



Designation: D 4367 – 99

Standard Test Method for Benzene in Hydrocarbon Solvents by Gas Chromatography¹

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1. Scope *

1.1 This test method covers the determination by gas chromatography of benzene at levels from 0.01 to 1 volume % in hydrocarbon solvents.

NOTE 1—For benzene levels lower than 0.01 volume %, use Test Method D 6229.

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.*

1.3 For hazard information and guidance, see the supplier's Material Safety Data Sheet. For specific hazard statements, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:

D 3606 Test Method for the Determination of Benzene and Toluene in Finished Motor and Aviation Gasoline by Gas Chromatography²

D 6229 Test Method for Trace Benzene in Hydrocarbon Solvents by Capillary Gas Chromatography³

E 260 Practice for Packed Column Gas Chromatography⁴

E 300 Practice for Sampling Industrial Chemicals⁵

3. Summary of Test Method

3.1 An internal standard, methyl ethyl ketone (MEK), is added to the material and then introduced into a gas chromatograph equipped with two columns connected in series. The specimen passes first through a column packed with the nonpolar phase, methyl silicone, which separates the components by boiling point. After octane has eluted, the flow through the nonpolar column is reversed, flushing out the components heavier than octane. The octane and lighter

components then pass through a column with the highly polar phase, 1,2,3-tris(2-cyanoethoxy)propane, that separates the aromatic and nonaromatic compounds. The eluted components are detected by a conventional detector and recorded on a strip chart. The peak areas are measured and the concentration of each component is calculated by reference to the internal standard.

4. Significance and Use

4.1 Benzene is classed as a toxic and carcinogenic material. A knowledge of the concentration of this compound may be an aid in evaluating the possible health hazards to persons handling and using hydrocarbon solvents, but this test method is not intended to evaluate such hazards.

5. Apparatus

5.1 *Chromatograph*—Any gas chromatographic instrument that has a backflush system and flame ionization detector and that can be operated at the conditions given in Table 1. The detector-recorder combination must produce a 4-mm deflection for a 1-μL specimen containing 0.05 volume % MEK when operated at maximum sensitivity.

5.2 *Columns*, one 0.8-m (2.5-ft) length of 3.2-mm (1/8-in.) outside diameter stainless steel tubing and one 4.6-m (15-ft) length of 3.2-mm (1/8-in.) outside diameter stainless steel tubing.

5.3 *Recorder, Strip Chart*—Potentiometer with a full-scale deflection of 1 mV, a full-scale response time of 2 s or less, and a maximum noise level of ±0.3 % of full scale.

5.4 *Microsyringe*, 5-μL capacity.

5.5 *Pipets*, measuring 1 and 2 mL, graduated in 0.01 mL; 5, 10, and 20-mL capacity.

5.6 *Flasks*, volumetric, 25 and 100-mL capacity.

5.7 *Vibrator*, electric.

5.8 *Vacuum Source*.

5.9 *Evaporator*, vacuum, rotary.

5.10 *Flask*, boiling, round-bottom, short-neck, with 24/40 T joint, 500-mL capacity. Suitable for use with the evaporator (see 5.9).

5.11 *Lamp*, infrared.

5.12 *Burets*, automatic, with integral reservoir, 25-mL capacity.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

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² Annual Book of ASTM Standards, Vol 05.02.

³ Annual Book of ASTM Standards, Vol 06.04.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Annual Book of ASTM Standards, Vol 15.05.

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