



# Standard Test Methods for Determination of Gel Content and Swell Ratio of Crosslinked Ethylene Plastics<sup>1</sup>

This standard is issued under the fixed designation D2765; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

1.1 The gel content (insoluble fraction) produced in ethylene plastics by crosslinking is determined by extracting with solvents such as decahydronaphthalene or xylenes. The methods described herein are applicable to crosslinked ethylene plastics of all densities, including those containing fillers, and all provide corrections for the inert fillers present in some of those compounds.

1.2 Test Method A, which permits most complete extraction in least time, is to be used for referee tests, but two alternative nonreferee Test Methods B and C are also described. Test Method B differs from the referee test method only in sample preparation; that is, it requires use of shavings taken at selected points in cable insulation, for example, rather than the ground sample required by the referee test method. Because the shaved particles are larger, less total surface per sample is exposed to the extractant, so this test method ordinarily yields extraction values about 1 to 2 % lower than the referee method. Test Method C requires that a specimen in one piece be extracted in xylenes at a constant temperature of 110°C. At this temperature and with a one-piece specimen, even less extraction occurs (from 3 to 9 % less than the referee test method), this method permits swell ratio (a measure of the degree of crosslinking in the gel phase) be determined.

1.3 Extraction tests are made on articles of any shape. They have been particularly useful for electrical insulations since specimens can be selected from those portions of the insulation most susceptible to insufficient crosslinking.

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

NOTE 1—This test method is equivalent to ISO 10147, Method B. It is

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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not equivalent to ISO 10147 in any other measurement or section.

1.5 *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 Sections 6, 9, and 24.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

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

D1998 Specification for Polyethylene Upright Storage Tanks

D3351 Test Method for Gel Count of Plastic Film (Withdrawn 2000)<sup>3</sup>

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

### 2.2 ISO Standard:

ISO 10147 Pipes and Fittings Made of Crosslinked Polyethylene (Pe-X)—Estimation of the Degree of Crosslinking by Determination of the Gel Content

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

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

3.1.2 *swell ratio*—the ratio of the gel volume in the swollen state to its volume in the unswollen state.

3.2 Terms as shown in Terminology D883 are applicable to this test method.

<sup>2</sup> 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.

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

\*A Summary of Changes section appears at the end of this standard

## 4. Summary of Test Methods

4.1 Specimens of the crosslinked ethylene plastic are weighed and then immersed in the extracting solvent at the temperature specified by the procedure selected and for the time designated by that procedure. After extraction, the specimens are removed, dried, and reweighed as directed. The amount of material extracted is calculated and, if desired, swell ratio determined by the means described in alternative Test Method C.

## 5. Significance and Use

5.1 Many important properties of crosslinked ethylene plastics vary with the gel content. Hence, determination of the gel content provides a means of both controlling the process and rating the quality of finished products.

5.2 Extraction tests permit verification of the proper 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:

5.2.1 The filler is not soluble in either decahydronaphthalene or xylenes at the extraction temperature.

5.2.2 The amount of filler present in the compound either is known or will be determined by other means.

5.2.3 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 extractant remains clear and free of filler.

5.3 A suitable antioxidant is added to the extractant to inhibit possible oxidative degradation at the extraction temperatures.

5.4 Before proceeding with this test method, reference shall be made to the specification of the material being tested. Any test specimen preparation, conditioning, dimensions, or testing parameters, or a 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 and is different than that measured by Test Method [D3351](#).

6.2 It has been reported that ultra-high molecular weight polyethylene fails to completely dissolve in this procedure at times.

6.3 Atmospheric pressure affects the boiling temperature of solvents. It is possible that laboratories performing these test methods at altitudes greater than 1500 m (5000 ft) above sea level will observe lower boiling temperatures for the solvent used. If this is observed it shall be noted in the results report.

### TEST METHOD A (REFEREE TEST METHOD)

## 7. Apparatus

7.1 The extraction apparatus shall be of the following general type, as illustrated in [Fig. 1](#):

7.1.1 *Round-Bottom Flask*, equipped with a suitable joint that has been demonstrated to provide an essentially leak free connection. For one or two determinations at one time, a 500-mL flask is appropriate. For several determinations at one time, but not exceeding six, a 2000-mL flask is suitable.

7.1.2 *Heating Mantle* to fit the flask and with sufficient heating capacity to boil decahydronaphthalene.

7.1.3 *Reflux Condenser* with ground-glass or cork joint to fit into flask.

7.1.4 *Ring Stand and Appropriate Clamps*.

7.2 *Grinding Equipment*, suitable for reducing the sample to a fineness between 30 and 60 mesh. A bench-top laboratory mill is satisfactory, although any procedure which will produce a sample of the required fineness without generating excessive heat is acceptable.

7.2.1 *U.S. No. 30 and U.S. No. 60 Sieves*.

7.2.2 *U.S. No. 120 Stainless Steel Wire Cloth*.

7.3 *Vacuum Oven*, with vacuum source capable of creating a vacuum of at least 710 mm (28 in.) Hg and equipped with a thermometer capable of measuring to 150°C.

7.4 *Analytical Balance*, capable of weighing to 0.0001 g.

## 8. Reagents

8.1 *Decahydronaphthalene (Decalin)*, anhydrous, boiling point 189 to 191°C.

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

8.3 Suitable stabilizers for the solvent include:

8.3.1 *2,2'-methylene-bis (4-methyl-6-tertiary butyl phenol)* (CAS# 119-47-1)

8.3.2 *Tetrakis-(methylene-(3,5-di-(tert)-butyl-4-hydroxycinnamate))methane* (CAS# 6683-19-8)

8.3.3 *1,3,5-trimethyl-2,4,6-tris(3,5-di-tert-butyl-4-hydroxybenzyl)benzene* (CAS# 1709-70-2)

8.3.4 *1,3,5-tris(3,5-di-tert-butyl-4-hydroxybenzyl)-1,3,5-triazine-2,4,6(1h,3h,5h)-trione* (CAS# 27676-62-6)

## 9. Safety Precautions

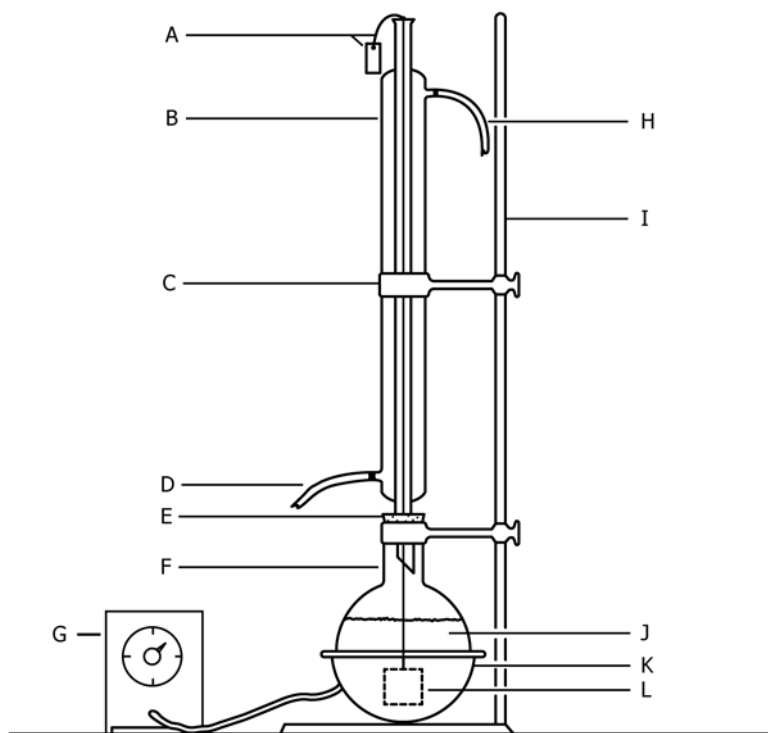
9.1 Xylenes and decahydronaphthalene are toxic and flammable solvents and shall be handled carefully. Use only in a ventilated hood. Check the effectiveness of the hood before starting the tests. Do not inhale the vapors. Excessive inhalation of the vapors has been known to cause dizziness or headache, or both. In the event of excessive inhalation, seek fresh clean air.

## 10. Test Specimens

10.1 At least two specimens each containing  $0.300 \pm 0.015$  g of ground polymer weighed to the nearest 0.0001 g shall be tested.

10.2 The test samples shall be ground to a fineness that will pass through a 30-mesh sieve. Shake this sieved material over a 60-mesh sieve and reject any material that passes through.

10.3 Test specimens originating from insulation cut from wire shall be homogeneous and consist of an equal mixture of samples representative of insulation nearest to the conductor and samples representative of insulation nearest the outer



- A—Identification tag and fine wire attached to cage.
- B—Reflux condenser.
- C—Ring stand clamp.
- D—Water inlet.
- E—Joint.
- F—Large-mouth round-bottom flask.
- G—Variable transformer.
- H—Water outlet.
- I—Ring stand.
- J—Decahydronaphthalene or xylene.
- K—Heating mantle.
- L—120-mesh wire cage containing the specimen.

NOTE 1—If a slightly higher degree of accuracy is desired (about 1 to 2 %) a modified Soxhlet Extractor may be employed wherein the specimen is held in an extraction thimble. The extraction thimble, with extra-coarse fritted glass disk sealed in, will have 45-mm body diameter, 130-mm height or equivalent (height must subsequently be cut to 75 mm). The fritted disk is extra-coarse with fused edge. Glass wool 13 to 19 mm thick ( $\frac{1}{2}$  to  $\frac{3}{4}$  in.) will be placed on the bottom of the extraction thimble to support one gram 30-60 mesh ground sample which is covered with 13 to 19 mm thick layer of glass wool, a fritted glass disk and a small glass weight. The extraction thimble assembly rests upon the 55-mm portion of the thimble previously cut off, the latter inserted into the modified Soxhlet Extractor.

FIG. 1 Extraction Apparatus

periphery of the construction unless specific portions of the insulation are being examined for insufficient crosslinking.

## 11. Procedure

11.1 Prepare a specimen holder by cutting a piece of 120-mesh stainless steel cloth measuring approximately 80 by 40 mm (3 by  $1\frac{1}{2}$  in.). Fold this to form a square measuring approximately 40 mm ( $1\frac{1}{2}$  in.). Fold two sides of this square closed by folding the cloth at the edges about 6 to 7 mm ( $\frac{1}{4}$  in.) and stapling those folds. In that manner, a pouch open at the top is obtained. Weigh this pouch (W1).

11.2 Place the ground and screened sample in the weighed pouch. Weigh the pouch and specimen (W2) to the nearest 0.0001 g. Fold over the open side of the pouch and staple the edge to form a cage. Weigh the cage and sample (W3).

NOTE 2—Compaction of specimens has been observed to negatively affect the repeatability of this method. Avoid compaction of the specimens and the specimen cages.

11.3 Fill a round-bottom flask as described in 7.1.1 with enough solvent to immerse the 120-mesh cage and sample completely during extraction. Dissolve 1 % an appropriate antioxidant (8.3) in the decahydronaphthalene or xylenes.

NOTE 3—A volume of 350-mL in a 500-mL flask or 1000-mL in a 2000-mL flask has been found to be sufficient.

11.4 Boil the decahydronaphthalene or xylenes vigorously enough to ensure good agitation of the solution. Twenty to 40 drops/min from the condenser, while using 1000 g of solvent, is a satisfactory boiling rate. Suspend the cage and specimen in the solvent so that the bottom edge of the cage is almost touching the bottom of the flask. A small wire, attached to the