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.

Designation: D4797 – 17 (Reapproved 2022)

Standard Test Methods for Gravimetric Analysis of White and Yellow Thermoplastic Pavement Marking[1]

This standard is issued under the fixed designation D4797; 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 gravimetric analysis of the binder and hydrochloric Acid (HCL) insoluble particles in white and yellow thermoplastic pavement markings. The HCL insoluble particles can be retroreflective optics, such as glass beads or some other type of retroreflective optic, or non-retroreflective particles such as silica sand, or a combination of any two or more of these materials.

1.2 This standard does not address the physical separation and the individual quantification of each component when a mixture of two or more HCL insoluble materials is present. Rather it requires the user to visually evaluate the HCL insoluble material (obtained from following this test method) and report the types of materials present.

1.3 This standard does not purport to address the titanium dioxide or lead chromate pigment measurement (after ashing) which is detailed in Test Methods D1394 and D126.

1.4 This standard will attempt to address the interference of organic pigments with the binder results.

1.5 The analytical procedures appear in the following order:

	Sections
Percent Binder	10
Percent Retroreflective Optics or Non-Retroreflective Particles	11

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

1.7 *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.*

1.8 *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]

D126 Test Methods for Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate and Chromium Oxide Green

D1394 Test Methods for Chemical Analysis of White Titanium Pigments

D7307 Practice for Sampling of Thermoplastic Pavement Marking Materials

D7308 Practice for Sample Preparation of Thermoplastic Pavement Marking Materials

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *ash, n*—the inorganic components of thermoplastic pavement marking including the pigment, retroreflective optics, and filler.

3.1.2 *binder, n*—the organic components (resinous components) of thermoplastic pavement marking that bind the pigments, retroreflective optics, and filler together as a unit.

3.1.3 *filler, n*—the inorganic components of thermoplastic pavement marking not including the pigments, retroreflective optics, or non-retroreflective particles that are considered functional.

3.1.4 *retroreflective optic, n*—functional particle that reflects and returns a relatively high proportion of light in a direction close to the light source. This characteristic is maintained over a wide variation of the angle made by the incident light ray and normal to the retroreflective surface. This includes a single

[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.

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component structure such as a spherical glass bead or a composite optic such as a core with a surface covered by small reflectors or such as a cluster of small reflectors bonded together.

3.1.5 *non-retroreflective particles, n*—functional particle that is insoluble in HCL, such as aluminum oxide, ground glass, quartz, etc., that are added for skid resistance or other non-retroreflective functional purpose.

3.1.6 *pigment, n*—titanium dioxide, lead chromate colorants, other inorganic or organic pigments, or combinations thereof.

3.1.7 *thermoplastic, n*—See *thermoplastic pavement marking*.

3.1.8 *thermoplastic pavement 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.

3.1.9 *hydrochloric acid (HCL) insoluble particles, n*—retroreflective optics, such as glass beads or some other type of retroreflective optic, or non-retroreflective particles such as silica sand, or a combination of any two or more of these materials.

4. Summary of Test Method

4.1 Thermoplastic pavement marking 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 non-stick surface such as a baking pan. The patties are then broken into pieces for ignition in a muffle furnace. The percent binder is calculated from the ashed specimen. The various tests for retroreflective optics, non-retroreflective particles, titanium dioxide, and lead chromate pigment can be performed later on the ashed residue. Determining the binder content of organic pigment containing thermoplastic may not be as straight forward. Specimen selection and preparation are the same for either sample type.

5. Significance and Use

5.1 The function of these test methods is to define the percent of binder and retroreflective optics or non-retroreflective particles in the composition of the thermoplastic pavement marking as defined by the applicable specification for the manufacture of a specific thermoplastic pavement marking. The subsequent sample, as a result of ashing can be used to later test for the presence of titanium dioxide, lead chromate and possibly organic pigments.

6. Apparatus

6.1 *Balance,* analytical, capable of weighing to 0.1 mg.

6.2 *Crucibles,* 30+ mL, porcelain or aluminum pan. (**Warning**—Some aluminum pans will degrade at high temperatures.)

6.3 *Desiccator.*

6.4 *Furnace (Muffle),* capable of maintaining 540 °C (1004 °F).

6.5 *Hot Plate or Heating Mantle,* capable of heating a can of thermoplastic to 218 °C (425 °F).

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

6.7 *Buchner Funnel.*

6.8 *Vacuum Flask and Rubber Hose.*

6.9 *Vacuum Pump.*

6.10 *Oven* capable of reaching 218 °C (425 °F).

6.11 *Microwave Oven.*

6.12 *400 mL Beaker* or acid proof container.

6.13 *Magnetic Stirring Bar.*

6.14 *Magnetic Stirring Plate.*

6.15 *Spatula.*

6.16 *Glass Beaker or Plastic Cup.*

7. Reagents

7.1 *Hydrochloric Acid Solution* (HCL) (1 + 1 concentrated HCL diluted with equal volume of water).

7.2 *Hydrochloric Acid, Concentrated* (HCL).

8. Sampling

8.1 Samples may be obtained in accordance with Practice D7307 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 thermoplastic pavement marking in accordance with Practice D7308 to 218 °C (425 °F) (or per manufacturers recommended processing temperature) under continuous agitation on a hot plate or stir every 15 min in an oven set at 218 °C (425 °F) or per manufacturer's recommended processing temperature.

NOTE 1—Thermoplastic pavement marking is manufactured in a wide variety of viscosities at 218 °C. Some viscosities are so low that the retroreflective optics settle quickly. In order to prevent any settlement during the sampling process, removing the test sample at a lower temperature is warranted. Some thermoplastic test samples are best poured as low as 160 °C (320 °F) as long as they can flow into patties.

9.2 Flow the sample out on a smooth clean non-stick surface and allow it to cool to room temperature. Patties approximately 3 mm (⅛ in.) thick are usually easy to break into specimens for the described analysis.

9.3 Break the specimen into small pieces and weigh to the nearest 0.1 mg into a weighed crucible that is at least twice the volume of specimen. The binder test can be done on samples as small as 10 g (0.353 g) and be effective. Larger samples can supply more retroreflective optics or non-retroreflective particles for their evaluation later if required.

9.4 Cover the crucible and place into a muffle furnace preheated to 540 °C (1004 °F) and ash for 1 hour or until no carbonaceous material remains.

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

iTeh Standards (https://standards.iteh.ai) Document Preview ASTM D4797-17(2022)

10. Percent Binder

10.1 *Interferences*—If yellow organic-pigment-containing thermoplastic pavement marking samples are ashed, the organic pigment may begin to degrade; therefore increasing the binder results. If the theoretical amount of organic pigment content of the sample is known and an assumption can be made as to whether some or all the organic pigment has degraded (depending on its degradation temperature and the amount of filler in the pigment), then that amount can be accounted for in the calculation in Eq 1.

10.2 *Procedure*—Weigh the crucible and ash (see Section 9) to the nearest 0.1 mg and calculate the percent binder D as follows:

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

where:

S = ashed weight of thermoplastic specimen, g, (crucible + ash wt.) – crucible wt.
W = weight of thermoplastic specimen, g, (crucible + thermoplastic specimen) – crucible wt.

11. Percent Retroreflective Optics (RO) or Non-retroreflective Particles (NRP) using Hydrochloric Acid (HCL)

11.1 *Procedure:*

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

11.1.2 After the ashed material has been weighed, transfer the ash to a 400-mL beaker or other acid-proof container and with minimal pressure, break apart the ashed specimen without crushing the retroreflective optics.

11.1.3 Add to the ash approximately 50 mL to 150 mL (1.7 oz. to 5 oz.) of cold 1 + 1 HCL and stir occasionally until most of the effervescence has ceased. **(Warning**—This is best performed under a vented hood. Also, this procedure can be performed using warmed HCL but is not necessary.)

11.1.4 Once the solution has stopped effervescing, dilute the contents with as much water as possible to allow the RO or NRP to settle. Decant the water and particulate carefully so as not to lose RO or NRP.

11.1.5 If all the ash residue has not gone into solution, reintroduce HCL solution and stir. Continuous agitation can best be achieved with a magnetic stirring bar and magnetic stirrer. Stirring with a spatula can achieve similar results.

11.1.6 Continue adding HCL or diluting with water and decanting until only beads and clear water are left in the cup/beaker.

11.1.7 Transfer the residue into a weighed 3-in. 45-µm (No. 325) sieve or a Buchner funnel containing a weighed corrugated coffee filter and wash with enough cold water to remove any residue. If the RO or NRP do not appear clean, then repeat the acid washing process.

11.1.8 Dry the sides and the bottom of the sieve with a paper towel and dry for 1 hour in a gravity oven preheated to 100 °C (212 °F). If using a Buchner funnel and coffee filters, a vacuum can be pulled on the system to evacuate the water and then the RO/NRP-containing-filters can be dried in a oven or microwave oven.

11.1.9 Place the sieve or coffee filter in a desiccator and cool to room temperature.

11.1.10 Weigh the sieve/filter and HCL insoluble particles (RO or NRP) to 0.1 mg and calculate the percent as follows (see Note 2):

NOTE 2—Most thermoplastic pavement marking ashed residue which is insoluble in HCL is a retroreflective optic.

$$\%HCL\ \ Insoluble\ Particles = (R/W) \times 100 \quad (2)$$

where:

R = weight of residue after acid wash (g) (not including sieve/filter wt.)
W = original weight of thermoplastic specimen (g) (not including crucible wt.)

11.1.11 After completing the calculation examine the sample of acid insoluble particles obtained during the test to determine the qualitative composition of the material. Include in the report the qualitative composition of the residue. For example, the HCL insoluble particles obtained from this test are composed of 100 % retroreflective optics, or the HCL insoluble particles obtained from this test are composed of both retroreflective optics and non-retroreflective particles.

12. Precision and Bias[3]

12.1 The precision of this test method is based on an interlaboratory study of ASTM D4797–12, Standard Test Methods for Gravimetric Analysis of White and Yellow Thermoplastic Traffic Marking, conducted in 2012. Seven laboratories participated in the study, testing four different thermoplastic materials. Every analyst was instructed to report four replicate test results in this study. Practice E691 was followed for the study design; the details are given in ASTM Research Report No. RR:D01-1166.

12.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same paint, obtained by the same operator using the same equipment on the same day in the same laboratory.

12.1.1.1 Repeatability limits are listed in Table 1 and Table 2.

12.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same paint, obtained by different operators using different equipment in different laboratories.

12.1.2.1 Reproducibility limits are listed in Table 1 and Table 2.

12.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

12.1.4 Any judgment in accordance with statements 12.1.1 and 12.1.2 would have an approximate 95 % probability of being correct.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1166. Contact ASTM Customer Service at service@astm.org.