



Designation: D 3616 – 95 (Reapproved 2000)

Standard Test Method for Rubber, Raw—Determination of Gel, Swelling Index, and Dilute Solution Viscosity¹

This standard is issued under the fixed designation D 3616; 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 This test method covers the determination of the dilute solution viscosity (DSV), the gel characteristics, and the swelling index of raw non-oil-extended and non-pigmented SBR and NBR.

1.2 This test method may be used to determine the dilute solution viscosity and gel characteristics of rubbers other than SBR and NBR, however, solvents other than 2-butanone and toluene may be required.

1.3 This test method is not intended for the measurement of micro-gel.

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.*

2. Referenced Documents

2.1 ASTM Standards:

D 446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers²

D 2857 Practice for Dilute Solution Viscosity of Polymers³

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries⁴

2.2 ISO Standard:

ISO/R1628 (Plastics) Directives for the Standardization of Methods for the Determination of the Dilute Solution Viscosity of Polymers⁵

2.3 Other Document:

“Reagent Chemicals, American Chemical Society Specifications”⁶

3. Summary of Test Method

3.1 A weighed sample is allowed to stand 16 to 20 h in a suitable solvent. The sol, or soluble portion, is removed from the rubber mixture and the viscosity is determined on this solution.

3.2 A portion of the sol is used for the determination of the dissolved rubber. This is obtained by evaporation of the solvent.

3.3 The swelling index of the gel is determined by comparing the dry gel mass to the swollen gel mass.

4. Significance and Use

4.1 These tests are particularly useful for quality control in the production of synthetic rubbers.

4.2 Percent gel is a measure of the amount of insoluble rubber present in the chosen solvent. Gel may be introduced intentionally in some rubbers. Variations from a normal level indicate changes in the polymerization system.

4.3 Swelling index is a measure of the type of gel. Gel with a low-swelling index is referred to as “hard gel” and usually indicates the presence of material that does not break down readily by milling. A high-swelling index normally indicates the presence of a “loose gel” that may be broken down easily by milling.

4.4 In low-gel or gel-free rubbers, the dilute solution viscosity correlates directly with the molecular weight. Since the viscosity is measured only on the soluble portion of the rubber, the use of DSV to predict molecular weight in rubbers having high gel values is meaningless.

5. Apparatus

5.1 *Borosilicate Weighing Bottle*, 45/12 standard taper, 40 mm in inside diameter, 100 mm high, with cover.

5.2 *Screen Rack*, consisting of five 300-μm (No. 50) circular screens mounted on a stainless steel tube to fit the weighing bottle (5.1). Borosilicate bottle, cover, and rack will be referred to hereafter as “the unit”.⁷(Fig. 1).

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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² *Annual Book of ASTM Standards*, Vol 05.01 (formerly D2515 redesignated D446 in 1977).

³ *Annual Book of ASTM Standards*, Vol 08.02.

⁴ *Annual Book of ASTM Standards*, Vol 09.01.

⁵ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

⁶ Available from the American Chemical Society, Washington, DC.

⁷ Available from the Spencer Machine and Tool Co., P.O. Box 298, 6205 Gheens Mill Rd., Jeffersonville, IN 47131, or manufactured according to the specifications in Fig. 1.

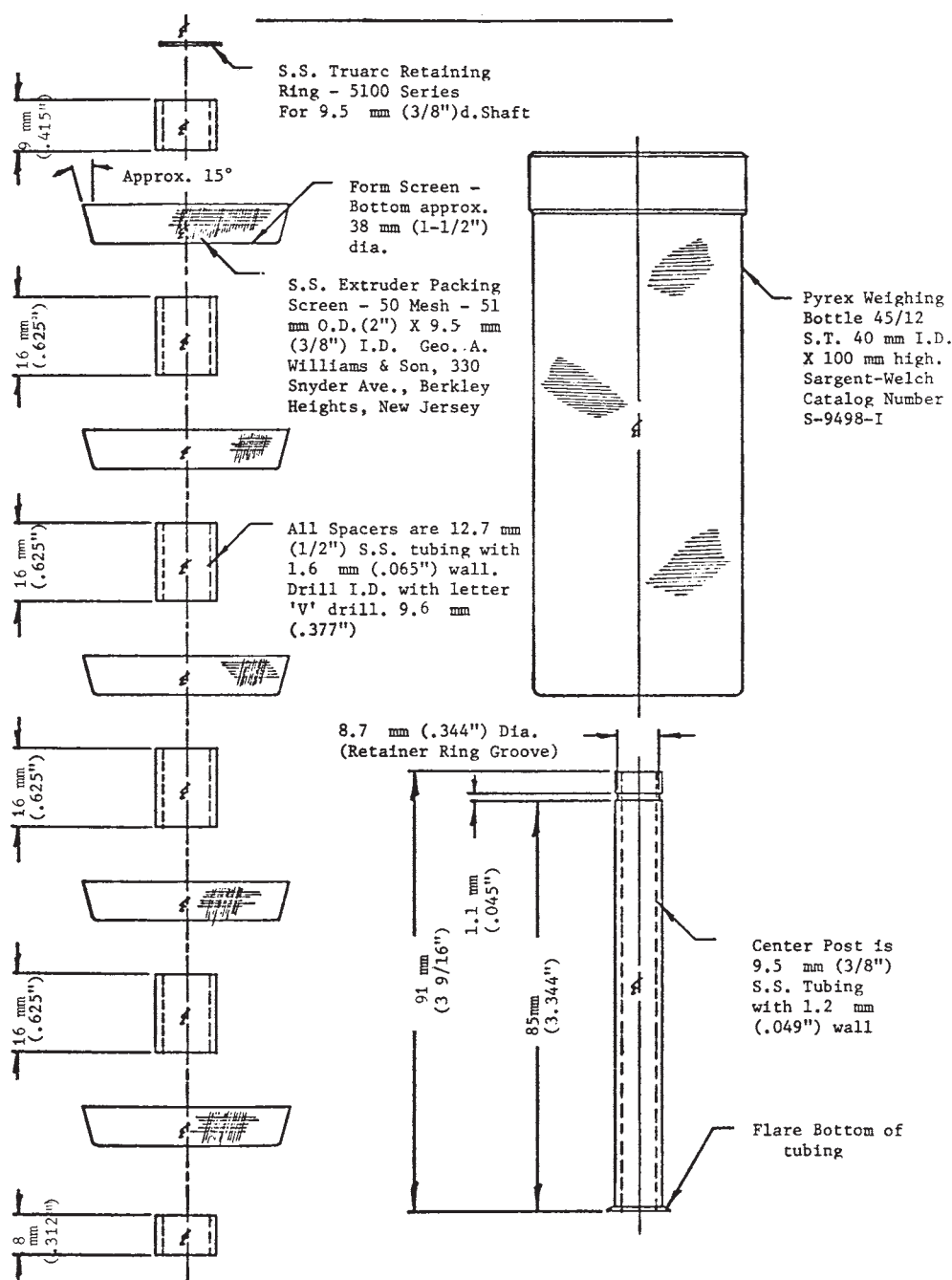


FIG. 1 Gel Test Apparatus

5.3 *Pipet*, capable of fitting within the tube of the screen rack (5.2).

5.4 *Ostwald Cannon-Fenske Viscometer*, size 100.

5.5 *Constant-Temperature Bath with Stirrer*, capable of operating at $25 \pm 0.1^\circ\text{C}$.

5.6 *Stop Watch or Electric Timer*, readable to 0.1 s.

5.7 *Additional Glassware*, sufficient to carry out the procedure as written. Class "A" pipets of 25 and 100-cm³ volume are mandatory.

5.8 *Disposable Aluminum Dishes*.

5.9 *Balance*, capable of accurately weighing to ± 0.1 mg.

5.10 *Screw-Cap Bottles*, 100-cm³ minimum capacity, or Erlenmeyer flasks, 125-cm³ capacity.

5.11 *Borosilicate Wool or Cotton*.

6. Reagents

NOTE 1—All recognized health and safety precautions shall be observed in carrying out the procedure as written.

6.1 *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. Other grades may be used, provided it is ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.