



Standard Test Method for Rubber—Chemical Analysis for Polystyrene Blocks In SBR (Styrene-Butadiene Rubber) and Styrene-Reinforced Latexes¹

This standard is issued under the fixed designation D 3314; 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 long polystyrene blocks in SBR rubbers and styrene-reinforced latexes.

1.2 Percent block styrene content may be determined in the range from 1 to 100 %.

1.3 This test method is intended for use on gel-free polymers, but it may be used on polymers containing gel, if it has been proven that gel does not interfere.

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.* For specific hazard statements, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:

D 1076 Specification for Rubber—Concentrated, Ammonia Preserved, Creamed, and Centrifuged Natural Latex²

D 1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis²

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens³

3. Summary of Test Method

3.1 The test method is based upon oxidative cleavage of the ethylenic bonds in the block copolymer by treating a 1,2-dichlorobenzene solution of the polymer with tertiary butyl hydroperoxide (2-methyl-2-hydroperoxypropane) with os-

mium tetroxide catalyst. The saturated portions of the polymer, which contain no ethylenic bonds, remain unattacked. The small fragments (low molecular weight aldehydes) and the low-molecular weight polystyrene fragments from scissions within the random copolymer block are soluble in methyl alcohol, whereas the detached high-molecular weight polystyrene from the styrene homopolymer block is insoluble in alcohol. It is therefore possible to separate the high-molecular weight polystyrene, which constitutes the homopolymer block, from the polymer solution.

4. Significance and Use

4.1 This test method is suitable for manufacturing control, development, and research studies.

5. Apparatus

5.1 *Erlenmeyer Flask*, or round-bottom flask for use with the heating mantle, of 250-cm³ capacity with ground-glass joint. An iodine flask is also satisfactory.

5.2 *Air Condenser*, for 5.1.

5.3 *Beaker*, 600-cm³.

5.4 *Graduated Cylinder*, 50-cm³.

5.5 *Pipet*, 1-cm³.

5.6 *Buret*, 100-cm³, for dispensing tertiary butyl hydroperoxide.

5.7 *Thermometer*, to 150°C range.

5.8 *Crucible*, Gooch-type, fritted-glass, medium-porosity, 25 to 50-cm³ size.

5.9 *Hot Plate*, capable of operating to provide 120 to 130°C to boiling solution, or heating mantle for round-bottom flasks operating in the same temperature range.

5.10 *Drying Oven*, capable of controlling at 100°C Type 1B oven in accordance with Specification E 145.

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.

Current edition approved June 10, 2002. Published July 2002. Originally published as D 3314 – 74. Last previous edition D 3314 – 92 (1997).

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

³ *Annual Book of ASTM Standards*, Vol 14.04.