and decanting until the water is nearly clear. Then transfer the residue into a weighed 3-in. 45-μm (No. 325) sieve and wash with 500 mL of cold water.

11.2.7 Dry the sides and the bottom of the sieve with a paper towel and dry for 1 h in a gravity oven preheated to 100°C.

11.2.8 Place the sieve in a desiccator and cool to room temperature. Weigh the sieve and glass beads to 0.1 mg and calculate the percent beads *G* as follows:

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.