Designation: D 5775 – 95 (Reapproved 2009)

Standard Test Method for Rubber—Determination of Bound Styrene in Styrene Butadiene Rubber by Refractive Index[1]

This standard is issued under the fixed designation D 5775; 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 styrene content, and is intended for general use on solid uncompounded styrene-butadiene copolymers, commonly referred to as SBR, prepared by the emulsion process. SBR polymers prepared in solution will require the use of different refractive index tables because the vinyl content of the butadiene differs from that in the emulsion polymers. It is applicable to polymers having less than 55 % bound styrene.

NOTE 1—The nomenclature used in this test method is in accordance with Practice D 1418.

1.2 *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:*[2]

D 1418 Practice for Rubber and Rubber Latices—Nomenclature

D 4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Summary of Test Method

3.1 A sample of dried ETA-extracted polymer is pressed between sheets of aluminum foil to provide sheeted rubber having a thickness of not more than 0.50 mm. The bound styrene content is calculated from the refractive index obtained at 25°C on this thinly sheeted rubber.

4. Significance and Use

4.1 The bound styrene test is a measure of the bound monomeric composition of the rubber. It is used as a check on the accuracy of monomer charging and also as a guide to the uniformity of the product, since the bound styrene content affects the physical properties.

5. Apparatus

5.1 *Spiders*, consisting of 13-mm squares of sheet aluminum or stainless steel having a Nichrome wire leg about 38 mm long attached to each corner. Where the extracting solvent is ETA-acidified with HCl, the spider and the legs should be made of tantalum.

5.2 *Abbe-Type Refractometer*, having fourth decimal place accuracy, whose refracting prism can be placed in a nearly horizontal position for measurement of the refractive index of solids. An Amici-type compensating prism for achromatization is necessary unless a sodium vapor lamp is used as a light source.

5.3 *Vacuum Oven*, capable of being evacuated to a pressure of 1.3 kPa (10 mm Hg) and of maintaining a temperature of 100°C.

5.4 *Aluminum Foil*, between 0.025 and 0.08 mm thick, having good tear strength.

5.5 *Glass Test Piece*, standard, for checking adjustment of the refractometer.

5.6 *Hydraulic Press*, that can be maintained at 100°C and can apply a force of at least 2.2 kN (500 lbf) on each specimen or of 100 kN (11 ton) if pressing plates as described in 5.7 are used.

5.7 *Pressing Plates (Optional Apparatus)*, 210 by 210 by 3 mm, with a wooden handle. One of the plates should have a 150-mm square area in the center of the plate milled out to a depth not to exceed 0.65 mm.

5.8 *Scissors*, small and sharp.

5.9 *Light Source*—The light source should be collimated to provide a beam at grazing incidence to the prism. If an incandescent light source is used, it shall be of low intensity, such as a flashlight bulb. A sodium vapor lamp may also be used. The light source requirement is that a clear sharp line with good contrast can be observed in the telescope of the refractometer. Attenuation or diffusion of the light for better viewing may be accomplished by placing crumpled tissue paper in the light path.

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[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] 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.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7. Reagents

7.1 *Acidified ETA*—Add 10 cm^3 of concentrated hydrochloric acid (HCl, density 1.19 mg/m^3) to a portion of ETA (see 7.3) and make the solution up to 1000 cm^3 with ETA.

7.2 *Alpha-Bromonaphthalene*.

7.3 *Ethanol-Toluene Azetrope (ETA)*—Mix 70 volumes of ethanol or of formula 3A ethanol and 30 volumes of toluene, reflux the mixture 4 h over calcium oxide, and distill. Discard the first and last portions, keeping only that distillate coming over within a range of 1°C. Refluxing and distilling are not necessary if anhydrous 3A ethanol is used or absolute grain alcohol is used.

8. Procedure

8.1 Sheet out the rubber to a thickness of 0.5 mm. Cut the sheeted rubber into strips approximately 13 mm wide and 25 mm long. Fasten one strip to each leg of the aluminum spider, thus allowing each portion of the rubber to be contacted on all sides by the solvent. Place the spider and strips in a 400-cm^3 flask into which 60 cm^3 of ETA have been placed (for alum-coagulated polymers, use acidified ETA and the tantalum spiders). Extract for 1 h at a temperature at which the solvent boils gently. Replace the solution with another 60 cm^3 of ETA or acidified ETA and extract for an additional 1 h, remove the spider from the flask, and dry the rubber to constant mass in the vacuum oven held at a pressure of about 1.3 kPa (10 mm Hg) and a temperature of 100 ± 5°C.

NOTE 2—It is important that the test specimens be extracted and dried thoroughly since either residual solvent or incompletely extracted materials will result in erroneous readings of the refractive index.

NOTE 3—Avoid plastication of the sample by overheating.

8.2 After the specimens have been dried thoroughly, remove them from the spiders. At this point, any one of several techniques for pressing the specimen may be used. The method of pressing may be modified to suit the type of rubber and the type of equipment available. The pressure and the time of pressing at 100°C may be varied. The specimen may be cooled to room temperature under pressure or removed from the press while hot. The time of hot pressing should never exceed 10 min, preferably not even 5 min. The conditions should be chosen so that the pressed specimen is homogeneous and so that a distinct line can be observed dividing the light and dark fields of the telescope when the refractive index is determined. Two general techniques are described for the pressing operation.

8.3 If the pressing is to be done between flat platens without a cavity, proceed as follows, modifying the details of the procedure to suit the sample: Prepare approximately 25-mm squares of the clean aluminum foil. Place a portion of one of the dried strips between two pieces of foil. Press the specimen between the foil squares with a force of between 2.2 and 5.6 kN (500 and 1500 lbf) at 100°C for from 3 to 10 min (preferably 3 to 5 min). If several specimens are pressed at one time, increase the applied force proportionally so that the pressure on each specimen is between about 3.5 and 10 MPa (500 and 1500 psi). Forces lower than the normal limits may be necessary with some rubbers. It also may be necessary, with some rubber samples, to allow the pressed specimen to cool under pressure while cooling the press platens with cold water.

8.4 If the cavity pressing plates described in Section 6 are used, proceed as follows: Place approximately 0.3 g of the dry extracted rubber between two sheets of aluminum foil about 50 mm square and fold the corners over once. Place this specimen between the pressing plates and place the plates in the press, held at 100°C. Close the platens without applying pressure and preheat for 1 min. Several specimens may be pressed at one time. Apply a force of about 100 kN (11 ton) for 3 min. Release the pressure, remove the specimens from the press, and allow them to cool.

8.5 The thickness of the final specimen to be measured shall not exceed 0.5 mm and may be much thinner. The ability to handle the pressed specimen and obtain a good refractive index reading are the only requirements with respect to specimen thickness.

8.6 Cut the specimen prepared in accordance with either 8.3 or 8.4 in half with sharp scissors and peel off one piece of the foil. Cut off a strip about 6 mm wide by 12 mm long with sharp scissors, such that one of the narrower ends is freshly cut. The second piece of foil may be removed, but it is frequently easier to handle the specimen with one foil piece left on the rubber.

9. Measurement of Refractive Index

9.1 Check the adjustment of the refractometer by means of the glass test piece pressed firmly against the prism with a drop of α-bromonaphthalene for contact liquid. The small light source should be collimated; the best readings are obtained with the glass test piece if the light is diffused through crumpled tissue paper. After this adjustment, clean the prism well with an alcohol and a lens paper.

9.2 The refractive index of the glass test piece and of the specimen must be measured at a known constant temperature, preferably 25°C, obtained by use of a constant-temperature room or by circulation of constant-temperature water through the prisms.

9.3 Place the specimen on the prism with the cut edge toward the light source approximately where the edge of the glass test piece was positioned. Remove the tissue paper from the light source. Press the specimen firmly on the prism by means of the finger and wait 1 min for the specimen to attain temperature equilibrium. It is also permissible to close the

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.