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Designation: 601

Standard Test Method for
Determination of Light Hydrocarbons in Stabilized Crude Oils by Gas Chromatography[1,2]

This standard is issued under the fixed designation D7900; 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.

ε1 NOTE—Subsection 1.1 was revised editorially in November 2018.

1. Scope*

1.1 This test method specifies a method to determine the boiling range distribution of hydrocarbons in stabilized crude oil up to and including n-nonane. A stabilized crude oil is defined as having a Reid Vapor Pressure equivalent to or less than 82.7 kPa. The results of this test method can be combined with those from Test Method D7169 and IP 545 to give a full boiling point distribution of a crude oil (see Appendix X3).

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information purposes only.

1.3 *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.*

1.4 *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:*[3]

D323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D5134 Test Method for Detailed Analysis of Petroleum Naphthas through n-Nonane by Capillary Gas Chromatography
D6729 Test Method for Determination of Individual Components in Spark Ignition Engine Fuels by 100 Metre Capillary High Resolution Gas Chromatography
D6730 Test Method for Determination of Individual Components in Spark Ignition Engine Fuels by 100–Metre Capillary (with Precolumn) High-Resolution Gas Chromatography
D6733 Test Method for Determination of Individual Components in Spark Ignition Engine Fuels by 50-Metre Capillary High Resolution Gas Chromatography
D7169 Test Method for Boiling Point Distribution of Samples with Residues Such as Crude Oils and Atmospheric and Vacuum Residues by High Temperature Gas Chromatography
E355 Practice for Gas Chromatography Terms and Relationships

2.2 *Energy Institute Standards:*[4]
IP 545 Crude Petroleum and Petroleum Products— Determination of Boiling Range Distribution of Crude Oil
IP 475 Manual Sampling
IP 476 Automatic Pipeline Sampling

2.3 *ISO Standard:*[5]
ISO 4259 Petroleum Products—Determination and Application of Precision Data in Relation to Methods of Test

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[1] 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.

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[2] This standard has been developed through the cooperative effort between ASTM and the Energy Institute, London. The IP and ASTM logos imply that the ASTM and IP standards are technically equivalent, but their use does not imply that both standards are editorially identical.

[3] 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.

[4] Information on Energy Institute Standards can be obtained from the Energy Institute at www.energyinst.org.

[5] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

TABLE 1 Typical Chromatographic Conditions

	Pre-column A	Pre-column B	Analytical	Accelerated Analytical
Column Length—metres	1.0 m	0.075 m	50 or 100 m	40 m
Column Internal Diameter—mm	2 mm	2.5 mm	0.25 mm	0.10 mm
Phase Loading	5 %	10 %		
Film Thickness			0.5 um	
Injection Volume			0.1 µL	0.1 µL
Injector Split Ratio			100 : 1	600 : 1
Injector Temperature	300 °C	100 °C		
Pre-column Temperature	200 °C	100 °C		
Injector Prog. Rate °C/min		50 °C/min		
Final Injector Temperature		300 °C		
Initial Oven Temperature			35 °C	35 °C
Hold Time			30 min	2.6 min
Oven Program Rate °C/min			2 °C/min	50 °C/min → 45 °C (hold time 3 min) 5 °C/min → 60 °C (hold time 3 min) 9.5 °C/min →
Final Oven Temperature			200 °C (hold time 20 min)	200 °C (hold time 1 min)
Flame Ionization Detector			300 °C	300 °C

3. Terminology

3.1 *Definitions*—This test method makes reference to many common gas chromatographic procedures, terms, and relationships. Detailed definitions can be found in Practice E355.

4. Summary of Test Method

4.1 An amount of internal standard is quantitatively added to an aliquot of the stabilized crude oil. A portion of this mixture is injected into a pre-column in series via a splitter with a capillary analytical column. When the n-nonane has quantitatively passed to the analytical column, the pre-column is back-flushed to vent the higher boiling components. The individual components are identified by comparison with reference chromatograms and a database of hydrocarbon compounds (see Appendix X1). The boiling point distribution up to and including n-nonane (n-C9) is calculated.

5. Significance and Use

5.1 Knowledge of the boiling point distribution of stabilized crude oils is important for the marketing, scheduling, and processing of crude oil in the petroleum industry. Test Method D7169 and IP 545 purport to give such a distribution in crude oils, but are susceptible to significant errors in the light ends portion of the distribution as well as in the mass recovery of the whole crude oil due to the interference imposed by the diluent solvent. This test method allows for more accurate determination of the front end of the boiling point distribution curve, in addition to providing important C1 to C9 (nonane) component level information, and more accurate mass recovery at C9 (nonane).

6. Apparatus

6.1 *Gas Chromatograph,* with the operational characteristics given in Table 1.

6.2 *Inlet*—A temperature programmable vaporizing (PVT) or split/splitless inlet.

6.2.1 *Carrier Gas Pneumatic Control*—Constant carrier gas pressure or flow control is required.

6.3 *Column*—A fused silica-bonded polydimethylsiloxane coated capillary column and pre-column are employed. See Table 1 for suggested columns. The analytical column shall elute hydrocarbons in a boiling point order. The eluate from the injector passes through the pre-column before eluting onto the analytical column.

6.4 *Data System*—A computer-based chromatography data system capable of accurately and repeatedly measuring the retention time and areas of eluting peaks. The system shall be able to acquire data at a rate adequate to accurately measure 10 to 20 points around an individual peak. For the accelerated methods (see Table 1), a sampling rate of at least 20 Hz is recommended.

6.5 *Sample Introduction*—Sample introduction by means of an automatic injection is highly recommended.

6.6 *Flame Ionization Detector (FID),* with sufficient sensitivity to detect 0.01 % mass n-heptane with a signal to noise of greater than five. When operating at this sensitivity level, detector stability shall be such that a baseline drift of not more than 1 % per hour is obtained. The detector shall be connected to the column so as to avoid any cold spots. The detector shall be capable of operating at a temperature equivalent to the maximum column temperature used.

6.7 *Pre-Column Configurations:*

6.7.1 *Heated Valve Switching Box Configuration*—For the isothermal 1 m pre-column, a heated valve box is needed with its own temperature control. The box will contain an automated six-port valve, which is used to back-flush the pre-column. The six-port valve should be made out of material that will not be corroded by the sample (some crude oils contain high amounts of sulfur components). The valve shall be situated in a heated isothermal oven and be attached to the injector, pre-column, splitter, analytical column, and the detector without any cold spots. An example configuration is given in Fig. X2.1 in Appendix X2. Alternatively, a Dean Switch type back-flush of the petroleum may also be employed in place of a rotary valve.

6.7.2 *Injection Port Back-Flush Configuration*—A temperature programmable injection port capable of containing a

7.5 cm pre-column, and this injection port must be equipped with a back-flush option. This injector can be connected directly to the capillary column (Fig. X2.2, Appendix X2) or via a splitter (Fig. X2.3, Appendix X2).

6.8 *Analytical Balance,* capable of weighing with an accuracy of 0.1 mg.

7. Reagents and Materials

7.1 *Gas Chromatograph Gases*—All of the following gases shall have a purity of 99.995 % (V∕V) or greater. (**Warning**—Gases are compressed. Some are flammable, and all gases are under high pressure.)

NOTE 1—These specifications can be obtained by proper use of filtering devices and meeting the FID specifications in 6.6.

7.1.1 *Carrier Gas*—Helium or hydrogen is required. Any oxygen present shall be removed, for example, by a suitable chemical filter. If hydrogen is employed as a carrier gas, the user is advised to follow all manufacturer's safety guidelines for its use. (**Warning**—Hydrogen is an extremely flammable gas under high pressure.)

7.1.2 *Detector Combustion Gases,* Air, Hydrogen, and Make-up Gas (Helium or Nitrogen). (**Warning**—Hydrogen is an extremely flammable gas under high pressure.) (**Warning**—Compressed air is a gas under high pressure and supports combustion.)

7.2 *Internal Standard*—The internal standard shall have baseline resolution from any adjacent eluting peaks. Hexene-1 or 3,3–dimethylbutene-1 (99 % pure) have been found to be suitable.

7.3 *Valve Timing Mixture/Splitter Linearity Mix*—A quantitative mixture of approximately 1 % mass of each normal alkane from pentane to decane in hexadecane (99+ % purity). Accurately record the mass (g) of each normal alkane as well as the hexadecane solvent and calculate the actual mass percent of each alkane in the mixture.

7.4 *Viscosity Agent,* Carbon disulfide, 99+ % pure, (**Warning**—Extremely flammable and toxic liquid) is used as a viscosity reduction agent in the preparation of samples.

8. Sampling

8.1 Samples to be analyzed by this test method must be obtained using the procedures outlined in Practice D4057 or Practice D4177 (IP 475 and IP 476, respectively).

8.2 The test specimen to be analyzed must be homogeneous and free of dust or undissolved material.

9. Preparation of Apparatus

9.1 *Chromatograph*—Place in service according to manufacturer's instructions. Typical operating conditions are given in Table 1.

9.2 *Column Preparation*—Condition analytical columns in accordance with manufacturer's instructions.

9.3 *System Performance Specification:*
9.3.1 *Skewness*—Determine the skew of the n-hexane peak by measuring the width of the leading part of the peak at 5 %
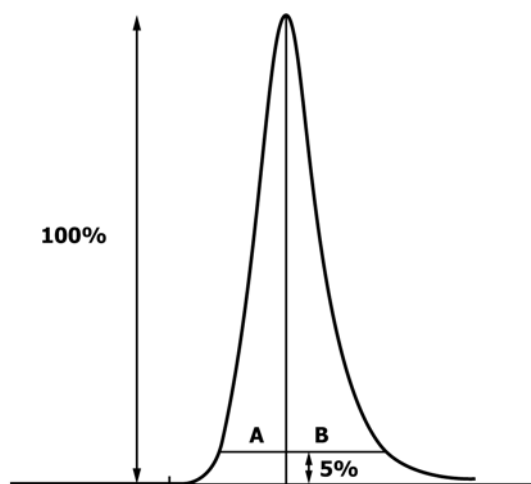

FIG. 1 Calculation of Peak Skewness (see 9.3.1)

peak height (A) and the width of the following part of the peak at 5 % peak height (B). The ratio (B)/(A) shall be not less than 1 or more than 4 (see Fig. 1).

9.3.2 *Column Resolution*—Determine the resolution between the internal standard and the nearest n-paraffin peak.

$$R = 2 \times (t2 - t1) / 1.699(w1 + w2) \quad (1)$$

where:

R = the column resolution,
$t1$ = the retention time of the first peak (peak 1),
$t2$ = the retention time of the second peak (peak 2),
$w1$ = the peak width at half height of peak 1, and
$w2$ = the peak width at half height of peak 2.

For example, if Hexene-1 is used as the internal standard, the resolution is determined between Hexene-1 and n-hexane. The resolution shall be at least 2.0.

9.3.3 *Detector Response Factor Calculations*—Calculate the flame ionization detector response factor relative to methane, which is considered to have a response factor of unity (1), for each hydrocarbon group type of a particular carbon number using Eq 2.

$$RRf = \frac{[(C_{aw} \times C_n) + (H_{aw} \times H_n)] \times 0.7487}{(C_{aw} \times C_n)} \quad (2)$$

where:

RRf = relative response factor for a hydrocarbon type group of a particular carbon number,
C_{aw} = atomic mass of carbon, 12.011,
C_n = number of carbon atoms in the hydrocarbon type group, of a particular carbon number,
H_{aw} = atomic mass of hydrogen, 1.008,
H_n = number of hydrogen atoms in the hydrocarbon type group of a particular carbon number, and
0.7487 = factor to normalize the result to a methane response of unity, (1).

9.3.4 *Determination of Back-Flush Time*—With the pre-column and analytical column in series, inject an aliquot of the pre-column switch test mixture (7.3) and determine the ratio of the alkanes.