



Standard Test Method for Determination of Trace Organic Impurities in Monomer Grade Vinyl Chloride by Capillary Column/Multidimensional Gas Chromatography¹

This standard is issued under the fixed designation D 5507; 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 is a general-purpose capillary-based test method for the determination of trace level impurities in high-purity vinyl chloride. This test method uses serially coupled capillary PLOT columns in conjunction with the multidimensional techniques of column switching and cryogenic trapping to permit the complete separation of the 11 key vinyl chloride impurities in a single 25-min run.

NOTE 1—There are no similar or equivalent ISO standards covering the primary subject of this test method.

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. Specific hazards statements are given in Section 8.*

2. Referenced Documents

2.1 ASTM Standards:

D 883 Terminology Relating to Plastics²

D 1600 Terminology for Abbreviated Terms Relating to Plastics²

F 307 Practice for Sampling Pressurized Gas for Gas Analysis³

3. Terminology

3.1 *Definitions*—Terminology is in accordance with Terminologies D 883 and D 1600 unless otherwise indicated.

4. Summary of Test Method

4.1 The liquid vinyl chloride sample or calibration standard is injected either directly using a high-pressure liquid sampling valve or alternately as an expanded gas. An appropriate volume

of the liquid or gas sample is injected to enable the required detection limits to be achieved. A preliminary GC separation is achieved on a 6-m pre-column, the purpose of which is to remove the bulk of the vinyl chloride peak from the trace peaks of interest. Two heart-cut transfers are made from this pre-column separation, which sends selected portions to a second column for additional separation. These two cuts incorporate 10 of the 11 trace impurities of interest, but they exclude 1,2 ethylene dichloride and the bulk of the vinyl chloride peak. The 1,2 EDC peak is eluted from the 6-m pre-column and detected at the first FID after the two cuts are made.

4.2 The components eluting to the two FID detectors are identified and quantitated by comparing their retention times and area counts to those obtained previously from a calibration standard run under identical conditions.

5. Significance and Use

5.1 The multidimensional approach permits all of the trace impurities to be well separated from the main vinyl chloride peak, thereby improving quantitative accuracy over established packed column methods.

5.2 The minimum detection limit (MDL) for all components of interest has been shown to be well below 500 ppb for this test method.

6. Apparatus

6.1 Instrumentation:

6.1.1 *HP 5890A*⁴ (or equivalent), equipped as follows:

6.1.1.1 *Split/Splitless Injector System*—Must be demonstrated to be free of discrimination effects induced by vapor viscosity differences if helium- or nitrogen-based gas standards are to be used for instrument calibration.

6.1.1.2 *Dual Flame-Ionization Detectors*.

6.1.1.3 *Column Switching Device* A pneumatics control system, available from Scientific Glass Engineering, Inc.,⁵ or equivalent.

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 08.01.

³ *Annual Book of ASTM Standards*, Vol 15.03.

⁴ Available from Hewlett-Packard Co., 3495 Deer Creek Road, Palo Alto, California 94304.

⁵ Scientific Glass Engineering 2007 Kramer Lane, Austin, Texas 78758.