



Designation: D3616 – 95 (Reapproved 2024)

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

This standard is issued under the fixed designation D3616; 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 DSV 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *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

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

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

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

2.2 Other Document:³

“Reagent Chemicals, American Chemical Society Specifications”

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 DSV 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.

³ Available from the American Chemical Society, 1155 Sixteenth Street, NW, Washington, DC, 20036.

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” (Fig. 1).

NOTE 1—The unit may be manufactured according to the specifications in Fig. 1.

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- cm^3 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- cm^3 minimum capacity, or Erlenmeyer flasks, 125- cm^3 capacity.

5.11 *Borosilicate Wool or Cotton*.

6. Reagents

NOTE 2—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.

6.2 The following solvents are used:

6.2.1 *2-Butanone (-Methyl Ethyl Ketone)*, for NBR rubbers, and

6.2.2 *Toluene*, for SBR rubbers.

7. Sampling

7.1 Select a representative sample of the rubber to be tested.

7.2 Do not mill the sample prior to testing except for rubbers in the powdered form (see 8.2).

NOTE 3—The presence of dusting agents may result in erroneous values for total solids or viscosity of the solution.

8. Procedure

8.1 Using clean scissors, cut the sample into strips less than 1 mm in thickness and about 5 mm long.

8.2 In the case of rubbers in powdered form, the sample may be massed or compacted by the use of a hand press with platens at 50°C . It may then be cut into appropriate strips. Rubbers that will not fall through the screen may be placed on the screen without prior treatment.

8.3 Weigh 0.39 to 0.41 g of the prepared sample to the nearest 0.1 mg and distribute evenly over the four lowest screens of the unit (5.2). Place the pieces near the center of each screen.

8.4 Gently place the screen rack into the borosilicate weighing bottle (5.1) and deliver 100 cm^3 of the chosen solvent into the bottle. Tip the container to assure complete wetting of each screen.

8.5 Cap the bottle and allow to stand for 16 to 20 h at $25 \pm 2^\circ\text{C}$, in the dark.

9. Gel

9.1 After 16 to 20 h, remove the cap from the unit, lift the screen, and examine for visible gel.

NOTE 4—Some rubbers will not go into solution as readily as others. It is necessary for the analyst to distinguish between undissolved polymer and true gel. A thinner or more porous sample will aid the solution and an extended standing period, up to 48 h, may be helpful in distinguishing gel from true polymer solution. It should be noted, however, that the precision statements in Section 13 were obtained using the time interval stated in 8.5.

9.2 Pipet the liquid from the unit into a screw-cap bottle or Erlenmeyer flask by inserting the pipet (5.3) through the center of the screen rack. Filter the liquid through cotton or borosilicate glass wool.

9.3 Pipet exactly 25 cm^3 of this liquid into a dried and weighed aluminum dish (5.8) and place the dish on a steam or electric hot plate at $100 \pm 10^\circ\text{C}$. Alternately heat and weigh the dish to the nearest 0.1 mg, until a constant mass is achieved. Record this value for use in Section 9.

9.4 Determine the gel content as follows:

$$A \times 4 = B \quad (1)$$

$$\text{Gel, \%} = \left(\frac{C - B}{C} \right) \times 100 \quad (2)$$

where:

A = mass of the dried sol, 25 cm^3 volume,

B = mass of the total dried sol, and

C = mass of the original sample.

9.5 A test result is the average of two determinations.

10. Swelling Index

10.1 Remove the final traces of solvent from the unit, using suction. Do not disturb the gel on the screens.

10.2 Cap the unit and weigh quickly to ± 1 g. Record this as the wet mass of the unit plus swollen gel.

10.3 Determine the wet mass of the unit alone, without sample, at frequent intervals, by filling and emptying the unit exactly as described in 8.4 and 9.2. Standing for 16 h is unnecessary.

10.4 Determine the swelling index as follows:

$$\text{Swelling index} = (E/D)$$