Designation: E 1618 – 01

# Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry[1]

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 Distillation[2]

E 1386 Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris by Solvent Extraction[2]

E 1387 Test Method for Flammable or Combustible Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography[2]

E 1388 Practice for Sampling of Headspace Vapors from Fire Debris Samples[2]

E 1412 Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris Samples by Passive Headspace Concentration[2]

E 1413 Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration[2]

E 1459 Guide for Physical Evidence Labeling and Related Documentation[2]

E 1492 Guide for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Laboratory[2]

## 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.

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

[1] 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 September 10, 2001. Published November 2001. Originally published as E 1618 – 94. Last previous edition E 1618 – 97.

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

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

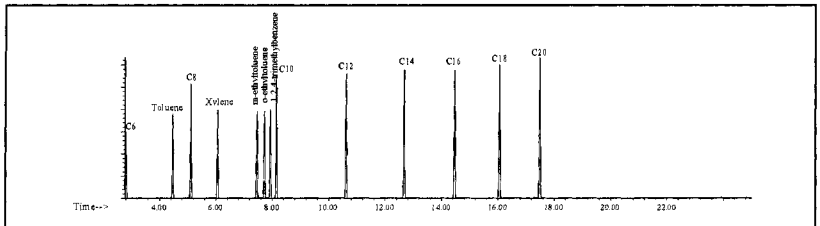
6.1 The test mixture shall consist of a minimum of the even-numbered normal alkanes (ranging from *n*-octane through *n*-eicosane), methylbenzene (toluene), 1,4-dimethylbenzene (*p*-xylene), 1-methyl-2-ethylbenzene (*o*-ethyltoluene), 1-methyl-3-ethyl benzene (*m*-ethyltoluene), and 1,2,4-trimethylbenzene (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.

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.




Chromatographic Conditions

| | |
|---|---|
| Instrument: HP 5890 Gas Chromatograph | Initial Temperature: 50C, 2.5 minutes |
| Detector: HP 5970 Mass Selective Detector | Rate: 15C/min |
| Column: 30 meter, Rtx-1 (polymethylsiloxane) | Final Temperature: 300C, 5.83 minutes |
| 0.25mm ID, 0.25 micron film thickness | Total Run Time: 25.00 minutes |
| Sample: 1.0 microliter of 1% solution in pentane | Split 20:1 |

NOTE: This is a typical chromatogram, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundance of individual components, depending on sample history, separation procedure, and chromatographic conditions.

**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 ($C_4$-$C_9$) | Medium ($C_8$-$C_{13}$) | Heavy ($C_8$–$C_{20+}$) |
|---|---|---|---|
| Gasoline-all brands, including gasohol | Fresh gasoline is typically in the range $C_4$-$C_{12}$ | | |
| Petroleum Distillates | Petroleum Ether<br>Some Cigarette Lighter Fluids<br>Some Camping Fuels | Some Charcoal Starters[B]<br>Some Paint Thinners<br>Some Dry Cleaning Solvents | Kerosene<br>Diesel Fuel<br>Some Jet Fuels<br>Some Charcoal Starters |
| Isoparaffinic Products | Aviation Gas<br>Specialty Solvents | Some Charcoal Starters<br>Some Paint Thinners<br>Some Copier Toners | Some Commercial Specialty Solvents |
| Aromatic Products | Some Paint and Varnish Removers<br>Some Automotive Parts Cleaners<br>Xylenes, Toluene-based products. | Some Automotive Parts Cleaners<br>Specialty Cleaning Solvents<br>Some Insecticide Vehicles<br>Fuel Additives | Some Insecticide Vehicles<br>Industrial Cleaning Solvents |
| Naphthenic Paraffinic Products | Cyclohexane based solvents/products | Some Charcoal Starters<br>Some Insecticide Vehicles<br>Lamp Oils | Some Insecticide Vehicles<br>Lamp Oils<br>Industrial Solvents |
| N-Alkanes Products | Solvents<br>Pentane<br>Hexane<br>Heptane | Some Candle Oils<br>Copier Toners | Some Candle Oils<br>Carbonless Forms<br>Copier Toners |
| De-Aromatized Distillates | Soem Camping Fuels | Some Charcoal Starters<br>Some Paint Thinners | Some Charcoal Starters<br>Odorless Kerosenes |
| Oxygenated Solvents | Alcohols<br>Ketones<br>Some Lacquer Thinners<br>Fuel Additives<br>Surface Preparation Solvents | Some Lacquer Thinners<br>Some Industrial Solvents<br>Metal Cleaners/Gloss Removers | |
| Others-Miscellaneous | Single Component Products<br>Soem Blended Products<br>Some Enamel Reducers | Turpentine Products<br>Some Blended Products<br>Various Specialty Products | Some Blended Products<br>Various Specialty Products |

[A]The 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.

[B]As 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.”

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 pervious 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 in 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)*:

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.

**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 |
| *n*-Alkylcyclohexanes | 82, 83 |
| Aromatic—alkylbenzenes | 91, 105, 119; 92, 106, 120 |
| Indanes | 117, 118; 131, 132 |
| Alkylnaphthalenes (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.

9.2.2 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.

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

**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. *n*-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. *n*-Pentylcyclohexane | 4292– 92– 6 |
| 12. Dodecane | 112– 40– 3 |
| 13. *n*-Hexylcyclohexane | 4292– 75– 5 |

**TABLE 5 Heavy Petroleum Distillate (HPD) Target Compounds**

| Compound | CAS Number |
|---|---|
| 1. Decane | 124–18–5 |
| 2. *n*-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. *n*-Pentylcyclohexane | 4292–92–6 |
| 7. Dodecane | 112–40–3 |
| 8. *n*-Hexylcyclohexane | 4292–75–5 |
| 9. 2-Methylnaphthalene | 91–57–6 |
| 10. 1-1–Methylnaphthalene | 90–12–0 |
| 11. Tridecane | 629–50–5 |
| 12. *n*-Heptylcyclohexane | 005617–41–4 |
| 13. 1,3-Dimethylnaphthalene | 575–41–7 |
| 14. Tetradecane | 629–59–4 |
| 15. *n*-Octylcyclohexane | 1795–15–9 |
| 16. 2,3,5-Trimethylnaphthalene | 2245–38–7 |
| 17. Pentadecane | 629–62–9 |
| 18. *n*-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 |

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*—$C_4$-$C_9$; the majority of the pattern occurs in the range $C_4$-$C_9$, no major peaks associated with the ignitable liquid exist above $C_{11}$.

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 (encompassing less than five n-alkanes) ignitable liquid products starting above $C_{11}$.

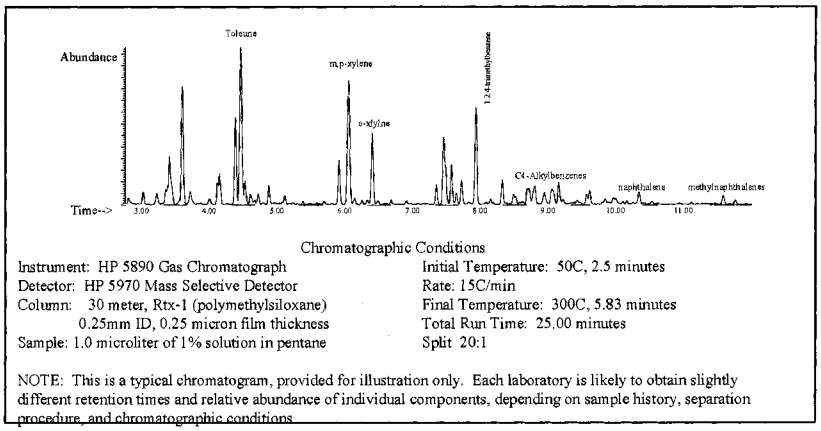
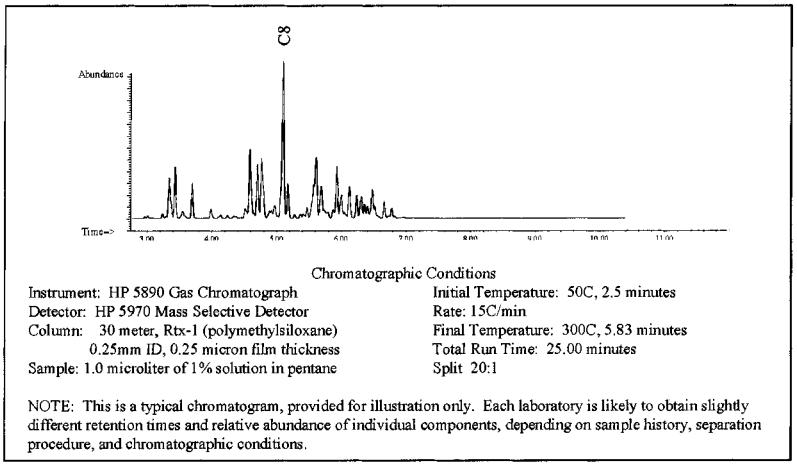



**FIG. 2 Example of a Gasoline Pattern; 50 % Evaporated Gasoline**

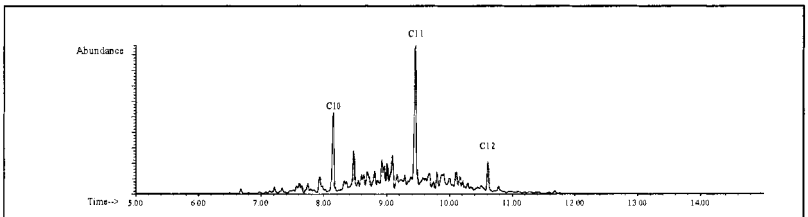



Chromatographic Conditions

| | |
|---|---|
| Instrument: HP 5890 Gas Chromatograph | Initial Temperature: 50C, 2.5 minutes |
| Detector: HP 5970 Mass Selective Detector | Rate: 15C/min |
| Column: 30 meter, Rtx-1 (polymethylsiloxane) | Final Temperature: 300C, 5.83 minutes |
| 0.25mm ID, 0.25 micron film thickness | Total Run Time: 25.00 minutes |
| Sample: 1.0 microliter of 1% solution in pentane | Split 20:1 |

NOTE: This is a typical chromatogram, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundance of individual components, depending on sample history, separation procedure, and chromatographic conditions.

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

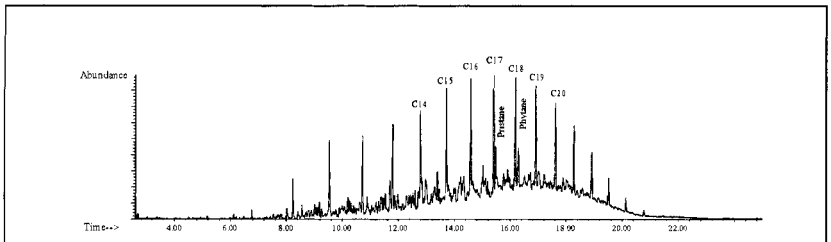



Chromatographic Conditions

| | |
|---|---|
| Instrument: HP 5890 Gas Chromatograph | Initial Temperature: 50C, 2.5 minutes |
| Detector: HP 5970 Mass Selective Detector | Rate: 15C/min |
| Column: 30 meter, Rtx-1 (polymethylsiloxane) | Final Temperature: 300C, 5.83 minutes |
| 0.25mm ID, 0.25 micron film thickness | Total Run Time: 25.00 minutes |
| Sample: 1.0 microliter of 1% solution in pentane | Split 20:1 |

NOTE: This is a typical chromatogram, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundance of individual components, depending on sample history, separation procedure, and chromatographic conditions.

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




Chromatographic Conditions

| | |
|---|---|
| Instrument: HP 5890 Gas Chromatograph | Initial Temperature: 50C, 2.5 minutes |
| Detector: HP 5970 Mass Selective Detector | Rate: 15C/min |
| Column: 30 meter, Rtx-1 (polymethylsiloxane) | Final Temperature: 300C, 5.83 minutes |
| 0.25mm ID, 0.25 micron film thickness | Total Run Time: 25.00 minutes |
| Sample: 1.0 microliter of 1% solution in pentane | Split 20:1 |

NOTE: This is a typical chromatogram, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundance of individual components, depending on sample history, separation procedure, and chromatographic conditions.

**FIG. 5 Example of Heavy Petroleum Distillate; Diesel Fuel**

10.1.3.4 It may 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 must be met:

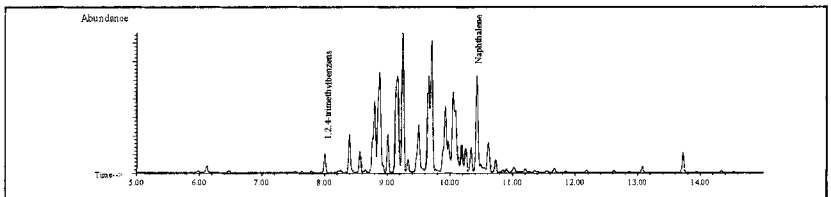
10.3 *Criteria for the Identification of Gasoline*:

10.3.1 *GENERAL*—All brands of gasoline including gasohol. Pattern characterized by abundant aromatics in specific pattern.

10.3.2 *ALKANE*—Present. Pattern may vary by brand, grade, and lot.

10.3.3 *CYCOLOALKANES*—Not present in significant amounts.

10.3.4 *AROMATIC*—Petroleum pattern comparable to that

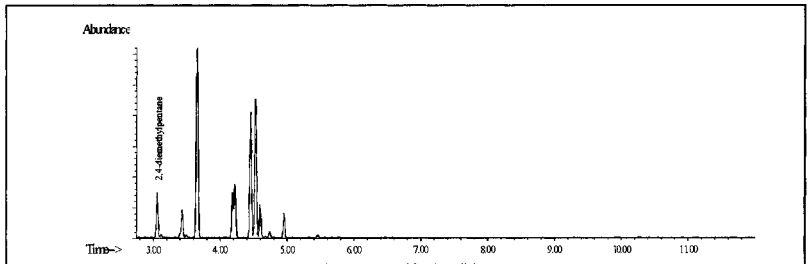



Chromatographic Conditions

| | |
|---|---|
| Instrument: HP 5890 Gas Chromatograph | Initial Temperature: 50C, 2.5 minutes |
| Detector: HP 5970 Mass Selective Detector | Rate: 15C/min |
| Column: 30 meter, Rtx-1 (polymethylsiloxane) | Final Temperature: 300C, 5.83 minutes |
| 0.25mm ID, 0.25 micron film thickness | Total Run Time: 25.00 minutes |
| Sample: 1.0 microliter of 1% solution in pentane | Split 20:1 |

NOTE: This is a typical chromatogram, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundance of individual components, depending on sample history, separation procedure, and chromatographic conditions.

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

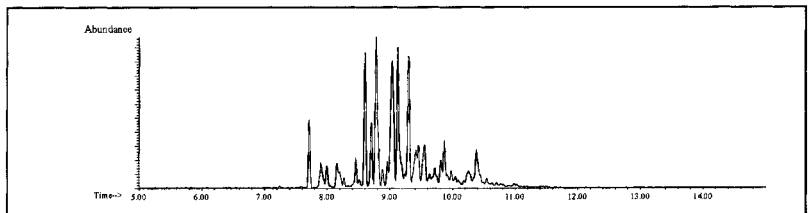



Chromatographic Conditions

| | |
|---|---|
| Instrument: HP 5890 Gas Chromatograph | Initial Temperature: 50C, 2.5 minutes |
| Detector: HP 5970 Mass Selective Detector | Rate: 15C/min |
| Column: 30 meter, Rtx-1 (polymethylsiloxane) | Final Temperature: 300C, 5.83 minutes |
| 0.25mm ID, 0.25 micron film thickness | Total Run Time: 25.00 minutes |
| Sample: 1.0 microliter of 1% solution in pentane | Split 20:1 |

NOTE: This is a typical chromatogram, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundance of individual components, depending on sample history, separation procedure, and chromatographic conditions.

**FIG. 7 Example of Light Isoparaffinic Product; Aviation Gas**




Chromatographic Conditions

| | |
|---|---|
| Instrument: HP 5890 Gas Chromatograph | Initial Temperature: 50C, 2.5 minutes |
| Detector: HP 5970 Mass Selective Detector | Rate: 15C/min |
| Column: 30 meter, Rtx-1 (polymethylsiloxane) | Final Temperature: 300C, 5.83 minutes |
| 0.25mm ID, 0.25 micron film thickness | Total Run Time: 25.00 minutes |
| Sample: 1.0 microliter of 1% solution in pentane | Split 20:1 |

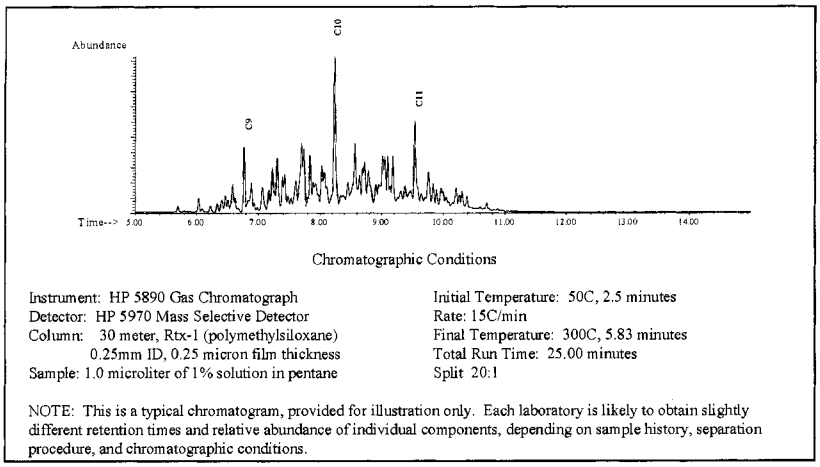
NOTE: This is a typical chromatogram, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundance of individual components, depending on sample history, separation procedure, and chromatographic conditions.

**FIG. 8 Example of Medium Isoparaffinic Product; Charcoal Starter**

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 must be present; above $C_7$, the aromatic concentration is generally substantially higher than the alkane concentration.

10.3.5 *CONDENSED RING AROMATIC*—Pattern comparable to known standard is usually present, including naphthalene, 1- and 2- methylnaphthalenes. These compounds may be absent in some gasolines. Indan (dihydroindene) and methyl indans are usually present.

10.3.6 **CAUTIONS:** The mere presence of 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 concentrations. Benzene, toluene, ethylbenzene, xylenes, cumenes, ethyltoluenes, and



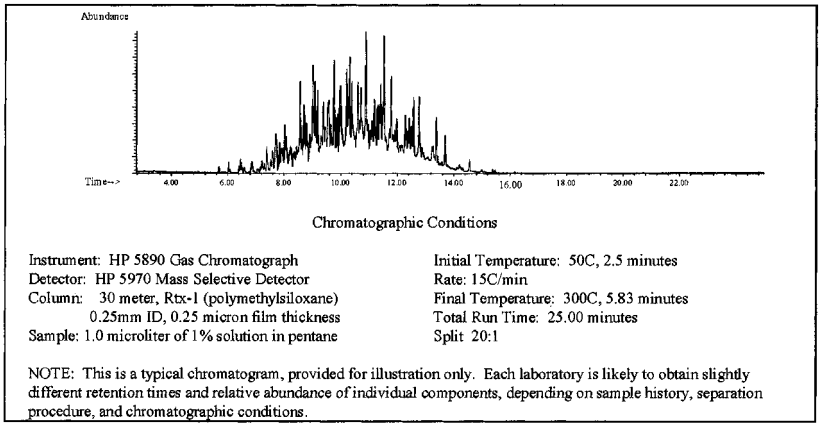

Chromatographic Conditions

Instrument: HP 5890 Gas Chromatograph
Detector: HP 5970 Mass Selective Detector
Column: 30 meter, Rtx-1 (polymethylsiloxane)
0.25mm ID, 0.25 micron film thickness
Sample: 1.0 microliter of 1% solution in pentane

Initial Temperature: 50C, 2.5 minutes
Rate: 15C/min
Final Temperature: 300C, 5.83 minutes
Total Run Time: 25.00 minutes
Split 20:1

NOTE: This is a typical chromatogram, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundance of individual components, depending on sample history, separation procedure, and chromatographic conditions.

**FIG. 9 Example of Medium De-aromatized Distillate; Odorless Paint Thinner**




Chromatographic Conditions

Instrument: HP 5890 Gas Chromatograph
Detector: HP 5970 Mass Selective Detector
Column: 30 meter, Rtx-1 (polymethylsiloxane)
0.25mm ID, 0.25 micron film thickness
Sample: 1.0 microliter of 1% solution in pentane

Initial Temperature: 50C, 2.5 minutes
Rate: 15C/min
Final Temperature: 300C, 5.83 minutes
Total Run Time: 25.00 minutes
Split 20:1

NOTE: This is a typical chromatogram, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundance of individual components, depending on sample history, separation procedure, and chromatographic conditions.

**FIG. 10 Example of a Heavy Naphthenic Paraffinic Product; Lamp Oil**

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 may indicate significant pyrolysis of the matrix and should make the recovery suspect. The presence of high levels of aromatics without the appropriate levels of alkanes may indicate an aromatic product.

10.4 *Criteria for the Identification of Distillates*:

10.4.1 *GENERAL*—Traditional distillates; pattern typified by a Gaussian distribution of peaks with aromatic compounds present.

10.4.2 *ALKANES*—Abundant. Predominant n-alkanes in normal distribution, saturated branched alkanes must be present.

10.4.3 *CYCLOALKANES*—Present, less abundant than alkanes. Pattern varies by boiling range and peak spread.

10.4.4 *AROMATICS*—Always present in medium and heavy distillates; less abundant than alkanes; pattern and abundance varies by boiling range and peak spread; may be present in light distillates.

10.4.5 *CONDENSED RING AROMATICS*—May be present based on boiling range and peak spread.

10.4.6 **CAUTIONS:** The absence of aromatics in products with abundant alkanes may be indicative of isoparrafinic products, naphthenic-paraffinic products or de-aromatized distillates.

10.5 *Criteria for the Identification of Isoparaffinic Products*:

10.5.1 *GENERAL*—Product comprised almost exclusively of branched chain aliphatic compounds (isoparaffins). The boiling range and pattern are dependent on the specific formulation.

10.5.2 *ALKANES*—Abundant. Pattern comparable to known isoparaffinic formulation. Characteristic isoparaffin product patterns present with no or insignificant levels of n-alkanes. The boiling range and component pattern are dependent on the specific formulation.

10.5.3 *AROMATIC*—Absent, or not present in significant concentrations.

10.5.4 *CYCLOALKANES*—Absent, or not present in significant concentrations. Note: Ions indicative of cycloparaffins are also present in smaller amounts in isoparaffinic compounds. “Cycloalkane” pattern representing isoalkanes maybe be present, but significantly less abundant than the alkane pattern.

10.5.5 *CONDENSED RING AROMATICS*—Not present.

10.6 *Criteria for the Identification of Aromatic Products*:

10.6.1 *GENERAL*—Products comprised almost exclusively of aromatic and/or condensed ring aromatic compounds. The

boiling range and pattern are dependent on the specific formulation.

10.6.2 *ALKANE*—Not present in significant amounts.

10.6.3 *CYCLOALKANES*—Not present in significant amounts.

10.6.4 *AROMATICS*—Abundant. Pattern comparable to known aromatic products. Pattern depends on formulation.

10.6.5 *CONDENSED RING AROMATICS*—May be present, pattern depends on formulation. Pattern comparable to known aromatic product.

NOTE: Light aromatic products may consist of single or few components. These compounds must be identified by both GC retention time and mass spectral identification.

10.7 *Criteria for the Identification of Naphthenic-Paraffinic Products*:

10.7.1 *GENERAL*—Products comprised mainly of branched chain (isoparaffinic) and cyclic (naphthenic) alkanes. The boiling range and pattern are dependent on the specific formulation.

10.7.2 *ALKANES*—Abundant. Normal alkanes may be notably absent or diminished. Depending on the feedstock, normal alkanes may be present, but at diminished levels compared to distillate products. Pattern comparable to known naphthenic-paraffinic products.

10.7.3 *CYCLOALKANES*—Abundant. Pattern comparable to known naphthenic-paraffinic products.

10.7.4 *AROMATICS*—Not present in significant amounts.

10.7.5 *CONDENSED RING AROMATICS*—Not present in significant amounts.

10.8 *Criteria for the Identification of Normal Alkane Products*:

10.8.1 *GENERAL*—Product comprised exclusively n-alkanes. The boiling range and pattern are dependent on the specific formulation.

10.8.2 *ALKANE*—Normal alkane product pattern present with no isoparaffins or only minor levels of isoparaffins. The boiling range and pattern are dependent on the specific formulation.

10.8.3 *CYCLOALKANES*—Not present in significant amounts.

10.8.4 *AROMATIC*—Not present in significant amounts.

10.8.5 *CONDENSED RING AROMATICS*—Not present in significant amounts.

Note: All major chromatographic peaks for this class must be identified by both GC retention time and mass spectral characteristics.

10.9 *Criteria for the Identification of De-aromatized Distillates*:

10.9.1 *GENERAL*—Products characterized by traditional distillate distribution with a notable absence of aromatic compounds.

10.9.2 *ALKANES*—Abundant. Predominant normal alkanes present with isoparaffinic compounds present. Pattern comparable to known distillate product.

10.9.3 *CYCLOALKANES*—Present. Pattern comparable to known distillate product.

10.9.4 *AROMATICS*—Absent or not present in significant amounts.

10.9.5 *CONDENSED RING AROMATICS*—Not present in significant amounts.

Note: Differentiation between distillates and de-aromatized distillates may not be possible without extracted ion profiling.

10.10 *Criteria for the Identification of Oxygenated Solvents*:

10.10.1 *GENERAL*—Products containing major oxygenated components may include mixtures of oxygenated compounds and other compounds or products. Major oxygenated compounds present before $C_8$; major compound(s) may include alcohols, esters, ketones. Other major compounds including toluene, xylene, and distillate formulations may also be present.

10.10.2 *ALKANE*—If in a mixture, may contain characteristic petroleum distillate pattern; pattern depends on formulation.

10.10.3 *CYCLOALKANES*—Pattern depends on formulation.

10.10.4 *AROMATIC*—Pattern depends on formulation.

10.10.5 *CONDENSED RING AROMATIC*—Not significant.

NOTE: All major oxygenated compounds must be identified by GC retention times and mass spectral characteristics.

10.10.6 **CAUTION:** The mere presence of oxygenated solvents such as alcohols or acetone does not necessarily indicate that a foreign ignitable liquid is present in the sample. There should be a large excess of the compound (at least one order of magnitude above the other peaks in the chromatogram) before the analyst should consider the finding of an oxygenated product significant.

10.11 *Miscellaneous/Other*—No classification system is likely to describe all possible ignitable liquids. There are numerous commercial and industrial products which are ignitable but which fall into more than one category or do not fall into any of the above categories other than miscellaneous. Many of these are synthetic mixtures consisting of only a few compounds, rather than distillation fractions, mass spectral identification is required in order to achieve identification.

## 11. Interpretation of Results

11.1 Pattern matching of extracted ion profiles or target compound chromatograms rarely gives perfect correlation with reference ignitable liquids. In general, the unknown pattern (if positive) will be skewed towards less volatile compounds for weathered samples or skewed towards more volatile compounds for incompletely recovered samples. Compounds may be missing from either the light end or the heavy end, or both. Under certain conditions, selective loss of classes of compounds may result from microbiological degradation. Compounds may also be added to the pattern when the pyrolysis of materials at the fire scene yields target compounds or compounds of the same type as those being compared. All of these circumstances must be taken into account by the analyst during visual pattern evaluation. It is therefore imperative that the analyst have a sufficient library of reference ignitable liquids, in successive stages of evaporation. A library of extracts from common substrate materials containing no foreign ignitable liquids should also be maintained.

11.2 *Interferences*:

11.2.1 *Extraneous Components*—Burned material from which the sample has been extracted usually contributes

extraneous components to extract. The amount and type of pyrolysis and combustion products formed during a fire depend on the substrate material and its fire history. They can consist of paraffinic, cycloparaffinic, aromatic, or condensed ring aromatic hydrocarbons, all of which will appear in the extracted ion profiles. Only those pyrolysis products that are themselves target compounds listed in Tables 3-5 will appear on the target compound chromatograms. The presence of these extraneous product components is acceptable when sufficient ignitable liquid product compounds remain to allow proper classification of the sample. When the pattern becomes overwhelmed by extraneous components, identification is not possible by this method.

11.2.2 Extracts that meet the criteria for heavy petroleum distillates should be reviewed carefully for "extraneous components" that elute near *n*-alkanes and are the result of polyolefin or high molecular weight hydrocarbon (asphalt) decomposition. Peaks representing the corresponding 1-alkene or 1, (*n*-1) diene, and having an abundance near the concentration (within 1/2 an order of magnitude) of the alkane, should be considered as indicating the presence of polyolefin or asphalt decomposition products rather than fuel oil products. Polyolefin decomposition products typically do not exhibit the same pattern of branched alkanes as fuel oils.

11.3 *Missing Components*—Exposure of the ignitable liquid to heat usually results in the preferential loss of lighter components, thereby enhancing the chromatographic pattern at the heavy end. Some sample preparation techniques may result in the preferential recovery of either the lighter or heavier components, resulting in the "loss" in the opposite end. Neither of these factors will cause the selective loss of intermediate components. The unexplainable absence of components from the middle of a pattern is generally sufficient grounds for a negative finding. Possible explanations for missing intermediate compounds include low sample concentration (compound below detection limit), compound did not meet target compound identification criteria (due to distortion of mass spectrum by co-eluting extraneous compound), and, in rare cases, selective loss due to digestion by microbes. Any such explanation for loss of compounds in the middle of a pattern must be scientifically supportable, and efforts should be made, if possible, to retrieve evidence of their existence from the data file or by reanalyzing the sample.

11.4 The presence of small quantities of some components common to a particular class of ignitable liquid product does not necessarily indicate the presence of that liquid in the debris at the time of the fire.

11.4.1 For example, the pyrolyzates of aromatic-containing polymers may include toluene and xylenes. The pyrolyzates of polyolefin plastic may include a homologous series of normal alkanes.

11.5 Certain ignitable liquid components may be found in some substrates common to fire scenes.

11.5.1 Examples include: normal alkane products found in linoleum and in carbonless paper forms; distillates found in some printed materials; and solvents used in some adhesives and coatings.

11.5.2 If there is suspicion that an ignitable liquid found might be indigenous to the substrate, the testing of an appropriate comparison sample, if available, may aid in determining whether or not an ignitable liquid is foreign to the substrate.

11.6 All of the components necessary for an identification by Practice E 1387 must be present in order for an identification to be made by this test method.

## 12. Report

12.1 Forensic laboratory reports must contain the following information: Identifying case reference numbers, the submitting agency's name and address, the date(s) of sample delivery, the name of the person(s) making the requested analysis, an itemized list describing the submitted samples, and the result of the laboratory examination.

12.1.1 The description of the evidence would seem to be merely a clerical matter, but it is important that the analyst be sure that the evidence is described accurately, not simply as it was identified by the submitting agent. Fire debris samples, especially, tend to appear similar from the outside. It is possible for samples coming from different locations within a fire scene, or even from different fire scenes, to be confused with each other.

12.1.1.1 While it may not be possible for the analyst to distinguish by visual inspection the difference between carpeting from the living room and carpeting from the hallway, it is possible to determine by visual inspection the difference between bedding from the master bedroom and carpeting from the hallway or concrete from the basement. The information that the analyst puts into the report should by verified by the analyst to the extent possible. An analyst's first hand observations, and information supplied by a submitter when a sample is delivered, should be easily distinguishable.

12.2 The results section should state which preparation techniques were used and which analytical techniques were used.

12.2.1 The results section should state list examples of commercial products and/or substrates that might contain the ignitable liquid identified.

12.3 The conclusion should give the scientist's opinion as to whether an ignitable liquid was identified in the sample. If a negative result was obtained, a disclaimer to the effect that negative results do not preclude the possibility that ignitable liquids were present at the fire scene may help to avoid misunderstanding by readers of the report.

12.3.1 In the case of a positive report, it may be appropriate to add a disclaimer to the effect that 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.

12.3.2 A conclusion, summarizing the results in terms understandable to a lay person, may be added to the report.

12.4 Certain words should not appear without explanation within the report. All extracts from organic materials are likely to contain" hydrocarbons." The word "hydrocarbon" should not appear in a report unless those hydrocarbons can be specifically identified and classified. The phrase "hydrocarbons from an unknown source" is expressly prohibited. Similarly, words such as "consistent with,"" in the boiling range of,"

"similar to," or "characteristic of" a particular ignitable liquid should not be used unless that liquid has been positively identified using the methods described in Section 10.

12.4.1 The analyst cannot determine the source of intended use of an ignitable liquid residue. For this reason, residues should not be characterized as "accelerants" by the analyst.

## 13. Precision and Bias

13.1 Since this is a qualitative test method, the terms precision and bias do not apply.

## 14. Keywords

14.1 fire debris samples; forensic sciences; gas chromatography; ignitable liquid residues; mass spectrometry

## ANNEX

**(Mandatory Information)**

## A1. SAMPLE STORAGE

A1.1 Store the original sample after extraction using appropriate procedures for handling and documentation.

A1.1.1 *Extract Storage Short-Term*—Extracts may be stored in a refrigerator in a stoppered tube to prevent evaporation.

A1.1.2 *Extract Storage Long-Term*—Long-term stability can be obtained by adding activated charcoal to the solvent containing the extract and allowing the solvent to slowly evaporate. The sample can later be reconstituted by addition of the solvent. A charcoal adsorption package, such as a C-strip, may serve the same purpose as added charcoal.