



Standard Test Method for Aromatics in Light Naphthas and Aviation Gasolines by Gas Chromatography¹

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1. Scope

1.1 This test method covers the determination of benzene, toluene, C_8 and heavier aromatics, and total aromatics in the concentration range from 1.0 to 30 % of the individual aromatic compounds listed above in the following products: (1) aviation gasolines described in Specification D 910; (2) reformer products having a final boiling point below 350°F (177°C); and (3) reformer feed and other petroleum products having a final boiling point below 300°F (149°C). The results are reported to the nearest 0.1 % by liquid volume.

NOTE 1—Test Method D 2600 should be used to determine aromatics in concentrations below 1.0.

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

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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. For specific hazard statements, see Notes 3, 4, 5, 6, 8, 9, 10, 11, 12, 13, 14, and Annex A1.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 910 Specification for Aviation Gasolines²
D 2600 Test Method for Aromatic Traces In Light Saturated Hydrocarbons by Gas Chromatography³
E 260 Practice for Packed Column Gas Chromatography⁴

3. Summary of Test Method

3.1 The sample is introduced into a gas chromatographic column containing a strongly polar liquid phase. The polar phase has very little affinity for saturated and olefinic hydrocarbons, while exhibiting a pronounced selectivity for aromatics. This selectivity results in the elution of all saturated and olefinic hydrocarbons in the products described above prior to the elution of benzene. Can either a thermal conductivity or flame ionization detector be used.

Calibration is performed by using synthetic blends of the aromatic compounds charged at the same constant volume as is used to analyze the sample. *n*-undecane or *n*-dodecane is used as an internal standard. Typical retention times are shown in Table 1 and a typical chromatogram is shown in Fig. 1.

NOTE 2—Reference should be made to Practice E 260.

NOTE 3: **Warning**—Naphthas and gasolines are extremely flammable, harmful if inhaled; see Annex A1.1. Precautionary statements for individual compounds follow in text.

4. Significance and Use

4.1 A knowledge of aromatic hydrocarbons in light naphthas and aviation gasolines is needed to assess product quality and meet specifications for aromatics as may be set by consumers and regulatory agencies.

5. Apparatus

5.1 *Chromatograph*—Any chromatographic instrument may be used which has the following performance and characteristics:

5.1.1 *Sensitivity*—An over-all sensitivity sufficient to detect 0.1 volume % of any aromatic compound of interest with a peak height of at least 2 mm without loss of resolution as defined in 5.1.3.

5.1.2 *Sample Inlet System*—A sample inlet system that allows a constant volume of liquid sample to be injected.

5.1.3 *Column*—Inject Blend 1, Table 2, into the chromatograph and record the chromatogram. (**Warning**—See Notes 4, 5, and 6.) Any column and conditions may be used provided, under conditions of the test, *n*-decane is eluted prior to benzene and the depth of the valley after *n*-decane is not less than 50 % of the height of the decane peak. The benzene, toluene, and C_8 and heavier aromatics must be completely resolved from each other, and the internal standard, *n*-undecane or *n*-dodecane, must be completely separated from all aromatic compounds.

NOTE 4: **Warning**—Benzene is a poison, carcinogen, harmful or fatal if swallowed; see Annex A1.2.

NOTE 5: **Warning**—Toluene and ethylbenzene are flammable, vapor harmful; see Annexes A1.3 and A1.4, respectively.

TABLE 1 Typical Retention Time, Minutes

Column	1	2	3	4	5
<i>n</i> -decane	2.3	6.8	2.6	2.0	10.2
Benzene	2.9	7.6	3.0	3.3	15
Toluene	4.5	11.4	4.3	5.3	19.1
Ethylbenzene	6.6	17.2	6.0	7.7	23.3
<i>n</i> -undecane	...	10.1	3.8	...	...
<i>n</i> -dodecane	5.85	...	...	...	...

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04 on Hydrocarbon Analysis.

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² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 05.02.

⁴ *Annual Book of ASTM Standards*, Vol 14.01.

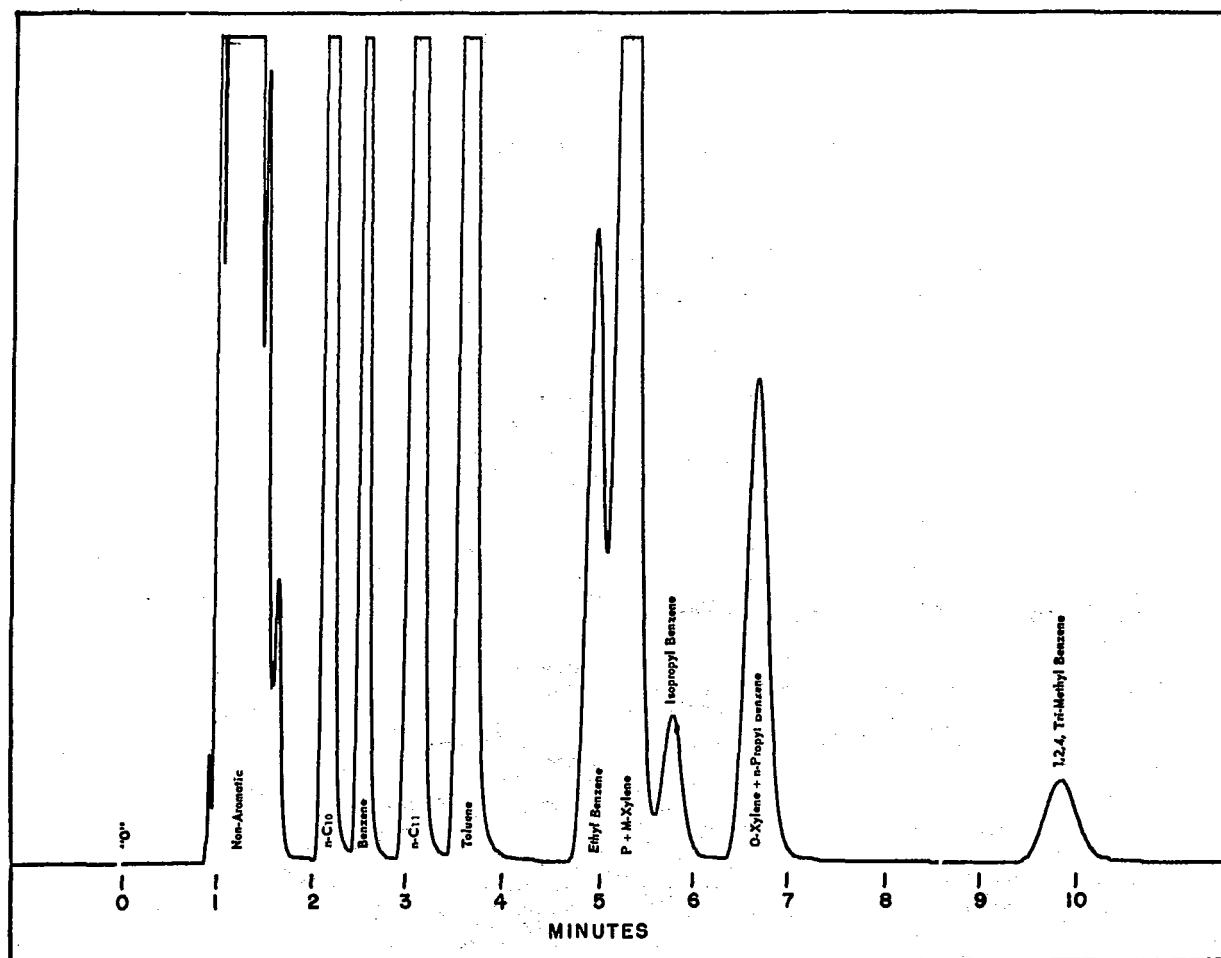


FIG. 1 Typical Chromatogram Using Table 3, Column 3

NOTE 6: Warning—*n*-Decane, *n*-undecane, and dodecane are combustible, vapor harmful; see Annex A1.5.

NOTE 7—Column, materials, and conditions that have been found suitable for this method are listed in Table 3. Typical instructions for preparing such columns may be found in Practice E 260.

5.2 *Strip Chart Recorder*—A recording potentiometer with a full-scale deflection of 10 mV or less should be used. The full-scale response of the recorder should not exceed 2 s. If a manual method of integration (such as triangulation, paper cut-out or planimeter) is employed, the chart speed should be at least 60 in./h (150 cm/h) in order to minimize errors in peak area measurement. This requirement is waived if a ball-and-disc or an electronic integrator is employed.

5.3 *Micro Syringe*, 10- μ L capacity, for sample introduction.

5.4 *Volumetric Flasks*.

5.5 *Volumetric Pipets*.

5.6 *Tubing*, copper, aluminum, or stainless steel.

5.7 *Analytical Balance*.

6. Reagents and Materials

6.1 *Liquid Phase for Column*—See Note 7.

6.2 *Solid Support*—See Note 7. If an open tubular column is used, a solid support is not necessary.

TABLE 2 Composition of Standard Blends, Volume Percent

Blend No.	<i>n</i> C ₁₀	Internal Standard	Benzene	Toluene	Ethyl Benzene
1	5	10.0	15.0	15.0	15.0
2	...	10.0	5.0	5.0	25.0
3	...	10.0	25.0	25.0	5.0
4	1	...	0.5	0.5	0.5

6.3 *Solvent for Liquid Phase*—Methylene chloride has been found satisfactory. (Warning—See Note 8.)

NOTE 8: Warning—Methylene chloride is harmful if inhaled, high concentrations may cause unconsciousness or death; see Annex A1.6.

6.4 *Carrier Gas*—See Note 7.

NOTE 9: Warning—Compressed gas under high pressure; see Annex A1.7.

6.5 *n*-Heptane or Isooctane, 99 mol % minimum containing less than 0.05 of aromatic material (Warning—See Notes 10 and 11.)

NOTE 10: Warning—*n*-Heptane is flammable, harmful if inhaled; see Annex A1.8.

NOTE 11: Warning—Isooctane is extremely flammable, harmful if inhaled; see Annex A1.9.

TABLE 3 Typical Columns and Conditions

	1	2	3	4	5
Column:					
Liquid phase, material	DEGS	PEG-400	TCEPE	TCEPE	TCEP
Weight percent	20	...	10	15	20
Solid support, material	Chrom. P	...	Chrom. P	Chrom. P	Chrom. P
Mesh size	60 to 80	...	60 to 80	60 to 80 (S)	60 to 80
Treatment	None	...	AW	AW	AW
Length, m	2.4	30.5	4.6	4.9	6.1
Inside diameter, mm	25.6	0.25	1.6	1.6	4.6
Temperature:					
Sample inlet system, °C	266	150	250	300	190
Detector, °C	102	86	175	260	215
Column:					
Isothermal, °C	102	86	100	100	...
Programmed, °C/min	...	...	...	...	4
Initial temperature, °C	...	...	...	...	50
Constant finally, min	...	...	...	...	5
Final temperature, °C	...	...	...	...	150
Carrier gas, material	helium	nitrogen	nitrogen	helium	helium
Flow rate, cm ³ /min	100	2.5	2.0	40	75
Detector, type	T.C.	F.I.	F.I.	F.I.	T.C.
Voltage (or mA)	12 V	...	...	...	200 mA
Recorder range, mV	1	10	5	1	1
Sample size, µL	5	2	5	1.5	1.5
Split, ratio	...	250:1	100:1	...	...
Speed, cm/h	1.0	1.0	1.0	1.0	1.0

Abbreviations:

DEGS—Diethylene glycol succinate
 PEG—Polyethylene glycol
 TCEPE—Tetracyano ethylated pentaerythritol
 TCEP—1,2,3 Tris (2 cyano ethoxy) propane
 Chrom. P—Chromosorb P
 AW—Acid Washed
 T.C.—Thermal conductivity
 F.I.—Flame ionization
 (S)—Sieved

6.6 Benzene, Toluene, and Ethylbenzene, 99.5 mol % minimum. (Warning—See Notes 12 and 13.)

NOTE 12: Warning—Benzene is a poison, carcinogen; see Annex A1.2.

NOTE 13: Warning—Toluene and ethylbenzene are flammable, vapor harmful; see Annexes A1.3 and A1.4, respectively.

6.7 *n*-Decane and Either *n*-Undecane or *n*-Dodecane—Either the C₁₁ or C₁₂ normal paraffin can be used as an internal standard according to the requirements in 5.1.3. (**Warning—**See Note 14.)

NOTE 14: Warning—Combustible, vapor harmful; see Annex A1.5.

7. Sample Handling

7.1 Due to the volatility of these materials and the tendency toward change in composition, store all samples and standard blends at a temperature of 45 to 60°F (7 to 15°C). Do not open the sample or standard container at liquid temperatures above 60°F (15°C). Transfers from the container to the chromatograph should be made only when the liquid in the container is within the temperature range indicated.

8. Calibration

8.1 Preparation of Standards—Prepare three standard blends indicated as Blend 1, 2, and 3 in Table 2. To do this, pipet the exact volume of each hydrocarbon indicated into a 100-mL flask. Fill to the 100-mL mark with one of the solvents listed in 6.5 and mix by inverting several times.

8.2 Select the instrument conditions and sample size so as to give the necessary sensitivity and resolution. Inject each

standard blend into the chromatograph at least twice, using the same volume of sample and instrument conditions. If a sensitivity of less than maximum is needed to measure the *n*-decane, internal standard and aromatic peaks, change the attenuation so that the peaks are measured at not less than 25 % of full scale.

8.3 On Blend 1 measure the height of the *n*-decane peak and the height of the valley between it and benzene. Express both on a common attenuation basis. Divide the height of the valley above the base line by the total height of the *n*-decane peak. This value must not exceed 0.5 for the column and conditions to be used.

8.4 Measure the area of all aromatic and internal standard peaks by any of the following means: triangulation, planimeter, paper cut-out, ball-and-disc integrator, or electronic integrator.

NOTE 15—Because the C₈ and heavier compounds may not be completely resolved, peak height times one half the width or retention time must not be used as a means of measuring the area.

8.5 If a different attenuation was used for part of the chromatogram, correct to a constant attenuation basis by multiplying the areas where a lower sensitivity was used by the ratio:

$$S_i/S_a$$

where:

S_i = the attenuation sensitivity used for the internal standard peak, and

S_a = attenuation sensitivity used for the aromatic peak.