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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Purity of Vinyl Chloride Monomer by Gas Chromatography¹

This standard is issued under the fixed designation D 3834; 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 The general availability of gas chromatography in practically every laboratory and the inherent sensitivity in the detection of trace constituents indicate that the technique would qualify as a standard test method. With this one-step technique, impurities can be determined in a range that includes major constituents and parts per million impurities.

1.2 Inherent in this test method is the limitation that the impurities must be volatile. Resolution of some of the individual impurities may be limited by the column selected. The use of several different types of columns may be necessary if the ultimate in resolution is required. A single column will normally give the required degree of separation.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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.* Specific hazards statements are given in Section 8.

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 ASTM Standards:

E 260 Practice for Packed Column Gas Chromatography²

E 355 Practice for Gas Chromatography Terms and Relationships²

3. Terminology

3.1 *Definitions*—All terms and practices used in this test method are in accordance with Practices E 260 and E 355.

4. Summary of Test Method

4.1 Conventional gas chromatographic techniques are used. A small sample is injected into a gas chromatographic column packed with a liquid-coated solid support or porous polymer beads. Passing over the support in a stream of helium, the sample is separated into its components, which are detected by a standard sensing device. The signal is recorded to indicate the

relative concentration of a component and the retention time.

5. Significance and Use

5.1 Vinyl chloride monomer (**Warning:** see 8.1) must be very pure to produce high-quality polymers with desirable appearance, stability, and generally desirable physical properties. Even trace concentrations of certain impurities, for example, conjugated dienes, interfere with the mechanism of polymerization. This test method is applied accordingly to process and quality control.

5.2 It is expected that this test method will be suitable for specifications, manufacturing control, and research and development. However, other specifications may be required to define a product completely.

6. Interferences

6.1 Retention times are dependent upon a large number of variables associated with the composition, length, and diameter of the column, the size of the sample, the temperature of the column, the carrier gas flow rate, and the temperature of the injection port. It is therefore quite possible to have two components or impurities with identical retention times. Only by changing columns or column conditions can these concurrent peaks be resolved.

6.2 Temperature programming, as recommended in this test method, must be precisely controlled to obtain reproducible retention times and reliable identification of constituents.

6.3 A given species has only one peak associated with it for identification; therefore, identification from that one peak by its retention time alone is risky unless coupled with other information. Such information might be obtained with a mass spectrometer or chemical tests for functional groups.

7. Apparatus

7.1 Many commercial instruments are suitable for this test; however, there are two basic requirements. The detectors must be equivalent in sensitivity to hydrogen flame ionization detectors and the column oven must be designed for temperature programming in the range from 40 to 150°C.

7.2 The basic equipment includes: injection ports, columns, column ovens, detectors, a recorder, a suitable attenuating device for keeping the peaks on scale, a temperature programming unit, and a syringe or liquid-sampling valve for injection of microlitres of sample onto the columns.

7.3 Dual columns are recommended for stable baselines.

7.4 An electronic or mechanical integrator is recommended

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

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² *Annual Book of ASTM Standards*, Vol 14.01.

for measurement of peak areas.

7.5 Recommended Columns—To provide greater versatility, two different types are recommended: one consists of a liquid phase on a solid support and the other is of commercially available porous polymer beads. Greater resolution of C-4 hydrocarbons is obtained with the former column, and better separation of some light components is observed with the latter column. Other columns with equivalent performance may be used.

7.5.1 Liquid Phase Column—A mixture consisting of 75 % Igepal CO-880³ and 25 % Antarox BL344 or LF330⁴ is applied to 60 to 80-mesh firebrick to produce a liquid content of 25 weight % and packed in 3.175-mm stainless steel tubing, 6 m long. Table 1 lists average retention times and relative retention times for some components possibly present in vinyl chloride monomer analyzed on the liquid phase column.

7.5.2 Porous Polymer Bead Column—A 2-m length of 4.76-mm stainless steel tubing is packed with 80- to 100-mesh

Porapak S⁵ or other equivalent commercial packing. Table 2 lists average retention times and relative retention time for some components possibly present in vinyl chloride monomer analyzed using a porous polymer column.

7.6 Other Operating Conditions:

7.6.1 Temperatures:

Liquid phase column:	
Injection port	125°C
Detector	260°C
Column	40°C until elution of vinyl chloride, then program at 6.0°C/min to 150°C

Porapak S column:	
Injection port	150°C
Detector	280°C
Column	50°C for 2 min, then programmed at 6.0°C/min to 150°C

7.6.2 Detector Type—Hydrogen flame ionization or other detector of equivalent sensitivity.

7.6.3 Sample Size—5 µL liquid or 0.5 mL gas.

7.6.4 Carrier Gas—Helium, 60 mL/min.

8. Hazards

8.1 Warning: Recent public information indicates that vinyl chloride monomer may be toxic to humans, both by skin contact and inhalation.

8.2 Precaution—Laboratory personnel are cautioned to use all reasonable care to prevent excessive contact with either liquid or gaseous forms of this material.

⁵ A trademark for porous polymer beads which may be obtained from GC Column Packing, Waters Associates Inc., Maple St., Milford, MA.

³ A trademark for a nonylphenoxy poly(ethyleneoxy)ethanol, which may be obtained from GAF Corp., Dyestuff and Chemical Div., 140 W. 51st St., New York, NY 10020.

⁴ A trademark for a series of surfactants which may be obtained from GAF Corp., Dyestuff and Chemical Div., 140 W. 51st St., New York, NY 10020.

TABLE 1 Average Retention Time and Relative Retention Time for Some Components Possibly Present in Vinyl Chloride Monomer Analyzed on the Liquid Phase Column

Components	RT, seconds	RRT
Ethylene/ethane	187	0.222
Propane	225	0.267
Propylene	257	0.305
Isobutane	281	0.334
Acetylene	347	0.412
Butane	365	0.434
Butene-1	417	0.495
Allene	456	0.542
<i>trans</i> -Butene	481	0.571
<i>cis</i> -Butene-2	549	0.652
1,3-Butadiene	648	0.770
Methyl acetylene	688	0.817
Methyl chloride	786	0.934
Vinyl chloride	842	1.00
Ethyl chloride	1166	1.385
Acetaldehyde	1269	1.559
Benzene	1899	2.183
EDC	2192	2.693

TABLE 2 Average Retention Time and Relative Retention Time for Some Components Possibly Present in Vinyl Chloride Monomer Analyzed Using a Porous Polymer Column

Components	RT, seconds	RRT
Methene	39	0.051
Ethylene	114	0.149
Acetylene		
Ethane	150	0.196
Propylene	490	0.641
Propane	527	0.690
Allene/methyl chloride	577	0.755
Vinyl chloride	764	1.00
1,3-Butadiene	923	1.208
<i>trans</i> -Butene-2	946	1.238
Ethyl chloride/ <i>cis</i> -butene-2	967	1.266
EDC	1830	2.395