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Standard Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption¹

This standard is issued under the fixed designation D 1319; 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 test method has been approved by the sponsoring committees and accepted by the cooperating societies in accordance with established procedures.

This standard has been approved for use by agencies of the Department of Defense to replace Method 3703 of Federal Test Method Standard No. 791b. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the determination of hydrocarbon types over the concentration ranges from 5 to 99 volume % aromatics, 0.3 to 55 volume % olefins, and 1 to 95 volume % saturates in petroleum fractions that distill below 600°F (315°C). This test method may apply to concentrations outside these ranges, but the precision has not been determined.

1.2 A more accurate procedure for chromatographically determining the aromatic content of olefin-free gasoline or gasoline fractions appears in Test Method D 936.

1.3 This test method is intended for use with full boiling range products. Cooperative data have established that the precision statement does not apply to narrow boiling petroleum fractions near the 600°F (315°C) limit. Such samples are not eluted properly, and results are erratic.

1.4 The applicability of this test method to products derived from fossil fuels other than petroleum, such as coal, shale, or tar sands, has not been determined, and the precision statement may or may not apply to such products.

1.5 The precision statement for this test method has been determined with unleaded fuels that do not contain oxygenates. It may or may not apply to automotive gasolines containing lead antiknock mixtures or oxygenated compounds, or both.

NOTE 1—For the determination of olefins below 0.3 volume %, other methods are available, such as Test Method D 2710.

1.6 *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, 8, 9, 10, 11, 12, and Annex A1.

2. Referenced Documents

2.1 ASTM Standards:

- D 770 Specification for Isopropyl Alcohol²
- D 936 Test Method for Aromatic Hydrocarbons in Olefin-Free Gasolines by Silica Gel Adsorption³
- D 1193 Specification for Reagent Water⁴
- D 1655 Specification for Aviation Turbine Fuels³
- D 2001 Test Method for Depentanization of Gasoline and Naphthas⁵
- D 2427 Test Method for Determination of C₂ Through C₅ Hydrocarbons in Gasolines by Gas Chromatography⁵
- D 2710 Test Method for Bromine Index of Petroleum Hydrocarbons by Electrometric Titration⁵
- D 3663 Test Method for Surface Area of Catalysts⁶
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁶
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁷

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

- 3.1.1 *saturates*—the volume percent of alkanes plus cycloalkanes.
- 3.1.2 *olefins*—the volume percent of alkenes, plus cycloalkenes, plus some dienes.
- 3.1.3 *aromatics*—the volume percent of monocyclic and polycyclic aromatics, plus aromatic olefins, plus some dienes, plus compounds containing sulfur, nitrogen, and oxygen atoms.

4. Summary of Test Method

- 4.1 Approximately 0.75 mL of sample is introduced into a

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 Analyses.

In the IP, this test method is under the jurisdiction of the Standardization Committee.

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² Annual Book of ASTM Standards, Vol 06.03.

³ Annual Book of ASTM Standards, Vol 05.01.

⁴ Annual Book of ASTM Standards, Vol 11.01.

⁵ Annual Book of ASTM Standards, Vol 05.02.

⁶ Annual Book of ASTM Standards, Vol 05.03.

⁷ Annual Book of ASTM Standards, Vol 11.02.

Documents

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special glass adsorption column packed with activated silica gel. A small layer of the silica gel contains a mixture of fluorescent dyes. When all the sample has been adsorbed on the gel, alcohol is added to desorb the sample and force it down the column. The hydrocarbons are separated according to their adsorption affinities into aromatics (including some diolefins and aromatics with olefinic side chains plus any sulfur, nitrogen, and oxygen compounds), olefins, and saturates. The fluorescent dyes are also separated selectively, with the hydrocarbon types, and make the boundaries of the aromatic, olefin, and saturate zones visible under ultraviolet light. The volume percentage of each hydrocarbon type is calculated from the length of each zone in the column.

5. Significance and Use

5.1 This test method provides information on the total volume percent of saturates, olefins, and aromatics in petroleum fractions. This information is important in characterizing the quality of petroleum fractions as gasoline blending components and as feeds to catalytic reforming processes. This information is also important in characterizing petroleum fractions and products from catalytic reforming and from thermal and catalytic cracking as blending components for motor and aviation fuels. This information is also important as a measure of the quality of fuels, such as specified in Specification D 1655.

6. Interferences

6.1 Dark samples sometimes require distillation to remove colored components that can interfere in reading the chromatographic zones.

6.2 Errors in the direction of high saturate values and low aromatic and low olefin values can result if the sample contains significant amounts of C_5 and lighter hydrocarbons. Such samples are to be depentanized by Test Method D 2001.

7. Apparatus

7.1 *Adsorption Columns*, with precision bore ("true bore" IP designation) tubing as shown on the right in Fig. 1, made of glass and consisting of a charger section with a capillary neck, a separator section, and an analyzer section; or with standard wall tubing, as shown on the left in Fig. 1.

7.1.1 The inner diameter of the analyzer section for the precision bore tubing shall be 1.60 to 1.65 mm and an approximately 100-mm thread of mercury shall not vary by more than 0.3 mm in any part of the analyzer section. In glass-sealing the various sections to each other, long-taper connections shall be made instead of shouldered connections. Support the silica gel by means of a 6-mm diameter portion of phosphor-bronze or equivalent 200-mesh wire gauze (BS 410:1943) located between the ball and socket of the 12/2 spherical joint and covering the analyzer outlet. The column tip attached to the 12/2 socket shall have a 2-mm internal diameter. Clamp the ball and socket together and ensure that the tip does not tend to slide from a position in a direct line with the analyzer section during the packing and subsequent use of the column.

7.1.2 For convenience, adsorption columns with standard wall tubing, as shown on the left in Fig. 1 can be used. When

TABLE 1 Sieve Acceptance Tests of Silica Gel

Sieve Size ^A		Particles Passing Through Sieve, Weight %
Sieve Number ^A	μm	
80	180	100
100	150	95 min
200	75	15 max

^A Detailed requirements for these sieves are given in Specification E 11, and BS410: 1943.

using standard wall tubing for the analyzer section, it is necessary to select tubing of uniform bore and to provide a leakproof connection between the separator and the analyzer sections. Calibrations of standard wall tubing would be impractical; however, any variations of 0.5 mm or greater, as measured by ordinary calipers, in the outside diameter along the tube can be taken as an indication of irregularities in the inner diameter and such tubing should not be used. Draw out one end of the tubing selected for the analyzer section to a fine capillary to retain the gel. Connect the other end of the analyzer section to the separator section with a 30-mm length of vinyl tubing, making certain that the two glass sections touch. To ensure a leakproof glass-to-vinyl seal with the analyzer section, it is necessary to heat the upper end of the analyzer section until it is just hot enough to melt the vinyl, then insert the upper end of the analyzer section into the vinyl sleeve. Alternatively, this seal can be made by securing the vinyl sleeve to the analyzer section by wrapping it tightly with soft wire.

7.2 *Zone-Measuring Device*—The zones may be marked with a glass-writing pencil and the distances measured with a meter rule, with the analyzer section lying horizontally. Alternatively, the meter rule may be fastened adjacent to the column. In this case, it is convenient to have each rule fitted with four movable metal index clips (Fig. 1) for marking zone boundaries and measuring the length of each zone.

7.3 *Ultraviolet Light Source*, with radiation predominantly at 3650 Å (365 nm) is required. A convenient arrangement consists of one or two 36-in. (915-mm) or 48-in. (1220-mm) units mounted vertically along the apparatus. Adjust to give the best fluorescence.

7.4 *Electric Vibrator*, for vibrating individual columns or the frame supporting multiple columns.

7.5 *Hypodermic Syringe*, 1-mL, graduated to 0.01 or 0.02 mL, with needle 4 in. (102 mm) in length. Needles of No. 18, 20, or 22-gage are convenient to use.

8. Reagents and Materials

8.1 *Silica Gel*, 100 to 200 mesh,⁸ manufactured to ensure minimum olefin polymerization and to conform to the sieve test requirements shown in Table 1. It may be necessary to screen-test the gel to make certain that the gel size specification is met and that the gel is homogeneous throughout the container. The surface area of the gel, determined by Test Method D 3663, shall be between 430 and 600 m²/g and have a pH between 5.5 and 7. Determine the pH of the silica gel as follows: Calibrate a pH meter with standard pH 4 and

⁸ Available from W. R. Grace and Co., Davison Chemical Division, Baltimore, MD 21203 by specifying Code 923.