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

This standard is issued under the fixed designation E1618; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

~~^{e1}Note~~ Editorial changes were made in November 2006.

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 the standard. ~~The values given in parentheses~~ No other units of measurement are for information only, 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:²

~~E1385 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Steam Distillation~~

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

~~E1387 Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography~~

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

~~E2154 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Solid Phase Microextraction (SPME)~~ 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 ~~target compound chromatograms (TCC)~~, TCCs, or combination thereof,

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

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.

4.1.2 ~~Due to~~ 4.1.2 Because of 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 (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 must 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 must 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 must 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 μ 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 (*m*-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 must 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

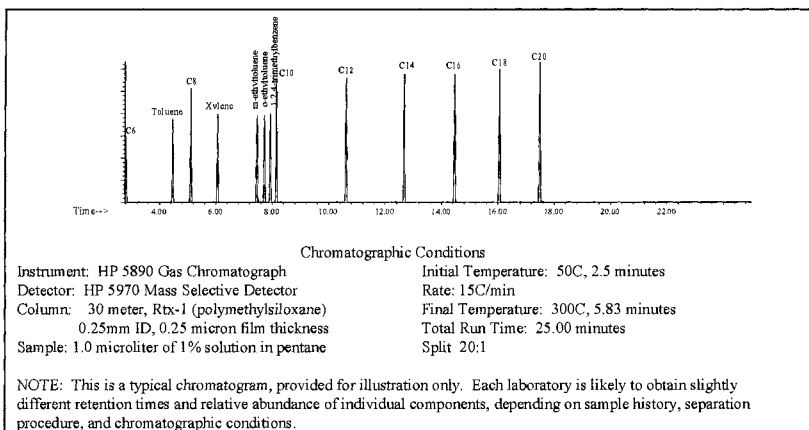


FIG. 1 Test Mixture Containing C8-C20 Normal Hydrocarbons, toluene, p-xylene, o-ethyltoluene, m-ethyltoluene, and 1,2,4-trimethylbenzene

TABLE 1 Ignitable Liquid Classification Scheme^A

Class	Light (C ₄₋₉)	Medium (C _{8-C₁₃})	Heavy (C _{8-C₂₀₊})
Class	Light (C _{4-C₉})	Medium (C _{8-C₁₃})	Heavy (C _{8-C₂₀₊})
Gasoline-all brands, including gasohol	Fresh gasoline is typically in the range C ₄ -C ₁₂		
Gasoline-all brands, including gasohol and E85	Fresh gasoline is typically in the range C ₄ -C ₁₂		
Petroleum Distillates (including De-Aromatized)	Petroleum Ether 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
Isoparaaffinic 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- Paraaffinic 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

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

^B Many of the examples can be prefaced by the word "some," as in "some charcoal starters."

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.

8. Sample Handling Procedure

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

8.2 Methods for isolating ignitable liquid residues from fire debris for analysis by this test method are described in Practices E1386, E1385, E1388, E1386, E1388, E1412, E1413, and E2154, E1412, E1413, and E2154.

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 dimethyldichlorosilane (DMCS)-treated columns.

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

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.

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

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

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

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

9. Data Analysis

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

9.1.1 The essential requirement for making a classification using this procedure is the matching of the sample chromatogram with a reference ignitable liquid chromatogram obtained under similar conditions, noting points of correlation or similarities.

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

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

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

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

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

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

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

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

9.1.78 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 relative intensity display for ions of significantly different abundance. It is best to overlay these mass chromatograms or use an absolute intensity output.

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

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

9.3 *Target Compound Analysis (TCC)(TCA):*

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 shall 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, provided 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. A single bar on the graph depicts each target compound.

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 that are 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 shall be made by the analyst on the basis of the mass spectra and relative retention times of the components in question by comparison to reference materials.

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

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

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

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

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

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

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

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-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-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

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

10. Ignitable Liquid Classification Scheme

10.1 Seven major classes of ignitable liquids may be identified by gas chromatography, mass spectrometry, extracted ion