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Idaho State Police
Forensic Services
Pocatello Laboratory

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Section I

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

1.0 Introduction

The analysis of evidence from fires of suspicious origin encompasses a variety of sample types. Evidence collected ranges from burnt fire debris and liquids recovered at the fire scene to the suspect's clothing. The examination of the evidence may involve only an identification of ignitable liquid/residue present or it may involve the comparison of a liquid recovered at the scene with a liquid recovered from the suspect. The approach taken to the analysis of fire scene evidence is, for these reasons, broad and general in nature.

2.0 Recovery Methods

2.1 Separation and Concentration of Ignitable Liquid Residues from Fire Evidence Samples by Passive Headspace Concentration

2.1.1 General

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

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

2.1.1.3 This method will recover the entire range of ignitable (flammable or combustible) liquids but may require solvent extraction to differentiate between a class IV and a class V ignitable liquid product (refer to 2.5.1).

2.1.1.4 This method is time efficient and does not consume or alter the sample.

2.1.2 ASTM Reference Method

Refer to ASTM standard practice E 1412-95, *Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration*.

2.1.3 Application

2.1.3.1 This procedure is useful for samples which may contain a light to medium range (C4-C14) petroleum distillate products as suggested by an indicative odor. This method will recover heavy range distillates but with the limitation discussed in section 2.5.1.

2.1.4 Sensitivity

2.1.4.1 Capable of isolating quantities smaller than $10\mu\text{L}$ of an ignitable liquid residue from a sample.

2.1.5 Limitations

2.1.5.1 A solvent extraction is required in order to differentiate a kerosene class product (class IV) from a heavy petroleum product (class V).

2.1.5.2 Samples that contain 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 which, based upon olfactory screening, are suspected of containing larger concentrations of light to medium range petroleum products.^{6,9}

2.1.6 Materials and Equipment

2.1.6.1 Solvents

2.1.6.1.1 ACS Carbon disulfide

Purity of solvents is assured by analyzing blanks on each batch of solvent by GC-MSD.

2.1.6.2 Collection device

2.1.6.2.1 Activated charcoal polymer strips (ACS)

Size used depends upon the presence, and intensity of, a characteristic ignitable liquid odor and the sample size. Approximate size 8 X10mm to 8 X 15mm.

2.1.6.2.2 Paper clips with serrated edges.

2.1.6.2.3 Securing of the ACS

Option One: Non-waxed dental floss.

Option Two: Magnet capable of securing paper clip to lid of friction lid can.

2.1.6.3 Heating system

Large oven which allows for uniform heating of friction lid cans at 50-60°C.

2.1.6.4 2mL widemouth autosampler vials, microinsert and closures.

2.1.7 Passive Adsorption Procedure

2.1.7.1 To suspend the ACS strip over the sample, either (1) place a magnet on the lid of the can or (2) tie a length of non-waxed dental floss onto a paper clip. The length of dental floss should be proportional to the size of can that the clip will be suspended into.

2.1.7.2 Secure appropriately sized charcoal strip in paper clip.

2.1.7.3 Open friction lid can or evidence pouch (Kapak)[®] and suspend charcoal strip above sample. Reseal the container.

2.1.7.4 If strong odor of petroleum product exists, allow strip to collect sample at room temperature for 4-16 hours or place into a 50-60°C, closely monitored, oven for 1-2 hours. If an odor is not present, place friction lid can into a 50-60°C for 16 to 48 hours.

2.1.7.5 At the completion of the adsorption period, remove can from oven and allow to cool for approximately 30 minutes. Transfer charcoal collection device directly from can into a labeled 2mL-widemouth autosampler vial.

2.1.8 Elution Procedure

2.1.8.1 To elute the compounds trapped upon the charcoal, add 200µL carbon disulfide (CS₂) to each vial. Seal vial and vortex. Place vial on its side to facilitate extraction. Vial may also be placed on rocker or rotator.

2.1.8.2 Transfer a portion of the carbon disulfide into labeled autosampler vial with microinsert, and seal. Place the vial containing the charcoal strip and the remaining extract into the refrigerator.

2.1.8.3 If the initial olfactory screening indicates a strong presence of an ignitable liquid in a sample, dilute extract with CS₂ prior to GC-MSD analysis.

2.1.9 Comparison Standards

2.1.9.1 Collection of petroleum product comparison standards with the charcoal strip device is periodically done to:

2.1.9.1.1 Check the adsorption efficiency of the charcoal.

2.1.9.1.2 Provides for a comparison standard collected in the same fashion as the case samples.

- 2.1.9.1.3 Preparation of charcoal strip comparison standards is as follows:
- 2.1.9.1.3.1 Place 1 μ L standard into a clean, unused quart sized friction lid can, and seal.
 - 2.1.9.1.3.2 Prepare charcoal strip standard as described for collection device above.
 - 2.1.9.1.3.3 Place prepared collection device into prepared friction lid can.
 - 2.1.9.1.3.4 Process charcoal strip as with case samples.

2.1.10 Charcoal Strip Blanks

- 2.1.10.1 A charcoal strip blank will be run with each batch of cans placed into the oven.
- 2.1.10.1.1 Prepare charcoal strip blank as described for collection device above.
 - 2.1.10.1.2 Place prepared collection device into a clean, empty quart sized friction lid can.
 - 2.1.10.1.3 Process charcoal strip as with case samples.

2.1.11 Analysis

- 2.1.11.1 Analyze extracts by GC-MSD as outlined in section 3.0 of this method.

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

2.2.1 General

- 2.2.1.1 This method serves to recover ignitable liquid residues from samples submitted as evidence in fire investigations.
- 2.2.1.2 This method applies the principle "Likes dissolves like". Any petroleum product in the sample is recovered with a short chain aliphatic hydrocarbon.
- 2.2.1.3 This method will recover the entire range of ignitable liquids.
- 2.2.1.4 This method will recover ignitable liquids over the entire range of concentrations.

2.2.2. ASTM Reference Method

Refer to ASTM standard practice E 1386-90, *Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction*.

2.2.3 Application

- 2.2.3.1 Samples which may contain a kerosene to heavy petroleum distillate product as indicated by:
 - 2.2.3.1.1 Strong indicative odor.
 - 2.2.3.1.2 GC/MSD data from analysis of ACS. A solvent extraction is required in order to differentiate a kerosene class product (class IV) from a heavy petroleum product (class V).
- 2.2.3.2 Samples which contain a high percentage of charred debris.
- 2.2.3.3 Samples which contain non-porous surfaces such as glass, or burned containers.

2.2.4 Sensitivity

- 2.2.4.1 This separation protocol is capable of isolating quantities smaller than 1 μ L of an ignitable liquid residue from a sample (ASTM E1386-95, 4.2)..

2.2.5 Limitations

- 2.2.5.1 The extracting solvent also recovering interfering compounds inherent in the fire debris hampers this method. 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.
- 2.2.5.2 The evaporation step in this method may lead to the loss of light petroleum product components.

2.2.6 Materials

- 2.2.6.1 Solvents
 - 2.2.6.1.1 Nanograde (or better) pentane or hexane and ACS Carbon disulfide.
 - 2.2.6.1.2 Purity of solvents is assured by analyzing blanks on each batch of solvent by GC-FID or GC-MSD
- 2.2.6.2 Filter Paper
 - 2.2.6.2.1 Whatman Grade 1 - Qualitative (27cm) or equivalent
 - 2.2.6.2.2 Whatman 1PS -Phase separation (15cm) or equivalent
- 2.2.6.3 Glassware
 - 2.2.6.3.1 Glass funnels
 - 2.2.6.3.2 25mL to 600mL beakers
 - 2.2.6.3.3 2 mL autosampler vials, inserts and seals

2.2.7 Solvent Extraction Procedure

- 2.2.7.1 Friction lid cans
Cover sample with solvent and place lid on loosely. Sample should be thoroughly moistened.
- 2.2.7.2 Kapak[®] -type pouches
Place pouch in 2-L beaker. Add sufficient solvent to thoroughly moisten sample.
- 2.2.7.3 Mix the sample and debris for approximately 5 minutes. A beaker may be used as a plunger to facilitate the extraction of flexible debris
- 2.2.7.4 Filter sample through phase separation paper supported with Whatman Grade 1 filter paper.
- 2.2.7.5 Allow sample to evaporate approximately 50%. Collect a 2mL sample. Place a portion of the partially evaporated sample into labeled autosampler vial with microinsert, and seal. Place the remaining partially evaporated sample into an additional autosampler vial for refrigerated storage.
- 2.2.7.6 Continue to evaporate remaining solvent. Concentrate extract down to ½ to 1mL. Place a portion of the sample into labeled autosampler vial with microinsert, and seal. Place the remaining sample into an additional autosampler vial for refrigerated storage.

2.2.8 Solvent Purity Check

- 2.2.8.1 A solvent blank will be run with each batch of solvent recovered extracts.

- 2.1.8.1.1 Place approximately 200mL of extraction solvent into a clean beaker.
- 2.1.8.1.2 Evaporate solvent to approximately 1mL.
- 2.1.8.1.3 The degree of solvent evaporation should be at least twice the extent used for questioned samples (ASTM E1386-95, 5.2.1).

2.2.9 Analysis

- 2.2.9.1 Analyze both the partially evaporated and evaporated solvent extracts by GC-MSD as described in section 3.0.

3.0 Analysis by Gas Chromatography-Mass Selective Detector (GC-MSD)

3.1 Instrument

Hewlett Packard 5890 Gas Chromatograph equipped with a Hewlett Packard 5971 Mass Selective Detector.

3.1.1 Column

25 meter Hewlett Packard Ultra 2 (5% PhMeSilicone) with 0.22 mm ID and 0.33 μ m film thickness.

3.1.2 Instrumental Parameters

- 3.1.2.1 Injector Temperature: 280°C
- 3.1.2.2 Detector Temperature: 300°C
- 3.1.2.3 Carrier Gas: Helium
- 3.1.2.4 Temperature Program:
 - Initial Temperature: 40°C
 - Initial Time: 4.00 min
 - Ramp Rate: 8°C/min
 - Final Temperature: 290°C
 - Final Time: 10.00 min
- 3.1.2.5 Sample size: 1.0 - 2.0 μ L

3.2 ASTM Reference Method

Refer to ASTM Method E-1387-95, *Standard Test Method for Ignitable Liquid Residues in Extracts from fire Debris Samples by Gas Chromatography* and E 1618-97, *Standard Guide for Identification of Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry*.

3.3 Preparation for Analysis of samples by GC/MSD

3.3.1 Perform AUTOTUNE.

Refer to maintenance manual for tune evaluation criteria.

3.3.2 Programming of GC/MSD sequence run.

- 3.3.2.1 Load ARSON sequence from sequence menu.

- 3.3.2.2 Select *Edit Sample Log Table* from sequence pull-down.
- 3.3.2.3 In Sample Log Table, program questioned samples into sequence by laboratory and sample number. Information should include method of recovery.
- 3.3.2.4 In Sample Log Table, program quality control samples into sequence by laboratory number or date prepared. Information should include method of recovery.
- 3.3.2.4.1 System Blanks for Charcoal Strips or Solvent Purity Check
Designate BLK-ACS, BLK-SE or similar notation.
- 3.3.2.4.2 In-between Sample Blanks
To ensure that carry-over between samples is not occurring, program a carbon disulfide solvent BLANK between each case sample. TIC from BLANK should be place into case file.
- 3.3.2.4.3 QC Test Mixture
To establish that the system has the capability to resolve compounds outlined in ASTM E1387-95 and ASTM E1618-97, an ASTM E1387-90 column resolution check mix (Restek #31224 or equivalent) is analyzed with each analysis run. Program test mixture into each sequence run. TIC and selected ion profiles (SIP) print-outs should be place in ASTM E1387-90 binder.
- 3.3.2.5 Ignitable Liquid Standards
- 3.3.2.5.1 In Sample Log Table, program appropriate ignitable liquid standards. The source and lot numbers for commercially obtained products should be included. The source of locally obtained fuels (gasoline, diesel fuel, etc) should be described.
- 3.3.2.5.2 A copy of the relevant standard(s) should be placed into the casefile.
- 3.3.3 Post-run Selected Ion Profiles Macros
- 3.3.3.1 Included with the ARSON GC/MSD method is a post-run macro provided by Alcohol, Tobacco and Firearms (ATF). This macro generates the TIC and individually printed selected ion profiles, two per page. Individual profiles allow the analyst to see less abundant ions indicative of certain classes of compounds in greater detail than summed profiles.^{6,13} This macro will initiate upon the completion of sample analysis. Ions included are as follows:

Alkanes	43	57	71	85
Aromatics	91	105	119	133
PNA*s*	128	142	156	

* Polynuclear aromatics (Naphthalenes)

3.3.3 Command Line Selected Ion Profiles Macros

3.3.3.1 Two macros are available which provide for further options for data presentation to assist with interpretation. The macros are accessed under data analysis using the command line.

3.3.3.1.1 *ARION*

On command line type **MACRO "ARION", GO.**

This macro provides a custom header on the TIC and one page with combined selected ion profiles for ions characteristic for key ignitable liquid groups

Alkanes	43	57	71	85
Aromatics	91	105	119	
Cycloalkane/alkene	55	69	82	83
Naphthalenes	128	142	156	

3.3.3.1.2 *CUSTOM*

On command line type **MACRO "CUSTOM", GO.**

This macro provides a custom header on each printout. Operator selects either a TIC or full-page version of the selected ion profiles indicated above for *ARION*. Macro also allows for defining of window size (X-axis) and abundance (Y-axis) which allows for enhancement of low abundance compounds.

4.0 Interpretation of Analytical Results

4.1 Petroleum Product Classification System

4.1.1 Classification system consists of six major classes of ignitable (flammable or combustible liquids).

Class Number (Class Name)	Peak Spread Based on	
	n-Alkane Carbon Numbers (Unvaporated Liquid)	Examples
1 Light Petroleum Distillates (LPD)	C ₄ -C ₁₁	Petroleum ether, Pocket lighter fuel, some rubber cement solvents, Skelly solvents, V M & P Naptha, Some camping fuels.
2 Gasoline	C ₄ -C ₁₂	All brands and grades of automotive gasoline, including gasohol.
3 Medium Petroleum Distillates (MPD)	C ₈ -C ₁₂	Mineral spirits. Some paint thinners. Some Charcoal starters, "Dry-cleaning" solvents. Some torch fuels. Some solvents for insecticides and polishes. Some lamp oils.
4 Kerosene	C ₉ -C ₁₇	Number 1 fuel oil, Jet-A (aviation) fuel, insect sprays. Some charcoal starters. Some torch fuels, Some paint thinners. Some solvents for insecticides and polishes, some lamp oils.
5 Heavy Petroleum Distillates (HPD)	C ₉ -C ₂₃	Number 2 fuel oil, Diesel fuel.
0. Miscellaneous	Variable	Single compounds. Turpentines, Specialty mixture that cannot be further classified into one of the categories below.
0.1 Oxygenated solvents	Variable	Alcohols, Esters, Ketones
0.2 Isoparaffins	Variable	Isoparaffin products. Some charcoal starters. Some copier fluids. Some aviation gasoline. Some lamp oils. Some solvents for insecticides and polishes. Some camping fuels.
0.3 Normal alkanes	Variable	Specialty products formulated from normal alkanes. Some lamp oils. Some solvents for insecticides and polishes.
0.4 Aromatic solvents	Variable	Light, medium and heavy "aromatic naptha" used as solvents for paints and plastics.
0.5 Naphthenic/paraffinic solvents	Variable	Specialty solvent/fuel products made from class 3 or Class 4 distillates treated to remove normal alkanes and aromatics, with higher cycloalkane content than isoparaffin products.

4.2 Procedure for Interpretation of Chromatograms

- 4.2.1 Obtain a chromatogram with the major peaks on-scale ($3/4$ to full scale).
- 4.2.2 Note in what region of the chromatogram the peaks reside.
 - 4.2.2.1 Examine chromatogram to determine in what retention time range the peaks are present (light, medium or heavy region).
 - 4.2.2.2 Examine chromatogram to determine the width of the carbon spread.
- 4.2.3 Compare sample chromatogram with chromatogram from a known standard obtained under similar conditions.
 - 4.2.3.1 Note significant points of correlation.
- 4.2.4 To establish carbon number range, compare the sample chromatogram with normal alkane chromatogram.
- 4.2.5 Be able to explain differences between an unknown and standard based on evaporation, interference from matrix, sample preparation techniques and/or bacterial degradation (soil samples). Unexplained differences should preclude identification as an ignitable liquid.

4.3 Criteria for Interpretation of Data

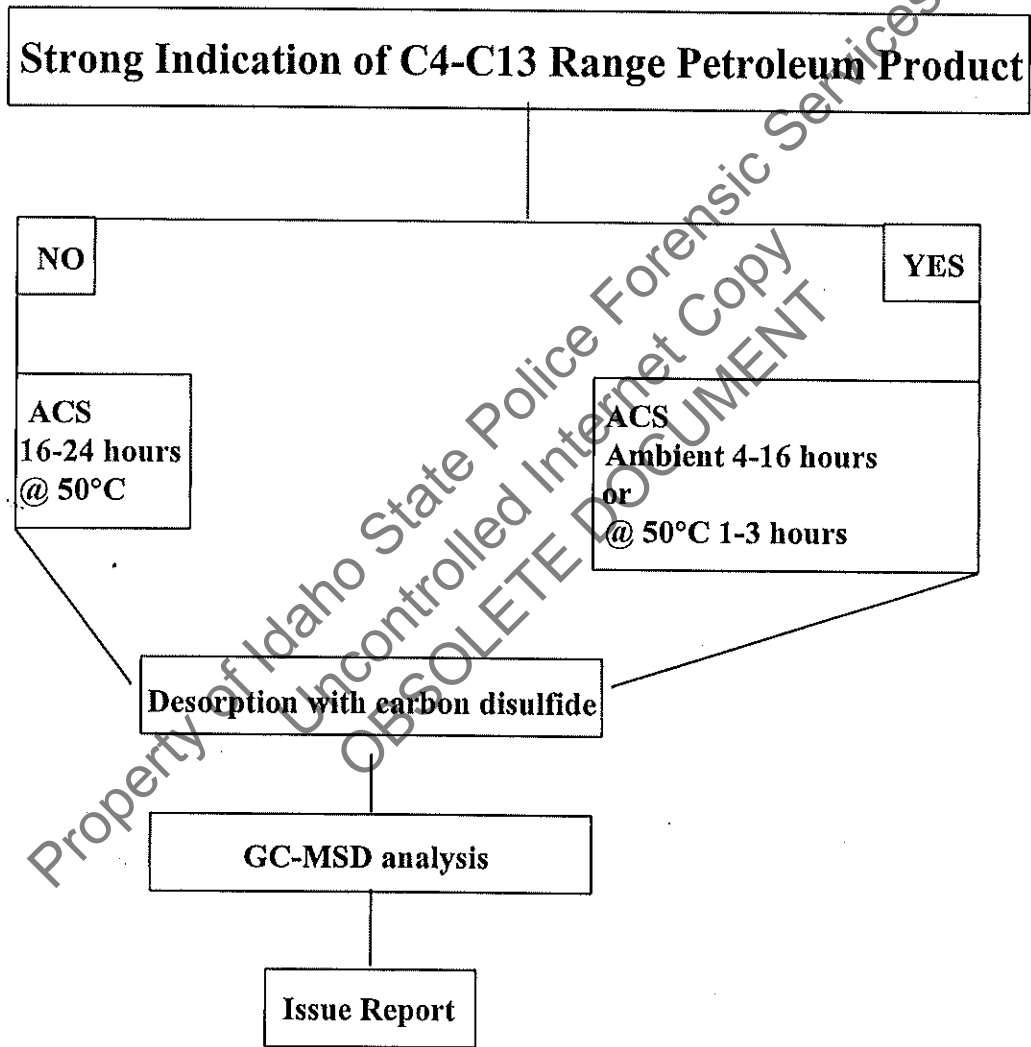
- 4.3.1 Refer to ASTM E 1618-97, *Standard Guide for Identification of Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry*, Sections 10 and 11.

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Analysis scheme for extraction of fire debris

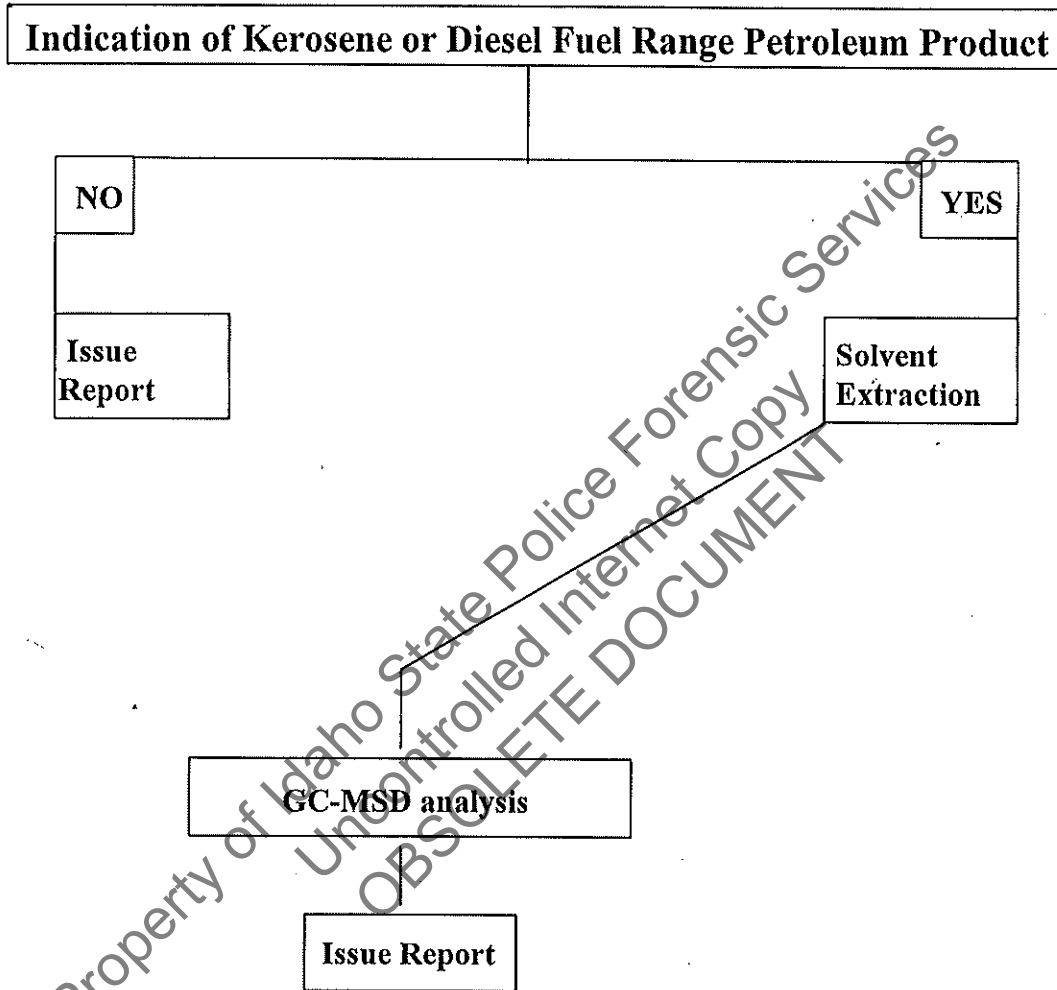
Olfactory
Screening

Case 1:



10 Analysis scheme for extraction of fire debris (cont.)

Case 2:



5.0 References

- 6.1 ASTM Method E-1412-91, **Standard Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris Samples by Passive Headspace Concentration.**
- 6.2 ASTM Method E-1412-95, **Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration.**
- 6.3 ASTM Method E-1386-90, **Standard Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris Samples by Solvent Extraction.**
- 6.4 ASTM Method E-1386-95, **Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction.**
- 6.5 ASTM Method E-1387-95, **Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography.**
- 6.6 ASTM Method E-1618-94, **Standard Guide for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.**
- 6.7 ASTM Method E-1618-97, **Standard Guide for Identification of Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.**
- 6.8 Dietz, W.R. **Improved charcoal packaging for accelerant recovery by passive diffusion.** J. Forensic Sci. 36(1):111-21;1991.
- 6.9 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.
- 6.10 Arson Accelerant Detection Course Materials, presented at Alcohol, Tobacco & Firearms Laboratory, Rockville, Maryland, May, 1993.
- 6.11 Arson Analysis Workshop Materials, presented at Northwest Association of Forensic Scientist's Fall Meeting, Salt Lake City, Utah, October, 1996.
- 6.12 Advanced Fire Debris Course Materials, presented at National Forensic Science Technology Center, St. Petersburg, Florida, December, 1996.
- 6.13 Newman, R.; Gilbert, M.; Lothridge, K. **GC-MS Guide to Ignitable Liquids.** Boca Raton, FL: CRC Press: 1998.