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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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# Standard Test Methods for Determination of 1,1,1-Trichloroethane and Methylene Chloride Content in Stabilized Trichloroethylene and Tetrachloroethylene[1]

This standard is issued under the fixed designation D 5320; 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 These test methods provide a means for determining low levels of 1,1,1-trichloroethane and methylene chloride in virgin or reclaimed trichloroethylene and tetrachloroethylene.

1.2 Two test methods are covered, as follows:

1.2.1 *Test Method A*—Capillary column gas chromatographic determination of 1,1,1-trichloroethane and methylene chloride in trichloroethylene and tetrachloroethylene.

1.2.2 *Test Method B*—Packed column gas chromatographic determination of 1,1,1-trichloroethane and methylene chloride in trichloroethylene and tetrachloroethylene.

1.3 *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.*

## 2. Summary of Test Methods

2.1 Samples of trichloroethylene or tetrachloroethylene are introduced into a gas chromatography (GC) equipped with either a thermal conductivity detector (TCD) or a flame ionization detector (FID). Calibration mixtures containing known concentrations of methylene chloride, 1,1,1-trichloroethane, or other impurities of interest are introduced into the chromatograph in the same manner. The amount of these impurities in the samples is then calculated by comparing the peak areas of these compounds from sample chromatograms with the corresponding peak areas from calibration standard chromatograms. Instrumental parameters are provided to allow the use of either capillary or packed gas chromatography columns.

## 3. Significance and Use

3.1 These test methods can be used to determine the impurity levels of 1,1,1-trichloroethane and methylene chloride in both trichloroethylene and tetrachloroethylene.

3.2 These test methods can be used to determine the impurity levels of other components, such as stabilizers, found in trichloroethylene and tetrachloroethylene. The other components shall be able to be resolved by recommended GC columns and the gas chromatograph shall be calibrated with known concentration of these compounds.

## 4. Interferences

4.1 Compounds that co-elute with 1,1,1-trichloroethane, methylene chloride, or other components of interest can interfere with the accuracy of these test methods.

## 5. Apparatus

5.1 *Gas Chromatograph*, temperature programmable and having the following minimum capabilities:

5.1.1 *Chart Recorder or Electronic Integrator*—An electronic integrator is preferable.

5.1.2 *Flame Ionization Detector and Capillary Split/Splitless Injection Port* (for Test Method A).

5.1.3 *Thermal Conductivity Detector and Packed Column Injection Port* (for Test Method B).

NOTE 1—This procedure was tested using the combinations of capillary column with flame ionization detector and packed GC column with thermal conductivity detector. It should be possible, however, to perform this analysis with either column and detector combination.

5.2 *Gas Chromatography Columns*:

5.2.1 *Column for Test Method A*—30-m by 0.53-mm inner diameter fused silica capillary, internally coated to a 3-µm thickness with a cross-linked polymethyl silicone.[2]

5.2.2 *Column for Test Method B*—10-ft by ⅛-in. outer diameter stainless steel packed with 3 % SP1500 on 80/100 mesh Carbopack B, or equivalent.

5.3 *Syringe*, 10 µL.

5.4 *Microsyringes*, assorted sizes for preparing calibration standards.

5.5 *Volumetric Flasks*, 100 mL.

5.6 *Flow Meter*, soap bubble or electronic type.

5.7 *Analytical Balance*, capable of weighing to four decimal places.

[1] These test methods are under the jurisdiction of ASTM Committee D-26 on Halogenated Organic Solvents and Fire Extinguishing Agents and are the direct responsibility of Subcommittee D26.04 on Test Methods.

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[2] J&W Scientific DB-1 fused silica capillary column (Cat# 125-1035) available from J&W Scientific, 91 Blue Ravine Rd., Folsom, CA 95630-4917, or Supelco (SPB-1 Cat# 2-5341M) available from Supelco, Inc., Supelco Park, Bellefonte, PA 16823-0048, have been found satisfactory for this purpose. Any equivalent may be used.