

Designation: D7753 - 12

# Standard Test Method for Hydrocarbon Types and Benzene in Light Petroleum Distillates by Gas Chromatography<sup>1</sup>

This standard is issued under the fixed designation D7753; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers and provides for the quantitative determination of total saturates, total olefins, total aromatics and benzene in light petroleum distillates having a final boiling point below 215°C by multidimensional gas chromatography. Each hydrocarbon grouping as well as benzene can be reported in both volume and mass percent.

1.2 This test method is applicable to light petroleum distillates such as oxygenate-free motor gasoline or spark ignition fuels, naphthas and hydrocarbon solvents over the content ranges from 1% (V/V) to 70 % (V/V) total olefins, 1 % (V/V) to 80 % (V/V) total aromatics and 0.2 to 10 % (V/V) benzene. This test method may apply to concentrations outside these ranges, but the precision has not been determined. Interlaboratory testing for precision used full range blending streams, such as FCC, reformates and spark ignition fuel or blended motor gasolines.

1.3 This test method is not intended to determine oxygenated components. Light petroleum distillate products such as motor gasoline may contain oxygenates. Oxygenates such as methyl *tert*-butyl ether (MTBE), *tert*-amyl methyl ether (TAME), ethyl *tert*-butyl ether (ETBE), ethanol and methanol etc. will coelute with specific hydrocarbon groups. If there is any suspicion the sample contains oxygenates, the absence of oxygenates should be confirmed by other standard test methods such as Test Methods D4815, D5599, or D6839 before using this test method.

1.4 This test method is not applicable for the determination of individual hydrocarbon components with the exception of benzene. Test Method D6733 may be used to determine a large number of individual hydrocarbons to complement this test method.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 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. Referenced Documents

- 2.1 ASTM Standards:<sup>2</sup>
- D4815 Test Method for Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and  $C_1$  to  $C_4$  Alcohols in Gasoline by Gas Chromatography
- **D5599** Test Method for Determination of Oxygenates in Gasoline by Gas Chromatography and Oxygen Selective Flame Ionization Detection
- D6733 Test Method for Determination of Individual Components in Spark Ignition Engine Fuels by 50-Metre Capillary High Resolution Gas Chromatography
- D6839 Test Method for Hydrocarbon Types, Oxygenated Compounds and Benzene in Spark Ignition Engine Fuels by Gas Chromatography

## 3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *aromatics*, *n*—mass or volume % of monocyclic aromatics and polycyclic aromatics (for example, naphthalenes), aromatic olefins and  $C_8^+$  cyclodienes compounds.

3.1.2  $C_7^+$  aromatics, *n*—mass or volume % of all other aromatics compounds (see 3.1.1) in sample not including benzene.

3.1.3 *olefins*, *n*—mass or volume % of alkenes, plus cycloalkenes and some di-olefins.

3.1.4 *olefins trap*, *n*—specific column utilized to selectively retain olefins from mixture of olefins and saturates. The trap must have good reversibility to capture and release olefins by changing the temperature.

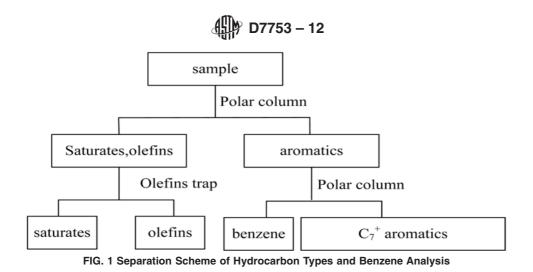
3.2 Acronyms:

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<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04.0L on Gas Chromatography Methods.

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<sup>&</sup>lt;sup>2</sup> 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.



3.2.1 *BCEF- N*,*N*-*bis*( $\alpha$ -*cyanoethyl*) *formamide*—gas chromatography stationary phase.

# 4. Summary of Test Method

4.1 Fig. 1 shows a separation scheme of the various hydrocarbon types and benzene analysis. The instrumental configuration is shown in Fig. 2. The valves are actuated at predetermined times to direct different components to different columns. As the analysis proceeds, different hydrocarbon types and benzene elute and are detected by a flame ionization detector (FID).

4.2 The mass concentration of different hydrocarbon types and benzene are determined by the application of average relative response factors to the areas of the detected peaks followed by normalization to 100%.

4.3 The volume percent concentration of different hydrocarbon types and benzene can be determined by the application of average density factors to the calculated mass concentration of the detected peaks followed by normalization to 100%.

4.4 This test method is not intended to determine compounds that contain oxygenates, such as ethanol, etc. Such oxygenates interfere with the analysis of the hydrocarbons.

4.5 Analysis time of a sample is approximately 15 min.

## 5. Significance and Use

5.1 Knowledge of the olefinic, aromatic, and benzene content is very important in quality specifications of petroleum products, such as spark ignition fuels (gasoline) and hydrocarbon solvents. Fast and accurate determination of hydrocarbon types and benzene of petroleum distillates and products is also important in optimization of process units.

5.2 This test method provides a fast standard procedure for determination of hydrocarbon types and benzene in light oxygenate-free petroleum distillates and products.

#### 6. Interferences

6.1  $C_{12}^{+}$  aliphatic hydrocarbon compounds (not including  $C_{12}$ ) may not be fully separated from benzene in the polar column, thus the determination of aromatics and benzene may be affected.

6.2 Different types of oxygenated compounds in some petroleum products will elute with specific hydrocarbon groups and interfre with the analysis of the hydrocarbons.

6.3 Commercial detergent, antioxidant, antiknock additives and dyes utilized in some petroleum products have been found not to interfere with this test method.

6.4 Dissolved water in samples has been found not to interfere with this test method.

## 7. Apparatus

7.1 The analysis system is comprised of a gas chromatograph with manual or automated sample injection, and specific hardware modifications. These modifications include columns, olefins trap, valves, and temperature controllers.

7.2 *Gas Chromatograph*—capable of temperature programmed operation at specified temperature, equipped with a vaporization inlet that can be a packed column inlet, a flame ionization detector (FID), and necessary flow controllers.

7.3 Sample Introduction System—manual or automatic injector, capable of injecting a 0.1  $\mu$ L volume of sample. Automated injector is recommended.

7.4 Gas Flow or Pressure Controllers—with adequate precision to provide reproducible flow rate of carrier gas to the chromatographic system, hydrogen and air for the flame ionization detector. Control of air pressure for automated valves operation is required.

7.5 *Data Acquisition System*—chromatographic workstation shall meet the following specifications:

7.5.1 Sampling rate of at least 10 points per second.

7.5.2 Capacity for 100 peaks for each analysis.

7.5.3 Normalized areas percent calculation with response factors.

7.5.4 Area summation of peaks that are split or of groups of components that elute at specific retention times.

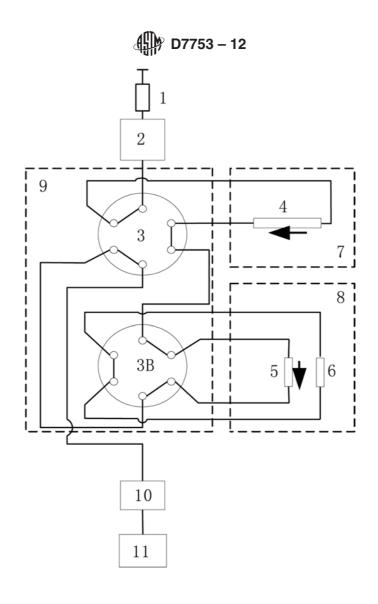
7.5.5 Noise and spike rejection capability.

7.5.6 Manual baseline adjusting function, as required.

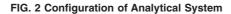
7.6 Valves—column and trap switching, automated rotary valves are recommended.

7.7 Gas Purifiers—to remove moisture and oxygen from carrier gas.

7.8 *Temperature Controllers*—the independent temperature control of the polar column, olefins trap, switching valves and sample connecting lines is required. All of the system components that contact the sample should be heated to a temperature that will prevent condensation of any sample component. Table 1 lists the system components and approximate operating



Note—Legend: 1—injector 2—vaporization room 3, 3B—valve 1 4—polar column 5—olefins trap 6—balance column 7—polar column oven 1 8—olefins trap oven 9—valves oven 10—FID 11—data processing unit



Component	Typical Operating Temperature, °C	Heating Mode
Polar Column Olefin Trap	100~120 125~210	isothermal temperature programmed ~40°C/min
Switching Valves Sample Lines	100~140 100~140	isothermal isothermal

temperatures. Some of the components operate isothermally, while others require temperature programming. Temperature control may be by any means that will meet the requirements listed in Table 1.

# 8. Reagents and Materials

8.1 Gases:

8.1.1 *Carrier Gas*—Nitrogen or Helium. ILS precision of this test method was obtained using nitrogen as the carrier gas. Better than 99.999% pure. (**Warning**—Compressed gases under high pressure.) Gas purifiers may be used to attain the required purity or to ensure a stable signal baseline.

8.1.2 *Hydrogen*—Better than 99.999% pure. (**Warning**— Extremely flammable gas under high pressure.) Gas purifiers may be used to attain the required purity or to ensure a stable signal baseline.