



Designation: D 5135 – 95

## Standard Test Method for Analysis of Styrene by Capillary Gas Chromatography<sup>1</sup>

This standard is issued under the fixed designation D 5135; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of the impurities in, and the purity of styrene by gas chromatography. It is applicable to styrene in the range from 99 to 100 % purity and to impurities at concentrations of 0.001 to 1.00 %. This test method may be used for lower purity but not all the impurities may be readily identified and the use of an internal standard becomes more difficult.

1.2 The following applies to all specified limits in this standard: for purposes of determining conformance with this standard, an observed value or a calculated value shall be rounded off “to the nearest unit” in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E 29.

1.3 *This standard does not purport to address all 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 a specific hazard statement, see Section 7.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 3437 Practice for Sampling and Handling Liquid Cyclic Products<sup>2</sup>

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>3</sup>

E 1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs<sup>3</sup>

#### 2.2 Other Document

OSHA Regulations—29CFR paragraphs 1910.1000 and 1910.1200<sup>4</sup>

### 3. Summary of Test Method

3.1 In this test method, the chromatogram peak area for each

impurity is compared to the peak area of the internal standard (*n*-heptane or other suitable known) added to the sample. From the response factors of these impurities relative to that of the internal standard and the amount of internal standard added, the concentration of the impurities are calculated. The styrene content is obtained by subtracting the total amount of all impurities from 100.00.

### 4. Significance and Use

4.1 This test method is designed to obtain styrene purity on the basis of impurities normally present in styrene and may be used for final product inspections and process control.

4.2 This test method will detect the following impurities: non-aromatic hydrocarbons containing ten carbons or less, ethylbenzene, *p*- and *m*-xylene, cumene, *o*-xylene, *n*-propylbenzene, *m*- and *p*-ethyltoluene, alpha-methyl-styrene, *m*- and *p*-vinyltoluene and others where specific impurity standards are available. Absolute purity cannot be determined if unknown impurities are present.

### 5. Apparatus

5.1 *Gas Chromatograph*—Any gas chromatograph having a flame ionization detector and a splitter injector suitable for use with a fused silica capillary column may be used, provided the system has sufficient sensitivity to obtain a minimum peak height response of 0.1 mV for 0.010 % internal standard when operated at the stated conditions. Background noise at these conditions is not to exceed 3  $\mu$ V.

5.2 *Column*—Capillary columns have been found to be satisfactory. For example, 60 m of 0.32-mm inside diameter polar-fused silica capillary internally coated to a 0.5- $\mu$ m thickness with a bonded (cross-linked) polyethylene glycol can be used (see Table 1 for parameters). Other columns may be used after it has been established that such a column is capable of separating all major impurities and the internal standard from the styrene under operating conditions appropriate for the column (see Practice E 1510).

5.3 *Electronic Integration*, with tangent capabilities is recommended.

5.4 *100-mL Volumetric Flask*.

5.5 *Microsyringes*, assorted volumes.

### 6. Reagents and Materials

6.1 *Carrier Gas*—a carrier gas (minimum purity of 99.95

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.07 on Styrene, Ethylbenzene, and C<sub>9</sub> and C<sub>10</sub> Aromatic Hydrocarbons.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.04.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>4</sup> Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.