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**Idaho State Police
Forensic Services
Trace Discipline**

Section I

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

1.1 BACKGROUND

The analysis of evidence from fires of suspicious origin encompasses a variety of sample types. The submitted samples may include burnt fire debris and liquids recovered at the fire scene, the suspect's clothing, and/or ignitable liquids found in the suspect's possession. The examination of the evidence may involve only an identification of ignitable liquid/residue present or it may involve the comparison of an ignitable liquid recovered at the scene with a liquid recovered from the suspect. Establishing the presence of an ignitable liquid residue in burnt debris recovered from a fire scene is 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 goal of this recovery process is to produce an extract as free from interferences as possible yet still recover a good representation of any ignitable liquid product present. Other considerations include recovery techniques that are sensitive and efficient. The analysis of recovered vapors and/or extracts by gas chromatograph equipped with a mass spectrometer 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. The presence of an ignitable liquid residue in fire debris or on a suspect's clothing should be considered in the context of the circumstances surrounding the fire. The detection of an ignitable liquid does not determine that the fire was arson without supporting information.

1.2 SCOPE

This method details the recovery, analysis, identification and reporting of ignitable liquids/residues from fire evidence samples as well as the care of the instrumentation utilized. Recovery is by heated headspace, passive headspace adsorption with activated charcoal strips (ACS) and/or solvent extraction. Analysis of the resulting extract is performed on a gas chromatograph equipped with a mass selective detector. The identification by GC-MSD is based on the interpretation of data provided in accordance with the outlined criteria for identification. For passive headspace adsorption, the practice of recovery with two ACS allows for one of the ACS to be placed with the evidence upon completion of analysis to be available for reanalysis. This analytical method is based upon corresponding American Society for Testing and Materials (ASTM) standard test methods and practices used in Forensic Science, that address the recovery and analysis of ignitable liquid residues (ILR) from fire evidence.

1.3 FIRE EVIDENCE HANDLING

1.3.1 Fire Evidence Containers

- 1.3.1.1 Fire evidence must be submitted in clean, airtight containers.
- 1.3.1.2 Debris samples and clothing must be packaged in a friction lid can and/or Kapak[®] or comparable heat sealable pouch material.
- 1.3.1.3 Glass vials should be used for liquids suspected of being or containing an ignitable liquid. The glass vial must be submitted in a friction lid can or heat sealable pouch material to prevent the contents of the vial from contaminating adjacent samples.
- 1.3.1.4 Items not properly packaged may be repackaged and/or not analyzed due to contamination issues. When the packaging has clearly compromised the integrity of the evidence, the sample(s) should not be analyzed.

1.3.2 Fire Evidence Storage

- 1.3.2.1 Fire evidence must not be stored near a heat source.
- 1.3.2.2 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 refrigerated or frozen until analysis.

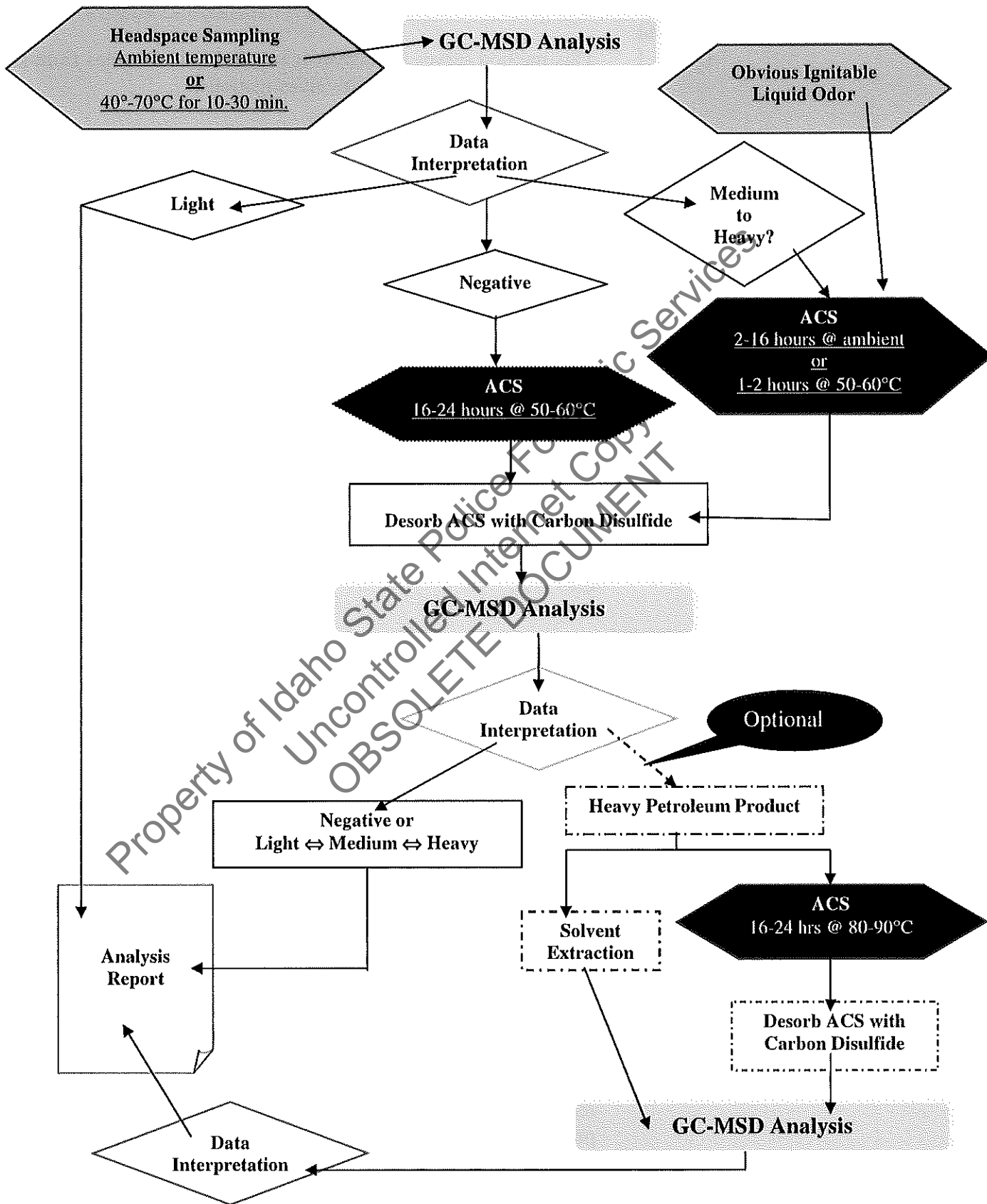
1.3.3 Description of Fire Evidence

- 1.3.3.1 On fire evidence worksheet, the analyst should note the type of container, whether the seals are intact, a brief description of the sample and any obvious ignitable liquid product odor.
- 1.3.3.2 When discernable, the nature of the substrate (carpet, wood, plastic, etc.) should be included in the description.
- 1.3.3.3 The evidence container labeling and contents should be compared against the evidence submittal form. Any discrepancies are to be noted in case notes.

1.3.4 Recovery Method Approach

- 1.3.4.1 Headspace sampling (1.4.1) should be done based on obvious/significant odors and/or case scenario. Based on headspace sampling data, additional techniques (1.4.2 and 1.4.3) may be indicated.
- 1.3.4.2 If headspace sampling is not warranted, the scheme should begin with recovery by passive headspace concentration with activated charcoal (1.4.2). Refer to analysis schemes on page 3 for options.

ANALYSIS SCHEMES FOR EXTRACTION OF FIRE DEBRIS



1.4 RECOVERY METHODS

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

1.4.1.1 **General**

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

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

1.4.1.2 **ASTM Reference Method**

This separation protocol was derived from ASTM standard practice E 1388-05: *Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples*.

1.4.1.3 **Application**

1.4.1.3.1 The screening of samples to determine relative ignitable liquid concentrations and possible ignitable liquid class and range prior to extraction with other techniques.

1.4.1.3.2 Samples suspected of containing light range constituents such as ketones, alcohols and chlorinated hydrocarbons which would not be detected by techniques involving a solvent.

1.4.1.3.3 Liquid samples suspected to contain a light range ignitable liquid (\pm C4 - C9).

1.4.1.4 **Sensitivity**

This separation protocol is the least sensitive of the recovery techniques covered in this analytical method.

1.4.1.5 **Limitations**

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

1.4.1.5.2 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.

1.4.1.5.3 This method is more effective in recovering light to medium range products than heavy range products. The higher temperatures required to recover heavy range products could result in the production of additional decomposition of the sample matrix.

1.4.1.6 **Reference Material**

1.4.1.6.1 Refer to section 1.4.1.12 for headspace sampling reference material preparation.

1.4.1.6.2 Refer to section 1.5.4.2 for headspace test mixture requirements.

1.4.1.7 **Safety Concerns**

1.4.1.7.1 Care should be taken with the handling headspace sampling syringes.

1.4.1.7.2 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.

1.4.1.8 **Equipment and Supplies**

1.4.1.8.1 Laboratory oven capable of providing uniform heating of samples from 40° to 70°C.

1.4.1.8.2 Hammer and nail.

1.4.1.8.3 Adhesive tape.

1.4.1.8.4 Gas tight syringes capable of sampling from 10µL to 500µL.

1.4.1.9 **Headspace Preparation - Non-liquid Samples**

1.4.1.9.1 Friction Lid Can

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

1.4.1.9.2 Kapak[®]-type Material

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-heat-sealed.

1.4.1.10 **Headspace Preparation - Liquid Samples**

1.4.1.10.1 Examine liquid and note the number of layers present.

1.4.1.10.2 If more than one layer is present, transfer 15µL of top layer of liquid to a quart size friction lid can with a taped sampling hole or other clean, airtight container that allows for headspace sampling.

1.4.1.10.3 If only one layer is present, remove a small portion of the liquid and place into an appropriately sized sealable glass extraction tube. Add an equal volume of carbon disulfide and mix. Note and record on analysis worksheet the number of layers present in extraction tube.

1.4.1.10.4 If upon addition of the solvent, one-layer results, transfer 15µL of liquid of original liquid sample to a quart size friction lid can with a

taped sampling hole or other clean, airtight container that allows for headspace sampling.

- 1.4.1.10.5 If upon addition of the solvent two layers result, remove a representative amount of the liquid, not to exceed approximately one-third of the original liquid sample, and place into a quart size friction lid can with a taped sampling hole or other clean, airtight container that allows for headspace sampling.

1.4.1.11 **Headspace Sampling**

1.4.1.11.1 If a significant odor of a petroleum product exists, first collect headspace sample at room temperature. The headspace may be resampled after heating.

1.4.1.11.2 If a significant odor is not present or if room temperature sampling was inadequate, place friction lid can into a 40° to 70°C closely monitored heating device for 10 to 30 minutes.

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

1.4.1.11.4 After inserting the gas tight syringe through the sampling hole in the friction lid can, slowly pump the syringe several times and inject 10 to 500µL into GC injection port. Immediately re-tape sampling hole.

1.4.1.11.5 Analyze by GC-MSD as described in section 1.5.

1.4.1.12 **Headspace Sampling of Reference Standard**

1.4.1.12.1 Place 15µL ignitable liquid reference standard into to a quart size friction lid can with a taped sampling hole or other clean, airtight container that allows for headspace sampling.

1.4.1.12.2 Process with headspace sampling.

1.4 RECOVERY METHODS

1.4.2 Separation of Ignitable Liquid Residues from Fire Evidence Samples by Passive Headspace Concentration with Activated Charcoal

1.4.2.1 **General**

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

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

1.4.2.1.3 This method recovers ignitable liquids for subsequent instrumental analysis by a gas chromatograph equipped with a mass selective detector (GC-MSD).

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

1.4.2.2 **ASTM Reference Method**

This separation protocol is derived from ASTM standard practice E 1412-07: *Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Activated Charcoal*.

1.4.2.3 **Application**

This procedure is useful for recovering light to medium range (>C6 → ≅C14) ignitable liquids. This method will recover heavier range distillates (>C14) with the limitation indicated in section 1.4.2.5.2.

1.4.2.4 **Sensitivity**

This separation protocol is capable of isolating quantities less than 0.1µL of an ignitable liquid residue from a sample.^{1,82}

1.4.2.5 **Limitations**

1.4.2.5.1 As of edition E 1618-01 and E 1387-01, changes were implemented which places class IV (kerosene) and V products together as Heavy Petroleum Distillates. For this reason, under the current 1618-06^{e1} the designation of Heavy Petroleum Distillates/Heavy Product Range does not require the completion of a solvent extraction.

1.4.2.5.2 Compounds longer than hexadecane (C16) may not be effectively volatilized for adsorption by ACS when using an oven temperature below 60°C.

1.4.2.5.3 This method may not fully differentiate a kerosene class product (formerly E 1618-97/E 1387-95 class IV) from a heavy petroleum product (formerly E 1618-97/E 1387-95 class V). When full differentiation is an issue the following two options, however, are available.

1.4.2.5.3.1 Option One

Perform a solvent extraction as described in section 1.4.3.

1.4.2.5.3.2 Option Two

Resample at a higher oven temperature (70° - 90°C). Resampling is discussed in section 1.4.2.12.

1.4.2.5.4 Samples containing high levels of light to medium range ignitable liquids are prone to displacement and thus loss of light petroleum product components. A shorter adsorption time and a larger ACS should be used for samples suspected of containing larger concentrations of light to medium range petroleum products based upon the presence of a significant petroleum product odor and/or available information.

1.4.2.6 **Equipment and Supplies**

1.4.2.6.1 Chemical fume hood

1.4.2.6.2 Individually packaged activated charcoal polymer strips (ACS) (Albrayco Laboratories ACS-150-6PACK or equivalent). Each strip is approximately 8 x 20mm for a total area of 160mm². ASTM E1412-07 recommends a minimum strip size of 100mm².

1.4.2.6.3 Metal, non-coated, paper clips

1.4.2.6.4 Magnet capable of securing paper clip to lid of friction lid can

1.4.2.6.5 Large laboratory oven for uniform heating of friction lid cans and Kapak-type pouches at 50-90°C.

1.4.2.6.6 2-mL wide-mouth automatic liquid sampler (ALS) vials preassembled with screw-top cap with PTFE/silicone septa

1.4.2.6.7 Glass vial microinserts

1.4.2.6.8 Polymer evidence bags/sheet

1.4.2.6.9 Heat sealer

1.4.2.7 **Reagents**

1.4.2.7.1 Carbon disulfide (CS₂) – Low Benzene

Solvent must meet American Chemical Society specifications.

1.4.2.8 **Reference Material**

1.4.2.8.1 Reference material can be run diluted or after recovery with this recovery technique as described in section 1.4.2.14.

1.4.2.8.2 Refer to section 1.5.4 for ignitable liquid test mixture and reference standards requirements and appropriate dilutions.

1.4.2.9 **Safety Concerns**

1.4.2.9.1 Carbon disulfide is an extremely flammable, volatile liquid. All routes of exposure to carbon disulfide have adverse affects 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.

1.4.2.9.2 For further information regarding the hazards of exposure to carbon disulfide refer to material safety data sheets (MSDS).

1.4.2.9.3 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).^{1.8.13}

1.4.2.10 **Passive Adsorption Procedure**

1.4.2.10.1 Secure two appropriately sized activated charcoal strips (ACS) in paper clips. One of these ACS will be used for analysis, the second for retention purposes.

1.4.2.10.1.1 Note lot number of ACS on analysis worksheet.

1.4.2.10.2 Size of ACS used depends upon the presence, and intensity of, a characteristic ignitable liquid odor and the sample size.

1.4.2.10.3 Open friction lid can or evidence pouch and quickly note any obvious odor present.

1.4.2.10.4 Suspend ACS over the sample. Secure paperclip by placing a magnet on the lid of the can or on the outside of the pouch material.

1.4.2.10.5 If a strong odor of an ignitable liquid exists, allow strip to collect sample at room temperature for 2-16 hours or place into a 50-60°C, **closely monitored**, oven for 1-2 hours.

1.4.2.10.7 If an obvious ignitable liquid odor is not present, place friction lid can into a 50-60°C laboratory oven for 16 to 24 hours.

1.4.2.10.8 As described in E 1412-07, important considerations for the optimum adsorption time for representative sampling and/or maximum sensitivity are the size of charcoal strip, the adsorption temperature and the ignitable liquid composition and concentration.

1.4.2.10.9 At the completion of the adsorption period, remove can from oven and allow to cool for approximately 30 minutes.

1.4.2.10.10 Label ALS vials with identifying information for both analysis and retention ACS.

1.4.2.10.11 Transfer each ACS from can directly into a labeled 2mL-wide-mouth automated sampler (ALS) vial and cap immediately.

1.4.2.11 **Elution Procedure - Analysis ACS**

1.4.2.11.1 To elute compounds trapped upon the charcoal, add 50 to 1000µL carbon disulfide (CS₂) to each vial. Immediately seal vial.

1.4.2.11.2 To facilitate extraction, place vial on its side.

- 1.4.2.11.3 Document the source, lot and volume of CS₂ used on analysis worksheet.
- 1.4.2.11.4 A portion of the carbon disulfide may be transferred into a labeled autosampler vial with microinsert. The vial containing the charcoal strip may be stored in the flammables refrigerator.
- 1.4.2.11.5 If initial examination indicates a significant odor of an ignitable liquid in the sample, the CS₂ extract may be diluted prior to GC-MSD analysis.
- 1.4.2.12 **Resampling of Item**
- 1.4.2.12.1 If initial GC-MSD data indicates that the strip was clearly overloaded, the sample can be resampled at ambient temperature or with a shortened adsorption time.
- 1.4.2.12.2 If initial examination of GC-MSD data indicates a kerosene or diesel fuel product, the sample may be resampled at 80 - 90°C for 16 to 24 hours.
- 1.4.2.13 **Packaging of Retention ACS**
- 1.4.2.13.1 Place vial containing retention ACS into polymer pouch material and heat seal.
- 1.4.2.13.2 Place heat-sealed pouch into corresponding evidence container.
- 1.4.2.14 **ACS Recovery of Reference Standard**
- 1.4.2.14.1 Place 1 to 10µL ignitable liquid reference standard into a clean, unused quart sized friction lid can, and seal.
- 1.4.2.14.2 Secure ACS in paper clip.
- 1.4.2.14.3 Use a magnet to secure prepared collection device onto lid of friction lid can and immediately seal lid.
- 1.4.2.14.4 Place friction lid can into a 50°C oven for 1 hour.
- 1.4.2.14.5 Process charcoal strip as with case samples.
- 1.4.2.15 **Quality Assurance**
- 1.4.2.15.1 Charcoal Strip Blanks
- 1.4.2.15.1.1 A charcoal strip blank must be run with each batch of cans placed into an oven. A blank ACS should be prepared for each oven used.
- 1.4.2.15.1.2 Prepare ACS as described for case samples.

1.4.2.15.1.3 Place prepared collection device into a clean, empty quart sized friction lid can.

1.4.2.15.1.4 Process charcoal strip as with case samples.

1.4.2.15.1.5 GC-MSD data for the charcoal strip blank must be either placed into casefile and/or centrally stored in the laboratory performing the analysis.

1.4.2.15.2 New ACS Lot

1.4.2.15.2.1 Each new lot of ACS should be checked for contamination and relative recovery prior to official use.

1.4.2.15.2.2 Contamination should be checked for by preparing a blank as described in sections 1.4.2.10 and 1.4.2.11.

1.4.2.15.2.3 Relative recovery should be verified with 5 μ L of 50% evaporated gasoline as described in sections 1.4.2.10 and 1.4.2.11.

1.4.2.15.2.4 GC-MSD data must be centrally stored in the laboratory performing the analysis.

1.4.2.15.3 New Elution Solvent Bottle

1.4.2.15.3.1 Relative contamination must be checked for in each new bottle of CS₂, regardless of lot.

1.4.2.15.3.2 For this check, evaporate a volume of CS₂ by 50%.

1.4.2.15.3.3 If present, the relative toluene concentration must be noted. Solvent should be free of other identifiable ignitable liquids.

1.4.2.15.3.4 GC-MSD data must be centrally stored in the laboratory performing the analysis.

1.4.2.16 **Analysis**

1.4.2.16.1 Analyze extracts by GC-MSD as outlined in section 1.5 of this method.

1.4 RECOVERY METHODS

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

1.4.3.1 **General**

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

1.4.3.1.2 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.

1.4.3.2 **ASTM Reference Method**

This separation protocol is derived from ASTM standard practice E 1386-00 (2005): *Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction*.

1.4.3.3 **Application**

1.4.3.3.1 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.

1.4.3.3.1.1 Although the updated classification system of E 1618-01 and E 1387-01 has class IV and V products classified together as Heavy Petroleum Distillates, a solvent extraction is required in order to fully differentiate a kerosene class product (formerly E 1618-97/E 1387-95 class IV) from a heavy petroleum product (formerly E 1618-97/ E 1387-95 class V).

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

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

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

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

1.4.3.3.6 Liquid samples suspected to contain an ignitable liquid.

1.4.3.4 **Sensitivity**

1.4.3.4.1 This separation protocol is capable of isolating quantities smaller than 1 μ L of an ignitable liquid residue from a sample (ASTM E1386-00 (2005), 4.2).

1.4.3.5 **Limitations**

- 1.4.3.5.1 Interfering compounds, inherent in the fire debris recovered by this method, may create GC-MSD data interpretation problems. These compounds are the result of pyrolysis of solid fuels present at the fire scene and the combustion of organic compounds liberated during the fire process.
- 1.4.3.5.2 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.
- 1.4.3.5.3 Lighter compounds may not be detected by this technique due to the requirement for a solvent delay during GC-MSD analysis. Any compound with a retention time \leq the solvent delay will not be detected by this technique.
- 1.4.3.5.4 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.

1.4.3.6 **Safety Concerns**

- 1.4.3.6.1 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.
- 1.4.3.6.2 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.
- 1.4.3.6.3 For further information regarding the hazards of exposure to extraction solvents refer to material safety data sheets (MSDS).
- 1.4.3.6.4 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.

1.4.3.7 **Equipment and Supplies**

- 1.4.3.7.1 Chemical fume hood
- 1.4.3.7.2 Tube rocker.

- 1.4.3.7.3 Filter Paper
- 1.4.3.7.3.1 Whatman Grade 1 - Qualitative (27cm) or equivalent
 - 1.4.3.7.3.2 Whatman IPS -Phase separation (15cm) or equivalent
- 1.4.3.7.4 Glassware
- 1.4.3.7.4.1 Assorted sizes of glass funnels
 - 1.4.3.7.4.2 Assorted 25mL to 2000mL glass beakers
 - 1.4.3.7.4.3 Assorted sizes of screw-top extraction tubes and screw caps (no adhesive liners).
 - 1.4.3.7.4.4 2-mL wide-mouth automatic liquid sampler (ALS) vials preassembled with screw-top cap with PTFE/silicone septa
 - 1.4.3.7.4.5 Glass vial microinserts
- 1.4.3.8 **Reagents**
- 1.4.3.8.1 Pentane (Pesticide Grade or equivalent)
 - 1.4.3.8.2 Carbon disulfide (ACS/Low Benzene)
- 1.4.3.9 **Solvent Extraction - Non-liquid Samples**
- 1.4.3.9.1 Transfer a representative portion of sample to an appropriately sized glass beaker, Kapak[®] -type pouch or friction lid can.
 - 1.4.3.9.2 Add sufficient solvent to thoroughly moisten sample. Suitable solvents are listed in 1.4.3.8. Carbon disulfide should only be used for small samples such as wicks.
 - 1.4.3.9.3 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.
 - 1.4.3.9.3.1 Note type, lot and source of extraction solvent on fire evidence worksheet.
 - 1.4.3.9.4 If necessary, filter solvent through phase separation paper supported with Grade 1 filter paper.
 - 1.4.3.9.5 Place \cong 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.
 - 1.4.3.9.6 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.
 - 1.4.3.9.7 Continue to evaporate remaining solvent. Concentrate extract down to approximately 1mL. Place the sample into labeled ALS vial.

1.4.3.10 **Solvent Extraction - Unknown Liquid Samples**

- 1.4.3.10.1 Examine liquid and note the number of layers present.
- 1.4.3.10.2 If more than one layer is present, transfer 2 μ L of top layer of liquid to a labeled ALS vial containing 2mL of carbon disulfide and mix.
- 1.4.3.10.3 If only one layer is present, remove a small portion of the liquid and place into an appropriately sized sealable glass extraction tube; add an equal volume of carbon disulfide and mix. Note and record on analysis worksheet the number of layers present in extraction tube.
- 1.4.3.10.4 If upon addition of the solvent, one layer results, transfer 2 μ L of liquid of original liquid sample to a labeled ALS vial containing 2mL of carbon disulfide and mix.
- 1.4.3.10.5 If upon addition of the solvent two layers result, remove a portion of the original liquid sample and place into an appropriately sized sealable glass extraction tube, add an equal volume of carbon disulfide and mix. Ideally at least 50% of the original sample liquid is not sampled.
- 1.4.3.10.5.1 Mix the sample for approximately one to five minutes.
- 1.4.3.10.5.2 If a significant amount of water is present, the solvent may be filtered through phase separation paper supported with Grade 1 filter paper.
- 1.4.3.10.5.3 For case samples possessing a significant odor, a 2mL aliquot of solvent should be sampled prior to evaporation. This sample may be diluted as necessary.
- 1.4.3.10.5.4 If necessary, allow solvent to evaporate approximately 50%. Transfer an \cong 1 to 2mL aliquot of the partially evaporated sample to a labeled ALS vial. Additional aliquots at higher levels of evaporation may be collected at the discretion of the analyst.
- 1.4.3.10.5.5 Continue to evaporate remaining solvent. Concentrate extract down to approximately 1mL. Place the sample into labeled ALS vial.
- 1.4.3.10.6 For all extraction situations, note lot and source of extraction solvent on fire evidence worksheet.

1.4.3.11 Solvent Purity Check

1.4.3.11.1 When solvent used for extraction is subjected to evaporation, the batch of samples should include a solvent blank.

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

1.4.3.11.1.2 Evaporate solvent to approximately 1mL.

1.4.3.11.1.3 The degree of solvent evaporation should be at least twice the extent used for questioned samples (ASTM E 1386-00 (2005), 5.2.1).

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

1.4.3.12 Analysis

1.4.3.12.1 Place each solvent extract into labeled ALS vial with microinsert, and cap.

1.4.3.12.2 Analyze extracts by GC-MSD as described in section 1.5. If a partially evaporated extract provides sufficient sensitivity, the more evaporated extract(s) need not be analyzed.

1.5 ANALYSIS OF RECOVERED EXTRACTS

1.5.1 General

1.5.1.1 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 with a gas chromatograph equipped with a mass selective detector (GC-MSD).

1.5.1.2 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.

1.5.2 ASTM Reference Method

This analysis approach is Derived from ASTM E 1618-06^{e1}: *Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry* and ASTM E 1387-01: *Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography*.

1.5.3 Apparatus

1.5.3.1 **Instrumentation**

Integrated system consisting of a Gas Chromatograph equipped with a Mass Selective Detector. Data system must be capable of handling macro programs to extract ion profiles to assist with the interpretation of data.

1.5.3.2 **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 1.5.4.1).

1.5.4 Chemicals and Reference Standards

1.5.4.1 **Column Resolution Test Mixture**

1.5.4.1.1 The minimum requirements for the test mixture are described in E 1618-06^{e1}, 6.4. Compliant test mixtures can be obtained through Restek (#31224), Cerilliant (ERR-002) or comparable vendor/product.

1.5.4.1.1.1 Place 200µL test mixture into 2mL ALS vial. Bring up to volume with carbon disulfide.

1.5.4.1.2 The Resolution Test Mixture should be compared to the previous run for retention time shifts and changes in abundances that may indicate instrumental problems and/or deterioration of the test mixture.

1.5.4.1.3 All constituents of this test mixture with the exception of hexane must be present.

1.5.4.2 **Headspace Test Mixture**

1.5.4.2.1 When headspace sampling is employed, in addition to the E1618 column resolution test mixture described in 1.5.4.1, a mixture of light range ignitable compounds must be included in each analysis run.

1.5.4.2.2 The mixture will contain Acetone, 2-Butanone, 2-Pentanone and 2-Hexanone.

1.5.4.2.3 Additional light range test mixtures may be analyzed at the discretion of the analyst.

1.5.4.3 **Ignitable Liquid Reference Material**

Ignitable liquids for use as reference material may be obtained as required from commercial and local sources. A reference standard 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.

1.5.4.3.1 Dilution of Reference Material

1.5.4.3.1.1 **Neat Ignitable Liquid Products**

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

1.5.4.3.1.2 **Diluted Commercial Products**

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

1.5.4.3.2 Authentication of Ignitable Liquid Reference Material

1.5.4.3.2.1 The authentication of ignitable liquid standards 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.

1.5.4.3.2.2 Authentication of standards is based upon interpretation of the TIC, EIP and mass spectral data obtained through the analysis of the reference standard.

1.5.4.3.2.3 When available, the data should be compared with the manufacture package insert/information packet. A MSDS alone is never be used as a sole basis for authentication.

1.5.4.3.2.4 When possible, newly obtained standards from local sources are to be compared to authenticated commercially available standards prior to official use.

1.5.4.3.2.5 Authentication documentation must be stored centrally.

1.5.4.3.3 Application of Ignitable Liquid Reference Material

1.5.4.3.3.1 Reference material need not be analyzed with each analysis run if the column resolution mixture (CRM) for the date of casework analysis agrees with the CRM for the date that the reference material was run on the GC-MSD.

1.5.4.3.3.2 The CRMs should show no significant shifts in retention time.

1.5.4.3.3.4 The CRM for the reference material run date must be included in the casefile.

1.5.5 GC/MSD Analysis

1.5.5.1 **Instrumental Parameters**

1.5.5.1.1 Refer to method printouts for current parameters.

1.5.5.1.2 Current and past method printouts must be centrally stored in the laboratory performing the analysis.

1.5.5.2 **MSD Tuning**

1.5.5.2.1 An *Autotune* and *System Verification* must be performed prior to initiating a new sequence run. Ideally each sequence should be bracketed by an AUTOTUNE.

1.5.5.2.2 All parameters evaluated for the *Autotune* and *System Verification* must fall within manufacturer recommended ranges.

1.5.5.2.3 *Autotunes* and *System Verifications* must be centrally stored in the laboratory performing the analysis.

1.5.5.3 **GC-MSD Quality Assurance Sample Requirements**

1.5.5.3.1 In-between Sample Blanks

1.5.5.3.1.1 Analyzing a solvent blank between case samples will indicate if any carry over is occurring.

1.5.5.3.1.2 For headspace sampling the corresponding amount of headspace may be injected.

1.5.5.3.1.3 For ACS recovery, a CS₂ solvent blank must be analyzed.

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

1.5.5.3.1.5 TIC from BLANK must be placed into case file. If any peaks are present, the MS for peaks of interest must also be placed in the case file.

1.5.5.3.2 Performance Verification Test Mixture

1.5.5.3.2.1 To establish that the system has the capability to resolve compounds as outlined in ASTM E 1618-06^{e1}/E 1387-01, a column resolution check mix should be analyzed at the beginning and the end of the analysis sequence.

1.5.5.3.2.2 A minimum of one ASTM test mix must be run with each sequence.

1.5.5.3.2.3 When headspace sampling is used, a mixture of commonly encountered light compounds must be included in the analysis run to verify detection ability.

1.5.5.3.2.4 Data for test mixtures must be centrally stored in the laboratory performing the analysis. A copy of the test mix data may also be placed into a casefile.

1.5.5.3.3 Helium Gas Purity

To prevent contamination from carrier gas, ultrapure ($\geq 99.995\%$) helium shall be used.

1.5.5.4 **GC-MSD Sequence**

1.5.5.4.1 Load SEQUENCE from sequence menu.

1.5.5.4.2 Select *Edit Sample Log Table* from sequence pull-down. The following is an example of the sample log table.

Sample Log Table for FIRE.M					
Line	Type	Vial	Date File	Method	Sample Name
1)	Sample	51	BLK-ACS	FIRE	QC ACS 02-01-10 ACS Lot No: 950674
2)	Blank	75	006-1-BLK	FIRE	Carbon Disulfide Blank Fisher Lot No: 950674
3)	Sample	53	006-1-ACS	FIRE	P20101234-1 ACS Lot No: 062200
4)	Blank	75	006-2-BLK	FIRE	P20101234-2 Pentane Blank Fisher Lot No: 4567
5)	Sample	54	006-2-SE	FIRE	P20101234-2 SE Pentane Lot No: 4567
6)	Sample	74	50EVGAS	FIRE	50% evap Sinclair gasoline Lot No:0500-1
7)	Sample	71	ASTM1618	FIRE	ASTM E1618 Column Resolution Mix Lot 2398

1.5.5.4.3 Source and lot number may be placed under sample log table *Miscellaneous Information*.

1.5.5.4.4 Case Samples

In Sample Log Table, program case samples into sequence by laboratory and sample number. Sample name description should provide an indication of method of recovery.

1.5.5.4.5 Quality Assurance Samples

In Sample Log Table, program quality control samples. Information must include lot number when applicable.

1.5.5.4.6 Ignitable Liquid Reference Standards

1.5.5.4.6.1 In Sample Log Table, program appropriate ignitable liquid standards.

1.5.5.4.6.2 The source and lot numbers for commercially obtained products must be included. The source of locally obtained fuels (gasoline, diesel fuel, etc.) must be

described. When available, the data of purchase (DOP) should be included.

1.5.6 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 types compounds found in ignitable liquids. The following MACROS are available to provide for options for data presentation to assist with interpretation. TICs and EIPs from case samples are compared against TICs and EIPs prepared for reference standards. EIPs may be used to assist with the interpretation of the TIC but must not be the sole basis for the conclusions reached.

1.5.6.1 **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.^{1.8.13}

1.5.6.1.1 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 as follows:

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

1.5.6.2 **Command Line Selected Ion Profiles Macros**

1.5.6.2.1 ARION2

1.5.6.2.1.1

The *ARION2* macro provides a custom header on the TIC and one page with combined selected ion profiles for ions characteristic for key ignitable liquid groups as indicated below.

Major Compound Types	Major Ion Fragments			
Alkanes	43	57	71	85
Aromatics	91	105	119	120
Cycloalkane/alkene	55	69	82	83
Naphthalenes	128	142	156	170

1.5.6.2.1.2

On command line type:

MACRO "ARION2", GO

1.5.6.2.2 INDANES

1.5.6.2.2.1

The *INDANES* macro provides a one page with combined selected ion profiles for ions characteristic for indane group as indicated below.

Major Compound Types	Major Ion Fragments			
Indanes	117	118	131	132

1.5.6.2.2.2 On command line type:
MACRO "INDANES", GO

1.5.6.2.3 CUSTOM

1.5.6.2.3.1 This macro provides a custom header on each printout. Operator selects either a full-page version of the selected ion profiles for alkanes, aromatics, cycloalkane/alkene, indanes and naphthalenes. Macro also allows for defining of window size (X-axis) and abundance (Y-axis). Adjustments made allow for enhancement of low abundance compounds.

1.4.6.2.3.2 On command line type:
MACRO "CUSTOM", GO

1.5.6.2.4 TIC

1.5.6.2.4.1 This macro provides TIC printout with a custom header. Macro allows for defining of window size (X-axis) and abundance (Y-axis). Adjustments made allow for enhancement of low abundance compounds.

1.5.6.2.4.2 On command line type:
MACRO "TIC", GO

1.5.7 Instrument Maintenance

1.5.7.1 Replacement parts and cleaning supplies required for GC-MSD maintenance should be stocked to reduce the time that an instrument is off-line. Refer to manufacturer's hardcopy or electronic instrument manuals and/or hardcopy or on-line catalog for ordering information.

1.5.7.2 Refer to manufacturer's hardcopy or electronic instrument manuals for maintenance indicators and instructions.

1.6 INTERPRETATION OF ANALYTICAL DATA

1.6.1 E 1618-06^{el} Classification system

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

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

1.6.1.2.1 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.

1.6.1.2.2 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.

Table 1: E 1618-06^{el} Ignitable Liquid Classification Scheme ^{1.8.4}

Class	Light {C ₄ - C ₉ }	Medium {C ₈ - C ₁₃ }	Heavy {C ₈ - C ₂₀₊ }
Gasoline All brands, including gasohol	Fresh gasoline is typically in the C ₄ -C ₁₂ range		
Petroleum Distillates	Petroleum Ether Some Cigarette Lighter Fluids Some Camping Fuels	Some Charcoal Starters 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 & 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
N-Alkanes Products	Solvents Pentane Hexane Heptane	Some Candle Oils Some Copier Toners	Some Candle Oils Some 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	
Other-Miscellaneous	Single Component Products Some Blended Products Some Enamel Reducers	Turpentine Products Some Blended Products Various Specialty Products	Some Blended Products Various Specialty Products

1.6.2 Identification of an Ignitable Liquid

- 1.6.2.1 Compare data from case sample TIC and EIPs with data from reference material obtained with the same GC-MSD parameters.
- 1.6.2.2 The characteristics which establish the presence of an ignitable liquid is set forth in Section 10 of ASTM E 1618-06^{e1}: *Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry*.
- 1.6.2.3 The characteristics are to be used as the basis for the identification of an ignitable by this analytical method.
- 1.6.2.4 The analyst must consider and be prepared to explain any observed differences between the unknown (questioned sample) and a known reference material.
 - 1.6.2.4.1 Variables that should be taken into account include the influence of evaporation, interference from burned matrix materials, sample preparation techniques and/or bacterial degradation (soil samples).
 - 1.6.2.4.2 Unexplained differences should preclude the identification as an ignitable liquid.
- 1.6.2.5 A copy of the relevant reference material supporting the conclusions reached must be placed into the casefile.

1.7 ANALYSIS REPORT

1.7.1 Fire Evidence Coversheet

1.7.1.1 Description of Evidence

1.7.1.1.1 Using information recorded on fire evidence worksheet, the description on the analysis coversheet must include the item number, the agency exhibit number (when available), condition of seals, the type of container and details of the type of evidence.

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

1.7.1.2 Analysis Results

1.7.1.2.1 Positive Results

1.7.1.2.1.1 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 in must be indicated for all classes except gasoline.

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

1.7.1.2.1.3 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.

1.7.1.2.2 Negative Results

1.7.1.2.2.1 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.

1.7.1.2.2.2 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.

1.7.1.2.3 Turpenes^{1.8.14}

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

1.7.1.2.3.1 When a comparison control sample is available and clearly shows the same distribution of turpene constituents, a questioned sample containing wood may be reported out as negative.

1.7.1.2.3.2 When in the absence of a comparison control sample, significant levels of turpenes are noted in a sample containing wood, a qualifier should be used. The qualifier should read to the effect that the sample contains turpenes, which may have originated from coniferous wood in the sample or from a medium range turpentine product.

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

1.7.1.2.3.4 When the sample clearly does not contain wood which would contribute to the presence of turpenes, a qualifying statement is not necessary. The sample

should be reported as containing a medium range turpentine product.

1.7.1.2.4 Questionable Results

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

1.7.1.2.4.2 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 1.7.1.2.4.1 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 control sample for the substrate it is unknown whether the ignitable liquid originated from (endogenous to) the substrate itself.

1.8 REFERENCES AND RECOMMENDED READING

- 1.8.1 ASTM Method E 1388-05, Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples.
- 1.8.2 ASTM Method E 1412-07, Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal.
- 1.8.3 ASTM Method E 1386-00 (2005), Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction.
- 1.8.4 ASTM Method E 1618-06^{e1}, Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.
- 1.8.5 ASTM Method E 1387-01, Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography.
- 1.8.6 Dietz, W.R. Improved Charcoal Packaging for Accelerant Recovery by Passive Diffusion. J. Forensic Sci. 36(1):111-21; 1991.

- 1.8.7 Newman, R.T.; Dietz, W.R.; Lothridge, K. 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. *J. Forensic Sci.* 41(3):361-370; 1996.
- 1.8.8 Arson Accelerant Detection Course Materials, presented at Alcohol, Tobacco & Firearms Laboratory, Rockville, Maryland, May, 1993.
- 1.8.9 Arson Analysis Workshop Materials, presented at Northwest Association of Forensic Scientist's Fall Meeting. Salt Lake City, Utah, October, 1996.
- 1.8.10 Advanced Fire Debris Course Materials, presented at National Forensic Science Technology Center, St. Petersburg, Florida, December, 1996.
- 1.8.11 Newman, R.; Gilbert, M.; Lothridge, K. GC-MS Guide to Ignitable Liquids. Boca Raton, FL: CRC Press: 1998.
- 1.8.12 Stauffer, E., *Concept of pyrolysis for fire debris analysts*, *Sci & Justice*, 43(1):29-40, 2003.
- 1.8.13 Laboratory Fire Standards and Protocols Committee Scientific Working Group for Fire and Explosions, Initial Assessment of Evidence, page 3, 8.1.5.
- 1.8.14 Trimpe, M.A., *Turpentine in Arson Analysis*, *J. Forensic Sci.*, 36(4):1059-1073, 1991.

Refer to fire evidence training plan for additional references.



Section I

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

Manual History

<u>Revision</u>	<u>Issue Date</u>	<u>History</u>	<u>Author</u>
0	01-1997	Original Issue	S.C. Williamson
1	01-07-1999	ASTM update.	S.C. Williamson
2	01-19-2001	Reformat, updates, refinements, Addition of magnet option to protocol.	S.C. Williamson
3	01-11-2002	Addition of safety section, reformatted GC/MSD maintenance schedule and method refinement	S.C. Williamson
4	11-05-2003	Update to most recent ASTM Standards	S.C. Williamson
5	02-22-2006	Quality assurance sections updated, headspace sampling section reinstated, addition of ACS retention requirement	S.C. Williamson
6	04-19-2006	Headspace test mix modified clarifications in authentication process.	S. C. Williamson
7	09-22-2010	Updated ASTM References and made associated changes, reviewed and clarified wording. (02/2010)	S. C. Williamson R. G. McDowell