



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

This standard is issued under the fixed designation E1618; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) 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 Although this test method is suitable for all samples, it is especially appropriate for extracts that contain high background levels of substrate materials or pyrolysis and combustion products. This test method is also suitable for the identification of single compounds, simple mixtures, or non-petroleum based ignitable liquids.

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

1.4 *This 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:*<sup>2</sup>

[E1386 Practice for Separation 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 Dy-](#)

[namic Headspace Concentration](#)

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

[E2451 Practice for Preserving Ignitable Liquids and Ignitable Liquid Residue Extracts from Fire Debris Samples](#)

## 3. Summary of Test Method

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

3.2 Post-run data analysis generates extracted ion profiles (mass chromatograms) characteristic of the chemical compound types commonly found in ignitable liquids. Additionally, specific 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 TCCs, 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 seven major classifications or one miscellaneous class, as described in this test method.

## 4. Significance and Use

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

4.1.1 The identification of an ignitable liquid residue in a fire scene does not necessarily lead to the conclusion that a fire was incendiary in nature. Further investigation may reveal a legitimate reason for the presence of ignitable liquid residues.

<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.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

4.2 Materials normally found in a building, upon exposure to the heat of a fire, will form pyrolysis and combustion products. Extracted ion profiling and 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 (see 6.4) is adequately separated.

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

5.2 *Mass Spectrometer*—Capable of acquiring mass spectra from  $m/z$  40 to  $m/z$  400 with unit resolution or better, with continuous data output. Values above  $m/z$  40 may not be sufficient to detect or identify some lower molecular weight compounds; for example, methanol, ethanol, acetone.

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

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

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

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

5.4 *Syringes*:

5.4.1 *For liquid samples*, a syringe capable of introducing a sample size in the range from 0.1 to 10.0  $\mu\text{L}$ .

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

## 6. Chemicals, Reagents, and Reference Materials

6.1 *Purity of Reagents*—Reagent grade or better chemicals shall be used in all tests. Unless otherwise indicated, it is

intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. 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.

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

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

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

6.4 *Test Mixture*—The test mixture shall consist of a minimum of the even-numbered normal alkanes (ranging from *n*-octane through *n*-eicosane), methylbenzene (toluene), 1,4-dimethylbenzene (*p*-xylene), 1-methyl-2-ethylbenzene (*o*-ethyltoluene), 1-methyl-3-ethylbenzene (*m*-ethyltoluene), and 1,2,4-trimethylbenzene (pseudocumene). Additional compounds 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 microliters/milliliter) in the chosen solvent (see 6.2). A typical chromatogram of the test mixture is shown in Fig. 1.

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

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

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

## 7. Equipment Calibration and Maintenance

7.1 Verify the consistent performance of the chromatographic instrument by using blanks and known concentrations of either prepared test mixture or other known ignitable liquids. Optimize gas flows periodically.

7.2 Tune and calibrate mass spectrometer.

7.2.1 Tune the mass spectrometer using perfluorotributylamine (PFTBA), or another appropriate calibration standard, according to the instrument manufacturer's specifications, prior to use. This should be done at least every day that the instrument is used or 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 basis to avoid sample contamination by carryover of residual material from previous sample injections.

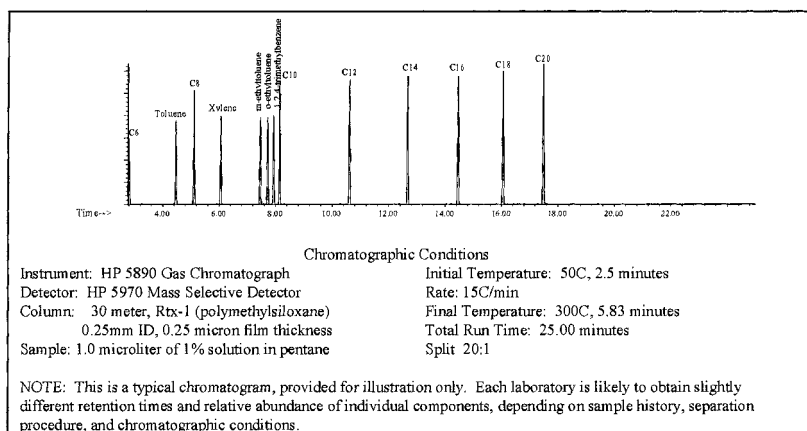


FIG. 1 Test Mixture Containing C8-C20 Normal Hydrocarbons, toluene, p-xylene, o-ethyltoluene, m-ethyltoluene, and 1,2,4-trimethylbenzene

TABLE 1 Ignitable Liquid Classification Scheme<sup>A</sup>

Class	Light (C <sub>4</sub> -C <sub>9</sub> )	Medium (C <sub>8</sub> -C <sub>13</sub> )	Heavy (C <sub>8</sub> -C <sub>20+</sub> )
Gasoline-all brands, including gasohol and E85		Fresh gasoline is typically in the range C <sub>4</sub> -C <sub>12</sub>	
Petroleum Distillates (including De-Aromatized)	Petroleum Ether Some Cigarette Lighter Fluids Some Camping Fuels	Some Charcoal Starters <sup>B</sup> Some Paint Thinners Some Dry Cleaning Solvents	Kerosene Diesel Fuel Some Jet Fuels Some Charcoal Starters
Isoparaffinic Products	Aviation Gas Some 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 Some Lamp Oils	Some Insecticide Vehicles Some Lamp Oils Industrial Solvents
Normal-Alkanes Products	Solvents Pentane Hexane Heptane	Some Candle Oils Some Copier Toners	Some Candle Oils Carbonless Forms Some Copier Toners
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 Some Specialty Products	Some Blended Products Some Specialty Products

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

<sup>B</sup> Many of the examples can be prefaced by the word "some," as in "some charcoal starters."

## 8. Sample Handling Procedure

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

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

8.3 Because of the volatility of solvents and analytes, care must be taken to ensure that samples do not evaporate or otherwise change composition prior to analysis. Septum seal

vials may be used for short term storage of any solvents or extracts. See Annex A1 for sample and extract storage guidance.

8.3.1 Avoid the use of water as a sealant.

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

8.5 Clean syringes thoroughly between injections to ensure no carryover.