



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 D5507; 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 is no known ISO equivalent to this standard.

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:*²

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

F307 Practice for Sampling Pressurized Gas for Gas Analysis

3. Terminology

3.1 *Definitions*—Terminology is in accordance with Terminologies D883 and D1600 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*^{3,4}(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.*

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

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The sole source of supply of the apparatus known to the committee at this time is Hewlett-Packard Co., 3495 Deer Creek Road, Palo Alto, California 94304.

⁴ If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

*A Summary of Changes section appears at the end of this standard

LPG VAPORIZING INJECTOR

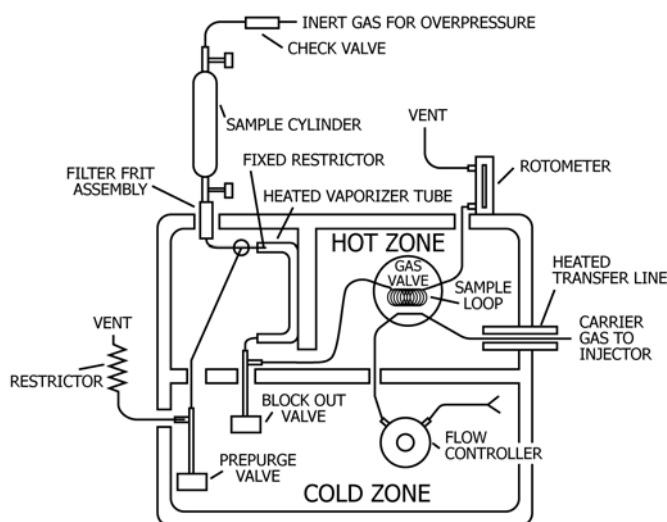


FIG. 1 Procedure B: On-Line Vaporization Using the LPG Vaporizing Injector

6.1.1.3 *Column Switching Device* A pneumatics control system, available from Scientific Glass Engineering, Inc.,^{4,5} or equivalent.

6.1.1.4 *Sub-Ambient Oven Temperature Control* (optional).

6.1.1.5 *LPG Vaporizing Injector*; available from Microanalytics Instrumentation,^{4,6} or equivalent (Fig. 1).

6.2 *Data System*—Dual HP 3396A Integrators^{3,4} (or equivalent) permit the acquisition, storage, and reduction of the output signals from the two FIDs simultaneously. After the initial method development, however, it is possible to consolidate the output to a single integrator using the instruments signal switching capability.

6.3 *Columns*:

6.3.1 *Pre-Column*—100 cm of 0.20-mm inside diameter fused silica fixed restrictor coupled to the front of a 6 m by 0.53-mm inside diameter GSQTM available from J & W Scientific^{4,7} (or equivalent).

6.3.2 *Analytical Column*—9 m by 0.53-mm inside diameter GSQTM available from J & W Scientific^{4,7} (or equivalent) plus 25 m by 0.53 mm inside diameter PORAPLOT UTM Chrompack^{4,8} (or equivalent).

6.4 *Syringes*—A range of high-quality gas-tight syringes representing volumes from 0.5 to 25 mL should be available. These syringes should be equipped with PTFE-tipped plunger seals and on and off syringe valves to prevent the loss of gas sample.

⁵ The sole source of supply of the apparatus known to the committee at this time is Scientific Glass Engineering 2007 Kramer Lane, Austin, Texas 78758.

⁶ The sole source of supply of the apparatus known to the committee at this time is Microanalytics Instrumentation, 2713 Sam Bass Rd., Round Rock, TX 78681.

⁷ The sole source of supply of the apparatus known to the committee at this time is J & W Scientific, 91 Blue Ravine Road, Folsom, California 95630-4714.

⁸ The sole source of supply of the apparatus known to the committee at this time is Chrompack Inc., 1130 Route 202, Raritan, NJ 08869.

7. Reagents and Materials

7.1 *Helium*—Carrier gas, zero grade, high quality. Traps should be placed in the supply lines leading to the gas chromatograph. These traps should reduce oxygen, moisture, and hydrocarbons to the lowest possible levels.

7.2 *Hydrogen*—Flame gas, high-purity (hydrocarbon free).

7.3 *Air*—Flame gas, high-purity (hydrocarbon free).

7.4 *Liquid CO₂*—Coolant, bone-dry grade, liquid-delivery, 1200-psi helium pad recommended.

7.5 *Standards*:

7.5.1 *Primary Standard*—The primary standard is a certified reference standard, which is blended into a stable nitrogen or helium matrix. The component concentrations should be prepared and reported on an as-in-vinyl chloride basis. The concentrations of the various components in this standard should also represent typical values expected for the particular process or sample. The following is a typical calibration standard composition:

Component	Mole, ppm	Weight, ppm
Ethylene	29.4	13.2
Propylene	20.0	13.5
Acetylene	6.8	2.8
Butane-1	6.8	6.1
Trans-butene-2	7.1	6.4
Cis-butene-2	7.5	6.7
1,3 butadiene	6.5	5.6
Methyl chloride	36.8	29.7
Vinyl acetylene	12.2	10.2
Ethyl chloride	15.9	16.4
1,2 ethylene dichloride	11.8	18.7
Nitrogen	balance	

7.5.2 *Secondary Standard*—The secondary standard is a vinyl chloride-based blend, which is used for method setup and day-to-day method calibration. This standard is prepared from actual vinyl chloride product, which is spiked where appropriate to yield the approximate levels represented in the nitrogen-based primary standard. The final concentrations should be determined by averaging the results from multiple runs, which are referenced to the primary standard. This calibration/recalibration process may be conducted using an alternate GC procedure.

8. Hazards

8.1 Appropriate caution must be exercised in handling the sample due to the suspected carcinogenicity of vinyl chloride. Any excess of sample beyond that actually injected into the column should be routed to a purge waste line to be passed to a vent hood or other suitable disposal location. This excess sample includes the inlet splitter vent flow and the sample-loop purge flow in the case in which a gas-valve injection is being made.

9. Sampling

9.1 This section is to be followed for all samples, including unknown samples and the synthetic standards.

9.2 Samples should be supplied to the laboratory in high-pressure sample cylinders, obtained using the procedure described in Practice F307 or similar standards.