



Designation: E1387 – 01

# Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography<sup>1</sup>

This standard is issued under the fixed designation E1387; 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 ( $\epsilon$ ) 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 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information 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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific precautionary statement, see 6.3.

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

**E1385** Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Steam Distillation<sup>3</sup>

**E1386** Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction

**E1388** Practice for Sampling of Headspace 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 and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration

**E1459** Guide for Physical Evidence Labeling and Related Documentation

**E1492** Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory

**E1618** Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry

## 3. Summary of Test Method

3.1 The sample extract or preparation is introduced into the gas chromatographic column containing a liquid phase suitable for the separation of common ignitable liquid components. The resulting chromatogram is interpreted by techniques of pattern recognition and pattern comparison described in this test method. Ignitable liquids may fall into one of eight major classifications, or into a “miscellaneous” category 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 a field investigator’s opinion regarding the origin, fuel load, and incendiary nature of a 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.

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 When the gas chromatographic pattern is not sufficiently complex, as described in 9.3, additional analytical techniques are required.

## 5. Apparatus

5.1 *Gas Chromatograph*—A chromatograph capable of using capillary columns and equipped with a flame ionization or mass spectral detector.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics. Current edition approved Sept. 10, 2001. Published December, 2001. Originally published as E1387 – 90. Last previous edition E1387 – 95. DOI: 10.1520/E1387-01.

<sup>2</sup> 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.

<sup>3</sup> Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

5.1.1 *Sensitivity*—The system shall be capable of detecting each component of the test mixture referenced in 6.5.

5.1.2 *Sample Inlet System*—A sample inlet system that allows a reproducible volume of liquid to be injected. The system may be operated in either split or splitless mode with capillary columns; the inlet system may use on-column technology.

5.1.3 *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.

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

5.2 *Data Station*—A computerized data station, capable of storing chromatographic data from sample runs. A strip chart recorder is acceptable.

5.3 *Syringes:*

5.3.1 *For liquid samples*—a syringe, capable of reproducibly introducing sample sizes in the range of 0.1 to 10.0 µL.

5.3.2 *For gas samples*—a gas-tight syringe capable of reproducibly introducing sample sizes in the range of 0.5 to 5 mL.

6. Chemicals, Reagents and Reference Materials

6.1 *Purity of Reagents*—Reagent grade 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.<sup>4</sup> Other grades may 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.

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, 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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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 a 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 *Combustion Gases*—Air and hydrogen (if a flame ionization detector is used).

6.5 *Test Mixture*—The test mixture shall consist of a minimum of the even-numbered normal alkanes (ranging 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 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 (0.05 µL/mL) in the chosen solvent (see 6.2). A typical chromatogram of the test mixture is shown in Fig. 1.

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

6.6.1 Typically, reference ignitable liquids are diluted 1:100 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.

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

7. Sample Handling

7.1 Methods for isolating ignitable liquid residues from fire debris for analysis are described in Practices E1385, E1386, E1388, E1412, and E1413.

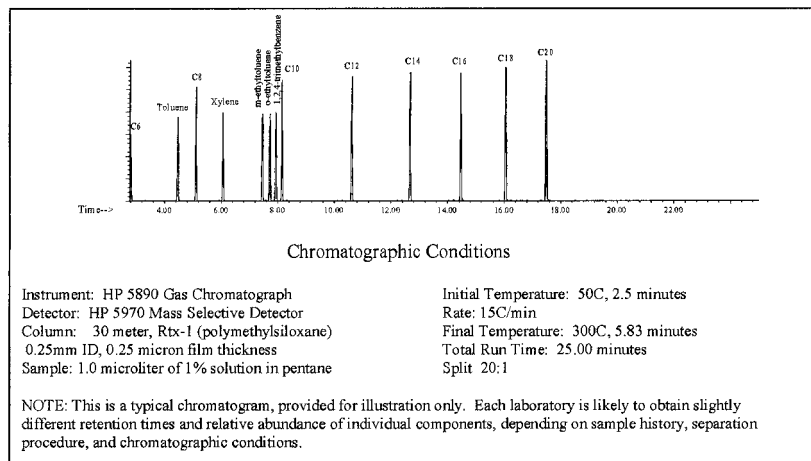


FIG. 1 Test Mixture containing C8-C20 normal hydrocarbons, toluene, p-xylene, o-ethyltoluene, m-ethyltoluene, and 1,2,4-trimethylbenzene

**TABLE 1 Ignitable Liquid Classification Schemes<sup>A</sup>**

Class	Light (C <sub>4-9</sub> )	Medium (C <sub>8-C<sub>13</sub></sub> )	Heavy (C <sub>9- C<sub>20+</sub></sub> )
Gasoline-all brands, including gasohol	Fresh gasoline is typically in the range C <sub>4</sub> -C <sub>12</sub>		
Petroleum Distillates	Petroleum Ether Some Cigarette Lighter Fluids <sup>B</sup> Some Camping Fuels	Some Charcoal Starters 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	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 Some Lamp Oils	Some Insecticide Vehicles Some Lamp Oils Industrial Solvents
Normal 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

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

<sup>B</sup>As can be noted, there are products found in multiple classifications such as “charcoal starters”. Therefore, many of the examples can be prefaced by the word “some”, as in “some charcoal starters.”

7.2 Due to the volatility of the solvents and the 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 seal vials may be used for storing any solvents or extracts.

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

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

7.3 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.

7.4 Clean syringes thoroughly between injections to ensure no carryover.

7.4.1 Conduct carryover studies, and maintain documentation that demonstrates the adequacy of laboratory procedures to prevent carryover.

7.4.2 Running solvent blanks between each sample is not necessary if studies demonstrate that the cleaning procedure is adequate to prevent carryover.

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

## 8. Calibration

8.1 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.

8.1.1 Maintain quality control documentation.

8.1.1.1 Reference ignitable liquids must be run under the same chromatographic conditions as those used to produce the sample chromatogram.

8.1.1.2 Every case file that includes a positive identification of an ignitable liquid or residue must include the reference chromatogram used to confirm the identification.

8.2 *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 replot chromatogram, using different parameters (attenuation or sample size) to achieve a good chromatogram.

8.2.1 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.

8.3 *Cleaning of the equipment:*

8.3.1 Change septa and clean or replace injector liners on a periodic basis to avoid sample contamination by “carry-over” of residual material from previous sample injections.

8.3.2 Clean syringes thoroughly between injections to ensure no carryover.

8.3.3 System Solvent blanks should be run at least daily to ensure no carry-over between samples or contamination.

#### 8.4 Reference Files:

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

### 9. Ignitable Liquid Classification Scheme

9.1 Eight major classes of ignitable liquids may be identified by gas chromatography when suitable peaks for comparison are present. These classes are outlined in 9.3 and Table 1. Typical chromatograms of many of these classes are shown in Figs. 2-10.

9.1.1 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 method.

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

9.1.3 Ignitable liquid classification of the questioned sample is determined by the classification of the reference ignitable liquid used for the comparison. Documented reference information or mass spectral analysis is required for the initial classification of the reference ignitable liquid.

9.1.3.1 Some ignitable liquids may require mass spectral techniques for proper identification/classification.

9.2 With the exception of the gasoline class, the major ignitable liquid classes may be divided into 3 subclasses based on n-alkane range. These subclasses are designated light, medium, and heavy products.

9.2.1 *Light product range*—C<sub>4</sub>-C<sub>9</sub>, the majority of the pattern occurs in the range C4-C9, no major peaks associated with the ignitable liquid exist above C11.

9.2.2 *Medium product range*—C<sub>8</sub>-C<sub>13</sub>, narrow range products, the majority of the pattern occurs in the range C8-C13, no major peaks associated with the ignitable liquid. Below C7 or above C14.

9.2.3 *Heavy product range*—C<sub>9</sub>-C<sub>20+</sub>, typically broad range products, the majority of the pattern occurs in the range C9-C23, with a continuous pattern spanning at least 5 n-alkanes. Also included in this subclass are narrow range (encompassing less than 5 n-alkanes) ignitable liquid products starting above C11.

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

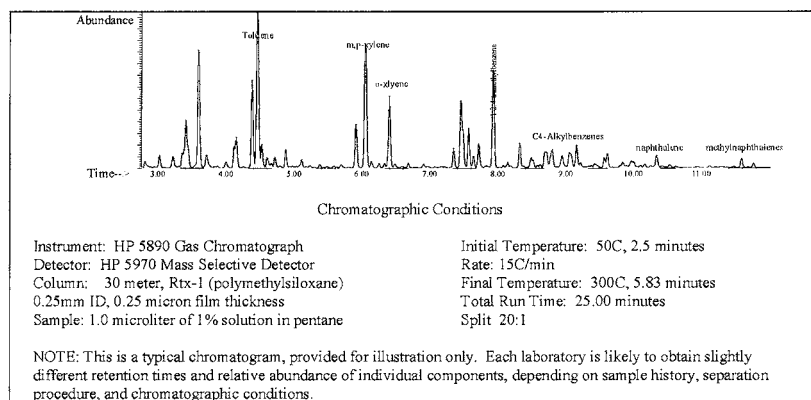
9.3 In order for an extract to be characterized as containing a particular class, the following minimum criteria must be met:

9.3.1 *Gasoline*—The C<sub>3</sub> alkylbenzene four-peak group, which represents *m*-ethyltoluene, *p*-ethyltoluene, 1,3,5-trimethylbenzene, *o*-ethyltoluene, and 1,2,4-trimethylbenzene (pseudocumene), must be present; this group occupies the range between C<sub>9</sub> and C<sub>10</sub> and is still present in gasolines that have lost as much as 90 % of their initial (“fresh”) weight by evaporation or combustion. Other peak groupings characteristic of gasoline including the C<sub>4</sub> alkylbenzenes and various aliphatic compounds must also be present.

**CAUTIONS:** The mere presence of these alkylbenzenes does not justify an identification of gasoline. These compounds must be present at approximately the same relative concentrations as are observed in samples of known gasoline. Many carpet samples that have been exposed to fire conditions contain these compounds in some concentration. Benzene, toluene, ethylbenzene, xylenes, C<sub>3</sub> alkylbenzenes, and naphthalenes, which are present in gasoline, are also sometimes found in fire debris samples containing no foreign ignitable liquid. The presence of high levels of other non-related compounds may indicate significant pyrolysis of the matrix and should make the recovery suspect.

Some aromatic products have patterns significantly similar to gasoline. The absence of significant but, less abundant aliphatic compounds is an indication that the product may be an aromatic solvent.

9.3.2 *Distillates*—Predominant peaks associated with a homologous series of normal alkanes in a Gaussian distribution of peak maxima with abundant but less significant isoparaffinic, cycloparaffinic, and aromatic compounds present. Light distillates may not exhibit a recognizable pattern, and may contain only one or two of the *n*-alkanes.



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

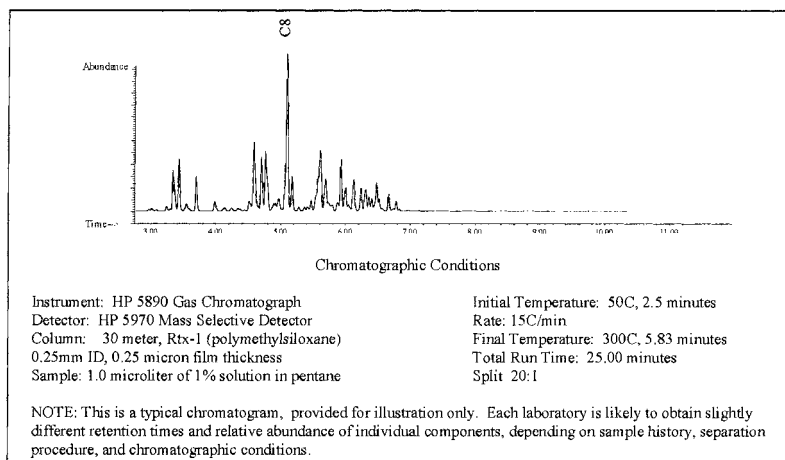


FIG. 3 Example of a Light Petroleum Distillate; Cigarette Lighter Fluid

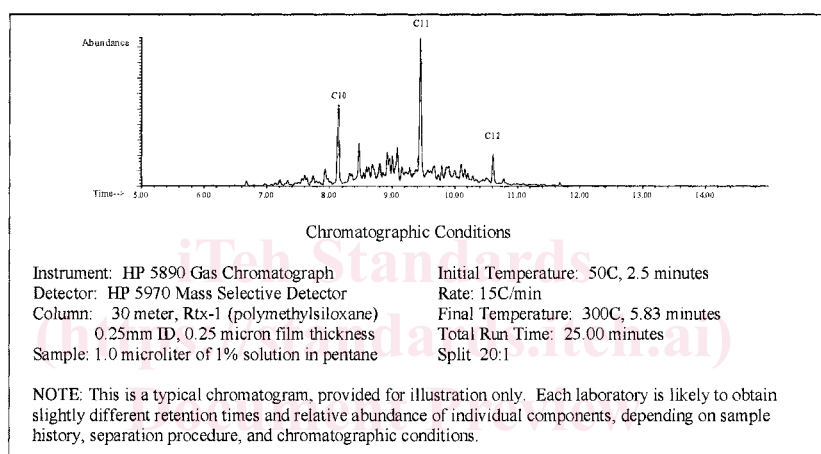


FIG. 4 Example of a Medium Petroleum Distillate Pattern; 50 % Evaporated Mineral Spirits

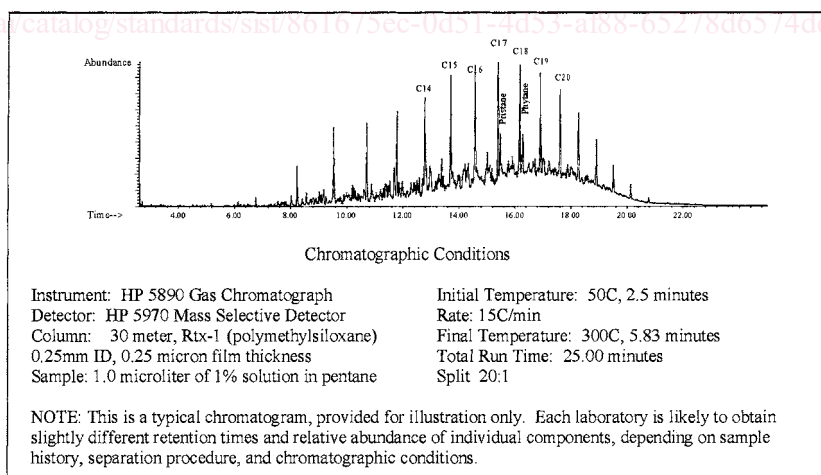


FIG. 5 Example of Heavy Petroleum Distillate; Diesel Fuel

**CAUTION:** The absence of specific less abundant compounds or disproportionate representation of the lesser abundant compounds may indicate the presence of a de-aromatized distillate or a naphthenic paraffinic product. The sole presence

of normal alkanes may indicate a normal alkane product. It may not be possible to distinguish between distillates and de-aromatized distillates without the use of mass spectrometry.



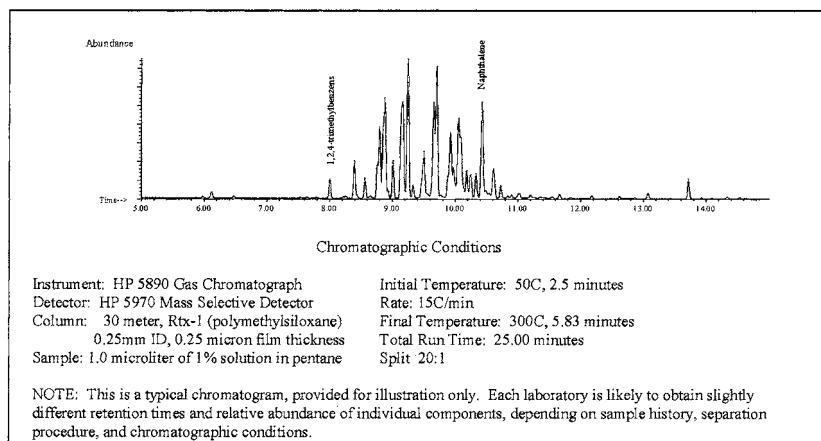


FIG. 6 Example of a Medium Aromatic Solvent; Fuel Additive

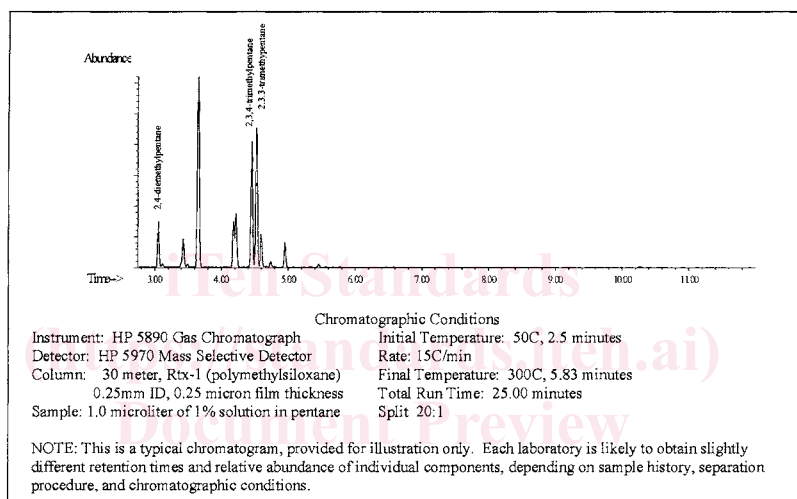


FIG. 7 Example of Light Isoparaffinic Product; Aviation Gas

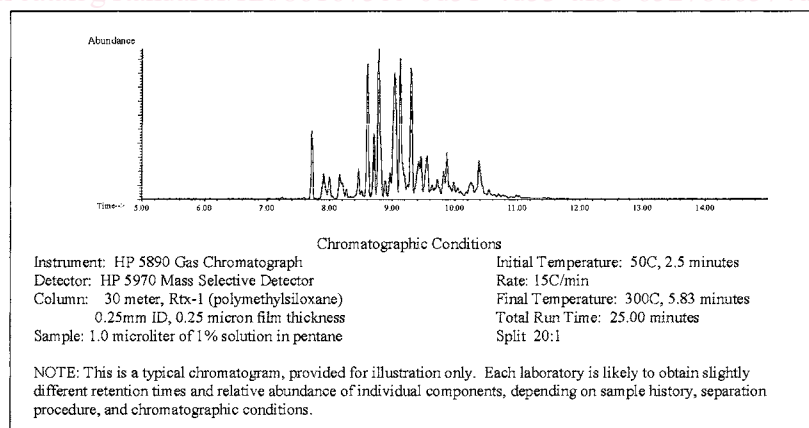


FIG. 8 Example of Medium Isoparaffinic Product; Charcoal Starter

9.3.3 *Isoparaffinic Products*—Products comprised almost exclusively of isoparaffinic compounds. The boiling range and pattern are dependent on formulation. The normal alkanes are not present or are significantly diminished.

9.3.4 *Aromatic Products*—Products comprised almost exclusively of aromatic compounds. The boiling range and pattern are dependent on formulation.

9.3.4.1 Light aromatic products may consist of single or few components. It may be necessary to identify these components by mass spectrometry.

**CAUTION:** Some aromatic products have patterns significantly similar to gasoline; the presence of significant but, less abundant aliphatic compounds is an indication that the product may be gasoline.