Designation: D 3447 – 01

Standard Test Method for Purity of Halogenated Organic Solvents[1]

This standard is issued under the fixed designation D 3447; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of common impurities found in halogenated organic solvents introduced during the manufacturing of the solvent. Although the absolute sensitivity may vary, sensitivity in the parts per million range may be achieved under the chromatographic conditions specified in 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.*

2. Summary of Test Method

2.1 This test method is based on a temperature-programmed gas chromatographic separation achieved on a packed column containing 30 % by weight silicone oil on 80/100 mesh diatomaceous earth.[2]

3. Significance and Use

3.1 This test method can be used to check the purity of halogenated organic solvents by determining the impurities present under the chromatographic conditions specified.

3.2 This test can be used for establishing manufacturing and purchasing specifications.

4. Apparatus

4.1 *Gas Chromatograph*, equipped with a 1-mV recorder or electronic integrator and a thermal conductivity or flame ionization detector.

4.2 *Column*, 6-m by 3.2-mm outside diameter stainless steel tubing packed with 30 % by weight silicone fluid[2] on 80/100 mesh diatomaceous earth or similar capillary (Example: 0.32 mm × 30 m DB5).[3]

4.3 *Syringe*, 10-µL gas chromatographic.

4.4 *Flow Meter*, soap-bubble type or equivalent.

4.5 *Helium*, research grade carrier gas.

5. Procedure

5.1 Install the column and adjust the helium carrier gas flow to 40 mL/min.

5.2 Set up the gas chromatograph according to the parameters listed as follows:

Injection port temperature	: 200°C
Detector temperature	: 220°C
Oven initial temperature	: 50°C
Oven final temperature	: 170°C
Temperature program rate	: 10°C/min

5.3 Inject 1.0 µL of sample into the gas chromatograph and start the temperature program. Simultaneously start the recording device to begin recording peak areas.

5.4 If an integrator is not used, record the chromatogram using the minimum detector attenuation setting necessary to keep each peak on-scale at all times.

5.5 Terminate the chromatogram after all peaks have eluted from the column.

6. Calculation

6.1 Obtain from the integrator the areas of all of the peaks eluted in the chromatogram and calculate the solvent purity as follows:

$$\text{purity, \%} = 100 - [(A \times 100)/(A + B)] \quad (1)$$

where:

A = summation of the peak areas of the impurities, and
B = peak area of trichlorotrifluoroethane.

6.2 If an integrator was not used, measure the area of each peak from the recorder strip chart as follows:

$$\text{peak area} = P_n \times P_{w1/2} \times \text{Atten} \quad (2)$$

where:

P_n = peak height,
$P_{w1/2}$ = peak width measured at ½ of the peak height,
Atten = detector attenuation setting.

Proceed to calculate the solvent purity as in 6.1.

6.3 The purity in area percent is a reasonable approximation of purity in weight percent when the concentration of impurities is low and when the molecular weights and thermal conductivities of the impurities are similar to the halogenated organic solvent being tested.

[1] This test method is under the jurisdiction of ASTM Committee D26 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] Thirty percent DC-200 on 80/100 mesh Chromosorb W has been found to be a satisfactory packing. Columns containing this packing may be obtained from most chromatography supply houses.

[3] Chromosorb W has been found satisfactory and can be obtained from most chromatography supply houses.

7. Precision and Bias

7.1 The 95 % confidence limits for analyses at the 220 and 1000-µg/g impurity level are ±9 and ±28 µg/g, respectively. This is based on the analyses of two impurities in two samples by four analysts on two different days.

NOTE 1—The precision and bias were determined using trichlorotrifluoroethane.

8. Keywords

8.1 gas chromatography; halogenated solvent; purity trichlorotrifluoroethane