



Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry¹

This standard is issued under the fixed designation E1618; 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 identification of residues of ignitable liquids in extracts from fire debris samples. Extraction procedures are described in the referenced documents.

1.2 Although this test method is suitable for all samples, it is especially appropriate for extracts that contain high background levels of substrate materials or pyrolysis and combustion products. This test method is also suitable for the identification of single compounds, simple mixtures, or non-petroleum based ignitable liquids.

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

1.4 This practice cannot replace knowledge, skill, or ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgment.

1.5 *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.*

1.6 *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:²

E1386 Practice for Separation of Ignitable Liquid Residues

from Fire Debris Samples by Solvent Extraction

E1388 Practice for Static Headspace Sampling of Vapors from Fire Debris Samples

E1412 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Activated Charcoal

E1413 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration onto an Adsorbent Tube

E2154 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Solid Phase Microextraction (SPME)

E2451 Practice for Preserving Ignitable Liquids and Ignitable Liquid Residue Extracts from Fire Debris Samples

3. Summary of Test Method

3.1 The sample is analyzed with a gas chromatograph (GC) which is interfaced to a mass spectrometer (MS) and a data system (DS) capable of storing and manipulating chromatographic and mass spectral data.

3.2 Post-run data analysis generates extracted ion profiles (mass chromatograms) characteristic of the chemical compound types commonly found in ignitable liquids. Additionally, specific compounds present within a class of ignitable liquid can be identified by their mass spectra and retention times.

3.2.1 The total ion chromatogram (TIC), extracted ion profiles (EIP) for the alkane, alkene, alcohol, aromatic, cycloalkane, ester, ketone, and polynuclear aromatic compound types, or identification of individual compounds, or combination thereof, are evaluated by visual pattern matching against known reference ignitable liquids.

3.2.2 Ignitable liquids can be grouped into one of seven major classifications or one miscellaneous class, as described in this test method.

4. Significance and Use

4.1 The identification of an ignitable liquid residue in samples from a fire scene can support the field investigator's opinion regarding the origin, fuel load, and incendiary nature of the fire.

4.1.1 The identification of an ignitable liquid residue in a fire scene does not necessarily lead to the conclusion that a fire

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² 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.

was incendiary in nature. Further investigation can reveal a legitimate reason for the presence of ignitable liquid residues.

4.1.2 Because of the volatility of ignitable liquids and variations in sampling techniques, the absence of detectable quantities of ignitable liquid residues does not necessarily lead to the conclusion that ignitable liquids were not present at the fire scene.

4.2 Materials normally found in a building, upon exposure to the heat of a fire, will form pyrolysis and combustion products. Extracted ion profiling and identification of specific compounds or classes of compounds described herein can facilitate the identification of an ignitable liquid in the extract by reducing interference by components generated as products of pyrolysis.

5. Apparatus

5.1 *Gas Chromatograph*—A chromatograph capable of using capillary columns and being interfaced to a mass spectrometer.

5.1.1 *Sample Inlet System*—A sample inlet system that can be operated in either split or splitless mode with capillary columns; the inlet system can use on-column technology.

5.1.2 *Column*—A capillary, bonded phase, methylsilicone or phenylmethylsilicone column or equivalent. Any column length or temperature program conditions can be used provided that each component of the test mixture (see 6.4) is adequately separated.

5.1.3 *GC Oven*—A column oven capable of reproducible temperature program operation in the range from 50 to 300°C.

5.2 *Mass Spectrometer*—Capable of acquiring mass spectra from m/z 40 to m/z 400 with unit resolution or better, with continuous data output. Values above m/z 40 can be insufficient to detect or identify some lower molecular weight compounds; for example, methanol, ethanol, acetone.

5.2.1 *Sensitivity*—The system shall be capable of detecting each component of the test mixture (see 6.4) and providing sufficient ion intensity data to identify each component, either by computer library search or by comparison with reference spectra.

5.3 *Data Station*—A computerized data station, capable of storing time sequenced mass spectral data from sample runs.

5.3.1 *Data Handling*—The data system shall be capable of performing, either through its operating system or by user programming, various data handling functions, including input and storage of sample data files, generation of extracted ion profiles, searching data files for selected compounds, and qualitative and semi-quantitative compound analysis.

5.3.2 *Mass Spectral Libraries*—The system shall be capable of retrieving a specified mass spectrum from a data file and comparing it against a library of mass spectra available to the data system. This capability is considered an aid to the analyst, who will use it in conjunction with chromatographic data and known reference materials to identify unknown components.

5.4 *Syringes*:

5.4.1 *For liquid samples*, a syringe capable of introducing a sample size in the range from 0.1 to 10.0 μL .

5.4.2 *For gas samples*, a gas-tight syringe capable of introducing a sample size in the range of 0.5 to 5 mL.

6. Chemicals, Reagents, and Reference Materials

6.1 *Purity of Reagents*—Reagent grade or better chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.³ Other grades can be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Solvent/Diluent*—Carbon disulfide, diethyl ether, pentane, or other solvent that will not interfere with the analysis. It is generally desirable to use a solvent whose volatility greatly exceeds that of the solute to facilitate sample concentration by evaporation, if necessary.

6.2.1 Use of a heavier solvent, such as toluene or tetrachloroethylene, is sometimes necessary when the compounds of interest have low molecular weights.

6.3 *Carrier Gas*—Helium or hydrogen of purity 99.995 % or higher.

6.4 *Test Mixture*—The test mixture shall consist of a minimum of the even-numbered normal alkanes (ranging from *n*-octane through *n*-eicosane), methylbenzene (toluene), 1,4-dimethylbenzene (*p*-xylene), 1-methyl-2-ethylbenzene (*o*-ethyltoluene), 1-methyl-3-ethylbenzene (*m*-ethyltoluene), and 1,2,4-trimethylbenzene (pseudocumene). Additional compounds can be included at the discretion of the analyst. The final test solution is prepared by diluting the above mixture such that the concentration of each component is 0.005 % volume/volume (0.05 $\mu\text{L}/\text{mL}$) in the chosen solvent (see 6.2). A typical chromatogram of the test mixture is shown in Fig. 1.

6.5 *Reference Ignitable Liquids*—Ignitable liquids shall be available for the various ignitable liquids represented in Table 1.

6.5.1 Typically, reference ignitable liquids are diluted 1:1000 in an appropriate solvent. Depending on the column capacity and injection technique, ignitable liquid solutions can be made somewhat more concentrated to ensure detection of minor compounds.

6.5.2 Certified ignitable liquid standards are not necessary. Most reference ignitable liquids can be obtained from commercial and retail sources.

7. Equipment Calibration and Maintenance

7.1 Verify the consistent performance of the chromatographic instrument by using blanks and known concentrations

³ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

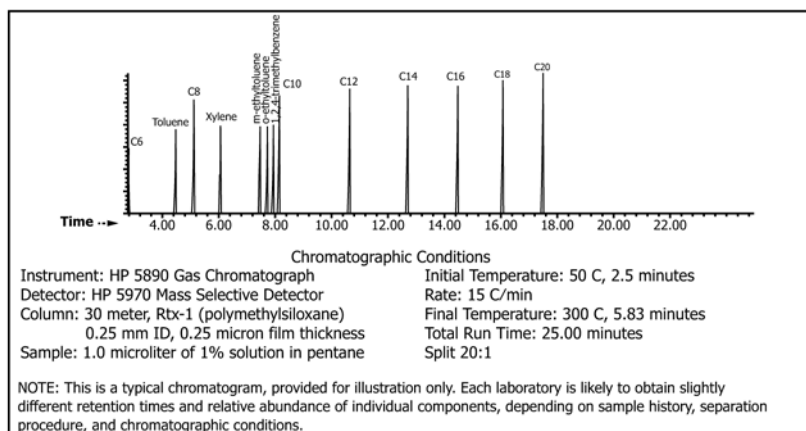


FIG. 1 Test Mixture Containing C₆–C₂₀ Normal Hydrocarbons, Toluene, *p*-Xylene, *o*-Ethyltoluene, *m*-Ethyltoluene, and 1,2,4-Trimethylbenzene

of either prepared test mixture or other known ignitable liquids. Optimize gas flows periodically.

7.2 Tune and calibrate mass spectrometer.

7.2.1 Tune the mass spectrometer using perfluorotributylamine (PFTBA), or another appropriate calibration standard, according to the instrument manufacturer’s specifications, prior to use. This should be done at least every day that the instrument is used or in accordance with manufacturer’s recommendations.

7.2.2 Maintain tuning documentation as a portion of the quality control documentation.

7.3 Cleaning the equipment.

7.3.1 Change septa and clean or replace injector liners on a periodic basis to avoid sample contamination by carryover of residual material from previous sample injections.

8. Sample Handling Procedure

8.1 Only samples of appropriate dilution should be analyzed on a GC-MS system.

8.2 Methods for isolating ignitable liquid residues from fire debris for analysis by this test method are described in Practices E1386, E1388, E1412, E1413, and E2154.

8.3 Because of the volatility of solvents and analytes, care must be taken to ensure that samples do not evaporate or otherwise change composition prior to analysis. Septum seal vials can be used for short term storage of any solvents or extracts. See Annex A1 for sample and extract storage guidance.

8.3.1 Avoid the use of water as a sealant.

8.4 Analyze solvent blanks at least once each day that the instrument is used; maintain these analysis records. This will verify the purity of the solvent and potentially detect carryover or contamination.

8.5 Clean syringes thoroughly between injections to ensure no carryover.

8.5.1 Conduct carryover studies periodically, and maintain records that demonstrate the adequacy of laboratory procedures to prevent carryover.

8.5.2 Running a solvent blank between samples is recommended but is not necessary if studies demonstrate that the cleaning procedure is adequate to prevent carryover.

8.6 Maintain reference files of known ignitable liquids that have been analyzed in the same manner as the questioned samples.

8.7 *Chromatogram Evaluation*—An adequate chromatogram with sufficient data for comparison work is one in which the peaks of interest are 50 to 100 % of full scale. Re-print the chromatogram, using different parameters (amplitude or presentation of data) to achieve an adequately scaled chromatogram in the targeted area of investigation.

9. Data Analysis

9.1 Initial data analysis consists of a visual comparison of the total ion chromatograms to reference ignitable liquid chromatograms as described below.

9.1.1 The essential requirement for making a classification using this procedure is the matching of the sample chromatogram with a reference ignitable liquid chromatogram obtained under similar conditions, noting points of correlation or similarities. Make all comparisons using adequate chromatograms as described in 8.7 and interpretation criteria described in Section 11, Interpretation of Results.

9.1.2 The use of externally generated libraries of chromatograms is not sufficient for identification of an ignitable liquid. Such libraries are intended only to give guidance for selection of reference ignitable liquids.

9.1.3 Pattern matching requires that the entire pattern used for comparison be displayed at the same sensitivity.

9.1.3.1 To provide sufficient detail for some comparisons, different amplitudes or presentations of the data could be necessary.

9.1.4 The carbon number range is determined by comparing the chromatogram to a reference or test mixture containing known normal alkanes.

9.1.5 Additional data analysis can be carried out using extracted ion profiling (mass chromatography), or identification of specific compounds, or both. See Appendix X1 for

TABLE 1 Ignitable Liquid Classification Scheme with Examples of Known Products for Each Class^{A, B, C}

Class	Light (C ₄ –C ₉)	Medium (C ₈ –C ₁₃)	Heavy (C ₉ –C ₂₀₊)
Gasoline — all brands, including gasohol and E85	Fresh gasoline is typically in the range C ₄ –C ₁₂		
Petroleum Distillates (including de-aromatized)	Petroleum Ether Cigarette Lighter Fluids Camping Fuels Naphtha	Charcoal Starters Paint Thinners Dry Cleaning Solvents Mineral Spirits Automotive Parts Cleaners Spray Lubricants Lamp Oils Deck Sealers Varnishes Kerosene Insecticides ^D	Kerosene Diesel Fuel Charcoal Starters Aviation Fuels Insecticides ^D Fuel Additives Lamp Oils Automotive Parts Cleaners
Isoparaffinic Products	Aviation Gasolines Lighter Fluids Charcoal Starters	Charcoal Starters Paint Thinners Copier Toners Mineral Spirits Solvent Cleaners Kerosene Lamp Oils Gun Oils	Spot Cleaners Penetrating Oils Insecticides ^D
Aromatic Products	Automotive Parts Cleaners Solvent Cleaners Xylenes Toluene-Based Products Lacquer Thinners	Automotive Parts Cleaners Specialty Cleaning Solvents Insecticides ^D Brush Cleaners	Insecticides ^D Adhesives
Naphthenic-Paraffinic Products	Cyclohexane-Based Solvents/Products	Charcoal Starters Insecticides ^D Lamp Oils Mineral Spirits Automotive Parts Cleaners	Insecticides ^D Lamp Oils
Normal Alkane Products		Candle Oils Copier Toners Lamp Oils Wax Cleaners	Candle Oils Lamp Oil Carbonless Forms Copier Toners
Oxygenated Solvents	Alcohols Ketones Lacquer Thinners Fuel Additives Surface Preparation Solvents Automotive Parts Cleaners Spray Adhesives Brush Cleaners	Metal Cleaners Gloss Removers Degreasers Furniture Strippers Cleaning Solvents Insecticides ^D	Biodiesels Fuel Additives Floor Finishes Insecticides ^D
Others-Miscellaneous	Single Component Products Enamel Reducers Lacquer Thinners Aviation Gasolines Racing Gasolines	Turpentine Products Mineral Spirits Fuel Additives Spray Lubricants Brush Cleaners Paint Thinners Citrus Cleaners Charcoal Starters	Lamp Oils Insecticides ^D Citrus Cleaners Automotive Parts Cleaners Kerosene Fuel Additives

^A The products listed in the various classes of Table 1 are examples of known commercial uses of these ignitable liquids. These examples are not intended to be all-inclusive. Reference literature materials can be used to provide more specific examples of each classification.

^B Many of the examples can be prefaced by the word “some,” as in “some charcoal starters.” It is not unusual for similarly labeled products to have examples in more than one class, as product labeling can be based upon chemical performance or property, and not necessarily on chemical composition.

^C The Ignitable Liquid Reference Collection (ILRC) is a resource with both an ignitable liquids database, available at <http://www.ilrc.ucf.edu> and a substrate database available at <http://www.ilrc.ucf.edu/substrate>.

^D Note that the active ingredient(s) in consumer products categorized here as “insecticides” are typically not inherently flammable, but the solvents and propellants used in some consumer products can be categorized as ignitable liquids.

tables of specific compounds that are commonly associated with particular classes.

9.1.6 Store the reference chromatogram(s) in the case file, along with the sample chromatogram(s).

9.1.7 The compounds that comprise ignitable liquids consist of six major types: normal alkane, branched alkane, cycloalkane, aromatic, polynuclear aromatic, and oxygenates. Other compounds could be present, but are not considered significant for the purposes of this test method.

9.1.8 Compounds of each type produce characteristic major ion fragments. These ions are listed in Table 2.

9.2 Extracted Ion Profiling (EIP):

9.2.1 A data station is used to extract and draw extracted ion profiles (mass chromatograms) for major ions characteristic of each compound type. Individual extracted ion profiles for two or more characteristic ions of the same functional groups or of similar magnitude can be summed to enhance the signal-to-noise ratio and to decrease interference by extraneous compounds that contain only one of the ions or to create summed profiles characteristic of specific classes of hydrocarbons.

9.2.1.1 Many data stations scale chromatograms so that the tallest peak is 100 % of full scale. It can be misleading to use a relative intensity display for ions of significantly different abundance. It is best to overlay these mass chromatograms or use an absolute intensity output.

9.2.2 Extracted ion profiles for an unknown sample are compared against the corresponding extracted ion profiles from reference ignitable liquids. This is generally done by visual pattern recognition as described in 9.1. Computerized pattern recognition techniques are also acceptable, provided the analyst visually verifies the results.

9.2.3 Major peaks in the extracted ion profiles should be identified by searching their mass spectra against a suitable library. The final identification shall be made by the analyst on the basis of the mass spectra and relative retention times of the components in question by comparison to reference ignitable liquids.

9.2.4 Extracted ion profiles can also be useful to distinguish compounds specific to individual classes of ignitable liquids as well as interfering compounds related to products of combustion and pyrolysis.

10. Ignitable Liquid Classification Scheme

10.1 Seven major classes of ignitable liquids can be identified by gas chromatography, mass spectrometry, extracted ion profiling (or extracted ion profile analysis), or a combination thereof, when recovered from fire debris. These classes are outlined in 10.2. Typical total ion chromatograms of many of these classes are shown in Figs. 2-10.

10.1.1 This test method is intended to allow identified ignitable liquids to be characterized as belonging to one of the classifications. Distinguishing between examples within any class could be possible, but such further characterization is not within the scope of this test method.

10.1.2 A miscellaneous category is included for those ignitable liquids that do not fall into one of the first seven major ignitable liquid classifications.

10.1.3 With the exception of the gasoline class, the major ignitable liquid classes can be divided into three subclasses based on boiling (*n*-alkane) range: light, medium and heavy.

10.1.3.1 *Light Product Range*—*n*-C₄–*n*-C₉; the majority of the pattern occurs in the range *n*-C₄–*n*-C₉, no major peaks associated with the ignitable liquid exist above *n*-C₁₁.

10.1.3.2 *Medium Product Range*—*n*-C₈–*n*-C₁₃; narrow range products, the majority of the pattern occurs in the range of *n*-C₈–*n*-C₁₃, no major peaks associated with the ignitable liquid below *n*-C₇ or above *n*-C₁₄.

10.1.3.3 *Heavy Product Range*—*n*-C₉–*n*-C₂₀₊, typically broad range products, the majority of the pattern occurs in the range C₉–C₂₃, with a continuous pattern spanning at least 5 consecutive *n*-alkanes. Also included in the subclass are narrow range (encompassing less than five *n*-alkanes) ignitable liquid products starting above *n*-C₁₁.

10.1.3.4 It could be necessary to characterize a product as “light to medium,” or “medium to heavy,” when the carbon number range does not fit neatly into one of the above categories. In such instances, the carbon number range should be reported.

10.2 In order for an extract to be characterized as containing a particular class, the following minimum criteria shall be met:

10.2.1 Criteria for the Identification of Gasoline:

10.2.1.1 *General*—All brands of gasoline including gasohol and E85. Pattern characterized by abundant aromatics in a specific pattern.

10.2.1.2 *Alkanes*—Present. Pattern can vary by brand, grade, and lot.

10.2.1.3 *Cycloalkanes*—Not present in significant amounts.

10.2.1.4 *Aromatics*—Petroleum pattern comparable to that of the reference ignitable liquids; 1-methyl-3-ethylbenzene (*m*-ethyltoluene), 1-methyl-4-ethylbenzene (*p*-ethyltoluene), 1,3,5-trimethylbenzene, 1-methyl-2-ethylbenzene (*o*-ethyltoluene), and 1,2,4-trimethylbenzene shall be present; above *n*-C₇, the aromatic concentration is generally substantially higher than the alkane concentration.

TABLE 2 Major Ions Present in Mass Spectra of Common Ignitable Liquids^{A,B,C,D,E}

Compound Type	<i>m/z</i>
Alkane	43, 57, 71, 85, 99
Cycloalkane and alkene	55, 69
<i>n</i> -Alkylcyclohexanes	82, 83
Aromatic—alkylbenzenes	91, 105, 119, 92, 106, 120, 134
Indanes	117, 118, 131, 132
Alkyl-naphthalenes (Condensed Ring Aromatics)	128, 142, 156, 170
Alkylstyrenes	104, 117, 118, 132, 146
Alkylanthracenes	178, 192, 206
Alkylbiphenyls/acenaphthenes	154, 168, 182, 196
Monoterpenes	93, 136
Ketones	43, 58, 72, 86
Alcohols	31, 45

^A Smith, R. M., “Arson Analysis by Mass Chromatography,” *Analytical Chemistry*, Vol 54, No. 13, November 1982, pp. 1399A–1409A.

^B Kelly, R. L., and Martz, R. M., “Accelerant Identification in Fire Debris by Gas Chromatography/Mass Spectrometry Techniques,” *Journal of Forensic Sciences*, Vol 29, No. 3, 1984, pp. 714–722.

^C Keto, R. O., and Wineman, P. L., “Detection of Petroleum-Based Accelerants in Fire Debris by Target Compound Gas Chromatography/Mass Spectrometry,” *Analytical Chemistry*, Vol 63, No. 18, September 15, 1991, pp. 1964–1971.

^D Keto, R. O., “GC/MS Data Interpretation for Petroleum Distillate Identification in Contaminated Arson Debris,” *Journal of Forensic Sciences*, Vol 40, No. 3, 1995, pp. 412–423.

^E McLafferty, F. W., and Turecek, F., *Interpretation of Mass Spectra*, 4th edition, University Science Books, Sausalito, California, 1993, pp. 233 and 247.

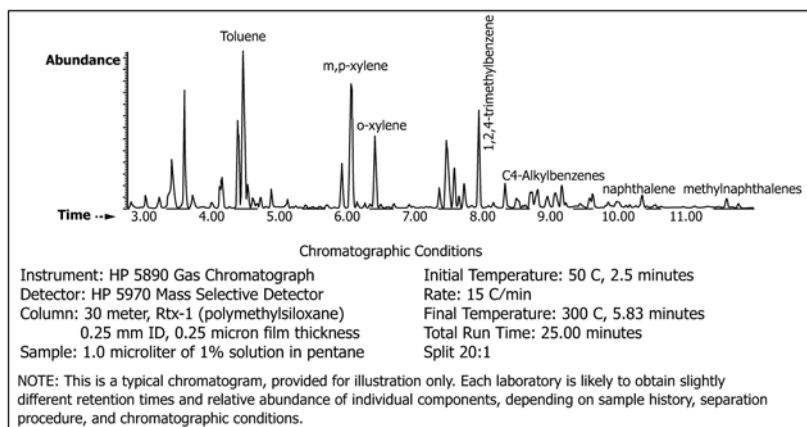


FIG. 2 Example of a Gasoline Pattern; 50 % Evaporated Gasoline

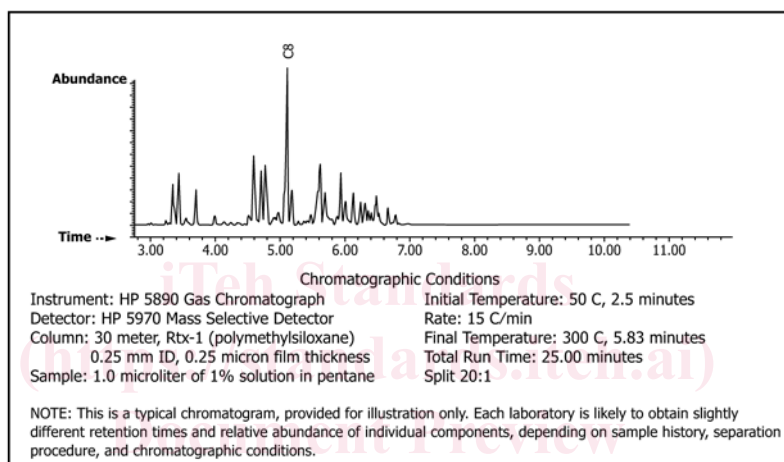


FIG. 3 Example of a Light Petroleum Distillate; Cigarette Lighter Fluid

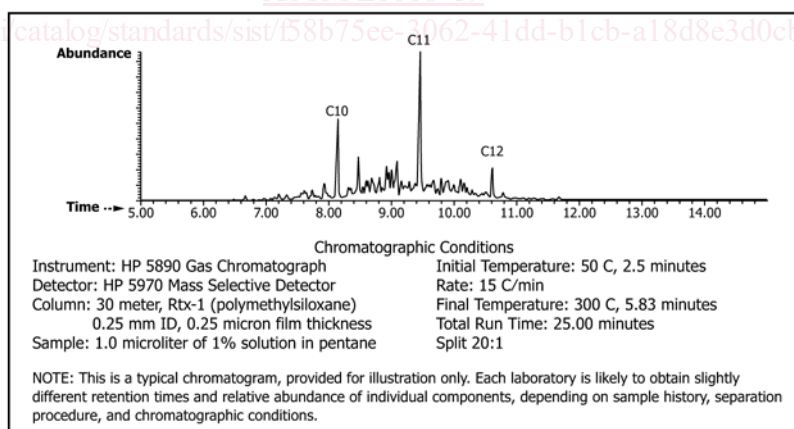


FIG. 4 Example of a Medium Petroleum Distillate Pattern; 50 % Evaporated Mineral Spirits

10.2.1.5 *Condensed Ring Aromatics*—Pattern comparable to known standard is usually present, including naphthalene, 1- and 2-methylnaphthalenes. These compounds could be absent in some gasolines. Indan (dihydroindene) and methyl indans are usually present.

10.2.1.6 **Warning**—The mere presence of alkylbenzenes does not justify an identification of gasoline. These compounds shall be present at approximately the same relative concentra-

tions as are observed in samples of known gasoline. Many carpet samples that have been exposed to fire conditions contain these compounds in some concentrations. Benzene, toluene, ethylbenzene, xylenes, cumenes, ethyltoluenes, and naphthalenes, which are present in gasoline, are also sometimes found in fire debris samples containing no foreign ignitable liquid residues. The presence of high levels of alkenes and oxygenates can indicate significant pyrolysis of the matrix