



Designation: D4457 – 02 (Reapproved 2008)

Standard Test Method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph¹

This standard is issued under the fixed designation D4457; 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 test method covers the determination of total amount of dichloromethane or 1,1,1-trichloroethane, or both, in paints and coatings. It has been evaluated for cellulose nitrate, alkyd, vinyl, and styrene-butadiene systems. It has not yet been evaluated for other formulations, but is believed to be applicable. The established working range of this test method is from 31 to 65 % for 1,1,1-trichloroethane and 32 to 78 % for dichloromethane. There is no reason to believe it will not work outside of these ranges. The presence of 1-propanol in paints and coatings requires the use of a different internal standard. (See also Practice E260.)

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

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.* Specific hazard statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*²

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)³

E260 Practice for Packed Column Gas Chromatography

¹ This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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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 last approved version of this historical standard is referenced on www.astm.org.

3. Summary of Test Method

3.1 Anhydrous 1-propanol (see 10.5) is added as an internal standard to suitable aliquot of the whole paint. The aliquot is then diluted with dimethylformamide and injected onto a gas chromatographic column containing a porous polymer packing that separates dichloromethane and 1,1,1-trichloroethane from other volatile compounds.

4. Significance and Use

4.1 Use of 1,1,1-trichloroethane and dichloromethane, which do not measurably contribute to the atmospheric oxidant level, is a way for industry to meet government or other regulations on volatile organic compounds. This test method is designed to determine the content of these halohydrocarbon solvents in paints and coatings. That content can subsequently be used in calculating the volatile organic compound content of a coating.

5. Apparatus

5.1 *Chromatograph*, any gas-liquid chromatographic instrument equipped with a thermal conductivity detector and capable of being temperature programmed (see Table 1). Optionally, a flame ionization detector may be used if the sample is diluted so that no more than 1000 ppm each of dichloromethane and 1,1,1-trichloroethane is present in the injected specimen.

5.2 *Recorder*, a recording potentiometer with a full-scale deflection of 10 mV, a full-scale response time of 2 s or less, and a maximum noise of ± 0.03 % of full scale.

5.3 *Pre-Column*, 40 in. (100 mm) long by $\frac{1}{8}$ in. (3.2 mm) outside diameter stainless steel, packed with glass wool, fitted on the entrance end of the column to retain any nonvolatile materials and minimize sludge buildup in the column.

5.4 *Column*, 4 ft (1.22 m) long by $\frac{1}{8}$ in. (3.2 mm) outside diameter stainless steel, packed with 80/100 mesh (150 to 180 μm) porous polymer packing material,⁴ or other suitable

⁴ Porapak R, a registered trademark of Waters Associates, Inc., Milford, MA, has been found satisfactory for this purpose.

material.

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