



Designation: E 1618 – 01

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 E 1618; 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 While this test method is suitable for all samples, it is especially appropriate for extracts that contain high background levels of substrate materials or pyrolysis products. This guide 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 the standard. The values given in parentheses are for information only.

1.4 *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:

E 1385 Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris Samples by Steam Distillation²

E 1386 Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris by Solvent Extraction²

E 1387 Test Method for Flammable or Combustible Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography²

E 1388 Practice for Sampling of Headspace Vapors from Fire Debris Samples²

E 1412 Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris Samples by Passive Headspace Concentration²

E 1413 Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration²

E 1459 Guide for Physical Evidence Labeling and Related Documentation²

E 1492 Guide for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Laboratory²

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 chemical components (target compounds) may be identified by their mass spectra and retention times. Semi-quantitative determination of target compounds which are identified by mass spectra and retention time may be used to develop target compound chromatograms (TCCs).

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 target compound chromatograms (TCC), or combination thereof, are evaluated by visual pattern matching against known reference ignitable liquids.

3.2.2 Ignitable liquids may be grouped into one of eight major petroleum 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 was incendiary in nature. Further investigation may reveal a legitimate reason for the presence of ignitable liquid residues.

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² *Annual Book of ASTM Standards*, Vol 14.02.

4.1.2 Due to the volatility of ignitable liquids and to 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 target compound identification techniques described herein may 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 may 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 may be used provided that each component of the test mixture 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 scanning from 40 to 400 m/e with unit resolution or better, with continuous data output. M/e values above 40 may not be sufficient to detect or identify some lower molecular weight compounds; for example, methanol, ethanol, acetone.

5.2.1 *Sensitivity*—The system must be capable of detecting each component of the test mixture referenced in 6.1 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 chromatographic and mass spectral data from sample runs.

5.3.1 *Data Handling*—The data system must 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 must be capable of retrieving a specified mass spectral scan 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 reproducibility introducing sample sizes in the range of 0.5 to 5 mL.

6. Chemicals, Reagents, and Reference Materials

6.1 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-ethyl benzene (*m*-ethyltoluene), and 1,2,4-trimethylbenzene (pseudocumene). Additional compounds may 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 (0.5 microlitre per millilitre) in the chosen solvent (see 6.3). A typical chromatogram of the test mixture in shown in Fig. 1.

6.2 *Reference Ignitable Materials*—Ignitable liquids must be available for the various ignitable liquids classes represented in Table 1.

6.2.1 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.

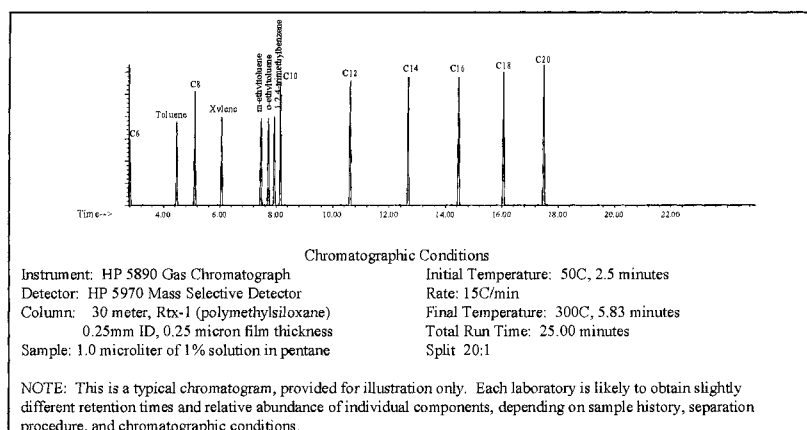


FIG. 1 Test Mixture Containing C8-C20 Normal Hydrocarbons, toluene, p-xylene, o-ethyltoluene, m-ethyltoluene, and 1,2,4-trimethylbenzene

TABLE 1 Ignitable Liquid Classification Scheme^A

Class	Light (C ₄₋₉)	Medium (C _{8-C₁₃})	Heavy (C _{8-C₂₀₊})
Gasoline-all brands, including gasohol	Fresh gasoline is typically in the range C ₄ -C ₁₂		
Petroleum Distillates	Petroleum Ether Some Cigarette Lighter Fluids Some Camping Fuels	Some Charcoal Starters ^B Some Paint Thinners Some Dry Cleaning Solvents	Kerosene Diesel Fuel Some Jet Fuels Some Charcoal Starters
Isoparaffinic Products	Aviation Gas Specialty Solvents	Some Charcoal Starters Some Paint Thinners Some Copier Toners	Some Commercial Specialty Solvents
Aromatic Products	Some Paint and Varnish Removers Some Automotive Parts Cleaners Xylenes, Toluene-based products.	Some Automotive Parts Cleaners Specialty Cleaning Solvents Some Insecticide Vehicles Fuel Additives	Some Insecticide Vehicles Industrial Cleaning Solvents
Naphthenic Paraffinic Products	Cyclohexane based solvents/products	Some Charcoal Starters Some Insecticide Vehicles Lamp Oils	Some Insecticide Vehicles Lamp Oils Industrial Solvents
N-Alkanes Products	Solvents Pentane Hexane Heptane	Some Candle Oils Copier Toners	Some Candle Oils Carbonless Forms Copier Toners
De-Aromatized Distillates	Some Camping Fuels	Some Charcoal Starters Some Paint Thinners	Some Charcoal Starters Odorless Kerosenes
Oxygenated Solvents	Alcohols Ketones Some Lacquer Thinners Fuel Additives Surface Preparation Solvents	Some Lacquer Thinners Some Industrial Solvents Metal Cleaners/Gloss Removers	
Others-Miscellaneous	Single Component Products Some Blended Products Some Enamel Reducers	Turpentine Products Some Blended Products Various Specialty Products	Some Blended Products Various Specialty Products

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

^BAs can be noted there are products found in multiple classifications such as “charcoal starters”. Therefore, many of the examples can be preface by the word “some”, as in “some charcoal starters.”

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

6.3 *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.

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

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

7. Equipment Calibration and Maintenance

7.1 Verify the consistent performance of the chromatographic instrument using known concentrations of known ignitable liquids or test mixtures as well as blanks. 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 per 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 base to avoid sample contamination by “carry-over” 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 E 1385, E 1386, E 1388, E 1412, and E 1413.

8.3 Due to the volatility of solvents and analytes, care must be taken to ensure that samples do not evaporate or otherwise change composition. Extracts in carbon disulfide may be covered with water prior to removing the extracts from the sample preparation hood. Alternatively, septum vials may be used for storing any solvents or extracts.

8.3.1 If water is used as a sealant, exercise care to avoid the introduction of water onto DMCS treated columns.

8.3.2 Avoid the use of water as a sealant if the presence of water soluble compounds is suspected.

8.4 Analyze solvent blanks at least every day that the instrument is used, and maintain documentation. A solvent blank 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, and maintain documentation that demonstrates the adequacy of laboratory procedures to prevent carryover.

8.5.2 Running solvent blanks between each sample 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*- A good chromatogram for comparison work is one in which the peaks of interest are 50 to 100% of full scale. Rerun samples, or re-plot chromatogram, using different parameters (attenuation or sample size) to achieve a good chromatogram.

8.7.1 In addition to the chromatogram described above, it is sometimes necessary to produce other, off-scale plots, in order to bring some features into view for comparison. Such off-scale plots may be required when there are one or more components present at a significantly higher concentration than the other components in the sample.

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.

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.

9.1.4 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 may be carried out using extracted ion profiling (mass chromatography), target compound analysis, or both.

9.1.6 The compounds that comprise ignitable liquids consist of six major types: alkane (both normal and branched), alkene, cycloalkanes, aromatic, polynuclear aromatic, and oxygenates. Other compounds may be present, but are not considered significant for the purposes of this method.

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

9.2 *Extracted ion Profiling (EIP)*:

TABLE 2 Major Ions Present in Mass Spectra of Common Flammable and Combustible Liquids^A

Compound Type	m/e
Alkane	43, 57, 71, 85
Cycloalkane and alkene	55, 69
<i>n</i> -Alkylcyclohexanes	82, 83
Aromatic—alkylbenzenes	91, 105, 119; 92, 106, 120
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 R. Martin Smith, *Analytical Chemistry*, Vol 54, No. 13, November 1982, pp 1399A–1409A.

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 may 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 may be misleading to use a common scale for ions of significantly different abundance.

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 that results are checked visually.

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 must 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.

9.3 *Target Compound Analysis (TCC)*:

9.3.1 Target compound analysis uses key specific compounds to characterize an ignitable liquid. These target compounds are listed in Table 3, Table 4, and Table 5.

TABLE 3 Gasoline Target Compounds

Compound	CAS Number
1. 1,3,5-Trimethylbenzene	108-67-8
2. 1,2,4-Trimethylbenzene	95-36-3
3. 1,2,3-Trimethylbenzene	526-73-8
4. Indane	496-11-7
5. 1,2,4,5-Tetramethylbenzene	95-93-2
6. 1,2,3,5-Tetramethylbenzene	527-53-7
7. 5-Methylindane	874-35-1
8. 4-Methylindane	824-22-6
9. Dodecane	112-40-3
10. 4,7-Dimethylindane	6682-71-9
11. 2-Methylnaphthalene	91-57-6
12. 1-1-Methylnaphthalene	90-12-0
13. Ethylnaphthalenes (mixed)	1127-76-0
14. 1,3-Dimethylnaphthalene	575-41-7
15. 2,3-Dimethylnaphthalene	581-40-8

TABLE 4 Medium Petroleum Distillate (MPD) Target Compounds

Compound	CAS Number
1. Nonane	111-84-2
2. Propylcyclohexane	1678-92-8
3. 1,3,5-Trimethylbenzene	108-67-8
4. 1,2,4-Trimethylbenzene	95-36-3
5. Decane	124-18-5
6. 1,2,3-Trimethylbenzene	526-7-8
7. <i>n</i> -Butylcyclohexane	1678-93-9
8. Trans-decalin	493-02-7
9. Undecane	1120-21-4
10. 1,2,3,5-Tetramethylbenzene	527-53-7
11. <i>n</i> -Pentylcyclohexane	4292-92-6
12. Dodecane	112-40-3
13. <i>n</i> -Hexylcyclohexane	4292-75-5

TABLE 5 Heavy Petroleum Distillate (HPD) Target Compounds

Compound	CAS Number
1. Decane	124-18-5
2. <i>n</i> -Butylcyclohexane	1678-93-9
3. Trans-decalin	493-02-7
4. Undecane	1120-21-4
5. 1,2,3,5-Tetramethylbenzene	527-53-7
6. <i>n</i> -Pentylcyclohexane	4292-92-6
7. Dodecane	112-40-3
8. <i>n</i> -Hexylcyclohexane	4292-75-5
9. 2-Methylnaphthalene	91-57-6
10. 1-1-Methylnaphthalene	90-12-0
11. Tridecane	629-50-5
12. <i>n</i> -Heptylcyclohexane	005617-41-4
13. 1,3-Dimethylnaphthalene	575-41-7
14. Tetradecane	629-59-4
15. <i>n</i> -Octylcyclohexane	1795-15-9
16. 2,3,5-Trimethylnaphthalene	2245-38-7
17. Pentadecane	629-62-9
18. <i>n</i> -Nonylcyclohexane	2883-02-5
19. Hexadecane	544-76-3
20. Heptadecane	629-78-7
21. Pristane	1921-70-6
22. Octadecane	593-45-3
23. Phytane	638-36-8
24. Nonadecane	629-92-5
25. Eicosane	112-95-8
26. Heneicosane	629-94-7

9.3.2 Semi-quantitative ratios for the target compounds must be derived and compared against standards to ensure not only their presence but also that their chromatographic patterns match. Computerized pattern matching techniques are acceptable, providing the analyst visually verifies results.

9.3.2.1 Target compound pattern recognition may be improved by the production of target compound chromatograms, which are graphical representations of semi-quantitative peak areas for the target compounds. Target compound data may be plotted as a bar graph, with the *x*-axis representing retention time and the *y*-axis representing peak area. Each target compound is depicted by a single bar on the graph.

9.3.2.2 Target compound chromatograms for unknown samples are compared to those generated for reference samples. The same pattern matching criteria for mass chromatography apply to target compound chromatography.

9.3.2.3 Major peaks in the TIC not accounted for by one of the target compound types may be identified by searching their mass spectra against a suitable library. The final identification must 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 materials.

9.3.2.4 While TCCs provide much useful information, a TCC should not be the sole basis for the identification of an ignitable liquid residue.

10. Ignitable Liquid Classification Scheme

10.1 Eight major classes of ignitable liquids may be identified by gas chromatography, mass spectrometry, ion profiling, or 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 may 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 eight major ignitable liquid classifications.

10.1.3 With the exception of the gasoline class, the major ignitable liquid classes may be divided into 3 subclasses based on boiling (*n*-hydrocarbon) range: Light, Medium and Heavy.

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

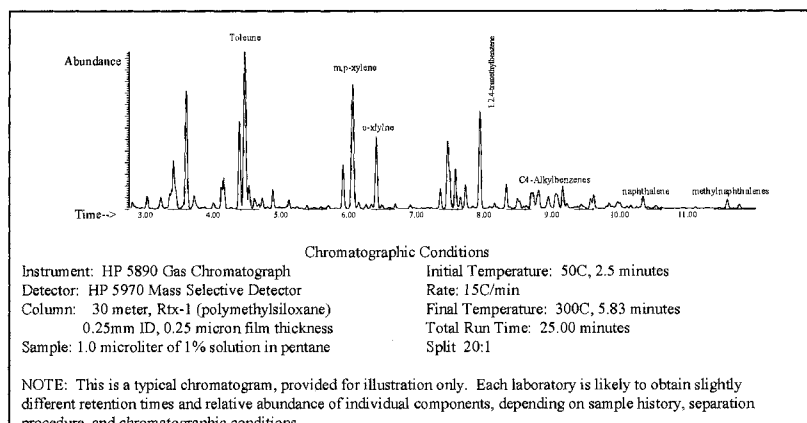


FIG. 2 Example of a Gasoline Pattern; 50 % Evaporated Gasoline

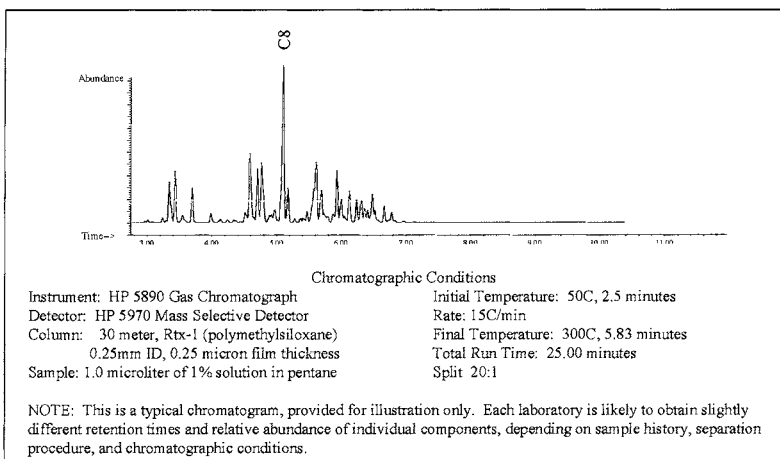


FIG. 3 Example of a Light Petroleum Distillate; Cigarette Lighter Fluid

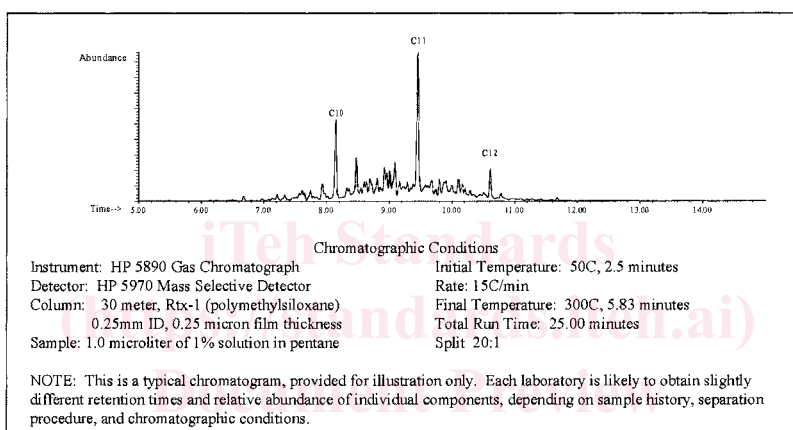


FIG. 4 Example of a Medium Petroleum Distillate Pattern; 50 % Evaporated Mineral Spirits

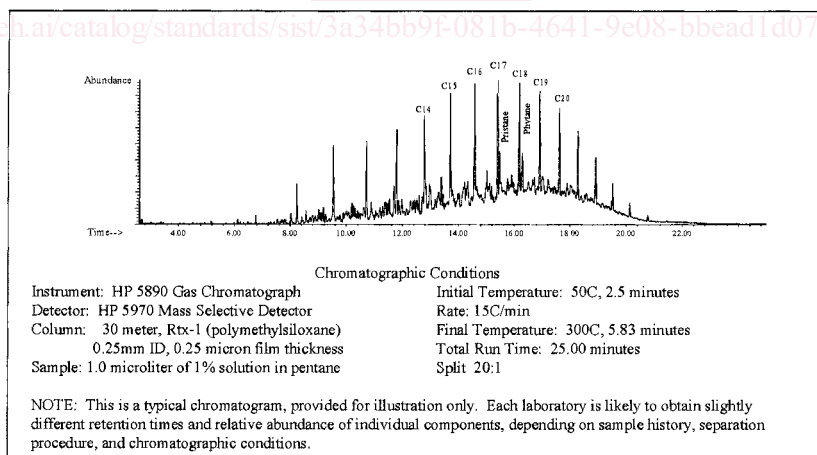


FIG. 5 Example of Heavy Petroleum Distillate; Diesel Fuel

10.1.3.2 *Medium product range*— C_8 - C_{13} ; narrow range products, the majority of the pattern occurs in the range of C_8 - C_{13} , no major peaks associated with the ignitable below C_7 or above C_{14} .

10.1.3.3 *Heavy product range*— C_9 - C_{20+} , typically broad range products, the majority of the pattern occurs in the range C_9 - C_{23} , with a continuous pattern spanning at least 5 consecutive n-alkanes. Also included in the subclass are narrow range