



Designation: D 4026 – 87 (Reapproved 2002)^{ε1}

Standard Test Method for Rubber Latex—Styrene-Butadiene Copolymer— Determination of Residual Styrene¹

This standard is issued under the fixed designation D 4026; 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.

^{ε1} NOTE—Keywords were added editorially in June 2002.

1. Scope

1.1 This test method covers the determination of the residual styrene content of styrene butadiene (SBR) latex. This test method is based upon direct injection of a diluted latex into a gas chromatograph. The amount of residual styrene is calculated using an internal standard technique.

1.2 The range of residual styrene covered is approximately 100 to 3000 mg/kg (ppm) with a lower detection limit of approximately 50 mg/kg (ppm).

1.3 *This standard does not purport to address all of the safety problems, 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 safety statements, see Section 8.)

2. Referenced Documents

2.1 ASTM Standards:

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

E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

E 260 Practice for Packed Column Gas Chromatography⁴

E 355 Practice for Gas Chromatography Terms and Relationships⁴

3. Summary of Test Method

3.1 A sample of latex is mixed with a wetting agent and an internal standard of vinyl toluene. It is then injected into a gas chromatograph equipped with a flame ionization detector. The amount of residual styrene is determined by the internal standard technique.

4. Significance and Use

4.1 The amount of residual styrene (unreacted styrene) in an SBR latex must be studied from health, safety, economic, and environment viewpoints. This test method is useful in studying these aspects of residual styrene and also in research, development, and factory processing problems.

5. Interferences

5.1 Materials that interfere with the complete separation of styrene and vinyl toluene must be absent. Matrix effects (anything in the latex that affects the release of styrene or vinyl toluene, or both) may be minimized by the use of an addition technique.

6. Apparatus

6.1 Gas Chromatograph:

6.1.1 Any gas chromatograph whose operating parameters and columns provide well-resolved, narrow, styrene and vinyl toluene peaks, free of interference, may be used. The chromatograph must be equipped with a flame ionization detector (FID) and preferably with some means of electronic or microprocessor type of integration. Detectors must be operated in the 200 to 300°C range and injection ports must be operated in the 200 to 210°C range.

NOTE 1—It is understood that the gas chromatograph will be operated in accordance with the manufacturer's instructions for optimum performance and that the equipment will be operated by persons knowledgeable in the techniques of gas chromatography. Practice E 260 and Recommended Practice E 355, manufacturer's literature, and standard texts on gas chromatography are especially helpful.

6.1.2 Equip the chromatograph with removable glass liners for easy cleaning. *Cleaning will be required when spurious peaks begin to appear and usually after about 20 injections.* If glass liners are not available, metal liners, packed with glass wool, may be used.

6.2 *Gas Chromatographic Columns, Packings, and Programs*—Any column, packing, or program listed in Table 1 is satisfactory, as long as it produces narrow, well-resolved

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² Annual Book of ASTM Standards, Vol 09.01.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Annual Book of ASTM Standards, Vol. 03.06.



TABLE 1 Parameters for the Gas Chromatograph

NOTE 1—Packings and supports may also be obtained from most companies that supply gas chromatographic supplies and equipment.

Chromatograph	A	B	C	D	E
Column:					
Glass	X				X
Stainless steel		X	X	X	
Length, cm	300	300	300	180	150
Outside diameter, mm	6	3	3	3	6
Inside diameter, mm	2	—	—	—	2
Packed with	20 % SP 2100 ^A or OV-101/0.1 % ^B Carbowax 1500 ^C	20 % SP 2100 or OV-101/0.1 % Carbowax 1500	10 % OV 225 ^B	10 % Apiezon L ^D	5 % SP 1200/ 1.7 % Bentone 34 ^E
Support type	Supelcoport ^A	Supelcoport	(1) Chromosorb W-HP ^F (2) Chromosorb P-NAW ^F	Chromosorb W	Supelcoport
Mesh size, μm^G	150/125	150/125	(1) 150/125 (2) 180/150	180/150	125
Carrier gas	helium	helium	helium	helium	helium
Carrier gas flow, cm^3/min	30	30	15	30	—
Detector, $^{\circ}\text{C}$	250	250	250	200	300
Program:					
Initial, $^{\circ}\text{C}$	80–90	80–90		50	85
Initial time, min	5	5		2	19
Rate, $^{\circ}\text{C}$	8	8		16	50
Final, $^{\circ}\text{C}$	120	120	135	150	165
Isothermal	No	No	Yes	No	No

^ASP 2100 and Supelcoport available from Supelco. Inc., Supelco Park, Bellefonte, PA 16823.^BOV-101 and 225 are available from Pierce Chemical Co., P.O. Box 117, Rockford, IL 61105.^CCarbowax 1500 is available from Union Carbide Corp., 270 Park Ave., New York, NY 10017.^DApiezon L is available from the James G. Biddle Co., Township Line & Jolly Rds., Plymouth Meeting, PA 19462.^EBentone 34 is available from the National Lead Co., Baroid Div., Box 1675, Houston, TX 77001.^FChromosorb P-NAW and W-HP are available from Johns-Mansville, 22 E. 40 St., New York, NY 10016.^GSpecification E 11; Sieve designations: No. 80 180 μm , No. 100 150 μm , No. 120 125 μm .

styrene and vinyl toluene peaks. Other columns, packings, and programs may be used, as long as they satisfy these requirements.

6.3 *Syringes*, capable of delivering 1 mm^3 (1 μL).

NOTE 2—Syringes may be cleaned by drawing water into the syringe barrel, then ejecting it, while heating the syringe needle with a small flame.

6.4 *Pipets* capable of accurately delivering 0.1 cm^3 .

6.5 *Glass Vials*, of 20- cm^3 capacity, equipped with self-sealing septa.

6.6 *Analytical Balance*, capable of weighing to ± 0.1 mg.

6.7 *Ordinary Laboratory Glassware*, necessary for carrying out this procedure.

6.8 *Mechanical Shaker*, that will accept the vials of 6.5. (Desirable, but not mandatory.)

7. Reagents and Materials

7.1 *Deionized Water* or equivalent.

7.2 *Nonionic Wetting Agent* (isooctyl phenyl polyethoxy ethanol).⁵

7.3 *Styrene* (*p*-tert butyl catechol, inhibited)—There shall be no chromatographic peaks that interfere with vinyl toluene and it shall show no turbidity when mixed with methanol.

7.4 *Vinyl Toluene* (polymerization grade—28 % *p*-vinyl toluene and 72 % *m*-vinyl toluene) and pure *o*-vinyl toluene.

Both chemicals elute from the chromatograph as one sharp peak and have the same response to the FID. Neither of these chemicals shall show turbidity when mixed with methanol.

8. Safety Precautions

8.1 Special care should be exercised in the use of compressed gases required for the operation of the gas chromatograph. Styrene and vinyl toluene should be handled in well-ventilated areas or in fume hoods, to minimize health and safety hazards.

9. Sampling and Selection of Test Portions

9.1 Since the use of this test method may be required for any purpose listed under Section 4, the analyst may choose the sample and test portion at his discretion. A necessary requirement is that the test portion be as representative of the sample as possible.

10. Calibration

10.1 For a series of analyses, sufficient stock and calibration solutions are made prior to test portion preparation. The concentrations of these solutions are tabulated as follows, but may be different if the analyst so chooses. In any case, the exact concentrations of styrene and vinyl toluene must be known.

10.2 *Stock Solutions*:

10.2.1 *Stock Solution A*—Dissolve 20 g of nonionic wetting agent (7.2) in 180 g of water (7.1).

⁵ Triton X100 has been found satisfactory. Available from Rohm and Haas, Philadelphia, PA 19105.