Designation: E1388 – 12

Standard Practice for
Sampling of Headspace Vapors from Fire Debris Samples[1]

This standard is issued under the fixed designation E1388; 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 practice describes the procedure for removing small quantities of ignitable liquid vapor from samples of fire debris by sampling the headspace of the debris container.

1.2 Separation and concentration procedures are listed in the referenced documents. (See Practices E1386, E1412, E1413, and E2154.)

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

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

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

3. Summary of Practice

3.1 The sample, preferably in its original container, is heated in order to volatilize any ignitable liquid residues present in the debris. After heating, the headspace is sampled and analyzed by gas chromatography-mass spectrometry.

4. Significance and Use

4.1 This procedure is particularly useful for screening fire debris samples to determine relative ignitable liquid concentrations and possible ignitable liquid class prior to extraction with other techniques.

4.2 This practice is useful when volatile oxygenated products such as alcohols or lacquer thinners are suspected.

4.3 This practice is the least sensitive of the sample preparation techniques and may not detect quantities of less than 10 µL of an ignitable liquid.

4.4 Because this separation takes place in a closed container, the sample remains in approximately the same condition in which it was submitted. Repeat and interlaboratory analyses are therefore possible.

4.5 Due to variables in the debris sample condition prior to headspace sampling, complete reproducibility of chromatograms may be difficult to obtain.

4.5.1 To obtain greater reproducibility, use one of the separation procedures which results in a solution of the ignitable liquid residue (see Practices E1386, E1412, and E1413).

4.5.2 To obtain greater sensitivity for petroleum distillates and oxygenated products, use solid phase microextraction. (See Practice E2154.)

4.6 High concentrations of highly volatile compounds may swamp the headspace, inhibiting the recovery of less volatile components.

5. Apparatus

5.1 *Heating System*—An oven, heating mantle or hot plate designed to fit the evidence container.

5.2 *Temperature Measuring Device*—A thermometer or thermocouple capable of measuring temperatures in the range from 40 to 150°C.

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[1] This practice in under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics

Current edition approved Feb. 1, 2012. Published March 2012. Originally approved in 1990. Last previous edition approved in 2005 as E1388 – 05. DOI: 10.1520/E1388-12.

[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.

5.3 A gas-tight syringe capable of reproducibility introducing sample sizes in the range from 0.5 to 5 mL.

5.4 Disposable 20 cc syringes.

5.5 Disposable 16–20 gauge syringe needles.

5.6 A drill, punch, or other device to puncture holes in sample containers.

5.7 Headspace sample vials.

6. Sample Preparation

6.1 Observe the appropriate procedures for handling and documentation of all submitted samples as described in Guide E1459 and Practice E1492.

6.1.1 Open and examine the fire debris sample in order to determine that it is consistent with its description.

6.1.1.1 Resolve any discrepancies between the submitting agency's description of the evidence and the analyst's observation prior to the completion of the report.

6.2 Punch or drill a small hole in the container lid and cover the hole with tape.

6.2.1 Alternatively, a rubber sleeve stopper may be placed in the hole.

6.3 If the sample is going to be heated, place the container in the heating system until the container reaches the desired temperature (not to exceed 90°C).

6.4 The syringe may be heated prior to sampling

7. Sampling Procedure

7.1 *Direct Headspace Sampling:*

7.1.1 Remove the container from the heating apparatus then push the syringe through the tape or rubber sleeve stopper into the hole in the container lid. Slowly pump the syringe several times, withdraw from the lid, and inject 0.5 to 2.0 mL of sample into the gas chromatograph. Analyze as described in Test Method E1618.

7.1.2 After the headspace sample has been removed, reseal the hole.

7.2 *Indirect Headspace Sampling Using Headspace Sample Vials:*

7.2.1 Some automated sample injection systems utilize headspace sample vials for delivery of the sample headspace into the gas chromatograph.

7.2.2 Assemble and seal (crimp or screw) lid onto headspace sample vial. Using a 20 cc syringe and needle, puncture the headspace vial septum and evacuate the contents of the vial. Repeat this procedure 3 to 4 times.

7.2.3 Remove the container from the heating apparatus then push the 20 cc syringe through the tape or rubber sleeve stopper into the hole in the container lid. Slowly pump the syringe several times, withdraw 20 cc of vapor and inject into headspace sample vial. Repeat this process 2 to 4 times.

7.2.3.1 If the headspace sample vial was properly sealed and evacuated, the vacuum created should pull the initial injection into the vial. The latter injections into the vial will require forcibly injecting the 20 cc sample.

7.2.3.2 The sequence method on the gas chromatograph should be setup to inject 0.5 to 2.0 mL of the sample into the gas chromatograph. Analyze as described in Test Method E1618.

7.2.4 After the headspace samples have been removed, reseal the hole in the container.

8. Keywords

8.1 fire debris samples; headspace

Designation: E1618 – 14

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 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.

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 practice cannot replace knowledge, skill, or ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgment.

1.5 *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]

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 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)

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.

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[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 July 1, 2014. Published July 2014. Originally approved in 1994. Last previous edition approved in 2011 as E1618 – 11. DOI: 10.1520/E1618-14.

[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.

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 µL.

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.

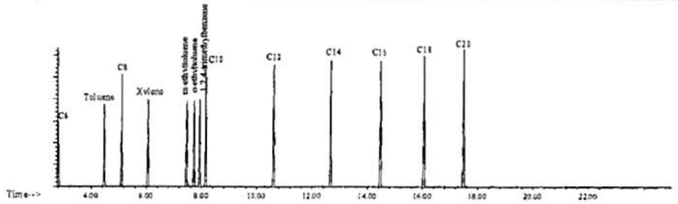
7. Equipment Calibration and Maintenance

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.



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) 0.25mm ID, 0.25 micron film thickness	Final Temperature: 300C, 5.83 minutes 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_9-C_{20+})
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
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

[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."

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-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.

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 samples 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*—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. 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: normal 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.

9.1.8 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.

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
n-Alkylcyclohexanes	82, 83
Aromatic—alkylbenzenes	91, 105, 119, 92, 106, 120, 134
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] 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.

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 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 (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 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.

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. *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-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

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 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.

9.3.2.4 While TCCs provide much useful information, a TCA 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 profiling (or extracted ion profile analysis), or a 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 seven major ignitable liquid classifications.

10.1.3 With the exception of the gasoline class, the major ignitable liquid classes may be divided into three subclasses based on boiling (*n*-alkane) range: light, medium and heavy.

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

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

10.1.3.3 *Heavy product range—n-C_9-n-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 n-C_{11}.

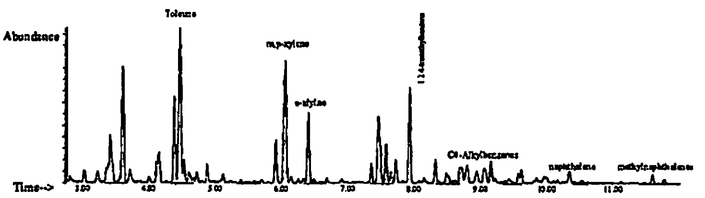
10.1.3.4 It may be necessary to characterize a product as "light to medium," or "medium to heavy," when the carbon number range does not fit neatly into one of the above categories. In such instances, the carbon number range should be reported.

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

10.2.1 *Criteria for the Identification of Gasoline:*

10.2.1.1 *General*—All brands of gasoline including gasohol and E85. Pattern characterized by abundant aromatics in a specific pattern.

10.2.1.2 *Alkanes*—Present. Pattern may vary by brand, grade, and lot.



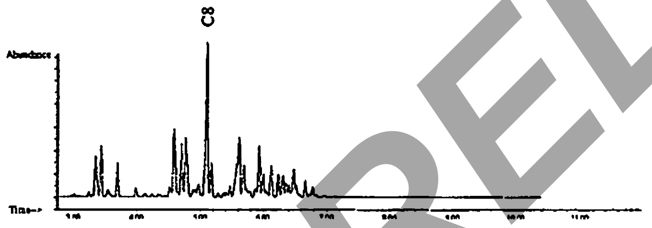
Chromatographic Conditions

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



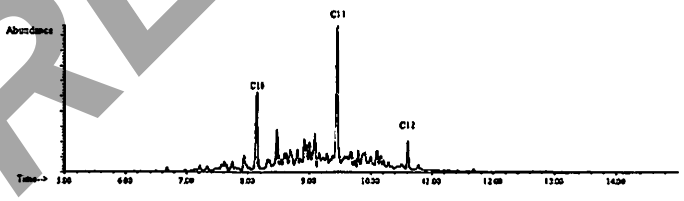
Chromatographic Conditions

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



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

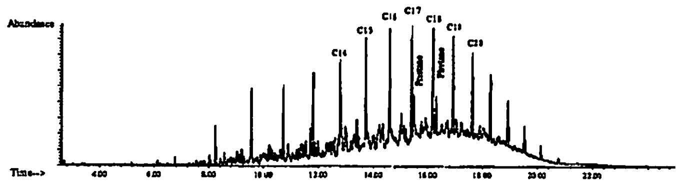
NOTE: This is a typical chromatogram, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundance of individual components, depending on sample history, separation procedure, and chromatographic conditions.

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

10.2.1.3 *Cycoloalkanes*—Not present in significant amounts.

10.2.1.4 *Aromatics*—Petroleum pattern comparable to that 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 shall be present; above *n*-C_7, the aromatic concentration is generally substantially higher than the alkane concentration.

10.2.1.5 *Condensed Ring Aromatics*—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.



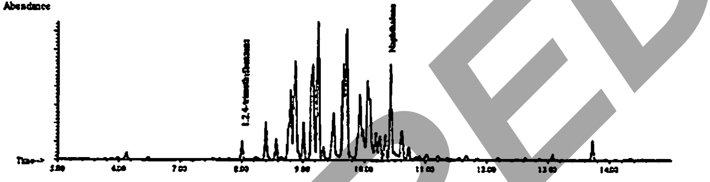
Chromatographic Conditions

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. 5 Example of Heavy Petroleum Distillate; Diesel Fuel



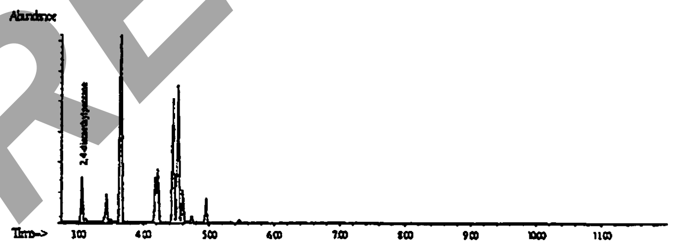
Chromatographic Conditions

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. 6 Example of a Medium Aromatic Solvent; Fuel Additive



Chromatographic Conditions

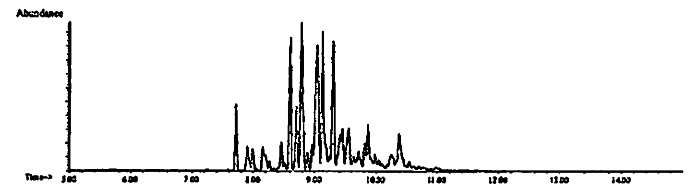
Instrument: HP 5890 Gas Chromatograph
Detector: HP 5970 Mass Selective Detector
Column: 30 meter, Rtx-1 (polymethylsiloxane)
0.25mm ID, 0.25 micron film thickness
Sample: 1.0 microliter of 1% solution in pentane

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. 7 Example of Light Isoparaffinic Product; Aviation Gas

10.2.1.6 **Warning**—The mere presence of alkylbenzenes does not justify an identification of gasoline. These compounds shall 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 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.



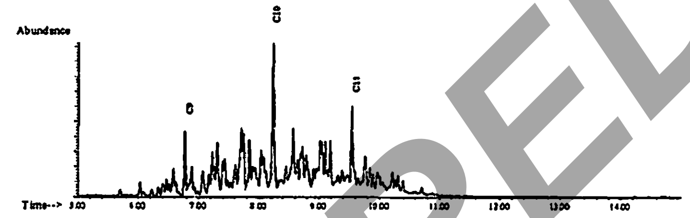
Chromatographic Conditions

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. 8 Example of Medium Isoparaffinic Product; Charcoal Starter



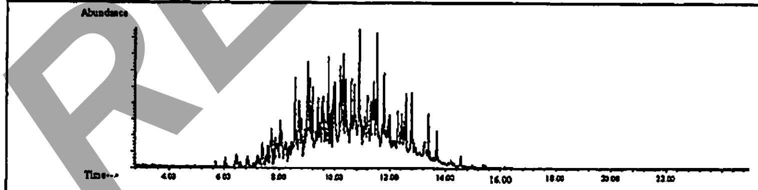
Chromatographic Conditions

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

10.2.2 *Criteria for the Identification of Distillates:*

10.2.2.1 *General*—Traditional distillates and de-aromatized distillates; Predominant pattern associated with a homologous series of *n*-alkanes in a Gaussian distribution of peaks. Light distillates may not exhibit a recognizable pattern and may contain only one or two of the *n*-alkanes.

10.2.2.2 *Alkanes*—Abundant. Predominant normal alkanes present with isoparaffinic compounds present.

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

10.2.2.4 *Aromatics*—Always present in traditional medium and heavy distillates; less abundant than alkanes; pattern and abundance varies by boiling range and peak spread; may be present in light distillates. In some products, the aromatic composition may be significantly reduced or completely absent (de-aromatized).

10.2.2.5 *Condensed Ring Aromatics*—May be present based on boiling range and peak spread.

10.2.3 *Criteria for the Identification of Isoparaffinic Products:*

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

10.2.3.2 *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.

10.2.3.3 *Aromatics*—Absent, or not present in significant concentrations.

10.2.3.4 *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 isoparaffins maybe be present, but significantly less abundant than the alkane pattern.

10.2.3.5 *Condensed Ring Aromatics*—Not present.

10.2.4 *Criteria for the Identification of Aromatic Products:*

10.2.4.1 *General*—Products comprised almost exclusively of aromatic or condensed ring aromatic compounds, or both. The boiling range and pattern are dependent on the specific formulation.

10.2.4.2 *Alkanes*—Not present in significant amounts.

10.2.4.3 *Cycloalkanes*—Not present in significant amounts.

10.2.4.4 *Aromatics*—Abundant. Pattern comparable to known aromatic products. Pattern depends on formulation.

10.2.4.5 *Condensed Ring Aromatics*—May be present, pattern depends on formulation. Pattern comparable to known aromatic product.

NOTE 1—Light aromatic products may consist of single or few components. These compounds shall be identified by both GC retention time and mass spectral identification.

10.2.4.6 **Warning**—The relative intragroup ratios of the isomers of xylenes and C_3-alkylbenzenes do not vary significantly among petroleum products. Therefore, the relative ratios of these compounds should match, or nearly match, the ratios found in petroleum products, if they are to be reported. Further, in the presence of styrene at a concentration significantly higher than that of toluene or xylenes, the finding of toluene or xylenes should not be considered significant.

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

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

10.2.5.2 *Alkanes*—Abundant. Branched alkanes present while normal alkanes may be notably absent or at diminished levels compared to distillate products. Pattern comparable to known naphthenic-paraffinic products.

10.2.5.3 *Cycloalkanes*—Abundant. Pattern comparable to known naphthenic-paraffinic products.

10.2.5.4 *Aromatics*—Not present in significant amounts.

10.2.5.5 *Condensed Ring Aromatics*—Not present in significant amounts.

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

10.2.6.1 *General*—Products comprised exclusively of *n*-alkanes. The boiling range and pattern are dependent on the specific formulation.

10.2.6.2 *Alkanes*—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.

10.2.6.3 *Cycloalkanes*—Not present in significant amounts.

10.2.6.4 *Aromatics*—Not present in significant amounts.

10.2.6.5 *Condensed Ring Aromatics*—Not present in significant amounts.

NOTE 2—All major chromatographic peaks for this class shall be identified by both GC retention times and mass spectral characteristics.

10.2.7 *Criteria for the Identification of Oxygenated Solvents:*

10.2.7.1 *General*—Products containing major oxygenated components may include mixtures of oxygenated compounds and other compounds or products. Major oxygenated compounds typically present before C8; major compound(s) may include alcohols, esters, and ketones. Other major compounds including toluene, xylene, and distillate formulations may also be present.

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

10.2.7.3 *Cycloalkanes*—Pattern depends on formulation.

10.2.7.4 *Aromatics*—Pattern depends on formulation.

10.2.7.5 *Condensed Ring Aromatics*—Not significant.

NOTE 3—All major oxygenated compounds shall be identified by GC retention times and mass spectral characteristics.

10.2.7.6 **Warning**—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 matrix peaks in the chromatogram) before the analyst should consider the finding of an oxygenated product significant.

10.2.8 *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; GC retention times and mass spectra of the components are 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, 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 or combustion of materials at the fire scene yields target compounds or compounds of the same type as those being compared. All of these circumstances shall be taken into account by the analyst during visual pattern evaluation. Therefore, it is imperative that the analyst has 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 oxygenated compounds, 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 one-half an order of magnitude when viewed in the alkene profile) 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 shall 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 some 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 an ignitable liquid is foreign to the substrate.

12. Report

12.1 This test method does not require a specific format for a forensic laboratory report for fire debris samples, but does indicate what specific information to include in a report.

12.1.1 Regardless of the format, a report shall include the following information:

(1) Unique case reference number.
(2) Identity of the laboratory issuing the report and the report date.
(3) Submitting agency's (requestor's) information.
(4) Date of submission to the laboratory.
(5) Itemized list of the submitted items.
(6) Description of the separation/extraction technique(s) employed.
(7) Description of the analytical technique(s) employed.
(8) Conclusion stating the result(s) of the laboratory examination(s).
(9) Name and signature of the individual(s) responsible for the analysis.

12.1.2 A report may include the following information:

(1) Statement of the analysis performed.
(2) Incident summary.
(3) Manner of submission to the laboratory.
(4) Disposition of the submitted items.
(5) Qualifying statements.
(6) Additional information or remarks.

NOTE 4—A sample report is provided as Annex A2.

12.2 *Criteria for Report Contents:*

12.2.1 *Unique Case Reference Number*—The report shall contain a case identifying number, such as a laboratory case number or agency case file number, which is unique to the submitted items.

12.2.1.1 In a report that contains multiple submissions (multiple case reference numbers), items shall be referenced by their item number and unique case reference number.

12.2.2 *Identity of the Laboratory Issuing the Report and the Report Date*—The report shall contain the name of the laboratory responsible for issuing the report and the date of the report. The report may also include the address and phone number of the issuing laboratory. The information may appear in the letterhead.

12.2.3 *Submitting Agency's (or Requestor's) Information*—The report shall contain the submitting agency's (or requestor's) name and may also include the agency's (or requestor's) case identifier, address, and investigator information.

12.2.4 *Date of Submission to the Laboratory*—The report shall contain the date the evidence was received by the laboratory.

12.2.5 *Itemized List of the Submitted Items*—The report shall include an itemized list describing the items received.

12.2.5.1 Each item shall be uniquely identified and include a brief description of the container type and its contents.

12.2.5.2 Each item shall be described as observed by the analyst, not simply as it appears on container labels or within the submitting paperwork.

12.2.5.3 The report shall describe the contents of each container as observed by the analyst or as the contents would appear to the court. Some contents may be unrecognizable and described in only general terms. For example: broken glass and burned carpet, clear yellow liquid, or charred unidentified debris.

12.2.5.4 The item description may reflect information based upon the submitter's description but shall be qualified by adding a statement such as "reported as" or "labeled as." The analyst's personal observations and descriptions of the evidence shall be easily distinguishable from the submitter's information, such as using quotation marks to identify the submitter's remarks.

12.2.6 *Description of the Separation/Extraction and Analytical Techniques Employed*—The report shall contain a brief description of the separation, extraction, concentration, and analytical techniques used. The appropriate ASTM standard(s) may be cited in lieu of a description.

12.2.7 *Conclusion Stating the Result(s) of the Laboratory Examination(s)*—The report shall contain a conclusion which clearly states the result(s) of the examination(s) in terms understandable to an investigator or lay person.

12.2.7.1 The results/conclusions section shall state the analyst's opinion as to whether or not an ignitable liquid or residue was identified/classified in the sample and the identity/classification of the liquid/residue.

12.2.7.2 A single results/conclusions section of the report may serve as the opinion in the report. A separate opinion section may appear in a report; however, restating the results in an opinion section is not necessary.

12.2.7.3 There is no universally accepted set of terms used in fire debris analysis conclusions. The results shall be scientifically accurate and convey the proper significance of the conclusions. Refer to Section 12.3 for acceptable terminology and phrases used in results/conclusions.

12.2.8 *Name and Signature of the Individual(s) Responsible for the Analysis*—The report shall contain the name and signature of the individual(s) responsible for the results, conclusions, or opinions within the report. The signature(s) may be electronic.

12.2.8.1 The signature(s) of the technical reviewer(s) may also appear on the report.

12.2.9 Reports may include the following:

12.2.9.1 *Statement of the Analysis Performed*—The report may include a statement or title reflecting the type of analysis conducted as per the request of the submitting agency or contributor. For example: Fire Debris Analysis Report, Chemical Analysis Report, Ignitable Liquid Tests, or Results of Fire Debris Analysis.

12.2.9.2 *Incident Summary*—Information such as date, location, victim(s), or subject(s) may be included to identify or associate the submitted items to an incident.

12.2.9.3 *Manner of Submission to the Laboratory*—The report may contain the method of delivery, such as personal delivery or registered mail.

12.2.9.4 *Disposition of the Submitted Items*—The report may contain the disposition of the submitted items. Examples of disposition are: returned to contributor, preserved, destroyed, and destroyed in 30 days unless otherwise notified.

12.2.9.5 *Qualifying Statements*—The laboratory/author may include an appropriate note, qualifier, or disclaimer in the report, examples of which appear in Section 12.3.

12.2.9.6 *Additional Information*—Remarks, special instructions, requests, or additional information may appear within a remarks section of the report. Example comments include: references to commercial products, requests for comparison samples, additional testing that may or can be performed upon receipt of additional items, and the description of secondary evidence.

12.2.9.7 Requested examinations by the submitting agency (requestor) that were not conducted may be noted with a reason for not performing the examination.

12.3 *Terminology Used in Reporting Results/Conclusions and Opinions*—There are many appropriate phrases and words that may be used to describe the results and conclusions of a fire debris or ignitable liquid analysis. The analyst has many options in reporting a positive or negative finding of an ignitable liquid or residue.

12.3.1 *Reporting Positive Results of an Ignitable Liquid or Residue:*

12.3.1.1 Some possible phrases include: was present in, was detected on, was identified in, was recovered from, and was found in. The meaning of all these phrases are interchangeable and represent a positive result or identification/classification of an ignitable liquid or ignitable liquid residue using the methods described in Sections 9, 10, and 11.

NOTE 5—Regardless of the choice of phrases used, there is no implied difference in the perceived level of confidence for a positive result.

12.3.1.2 The reporting of a positive result does not preclude the contribution of ignitable liquids from the matrix of other material present in the debris. In general, the source of the ignitable liquid cannot be determined and therefore shall not be stated. It may be appropriate to add a qualifying statement to the report finding. Refer to 12.3.6.

12.3.1.3 The results section may list examples of commercial products or substrates, or both, that might contain the ignitable liquid identified. Examples of positive results are:

> A medium petroleum distillate (MPD) was detected in Item #1. Examples of commercial products that contain MPDs include some charcoal lighters and paint thinners.

> Gasoline was identified on Item #2.

12.3.2 *Report Terminology Using the Ignitable Liquid Classification Scheme (Table 1):*

12.3.2.1 *Gasoline* is a distinct class of ignitable liquid and shall be named as such in the report.

12.3.2.2 *Petroleum Distillates*—These products are distilled from petroleum (crude oil) and are consequently named petroleum distillates. They are further characterized by their *n* -alkane range. Table 1 classifies distillates into light, medium, and heavy distillates based upon their n-alkane range. Some distillates may not fit clearly into a single category, so it may be necessary to describe the ignitable liquid as "light to medium," "medium to heavy," or simply state the *n* -alkane range of the material.

(1) The report may list examples of commercial products or substrates that may contain such liquids. For example:

> A heavy petroleum distillate (HPD) was recovered from Item #22. Examples of HPDs include kerosene, diesel fuel, and some charcoal starters.

> A medium petroleum distillate in the range of C10 to C13 was detected in Item #4. Examples of this distillate include some paint thinners and some specialty solvents. Medium petroleum distillates are also found in some shoe polish, wood staining products, insecticides, and automotive cleaner products.

12.3.2.3 *Other Ignitable Liquids*—Commercial products can be variations of petroleum products or derived from non-petroleum sources. These include: isoparaffinic products, aromatic products, naphthenic-paraffinic products, *n*-alkane products, oxygenated products, and miscellaneous products.

(1) These products may also be reported by assigning an *n*-alkane range, or may be classified as light, medium, or heavy. For example:

> A medium isoparaffinic product like that found in some lamp oils or toner solvents was present on Item #7.

12.3.2.4 *Single Compounds or Simple Mixtures*—Some commercial products contain a single compound or mixture of a few compounds. These ignitable liquids shall be reported based upon identification of the compounds rather than classification. For example:

> Isopropyl alcohol and methyl alcohol were identified in Item #7 (plastic bottle of clear liquid).

12.3.2.5 *Terminology for Residues of Ignitable Liquids*—In many cases components of an ignitable liquid may be reduced or lost due to evaporation or other environmental condition, such as microbial degradation. The terms "residue," "weathered," or "evaporated" may be used to refer to recovered products. However, do not use a term such as "fire-aged" as it implies that fire was the only reason for the recovered condition of the ignitable liquid.

12.3.3 *Special Materials and Matrices:*

12.3.3.1 Debris and matrices of the submitted items sent in for fire debris analysis may contain ignitable liquids. Some commercial products that are ignitable liquids, such as turpentine, toluene, polish removers, rubbing alcohol, and some cleaning solvents are also common to items submitted for analysis, such as wood (terpenes), shoes (toluene), and clothing (alcohols).

12.3.3.2 The analyst shall use caution and not report positive results if the submitted items may contain a matrix which is known to contain an ignitable liquid of the type detected. If the laboratory elects to report matrix compounds, such as terpenes in wood and alcohols in clothing, the report shall reflect the nature of the ignitable liquid. Examples of report conclusions for items with matrices that contain known ignitable liquids include:

> No ignitable liquids were detected on Items #7 and #8 (sneakers) other than compounds associated with the items.

> Toluene was identified on Items #3 and #4 (sneakers). Toluene is found in glue commonly used in the manufacturing of shoes.

> No ignitable liquids were detected on Item #11 (pine studs) except for terpenes which are common to softwoods.

12.3.4 *Reporting Negative Findings of an Ignitable Liquid:*

12.3.4.1 Several phrases may be used when no ignitable liquid was identified on an item. The word "detected" could be used to describe no response great enough to be identified by gas chromatography-mass spectrometry, while the word "identified" may be used when compounds are detected but are not associated with an ignitable liquid and most likely correspond to interfering products. Examples include:

> No ignitable liquids were detected in Item #18.

> No ignitable liquids were identified on Item #6.

12.3.4.2 The use of the phrase "No ignitable liquids were present" is not recommended as an ignitable liquid may be present below the detectable limits of the method.

12.3.4.3 For clarification purposes, the analyst may elect to report to the submitter that something other than an ignitable liquid was detected or identified. For example:

> The analysis identified volatile compounds consistent with the interfering products associated with a variety of synthetic materials. These compounds may be responsible for the positive alert on the investigator's monitoring equipment at the scene.

12.3.5 *Misleading Terminology:*

12.3.5.1 Certain words shall not appear without explanation within the report. All extracts from organic materials are likely to contain "hydrocarbons." The word "hydrocarbon" shall 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 shall not be used unless that liquid has been positively identified and classified using the methods described in Sections 9, 10, and 11. Examples of misleading terminology include:

> Item #22 contained hydrocarbons in the range of gasoline.

> Hydrocarbons from an unknown source were detected in Item #12.

> Flammable compounds were present on Item #6.

> Some components consistent with gasoline were identified on Item #9.

12.3.5.2 Common interfering compounds resulting from pyrolysis, combustion, or distillation of a substrate at the fire scene are not normally reported except when a significant quantity of an unexplainable product is detected.

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

12.3.6 *Qualifying Statements:*

12.3.6.1 A finding of either a positive or negative result/conclusion shall be scientifically accurate and supported by the analytical data. Positive and negative results often cannot be reported in simple terms because of the presence of matrix and interfering compounds. The analyst may use qualifiers or disclaimers to convey the proper significance of both positive and negative results.

12.3.6.2 Qualifying statements shall be used when necessary and not routinely.

12.3.6.3 Examples of qualifying statements that may be used with positive findings when the matrix cannot be ruled out as a contributor of an ignitable liquid:

> 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.

(1) If the analyst or laboratory elects to report a positive ignitable liquid finding of a known matrix component, a qualifying statement shall be used. For example:

> A medium isoparaffinic product like that found in some lamp oils or toner solvents was present on Item #7.Toluene was identified on Item #5 (tennis shoes). Toluene is an ignitable liquid but is a component of some glues used in the manufacturing of shoes.
>
> Terpenes were identified in Item #8 (charred wood and debris). Terpenes are a natural component in some softwoods and are also found in turpentine solvents and pine-based cleaners.

12.3.6.4 An example of a qualifying statement that may be used with negative findings:

> The absence of an ignitable liquid residue does not preclude the possibility that ignitable liquids were present at the fire scene. Ignitable liquids are volatile compounds that may have evaporated, been totally consumed in a fire, environmentally altered or removed, or otherwise indistinguishable from background materials.

(1) A qualifying statement shall only include remarks appropriate for the specimen(s) analyzed. For example, if an item was not burned or involved in a fire, the qualifying statement shall not reference having "been totally consumed in a fire."

12.3.7 *Additional Remarks:*

12.3.7.1 Additional information may be included in a report for further clarification. For example, if a distinctive set of compounds were identified in a sample, the following additional investigative information could be given:

> Examples of commercial products that contain methyl alcohol, acetone, toluene, and methylene chloride include some paint and varnish removers.
>
> The ignitable liquid and components identified in Item #2 are consistent with the ingredients found in "John Smith's Oven Cleaner" or similar product.

12.3.7.2 Other information or comments that may appear in the remarks section include contact information of the analyst (phone number), websites and references to product information, additional testing, testing that could not be conducted, and damage to the submitted items during shipment/delivery. Examples include:

> The liquid in Item #6 (bottle) contained a mixture of gasoline and a medium petroleum distillate. The request to determine the flashpoint of the liquid was not conducted as this laboratory does not have the required equipment to conduct the requested test.
>
> Items #1 through #5 were not examined for the presence of ignitable liquids because they were improperly packaged. Samples from fire scenes to be tested for ignitable liquids should be packaged in metal paint cans, heat-sealable arson bags, or glass jars.
>
> Item #3 was not examined as the glass container was broken upon receipt by the laboratory.

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

ANNEXES

(Mandatory Information)

A1. SAMPLE AND EXTRACT STORAGE

A1.1 After extraction, store the original sample using appropriate procedures for handling and documentation.

A1.1.1 *Extract Storage Short-Term*—Extracts, liquid samples, or solvents may be stored in septum seal or screw cap glass vials with PTFE-lined seals to prevent evaporation.

A1.1.2 *Extract Storage Long-Term*—Follow the procedures set forth in Practice E2451 for long-term storage of extracts or liquid samples.

A2. SAMPLE LABORATORY REPORT

A2.1 See Table A2.1.

INTERNATIONAL

Designation: E1386 − 15

Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction[1]

This standard is issued under the fixed designation E1386; 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 practice covers the procedure for removing small quantities of ignitable liquid residue from samples of fire debris using solvent to extract the residue.

1.2 This practice is suitable for successfully extracting ignitable liquid residues over a wide range of concentrations.

1.3 Alternate separation and concentration procedures are listed in the referenced documents (Practices E1388, E1412, E1413, and E2154).

1.4 This practice offers a set of instructions for performing one or more specific operations. This standard cannot replace knowledge, skill, or ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgment.

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

1.6 *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 hazard statement, see 5.5.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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 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

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 Practice

3.1 A sample of fire debris is extracted with an organic solvent. The extract is filtered and concentrated as necessary.

4. Significance and Use

4.1 This practice is useful for preparing extracts from fire debris for later analysis by gas chromatography-mass spectrometry (GC/MS).

4.2 This is a very sensitive separation procedure, capable of isolating quantities smaller than 1 μL of ignitable liquid residue from a sample.

4.3 This practice is particularly useful when the potential for fractionation during separation must be reduced, as when attempting to distinguish between various grades of fuel oil.

4.4 This practice is particularly useful for the extraction of nonporous surfaces such as glass, or the interior of burned containers. It is also particularly well suited to the extraction of ignitable liquid residues from very small samples, very large samples, or samples that are not suitable for heating.

4.5 This practice is not specific to ignitable liquids and can be hampered by coincident extraction of interfering compounds present in the fire debris samples.

4.6 This practice may not be useful for the extraction of some extremely volatile ignitable liquids, which may evaporate during the concentration step.

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[1] This practice is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of E30.01 on Criminalistics

Current edition approved May 1, 2015. Published June 2015. Originally approved in 1990. Last previous edition approved in 2010 as E1386 – 10. DOI: 10.1520/E1386-15.

[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.

4.7 This is a destructive technique. Whenever possible, this technique should only be used when a representative portion of the sample can be preserved for reanalysis. Those portions of the sample subjected to this procedure may not be suitable for resampling. If destruction of the sample is an issue, consider using passive headspace concentration as described in Practice E1412.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade or better chemicals shall be used in all tests. 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.[3] 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.

5.2 *Solvent*—A suitable single component solvent (polar or non-polar), with high volatility to allow concentration by evaporation, such as carbon disulfide, pentane, methylene chloride, or diethyl ether.

NOTE 1—Polar oxygenated solvents (such as alcohols) may not be soluble in non-polar solvents (such as pentane). In order to examine for the presence of polar oxygenated solvents, it may be necessary to perform an additional extraction technique, such as analyzing a sample from the headspace (Practice E1388) prior to performing a solvent extraction.

5.2.1 Check solvent purity by evaporating to at least twice the extent used in the analysis and analyzing the evaporated solvent in accordance with Test Method E1618.

5.2.2 Read and follow the safety precautions described in the safety data sheet (SDS) of the extraction solvent that is used.

5.3 *Filter paper or filter apparatus,* free of extractable hydrocarbons.

5.4 *Beakers, vials, or other extraction containers,* free of extractable hydrocarbons.

5.5 *Compressed dry nitrogen, filtered air, or inert gas.* (**Warning**—These gases are stored under high pressure.)

6. Procedure

6.1 Observe the appropriate procedures for handling and documentation of all submitted samples as described in Practice E1492 and Guide E1459.

6.1.1 Open and examine the fire debris sample in order to determine that it is consistent with its description.

6.1.1.1 Resolve any discrepancies between the submitting agent's description of the evidence and the analyst's observation with the submitting agent prior to the completion of the report.

6.2 Select an appropriate size container to perform the extraction.

6.3 If possible, select a representative portion of the sample to extract.

6.3.1 Record in the case notes a description of the portion of the sample extracted.

6.4 Add sufficient solvent to thoroughly moisten the sample.

6.5 A record of the solvent manufacturer, grade and lot number used shall be maintained.

6.6 Mix the solvent and debris. An extraction time of less than one minute is usually sufficient.

6.6.1 Simple rinsing of nonporous surfaces may be sufficient for adequate extraction of residues.

6.6.2 Multiple small volume extractions may be used; this technique is more efficient than a single large volume extraction. The multiple small volumes may be combined and concentrated as necessary.

6.7 Decant the solvent and filter if necessary.

6.8 If needed, evaporate the solvent to an appropriate volume.

6.8.1 Sufficiently concentrated samples do not require evaporation.

6.8.2 Evaporation techniques may include dry nitrogen, filtered air, inert gas, vacuum techniques or simple evaporation.

6.9 Code or label an appropriately sized vial in accordance with Guide E1459.

6.10 Transfer the solvent into the prepared container and cap.

6.11 Prepare a system blank to approximate the most reduced sample. An equivalent amount of the extraction solvent shall be analyzed (reduced and filtered if necessary) in the same manner as sample(s) under analysis.

6.12 Follow the extract storage procedures described in Practice E2451.

7. Keywords

7.1 fire debris samples; solvent extraction

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[3] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC, http://www.chemistry.org. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, http://www.usp.org.

Designation: E1388 − 12

Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples[1]

This standard is issued under the fixed designation E1388; 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 practice describes the procedure for removing small quantities of ignitable liquid vapor from samples of fire debris by sampling the headspace of the debris container.

1.2 Separation and concentration procedures are listed in the referenced documents. (See Practices E1386, E1412, E1413, and E2154.)

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

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

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

3. Summary of Practice

3.1 The sample, preferably in its original container, is heated in order to volatilize any ignitable liquid residues present in the debris. After heating, the headspace is sampled and analyzed by gas chromatography-mass spectrometry.

4. Significance and Use

4.1 This procedure is particularly useful for screening fire debris samples to determine relative ignitable liquid concentrations and possible ignitable liquid class prior to extraction with other techniques.

4.2 This practice is useful when volatile oxygenated products such as alcohols or lacquer thinners are suspected.

4.3 This practice is the least sensitive of the sample preparation techniques and may not detect quantities of less than 10 μL of an ignitable liquid.

4.4 Because this separation takes place in a closed container, the sample remains in approximately the same condition in which it was submitted. Repeat and interlaboratory analyses are therefore possible.

4.5 Due to variables in the debris sample condition prior to headspace sampling, complete reproducibility of chromatograms may be difficult to obtain.

4.5.1 To obtain greater reproducibility, use one of the separation procedures which results in a solution of the ignitable liquid residue (see Practices E1386, E1412, and E1413).

4.5.2 To obtain greater sensitivity for petroleum distillates and oxygenated products, use solid phase microextraction. (See Practice E2154.)

4.6 High concentrations of highly volatile compounds may swamp the headspace, inhibiting the recovery of less volatile components.

5. Apparatus

5.1 *Heating System*—An oven, heating mantle or hot plate designed to fit the evidence container.

5.2 *Temperature Measuring Device*—A thermometer or thermocouple capable of measuring temperatures in the range from 40 to 150°C.

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[1] This practice in under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics

Current edition approved Feb. 1, 2012. Published March 2012. Originally approved in 1990. Last previous edition approved in 2005 as E1388 – 05. DOI: 10.1520/E1388-12.

[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.

5.3 A gas-tight syringe capable of reproducibility introducing sample sizes in the range from 0.5 to 5 mL.

5.4 Disposable 20 cc syringes.

5.5 Disposable 16–20 gauge syringe needles.

5.6 A drill, punch, or other device to puncture holes in sample containers.

5.7 Headspace sample vials.

6. Sample Preparation

6.1 Observe the appropriate procedures for handling and documentation of all submitted samples as described in Guide E1459 and Practice E1492.

6.1.1 Open and examine the fire debris sample in order to determine that it is consistent with its description.

6.1.1.1 Resolve any discrepancies between the submitting agency's description of the evidence and the analyst's observation prior to the completion of the report.

6.2 Punch or drill a small hole in the container lid and cover the hole with tape.

6.2.1 Alternatively, a rubber sleeve stopper may be placed in the hole.

6.3 If the sample is going to be heated, place the container in the heating system until the container reaches the desired temperature (not to exceed 90°C).

6.4 The syringe may be heated prior to sampling

7. Sampling Procedure

7.1 *Direct Headspace Sampling:*

7.1.1 Remove the container from the heating apparatus then push the syringe through the tape or rubber sleeve stopper into the hole in the container lid. Slowly pump the syringe several times, withdraw from the lid, and inject 0.5 to 2.0 mL of sample into the gas chromatograph. Analyze as described in Test Method E1618.

7.1.2 After the headspace sample has been removed, reseal the hole.

7.2 *Indirect Headspace Sampling Using Headspace Sample Vials:*

7.2.1 Some automated sample injection systems utilize headspace sample vials for delivery of the sample headspace into the gas chromatograph.

7.2.2 Assemble and seal (crimp or screw) lid onto headspace sample vial. Using a 20 cc syringe and needle, puncture the headspace vial septum and evacuate the contents of the vial. Repeat this procedure 3 to 4 times.

7.2.3 Remove the container from the heating apparatus then push the 20 cc syringe through the tape or rubber sleeve stopper into the hole in the container lid. Slowly pump the syringe several times, withdraw 20 cc of vapor and inject into headspace sample vial. Repeat this process 2 to 4 times.

7.2.3.1 If the headspace sample vial was properly sealed and evacuated, the vacuum created should pull the initial injection into the vial. The latter injections into the vial will require forcibly injecting the 20 cc sample.

7.2.3.2 The sequence method on the gas chromatograph should be setup to inject 0.5 to 2.0 mL of the sample into the gas chromatograph. Analyze as described in Test Method E1618.

7.2.4 After the headspace samples have been removed, reseal the hole in the container.

8. Keywords

8.1 fire debris samples; headspace

Designation: E1412 – 16

Standard Practice for
Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal[1]

This standard is issued under the fixed designation E1412; 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 practice describes the procedure for separation of small quantities of ignitable liquid residues from samples of fire debris using an adsorbent material to extract the residue from the static headspace above the sample, then eluting the adsorbent with a solvent.

1.2 While this practice is suitable for successfully extracting ignitable liquid residues over the entire range of concentration, the headspace concentration methods are best used when a high level of sensitivity is required due to a very low concentration of ignitable liquid residues in the sample.

1.2.1 Unlike other methods of separation and concentration, this practice is essentially nondestructive.

1.3 Alternate separation and concentration procedures are listed in the referenced documents (see Practices E1386, E1388, E1413, and E2154).

1.4 This practice does not replace knowledge, skill, ability, experience, education, or training and should be used in conjunction with professional judgment.

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

1.6 *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]

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
E1413 Practice for Separation 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
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 Practice

3.1 Charcoal in some form of an adsorption package is placed in the sample container to adsorb ignitable liquid residues. The container may be heated or left at ambient temperature. The charcoal is removed and eluted with a suitable elution solvent as listed in 6.3.

4. Significance and Use

4.1 This practice is useful for preparing extracts from fire debris for later analysis by gas chromatography mass spectrometry.

4.2 This is a very sensitive separation procedure, capable of isolating quantities smaller than 1⁄10 µL of ignitable liquid residue from a sample.

5. Apparatus

5.1 *Heating System*—An oven, or a heating mantle to fit the evidence container (or a hot plate).

5.1.1 An oven is recommended to achieve a constant temperature throughout the system.

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[1] This practice 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 Jan. 15, 2016. Published February 2016. Originally approved in 1991. Last previous edition approved in 2012 as E1412 – 12. DOI: 10.1520/E1412-16.

[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.

5.2 *Temperature Measuring Device*—A thermometer or thermocouple capable of measuring temperatures in the range of 40 to 100°C.

5.3 *Adsorption Package.*

5.3.1 Commercial charcoal adsorption packages are available from several companies. These packages, in the form of polymer strips or small charcoal canisters or "C-bags," are used to adsorb organic vapors.

5.3.1.1 The minimum recommended polymer strip size is 10 mm by 10 mm, or 100 mm^2.

5.3.2 *Non-Commercial Adsorption Packages.*

5.3.2.1 *C-Bags*—Prepare C-bags by encapsulating 0.2 g of activated charcoal within a folded sheet of high strength, light weight, high porosity tissue paper, such as that commonly used for making tea bags.[3]

5.3.3 *Storage of Adsorption Packages*—To prevent contamination, store all adsorption packages away from any sources of organic vapors prior to and after sampling.

6. Reagents and 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.[4] 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 *Adsorption Package:*

6.2.1 *C-Bags:*

6.2.1.1 4 by 5 in. (approximate) high strength, light weight, high porosity filter paper.

6.2.1.2 Activated charcoal.

6.2.2 Commercial charcoal adsorption package.

6.2.3 Check charcoal media purity by, at a minimum, desorbing a representative unit using the same elution solvent as will be used for questioned samples and analyzing in accordance with Test Method E1618.

6.3 *Elution Solvent*—Suitable elution solvents are carbon disulfide, *n* –pentane, or diethyl ether. Use of a heavier solvent, such as toluene or tetrachloroethylene, is sometimes necessary when the compounds of interest have low molecular weights.

6.3.1 Check solvent purity by evaporating to at least twice the extent used in the analysis and analyzing the evaporated solvent in accordance with Test Method E1618.

6.3.2 Read and follow the safety precautions described in the safety data sheet (SDS) of the extraction solvent that is used.

7. Sample Preparation/Adsorption Procedure

7.1 Observe the appropriate procedures for handling and documentation of all submitted samples as described in Practice E1492.

7.1.1 Open and examine the fire debris sample in order to determine that it is consistent with its description.

7.1.1.1 Resolve any discrepancies between the submitting agency's description of the evidence and the analyst's observation prior to the completion of the report.

7.2 Place an adsorbent package in the evidence container according to laboratory protocols and reseal the container. Suspend the adsorbent package above the sample whenever possible.

7.3 Heat the container to a temperature of 50 to 80°C, for 2 to 24 h. The longer times or higher temperatures, or both, are required for the adsorption of higher boiling point compounds or for the adsorption of very small quantities of volatile hydrocarbons. The adsorption temperature and duration may vary based on the sample.[3, 5]

7.3.1 When other evidentiary considerations arise (such as document or latent print examinations) it may be appropriate to conduct the adsorption at ambient temperature (approximately 20°C) for extended periods (24 h or longer) to minimize damage.

7.3.2 Room temperature adsorption may also be appropriate to detect low molecular weight compounds.

7.3.3 The optimum adsorption time for maximum sensitivity will depend on the adsorption package and temperature selected. Temperatures lower than 60°C may be insufficient to volatilize compounds heavier than C16.

7.3.4 Temperatures in excess of 80°C may result in disproportionate recovery of higher molecular weight compounds with the displacement of lower molecular weight compounds.

7.3.5 The optimum adsorption time for representative sampling or maximum sensitivity, or both, will depend on the adsorption package, the adsorption temperature, and the ignitable liquid composition and concentration.

7.3.5.1 Adsorption times for routine screening of samples are typically in the range of 8 to 24 h.

7.3.5.2 Data that appears overloaded or excessively displaced may be corrected by resampling at ambient temperatures, or with shortened adsorption times (1 to 4 h), or by performing a solvent extract on a portion of the debris in accordance with Practice E1386.

7.4 A known amount (typically, 0.1 µL to 0.5 µL) of an internal standard may be added to the sample in order to evaluate the efficiency of the procedure.

7.4.1 Internal standards are typically prepared using a single compound that is easily identified (such as 3-phenyltoluene or diphenylmethane) dissolved in the eluting solvent.

7.4.2 If an internal standard is added, the compound and the quantity added shall be documented along with the manufacturer, grade, and lot number used.

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[3] Dietz, W. R., "Improved Charcoal Packaging for Accelerant Recovery by Passive Diffusion," *Journal of Forensic Sciences*, Vol 35, 1991, pp. 111–121 (Unk).

[4] *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

[5] Newman, R, et al., "The Use of Activated Charcoal Strips for Fire Debris Extractions by Passive Diffusion. Part 1: The Effects of Time, Temperature, Strip Size and Sample Concentration," *Journal of Forensic Sciences*, Vol 41, 1996, pp.

7.5 Document the adsorption parameters including the type and amount of adsorbent used, adsorption temperature and adsorption time.

8. Elution Procedure

8.1 Remove the adsorption package from the evidence container. If it is not to be eluted immediately, store the adsorption package in a clean, vapor-tight container.

8.2 Follow the evidence documentation and handling procedures described in Guide E1459 and Practice E1492.

8.3 Place the adsorption substrate in a properly labeled container and desorb with a minimal amount of eluting solvent required for instrumental analysis (typically 50 to 1000 µL).

8.4 Document the type and approximate volume of solvent used to desorb the analyte(s) from the adsorbent.

9. Sealing

9.1 A septum seal or screw cap glass vial may be used for collecting and sealing the extract for analysis.

10. Extract and Adsorbent Storage

10.1 Refer to Practice E2451 for short term and long term storage of ignitable liquid extracts.

11. Blanks and Standards

11.1 Run frequent blanks on each lot of adsorbent packages.

11.1.1 Analyze an absorbent package blank by eluting the adsorbent with 50 to 1000 µL of solvent and analyze according to Test Method E1618.

11.1.2 Prepare a system blank by placing an adsorbent package into a clean dry evidence container and running the adsorbent procedure as described in Section 7. Elute the adsorption package according to Section 8, and analyze the extract according to Test Method E1618.

11.1.2.1 If an internal standard is routinely used, include an internal standard in the blank.

11.2 When necessary, charcoal may be activated and cleaned by heating in a 400°C oven for 4 h, then cooling in a desiccator.

11.3 Periodically check the adsorption efficiency by running this procedure on a sample containing a known volume of standard ignitable liquid.

12. Keywords

12.1 activated charcoal; fire debris samples; passive headspace concentration

Designation: E1413 – 13

Standard Practice for
Separation of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration[1]

This standard is issued under the fixed designation E1413; 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 practice describes the procedure for separation of small quantities of ignitable liquid residues from fire debris samples using the method of dynamic headspace concentration.

1.2 Dynamic headspace concentration uses adsorption and subsequent solvent elution or thermal desorption.

1.3 Both positive and negative pressure systems for adsorption are described, as well as a thermal desorption system.

1.4 While this practice is suitable for successfully extracting ignitable liquid residues over the entire range of concentration, the headspace concentration methods are best used when a high level of sensitivity is required due to a very low concentration of ignitable liquid residues in the sample.

1.5 Alternate separation and concentration procedures are listed in Section 2. If archival of the extract is of importance, then this practice's thermal desorption procedure, SPME (Practice E2154), and headspace (Practice E1388) sample separation techniques are not recommended unless a portion of the extract can be split and retained. In order to have an archivable extract, then this practice's sample collection on charcoal, solvent extraction (Practice E1386), or passive headspace concentration (Practice E1412) is recommended.

1.6 This practice offers a set of instructions for performing one or more specific operations. This standard cannot replace knowledge, skill, or ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgment.

1.7 *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]

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
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
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 Practice

3.1 The sample, preferably in its original container, is heated, forcing volatile compounds to vaporize. The headspace in the sample is then drawn or pushed through a tube containing an adsorption media (typically activated charcoal or Tenax) which adsorbs the vaporized compounds.

3.2 Other solid adsorbents and collection systems can be used as long as the method has been validated with a wide variety of ignitable liquids with a range of both flash points and polarity.

4. Significance and Use

4.1 This practice is useful for preparing extracts from fire debris for later analysis by gas chromatography-mass spectrometry (GC-MS), see Test Method E1618.

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[1] This practice 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 Aug. 15, 2013. Published September 2013. Originally approved in 1991. Last previous edition approved in 2007 as E1413 – 07. DOI: 10.1520/E1413-13.

[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.

4.2 This is a very sensitive separation procedure, capable of isolating quantities smaller than 0.1 µL of ignitable liquid residue from a sample

4.2.1 Actual recovery will vary, depending on several factors, including adsorption temperature, container size, and competition from the sample matrix.

4.3 This is a potentially destructive technique. Portions of the sample subjected to this procedure may not be suitable for re-sampling. Therefore, a portion of the sample extract should be saved for potential future analysis. Consider using passive headspace concentration as described in Practice E1412.

5. Apparatus

5.1 *Positive Pressure Apparatus:*

5.1.1 *Sample Pressurization Device*—A system capable of delivering pressurized dry nitrogen (or other inert gas) at up to 40 psi (276 kPa) to an orifice to be inserted into the bottom of the sample container.

5.1.1.1 A needle valve capable of fine control of the flow rate of the dry nitrogen, at up to 1500 cc/min.

5.1.1.2 A flow meter capable of measuring the flow of dry nitrogen through the end of the adsorption tube at the rate of 0 to 1500 cc/min.

5.1.1.3 *Container Closure*—A device suitable for sealing the container and directing the effluent nitrogen (or other inert gas) and vapors to the adsorption tube.

5.2 *Negative Pressure Apparatus:*

5.2.1 *Inlet and Outlet System*—A tube containing approximately 1 cm of charcoal is fitted into the lid of the original container with a suitable penetrable seal. This serves as a filter for incoming room air. A vacuum is pulled on an adsorption tube also fitted into the lid of the original container with a suitable penetrable seal.

5.2.2 A vacuum system capable of pulling between 200 and 1500 cc/min on the sample collection tube.

5.2.3 A flow meter capable of measuring the flow of air through the end of the charcoal tube at the rate of 200 to 1500 cc/min.

5.3 *Thermal Desorption Apparatus:*

5.3.1 *Sampling System*—An air-tight syringe is connected to the back-end of a Tenax tube. The front end of the Tenax tube is fitted into the heated container.

5.3.2 In order to prevent leakage and contamination, the system should be designed to minimize loss of vapors from the container. For example by means of a septum mounted on top of the lid of the container.

5.3.3 *Thermal Desorption Device*—A system capable of desorbing the volatiles by means of elevated temperature and trapping the volatiles in a cold-trap. This apparatus is directly coupled to a GC-MS.

5.4 *Adsorption Tubes:*

5.4.1 *Charcoal Tubes*—Suitable charcoal filter and sample adsorption tubes may be made by inserting a small (approximately 1 cm) plug of glass wool or cotton in the bottom of a 2.5 to 5 cm of activated charcoal, and finally, holding the charcoal in place with an additional plug of glass wool or cotton.

5.4.1.1 Alternatively, charcoal tubes are available from commercial sources.

5.4.2 *Tenax Tubes*—Suitable Tenax tubes are commercially available. Note that solvent extraction of these tubes results in complications in the desorbing phase because certain solvents cause the dissolution of the Tenax. Tenax should be employed when thermal desorption is to be performed.

5.5 *Heating System*—A heating mantel designed to fit the evidence container or an oven or a hot plate.

5.5.1 An oven may be set up with any number of stations to allow for multiple sample preparation.

5.6 *Temperature Measuring Device*—A thermometer or thermocouple capable of measuring temperatures in the range of 40 to 150°C.

6. Reagents and 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.[3] 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 *Solid Adsorbent:*

6.2.1 *Activated Charcoal* (coconut).

6.2.1.1 Charcoal may be activated and cleaned by heating in a 400°C oven for approximately 4 hours and cooling in a desiccator.

6.2.1.2 Test each new or reconditioned batch of charcoal for residual hydrocarbons by analyzing a concentrated extract of blank adsorption media according to Test Method E1618.

6.2.2 *Tenax:*

6.2.2.1 Tenax may be cleaned and conditioned according to the manufacturer's instructions.

6.2.2.2 Test each new or reconditioned batch of Tenax for residual hydrocarbons by analyzing a concentrated extract of blank adsorption media according to Test Method E1618.

6.3 *Glass Wool,* or cotton, free of extractable hydrocarbons.

6.4 *Extraction solvent* (for example, carbon disulfide, pentane, ethyl ether).

6.4.1 Read and follow the safety precautions described in the Safety Data Sheets (SDS) of the extraction solvent that is used.

6.4.2 Test each new lot number of the extraction solvent by analyzing a concentrated portion according to Test Method E1618.

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[3] *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

6.5 Pasteur pipettes and glass vials free of extractable hydrocarbons.

7. Sample Preparation

7.1 Observe the appropriate procedures for handling and documentation of all submitted samples (Guide E1459 and Practice E1492).

7.1.1 Examine the fire debris sample in order to determine that it is consistent with its description.

7.1.1.1 Resolve any discrepancies between the submitting agent's description of the evidence and the analyst's observation with the submitting agent prior to the completion of the report.

7.2 This is a potentially destructive technique. Portions of the sample subjected to this procedure may not be suitable for re-sampling. Therefore, a portion of the sample extract should be saved for potential future analysis. Consider using passive headspace concentration as described in Practice E1412 which is essentially nondestructive.

7.3 The system should be designed so that the majority of the samples can be extracted from the container in which they are delivered to the laboratory.

7.3.1 Alternatively, the sample or a portion of the sample can be placed in an appropriate, clean sampling container which is designed to be flushed by positive or negative pressure.

8. Adsorption Procedure

8.1 A laboratory typically has one method of adsorption and elution that is employed. The most common combinations in use are charcoal adsorption followed by solvent elution, and Tenax adsorption followed by thermal desorption. Both adsorbents can be collected using either a positive or a negative pressure system. Other solid adsorbents and collection systems can be used as long as the method has been validated with a wide variety of ignitable liquids with a range of both flash points and polarity.

8.1.1 The volume of air sampled is generally less when thermal desorption is employed as the entire collected sample is commonly injected into the GC-MS system for analysis.

8.1.2 With thermal desorption there is typically no material that can be archived once GC-MS analysis has been performed. If archival of a portion of the sample extract is needed, then a different adsorbent and elution combination should be chosen either following this practice, or following one of the other sample collection methods for fire debris analysis (Practices E1386 or E1412). Some instrument manufacturers now have the facility to collect the unused sample from the split; if your instrument has such capability, then there will be material available for archival when thermal desorption is used.

8.2 *Positive Pressure:*

8.2.1 Place the sample container in the heating system and connect to the inert gas source (commonly nitrogen) which will introduce the gas into the bottom of the container, then connect the exhaust line to the adsorbing tube.

8.2.1.1 Direct connection of the adsorption tube to the sample container is possible, unless an oven is used. Placement intervening line which must be carefully cleaned between uses to remove any adsorbed or condensed residues.

8.2.2 *Pressurization*—Deliver the inert gas to the sample through a ⅛ in. (3.2 mm) line on the inlet side of the container at a pressure of 40 psig (276 kPa) upstream of the needle valve controlling the flow rate. Connect a ¼ in. (6.35 mm) line on the downstream side of the container to the charcoal tube. Attach the flow meter to the outlet side of the charcoal tube, and use the needle valve to adjust the flow to the rate determined to be optimum for this system.

8.2.2.1 Determine the optimum system conditions (flow rate, temperature and time) by placing known quantities of gasoline, kerosene, mineral spirits, and diesel fuel onto exemplar substrate material such as squares of carpeting or filter paper. Determine the percent recovery by gas chromatographic analysis. The conditions resulting in the highest percent recovery is the optimum.

8.2.2.2 Remove the flow meter once the flow is adjusted. Check the flow at least once after the sample has reached the final temperature.

8.2.3 Place the temperature measuring device in an appropriate location for the system used in order to monitor the temperature of the sample. Turn on the heating system and increase the temperature to no more than 150°C. At this temperature, most petroleum distillates can be volatilized in less than one hour, and there will be a minimum of pyrolysis of common polymers.

8.2.3.1 Allow 20 min for the sample to reach temperature, and an additional one hour flushing the sample out. At this point, turn off the gas. The charcoal adsorption tube is now ready for elution.

8.2.3.2 The adsorption time may be reduced if it can be shown that equal or superior recovery rates are achieved in the reduced time period.

8.3 *Negative Pressure:*

8.3.1 Place the inlet air filtering adsorption tube and the outlet adsorption tubes in the lid of the evidence container.

8.3.2 Place the temperature measuring device in an appropriate location for the system used in order to monitor the temperature of the sample.

8.3.3 Apply a vacuum to the sample adsorption tube. The flow through the tube should be between 200 and 1500 cc/min.

8.3.4 Determine the optimum system conditions (flow rate, temperature and time) for this system by placing known quantities of gasoline, kerosene, mineral spirits, and diesel fuel onto exemplar substrate material such as squares of carpeting or filter paper. Determine the percent recovery by gas chromatographic analysis. The conditions resulting in the highest percent recovery is the optimum.

8.3.4.1 An example of system conditions could be to heat the evidence container to 90 to 95°C and hold at that temperature for 15 to 25 minutes with the flow at 500 cc/min. Longer times may be necessary for heavily loaded cans. Turn off the vacuum, and the sample adsorption tube is ready to be desorbed.

8.4 *Thermal Desorption:*

8.4.1 Heat the sample to the optimum temperature for your

8.4.2 Place the temperature measuring device in an appropriate location for the system used in order to monitor the temperature of the sample.

8.4.3 Remove the caps from the Tenax tube.

8.4.4 Connect the syringe to the back-end of the Tenax tube. Connect the front-end of the Tenax tube to the heated container.

8.4.5 Pull the plunger of the syringe and sample the headspace vapors onto the Tenax adsorption tube. Remove the Tenax system from the container and close the container. Cap the Tenax tube. The Tenax tube is ready for thermal desorption.

8.4.6 Determine the optimum system conditions (temperature, time and sample size) for this system by placing known quantities of gasoline, kerosene, mineral spirits, and diesel fuel onto exemplar substrate material such as squares of carpeting or filter paper. Determine the percent recovery by gas chromatographic analysis. The temperature-volume combination resulting in the highest percent recovery is the optimum.

9. Desorption Procedure

9.1 Follow the evidence documentation, labeling and handling procedures described in Guide E1459 and Practice E1492.

9.2 *Desorption:*

9.2.1 Any method of desorption may be used if it can be shown that adsorbed residues are effectively eluted.

9.2.2 *Charcoal Tube (Solvent Elution)*—Remove the charcoal tube (or other solid adsorbent) from the sample outlet line and insert into the properly labeled test tube. Allow to cool to room temperature. Trickle approximately 1 mL of elution solvent into the charcoal tube. If possible, allow the solvent to remain in the charcoal for approximately 5 minutes. An aspirator bulb may be necessary in order to push the solvent through the charcoal. Collect the solvent into a GC vial or clean test tube.

9.2.2.1 Concentration of the solvent extract can be performed using a stream of gas (such as hydrocarbon free air or nitrogen) over the solvent.

9.2.2.2 Alternatively, the charcoal (or other solid adsorbent) may be removed from the tube and agitated in a container with elution solvent.

9.2.3 *Tenax Tube (Thermal Desorption)*—The thermal desorption device coupled to a GC-MS is used for the desorption and direct analysis of the sampled volatiles. The Tenax tube is heated and the sample is collected in a cold-trap. The collected sample is directly injected in the GC-MS.

10. Sealing

10.1 A septum seal vial or screw cap glass vial with PTFE-lined seals may be used for collecting and sealing the elution solvent extract.

10.2 Refer to Practice E2451 for short term and long term storage of ignitable liquid extracts.

11. Blanks and Standards

11.1 Frequent blanks must be run on every apparatus used for this procedure.

11.1.1 Analyze an absorbent package blank by desorbing a unit according to the same method employed with samples and analyze according to Test Method E1618.

11.1.2 Prepare a system blank by connecting an empty container to the adsorption apparatus, and running the adsorption procedure described in Section 8 for the normal length of time. Desorb the adsorbent tube according to Section 9 (including concentration, if used), and analyze the extract according to Test Method E1618.

11.1.2.1 If an internal standard is routinely used, include an internal standard in the blank.

11.2 *Cleaning and Regeneration:*

11.2.1 Charcoal may be activated and cleaned by heating in a 400°C oven for 4 h and cooling in a desiccator.

11.2.2 Tenax may be cleaned and conditioned according to the manufacturer's instructions.

11.3 Check the adsorption efficiency periodically by running this procedure on a sample containing a known volume of standard ignitable liquid.

12. Keywords

12.1 activated charcoal; dynamic headspace concentration; elution; fire debris samples; forensic science; ignitable liquid residues; Tenax; thermal desorption

Designation: E2154 − 15a

Standard Practice for
Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Solid Phase Microextraction (SPME)[1]

This standard is issued under the fixed designation E2154; 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 practice describes the procedure for removing small quantities of ignitable liquid residues from samples of fire debris. An adsorbent material is used to extract the residue from the static headspace above the sample. Then, analytes are thermally desorbed in the injection port of the gas chromatograph (GC).

1.2 This practice is best suited for screening fire debris samples to assess relative ignitable liquid concentration and for extracting ignitable liquid from aqueous samples.

1.3 This practice is suitable for extracting ignitable liquid residues when a high level of sensitivity is required due to a very low concentration of ignitable liquid residues in the sample.

1.3.1 Unlike other methods of separation and concentration, this method recovers a minimal amount of the ignitable residues present in the evidence, leaving residues that are suitable for subsequent resampling.

1.4 Alternate separation and concentration procedures are listed in Section 2.

1.5 This standard cannot replace knowledge, skill, or ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgment.

1.6 *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]

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 of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration

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 Practice

3.1 A fiber coated with a polydimethylsiloxane stationary phase is exposed to the headspace of the fire debris sample container to extract ignitable liquid residues. The fiber, which is housed in a needle similar to a syringe needle, is introduced directly in the injection port of a gas chromatograph to thermally desorb the analytes.

4. Significance and Use

4.1 This practice is suited ideally for screening samples for the presence, relative concentration, and potential class of ignitable liquid residues in fire debris.

4.2 This is a very sensitive separation procedure, capable of isolating small quantities of ignitable liquid residues from a

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[1] This practice 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. 1, 2015. Published September 2015. Originally approved in 2001. Last previous edition approved in 2015 as E2154 – 15. DOI: 10.1520/E2154-15A.

[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.

sample, that is, a 0.1 µL spike of gasoline on a cellulose wipe inside of a 1-gal can is detectable.

4.3 Actual recovery will vary, depending on several factors, including adsorption temperature, container size, competition from the sample matrix, ignitable liquid class and relative ignitable liquid concentration.

4.4 Because this separation takes place in a closed container, the sample remains in approximately the same condition in which it was submitted. Repeat and interlaboratory analyses, therefore, may be possible. Since the extraction is nonexhaustive, the technique permits reanalysis of samples.

4.5 This practice is intended for use in conjunction with other extraction techniques described in Practices E1386, E1388, E1412, and E1413.

4.6 The extract is consumed in the analysis. If a more permanent extract is desired, one of the separation practices described in Practices E1386, E1412, or E1413 should be used.

5. Apparatus

5.1 *Heating System,* such as, an oven or heating mantle to fit the evidence container (or a hot plate).

5.2 *Temperature Measuring Device,* such as, a thermometer or thermocouple capable of measuring temperatures in the range of 40 to 110°C.

5.3 *SPME Apparatus,* such as, a solid phase microextraction fiber holder.

5.4 *SPME Fiber*—Several polymer coatings can be used to extract volatiles such as ignitable liquid residues. A100 µm polydimethylsiloxane (PDMS) coating has been shown to perform well for most C_{10}-C_{25} compounds while a 85 µm Polyacrylate (PA) and a combined 75 µm Carboxen/PDMS has been shown to perform well for C_1-C_{10} compounds (**1**).[3]

5.5 *Punch.*

5.6 *Rubber Sleeve Septum.*

6. Sample Preparation/Analysis Procedure

6.1 Observe the appropriate procedures for handling and documentation of all submitted samples as described in Practice E1492.

6.1.1 Open and examine the fire debris sample to determine that it is consistent with its description.

6.1.1.1 Resolve any discrepancies between the submitting agent's description of the evidence and the analyst's observation with the submitting agent prior to the completion of the report.

6.2 Verify that the fiber is free of ignitable residue components and other contaminants.

6.2.1 Introducing the fiber into the GC under the injector desorption and chromatographic conditions for ignitable residue analysis. This fiber residue "blank" always should be evaluated before residue sampling when performing a manual injection and desorption.

6.2.2 When performing batch analysis of fiber blanks for autosampler apparatus, all fibers determined to be clean by analysis should be promptly sealed in a vapor-tight container(s) until ready for use.

6.3 If the sample container has a metal lid, make a hole in the lid and install a rubber sleeve septum in the opening.

6.4 Heat the container until the sample reaches the desired temperature (approximately 20-30 min, but longer equilibration times may be needed depending on the heating method or the amount and type of matrix present in the can, or both).

6.4.1 Temperatures lower than 60°C may be insufficient to volatilize compounds above C_{16}.

6.4.2 Temperatures above 80°C may result in a significant discrimination against high volatility compounds when the 100 µm PDMS fiber is used.

6.5 Remove the container from the heating apparatus.

6.6 Immediately puncture the rubber sleeve septum or plastic evidence bag with the needle of the SPME apparatus.

6.7 Expose the SPME fiber to the headspace of the container for the desired sampling duration.

6.7.1 The optimum exposure time for maximum sensitivity will depend on the temperature and the concentration and composition of the volatile compound present in the sample headspace.

6.7.2 Exposure times for routine screening of samples typically are in the range of 5-15 min.

6.7.3 Residue extracts that provide off-scale or poorly resolved chromatographic responses should be resampled at lower temperatures or shorter exposure times.

6.8 Secure the fiber in the needle by withdrawing it into the SPME apparatus. Remove the apparatus from the sample container.

6.8.1 Rubber sleeve septa are self-sealing. Puncture holes in evidence bags should be sealed after sampling.

6.9 Analyze the adsorbate by exposing the fiber in the injection port of a gas chromatograph. Desorb the fiber for approximately 1.5 to 4 min between 200°C and 260°C. Analysis is performed as described in Test Method E1618.

6.9.1 Some instruments can desorb fibers using an autosampler apparatus. The acceptability of the autosampler parameters shall be verified using known ignitable liquids and fiber blanks within a sequence.

6.10 Repeat each step in Section 6 as necessary for each remaining fire debris sample.

6.10.1 Previously analyzed fiber blanks are analyzed during an autosampler sequence. At least one fiber blank must be analyzed, preferably near the end of the sequence, to verify that fibers were not contaminated during storage or sample processing.

6.11 Record the adsorption parameters including the expo-

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[3] The boldface numbers in parentheses refer to a list of references at the end of

7. Blanks and Standards

7.1 Analysis of a fiber blank (6.2) is required immediately before each manual sample extraction and batch analysis of fiber blanks prior to sample extractions when using autosampler apparatus.

7.1.1 If an internal standard is routinely used, include the internal standard in the blank run by placing the internal standard in an empty container and exposing the SPME fiber as described in Section 6.

7.2 Periodically check the adsorption efficiency of the fiber by running this procedure on a sample containing a known volume of an ignitable liquid standard.

7.3 An ignitable liquid standards library should be maintained with neat and evaporated commonly ignitable liquids recovered from sample containers by this technique at various concentrations.

8. Keywords

8.1 fire debris samples; passive headspace concentration; solid phase microextraction (SPME)

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(5) Almirall, J.R., Wang, J., Lothridge, K., and Furton, K.G., "The Detection and Analysis of Flammable or Combustible Liquid Residues on Human Skin," *Journal of Forensic Science*, JFSCA, Vol 45, No. 2, March 2000, pp. 453–461.

Designation: E2451 – 13

Standard Practice for
Preserving Ignitable Liquids and Ignitable Liquid Residue Extracts from Fire Debris Samples[1]

This standard is issued under the fixed designation E2451; 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 practice describes procedures for preserving residues of ignitable liquids in extracts obtained from fire debris samples and questioned ignitable liquid samples. Extraction procedures are described in the Referenced Documents.

1.2 This practice does not attempt to address all the issues regarding the short-term or long-term storage of ignitable liquid samples and ignitable liquid extracts from fire debris samples. The changes that may occur under various storage conditions have not been fully documented.

1.3 This practice cannot replace knowledge, skill, or ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgment.

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]

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 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

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

3. Summary of Practice

3.1 Extracts obtained from fire debris samples and questioned liquids analyzed for the presence of ignitable liquid residues are retained and preserved for potential reanalysis by Test Method E1618.

4. Significance and Use

4.1 The archiving of extracts recovered from fire debris or liquids submitted in a fire investigation provides a mechanism to preserve extracts and liquids for reanalysis in the event that sample loss, sample degradation, or failure of the fire debris container occurs during post-analysis storage of fire debris evidence.

4.2 The nature of some extraction procedures, which may preclude reanalysis, is considered.

4.3 Changes to a preserved sample extract and the length of time it remains viable under storage conditions are unknown.

4.4 The concentration and composition of the ignitable liquid residue or the use of an alternate extraction/concentration technique used to preserve a sample extract of the fire debris sample may result in different findings between the analysis of the preserved sample and the original analysis.

5. Materials

5.1 *Airtight and Vapor-Tight, Volatile-Free Storage Containers*—The following are suggested container types:

5.1.1 Septum crimp vials with PTFE-lined seals.

5.1.2 Screw cap glass vials with PTFE-lined seals.

5.1.3 Polymer evidence bags (this does not include polyethylene or polypropylene-type containers).

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[1] This practice 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 June 1, 2013. Published July 2013. Originally approved in 2008. Last previous edition approved in 2008 as E2451 – 08. DOI: 10.1520/E2451-13.

[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.

5.2 *Adsorption Media:*

5.2.1 Activated charcoal (coconut).

5.2.2 Activated charcoal strips.

6. Procedure

6.1 Passive Headspace Concentration with Activated Charcoal (Practice E1412).

6.1.1 *Single-Strip Adsorption:*

6.1.1.1 After analysis, the eluted and analyzed extract may be retained for preservation. Readsorb the extract onto the charcoal strip by allowing the eluent to evaporate. Store the charcoal strip in an appropriate container for preservation.

6.1.1.2 Alternatively, after adsorption and before elution, divide the strip. Use one portion of the strip for analysis. Place the remaining portion in an appropriate container for preservation.

6.1.2 *Two-Strip Adsorption:*

6.1.2.1 After adsorption, use one strip for analysis. Place the second strip in an appropriate container for preservation.

NOTE 1—This procedure requires that the two strips be adsorbed simultaneously.

6.1.3 *Adsorbent Package Adsorption:*

6.1.3.1 After analysis, the eluted and analyzed extract may be retained for preservation. Readsorb the extract onto the adsorbent package by allowing the eluent to evaporate. Store the adsorbent in an appropriate container for preservation.

6.2 Dynamic Headspace (Practice E1413).

6.2.1 After analysis, the eluted and analyzed extract may be retained for preservation. Readsorb the extract onto the adsorbent by allowing the eluent to evaporate. Store the adsorbent in an appropriate container for preservation.

6.3 Solvent Extraction (Practice E1386).

6.3.1 After analysis, the analyzed extract may be retained for preservation. Adsorb the extract or a portion of the extract on an adsorption medium and store in an appropriate container for preservation.

6.3.2 Place any remaining extract in an appropriate container for preservation.

6.4 Liquid Samples.

6.4.1 Place the liquid, a portion of the liquid, extract, or recovered distillate in an appropriate container for preservation.

6.4.2 Alternatively, adsorb a portion of the liquid, extract, or recovered distillate on an adsorption medium and store in an appropriate container for preservation.

6.5 SPME and Headspace Sampling (Practices E2154 and E1388).

6.5.1 Extracts using these sampling techniques are consumed by the analysis and are not amenable to preservation.

6.5.2 Alternative separation and concentration practices may be used when the preservation of extracts is desired.

7. Storage of Liquid Samples and Extracts

7.1 Label all vials containing samples or extracts according to Guide E1459. Notations regarding any sample or extract preservation should be maintained in the case notes and in accordance with laboratory procedures.

7.1.1 Preserved samples and extracts may be released/returned with the evidence as an attachment or enclosure.

7.1.2 Alternatively, preserved samples and extracts may be cataloged and stored.

7.2 Preserved samples and extracts may be stored in containers as noted in 5.1. Chemical compatibility, vapor pressure, contamination, mechanical strength, and storage conditions are among the considerations in selection of storage containers.

7.3 Preserved samples and extracts may be stored at room temperature, under refrigeration, or frozen. Effects of temperature and freeze-thaw conditions on the stability and viability of samples, effectiveness of container sealing, and container integrity are factors to consider in selection of thermal storage conditions.

7.4 If not returned with the evidence, store preserved samples and extracts at the laboratory for a period of time as directed by laboratory policy and federal, state, or local laws. Follow the procedures set forth in Practice E1492 to ensure that the chain of custody is protected.

8. Keywords

8.1 analysis; archiving; extract preservation; fire debris; forensic science; ignitable liquid residue; sample extract; sample preservation; storage

TABLE A2.1 Sample Laboratory Report

[Laboratory Name and address if not already in a letterhead]
[Laboratory or Case file number]
[Date of report]

[Report recipient]
[Agency or Requestor]
[Address]
[City, State, Zip]

Subject (Incident Summary)
[Case Identifier]
[Fire Date]
[Fire Location]
Analysis of Fire Evidence

Background
The following items were received on [Date received] via [method of delivery].

Item 1. [Size of container] [description of container, that is, can, jar, bag] containing [description of material, *that is, charred wood, burned carpet, debris, etc. as determined by the individual who analyzed the sample*] labeled as "removed from [Post-fire location of the sample as *described by the person who collected the sample.*]"

(Example: one-gallon can containing burned carpet labeled as "removed from the master bedroom.")

Continue to describe additional samples.

[Agency/*Laboratory Name*] was requested to analyze the samples to check for the presence of ignitable liquid residues.

Test Methods and Results
The sample was (samples were) separated according to [describe separation procedure(s) or cite ASTM method(s), for example, Practices E1386 – yy, E1412 – yy, E2154 – yy], and analyzed by [describe analytical procedure(s) or cite ASTM method(s), for example, Test Method E1618 – yy].

For a Positive Sample:
Gas chromatography-mass spectrometry (GC-MS) of [a solvent extract, or concentrated headspace vapors] from the sample detected [identify the class of materials identified] (*optional*) having a carbon number range of [C_X through C_Y.]

Examples of ignitable liquids in this class include [provide examples of the class of materials.]

For a Negative Sample:
Gas chromatography-mass spectrometry (GC-MS) of [a solvent extract, or concentrated headspace vapors] from the sample failed to detect any ignitable liquid residues.

Qualifying Statements

Conclusion
[Ignitable liquid residue] was detected in item(s) [ ].

Evidence Disposition
The sample has (samples have) [been returned to the submitting agency, via method of delivery] [been placed in the laboratory storage facility].

Prepared by [signature and printed name of the individual who is responsible for the analysis].

(*optional*) Reviewed by [*signature and printed name of the individual who reviewed the analysis*].