



Designation: D7567 – 09

Standard Test Method for Determining Gel Content in Crosslinked Ethylene Plastics Using Pressurized Liquid Extraction¹

This standard is issued under the fixed designation D7567; 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 The gel content of crosslinked ethylene plastics is determined by solvent extraction with xylene or toluene. This test method is applicable to ethylene plastics of all densities, including those containing fillers; provided the fillers are insoluble in the extraction solvent(s).

1.2 This test method uses pressurized liquid extraction (PLE) to increase the speed and reduce the amount of solvent required for solvent extraction. The results of this method are similar to Test Method D2765.

1.3 Extraction tests shall be performed on samples of any shape (see 7.2). Specimens shall be selected from those portions of the article most susceptible to insufficient crosslinking or selected from portions representative of the entire article. This test method has been developed for production and quality control of crosslinked polyethylene pipe but may be applicable to other products such as multilayer materials and fibers.

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

1.5 This method makes use of minimal reagents and allows for solvent recovery and re-use. Due to the use of minimal reagents, health and safety concerns are minimized in comparison to other methods.

1.6 *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.* Specific precautionary statements are given in Section 10.

NOTE 1—There is no known ISO equivalent to this test method. This test method is similar to Test Method D2765.

2. Referenced Documents

2.1 ASTM Standards:²

D297 Test Methods for Rubber Products—Chemical Analysis

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

D1603 Test Method for Carbon Black Content in Olefin Plastics

D2765 Test Methods for Determination of Gel Content and Swell Ratio of Crosslinked Ethylene Plastics

D3351 Test Method for Gel Count of Plastic Film (Withdrawn 2000)³

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

3. Terminology

3.1 Description of Terms Specific to This Standard:

3.1.1 *gel content*—the percentage by mass of polymer insoluble in a specified polymer after extraction under the specified conditions.

3.1.2 *soluble*—capable of being loosened or dissolved, susceptible of being dissolved in or as if in a fluid. (See Webster's Ninth New Collegiate Dictionary, 1988.)

4. Summary of Test Method

4.1 Specimens of the crosslinked ethylene plastic are weighed, placed in an extraction cell, and extracted under pressure at a specified temperature for a specified time. After extraction, the specimens are removed, dried, and reweighed. The amount of material extracted is calculated.

5. Significance and Use

5.1 Many important properties of crosslinked ethylene plastics vary with the gel content. Hence, determination of the gel

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods and is the direct responsibility of Task Group D20.70.01 on Physical Properties.

Current edition approved Nov. 15, 2009. Published December 2009. DOI: 10.1520/D7567-09.

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.

³ The last approved version of this historical standard is referenced on www.astm.org.

content provides a means of both controlling the process and rating the quality of finished products.

5.2 Extraction tests permit verification of the desired gel content of any given crosslinked ethylene plastic and they also permit comparison between different crosslinked ethylene plastics, including those containing fillers, provided that, for the latter, the following conditions are met: The filler is not soluble in xylene or toluene at the extraction temperature and the amount of filler present in the compound is known or can be determined.

5.3 Sufficient crosslinking has been achieved to prevent migration of filler during the extraction. Usually it has been found that, at extraction levels up to 50 %, the extraction solvent remains clear and free of filler.

5.4 Since some oxidative degradation of the material can occur at the extraction temperatures used in this procedure, despite the use of a closed cell which allows for minimal introduction of oxygen, a suitable antioxidant is added to the solvent to inhibit such degradation.

5.5 Before proceeding with this method, reference should be made to the specification of the material being tested. Any test specimen preparation, conditioning, dimensions, or testing parameters, or combination thereof, covered in the materials specification shall take precedence over those mentioned in this test method. If there are no material specifications, then the default conditions apply

6. Precautions

6.1 This test method measures a much larger three-dimensional polymer network than that measured by Test Method **D3351** and should not be confused with it.

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and 50 ± 10 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice **D618**, for those tests where conditioning is required. In cases of disagreement, the tolerances shall be $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) and ± 2 % relative humidity.

7.2 Because conditioning typically will result in an increase in the gel content of ethylene polymers grafted with silane, conditioning may not be required if this method is being used to prove that a minimum gel content percentage has been achieved. When this method is used for production monitoring purposes, no conditioning is required.

7.3 *Test Conditions*—Conduct tests in the standard laboratory atmosphere of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and 50 ± 5 % relative humidity unless otherwise specified in the test methods. In case of disagreement, the tolerances shall be $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) and ± 2 % relative humidity.

8. Apparatus

8.1 The extraction apparatus shall be of the following general type:

8.1.1 *Pressurized Liquid Extractor*, with metal extraction cells of 10, 11, 22, 34, 66, or 100-mL capacity, such as an ASE 100, 150, 300, or 350.⁴

NOTE 2—For one to three specimens, a 34-mL extraction cell has been used. Up to six specimens have been extracted using a 66-mL cell and up to nine specimens have been extracted using a 100-mL extraction cell.

8.1.1.1 *Specifications*. The PLE instrument shall allow programming of extraction methods up to 200°C and operate in both dynamic and static extraction modes.

(1) The PLE instrument shall allow programming of at least five static cycles.

8.2 *Planer, Lathe, or Mill*, suitable for producing shavings of the sample of less than or equal to 0.1 mm thickness and no fines of less than 60 mesh.

NOTE 3—A commercial electric wood planer with appropriate safety guards added is satisfactory, although any procedure which will produce a sample of the required thickness without generating excessive heat may be used.

8.3 *Calipers*, calibrated with precision of ± 0.02 mm or better.

8.4 *Analytical Balance*, calibrated to ± 0.001 g or better.

8.5 *US. No. 150 Stainless Steel Wire Cloth*. This mesh number allows easy cutting and folding for production of specimen pouches, and importantly, allows the specimens to swell and to be completely immersed in solvent.

8.6 *Stapler*.

8.7 *Scissors*.

8.8 *Oven*, suitable for drying samples at up to 200°C with a thermometer capable of measuring up to 200°C . A vacuum oven is well suited for this purpose; however any oven that has adequate ventilation to prevent build-up of solvent vapor may be used.

9. Reagents

9.1 *Xylenes*, ACS reagent grade, boiling point 138°C to 141°C .

9.2 *Toluene*, technical grade, boiling point 110°C .

9.3 *Antioxidant*, 2,2-methylene-bis (4-methyl-6-tertiary butyl phenol), or similar phenolic antioxidant.

10. Safety Precautions

10.1 Xylene and toluene are toxic and flammable solvents and as such should be handled carefully. Use only in a ventilated hood. Check the effectiveness of the hood before starting the tests. Do not inhale the vapors. Excessive inhalation of the vapors may cause dizziness or headache, or both. In the event of excessive inhalation, seek fresh clean air.

⁴ The sole source of supply of the apparatus known to the committee at this time is Dionex Corporation, 1228 Titan Way, P.O. Box 3603 Sunnyvale, CA 94088-3603 Tel: (408) 737-0700. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.