Idaho State Police Forensic Services

FIRE EVIDENCE
ANALYTICAL METHODS

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Revision History

Revision #	Description of Changes
1	Original issue of Combined method: numbering change to fit new template and minor grammatical changes
2	Changed 1.1, 1.2, 2.1, 4.1.2.1, 4.1.3.1, 4.1.4, 4.1.5, 4.2.1.3.1, 4.2.1.3.2, 4.2.1.3.3, 4.2.1.9, 4.2.1.10.1, 4.2.1.10.5., 4.2.2.2, 4.2.2.6, 4.2.2.7, 4.2.2.10.7, 4.2.2.12, 4.2.2.13, 4.2.2.14.2.1, 4.2.2.15, 4.3.4.3.3.2, 4.3.5.3.1.5, 4.3.5.3.2.2, corrected numbering in 4.2.2.13, added 4.3.4.1.1.2, 4.3.4.1.1.3, 4.3.4.1.1.4, 4.3.5.3.3, 4.3.5.4
3	Changed 1.1, 4.1.1.3, 4.2.1.10.2, 4.2.2.1.2, 4.2.2.10.7, 4.2.2.12, 4.2.2.14.1.4, 4.2.2.14.3.2, 4.5.1.2.3-all
4	Added to 4.2.1.8 & 4.2.2.6
5	Changed 4.2.2.14.1.2, 4.2.2.14.2.4, 4.3.4.3.2.5, 4.3.4.3.3, 4.3.5.3.2.1, 4.3.5.3.2.3
6	Changed 4.2.1.10.1, 4.3.4.1.2
7	Changed 4.3.5.2
8	Change 1.2
9	Changed 4.2.1.3.1, 4.2.1.10.4, 4.2.1.11, 4.3.4.1.2, updated Table 1 and ASTM versions throughout
10	General grammatical editing; updated 1.2, 4.1.1.1, 4.1.1.4, 4.2.2.6, 4.2.2.12, 4.2.3.11.1.3, 4.3.5.2, 4.3.5.3.2.3, 4.3.6.1.1; deleted 4.2.1.4, 4.2.2.4, 4.2.3.4
11	Add 1.1, 3.2, 4.1.5.2, 4.2.1.6.3, 4.2.3.5.2; Change 1.3, 2.1, 4.1.1.3, 4.1.3.1, 4.2.1.2.3, 4.2.1.3.1, 4.2.1.4.1, 4.2.1.7.2, 4.2.1.7.4, 4.2.2.2.1, 4.2.2.2.2, 4.2.2.7.2, 4.2.2.7.4, 4.2.2.7.7, 4.2.2.9, 4.2.2.11.2.3, 4.2.3.2.7, 4.2.3.6.2, 4.2.3.7.3, 4.2.3.8.1, 4.3.3.2.2.1, 4.3.3.2.2.2, 4.3.4.2, 4.3.4.3.2.2, 4.4.2.4.1, 4.5.1.2.3.1-3; Deleted 4.2.1.3.1, 4.2.2.5.1, 4.2.2.5.2; Moved 4.2.1.11 to 4.2.1.3.1, 4.3.4.2 to 4.2.1.3.2, and 4.3.5.3.1.2 to 4.2.1.3.3; Cut 4.2.1.3.1, 4.2.2.5.1, and 4.2.2.5.2 Assigned numbers to bulleted item, combined General and ASTM Method into “Background/Reference” and combined Application and Limitation into “Scope”. Renumbered document due to combined sections.
12	Add 4.2.1.1.1; Ketone and Alcohol to 4.3.5.1.1 Change 4.2.1.1.1, 4.2.1.3.2.1-3, 4.2.1.6.3, 4.2.1.7.1, 4.2.1.7.2, 4.2.2.4.2, 4.2.2.7.4, 4.2.2.7.9, 4.2.2.11.2.3 Delete 4.2.1.7.1

Recovery, Analysis and the Identification of Ignitable Liquid Residues from Fire Evidence

1.0 Background/References

1.1 This method is based upon corresponding American Society for Testing and Materials (ASTM) standard test methods used in Forensic Science that address the recovery, analysis, and identification of ignitable liquid residues. *ASTM methods referenced in this method are current revisions unless otherwise noted. Refer to the Fire Evidence Training Plan and current literature for additional references.*

1.2 The analysis of evidence from fires of suspicious origin encompasses a variety of sample types. The submitted samples may include burnt fire debris, liquids recovered at the fire scene, the suspect's clothing, and/or ignitable liquids found in the suspect's possession. Establishing the presence of an ignitable liquid or ignitable liquid residue in burnt debris recovered from a fire scene can be an important investigative tool for the investigator tasked with establishing the cause and origin of a fire. The techniques used to recover the ignitable residue in debris are complicated by the large contribution of the vast variety of materials that are concurrently subjected to the fire process. The analysis of recovered vapors and/or extracts by GC-MS not only provides an analytical tool to identify the ignitable liquid present but also the capability to use characteristic ions in extracted ion profiling (EIP) to identify ignitable liquid residues when high background levels of substrate materials or pyrolysis products are present.

1.3 References

1.3.1 ASTM Method E1388, Standard Practice for Static Headspace Sampling of Vapors from Fire Debris Samples

1.3.2 ASTM Method E1412, Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Activated Charcoal

1.3.3 ASTM Method E1386 Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction

1.3.4 ASTM Method E1618, Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry

1.3.5 ASTM Method E3197, Standard Terminology Relating to Examination of Fire Debris

1.3.6 Stauffer: Dolan: Newman, *Fire Debris Analysis,* 2008 Academic Press

1.3.7 ASTM Method E2451-, Standard Practice for Preserving Ignitable Liquids and Ignitable Liquid Residue Extracts from Fire Debris Samples

1.3.8 Lentini: Dolan: Cherry, *The Petroleum- Laced Background*, J Forensic Sci 2000: 45 (5) 968-989

1.3.9 ASTM Method E2881, Standard Test Method for Extraction and Derivatization of Vegetable Oils and Fats from Fire Debris and Liquid Samples with Analysis by Gas Chromatography-Mass Spectrometry

2.0 Scope

2.1 This method details the sampling, analysis, identification and reporting of ignitable liquids/residues from fire evidence samples as well as the care of the instrumentation utilized. Sampling is performed using any combination of the following; heated headspace, passive adsorption elution (PAE) with activated charcoal strips (ACS), or solvent extraction. Analysis of the resulting extract is performed using gas chromatography-mass spectrometry (GC-MS). The identification by GC-MS is based on the interpretation of data provided in accordance with the outlined criteria for identification. For PAE and solvent extraction the ACS, or extract is to be returned with the evidence for possible reanalysis. Extracts of diluted liquid samples do not need to be returned unless less than half of the original sample remains.

3.0 Equipment/Reagents

3.1 See specified sub-section for equipment and reagents.

3.2 All reference standards and reference materials should be stored according to manufacturer's recommendations. These materials should be kept in an explosion proof refrigerator or flammables cabinet and maintained at or below the recommended temperature. These items will not be shipped by ISPFS.

4.0 Procedure

4.1 Fire Evidence Handling

4.1.1 Fire Evidence Containers

4.1.1.1 Fire evidence must be submitted in clean, unused airtight containers.

4.1.1.2 Debris samples and clothing must be packaged in either a friction lid can, glass jar or a heat sealable polyethylene or nylon bag that is designed for use with fire evidence.

4.1.1.3 Glass or plastic vials should be used for liquids suspected of being or containing an ignitable liquid. The vial should be submitted in a friction lid can or heat sealable pouch with adsorbent material to prevent the contents of the vial from contaminating adjacent samples and to protect the vial from damage/preserve the sample in case of breakage.

4.1.1.4 Items not properly packaged may be repackaged and/or not analyzed due to contamination issues or loss of evidence. When the packaging has clearly compromised the integrity of the evidence, the sample(s) should not be analyzed. Any issue with the packaging that affects analysis will be listed on the report.

4.1.2 Fire Evidence Storage

4.1.2.1 Fire evidence should be stored under refrigeration until analysis. Exceptions can be made on a case-by-case basis after consultation with a trained analyst depending on the nature of the evidence.

4.1.2.2 Fire evidence said to contain substrate materials likely to contribute to the degradation of petroleum products such as manure, vegetation and/or soil, should be frozen until analysis. After consultation with a trained analyst exceptions can be made on a case-by-case basis depending on the nature of the evidence.

4.1.3 Description of Fire Evidence

4.1.3.1 The analyst shall note the type of container, condition of seals, a brief description of the sample and any obvious ignitable liquid product odor. If headspace sampling is used where a friction lid can is not opened before sampling, then after the analysis is complete the can will be opened so that an accurate description of the contents can be obtained. At that time, replace the punctured lid, if possible. If a replacement lid is not available, place the item in an arson bag.

4.1.3.2 When discernible, the nature of the substrate (carpet, wood, plastic, etc.) should be included in the description.

4.1.3.3 The evidence container labeling and contents shall be compared against the information provided by the agency. Any discrepancies are to be noted in case notes. If a major difference is observed the agency must be notified.

4.1.4 Recovery Method Approach

PAE is the sampling method that is employed most often. Headspace and solvent extraction will be done on a case specific basis based on information provided by the submitting agency and analyst experience.

4.1.5 Liquid Sample Screening

4.1.5.1 A drop or two of liquid sample is placed on glass wool and then exposed to flame. Observations are noted, i.e. does it ignite, color of flame, presence of smoke, soot, residue, etc. The report will state whether or not the sample ignited.

4.1.5.2 (Optional) Test the miscibility of the sample with known liquids. Most organic liquids are immiscible with water. Many common oxygenated compounds are miscible with water.

4.2 RECOVERY METHODS

4.2.1 **Separation of Ignitable Liquid Residues from Fire Evidence Samples by Headspace Sampling**

4.2.1.1 Background/References

4.2.1.1.1 This method serves to recover ignitable liquid and/or liquid residues from samples submitted as evidence in fire investigations.

4.2.1.1.2 This method involves sampling vapors in the headspace of a fire evidence collection container.

4.2.1.1.3 This separation protocol was derived from ASTM standard practice E1388: Standard Practice for Static Headspace Sampling of Vapors from Fire Debris Samples.

4.2.1.2 Scope

4.2.1.2.1 Headspace sampling should be used on samples suspected of containing light range (+/- C4-C9) constituents such as ketones, alcohols and chlorinated hydrocarbons which could have a reduced response using techniques involving a solvent.

4.2.1.2.2 If after sampling with Heated Headspace the sample is negative or the sample indicates the presence of a medium weight product/compound then the sample must be analyzed using a PAE.

4.2.1.2.3 Interfering compounds inherent in the fire debris recovered by this method may create GC-MS data interpretation problems. These compounds are the result of pyrolysis of solid fuels present at the fire scene, the combustion of organic compounds liberated during the fire process, and substrate background products.

4.2.1.2.4 This method lacks the sensitivity of other available recovery techniques. A negative result from the application of this technique should be interpreted as only indicating that a significant amount of a light range ignitable liquid may not be available for detection and that further analysis is required.

4.2.1.2.5 This method is more effective in recovering light to medium range products than heavy range products

4.2.1.3 Headspace Sampling Quality Assurance

4.2.1.3.1 For headspace sampling of reference material, place 15uL ignitable liquid reference material into to a quart size friction lid can, seal, and heat at both the time and temperature used for the samples. The volume used may need to be adjusted depending on response.

4.2.1.3.2 Headspace Controls

4.2.1.3.2.1 When headspace sampling is employed, a reference material of a light range ignitable compound and process blank must be analyzed prior to case samples.

4.2.1.3.2.2 A reference material of the analyte identified must be included in the run.

4.2.1.3.2.3 Additional reference materials may be analyzed at the discretion of the analyst.

4.2.1.3.2.4 For headspace sampling the corresponding amount of air from an empty evidence container, similar to the sample, will be injected using the same syringe as the sample as a process (system) blank.

4.2.1.4 Safety Concerns

4.2.1.4.1 Care should be taken with the handling heated headspace sampling syringes.

4.2.1.4.2 Given the unknown nature of samples, olfactory screening may pose a health risk and is not recommended. Note obvious odors to aid in the selection of parameters applied to the recovery technique.

4.2.1.5 Equipment and Supplies

4.2.1.5.1 Laboratory oven capable of providing uniform heating of samples from 60° to 90°C.

4.2.1.5.2 Every two years the thermometer in the oven is compared to a NIST traceable thermometer. The accuracy must be within (+/-) 5 °C. This verification may be done by either laboratory staff or by external personnel.

4.2.1.5.3 Hammer and nail, awl, or punch.

4.2.1.5.4 Foil adhesive tape.

4.2.1.5.5 Gas tight syringes capable of sampling from 250µL to 1mL.

4.2.1.6 Headspace Preparation

4.2.1.6.1 Friction Lid Can

Prepare the can for headspace sampling by using the hammer and nail/awl/punch to punch a small hole in the friction lid. The hole must be immediately covered with foil adhesive tape.

4.2.1.6.2 Plastic Fire Debris Bag

If the sample is submitted in polymer pouch material, insert needle directly through pouch material for sampling. Place a piece of tape over hole until pouch can be re-heatsealed.

4.2.1.6.3 Container or Vial of liquid

Packaging of liquid sample can be opened and needle used to draw from headspace or place 15ul on a laboratory wipe in a friction lid can and processed by 4.2.1.6.1. Headspace sampling of a liquid should only be performed at room temperature.

4.2.1.7 Headspace Sampling

4.2.1.7.1 Samples may be collected at room temperature or placed in a 60° to 90°C oven, as appropriate..

4.2.1.7.2 If the sample is heated, the gas tight syringe shall be placed into the oven at the same temperature the sample is being heated at.

4.2.1.7.3 After inserting the needle of the gas tight syringe through the can sampling hole or arson bag, slowly pump the syringe several times and inject 500uL to 1 mL into GC injection port. Immediately re-tape sampling hole. Smaller volumes may be injected as necessary to reduce the risk of instrument overload.

4.2.1.7.3.1 Due to the gas tight syringe being inserted through adhesive, maintenance may need to be done to the injection port and the needle of the syringe may need to be cleaned with MeOH.

4.2.1.7.4 Analyze by GC-MS as described in section 4.3.4 using the following acquisition parameters:

Initial Temp 35°C	
Hold	1.5 min
Rate 1	10°C/min to 100°C
Rate 2	25°C/min to 300°C
Hold 2 min	
Injector 280°C	
Split	50:1
Aux temp 310°C	
Scan mass 30-350	
Detector shut off 10min	

The analyst may vary GC/MS instrument parameters without performance verification other than a positive control. This includes parameters such as changing the inlet temperature, temperature ramp and/or hold time, pressure program, injection volume, split ratio, and ion scan range. Acquisition parameters are stored with the data files.

4.2.2 **Separation of Ignitable Liquid Residues from Fire Evidence Samples by Passive Adsorption-Elution (PAE) with Activated Charcoal**

4.2.2.1 Background/References

4.2.2.1.1 This method serves to recover ignitable liquid residues from samples submitted as evidence in fire investigations.

4.2.2.1.2 This method utilizes polymer strips impregnated with activated charcoal (ACS) to adsorb, and thus trap, ignitable liquid residues.

4.2.2.1.3 This method recovers ignitable liquids for subsequent instrumental analysis using gas chromatography-mass spectrometry (GC-MS).

4.2.2.1.4 This method is time efficient and is essentially non-destructive to the sample.

4.2.2.1.5 This separation protocol is derived from ASTM standard practice E1412: Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Activated Charcoal.

4.2.2.2 Scope

4.2.2.2.1 This procedure is useful for recovering light to medium range (>C6 → ≅C14) ignitable liquids. Compounds longer than hexadecane (C16) may not be effectively volatized for adsorption by ACS.

4.2.2.2.2 Samples containing high levels of light to medium range ignitable liquids are prone to displacement and thus loss of light petroleum product components.

4.2.2.3 Equipment and Supplies

4.2.2.3.1 Chemical fume hood

4.2.2.3.2 Activated charcoal polymer strips (ACS) (Albrayco Laboratories ACS-150-6PACK or equivalent). Each strip is approximately 10 x 20mm for a total area of 200mm^2. ASTM E1412- recommends a minimum strip size of 100mm^2.

4.2.2.3.3 Metal, non-coated, paper clips or ornament hangers.

4.2.2.3.4 Magnet capable of securing paper clip to lid of friction lid can.

4.2.2.3.5 Large laboratory oven for uniform heating of friction lid cans and fire debris bags at 50-80°C.

4.2.2.3.6 Every two years the thermometer in the oven is compared to a NIST traceable thermometer. The accuracy must be within (+/-) 5 °C. This verification may be done by either laboratory staff or by external personnel.

4.2.2.3.7 2-mL wide-mouth automatic liquid sampler (ALS) vials with PTFE/silicone septa

4.2.2.3.8 Glass pipettes

4.2.2.3.9 Glass vial 350-400ul microinserts
4.2.2.3.10 Nylon evidence bags/sheet
4.2.2.3.11 Heat sealer

4.2.2.4 Reagents

4.2.2.4.1 Carbon disulfide (CS_2)

Solvent must meet American Chemical Society (ACS) specifications and/or be 99.9% pure. Each bottle must be checked prior to use as described in section 4.2.2.11.3.

4.2.2.4.2 Thiophene internal standard is added to carbon disulfide at a 100ppm concentration (50ul thiophene to 500mls of CS_2)

Thiophene must meet American Chemical Society (ACS) specifications.

4.2.2.5 Reference Material

4.2.2.5.1 Reference material can be run diluted or after recovery with this recovery technique as described in section 4.2.2.10.

4.2.2.5.2 Refer to section 4.3.3 for ignitable liquid test mixture and reference material requirements and appropriate dilutions.

4.2.2.6 Safety Concerns

4.2.2.6.1 Carbon disulfide is an extremely flammable, volatile liquid. All routes of exposure to carbon disulfide have adverse effects on the central nervous system with a NFPA health rating of 3 (severe). Care should be taken to protect all routes of exposure from contact with carbon disulfide; as well as keeping the solvent well away from heat sources.

4.2.2.6.2 For further information regarding the hazards of exposure to carbon disulfide refer to safety data sheets (SDS).

4.2.2.6.3 Given the unknown nature of case samples, olfactory screening may pose a health risk and is not recommended. Obvious odors should however be noted to aid in the selection of parameters applied to the recovery technique(s).

4.2.2.7 Passive Adsorption Procedure

4.2.2.7.1 Secure an activated charcoal strip (ACS) with a paper clip or hanger.

4.2.2.7.1.1 Note lot number of ACS on analysis worksheet.

4.2.2.7.2 Open friction lid can or arson bag and quickly note contents and any obvious odor present.

4.2.2.7.3 Suspend ACS over the sample, seal container, place into arson bag or secure lid with clips, to prevent loss.

4.2.2.7.4 Heat container for 2-24 hours at 60-90° C. Record time and temperature conditions in notes. Room temperature analysis can be performed on a case by case basis at the analyst's discretion.

4.2.2.7.5 As described in E1412, important considerations for the optimum adsorption time for representative sampling and/or maximum sensitivity are the adsorption temperature and the ignitable liquid composition and concentration.

4.2.2.7.6 At the completion of the adsorption period, remove the evidence from the oven and allow to cool to room temperature.

4.2.2.7.7 Label two ALS vials containing microinserts. Using the same pipette, rinse one with solvent and place the solvent into the second vial which is used as a sample blank. Into the first vial add the ACS and then solvent and cap immediately. The ACS strip may be cut in half prior to placing in the vial with solvent. Half is placed in the first vial and half is placed in a third vial for retention. Change gloves between samples.

4.2.2.7.8 Document the source and lot number of CS_2 and thiophene used on the analysis worksheet.

4.2.2.7.9 After initial examination of the sample, the CS_2 extract may be diluted for GC-MS analysis. Record the approximate dilution volume.

4.2.2.8 Resampling of Item

If initial GC-MS data indicates that the strip was overloaded, the sample may be resampled at ambient temperature or with a shortened adsorption time.

4.2.2.9 Packaging of Retention ACS

The retained unextracted half of the ACS or the whole ACS after evaporating the solvent, returned to evidence or place into a new evidence envelope to be returned to the submitting agency. If the vial is placed in a new envelope, be sure to create the new item of evidence in the ILIMS system before the report is written.

4.2.2.10 ACS Recovery of Reference Material

4.2.2.10.1 Place 1 to 10µL ignitable liquid reference material into a clean, unused quart sized friction lid can, and seal.

4.2.2.10.2 Secure ACS on paper clip or hanger.

4.2.2.10.3 Place friction lid can into the oven for the same time and temperature as the samples.

4.2.2.10.4 Process charcoal strip as with case samples.

4.2.2.11 Quality Assurance

4.2.2.11.1 Charcoal Strip Blanks (preparation blank)

4.2.2.11.1.1 A charcoal strip blank must be run with each case.

4.2.2.11.1.2 Place ACS and hanging device into a heat sealable bag or friction lid can.

4.2.2.11.1.3 Process charcoal strip with a solvent blank.

4.2.2.11.1.4 GC-MS data (TIC) for the preparation blank must be placed into case file.

4.2.2.11.2 New ACS Lot

4.2.2.11.2.1 Each new lot of ACS must be checked for contamination and relative recovery prior to extraction of samples.

4.2.2.11.2.2 Contamination should be checked for by preparing a blank as described in section 4.2.2.7.

4.2.2.11.2.3 Relative recovery should be verified with 10µL of 50% gasoline and 50% heavy petroleum distillate..

4.2.2.11.2.4 ACS lot GC-MS data must be centrally stored on the common forensic drive.

4.2.2.11.3 New Elution Solvent Bottle

4.2.2.11.3.1 Relative contamination must be checked for in each new bottle of CS_2, regardless of lot, both before and after addition of thiophene.

4.2.2.11.3.2 Evaporate a volume of CS2 by 50% and analyze. If the results are acceptable, add thiophene to the bottle and analyze again without evaporation

4.2.2.11.3.3 If present, the relative toluene and or benzene concentration must be noted. Solvent should be free of other identifiable ignitable liquids.

4.2.2.12 Analysis

Analyze extracts by GC-MS as outlined in section 4.3 of this method using the following acquisition parameters:

Initial Temp 35°C	
Hold	1.5 min
Rate 1	10°C/min to 100°C
Rate 2	25°C/min to 300°C
Hold 2 min	
Injector 280°C	
Split	50:1
Aux temp 310°C	
Scan mass 30-350°C	

The analyst may vary GC/MS instrument parameters without performance verification other than a positive control. This includes parameters such as changing the inlet temperature, temperature ramp and/or hold time, pressure program, injection volume, split ratio, and ion scan range. Acquisition parameters are stored with the data files.

4.2.3 **Separation and Concentration of Ignitable Liquid Residues from Fire Evidence Samples by Solvent Extraction**

4.2.3.1 Background/References

4.2.3.1.1 This method serves to recover ignitable liquid residues from samples submitted as evidence in fire investigations.

4.2.3.1.2 This method applies the principle "Like dissolves like". Ignitable liquid/residue in the sample is recovered with an organic solvent which is filtered and concentrated if necessary.

4.2.3.1.3 This separation protocol is derived from ASTM standard practice E1386-Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction.

4.2.3.2 Scope

4.2.3.2.1 This recovery technique may be applied to samples which may contain heavy petroleum distillates as indicated by a strong odor and/or GC/MSD data from analysis of passive headspace ACS recovered extract.

4.2.3.2.2 Samples which contain a high percentage of charred debris and tend to hold ignitable liquid residues more tightly.

4.2.3.2.3 Samples which contain non-porous surfaces such as glass or burned containers.

4.2.3.2.4 Aqueous samples and debris samples containing a significant amount of water.

4.2.3.2.5 Samples which have a significant odor of an ignitable liquid.

4.2.3.2.6 Liquid samples suspected to contain an ignitable liquid.

4.2.3.2.7 Interfering compounds, inherent in the fire debris recovered by this method, may create GC-MS data interpretation problems. These compounds are the result of pyrolysis of solid fuels present at the fire scene, the combustion of organic compounds liberated during the fire process, and substrate background products.

4.2.3.2.8 The evaporation step in this method may lead to the loss of light ignitable liquid products or lighter components of medium to heavy range products. Close monitoring of the evaporation process should reduce losses.

4.2.3.2.9 Lighter compounds may not be detected by this technique due to the requirement for a solvent delay during GC-MS analysis. Any compound with a retention time during the solvent delay will not be detected by this technique.

4.2.3.2.10 Due to the nature of the solvent extraction process, this technique may render the extracted sample unsuitable for resampling. For this reason, only a representative portion of the sample should be subjected to this technique and the extracts returned with the evidence.

4.2.3.3 Safety Concerns

4.2.3.3.1 Pentane is an extremely flammable, volatile liquid. Although pentane only has a NFPA health rating of 1 (slight), exposure through inhalation and ingestion has an adverse effect on the central nervous system. Skin contact causes irritation. Care should be taken to protect all routes of exposure from contact with pentane as well as keeping the solvent well away from heat sources.

4.2.3.3.2 For further information regarding the hazards of exposure to extraction solvents refer to safety data sheets (SDS).

4.2.3.3.3 Given the unknown nature of samples, olfactory screening may pose a health risk and is not recommended. Note obvious odors to aid in the selection of parameters applied to the recovery technique.

4.2.3.4 Equipment and Supplies

4.2.3.4.1 Chemical fume hood

4.2.3.4.2 Tube rocker.

4.2.3.4.3 Filter Paper

4.2.3.4.3.1 Whatman Grade 1 - Qualitative (27cm) or equivalent

4.2.3.4.3.2 Whatman 1PS -Phase separation (15cm) or equivalent

4.2.3.4.4 Glassware

4.2.3.4.4.1 Assorted sizes of glass funnels

4.2.3.4.4.2 Assorted 25mL to 2000mL glass beakers

4.2.3.4.4.3 Assorted sizes of screw-top extraction tubes and screw caps (no adhesive liners).

4.2.3.4.4.4 2-mL wide-mouth automatic liquid sampler (ALS) vials preassembled with screw-top cap with PTFE/silicone septa

4.2.3.4.4.5 Glass vial microinserts

4.2.3.5 Reagents

4.2.3.5.1 Pentane (Pesticide Grade or equivalent)

4.2.3.5.2 Carbon disulfide (CS_2)

Solvent must meet American Chemical Society (ACS) specifications and/or be 99.9% pure. Each bottle must be checked prior to use as described in section 4.2.2.11.3.

4.2.3.6 Solvent Extraction - Non-liquid Samples

4.2.3.6.1 Transfer a representative portion of sample to an appropriately sized glass beaker.

4.2.3.6.2 Add sufficient pentane or carbon disulfide to thoroughly wet the sample.

4.2.3.6.3 Mix/soak the sample for approximately one to five minutes. A beaker may be used as a plunger to facilitate the extraction of flexible material. Note lot and source of extraction solvent on fire evidence worksheet.

4.2.3.6.4 If necessary, filter solvent through phase separation paper supported with Grade 1 filter paper.

4.2.3.6.5 Place ≅2mL aliquot of solvent into a 2mL ALS vial prior to evaporation. Additional evaporation may not be necessary. This sample may be diluted as necessary.

4.2.3.6.6 If necessary, allow solvent to evaporate approximately 50%. Transfer an aliquot of the partially evaporated sample to a glass insert in a labeled ALS vial. Additional aliquots at higher levels of evaporation may be collected at the discretion of the analyst.

4.2.3.6.7 Continue to evaporate remaining solvent. Concentrate extract down to approximately 1mL. Place the sample into labeled ALS vial.

4.2.3.7 Solvent Extraction - Unknown Liquid Samples

4.2.3.7.1 Examine liquid and note the number of layers present.

4.2.3.7.2 If more than one layer is present, transfer one drop of the organic layer of liquid to a labeled ALS vial containing 2mL of carbon disulfide and mix.

4.2.3.7.3 If only one layer is present or you choose to analyze the aqueous layer of a two-layer liquid after analyzing the organic layer, extract/dilute an aliquot with carbon disulfide and analyze.

4.2.3.7.4 For all extraction situations, note lot and source of extraction solvent on fire evidence worksheet.

4.2.3.8 Solvent Purity Check

4.2.3.8.1 When solvent used for extraction is subjected to evaporation, the batch of samples should include a process blank, to include solvent, phase separation paper and filter paper, as appropriate.

4.2.3.8.1.1 Place approximately 200mL of extraction solvent into a clean beaker.

4.2.3.8.1.2 Evaporate solvent to approximately 1mL.

4.2.3.8.1.3 The degree of solvent evaporation should be the same conditions as used for questioned samples and in accordance with ASTM E1618 (ASTM E1386, 5.2.2).

4.2.3.8.1.4 When the extraction solvent is used only to dilute a suspected ignitable liquid, it does not have to be subjected to an evaporation check.

4.2.3.9 Analysis

Analyze extracts by GC-MS as described in section 4.3. If a partially evaporated extract provides sufficient sensitivity, the more evaporated extract(s) need not be analyzed.

4.3 ANALYSIS OF RECOVERED EXTRACTS

4.3.1 Background/References

4.3.1.1 To detect the presence of an ignitable liquid in fire evidence samples, extracts recovered by headspace sampling, activated charcoal and/or solvent extraction are analyzed using gas chromatography-mass spectrometry (GC-MS)

4.3.1.2 Post-run macros, which process the data to generate extracted ion profiles, can be utilized to assist with the detection and identification of ignitable liquid residues.

4.3.1.3 This analysis approach is derived from ASTM E1618 Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry

4.3.2 Equipment

4.3.2.1 Instrumentation

Integrated system consisting of a gas chromatograph-mass spectrometry. Data system must be capable of handling macro programs to extract ion profiles to assist with the interpretation of data.

4.3.2.2 Column

30 meter Agilent HP-5MS [(5% PhMeSilcone) with 0.25 mm ID and 0.25 μm film thickness], or comparable. The column, with appropriate carrier gas flow and temperature program, must have the capability to adequately separate the components of the column resolution test mixture (refer to 4.3.3.1).

4.3.3 Chemicals and Reference Materials

4.3.3.1 ASTM Test Mixture (Test Mix)

4.3.3.1.1 The minimum requirements for the test mixture are described in E1618-19, 6.4. Compliant test mixtures can be obtained through Restek (#31224), Cerilliant (ERR-002) or an equivalent vendor/product.

4.3.3.1.1.1 Dilute stock test mixture 10:1 with carbon disulfide/thiophene.

4.3.3.1.1.2 Authenticate each component (except hexane) via library search (>85%) each time a new ampule is opened. The overall pattern should be similar to the previous lot/ampule.

4.3.3.1.1.3 Document the authentication of the test mix by initialing and dating the data printout which will be centrally stored.

4.3.3.1.1.4 Since the contents of each ampule are authenticated, any manufacturers' expiration dates can be ignored.

4.3.3.1.2 The dilute test mix will be analyzed by direct injection and the TIC results will be placed in the case file.

4.3.3.1.3 All constituents of this test mix with the exception of hexane must be present.

4.3.3.1.4 The test mix should be stored in the freezer.

4.3.3.2 Ignitable Liquid Reference Material

Ignitable liquids for use as reference material may be obtained as required from commercial and local sources. A reference material collection including weathered/evaporated commonly encountered ignitable liquids must be available. Reference material may be diluted as described below or neat products may be recovered by either headspace sampling or ACS.

4.3.3.2.1 Dilution of Reference Material

4.3.3.2.1.1 Neat Ignitable Liquid Products

For most neat liquids a 1:1000 dilution is appropriate. Single component liquids may require a 1:4000 dilution. Additional dilutions may be made as required.

4.3.3.2.1.2 Diluted Commercial Products

Most commercially obtained ignitable liquid reference materials should be diluted 1:10. Dilution should be adjusted as necessary.

4.3.3.2.2 Authentication of Ignitable Liquid Reference Material

4.3.3.2.2.1 The authentication of ignitable liquid reference materials presents a unique problem since the standard may be a multi-component mixture whose identification is based largely on total ion chromatograms (TIC) and extracted ion profiles (EIP) or a single component product whose identification is based mostly on the comparison of mass spectra.

4.3.3.2.2.2 Authentication of reference materials is based upon interpretation of the TIC, EIP and mass spectral data obtained through the analysis of the reference material.

4.3.3.2.2.3 When available, the analytical data should be compared with the manufacturer’s package insert/information packet or SDS.

4.3.3.2.2.5 Authentication documentation must be stored in the laboratory performing the analysis.

4.3.3.2.3 Application of Ignitable Liquid Reference Material

Appropriate ignitable liquid reference material(s) (IRM(s)) from the reference collection will be run within seven days of the samples.

4.3.4 GC/MSD Analysis

4.3.4.1 Instrumental Parameters

Instrumental parameters are stored electronically as part of the data file.

4.3.4.2 MSD Tuning

4.3.4.2.1 An Autotune must be performed monthly (maximum 45 days prior to sample analysis). A tune evaluation will be run each week the instrument is used. A week is defined as any seven-day period from the last tune evaluation.

4.3.4.2.2 All parameters evaluated for the Autotune/Tune Evaluation must fall within manufacturer recommended ranges.

4.3.4.2.3 Autotunes/Tune Evaluations must be centrally stored in the laboratory performing the analysis.

4.3.4.3 GC-MS Quality Assurance Sample Requirements

4.3.4.3.1 Sample Blanks

4.3.4.3.1.1 Analyzing a sample blank between case samples will indicate if any carry over is occurring.

4.3.4.3.1.2 For ACS recovery, a CS_2 solvent, with thiophene, blank must be analyzed.

4.3.4.3.1.3 For solvent extraction, the solvent used for extraction must be used for the solvent blank.

4.3.4.3.1.4 TIC from BLANK must be placed into case file. If any peaks of interest are present, the MS for peaks of interest must also be placed in the case file. Peaks of interest are defined as compounds or mixtures of compounds that would be identified as ignitable liquids.

4.3.4.3.1.5 If peaks of interest are identified in the blank that could affect the interpretation of sample results, the blank and sample may need to be reinjected and/or re-extracted.

4.3.4.3.2 Performance Verification Test Mixture

4.3.4.3.2.1 To establish that the system has the capability to resolve compounds as outlined in ASTM E1618, a test mix must be analyzed within seven days prior to the beginning of the analysis sequence.

4.3.4.3.2.2 When headspace sampling is used at least one of the commonly encountered light compounds must be included in the analysis run to verify detection ability, if a light weight compound is detected, the reference material run for the case will be acceptable.

4.3.4.3.2.3 The ASTM test mixture will be evaluated and documented in the case file.

4.3.4.3.3 Carrier Gas Purity

To prevent contamination, a carrier gas of appropriate quality will be used.

4.3.4.4 Maintenance

Maintenance of the GC/MS shall be performed according to the schedules outlined in the Controlled Substances manual.

4.3.5 GC/MSD Data Analysis

Data processing capabilities should include both the generation of total ion chromatograms (TIC) and extracted ion profiles (EIP) of characteristic major ion fragments for each of the major type of compounds found in ignitable liquids. TICs and EIPs from case samples are compared against TICs and EIPs prepared for reference materials. EIPs may be used to assist with the interpretation of the TIC but must not be the sole basis for the conclusions reached.

4.3.5.1 Deuser.mac

The macro generates the TIC and individually printed selected ion profiles. Individual profiles allow the analyst to see less abundant ions indicative of certain classes of compounds in greater detail than summed profiles.

4.3.5.1.1 This macro may be set up to run at the end of an analysis run or can be accessed in Data Analysis. In Data Analysis, load the method containing the *deuser* macro and a data file. From the method pull down, select run. Ions included are at a minimum:

Major Compound Types	**Major Ion Fragments**			
Alkanes	43	57	71	85
Aromatics	91	105	119	133
Cycloalkane/alkene	55	69	82	83
Indanes	117	118	131	132

PNAs(Polynuclear aromatics/naphthalenes)	128	142	156	170
Ketones	43	58	72	86
Alcohols	31	45		

4.3.6 Instrument Maintenance

4.3.6.1 Refer to the Controlled Substances Analytical Method for maintenance indicators, schedules, and requirements.

4.4 INTERPRETATION OF ANALYTICAL DATA

4.4.1 *E1618* Classification system

4.4.1.1 Classification system consists of seven major classes of ignitable liquids and one miscellaneous class.

4.4.1.2 All classes except gasoline are further differentiated into light, medium or heavy subclasses based on carbon spread/n-hydrocarbon boiling range.

4.4.1.2.1 The subclasses allow for the characterization of products, which fall between classes due to evaporation/weathering. A product may be described as "light to medium" or "medium to heavy" when the carbon number range does not fit neatly into one of the below categories. If this option is appropriate, the carbon number range should be reported.

4.4.1.2.2 The products listed in *Table 1* are examples of commercially available products which fall within each classification and are not intended to be all-inclusive. Additional references should be used to provide more specific examples for each class.

4.4.2 Identification of an Ignitable Liquid

4.4.2.1 Compare data from case sample TIC and EIPs with data from reference material obtained with the same GC-MS parameters.

4.4.2.2 The characteristics which establish the presence of an ignitable liquid is set forth in Section 10 of ASTM E1618: Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.

4.4.2.3 The characteristics are to be used as the basis for the identification of an ignitable liquid by this analytical method.

4.4.2.4 The analyst must consider and be prepared to explain any observed differences between the unknown (questioned sample) and a known reference material.

4.4.2.4.1 Variables that should be taken into account include the influence of evaporation, interference from burned substrate materials, sample preparation techniques and/or bacterial degradation (soil samples).

4.4.2.4.2 Unexplained differences should preclude the identification as an ignitable liquid.

4.4.2.5 A copy of the relevant reference material supporting the conclusions reached must be placed into the casefile.

Table 1: *E1618* Ignitable Liquid Classification Scheme with Examples of Known Products for Each Class

Class	**Light {C_4 - C_9}**	**Medium {C_8 – C_{13}}**	**Heavy {C_8 – C_{20+}}**
Gasoline All brands, including gasohol and E-85	Fresh gasoline is typically in the C_4 –C_{12} range		
Petroleum Distillates (including de-aromatized)	Petroleum Ether Cigarette Lighter Fluids Camping Fuels Naphtha	Charcoal Starters Paint Thinners Dry Cleaning Solvents Mineral Spirits Automotive Parts Cleaners Spray Lubricants Lamp Oils Deck Sealers Varnishes Kerosene Insecticides	Kerosene Diesel Fuel Charcoal Starters Aviation Fuels Insecticides Fuel Additives Lamp Oils Automotive Parts Cleaners
Isoparaffinic Products	Aviation Gasolines Lighter Fluids Charcoal Starters	Charcoal Starters Paint Thinners Copier Toners Mineral Spirits Solvent Cleaners Kerosene Lamp Oils Gun Oils	Spot Cleaners Penetrating Oils Insecticides
Aromatic Products	Automotive PartsCleaners Solvent Cleaners Xylenes Toluene-based Products Lacquer Thinners	Automotive Parts Cleaners Specialty Cleaning Solvents Insecticides Brush Cleaners	Insecticides Adhesives
NaphthenicParaffinic Products	Cyclohexane based solvents/products	Charcoal Starters Insecticides Lamp Oils Mineral Spirits Automotive Parts Cleaners	Insecticides Lamp Oils
Normal Alkanes Products		Candle Oils Copier Toners Lamp Oils Wax Cleaners	Candle Oils Lamp Oil Carbonless Forms Copier Toners
Oxygenated Solvents	Alcohols Ketones Lacquer Thinners	Metal Cleaners Gloss Removers Degreasers	Biodiesels Fuel Additives Floor Finishes

	Fuel Additives Surface Preparation Solvents Automotive Parts Cleaners Spray Adhesives Brush Cleaners	Furniture Strippers Cleaning Solvents Insecticides	Insecticides
Other-Miscellaneous	Single Component Products Enamel Reducers Lacquer Thinners Aviation Gasolines Racing Gasolines	Turpentine Products Mineral Spirits Fuel Additives Spray Lubricants Brush Cleaners Paint Thinners Citrus Cleaners Charcoal Starters	Lamp Oils Insecticides Citrus Cleaners Automotive Parts Cleaners Kerosene Fuel Additives

4.5 ANALYSIS REPORT

4.5.1 Fire Evidence Notes

4.5.1.1 Description of Evidence

4.5.1.1.1 In the notes the description of the evidence shall contain at least the following information, the item number, the agency exhibit number (when available), condition of seals, and type of container and details of the type of evidence.

4.5.1.1.2 The description may include the location the evidence was said to have been recovered.

4.5.1.2 Analysis Results

4.5.1.2.1 Positive Results

4.5.1.2.1.1 When the criteria for identification of an ignitable liquid are met, the analysis results must indicate the class which the ignitable liquid falls. The carbon range (light, medium, or heavy) as described must be indicated for all classes except gasoline.

4.5.1.2.1.2 Following the class designation, a listing of potential sources of the ignitable liquid must be compiled and listed.

4.5.1.2.1.3 A disclaimer indicating that positive results should be considered in context with all available information and do not automatically lead to the conclusion that a fire was incendiary in nature, may be added to the analysis report.

4.5.1.2.2 Negative Results

4.5.1.2.2.1 When the criteria for identification of an ignitable liquid are not met, the analysis results must indicate that no ignitable liquids were detected in the sample.

4.5.1.2.2.2 A disclaimer indicating that negative results do not preclude the possibility that ignitable liquids were present at the fire scene, may be added to the analysis report.

4.5.1.2.3 Terpenes

Turpentine is a volatile essential oil obtained from the oleoresin of coniferous trees. The presence of the naturally occurring alkenes, terpenes (oleoresins) should be considered in context with the makeup of the sample.

4.5.1.2.3.1 When a comparison sample is available and clearly shows the same distribution of terpene constituents, a questioned sample containing coniferous plant material may be reported out as negative.

4.5.1.2.3.2 When in the absence of a comparisonsample, significant levels of terpenes are noted in a sample containing coniferous plant material, a qualifier should be used. The qualifier should read to the effect that the sample contains terpenes, which may have originated from coniferous plant material in the sample or from a medium range turpentine product.

4.5.1.2.3.3 When a comparison sample of the involved wood is available and does not indicate the presence of terpenes, it is at the discretion of the analyst as to whether the qualifier is used.

4.5.1.2.3.4 When the sample clearly does not contain coniferous plant material which would contribute to the presence of terpenes, a qualifying statement is not necessary. The sample should be reported as containing a medium range turpentine product.

4.5.1.2.4 Questionable Results

4.5.1.2.4.1 In situations where the analysis data is inconclusive, the analysis results may indicate that no ignitable liquids were *identified* in the sample.

4.5.1.2.4.2 When a comparison (substrate) sample is not available and the material is one known to have the potential of contributing the ignitable liquid identified, two options are available. Examples of this include distillates in newspaper and varnished flooring.

Option One:

The sample can be reported out as *no ignitable liquids identified* (see 4.5.1.2.4.1 above).

Option Two:

The ignitable liquid may be identified but a qualifying statement must be placed on the analysis report. When appropriate, the qualifier must state that the sample was found to contain the ignitable liquid but due to the lack of a comparison sample for the substrate it is unknown whether the ignitable liquid originated from (endogenous to) the substrate itself.