



Designation: D 2765 – 95

Standard Test Methods for Determination of Gel Content and Swell Ratio of Crosslinked Ethylene Plastics¹

This standard is issued under the fixed designation D 2765; 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 (insoluble fraction) produced in ethylene plastics by crosslinking can be determined by extracting with solvents such as decahydronaphthalene or xylene. Such extraction test methods are described herein. They 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. The first of these 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. The second of the alternative test methods requires that a specimen in one piece be extracted in xylene 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) but swell ratio (a measure of the degree of crosslinking in the gel phase) can be determined.

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

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

NOTE 1—There is no equivalent ISO Method.

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 appro-*

priate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Sections 6, 10, and 25.

2. Referenced Documents

2.1 ASTM Standards:

- D 297 Test Methods for Rubber Products—Chemical Analysis²
- D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing³
- D 883 Terminology Relating to Plastics³
- D 1603 Test Method for Carbon Black in Olefin Plastics³
- D 1998 Specification for Polyethylene Upright Storage Tank³
- D 3351 Test Method for Gel Count of Plastic Film⁴
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁵

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 *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.)

3.1.3 *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 D 883 are applicable to this test method.

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

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² *Annual Book of ASTM Standards*, Vol 09.01.

³ *Annual Book of ASTM Standards*, Vol 08.01.

⁴ *Annual Book of ASTM Standards*, Vol 08.02.

⁵ *Annual Book of ASTM Standards*, Vol 14.02.

amount of material extracted is calculated and, if desired, swell ratio also may be 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 xylene at the extraction temperature.

5.2.2 The amount of filler present in the compound either is known or can be determined.

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 Since some oxidative degradation of the material can occur at the reflux temperature of the extractants, a suitable antioxidant is added to the extractant to inhibit such degradation.

5.4 Before proceeding with this test method, reference should 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 than that measured by Test Method D 3351 and should not be confused with it.

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

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 $50 \pm 5\%$ relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618, 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 $\pm 2\%$ relative humidity.

7.2 *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 $50 \pm 5\%$ relative humidity, unless otherwise specified in the test methods. In cases of disagreement, the tolerances shall be $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) and $\pm 2\%$ relative humidity.

TEST METHOD A (REFEREE TEST METHOD)

8. Apparatus

8.1 The extraction apparatus shall be of the following general type, as illustrated in Fig. 1:

8.1.1 *Round-Bottom Flask*, with large-mouth ground-glass or cork joint. 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.

8.1.2 *Heating Mantle* to fit the flask and with sufficient heating capacity to boil decahydronaphthalene (boiling point 190 to 193°C) or xylene (boiling point 138 to 141°C).

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

8.1.4 *Ring Stand and Appropriate Clamps*.

8.1.5 *Grinding Equipment*, suitable for reducing the sample to a fineness between 30 and 60 mesh. A bench-top laboratory mill⁶ is satisfactory, although any procedure which will produce a sample of the required fineness without generating excessive heat may be used.

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

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

8.1.8 *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 150°C .

NOTE 2—If a slightly higher degree of accuracy is desired (about 1 to 2 %) a modified Soxhlet Extractor; Kontes Catalog No. 58610, Size C, Kontes Glass Co., Vineland, NJ or equivalent may be employed wherein the specimen is held in an extraction thimble. The extraction thimble, with extra-coarse fritted glass disk sealed in; Corning Glass Catalog No. 33950, 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; Corning Glass Catalog No. 31000, 40-mm diameter or equivalent. Glass wool 13 to 19 mm thick ($\frac{1}{2}$ to $\frac{3}{4}$ in.) 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.

9. Reagents

9.1 *Decahydronaphthalene*, practical, boiling point 190 to 193°C .

9.2 *Xylene*, ACS reagent grade, boiling point 138 to 141°C .

9.3 *2,2'-methylene-bis (4-methyl-6-tertiary butyl phenol)*.⁷

10. Safety Precautions

10.1 Xylene and decahydronaphthalene 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.

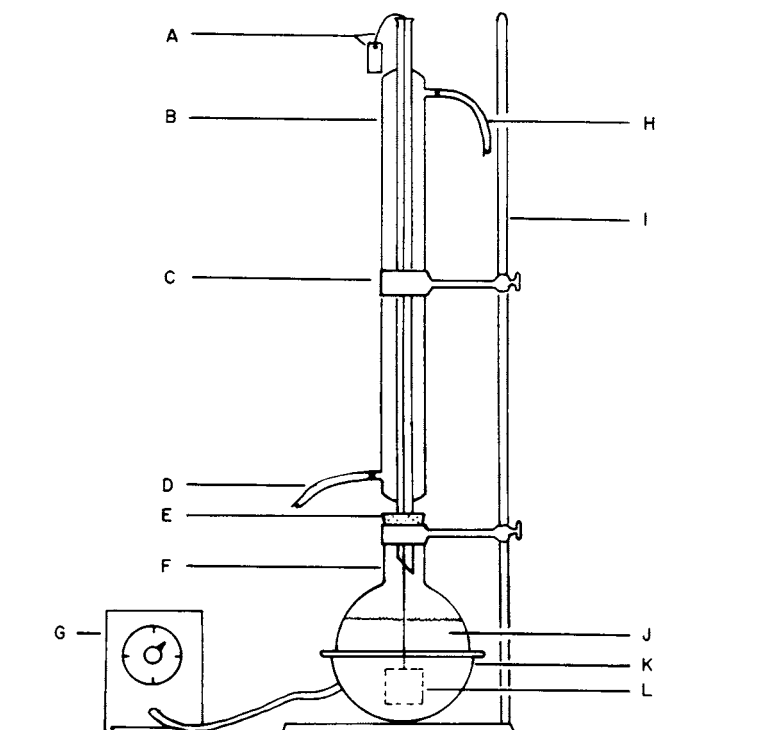
11. Test Specimens

11.1 At least two specimens each containing 0.300 ± 0.015 g of ground polymer weighed to the nearest 0.001 g shall be tested.

⁶ A Wiley Cutting Mill, Intermediate Model, or equivalent, Catalog No. 8-338, available from Fisher Scientific Co., 711 Forbes Ave., Pittsburgh, PA 15219, has been found satisfactory for this purpose.

⁷ Antioxidant, available from Cytec under the trade name Cyanox 2246, has been found satisfactory for this purpose. Cytec Industries, Polymer Additive Department, 5 Garret Mountain Plaza, West Paterson, NJ 07424.

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- A—Identification tag and fine wire attached to cage.
- B—Reflux condenser.
- C—Ring stand clamp.
- D—Water inlet.
- E—Ground-glass or cork 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.

FIG. 1 Extraction Apparatus

11.2 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.

11.3 Test specimens originating from insulation cut from wire should 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 periphery of the construction unless specific portions of the insulation are being examined for insufficient crosslinking.

12. Procedure

12.1 Prepare a specimen holder by cutting a piece of 120-mesh stainless steel cloth measuring approximately 80 by 40 mm (3 by 1½ in.). Fold this to form a square measuring approximately 40 mm (1½ in.). Fold two sides of this square closed by folding the cloth at the edges about 6 to 7 mm (¼ in.) and stapling those folds. In that manner, a pouch open at the top is obtained. Weigh this pouch (W1).

12.2 Place approximately 0.3 g of the ground and screened sample in the weighed pouch. Weigh the pouch and specimen (W2). Fold over the open side of the pouch and staple the edge to form a cage. Weigh the cage and sample (W3).

12.3 Fill a round-bottom flask as described in 8.1.1 with enough solvent to immerse the 120-mesh cage and sample completely. Three hundred fifty grams in a 500-mL flask or 1000 g in a 2000-mL flask is sufficient. Dissolve 1 % of antioxidant in the decahydronaphthalene or xylene to inhibit further crosslinking of the specimen.

12.4 Boil the decahydronaphthalene or xylene 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 cage and extending through the reflux condenser can be used for suspending the cage. Extract the specimen for 6 h in decahydronaphthalene or for 12 h in xylene.

NOTE 3—Since most of the extraction occurs within less than the specified time, the period of extraction may be shortened for control tests on the basis of experience. If this is done, the time of extraction must be reported. For referee tests, extraction time is to be as specified by this test method.

12.5 After extraction, place the cage and specimen immediately in a vacuum oven preheated to 150°C. Dry the