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An American National Standard

Standard Test Methods for Comparison of Waterborne Petroleum Oils by Gas Chromatography[1]

This standard is issued under the fixed designation D 3328; 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 comparison of petroleum oils recovered from water or beaches with oils from suspect sources by means of gas chromatography **(1, 2, 3)**.[2] Such oils include distillate fuel, lubricating oil, and crude oil. The test method described is for capillary column analyses using either single detection (flame ionization) or dual detection (flame ionization and flame photometric) for sulfur containing species.

1.2 This test method provides high resolution for critical examination of fine structure that is resistant to weathering. The flame-photometric detection for sulfur components is an adjunct, not a substitute, for flame-ionization detection in the identification of waterborne petroleum oils **(4-12)**. For this reason, flame photometric detection is optional.

1.3 *This standard does not purport to address 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:*

D 1129 Terminology Relating to Water[3]

D 1193 Specification for Reagent Water[3]

D 2549 Test Method for Separation of Representative Aromatics and Nonaromatics Fractions of High-Boiling Oils by Elution Chromatography[4]

D 3325 Practice for Preservation of Waterborne Oil Samples[5]

D 3326 Practices for Preparation of Samples for Identification of Waterborne Oils[5]

D 3415 Practice for Identification of Waterborne Oils[5]

D 4489 Practices for Sampling of Waterborne Oils[5]

D 5739 Practice for Oil Spill Source Identification by Gas Chromotography and Positive Ion Electron Impact Low Resolution Mass Spectrometry[5]

E 355 Practice for Gas Chromatography Terms and Relationships[6]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Practice D 3415, Terminology D 1129, and Practice E 355.

4. Significance and Use

4.1 Identification of a recovered oil is determined by comparison with known oils, selected because of their possible relationship to the particular recovered oil. The known oils are collected from suspected sources. Samples of such known oils *must* be collected and submitted along with the unknown for analysis. At present, identification of the source of an unknown oil by itself cannot be made (for example, from a library of known oils).

4.2 The use of a flame-photometric detector in addition to the flame-ionization detector provides a second, independent profile of the same oil, that is, significantly more information is available from a single analysis with dual detection.

4.3 Many close similarities (within uncertainties of sampling and analysis) will be needed to establish identity beyond a reasonable doubt. The analyses described will distinguish many, but not all samples. For cases in which this method does not clearly identify a pair of samples, and for important cases where additional comparisons are needed to strengthen conclusions, other analyses will be required (refer to Practice D 3415). In particular, Practice D 5739 is useful for such cases.

5. Interferences

5.1 Compounds that have the same retention time as petroleum hydrocarbons will interfere in the comparison of the unknown with known oils. This is particularly true if animal fat or vegetable oil, naturally occurring hydrocarbons, or spill-treatment chemicals are present in relatively large amounts. Independent analysis, for example, infrared spectroscopy, will

[1] These test methods are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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[2] The boldface numbers in parentheses refer to the references at the end of these test methods.

[3] *Annual Book of ASTM Standards,* Vol 11.01.

[4] *Annual Book of ASTM Standards,* Vol 05.01.

[5] *Annual Book of ASTM Standards,* Vol 11.02.

[6] *Annual Book of ASTM Standards,* Vol 14.02.

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

establish the presence of these contaminants if their presence is suspected. Animal or vegetable oils can be removed effectively by Test Method D 2549 or by Practices D 3326 (Method D).

NOTE 1—Test Method D 2549 will also remove the aromatic fraction.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society. [7]

6.2 Unless otherwise indicated references to water shall be understood to mean reagent water conforming to Specification D 1193, Type II.

6.3 *Air*—For use with the flame-ionization and flame-photometric detectors; may be obtained using a laboratory pure air generator, or from a zero grade tank supply.

6.4 *Carrier Gas*— High-purity grade helium is used as carrier gas.

6.5 *Cyclohexane*— High-purity (HPLC-grade). For sample preparation and for use in reference standards.

6.6 *Hydrogen*—For use with the flame-ionization and flame-photometric detectors; may be obtained using a hydrogen generator, or from a prepurified grade tank supply.

6.7 *Methylene Chloride*—For use in reference standards and glassware cleaning.

6.8 *Normal Alkane Standards*—Normal alkanes, decane through hexatriacontane, for use as reference compounds.

6.9 *Thiophene*—For use in optimization of flame-photometric detector.

7. Reference Standards

7.1 *Normal Paraffinic Hydrocarbons*—Prepared mixtures of approximately decane to hexatriacontane, or selected individual normal paraffins, are run under normal analysis conditions to determine retention times of compounds.

7.2 *Resolution Mixture*—Equal mixtures of *n*-heptadecane, *n*-octadecane, pristane and phytane in solution. See Annex A1 for details (A1.2.1).

8. Sampling

8.1 Collect a representative sample in accordance with Practice D 4489.

8.2 If the sample is not to be analyzed within 1 week, it should be preserved in accordance with Practice D 3325 because of the possibility of bacterial decomposition of normal paraffins in the sample.

8.3 The sample should be prepared for analysis in accordance with Practices D 3326, because of the great variety of materials and circumstances associated with collecting petroleum oils from the environment. For heavier oils, a procedure to deasphalt the oil may be necessary.

[7] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

9. Summary of Test Method

9.1 This test method uses a gas chromatographic capillary column system for the separation of petroleum hydrocarbons. The effluent of the column may be detected with a flame-ionization detector, or it may be split (1 + 2) between a flame ionization and a flame-photometric detector. The flame photometric detector is equipped with a narrow bandpass interference filter for spectral isolation of the sulfur emission at 394 nm. The relative peak size of each component (as indicated by retention time) of recovered oil is compared visually with the relative peak size of each component (of like retention time) of the suspected source.

NOTE 2—This duel detector method is based on the early work done by Kahn **(13)**, Garza **(4)**, and Adlard **(7)**.

9.2 In this test method, elution of characteristic hydrocarbons occurs generally in order of increasing boiling point.

10. Apparatus

10.1 *Chromatographic Column*—Fused silica capillary column with bonded phase SE-30 or equivalent, 30 m by 0.32 mm inside diameter (0.1 µm film thickness).

NOTE 3—Other columns, providing equivalent or better resolution may be substituted (see Annex A1), but the analysis time will be increased with longer columns.

10.2 *Gas Chromatograph*—A commercial or custom designed gas chromatograph with heated injection and detector zones and a column oven capable of being programmed from 75°C to at least 325°C for heavier oils (higher boiling than gasolines, jet fuels, etc.).

10.2.1 For light distillate fuels, the chromatograph must be capable of programming from 50°C and also be capable of maintaining isothermal control at 50°C.

10.2.2 *Carrier Gas Pressure Regulator* is substituted pressure regulator for the mass flow controllers to give more precise rates in the low flow ranges (1 to 5 mL/mm).

10.2.3 *Injection Port*— The use of glass injector inserts that can be replaced or cleaned frequently, or both, will prolong the useful life of the column **(3)**.

10.2.4 *Detectors*—A hydrogen-flame ionization detector is always used for analyses. A flame-photometric detector with a 394 nm bandpass filter is used for dual detection **(9, 10, 11, 12)**.

10.2.5 *Carrier Gas Makeup* is required at the effluent of the column with a temperature independent mass flow controller.

10.2.6 *Effluent Splitter*—An effluent splitter with a split ratio of 1 + 2 (FID/FPD) is required for dual detection.

10.2.7 *Bleeder for Reference Compound*—A device for in-line bleed of a reference compound (thiophene and cyclohexane) into the carrier flow for detector optimization is required, when using a flame-photometric detector.

10.2.8 *Recorder*, or an integrator or computer data handling system capable of acquiring data at a rate compatible with the high resolution of the capillary column. Alternatively, a strip-chart recorder is required to measure detector response at full-scale range of 1 mV with a response time of 1 s (or less). A second recorder, or dual-pen recorder, is required for dual detection..

10.3 *Syringe*—A microsyringe of 0.5 to 1 µL capacity.

10.4 *Gas Traps*—Any commerically available gas filter traps to be placed in line to remove trace hydrocarbon and water impurities from the helium, hydrogen, nitrogen, and air gas supplies.

10.5 *FPD Linearizer*— Optional accessory to facilitate comparison of FPD chromatograms.

10.6 *Glass Insert*, packed with glass wool (optional).

NOTE 4—For instruments that can use this instrument, splitless injection of an oil in cyclohexane solution simplifies the analysis by eliminating the need to deasphalt most oil samples.

11. Preparation of Chromatograph

11.1 Install the column in the chromatograph, as described in the manufacturer's instructions.

11.2 Shut off the downstream end of the system and pressurize the carrier gas supply to a gage pressure of approximately 15 psi (103 kPa) above the operating pressure. Shut off the cylinder valve and observe the pressure gage. Consider the system tight if no pressure drop is noted in 10 to 15 min. Use a small amount of aqueous soap solution to locate minor leaks. Do not use the soap solution near the ionization detector.

11.3 *Column Conditioning for New Columns*:

NOTE 5—For previously conditioned columns, proceed to 11.3.4.

11.3.1 Disconnect the column at the detector end to avoid deposition of volatiles on the detector(s) during conditioning.

11.3.2 For new columns, follow the manufacturer's instructions for column conditioning.

11.3.3 Adjust the carrier gas flow as indicated in Table 1.

11.3.4 Adjust the hydrogen and air flow, and the air/hydrogen flow ratio to the detector(s), as specified for the instrument being used. Ignite the flame(s) (see 11.4 for optimization).

11.3.5 Adjust the carrier gas flow as indicated in Table 1.

11.3.6 Program the column temperature as indicated in Table 1, and hold at the maximum temperature while monitoring the effluent. If there are no peaks in the chromatogram and there is minimal baseline shift at high temperatures, then the column is ready for use; otherwise, recondition it.

11.3.7 Return the oven temperature to 75°C.

11.3.8 If the column is to be moved or stored, disconnect and seal the ends of the column. When the column is to be reused, even after conditioning, it is always necessary to cycle through the temperature program to remove any accumulated volatiles.

11.4 *Optimization of Detectors*—Adjust hydrogen and air flows to give optimal detector responses for a given signal provided by the reference compound bleeder (10.2.7). Use cyclohexane for FID optimization and thiophene for the FDP optimization.

12. Operating Conditions for Analysis (Notes 6-8)

NOTE 6—One of the problems frequently encountered with the flame photometric detector is "flameout" when large amounts of solvent are injected with the sample. The recommended sample preparation procedure avoids this problem at the same time that it permits the use of small samples. For those who may encounter this problem, a simple modification has been suggested **(8)** which consists of reversing the hydrogen gas and air/oxygen gas inlets to the detector.

NOTE 7—For oil identification under the recommended procedure, air has been found satisfactory for combustion for the FPD, that is, oxygen is not necessary.

NOTE 8—See the manufacturer's manual for maintenance information for the FPD. Present flame photometric units should not be heated above 250°C, unless the photometer is removed from the heated zone by fiber optics.

12.1 Operating conditions are summarized in Table 1; apparatus operated under these conditions should achieve partial resolution of two pairs of normal and isoprenoid hydrocarbons found in many, but not all, crude oils and certain petroleum products. In order of emergence from the column, these are heptadecane and pristane, and octadecane and phytane.

12.1.1 Periodically analyze the resolution mixture to test the column performance, monitor the instrument performance and

TABLE 1 Operating Conditions for Chromatographic Columns (11, 12, 13)

Column	30 m by 0.32 mm ID by 0.1 μm film thickness, fused capillary
Packing	bonded phase SE-30, or equivalent
Carrier gas:	helium
Flow, mL/min:	
Column	1 to 2
Makeup gas	40
Temperature, °C:	
Injection port	250
Column:	
Heavier oils:	
Initial	60 hold 4 min
Final	280 (FID) 250 (FID/FPD) hold 30 min
Lighter oils:	
Initial	40 hold 10 min
Final	280 hold 10 min
Detector	300 (FID) 250 (FID/FPD)
Program Rate[A]	3–8 [A]
Chart speed, in/min (mm/min)	2.5 (10)
Sensitivity, mV	1
Sample size, μL[A]	1.0 (cyclohexane solution)
Effluent split ration (FPD procedures)	1 + 2 (FID/FPD)

[A] The precise rate is dictated by the design of the gas chromatograph.

thermally equilibrate the system (see Annex A1 for details). After system maintenance, new column installation or change of flow conditions, analyze the resolution test mix.

NOTE 9—See for the results expected on a new, properly conditioned capillary column. A properly functioning column should provide more than 1600 analyses, depending on the type of oil analyzed.

13. Component Identification

13.1 In most instances, it is unnecessary to identify individual components when comparing chromatograms of a spill with its source; it is sufficient to note their degree of match. Identification of the usually dominant normal paraffin hydrocarbons is readily achieved by comparing their retention times with those from known *n*-alkane standards.

13.1.1 Identification of peaks other than normal paraffins is not achieved, except in rare cases.

13.1.2 Comparison of peaks with the same retention times in the known and unknown oils is also made with respect to relative peak sizes of adjacent peaks.

13.2 To determine the retention time of normal paraffins, the following procedure is recommended:

13.2.1 With the column at the initial operating temperature, inject 1.0 µL of the known mixture of normal paraffins (10.1).

NOTE 10—This test method, as written, does not require the use of a computer-controlled instrument with an autosampler. Such an instrument may be used. If so, follow the manufacturer's operating instructions.

13.2.2 Turn on the recorder or integrator and mark the injection point on the recorder chart.

13.2.3 Adjust the instrument attenuation so that the maximum peak heights are on scale.

13.2.4 When the temperature program is complete and the baseline has stabilized, cool the oven to the initial temperature.

13.2.5 Measure the retention time in minutes to at least two significant figures for each normal paraffin in the known mixture.

14. Procedure for a Sample

14.1 First, cycle the instrument through its program to test the column and instrument performance (12.1) and thermally equilibrate the oven.

14.2 For the strip-chart recorder, zero the recorder, if necessary, and make appropriate notations at the beginning of the chromatogram (sample name, reference number, date, amplifier attenuations).

14.3 For light distillate oils (such as gasoline, jet fuels, kerosines, and No. 2 fuel oils), inject 1.0 µL of sample directly into the injection port with the column at initial operating temperature. For heavier oils, a deasphalted sample (see Practice D 3326) is required.

14.4 Start the recorder and the temperature program. Mark the injection point on the recorder chart. For computerized instruments, star the run.

14.5 Adjust the attenuation so that the highest peak is retained on scale and constant baselines are achieved after the analysis. Obtain a complete chromatogram at a single attenuation, repeating if necessary until a satisfactory chromatogram is obtained.

14.6 After resetting the initial conditions, another sample may be analyzed. Analyze all spill samples as well as samples of known origin, that is, from potential sources.

14.7 After completion of the analysis, record the following information for each set of chromatograms: column length and diameter; liquid phase and weight percent; support material and mesh size; initial and final column temperatures; programming rate; carrier gas flow rate; detector manifold and injection port temperatures; FPD heater temperature (if used); hydrogen and air flow rates; injection port split ratio and effluent split ratio (if employed); sample size and amplifier ranges. For computerized instruments, the conditions may be saved with the electronic data.

15. Interpretation

15.1 *Basis of Matching*—The matching of oil samples is essentially a profiling technique based on the premise that identical oils give identical chromatograms. Normally, the matching of a spilled oil to a suspect oil can be accomplished by comparison of the chromatograms for each of the oils in a spill case.

15.2 *Chromatogram Features*—The major features of a chromatogram used for comparison are illustrated in Fig. 1 (gas chromatograms of a crude oil).

15.2.1 The FID curve shows a typical separation with the features of a homologous series of normal paraffins, the isoprenoid hydrocarbons pristane and phytane, the unresolved envelope and other resolved peaks. All of these features are used to characterize an oil.

15.2.2 The FPD chromatogram has fewer readily ascribed characteristics; it gives the overall sulfur profile generated by the detector. It is useful not only qualitatively, but semiquantitatively. And FPD chromatogram is not shown.

15.3 *Weathering Effects*:

15.3.1 When an oil is spilled on open water, or a relatively small amount of oil is widely dispersed in an area such as a bilge tank, weathering will progress rapidly. A thin slick on open water may lose significant amounts of its components up to *n*-C_{15} (271°C atmospheric boiling point) within 48 h of being spilled. It is important to be cognizant of the effects of weathering when analyzing spill samples more than a few hours old. It is advisable to compare only those portions of chromatograms boiling above pentadecane in order to minimize the difference resulting from changes due to weathering.

15.3.2 Light distillate fuels cannot survive heavy weathering and have few hydrocarbons above C_{15}. Comparison of the residues of these oils can only be done qualitatively—from about C_8–C_{15}.

15.4 *Comparison of Chromatograms*:

15.4.1 Normally a direct comparison of chromatograms, considering the features enumerated above, will suffice for establishing identity or nonidentity between samples. The comparison involves a peak-for-peak matching, noting differences or similarities in relative peak size. If the chromatograms are the same on the basis of peak-for-peak matching, there is a high degree of probability that the samples are from the same source. A mismatch is obtained when the curves are different. The differences may be due to the presence of one or more components in one sample relative to another or consistent

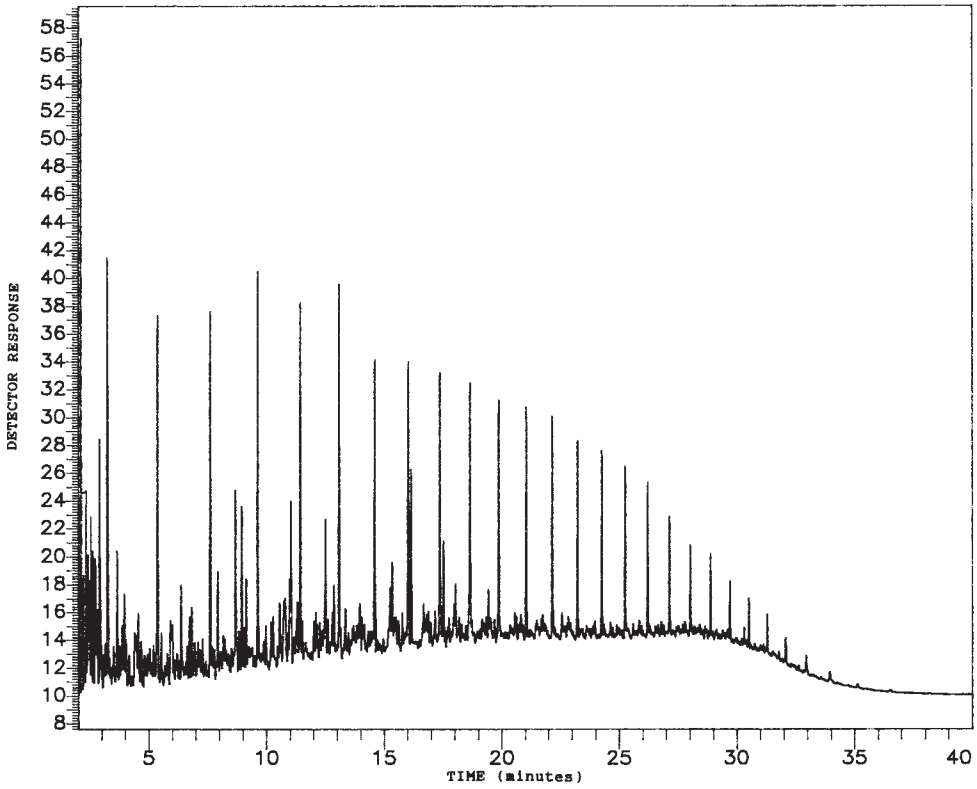

FIG. 1 Representative Chromatogram from Capillary Column

differences in relative intensities of peak responses, or both. Spill samples may contain components such as bilge cleaning detergents, plasticizers, paint vehicles, etc. The presence of one or two components in a spill sample, which are absent in a suspect, is not an absolute indication of nonidentity.

15.4.2 Additional interpretation of capillary chromatographs is based on fine structure, the pattern of many small peaks resolved, or partially resolved, in addition of *n*—alkanes and isoprenoids. This structure, referred to as "grass," is reproducible and reveals differences among oils that are more resistant to weathering.

16. Report

16.1 Based upon the visual comparison of chromatograms, and after considering 8.2, 15.3, and 15.4, report the results of the comparison of the spill sample and the sample of unknown origin as belonging to one of the categories below:

16.1.1 *Match* (M)—Like the sample submitted for comparison, that is, the chromatographic pattern are virtual overlays.

16.1.2 *Probable Match* (PM)—The chromatographic pattern is similar to that of the samples submitted for comparison, except: (*a*) for changes which could be attributed to weathering (specific low molecular weight peak losses), or (*b*) differences attributable to specific contamination.

16.1.3 *Indeterminate* (I)—The chromatographic patten is somewhat similar to that of the sample submitted for comparison, except for certain differences as in 16.1.2 of such magnitude that it is impossible to ascertain whether the unknown is the same oil heavily weathered, or a totally different oil.

16.1.4 *Nonmatch* (NM)—Unlike the samples submitted for comparison.

17. Precision and Bias

17.1 No statement is made about either the precision or bias of this test method since the result merely states whether there is conformance to the criteria for comparison specified in the procedure.

18. Test Sample (Spilled oil)

18.1 For splitless injection, prepare a solution of approximately 10 µL of oil in 1 mL of cyclohexane. An injection volume of 1 µL of this solution is used.

19. Quality Assurance

19.1 If the analysis of the quality control sample described in 5.5 of Practice D 3415 does not meet the criteria for a match, the results of all the comparisons are invalid.

20. Keywords

20.1 gas chromotograph; oil analysis; oil identification; spilled oils; waterborne oils

ANNEX

(Mandatory Information)

A1. COLUMNS

A1.1 Column Performance

A1.1.1 The level of performance of the chromatographic system, in particular the gas chromatographic column, can be quantitated by calculation of the resolution of specific compounds. The term "resolution" is defined in Practice E 355. The resolution values for normal *n*-alkanes C-17 and C-1 from pristane and phytane, respectively, are used in defining column performance for this test method.

A1.2 Procedure

A1.2.1 A resolution mixture is prepared consisting of 150 ng/µL each of normal alkanes *n*-heptadecane (*n*—C17), *n*-octadecane (*n*—C18), pristane and phytane dissolved in cyclohexane. Gentle warming of the solutes may be necessary to carry out their transfer.

A1.2.2 Instrumental conditions, gas flows, and temperature programming are exactly the same as for the analysis of samples (see Section 12). A 1.0 µL injection volume is used for analysis and will give adequate response at normal instrument conditions used for oil samples. The resultant peaks are of the approximate size of the same peaks that will be found for many oil samples.

A1.2.3 The resolution for the peak pairs *n*-C17 and pristane and for *n*-C18 and phytane are determined using Practice E 355.

NOTE A1.1—A faster chart speed of 100 to 200 mm/min will improve the measurement of peak width. The measurement of peak width at half height may be necessary when peak tailing occurs; this measurement should be doubled for use in resolution equations.

A1.3 Performance Standards

A1.3.1 The resolution of components for a well performing column will give resolution values for capillary columns of 80 % or greater for the peak pairs *n*-C17 and pristane and for *n*-C18 and phytane.

A1.3.2 Columns should be replaced and operating conditions thoroughly checked should resolution values become less than 50 % for either peak pair.

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SUMMARY OF CHANGES

This section identifies the location of selection changes to test methods that have been incorporated since the last issue. For the convenience of the user, Committee D19 has highlighted those changes that may impact the use of these test methods. This section may also include descriptions of the changes or reasons for the changes, or both.

(*1*) This test method, formerly Test Method B, has been revised to reflect the widespread acceptance and use of fused silica capillary columns. Former Test Method A, for lower resolution packed column chromatography, has been dropped from this test method.