



**Idaho State Police  
Forensic Services  
Trace Section**

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**Section I**

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

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**1.0 SCOPE**

This method details the recovery, analysis, and identification of ignitable liquid residues from fire evidence samples as well as the care of the instrumentation utilized. Recovery is by passive headspace concentration with activated charcoal and/or solvent extraction, analysis is performed on a gas chromatograph equipped with a mass selective detector and the identification is based on the interpretation of data provided in accordance with the outlined criteria for identification. This method is based upon ASTM standard test methods and practices.

**2.0 BACKGROUND**

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. 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 which is as free from interferences as possible but still recover a good representation of the ignitable liquid product present. Other considerations include recovery techniques that are sensitive and time efficient. The analysis of recovered 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 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 context of the circumstances surrounding the fire.

**3.0 RECOVERY METHODS**

**3.1 Separation of Ignitable Liquid Residues from Fire Evidence Samples by Passive Headspace Concentration With Activated Charcoal**

**3.1.1 General**

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

- 3.1.1.2 This method utilizes activated charcoal impregnated polymer strips (ACS) to adsorb, and thus trap, ignitable liquid residues.
- 3.1.1.3 This method will recover ignitable (flammable or combustible) liquids for instrumental analysis by a gas chromatograph equipped with a mass selective detector (GC-MSD).
- 3.1.1.4 This method is time efficient and is essentially non-destructive of the sample.

3.1.2 ASTM Reference Method

Refer to ASTM standard practice E 1412-00, *Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal*.

3.1.3 Application

3.1.3.1 This procedure is useful for samples which may contain a light to medium range (C4-C14) petroleum distillate product as suggested by an indicative odor. This method will recover heavy range distillates but with the limitation indicated in section 3.1.5.1.

3.1.4 Sensitivity

3.1.4.1 Capable of isolating quantities less than 0.1 $\mu$ L of an ignitable liquid residue from a sample.

3.1.5 Limitations

3.1.5.1 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). In the updated classification system of E-1618-01 and E-1387-01, class IV and V products are classified together as Heavy Petroleum Distillates. For this reason, under E-1618-01 and E-1387-01, the designation of Heavy Petroleum Distillates/Heavy Product Range does not require the completion of a solvent extraction.

3.1.5.2 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.<sup>7.9</sup>

3.1.6 Safety Concerns

3.1.6.1 Carbon disulfide is an extremely flammable, volatile liquid which by all routes of exposure has adverse effects on the central nervous system with a

NPFA 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.

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

### 3.1.7 Materials and Equipment

#### 3.1.7.1 **Solvents**

3.1.7.1.1 Carbon disulfide (Fisher C184-500 or equivalent).  
Purity of solvents is assured by analyzing blanks on each batch of solvent by GC-MSD.

#### 3.1.7.2 **Collection device**

3.1.7.2.1 Activated charcoal polymer strips (ACS) (Albrayco Laboratories ACS-150-6PACK or equivalent)  
Size of ACS used depends upon the presence, and intensity of, a characteristic ignitable liquid odor and the sample size. Approximate size is 8 X 20mm. ASTM E1412-00 recommends a minimum strip size of 100mm<sup>2</sup>.

3.1.7.2.2 Metal, non-coated, paper clips.

#### 3.1.7.2.3 Securing of the ACS

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

Option Two: Non-waxed dental floss.

#### 3.1.7.3 **Heating system**

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

#### 3.1.7.4 **Glassware**

3.1.7.4.1 2-mL widemouth autosampler vials preassembled with screw-top cap with PTFE/silicone septa (Agilent 5182-0865 or equivalent).

3.1.7.4.2 Glass vial microinserts (Agilent 5183-2088 or equivalent).

### 3.1.8 Passive Adsorption Procedure

3.1.8.1 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.

3.1.8.2 Secure appropriately sized charcoal strip in paper clip.

3.1.8.3 Open friction lid can or evidence pouch (Kapak)<sup>®</sup> and suspend charcoal

- 3.1.10.2.2 Prepare charcoal strip standard as described for collection device above.
- 3.1.10.2.3 Place prepared collection device into prepared friction lid can.
- 3.1.10.2.4 Process charcoal strip as with case samples.

### 3.1.11 Charcoal Strip Blanks

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

### 3.1.12 Analysis

- 3.1.12.1 Analyze extracts by GC-MSD as outlined in section 4.0 of this method.

## 3.0 RECOVERY METHODS

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

#### 3.2.1 General

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

#### 3.2.2 ASTM Reference Method

Refer to ASTM standard practice E 1386-00, *Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction*.

#### 3.2.3 Application

- 3.2.3.1 Samples which may contain a kerosene to heavy petroleum distillate product as indicated by:
  - 3.2.3.1.1 Strong indicative odor.
  - 3.2.3.1.2 GC/MSD data from analysis of ACS. 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).

strip above sample. Securely reseal the container.

- 3.1.8.4 If strong odor of petroleum product 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. If the GC-MSD data indicates that the strip was clearly overloaded, the sample can be resampled at ambient temperature or with a shortened adsorption time.
- 3.1.8.5 If an odor is not present, place friction lid can into a 50-60°C for 16 to 24 hours.
- 3.1.8.6 As described in E1412-00, 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.
- 3.1.8.7 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 automated sampler (ALS) vial.

### 3.1.9 Elution Procedure

- 3.1.9.1 To elute the compounds trapped upon the charcoal, add approximately 100 to 1000 $\mu$ L carbon disulfide (CS<sub>2</sub>) to each vial. Seal vial and vortex. Place vial on its side to facilitate extraction. Vial may also be placed on rocker or rotator. Document the volume of CS<sub>2</sub> used on analysis worksheet.
- 3.1.9.2 Transfer a portion of the carbon disulfide into labeled autosampler vial with microinsert, and cap. Place the vial containing the charcoal strip and the remaining extract into the flammables refrigerator.
- 3.1.9.3 If the initial olfactory screening indicates the strong presence of an ignitable liquid in a sample, dilute extract with CS<sub>2</sub> prior to GC-MSD analysis.

### 3.1.10 Comparison Standards

- 3.1.10.1 Collection of petroleum product comparison standards with the charcoal strip device is periodically done to:
  - 3.1.10.1.1 Check the adsorption efficiency of the charcoal.
  - 3.1.10.1.2 Provide for a comparison standard collected in the same fashion as the case samples.
- 3.1.10.2 Preparation of charcoal strip comparison standards is as follows:
  - 3.1.10.2.1 Place 1 $\mu$ L standard into a clean, unused quart sized friction lid can, and seal.

In the updated classification system of E1618-01 and E-1387-01, class IV and V products are classified together as Heavy Petroleum Distillates.

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

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

### 3.2.4 Sensitivity

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

### 3.2.5 Limitations

3.2.5.1 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.

3.2.5.2 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.

### 3.2.6 Safety Concerns

3.2.6.1 Carbon disulfide is an extremely flammable, volatile liquid which by all routes of exposure has adverse affects on the central nervous system with a NPFA 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.

3.2.6.2 Pentane is an extremely flammable, volatile liquid. Although pentane only has a NPFA health rating of 1 (slight), exposure though inhalation and ingestion has an adverse affect 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.

3.2.6.3 For further information regarding the hazards of exposure to carbon disulfide refer to ASTM E 752, *Practice for Safety and Health Requirements Relating to Occupational Exposure to Carbon Disulfide*. For information on both pentane and carbon disulfide, refer to the material safety data sheets (MSDS).

### 3.2.7 Materials

#### 3.2.7.1 **Solvents**

- 3.2.7.1.1 Pentane (Fisher P400-4 or equivalent)
- 3.2.7.1.2 Hexane (Fisher H300-4 or equivalent)
- 3.2.7.1.3 Carbon disulfide (Fisher C184-500 or equivalent).
- 3.2.7.1.4 Purity of solvents is assured by analyzing blanks on each batch of solvent by GC-MSD.

#### 3.2.7.2 **Filter Paper**

- 3.2.7.2.1 Whatman Grade 1 - Qualitative (27cm) or equivalent
- 3.2.7.2.2 Whatman 1PS -Phase separation (15cm) or equivalent

#### 3.2.7.3 **Glassware**

- 3.2.7.3.1 Glass funnels
- 3.2.7.3.2 25mL to 2000mL beakers
- 3.2.7.3.3 2-mL widemouth autosampler vials preassembled with screw-top cap with PTFE/silicone septa (Agilent 5182-0865 or equivalent).
- 3.2.7.3.4 Glass vial microinserts (Agilent 5183-2088 or equivalent).

### 3.2.8 Solvent Extraction Procedure

#### 3.2.8.1 **Friction lid cans**

Cover sample with solvent and place lid on loosely. Sample should be thoroughly moistened.

#### 3.2.8.2 **Kapak<sup>®</sup> -type pouches**

Place pouch in 2-L beaker. Add sufficient solvent to thoroughly moisten sample.

3.2.8.3 Mix/soak the sample and debris for approximately one to five minutes. A beaker may be used as a plunger to facilitate the extraction of flexible debris

3.2.8.4 Filter sample through phase separation paper supported with Grade 1 filter paper.

3.2.8.5 Samples with a significant odor should be sampled prior to evaporation.

3.2.8.6 Allow sample to evaporate approximately 50%. Place a 2mL portion of the partially evaporated sample into labeled automated liquid sampler (ALS) vial.

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

Ignitable Liquid	Vendor
Pocket Light Fluid, Ronsonol	Cerilliant CSQ-2130A
Gasoline, Texaco	Cerilliant CSQ-2130B
Mineral Spirits, Klean-Strip	Cerilliant CSQ-2130C
Kerosene, Sunnyside	Cerilliant CSQ-2130D
Diesel, Texaco	Cerilliant CSQ-2130E
Liquid Sandpaper, Klean-Strip	Cerilliant CSQ-2130F
Lacquer Thinner, Klean-Strip	Cerilliant CSQ-2130G
Isopar H, Exxon	Cerilliant CSQ-2130H
Lamp Oil, Lamplight Farms	Cerilliant CSQ-2130I
Concrete and Drive Cleaner, Klean-Strip	Cerilliant CSQ-2130J
Odorless Charcoal Starter, Kingsford,	Cerilliant CSQ-2130K
LPA-142, Condea Vista	Cerilliant CSQ-2130L
Unweathered, 25% weathered, 50% weathered and 75% weathered gasoline	Restek 30096, 30097, 30098, 30099
Unweathered, 25% weathered, 50% weathered and 75% weathered mineral spirits	Restek 31225, 31226, 31227, 31228
Unweathered, 25% weathered, 50% weathered and 75% weathered diesel	Restek 31233, 31234, 31235, 31236
C-7 to C-10 n-paraffin mixture	Alltech 628002 or equivalent
C-9 to C-12 n-paraffin mixture	Alltech 628003 or equivalent
C-11 to C-14 n-paraffin mixture	Alltech 628003 or equivalent
C-12 to C-18 n-paraffin mixture	Alltech 628003 or equivalent
Aromatic Hydrocarbon Mixtures	Alltech 629001, 629002, 629003, 629004, 629005, 629006, or equivalent



### 3.2.9 Solvent Purity Check

- 3.2.9.1 Each batch of samples should include a solvent blank.
  - 3.2.9.1.1 Place approximately 200mL of extraction solvent into a clean beaker.
  - 3.2.9.1.2 Evaporate solvent to approximately 1mL.
  - 3.2.9.1.3 The degree of solvent evaporation should be at least twice the extent used for questioned samples (ASTM E1386-00, 5.2.1).

### 3.2.10 Analysis

- 3.2.10.1 Place each solvent extract into labeled ALS vial with microinsert, and cap.
- 3.2.10.2 Analyze both the partially evaporated and evaporated solvent extracts by GC-MSD as described in section 3.0.

## 4.0 ANALYSIS OF RECOVERED EXTRACTS

### 4.1 **General**

- 4.1.1 To detect the presence of an ignitable liquid residue in fire evidence samples, extracts recovered by either activated charcoal or solvent extraction are analyzed with a gas chromatograph equipped with a mass selective detector (GC-MSD).
- 4.1.2 Post-run macros, which process the data to generate extracted ion profiles, assist with the detection and identification of ignitable liquid residues.

### 4.2 **ASTM Reference Method**

Refer to ASTM E 1618-01, *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*.

### 4.3 **Chemicals and Reference Standards**

#### 4.3.1 Column Resolution Test Mixture

- 4.3.1.1 Obtain through Restek (#31224), Cerilliant (ERR-002) or equivalent vendor.
- 4.3.1.2 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.

#### 4.3.2 Ignitable Liquid Standards Sources and Authentication

- 4.3.2.1 Standards should be obtained as required from commercial and local sources. The following are representative of established classes of ignitable liquids but is not an all-inclusive list. Refer to *Ignitable Liquid Inventory* for a list of in-house standards.

#### 4.3.3 Authentication of Ignitable Liquid Standards

4.3.3.1 The authentication of ignitable liquid standards presents a unique problem since the standard is a multi-component mixture whose identification is largely based on total ion chromatograms (TIC) and extracted ion profiles (EIP). Authentication of standards from commercial sources should be based upon interpretation of the TIC and EIP data obtained through the analysis of the standard. The interpretation should use the same process applied when interpreting the data from a casework sample. In addition, the TIC and EIP data should be compared with the manufacture package insert/information packet. This overall process should verify that the standard is classified correctly. Newly obtained standards from local sources should be compared to authenticated commercially available standards prior to official use.

#### 4.4 Apparatus

##### 4.4.1 Instrumentation

Integrated system consisting of Hewlett Packard 5890 Gas Chromatograph equipped with a Hewlett Packard 5971 Mass Selective Detector operated by data station software. Data system must be capable of handling macro programs to extract ion profiles to assist with the interpretation of data.

##### 4.4.2 Column

25 meter Hewlett Packard HP-5 [(5% PhMeSilcone) with 0.22 mm ID and 0.33  $\mu$ m film thickness] or equivalent. 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 3.3.2.4.3).

#### 4.5 GC/MSD Analysis

##### 4.5.1 Instrumental Parameters

- Injector Temperature: 280°C
- Detector Temperature: 300°C
- Carrier Gas: Helium
- **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
- Sample size: 1.0 - 2.0 $\mu$ L

##### 4.5.2 Preparation for Analysis of samples by GC/MSD

###### 4.5.2.1 **Perform Autotune**

Refer to section 4.6 for *Autotune* evaluation criteria.

4.5.2.2 **Program GC-MSD Sequence**

4.5.2.2.1 Load ARSON sequence from sequence menu.

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

Sample Log Table for ARSON.M					
Line	Type	Vial	Date File	Method	Sample Name
1)	Sample	51	BLK-ACS	ARSON	QC ACS 02-01-01 ACS LOT# 062200
2)	Blank	75	BLANK	ARSON	Carbon Disulfide Blank Fisher Lot#950674
3)	Sample	53	006-2ACS	ARSON	P2001006 (1) ACS lot# 062200/CS2 lot#950674
4)	Blank	75	BLANK	ARSON	Carbon Disulfide Blank Fisher Lot#950674
5)	Sample	54	006-2SE	ARSON	P2001006 (2) Solvent e/Pentane lot#962473
6)	Sample	74	50EVGAS	ARSON	50% evap Sinclair gasoline lot#0500-1/CS2
7)	Sample	71	ASTM1387	ARSON	ASTM E1387 Column Resolution Mix*/CS2

\* Program source and lot number under sample log table *Miscellaneous Information*.

4.5.2.2.3 In Sample Log Table, program questioned samples into sequence by laboratory and sample number. Sample name description should include method of recovery.

4.5.2.2.4 Quality Control Samples

4.5.2.2.4.1 In Sample Log Table, program quality control samples into sequence by laboratory number or date prepared. Information should include method of recovery.

4.5.2.2.4.2 For system blanks for charcoal strips or solvent purity check, designate BLK-ACS, BLK-SE or similar notation.

4.5.2.2.4.3 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.

4.5.2.2.4.4 QC Test Mixture

To establish that the system has the capability to resolve compounds outlined in ASTM E1618-01/E1387-01, a column resolution check mix is analyzed with each analysis run. Program test mixture into each sequence run. TIC and extracted ion profiles (EIP) print-outs should be placed into the centrally stored *Fire Analysis Column Resolution Mixture* binder.

4.5.2.2.5 Ignitable Liquid Standards

4.5.2.2.5.1 In Sample Log Table, program appropriate

ignitable liquid standards following the questioned samples. 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.

4.5.2.2.5.2 A copy of the relevant standard(s) should be placed into the casefile.

#### 4.5.3 Post-run Selected Ion Profiles Macro

4.5.3.1 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.<sup>7,13</sup> 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
PNAs*	128	142	156	

\* Polynuclear aromatics (Naphthalenes)

#### 4.5.4 Command Line Selected Ion Profiles Macros

4.5.4.1 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.

##### 4.5.4.1.1 *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	

##### 4.5.4.1.2 *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.

## 4.6 Maintenance Schedule for Hewlett Packard 5890 Gas Chromatograph/5971 Mass Selective Detector

### 4.6.1 When-in-use Maintenance

#### 4.6.1.1 **Autotune**

##### 4.6.1.1.1 Tune Requirements

Autotune should be performed prior to a sequence run and whenever the instrument pauses. Ideally, each sequence should be bracketed by autotunes.

#### 4.6.1.2 **Interpretation of Autotune report**

##### 4.6.1.2.1 Tune Report

The Tune Report is printed as a result of performing an Autotune on the MSD. The report includes the final values of the MS parameters which were set by the tuning process. In addition the report includes the actual mass spectral data obtained using these parameters.

##### 4.6.1.2.2 Interpretation

The major parameters which are used to evaluate the performance of the MSD are peak width, mass peaks, relative abundance and isotope ratio.

##### 4.6.1.2.2.1 **Peak Width ( $PW$ )**

- Peaks of the ions mass 69, 219 and 502 resulting from the fragmentation of PFTBA are plotted on the top section of the Tune Report.
- Peak widths should fall within a range of 0.4 to 0.6.
- If the peak width noted on the Tune Report falls outside of the acceptable range, troubleshooting procedures should be initiated.

##### 4.6.1.2.2.2 **Mass Peaks**

- The lower portion of the Tune Report illustrates the PFTBA spectrum acquired during the Autotune.
- Target values for the tune masses are  $69 \pm 0.2$  amu,  $219 \pm 0.2$  amu and  $502 \pm 0.2$  amu.
- If the mass assignments do not fall in this range, troubleshooting procedures should be initiated.

4.6.1.2.2.3 **Relative Abundance**

- The lower portion of the Tune Report provides the abundance of each tune mass reported as a percentage of the most abundant mass. Acceptable values are as follows:

<i>Mass</i>	<i>Relative Abundance</i>
69.0	100%
219.0	>30%
502.0	>1%

- If the relative abundance of each mass falls below the acceptable abundance, troubleshooting procedures should be initiated. A relative abundance for mass 502 of 1% or less may indicate a dirty ion source.

4.6.1.2.2.4 **Isotope Ratio**

- The lower portion of the Tune Report will indicate the isotope ratio reported for the tune masses 69, 219 and 502. Acceptable ranges for isotope ratios are as follows:

<i>Isotope Mass</i>	<i>Isotope Ratio</i>
70.0	0.5 to 1.5%
220.0	2 to 8%
502.9	5-12%

- If the isotope ratios reported on the Tune Report do not fall within these limits, trouble-shooting procedures should be initiated.

4.6.2 **Overview of Troubleshooting**

In the advent that the MSD reports values for any of the above parameters which fall outside of the acceptable ranges, troubleshooting procedures will be initiated to determine the origin of the problem. Commonly encountered reasons for the MSD to not pass the tune criteria include:

<i>Potential Source of Problem</i>	<i>Manual Section Reference</i>
Calibration vial is empty	Hardware manual p. 4-40, 4-41
Excessive foreline or vacuum manifold pressure	Hardware manual p. 4-6, 4-14 to 4-15, 4-24 to 4-25
Dirty ion source	Hardware manual p. 4-45, 4-49, 4-52, 4-55
Calibration valve is not working correctly	Hardware manual p. 4-37, 4-40, 4-41
Bad signal cable connection	Hardware manual p. 4-36
Filament has failed or is not connected properly	Hardware manual p. 4-26 to 4-27
Bad ion source wiring connection	Hardware manual p. 4-36
Bad detector wiring connection	Hardware manual p. 4-40
Failed electron multiplier horn	Hardware manual p. 4-31, 4-33

For Additional trouble shooting information refer 5890 operating manuals and the hardware manual for the 5971.

4.6.3 As Needed Maintenance

<i>Task</i>	<i>Indications</i>	<i>Manual Reference</i>
Replace inlet/injection port seal Options: 1. Merlin Microseal™ Septum 2. Septa	<ul style="list-style-type: none"> <li>▪ Longer or shifting retention time</li> <li>▪ Loss of response</li> <li>▪ Noisy detector signal</li> </ul> Autotune indicates an air leak	Reference Manual p. 8-8 to 8-9
Replace inlet/injection port liner and O-ring	<ul style="list-style-type: none"> <li>▪ Loss of response</li> <li>▪ Visual Inspection</li> </ul>	Operating Manual p. 2-4 to 2-5 Reference Manual p. 1-17 to 1-19, 6-8 to 6-9
Replace inlet/injection port base seal	<ul style="list-style-type: none"> <li>▪ Ghost peaks</li> <li>▪ Visual Inspection</li> </ul>	Reference Manual p. 8-13
Clean inlet reducing nut	<ul style="list-style-type: none"> <li>▪ When replacing inlet base seal and/or column</li> </ul>	Reference Manual p. 35,54-55
Replace ion source filaments	<ul style="list-style-type: none"> <li>▪ Evaluation of Autotune</li> </ul>	Hardware p. 5-34 to 5-35
Clean ion source	<ul style="list-style-type: none"> <li>▪ Evaluation of Autotune</li> </ul>	Hardware p. 5-22 to 5-29
Replace column	<ul style="list-style-type: none"> <li>▪ Evaluation of chromatography and Autotune</li> </ul>	GC inlet: Operating Manual p. 2-2 to 2-3, 2-14 to 2-15. MSD: Hardware p. 3-4 to 3-5
Replace or clean split/splitless vent line trap	<ul style="list-style-type: none"> <li>▪ Evaluation of split ratio</li> <li>▪ Clogged trap will not provide proper split ratio</li> </ul>	Reference Manual General on split mode, p. Cleaning procedure following.
Lubricate seals-Side plate O-ring	<ul style="list-style-type: none"> <li>▪ Autotune indicates an air leak</li> </ul>	Hardware p. 138-139

4.6.4 Weekly Maintenance

<i>Task</i>	<i>Manual Reference</i>
Check foreline pump oil fluid level	Hardware p. 5-8 to 5-11

4.6.5 Six Month Maintenance

<i>Task</i>	<i>Manual Reference</i>
Pump Maintenance <ul style="list-style-type: none"> <li>▪ Drain and replace foreline pump fluid</li> <li>▪ Remove and inspect oil trap, refill or replace</li> </ul>	Hardware p. 5-12 to 5-13
Check PFTBA calibration vial <ul style="list-style-type: none"> <li>▪ Refill if necessary</li> </ul>	Hardware p. 5-20 to 5-21

## 5.0 INTERPRETATION OF ANALYTICAL RESULTS

### 5.1 Petroleum Product Classification System

#### 5.1.1 E1618-01 and E1387-01 Classification system

5.1.1.1 Classification system consists of nine major classes of ignitable liquids.

5.1.1.2 All but gasoline are further differentiated into three subclasses based on carbon spread/n-hydrocarbon boiling range. In addition, this system allows for the better 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.

5.1.1.3 The products listed in the table below 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<sup>7-16</sup>.

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**E1618-01/E1387-01 Ignitable Liquid Classification Scheme** <sup>7.16, 7.17</sup>

Class	Light {C <sub>4</sub> - C <sub>9</sub> }	Medium {C <sub>8</sub> - C <sub>13</sub> }	Heavy {C <sub>8</sub> - C <sub>20+</sub> } <sup>7.16</sup> Heavy {C <sub>9</sub> - C <sub>20+</sub> } <sup>7.17</sup>
Gasoline All brands, including gasohol	Fresh gasoline is typically in the C <sub>4</sub> -C <sub>12</sub> 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 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 Carbonless Forms Copier Toners
De-Aromatized Distillates	Some Camping Fuels	Some Charcoal Starters Some Paint Thinners	Some Charcoal Starters Odorless Kerosenes
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

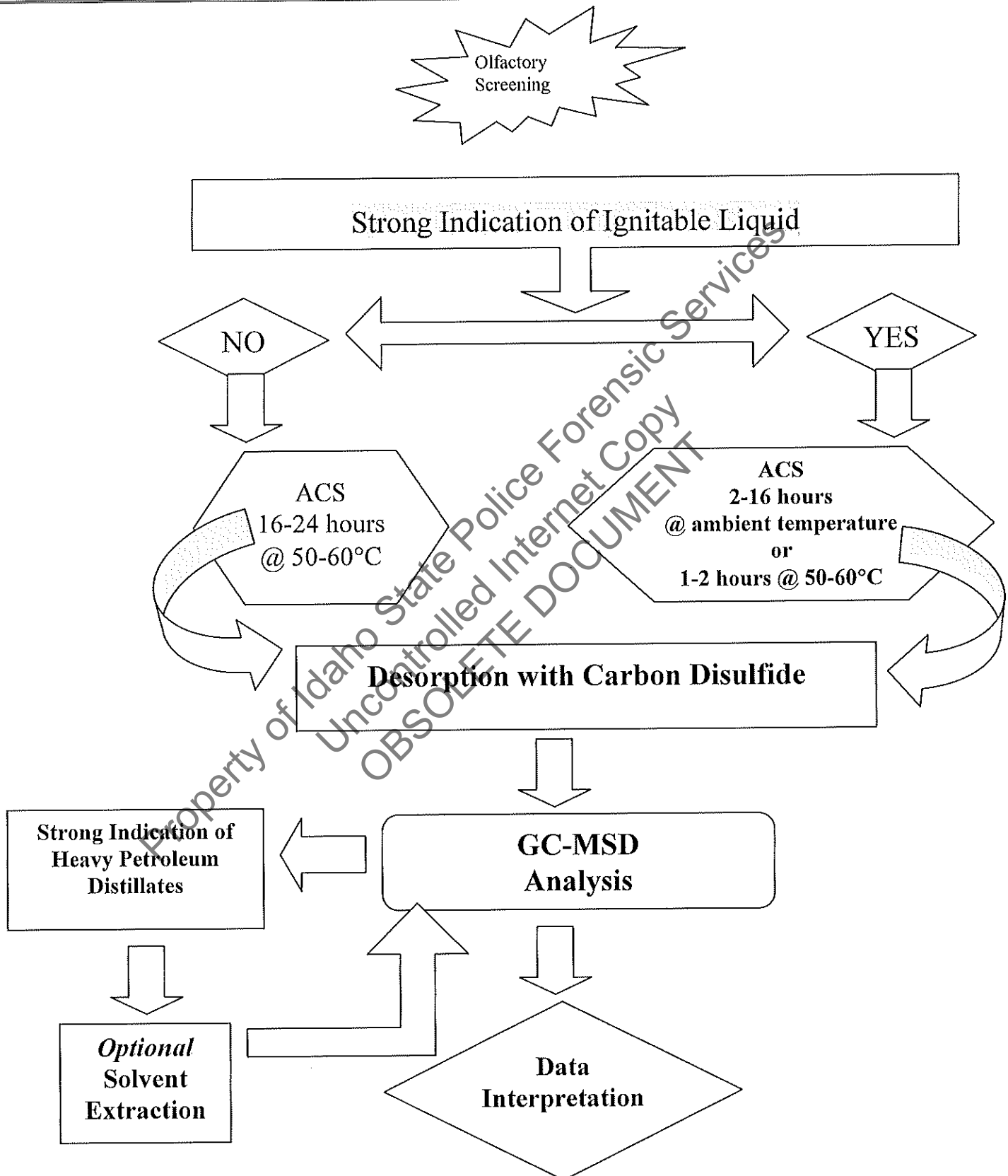
## 5.2 Procedure for Interpretation of Chromatograms

- 5.2.1 Obtain a chromatogram with the major peaks on-scale (three-quarters to full scale).
- 5.2.2 Note in what region of the chromatogram the peaks reside.
  - 5.2.2.1 Examine chromatogram to determine in what retention time range the peaks are present (light, medium or heavy region).
  - 5.2.2.2 Examine chromatogram to determine the width of the carbon spread.
- 5.2.3 Compare sample chromatogram with chromatogram from a known standard obtained under similar conditions.
  - 5.2.3.1 Note significant points of correlation.
- 5.2.4 To establish carbon number range, compare the sample chromatogram with normal alkane chromatogram.
- 5.2.5 The analyst must consider and be prepared to explain any observed differences between the unknown (questioned sample) and a standard. 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). Unexplained differences should preclude the identification as an ignitable liquid.

## 5.3 Criteria for Interpretation of Data

- 5.3.1 Refer to ASTM E 1618-01, *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*.

## 6.0 ANALYSIS SCHEME FOR EXTRACTION OF FIRE DEBRIS



## 7.0 REFERENCES

- 7.1 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.**
- 7.2 ASTM Method E-1412-95, **Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration.**
- 7.3 ASTM Method E-1386-90, **Standard Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris Samples by Solvent Extraction.**
- 7.4 ASTM Method E-1386-95, **Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction.**
- 7.5 ASTM Method E-1387-95, **Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography.**
- 7.6 ASTM Method E-1618-94, **Standard Guide for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.**
- 7.7 ASTM Method E-1618-97, **Standard Guide for Identification of Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.**
- 7.8 Dietz, W.R. **Improved Charcoal Packaging for Accelerant Recovery by Passive Diffusion.** J. Forensic Sci. 36(1):111-21;1991.
- 7.9 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.
- 7.10 Arson Accelerant Detection Course Materials, presented at Alcohol, Tobacco & Firearms Laboratory. Rockville, Maryland, May, 1993.
- 7.11 Arson Analysis Workshop Materials, presented at Northwest Association of Forensic Scientist's Fall Meeting. Salt Lake City, Utah, October, 1996.
- 7.12 Advanced Fire Debris Course Materials, presented at National Forensic Science Technology Center, St. Petersburg, Florida, December, 1996.
- 7.13 Newman, R.; Gilbert, M.; Lothridge, K. **GC-MS Guide to Ignitable Liquids.** Boca Raton, FL: CRC Press: 1998.

- 7.14 ASTM Method E-1386-00, **Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction**
- 7.15 ASTM Method E-1412-00, **Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal.**
- 7.16 ASTM Method E-1618-01, **Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.**
- 7.17 ASTM Method E1387-01, **Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography.**

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