



Designation: D5580 – 21

Standard Test Method for Determination of Benzene, Toluene, Ethylbenzene, *p/m*-Xylene, *o*-Xylene, C₉ and Heavier Aromatics, and Total Aromatics in Finished Gasoline by Gas Chromatography¹

This standard is issued under the fixed designation D5580; 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 benzene, toluene, ethylbenzene, the xylenes, C₉ and heavier aromatics, and total aromatics in finished motor gasoline by gas chromatography.

1.2 The aromatic hydrocarbons are separated without interferences from other hydrocarbons in finished gasoline. Non-aromatic hydrocarbons having a boiling point greater than *n*-dodecane may cause interferences with the determination of the C₉ and heavier aromatics. For the C₈ aromatics, *p*-xylene and *m*-xylene co-elute while ethylbenzene and *o*-xylene are separated. The C₉ and heavier aromatics are determined as a single group.

1.3 This test method covers the following concentration ranges, in liquid volume %, for the preceding aromatics: benzene, 0.1 % to 5 %; toluene, 1 % to 15 %; individual C₈ aromatics, 0.5 % to 10 %; total C₉ and heavier aromatics, 5 % to 30 %, and total aromatics, 10 % to 80 %.

1.4 Results are reported to the nearest 0.01 % by either mass or by liquid volume.

1.5 This test method includes a relative bias section for U.S. EPA spark-ignition engine fuel regulations reporting for benzene based on Practice D6708 accuracy assessment between Test Method D5580 and Test Method D3606 as a possible Test Method D5580 alternative to Test Method D3606. The Practice D6708 derived correlation equation is only applicable for fuels in the benzene concentration range from 0.0 % to 2.31 % by volume as measured by Test Method D5580. The applicable Test Method D3606 range for benzene is from 0.0 % to 2.38 % by volume as reported by Test Method D3606.

1.6 This test method includes a relative bias section for U.S. EPA spark-ignition engine fuel regulations for total aromatics reporting based on Practice D6708 accuracy assessment be-

tween Test Method D5580 and Test Method D5769 as a possible Test Method D5580 alternative to Test Method D5769. The Practice D6708 derived correlation equation(s) is only applicable for fuels in the total aromatic concentration range from 5.4 % to 31.6 % by volume as measured by Test Method D5580 and a distillation temperature T₉₅, at which 95 % of the sample has evaporated, as measured by Test Method D86 is in the range of 149.1 °C to 196.6 °C (300.4 °F to 385.9 °F).

1.6.1 The applicable Test Method D5769 range for total aromatics is from 3.7 % to 29.4 % by volume as reported by Test Method D5769 and the distillation temperature T₉₅, at which 95 % of the sample has evaporated, when tested according to Test Method D86 ranged from 149.1 °C to 196.6 °C (300.4 °F to 385.9 °F).

1.7 This test method includes a relative bias section for spark-ignition engine fuels (gasolines) for benzene reporting based on Practice D6708 accuracy assessment between Test Method D5580 and Test Method D5769 as a possible Test Method D5580 alternative to Test Method D5769. The Practice D6708 derived correlation equation for benzene is applicable in the test method inclusive valid reporting concentration ranges, as determined from Practice D6708 data set and precision working limits of Test Method D5580, from 0.08 % to 2.34 % by volume as measured by Test Method D5580.

1.8 Many of the common alcohols and ethers that are added to gasoline to reduce carbon monoxide emissions and increase octane, do not interfere with the analysis. Ethers such as methyl *tert*-butylether (MTBE), ethyl *tert*-butylether (ETBE), *tert*-amylmethylether (TAME), and diisopropylether (DIPE) have been found to elute from the precolumn with the nonaromatic hydrocarbons to vent. Other oxygenates, including methanol and ethanol elute before benzene and the aromatic hydrocarbons. 1-Methylcyclopentene has also been found to elute from the precolumn to vent and does not interfere with benzene.

1.9 The values stated in SI units are to be regarded as standard.

1.9.1 *Exception*—The values given in parentheses are for information only.

*This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0L on Gas Chromatography Methods.

Current edition approved April 1, 2021. Published April 2021. Originally approved in 1994. Last previous edition approved in 2020 as D5580 – 15 (2020). DOI: 10.1520/D5580-21.

1.10 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.11 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure

D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

D3606 Test Method for Determination of Benzene and Toluene in Spark Ignition Fuels by Gas Chromatography

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D5769 Test Method for Determination of Benzene, Toluene, and Total Aromatics in Finished Gasolines by Gas Chromatography/Mass Spectrometry

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants

D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

E355 Practice for Gas Chromatography Terms and Relationships

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *aromatic*, *n*—any organic compound containing a benzene ring.

3.1.2 *low-volume connector*, *n*—a special union for connecting two lengths of narrow bore tubing 1.6 mm (0.06 in.) outside diameter and smaller; sometimes this is referred to as zero dead volume union.

3.1.3 *narrow bore tubing*, *n*—tubing used to transfer components prior to or after separation; usually 0.5 mm (0.02 in.) inside diameter and smaller.

3.1.4 *split ratio*, *n*—in capillary gas chromatography, the ratio of the total flow of carrier gas to the sample inlet versus the flow of the carrier gas to the capillary column, expressed by:

$$\text{split ratio} = (S + C)/C \quad (1)$$

where:

S = flow rate at the splitter vent, and

C = flow rate at the column outlet.

3.1.5 *1,2,3-tris-2-cyanoethoxypropane (TCEP)*, *n*—a polar gas chromatographic liquid phase.

3.1.6 *wall-coated open tubular (WCOT)*, *n*—a type of capillary column prepared by coating the inside wall of the capillary with a thin film of stationary phase.

4. Summary of Test Method

4.1 A two-column chromatographic system equipped with a column switching valve and a flame ionization detector is used. A reproducible volume of sample containing an appropriate internal standard such as 2-hexanone is injected onto a precolumn containing a polar liquid phase (TCEP). The C₉ and lighter nonaromatics are vented to the atmosphere as they elute from the precolumn. A thermal conductivity detector may be used to monitor this separation. The TCEP precolumn is backflushed immediately before the elution of benzene, and the remaining portion of the sample is directed onto a second column containing a nonpolar liquid phase (WCOT). Benzene, toluene, and the internal standard elute in the order of their boiling points and are detected by a flame ionization detector. Immediately after the elution of the internal standard, the flow through the nonpolar WCOT column is reversed to backflush the remainder of the sample (C₈ and heavier aromatics plus C₁₀ and heavier nonaromatics) from the column to the flame ionization detector.

4.2 The analysis is repeated a second time allowing the C₁₂ and lighter nonaromatics, benzene and toluene to elute from the polar TCEP precolumn to vent. A thermal conductivity detector may be used to monitor this separation. The TCEP precolumn is backflushed immediately prior to the elution of ethylbenzene and the remaining aromatic portion is directed into the WCOT column. The internal standard and C₈ aromatic components elute in the order of their boiling points and are detected by a flame ionization detector. Immediately after o-xylene has eluted, the flow through the nonpolar WCOT column is reversed to backflush the C₉ and heavier aromatics to the flame ionization detector.

4.3 From the first analysis, the peak areas of benzene, toluene, and the internal standard (2-hexanone) are measured and recorded. Peak areas for ethylbenzene, *p/m*-xylene, *o*-xylene, the C₉ and heavier aromatics, and internal standard are measured and recorded from the second analysis. The backflush peak eluting from the WCOT column in the second analysis contains only C₉ and heavier aromatics.

4.4 The flame ionization detector response, proportional to the concentration of each component, is used to calculate the amount of aromatics that are present with reference to the internal standard.

5. Significance and Use

5.1 Regulations limiting the concentration of benzene and the total aromatic content of finished gasoline have been

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.

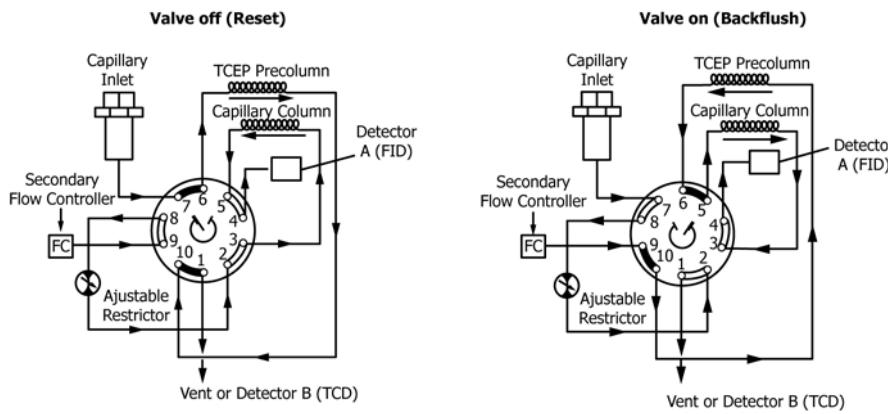


FIG. 1 Valve Diagram, Aromatics in Gasoline

established for 1995 and beyond in order to reduce the ozone reactivity and toxicity of automotive evaporative and exhaust emissions. Test methods to determine benzene and the aromatic content of gasoline are necessary to assess product quality and to meet new fuel regulations.

5.2 This test method can be used for gasolines that contain oxygenates (alcohols and ethers) as additives. It has been determined that the common oxygenates found in finished gasoline do not interfere with the analysis of benzene and other aromatics by this test method.

6. Apparatus

6.1 *Chromatographic System*—See Practice E355 for specific designations and definitions. Refer to Fig. 1 for a diagram of the system.

6.1.1 *Gas Chromatograph (GC)*, capable of operating at the conditions given in Table 1, and having a column switching and backflushing system equivalent to Fig. 1. Carrier gas pressure and flow control devices shall be capable of precise control when column head pressures and flow rates are low.

6.1.2 *Sample Introduction System*, capable of introducing a representative sample into the gas chromatographic inlet. Microlitre syringes and automatic syringe injectors have been used successfully.

6.1.3 *Inlet System, (splitting type)*—Split injection is necessary to maintain the actual chromatographed sample size within the limits required for optimum column efficiency and detector linearity.

6.1.3.1 Some gas chromatographs are equipped with on-column injectors and autosamplers which can inject submicrolitre sample sizes. Such systems can be used provided that column efficiency and detector linearity are comparable to systems with split injection.

6.1.4 *Detector*—A flame ionization detector (Detector A) is employed for quantitation of components eluting from the WCOT column. The flame ionization detector used for Detector A shall have sufficient sensitivity and stability to detect 0.01 % by volume of an aromatic compound.

6.1.4.1 It is strongly recommended that a thermal conductivity detector be placed on the vent of the TCEP precolumn (Detector B). This facilitates the determination of valve

TABLE 1 Typical Chromatographic Operating Parameters 130

Temperatures	
Injection port (split injector)	200 °C
FID (Detector A)	250 °C
TCD (Detector B)	200 °C
Nonpolar WCOT capillary	
Initial	60 °C (6 min)
Program rate	2 °C/min
Final	115 °C (hold until all components elute)
Polar TCEP precolumn (temperature to remain constant before time to BACKFLUSH, T1 or T2. Do not exceed maximum operating temperature.)	60 °C or same as nonpolar WCOT capillary if TCEP/WCOT columns contained in identical heated zone.
Valve	>115 °C or same as nonpolar WCOT capillary if valve and WCOT column contained in identical heated zone.
Flows and Conditions	
Carrier gas	helium
Flow to TCEP precolumn (split injector)	10 mL/min
Flow to WCOT capillary (auxiliary flow)	10 mL/min
Flow from split vent	100 mL/min
Detector gases	as necessary
Split ratio	11:1
Sample size	1 µL

BACKFLUSH and RESET times (10.5) and is useful for monitoring the separation of the polar TCEP precolumn.

6.1.5 *Switching and Backflushing Valve*, to be located within a temperature-controlled heated zone and capable of performing the functions in accordance with Section 10, and illustrated in Fig. 1. The valve shall be of low internal volume design and not contribute significantly to deterioration of chromatographic resolution.

6.1.5.1 A 10-port valve with 1.6 mm (0.06 in.) outside diameter fittings is recommended for this test method. Alternatively, and if using columns of 0.32 mm inside diameter or smaller, a valve with 0.8 mm (0.03 in.) outside diameter fittings should be used.

6.1.5.2 Some gas chromatographs are equipped with an auxiliary oven which can be used to contain the valve. In such a configuration, the valve can be kept at a higher temperature than the polar and nonpolar columns to prevent sample condensation and peak broadening. The columns are then located in the main oven and the temperature can be adjusted for optimum aromatic resolution.



6.1.5.3 An automatic valve switching device is strongly recommended to ensure repeatable switching times.

6.2 Data Acquisition System:

6.2.1 *Integrator or Computer*, capable of providing real-time graphic and digital presentation of the chromatographic data are recommended for use. Peak areas and retention times can be measured by computer or electronic integration.

6.2.1.1 It is recommended that this device be capable of performing multilevel internal-standard-type calibrations and be able to calculate the correlation coefficient (r^2) and linear least square fit equation for each calibration data set in accordance with 11.4.

6.3 Chromatographic Columns (two columns are used):

6.3.1 *Polar Precolumn*, to perform a pre-separation of the aromatics from nonaromatic hydrocarbons in the same boiling point range. Any column with equivalent or better chromatographic efficiency and selectivity in accordance with 6.3.1.1 can be used.

6.3.1.1 *TCEP Micro-Packed Column*, 560 mm (22 in.) by 1.6 mm ($\frac{1}{16}$ in.) outside diameter by 0.76 mm (0.030 in.) inside diameter stainless steel tube packed with 0.14 g to 0.15 g of 20 % (mass/mass) TCEP on 80/100 mesh Chromosorb P(AW). This column was used in the cooperative study to provide the precision and bias data referred to in Section 15.

6.3.2 *Nonpolar (Analytical) Column*—Any column with equivalent or better chromatographic efficiency and selectivity in accordance with 6.3.2.1 can be used.

6.3.2.1 *WCOT Methyl Silicone Column*, 30 m long by 0.53 mm inside diameter fused silica WCOT column with a 5.0 μm film thickness of cross-linked methyl siloxane.

7. Reagents and Materials

7.1 *Carrier Gas*, appropriate to the type of detector used. Helium has been used successfully. The minimum purity of the carrier gas used must be 99.95 mol %. Additional purification may be necessary to remove trace amounts of oxygen. (Warning—Helium is usually supplied as a compressed gas under high pressure.)

7.2 *Methylene Chloride*—Used for column preparation. Reagent grade, free of nonvolatile residue. (Warning—Harmful when ingested or inhaled at high concentrations.)

7.3 *2,2,4-Trimethylpentane (isooctane)*—Used as a solvent in the preparation of the calibration mixture. Reagent grade. (Warning—Isooctane is flammable and can be harmful or fatal when ingested or inhaled.)

7.4 *Standards for Calibration and Identification*, required for all components to be analyzed and the internal standard. Standards are used for establishing identification by retention time as well as calibration for quantitative measurements. These materials shall be of known purity and free of the other components to be analyzed. (Warning—These materials are flammable and may be harmful or fatal when ingested or inhaled.)

8. Preparation of Columns

8.1 TCEP Column Packing:

8.1.1 Use any satisfactory method, that will produce a column capable of retaining aromatics from nonaromatic components of the same boiling point range in a gasoline sample. The following procedure has been used successfully.

8.1.2 Completely dissolve 10 g of TCEP in 100 mL of methylene chloride. Next add 40 g of 80/100 mesh Chromosorb P(AW) to the TCEP solution. Quickly transfer this mixture to a drying dish, in a fume hood, without scraping any of the residual packing from the sides of the container. Constantly, but gently, stir the packing until all of the solvent has evaporated. This column packing can be used immediately to prepare the TCEP column.

8.2 Micro-packed TCEP Column:

8.2.1 Wash a straight 560 mm (22 in.) length of 1.6 mm ($\frac{1}{16}$ in.) outside diameter, 0.76 mm (0.030 in.) inside diameter stainless steel tubing with methanol and dry with compressed nitrogen.

8.2.2 Insert 6 to 12 strands of silvered wire, a small mesh screen or stainless steel frit inside one end of the tube. Slowly add 0.14 g to 0.15 g of packing material to the column and gently vibrate to settle the packing inside the column. Insert silvered wire, mesh screen, or frit to the other end of the tube to prevent the packing material from falling. When strands of wire are used to retain the packing material inside the column, leave 6.0 mm (0.25 in.) of space at the top of the column.

8.3 *WCOT Methyl Silicone Column*—It is suggested that this column be purchased directly from a suitable capillary column manufacturer (see 6.3.2.1).

9. Sampling

9.1 Every effort should be made to ensure that the sample is representative of the fuel source from which it is taken. Follow the recommendations of Practice D4057, or its equivalent, when obtaining samples from bulk storage or pipelines.

9.2 Appropriate steps should be taken to minimize the loss of light hydrocarbons from the gasoline sample to be analyzed. Upon receipt in the laboratory, chill the sample in its original container from 0 °C to 5 °C (32 °F to 40 °F) before and after sub-sampling is performed.

9.3 If necessary, transfer the chilled sample to a vaportight container and store at 0 °C to 5 °C (32 °F to 40 °F) until needed for analysis.

10. Preparation of Apparatus and Establishment of Conditions

10.1 *Assembly*—Connect the TCEP and WCOT column to the valve system (Fig. 1) using low-volume connectors and narrow bore tubing. It is important to minimize the volume of the chromatographic system that comes in contact with the sample, otherwise peak broadening will occur.

10.2 *Initial Operating Conditions*—Adjust the operating conditions initially to approximately those listed in Table 1, but do not turn on the detector circuits. Check the system for leaks before proceeding further.

10.2.1 If different polar and nonpolar columns are used, or WCOT capillary columns of smaller inner diameter or different

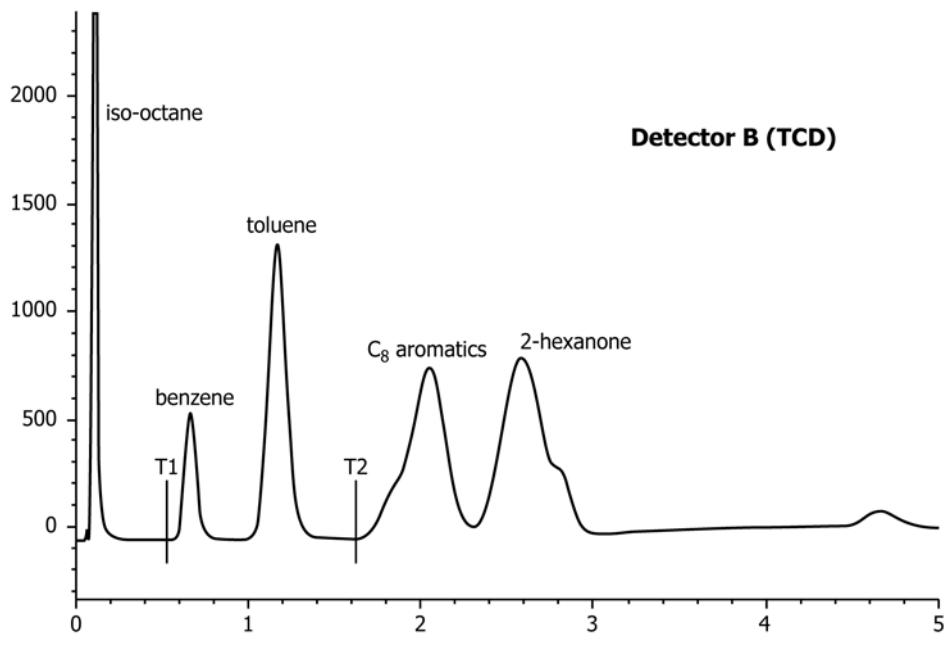


FIG. 2 Determination of Precolumn Backflush Times, T1 and T2

film thickness, or both, are used, it may be necessary to use different optimum flows and temperatures.

10.2.2 Conditions listed in **Table 1** are applicable to the columns described in **6.3**. If a WCOT column of a different film thickness is used, the conditions chosen for the analysis must sufficiently separate toluene from the internal standard (first analysis) and ethylbenzene from the xylenes (second analysis).

10.3 Flow Rate (Carrier Gas) Adjustments:

10.3.1 Attach a flow measuring device to the precolumn vent (or Detector B) with the valve in the RESET or forward flow position and adjust the pressure of the capillary injection port (**Fig. 1**) to give approximately 10 mL/min flow (17 psi to 20 psi). Soap bubble flow meters are suitable. This represents the flow through the polar precolumn.

NOTE 1—The word “approximately” implies to get as close as possible to the stated column flows to initiate the further optimization of the system.

10.3.2 Attach a flow measuring device to the split injector vent and adjust the flow from the split vent using the flow controller to provide a flow of approximately 100 mL/min. Recheck the column vent flow set in **10.3.1** and adjust, if necessary. The split ratio should be approximately 11:1. (See **Note 1**.)

10.3.3 Switch the valve to BACKFLUSH position and adjust the variable restrictor to give the same precolumn vent flow set in **10.3.1**. This is necessary to minimize flow changes when the valve is switched.

10.3.4 Switch the valve to the RESET position and adjust the auxiliary flow controller to give a flow of approximately 10 mL/min at the Detector A (FID) exit. (See **Note 1**.)

10.4 *Detector Setup*—Depending on the particular type of instrumentation used, adjust the hydrogen, air, and makeup flows to the flame ionization detector and ignite the flame. If a thermal conductivity detector (Detector B) is being used to

monitor the vent effluent in the valve RESET position, set the reference flow and turn on the detector circuit.

10.5 Valve Backflush and Reset Times:

10.5.1 The time to BACKFLUSH and RESET the valve will vary slightly for each column system and must be determined as described in **10.5.1.1**, **10.5.1.2**, and **10.5.1.3**. The start time of the integrator or computer system and valve timer must be synchronized with the injection to accurately reproduce the backflush time. This procedure assumes that a thermal conductivity detector is installed on the precolumn vent line as Detector B (see **6.1.4.1**). If a detector is not available, the appropriate valve BACKFLUSH times, T1 and T2, must be determined experimentally. If the BACKFLUSH times, T1 and T2, are not set correctly (switched too late), it is possible that part of the benzene and ethylbenzene peaks will be vented.

10.5.1.1 Adjust the valve to RESET (forward flow) and inject 1.0 μ L of a blend containing approximately 5 % each of benzene, ethylbenzene, *o*-xylene, and 2-hexanone in *iso*octane. This mixture is used to set the valve timing, therefore, the exact concentration need not be known. Alternatively, the calibration mixture can be used for this test. Determine retention time in seconds at which benzene and ethylbenzene start to elute as measured by Detector B. Subtract 6 s from each of these and call these times to BACKFLUSH, T1 and T2, respectively. The correct time for T1 and T2 is just prior to the elution of benzene and ethylbenzene from the TCEP precolumn.

NOTE 2—**Fig. 2** is an example chromatogram illustrating the elution of a calibration mixture from the polar precolumn using the procedure described in **10.5.1.1**. Times to BACKFLUSH, T1 and T2, are indicated on the chromatogram. The times to BACKFLUSH, T1 and T2, should be optimized for each chromatographic system.

10.5.1.2 Reinject the calibration blend and turn the valve to BACKFLUSH at time T1. When the internal standard peak (2-hexanone) returns to baseline switch valve back to RESET (forward flow) position. Call this time T3.



10.5.1.3 Reinject the calibration blend and BACKFLUSH at time T2. When the *o*-xylene peak returns to baseline, switch the valve back to RESET (forward flow). Call this time T4.

10.6 Polar Precolumn Selectivity Check:

10.6.1 The selectivity of the polar precolumn is critical to allow for accurate determination of the C₉ and heavier aromatics without non-aromatic interferences. The selectivity must be verified so that for the second analysis, when the time to BACKFLUSH T2 is properly adjusted, all of the C₁₂ and lighter nonaromatic hydrocarbons are vented from the polar precolumn while the heavier aromatics are retained. The following test can be used to verify the precolumn performance.

10.6.1.1 Prepare a blend containing approximately 1.7 % *n*-dodecane in 2,2,4-trimethylpentane (*isooctane*). *n*-Dodecane is used to represent the high boiling nonaromatic hydrocarbons in gasoline. Inject 1.0 μ L of the mixture under the conditions specified in 10.2 to 10.5 and actuate the valve at time T2 (BACKFLUSH) and time T4 (RESET). Record the signals from both the flame ionization (Detector A) and thermal conductivity (Detector B) detectors. Verify that *n*-dodecane fully elutes from the polar precolumn before BACKFLUSH time T2. When monitoring the thermal conductivity detector (Detector B), the *n*-dodecane peak should return to baseline before BACKFLUSH time T2. If not, part of the *n*-dodecane peak will be backflushed to the non-polar WCOT column and be detected by the flame ionization detector after the valve RESET time T4. If a thermal conductivity detector is not available on the precolumn vent line, the chromatogram obtained by the flame ionization detector can be used to verify that all the *n*-dodecane is being vented. This chromatogram should not show any significant response from *n*-dodecane after the RESET time T4.

10.6.1.2 If all of the *n*-dodecane peak is not completely vented from the polar precolumn, as measured by the thermal conductivity or flame ionization detector, recheck instrument parameters and valve backflush times (10.5) or replace the polar precolumn. If the valve is contained in a separate isothermal heated zone, it may be necessary to use a higher temperature to prevent absorption of small amounts of *n*-dodecane on the rotor or transfer tubing surfaces.

11. Calibration

11.1 *Preparation of Calibration Samples*—Prepare multi-component calibration standards of benzene, toluene, ethylbenzene, *o*-xylene, and 1,2,4-trimethylbenzene at concentrations of interest by mass in accordance with Practice D4307. *O*-xylene is used to represent the three xylenes while 1,2,4-trimethylbenzene is used for the C₉ and heavier aromatics. For each aromatic component, use at least five calibration points and ensure that the concentration of each aromatic component is within its calibration range. For convenience, a fixed volume of the calibration components may be dispensed and the mass of each obtained. As an example, for benzene, calibration concentrations of 0.1 %, 0.5 %, 1.0 %, 2.0 %, and 5 % by volume can be used. For toluene: 1.0 %, 2.5 %, 5.0 %, 10.0 %, and 15.0 % by volume. For ethylbenzene, *o*-xylene, and 1,2,4-trimethylbenzene: 0.5 %, 1.0 %, 2.5 %, 5.0 %, and 10 %

TABLE 2 Relative Densities for Interconversion of Mass-Volume

Component	Relative Density (15.56/15.56 °C) ^A
Benzene	0.8845
Toluene	0.8719
Ethylbenzene	0.8717
<i>p/m</i> -Xylene	0.8679
<i>o</i> -Xylene	0.8848
1,2,4-Trimethylbenzene	0.8806
C ₉ plus aromatics	0.8764
2-hexanone	0.8162

^A "Physical Constants of Hydrocarbons C₁–C₁₀," STP 109A, ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428–2959. The mixed xylene (*p/m*-xylene) density based upon a 1:3 ratio of *p*-xylene to *m*-xylene. C₉ plus aromatics based upon the average relative density values of the 30 C₉–C₁₀ aromatics.

by volume can be used. The relative densities listed in Table 2 shall be used as a guide in determining the proper mass of aromatic components that needs to be dispensed in order to arrive at a target volume percent concentrations.

11.2 Before preparing the standards, determine the purity of the aromatics by capillary GC and make corrections for the impurities found. Whenever possible, use stocks of at least 99.9 % purity.

11.3 Prepare standards by transferring a fixed volume of aromatic component using pipettes, eye droppers, or syringes to 100 mL volumetric flasks or 100 mL septum-capped vials as follows. Cap and record the tare weight of the volumetric flask or vial to 0.1 mg. Remove the cap and carefully add the aromatic components to the flask or vial starting with the least volatile (1,2,4-trimethylbenzene). Cap the flask and record the net mass (W_i) of the aromatic component added to 0.1 mg. Repeat the addition and weighing procedure for each aromatic component. Do not exceed 50 % by volume for all aromatics added. Similarly, add 10 mL of the internal standard, 2-hexanone, and record its net mass (W_s) to 0.1 mg. Dilute each standard to the mark with aromatics free 2,2,4-trimethylpentane (*isooctane*). Store the capped calibration standards in a refrigerator at 0 °C to 5 °C (32 °F to 40 °F) when not in use.

11.4 *Calibration Procedure*—With the valve initially in the RESET mode, chromatograph each of the calibration mixtures (11.1) twice using valve timing procedures in accordance with 10.5. For the first analysis use times T1 (BACKFLUSH) and T3 (RESET) to actuate the valve. For the second analysis use times T2 (BACKFLUSH) and T4 (RESET) to actuate the valve.

NOTE 3—The first analysis is used to calibrate the gas chromatograph for benzene and toluene. The second analysis is used to calibrate for ethylbenzene, the three xylenes (the calibration curve of *o*-xylene is used to calibrate the three xylenes), and the C₉ and heavier aromatics (1,2,4-trimethylbenzene).

11.4.1 *Linearity Test*—Analyze the calibration standards in accordance with the procedure in 11.4. Measure the peak areas of benzene, toluene, and internal standard peaks from the first analysis. From the second analysis measure the peak areas

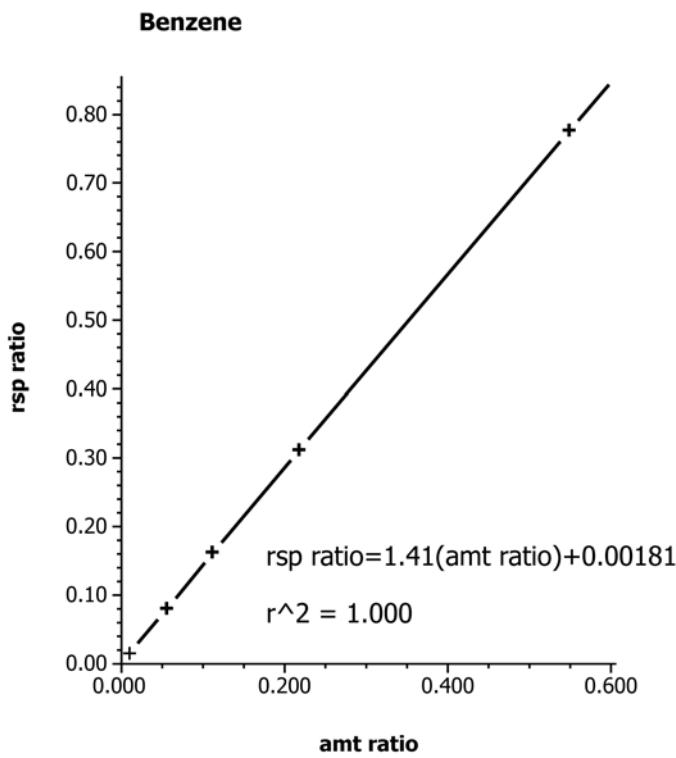


FIG. 3 Typical Benzene Calibration Curve

of internal standard, ethylbenzene, *o*-xylene, and 1,2,4-trimethylbenzene. Determine the response ratio (rsp_i) and amount ratio (amt_i) for each component in each standard using Eq 2 and Eq 3.

$$rsp_i = (Ai/As) \quad (2)$$

where:

Ai = area of aromatic component, and
 As = area of internal standard.

$$amt_i = (Wi/Ws) \quad (3)$$

where:

Wi = mass of aromatic component, and
 Ws = mass of internal standard.

11.4.1.1 Prepare a calibration curve for each aromatic component by plotting the response ratios (rsp_i), as the y-axis, versus the amount ratios (amt_i), as the x-axis. Fig. 3 is an example of such a plot.

11.4.1.2 Calculate the correlation coefficient r^2 value for each aromatic component in the calibration using Eq 4. The r^2 value should be at least 0.990 or greater. If the above criteria for r^2 is not met, rerun the calibration or check instrument parameters and hardware.

$$r^2 = \frac{(\sum xy)^2}{(\sum x^2)(\sum y^2)} \quad (4)$$

where:

$$x = X_i - \bar{x} \quad (5)$$

$$y = Y_i - \bar{y} \quad (6)$$

TABLE 3 Example of Data Set for r^2 Calculation^A

X_i	Y_i	$x = X_i - \bar{x}$	$y = Y_i - \bar{y}$	xy	x^2	y^2
1.0	0.5	-2.0	1.0	2.0	4.0	1.0
2.0	1.0	-1.0	-0.5	0.5	1.0	0.25
3.0	1.5	0.0	0.0	0.0	0.0	0.0
4.0	2.0	1.0	0.5	0.5	1.0	0.25
5.0	2.5	2.0	1.0	2.0	4.0	1.0

$$\bar{x} = 3$$

$$\bar{y} = 1.5$$

$$(\sum xy)^2 = 25.0$$

$$\sum x^2 = 10.0$$

$$\sum y^2 = 2.5$$

$$r^2 = \frac{(\sum xy)^2}{(\sum x^2)(\sum y^2)}$$

$$r^2 = \frac{25.0}{(10.0)(2.5)} = 1.0$$

and:

X_i = amt_i ratio data point,

\bar{x} = average values for all amt_i data points,

Y_i = corresponding rsp_i ratio data point, and

\bar{y} = average values for all rsp_i data points.

11.4.1.3 Table 3 gives an example on the calculation of r^2 for an ideal data set.

11.4.2 Linear Least Square Fit—For each aromatic i calibration data set, obtain the linear least square fit equation in the form:

$$(rsp_i) = (m_i)(amt_i) + b_i \quad (7)$$

where:

rsp_i = response ratio for aromatic i (y-axis),

m_i = slope of linear equation for aromatic i ,

amt_i = amount ratio for aromatic i (x-axis), and

b_i = y-axis intercept.

11.4.2.1 The values m_i and b_i are calculated as follows:

$$m_i = \sum xy / \sum x^2 \quad (8)$$

and:

$$b_i = \bar{y} - m_i \bar{x} \quad (9)$$

11.4.2.2 For the example in Table 3:

$$m_i = 5/10 = 0.5 \quad (10)$$

and

$$b_i = 1.5 - (0.5)(3) = 0 \quad (11)$$

11.4.2.3 Therefore, the least square equation (Eq 7) for the example in Table 3 is:

$$(rsp_i) = 0.5(amt_i) + 0 \quad (12)$$

NOTE 4—Normally the b_i value is not zero and can be positive or negative. Fig. 3 gives an example of linear least square fit for benzene and the resulting equation in the Eq 7.

11.4.3 Y-Intercept Test—For an optimum calibration, the absolute value of the y-intercept (b_i) must be at a minimum. In this case, Ai approaches zero when w_i is less than 0.1 % by mass. In practice, this means the mass % (w_i) calculated for an aromatic with zero peak area must be close to zero. The equation to determine the mass % aromatic i , or w_i , reduces to Eq 13. The y-intercept can be tested using Eq 13 below:

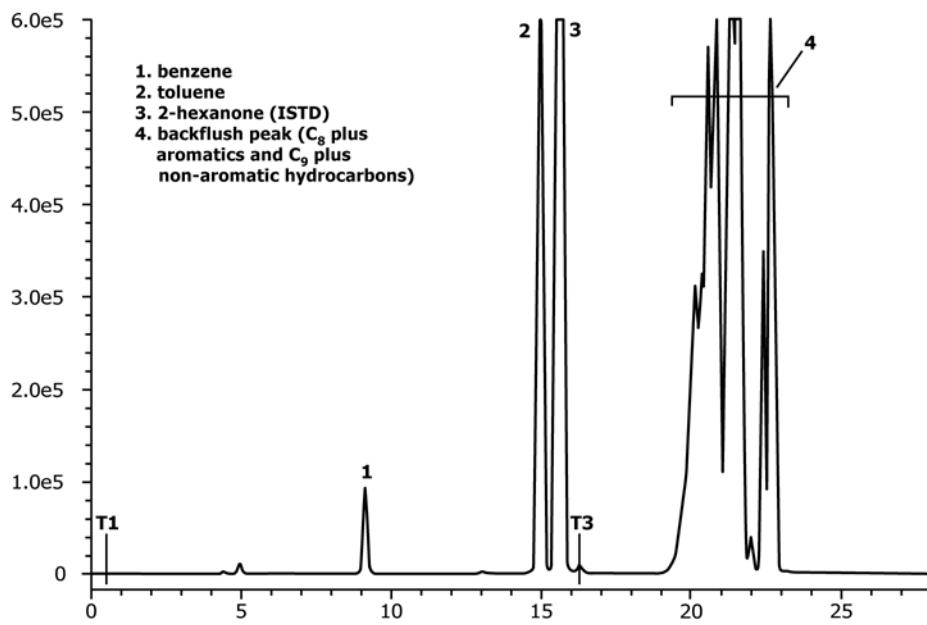


FIG. 4 Aromatics in Gasoline, Analysis No. 1

$$w_i = (b_i/m_i)(W_s/W_g)100\% \quad (13)$$

where:

w_i = mass % aromatic i ,

W_s = mass of internal standard added, g, and

W_g = mass of gasoline samples, g.

NOTE 5—Since in practice W_s and W_g vary slightly from sample to sample, use an average value as indicated below.

For benzene, w_i must be less than 0.02 % by mass. For the other aromatics, w_i must be less than 0.2 % by mass. If any of the w_i values exceed the mass % limit, rerun the calibration procedure for aromatic i , or check instrument parameters and hardware.

The following gives an example of the calculation for the y-intercept (b_i) test using the data from Fig. 3 for aromatic i (benzene) for which $b_i = 0.0018$ and $m_i = 1.41$. From 13.1, a typical sample preparation may contain approximately $W_s = 0.8$ g (1.0 mL) of internal standard and $W_g = 6.75$ g (9.0 mL) of a gasoline sample. Substituting these values into Eq 13 yields:

$$w_i = 0.01\% \text{ by mass} \quad (14)$$

$$w_i = (0.0018/1.41)(0.8/6.75)100\%$$

Since w_i is less than 0.02 % by mass, the y-intercept (b_i) has an acceptable value for benzene. Similarly, determine w_i for all other aromatics.

12. Column Conditioning

12.1 Both the TCEP and WCOT columns are to be briefly conditioned before use. Connect the columns to the valve (see Fig. 1 and 10.1) in the chromatographic oven. Adjust the carrier gas flows in accordance with 10.3 and place the valve in the RESET position. After several minutes, increase the column oven temperature to 120 °C and maintain these conditions for 20 min. Cool the columns below 60 °C before shutting off the carrier gas.

13. Procedure

13.1 *Preparation of Sample*—Transfer 1 mL of internal standard (W_s) by a volumetric pipette into a tared and capped 10 mL volumetric flask or capped vial. Record the net mass of the internal standard added to the nearest 0.1 mg. Retare the capped flask or vial. Fill the volumetric flask or vial with 9 mL of chilled sample, cap, and record the net mass (W_g) of the sample added. Mix thoroughly. If using an automatic sampler then transfer an aliquot of the solution into a glass GC vial. Seal the GC vial with a TFE-fluorocarbon-lined cap. If the sample is not immediately analyzed, store at 0 °C to 5 °C (32 °F to 40 °F).

13.2 *Chromatographic Analysis*—Introduce an aliquot of the sample, containing internal standard, into the gas chromatograph using the same technique and sample size as used for the calibration analysis. An injection volume of 1 µL with a 11:1 split ratio has been used successfully. Chromatograph the sample twice using valve timing procedures in accordance with 10.5. Use times T1 and T3 for the first analysis to BACK-FLUSH and RESET the valve. Use times T2 and T4 for the second analysis.

13.3 *Interpretation of Chromatogram*—Compare the retention times of sample components to those of the calibration analysis to determine the identities of the aromatics. Identify benzene, toluene, and the internal standard from the first analysis. Identify the internal standard, ethylbenzene, *p*/*m*-xylene, *o*-xylene, C₉ and heavier aromatic composite from the second analysis. Refer to Fig. 4 and Fig. 5 for sample chromatograms.

NOTE 6—Gasolines containing styrene will have an additional peak in the chromatogram. Styrene elutes just before *o*-xylene; the two peaks are partially, but not baseline, resolved. If quantitation of styrene is desired, use the *o*-xylene calibration. To identify the *p*/*m* xylenes, a calibration standard may be spiked with *p*-xylene and *m*-xylene.

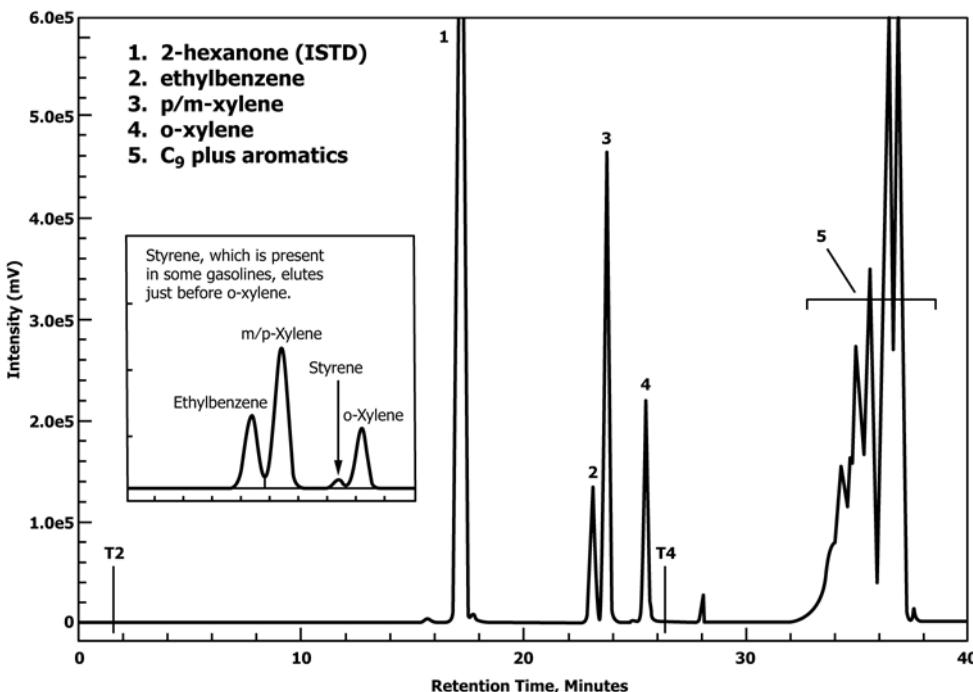


FIG. 5 Aromatics in Gasoline, Analysis No. 2

14. Calculation and Report

14.1 *Mass Concentration of Aromatics*—After identifying the peaks, measure the areas of benzene, toluene, and the internal standard from the first analysis and the internal standard, ethylbenzene, *p/m*-xylene, *o*-xylene, C₉ and heavier aromatics from the second analysis. Using the slope and y-intercept of the least square fit calibrations in 11.4.2, calculate the mass of each aromatic component (*W_i*) in the gasoline samples using the response ratio (*rsp_i*) of the areas of the aromatic component to the internal standard as follows:

$$W_i = \left[\left(\frac{A_i}{A_s} - b_i \right) / m_i \right] W_s \quad (15)$$

where:

W_i = mass of aromatic component *i*,

A_i = area of aromatic component *i*,

A_s = area of internal standard, and

W_s = mass of internal standard added.

14.1.1 To obtain mass percent (*w_i*) results for each component:

$$w_i = \frac{W_i(100)}{W_g} \quad (16)$$

where:

W_g = mass of gasoline sample.

14.1.2 Report the mass percent (*w_i*) results of the following aromatics to the nearest 0.01 %; benzene, toluene, ethylbenzene, *p/m*-xylene, *o*-xylene, C₉ and heavier aromatics.

14.1.3 To obtain the total mass percent aromatics, sum the mass percent (*w_i*) results of all the individual aromatic components *i*.

TABLE 4 Repeatability Estimates for Aromatics in Gasoline

Component	Range, (mass %)	Repeatability (X = mass %)
Benzene	0.14–1.79	0.0265(X ^{0.65})
Toluene	2.11–10.08	0.0301(X ^{0.5})
Ethylbenzene	0.57–2.65	0.029
P/M-xylene	2.06–9.59	0.071
O-xylene	0.77–3.92	0.0296(X ^{0.5})
C ₉ plus aromatics	8.32–25.05	0.0145(X+5.157)
Total aromatics	16.34–49.07	0.0899(X ^{0.5})

TABLE 5 Reproducibility Estimates for Aromatics in Gasoline

Component	Range, (mass %)	Reproducibility (X = mass %)
Benzene	0.14–1.79	0.1229(X ^{0.65})
Toluene	2.11–10.08	0.0926(X ^{0.5})
Ethylbenzene	0.57–2.65	0.163
P/M-xylene	2.06–9.59	0.452
O-xylene	0.77–3.92	0.1168(X ^{0.5})
C ₉ plus aromatics	8.32–25.05	0.070(X+5.157)
Total aromatics	16.34–49.07	0.2851(X ^{0.5})

14.2 *Volumetric Concentration of Aromatic Components*—If the volumetric concentration of each aromatic component *i* is desired, calculate the volumetric concentration in accordance with Eq 15:

$$v_i = w_i \left(\frac{D_f}{D_i} \right) \quad (17)$$

where:

v_i = volume percent of each aromatic component to be determined,

D_f = relative density of the fuel under study as determined in accordance with Test Method D1298 or Test Method D4052, and

TABLE 6 Volume % Precision for Benzene and Total Aromatics^A

	Range, (Volume %)	Repeatability	Reproducibility
Benzene	0.11–1.5	0.0259(X ^{0.64})	0.1087(X ^{0.64})
Total Aromatics	14–43	0.0825(X ^{0.5})	0.2619(X ^{0.5})

^A The precision in Table 6 was obtained with a constant sample density for each sample provided to the round robin participants to convert mass % to volume %.

D_i = relative density at 15.56 °C (60 °F) of the individual aromatics (Table 2).

14.2.1 Report the volume percent results (v_i) of the following aromatic components to the nearest 0.01 %; benzene, toluene, ethylbenzene, *p/m*-xylene, *o*-xylene, and C₉ and heavier aromatics.

14.2.2 To obtain total volume percent aromatics, sum the volume percent (v_i) results of all the individual aromatic components i .

15. Precision and Bias³

15.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test reports is as follows:³

15.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method exceed the following values in only one case in twenty. See Table 4.

15.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical material, would in the long run, exceed the following values in one case in twenty. See Table 5.

15.1.3 *Bias*—Since there is no accepted reference material suitable for determining bias for the procedure in this test method, no statement of bias is being made.

15.2 *Relative Bias*—A relative bias assessment of Test Method D5580 versus Test Method D3606 for the determination of benzene in spark ignition fuel was conducted using data from the ASTM D02 Interlaboratory Crosscheck Program. The assessment was performed in accordance with the requirements of Practice D6708 with a successful outcome. It was based on measurements of benzene in spark ignition fuels supplied between January 2006 and October 2014 by the Reformulated Gasoline program, plus fuels distributed between March 2004 and October 2014 by the Motor Gasoline program of the ASTM Proficiency Testing Program (Interlaboratory Crosscheck Program) by participating laboratories and is documented in Research Report RR:D02-1815.⁴

NOTE 7—In the United States, the EPA requires the measurement of benzene in spark-ignition engine fuels by Test Method D3606. Effective

³ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1329. Contact ASTM Customer Service at service@astm.org.

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1815. Contact ASTM Customer Service at service@astm.org.

Jan. 1, 2016, there is an allowance in the regulation to use other test methods if they are formally correlated with the specified test method by a consensus organization, for example, ASTM. This relative bias statement is intended to satisfy the requirement and allow use of Test Method D5580 bias-corrected results in the stated concentration ranges in place of Test Method D3606 for benzene.

15.2.1 The degree of agreement between results from Test Method D5580 and Test Method D3606 can be further improved by applying a correlation equation (Eq 18) (Research Report RR:D02-1815),⁴ and this correlation equation shall be utilized when reporting compliance with EPA fuels program. There were no discernable sample-specific biases as defined in Practice D6708.

15.2.2 Correlation Equation:

$$\text{Predicted Test Method D3606} =$$

$$\text{Bias-corrected Test Method D5580} =$$

$$C_{\text{D5580}} - 0.003 \quad (18)$$

where:

C_{D5580} = unrounded volume percent benzene as reported by Test Method D5580, and

$\text{Predicted Test Method D3606}$ = the outcome from Eq 18 rounded to the reported resolution of Test Method D3606.

15.2.2.1 The correlation equation is only applicable for fuels in the concentration range from 0.0 % to 2.31 % by volume as reported by Test Method D5580.

15.2.2.2 The correlation equation is applicable for fuels that when determined by Test Method D3606 are in the concentration range of 0.0 % to 2.38 % by volume.

NOTE 8—The Test Method D3606 concentration range used to develop the Practice D6708 assessment may not cover the entire scope indicated in the scope of Test Method D3606 for benzene.

NOTE 9—The correlation equation was developed from a variety of fuel samples from the ASTM Interlaboratory Crosscheck programs; however, it is recommended that the correlation equation be verified for samples of interest to ensure applicability.

15.3 *Relative Bias*—A relative bias assessment of Test Method D5580 versus Test Method D5769 for the determination of total aromatics in spark-ignition engine fuel was conducted using data from the ASTM D02 Interlaboratory Crosscheck Program. The assessment was performed in accordance with the requirements of Practice D6708 with a successful outcome. It was based on measurements of total aromatics in spark-ignition engine fuels supplied to the ASTM Proficiency Test Program by participating laboratories between February 2007 and October 2014 and is documented in Research Report RR:D02-1814.⁵

NOTE 10—In the United States, the EPA requires the measurement of total aromatics in spark-ignition engine fuels by Test Method D5769. Effective Jan. 1, 2016, there is an allowance in the regulation to use other test methods if they are formally correlated with the specified test method by a consensus organization, for example, ASTM. This relative bias

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1814. Contact ASTM Customer Service at service@astm.org.



statement is intended to satisfy the requirement and allow use of Test Method D5580 bias-corrected results in the stated concentration ranges in place of Test Method D5769 for total aromatics content.

15.3.1 The degree of agreement between results from Test Method D5580 and Test Method D5769 can be further improved by applying a correlation equation (Eq 19 or Eq 20) (Research Report RR:D02-1814),⁵ and this correlation equation shall be utilized when reporting compliance with EPA fuels program. Sample-specific bias, as defined in Practice D6708, was observed for some samples after applying the bias-correction for the material types and property range listed below.

15.3.2 Correlation Equation:

Predicted Test Method D5769 =

$$\text{bias-corrected Test Method D5580} = \\ C_{D5580} - [0.0867 \times (T_{95} - 153.5)] \quad (19)$$

for T_{95} expressed in degrees Celsius, or

Predicted Test Method D5769 =

$$\text{bias-corrected Test Method D5580} = \\ C_{D5580} - [0.0482 \times (T_{95} - 307.9)] \quad (20)$$

for T_{95} expressed in degrees Fahrenheit.

where:

Predicted Test Method D5769	= the Test Method D5769 predicted volume percent,
C_{D5580}	= the Test Method D5580 reported total aromatics volume percent, and
T_{95}	= the distillation temperature at which 95 % of the sample has evaporated when tested in accordance with Test Method D86.

15.3.2.1 The correlation equation is only applicable for fuels in the concentration range from 5.4 % to 31.6 % by volume as reported by Test Method D5580 and T_{95} values ranging from 149.1 °C to 196.6 °C (300.4 °F to 385.9 °F).

15.3.2.2 The correlation equation is applicable for fuels that when determined by Test Method D5769 are in the concentration range of 3.7 % to 29.4 % by volume and T_{95} values ranging from 149.1 °C to 196.6 °C (300.4 °F to 385.9 °F).

NOTE 11—The Test Method D5769 concentration range used to develop the Practice D6708 assessment may not cover the entire scope indicated in the scope of Test Method D5769 for total aromatics.

NOTE 12—The correlation equation was developed from a variety of fuel samples from the ASTM Interlaboratory Crosscheck programs; however, it is recommended that the correlation equation be verified for samples of interest to ensure applicability.

15.4 Relative Bias—A relative bias assessment of Test Method D5580 versus Test Method D5769 for the determination of benzene in spark ignition fuels (gasolines) was conducted in accordance with the requirements of Practice D6708 with a successful outcome. It was based on measurements of benzene in spark ignition fuels (gasolines) supplied between January 2006 and October 2014 from the ASTM D02 Reformulated Gasoline and the Motor Gasolines Interlaboratory Crosscheck Programs of the ASTM Proficiency Testing Pro-

gram by participating laboratories and is documented in Research Report RR:D02-2016.⁶

NOTE 13—Certain fuel regulations require the measurement of benzene in spark-ignition engine (gasolines) fuels. There may be an allowance in these regulations to use other test methods if they are formally correlated with the specified test method by a consensus organization, for example, ASTM. This relative bias statement may satisfy such requirement and allow use of Test Method D5580 bias-corrected results in the stated concentration ranges in place of Test Method D5769 for benzene. Users should review pertinent regulations related to such use.

15.4.1 The degree of agreement between results from Test Method D5580 and Test Method D5769 can be further improved by applying a correlation equation (Eq 21) (Research Report RR:D02-2016).⁶ There were no discernable sample-specific biases as defined in Practice D6708.

15.4.2 Correlation Equation:

Predicted Test Method D5769 benzene =

$$\text{bias-corrected Test Method D5580 benzene} = \\ C_{D5580_benzene} - 0.002 \quad (21)$$

where:

$C_{D5580_benzene}$	= unrounded volume percent benzene as reported by Test Method D5580, and
Predicted Test Method D5769	= the outcome from Eq 21 rounded to the reported resolution of Test Method D5769.

NOTE 14—The bias correction is smaller than the reporting resolution of the D5580 and D5769 test methods (both are 0.01 % by volume). After applying the bias correction, the predicted D5769 benzene results are reported to nearest 0.01 % volume.

15.4.2.1 The correlation equation is only applicable for fuels in the inclusive valid reporting concentration range, per Practice D6300 section 8.4.5, test working limits and the Practice D6708 sample robust mean values, from 0.08 % to 2.34 % by volume as reported by Test Method D5580.

NOTE 15—The correlation equation was developed from a variety of fuel samples from the ASTM Interlaboratory Crosscheck programs; however, it is recommended that the correlation equation be verified for samples of interest to ensure applicability.

15.4.2.2 *Between Methods Reproducibility (R_{XY})*—The random error associated with the difference between the two results obtained by different operators using different apparatus and applying the two methods X (D5580) and Y (D5769), respectively, each obtaining a single result on an identical test sample, when the methods have been assessed and an appropriate bias-correction has been applied in accordance with Practice D6708, is defined as the 95 % confidence limit for the difference between two such single and independent results and can be estimated by Eq 22 where X = reproducibility of Test Method D5580 and Y = reproducibility of Test Method D5769.

$$R_{XY} = \sqrt{(R_X^2 + R_Y^2)/2} \quad (22)$$

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-2016. Contact ASTM Customer Service at service@astm.org.

16. Keywords

16.1 aromatics; benzene; ethylbenzene; gas chromatography; gasoline; toluene; xylenes

SUMMARY OF CHANGES

Subcommittee D02.04 has identified the location of selected changes to this standard since the last issue (D5580 – 15 (2020)) that may impact the use of this standard. (Approved April 1, 2021.)

(1) Added new subsections 1.7 and 15.4.

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