



Standard Test Method for 1,1,1-Trichloroethane Content¹

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ϵ^1 NOTE—Section 10, Keywords, was added in June 1994.

1. Scope

1.1 This test method provides a basis for the determination of the 1,1,1-trichloroethane (weight %) in the presence of stabilizers and impurities, in virgin or reclaimed 1,1,1-trichloroethane products.

2. Referenced Documents

2.1 ASTM Standards:

E 260 Practice for Packed Column Gas Chromatography²

3. Summary of Test Method

3.1 Gas chromatographic scans of 1,1,1-trichloroethane of known purity and the sample under test are prepared. The 1,1,1-trichloroethane concentration of the unknown is calculated by comparison of the respective elution peak areas.

4. Significance and Use

4.1 This test method provides an analysis in weight percent of the 1,1,1-trichloroethane concentration of virgin, or reclaimed 1,1,1-trichloroethane. Compounds that co-elute with 1,1,1-trichloroethane may interfere with this test.

5. Apparatus

5.1 *Gas Chromatograph* having the following minimum capabilities:

5.1.1 Temperature programmable column oven with control systems capable of maintaining the chromatographic column at the prescribed temperature $\pm 0.5^\circ\text{C}$.

5.1.2 Heated sample injection port.

5.1.3 Differential katharometer (hot wire), thermistor-type thermal conductivity detector or hydrogen flame detector.

5.1.4 Heated detector section.

5.2 *Syringe*, 10 μL .

5.3 *Chromatographic Column*, of suitable length and packing similar to:

5.3.1 *Column*—Annealed copper or stainless steel tubing, 20 ft by $\frac{1}{8}$ in. (2.4 m by 3.2 mm) in outside diameter.

5.3.2 *Packing*—Chromosorb W HP,³ 80/100 mesh coated with 20 weight % OV101⁴ (or equivalent, polydimethyl siloxane) as the liquid phase.

5.3.3 Or other columns or packings capable of providing separation of 1,1,1-trichloroethane from stabilizers and process impurities.

5.4 *Carrier Gas*—Dry helium of 99.95 mol % purity.

5.5 *Standard*—Uninhibited 1,1,1-trichloroethane of known high purity of at least 98.0 %.

5.6 *Flow Meter*—Capable of measuring in the prescribed gas flow ranges.

6. Suggested Analysis Conditions

6.1 The following analysis conditions are recommended:

Initial Column temperature	85°C
Initial hold time	4 min
Rate	8°C/min
Final temperature	160°C
Final hold time	16 min
Detector temperature	250°C
Injector temperature	250°C
Carrier gas and flow	20 He/20 cm ³ /min
Sample size	2 μL

NOTE 1—With a hydrogen flame detector, sample size should be reduced to around 0.2 μL to prevent overloading the detector.

6.1.1 Employing the conditions recommended, all constituents of 1,1,1-trichloroethane solvents should elute from the column within approximately 30 min.

7. Analytical Procedure

7.1 Chromatograph 2- μL samples of both the standard 1,1,1-trichloroethane and unknown material under the conditions described in 6.1 or equivalent. Both analyses must be conducted under identical operating conditions. Determine peak areas of the eluted 1,1,1-trichloroethane in each sample. Calculate weight percentage of 1,1,1-trichloroethane in the sample as shown in 8.1.

¹ This test method is under the jurisdiction of ASTM Committee D-26 on Halogenated Organic Solvents and Fire Extinguishing Agents and is the direct responsibility of Subcommittee D26.04 on Test Methods.

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

³ Johns-Manville product available from any chromatographic supply company.

⁴ Available from any chromatographic supply company.

8. Calculation

8.1 Calculate the percentage of 1,1,1-trichloroethane in the sample as follows:

$$1,1,1\text{-TCE \%} = \frac{\text{peak area sample} \times \% \text{ assay standard}}{\text{peak area standard sample}} \quad (1)$$

9. Precision and Bias

9.1 The precision and bias of this test method have not been

determined. Good sample volume reproducibility is critical to overall precision of the method.

10. Keywords

10.1 assay; chlorinated; gas chromatography; halogenated; 1,1,1-trichloroethane; purity; solvent

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