Technical Procedure for the Examination of Fire Debris Evidence

1.0 Purpose – This technical procedure shall be followed for the examination of fire debris evidence.

2.0 Scope – This procedure applies to fire debris samples as well as other volatile compounds, semi-volatile compounds and single component or very light products such as alcohols, acetone, ethylene glycol or other solvents.

3.0 Definitions – N/A

4.0 Equipment, Materials, and Reagents

4.1 Equipment

- Oven
- Gas Chromatograph-Flame Ionization Detector (GC-FID)
- Gas Chromatograph-Mass Spectrometer (GC-MS)
- Ultrasonic cleaner or vortex mixer
- NIST Traceable Thermometer

4.2 Materials

- Pasteur Pipettes
- Screw top glass vials
- Auto sampler vials, 150 µL vial inserts, and crimp seals
- Vial crimper and decrimper
- Razor blades
- Weigh pans
- Small diameter wire
- Rubber septa
- Syringeless filters
- 2.5 mL airtight syringe
- In-house reference collection for fire debris examinations
- 100 % diesel fuel reference material

4.3 Reagents

- Carbon disulfide - Reagent A.C.S. grade
- Petroleum ether – Optima Grade
- Activated charcoal strips
- Acetone – Reagent A.C.S. grade

5.0 Procedure

5.1 Analytical Approach

5.1.1 Review the request for analysis. Items with a request for both fire debris examination and DNA analysis must be addressed prior to extraction.

5.1.2 Open the evidence container. Describe the contents and any ignitable liquid odors.

5.1.3 Based on the sample material, type of odor present and training and experience, the Forensic Scientist shall determine the most appropriate extraction technique as described in **5.2**.

5.1.4 Once the appropriate extraction technique is performed, the resulting extract undergoes instrumental analysis based on the guidelines as described in **5.3**.

5.1.5 At the completion of the examination, the Forensic Scientist shall issue a report stating his or her findings using **5.4** and **5.5** as guides.

5.2 Extraction Techniques

5.2.1 Neat (Liquid) Sample

5.2.1.1 Using a Pasteur pipette, transfer a sample of the liquid to an autosampler vial and cap with a crimp seal. A vial insert may be used for smaller sample amounts.

5.2.1.2 A syringeless filter may be used to remove particulate material or debris present in the sample.

5.2.1.3 Analyze the sample based on **5.3**.

5.2.2 Heated Headspace Analysis

5.2.2.1 For samples packaged in a can or jar, puncture a small hole in the lid and insert a rubber septum. Nylon bags can be sampled through the bag wall.

5.2.2.2 Heat a 2.5mL airtight syringe in an oven at approximately 70 °C for 10-15 minutes.

5.2.2.3 Prepare the GC-MS by loading the Alcohol method. See the Trace Evidence Section Technical Procedure for Gas Chromatography–Mass Spectrometry.

5.2.2.4 Remove the syringe from the oven. Draw approximately 1.0 mL of air. Immediately inject the air blank into the GC-MS and begin data collection.

5.2.2.5 Heat the sample and the same syringe in an oven at approximately 70 °C for 10-15 minutes.

5.2.2.6 Confirm the air blank has no peaks that would interfere with the analysis.

5.2.2.7 Remove the sample and syringe from the oven. Insert the syringe into the container through the rubber septum or nylon bag wall and slowly pump the syringe several times.

5.2.2.8 Draw approximately 1.0 mL of vapor from the container. Immediately inject the sample into the GC-MS and begin data collection.

5.2.3 Solvent Extraction

5.2.3.1 Using a Pasteur pipette add an adequate amount of appropriate solvent (usually petroleum ether or carbon disulfide) and rinse the debris several times.

5.2.3.2 Separate the solvent from any water that may be present and collect a sample of the organic layer.

5.2.3.3 If a large volume of solvent is used to rinse the debris, the solvent extract may be concentrated by heating lightly or by evaporating with a stream of clean nitrogen gas or air.

5.2.3.4 A syringeless filter may be used to remove particulate material or debris present in the sample.

5.2.3.5 Analyze a sample of the organic layer based on **5.3**.

5.2.4 Diffusive Flammable Liquid Extraction (DFLEX) or Adsorption Elution Extraction (Passive) with Charcoal Strips

5.2.4.1 Inspect the fire debris sample container for the presence of the red DFLEX device alert label. If a label is found, proceed to **5.2.4.3**.

5.2.4.2 If an alert label is not found, open and add a DFLEX extraction device or suspend an activated charcoal strip in the evidence container. If using a charcoal strip, small diameter wire shall be used to suspend the strip in a metal paint can or nylon bag. Avoid touching the DFLEX device or charcoal strip. Reseal the evidence container.

5.2.4.3 Place the fire debris container with extraction device into an oven at approximately 70 °C for approximately sixteen (16) hours.

5.2.4.4 After approximately sixteen (16) hours, remove the fire debris container with extraction device from the oven and allow it to cool to room temperature.

5.2.4.5 Once cool, remove the DFLEX device or charcoal strip from the container. If DFLEX device is used, cut through the membrane using a clean razor blade and remove the charcoal strip.

5.2.4.6 Cut the strip into pieces that will fit into and lie on the bottom of a screw top glass vial.

5.2.4.7 Add carbon disulfide to the vial to cover the pieces of charcoal strip. When attempting to extract ethylene glycol, substitute acetone for carbon disulfide.

5.2.4.8 Agitate the sample for 15 minutes using an ultrasonic cleaner or vortex mixer.

5.2.4.9 Analyze a sample based on **5.3.**

5.3 Guidelines for Instrumental Analysis

5.3.1 Samples suspected to contain light solvents, such as alcohols and acetone, shall be analyzed by the Trace Evidence Section Technical Procedure for Gas Chromatography–Mass Spectrometry utilizing a method with no solvent delay. This method may be applied to all items in the case. These samples do not require analysis by the Trace Evidence Section Technical Procedure for Gas Chromatography.

5.3.2 Samples with ignitable liquid odors may be analyzed only using the Trace Evidence Section Technical Procedure for Gas Chromatography–Mass Spectrometry. If analyzed by GC-MS, these samples do not require analysis by the Trace Evidence Section Technical Procedure for Gas Chromatography.

5.3.3 For Heated Headspace samples, the extract shall be analyzed by the Trace Evidence Section Technical Procedure for Gas Chromatography–Mass Spectrometry using the Alcohol method.

5.3.4 All other samples shall be analyzed by the Trace Evidence Section Technical Procedure for Gas Chromatography. If the sample is negative, the examination may be concluded. If additional analysis is required, the extract shall be analyzed further using the Trace Evidence Section Technical Procedure for Gas Chromatography–Mass Spectrometry.

5.4 Guidelines for Classification

5.4.1 ASTM procedures and classifications shall be used as a guide for flammable/combustible liquid identifications. All results shall be based on the Forensic Scientist's knowledge and experience and the case being examined. Results shall be in agreement with the technical reviewer.

5.4.2 When possible, an unknown sample shall be compared to a known reference material in order to confirm the classification. However, no classification system is likely to describe all possible samples. Other techniques, such as GC-MS and extracted ion profiles may be used to specifically identify target components of a flammable/combustible liquid.

5.4.3 Classification Criteria

5.4.3.1 Light Petroleum Distillates (LPD)

- At a minimum, 4 major peaks in the C4 to C9 range.
- No major peaks associated with the ignitable liquid exist above C11.
- Petroleum distillate pattern shall be comparable to that of known reference materials.

5.4.3.2 Gasoline

- The m-ethyltoluene/pseudocumene 5-peak group must be present; this group occupies the range between C9 and C10.
- Higher peak groupings characteristic of gasoline, such as tetramethylbenzene and 1- and 2-methylnaphthalene, with cutoff between C12 and C13, should also be present.
- Hydrocarbon pattern shall be comparable to that of known reference materials.

5.4.3.3 Medium Petroleum Distillates (MPD)

- Pattern starts between C8 and C10, ends near C13, and contains at least 3 consecutive n-alkane peaks between C8 and C13.
- Petroleum distillate pattern shall be comparable to that of known reference materials.

5.4.3.4 Medium – Heavy Petroleum Distillate (M-HPD)

- Pattern starts between C8 and C10 and ends between C14 and C17.
- Petroleum distillate pattern shall be comparable to that of known reference materials.

5.4.3.5 Heavy Petroleum Distillates (HPD)

- Pattern starts above C8.
- At a minimum, 5 consecutive n-alkane peaks between C9 and C23 shall be present.
- Also included in the subclass are narrow range (encompassing less than five n-alkanes) distillates starting above C11.
- Petroleum distillate pattern shall be comparable to that of known reference materials.
- Kerosene and diesel fuel shall be included within the HPD classification.

5.4.3.5.1 Kerosene

- Pattern starts above C8.
- At a minimum, 5 consecutive n-alkane peaks between C9 and C17 must be present.
- Petroleum distillate pattern shall be comparable to that of known reference materials.

5.4.3.5.2 Diesel Fuel

- Pattern starts above C8 and extends to C23.
- At a minimum 5 consecutive n-alkane peaks between C9 and C23 must be present.
- Petroleum distillate pattern shall be comparable to that of known reference materials.

5.4.3.6 Aromatic Products

- Product comprised almost exclusively of aromatic and/or condensed ring aromatic compounds. GC-MS confirmation shall be required.
- Petroleum distillate pattern shall be comparable to that of known reference materials.

5.4.3.7 Normal Alkane Products (n-alkane)

- Product comprised of only normal alkanes. GC-MS confirmation shall be required.
- Petroleum distillate pattern shall be comparable to that of known reference materials.

5.4.3.8 Isoparaffinic Products

- Product comprised almost exclusively of branched chain aliphatic compounds. GC-MS confirmation shall be required.
- Petroleum distillate pattern shall be comparable to that of known reference materials.

5.4.3.9 Naphthenic – Paraffinic Products

- Products are mainly comprised of branched chain and cyclic alkanes. GC-MS confirmation shall be required.
- n-Alkanes may be absent or diminished in concentration.
- Petroleum distillate pattern shall be comparable to that of known reference materials.

5.4.3.10 Oxygenated Solvents

- Comprised of one or more oxygenated compounds typically present before C8. GC-MS confirmation shall be required.
- May include mixtures of oxygenated compounds or other compounds such as toluene, xylene and distillate formations.
- Oxygenated compound must be an order of magnitude greater than the other ignitable liquid components and/or sample background.

5.4.3.11 Single-component samples such as alcohols, ethylene glycol or toluene may be identified by GC-MS identification of the components.

5.4.3.12 Mixtures and simple multi-component samples such as miscellaneous products and oxygenated solvents may be identified by GC-MS identification of the components and/or classifications.

5.4.3.13 Motor oil may be identified by GC-MS identification of the components and comparison to a known reference material.

5.4.3.14 Paraffin products such as candle wax may be identified by GC-MS identification of the components and comparison to a known reference material.

5.5 Guidelines for Fire Debris Analysis Result Statements

5.5.1 A methodology statement shall be added to all reports in which analysis was performed.

5.5.1.1 Example: The following methodologies were used in the examination of this case: visual examination, odor assessment, GC-FID and GC-MS.

5.5.2 Positive

5.5.2.1 Conclusion for items in which the sample has been identified through analysis. See **6.0** for limitations on reporting results.

5.5.2.1.1 Example: Examination of Item A revealed the presence of residual gasoline.

5.5.2.1.2 Example: Examination of Item A revealed the presence of a heavy petroleum distillate. Heavy petroleum distillates include kerosene, diesel fuel and some charcoal starters.

5.5.2.2 Samples examined using the GC-MS shall be reported as the specific compound in cases of single component samples and simple mixtures.

5.5.2.2.1 Example: Examination of Item A revealed the presence of toluene, xylene, and ethyl benzene.

5.5.3 Negative

5.5.3.1 Conclusion for items in which analysis of the sample resulted in a negative finding.

5.5.3.1.1 Example: Examination of Item A failed to reveal the presence of an ignitable liquid.

5.5.4 Not Examined

5.5.4.1 Samples for fire debris analysis must be packaged in vapor-tight containers to prevent contamination or loss of sample. If a sample is not packaged in a vapor-tight container or is packaged in a container that has been punctured, broken or rusted through, no analysis shall be performed except as described in **5.9.2.1**.

5.5.4.1.1 Example: Item A was not examined due to improper packaging. Proper packaging for fire debris evidence includes properly sealed nylon bags, glass jars with air tight lids, and lined metal paint cans.

5.5.4.1.2 Example: The packaging for Item A was no longer airtight due to holes in the metal can; therefore, no analysis could be performed.

5.5.4.2 If multiple items are representative of the same sample (i.e., Item B is the liquid removed from the bottle in Item A), one or more of the items may be returned without examination as described in 5.9.2.2.

5.5.4.2.1 Example: Item A was not examined.

5.6 Standards and Controls

5.6.1 There shall be at least one solvent blank run for each case.

5.6.1.1 If the sample being analyzed is a neat sample, petroleum ether or carbon disulfide shall be acceptable for the blank.

5.6.1.2 When an extraction is performed, the same solvent used for the extraction shall be used for the blank.

5.6.1.3 If multiple extraction techniques are used within the same case, a solvent blank shall be run between extraction types.

5.6.1.4 The resulting data from the blanks shall have no peaks that interfere with the analysis. If such peaks are detected, a new extraction shall be performed.

5.6.2 Quality assurance checks shall be performed upon receipt of a new lot of reagent.

5.6.2.1 The purity of a new lot of solvent, such as carbon disulfide or acetone, shall be verified prior to use by analyzing an aliquot of the solvent at 50% evaporation by GC-MS.

5.6.2.2 The efficiency and performance of a new lot of activated charcoal strips shall be verified prior to use by analyzing a control blank and a positive control. The control blank consists of eluting an unused activated charcoal strip and analyzing by GC-MS. The positive control consists of extracting an activated charcoal strip in a container spiked with 100% diesel fuel following **5.2.4** and analyzing by GC-MS.

5.6.3 For extractions following **5.2.4** using bulk-packed activated charcoal strips, an unused activated charcoal strip shall be eluted and examined as a control blank by GC-FID. If the case was examined by GC-MS only, then the control blank shall be run on the GC-MS. The resulting data from the control blank shall have no peaks that interfere with the analysis and shall be included with the case. If such peaks are detected, a new extraction shall be performed.

5.6.4 If the case was examined by GC-FID, a Performance Check Standard (100 % diesel fuel standard) shall be run and the resulting data shall be included with the case. If the case was examined by GC-MS only using a liquid sample injection, a Performance Check Standard (100 % diesel fuel standard) shall be run on the GC-MS and the resulting data shall be included with the case.

5.7 **Calibrations** – No additional calibrations or performance checks are required. See associated technical procedures for instrumental performance checks.

5.8 **Maintenance** – No additional maintenance is required. See associated technical procedures for instrumental maintenance procedures.

5.9 Sampling and Sample Selection

5.9.1 No sampling is performed. When sample selection occurs, it shall be based on the Forensic Scientist's training and experience.

5.9.2 In general, every item that is submitted for examination shall be processed with the following exceptions:

5.9.2.1 Items that are improperly packaged shall not be examined unless the item is a closed container with the odor of an ignitable liquid.

5.9.2.2 If a sample is removed from a container and both items are submitted for examination, the analysis will conclude after the first positive result.

5.10 **Calculations** – N/A

5.11 **Uncertainty of Measurement** – N/A

6.0 Limitations

6.1 If a thermal extraction procedure (headspace analysis, charcoal strip or DFLEX) is performed on the debris, kerosene cannot be a conclusion. In this case Heavy Petroleum Distillate shall be noted as the conclusion. If diesel fuel is indicated by this extraction, diesel fuel can be concluded from this type of extraction.

6.2 The thermal extraction procedures require a temperature of approximately 70 °C. The temperature readings for ovens used for fire debris examination shall be monitored and recorded once a month. The temperature reading shall also be monitored and recorded once every 12 months with a NIST traceable thermometer. The acceptable temperature range for an oven shall be 60 °C to 80 °C. If the temperature is outside of the range, the oven temperature shall be adjusted and allowed to equilibrate until a temperature within this range is acquired.

7.0 Safety

7.1 Refer to Appendix 1 for chemical hygiene and safety precautions.

7.2 Glass pipettes and razor blades are sharp and can be dangerous.

7.3 Burns may result from contact with hot items such as containers and syringes.

7.4 Syringe needles are sharp and can easily puncture skin.

7.5 Care shall be exercised when using the above-listed items or procedures.

7.6 Olfactory analysis may pose a health risk.

8.0 References

ATF National Laboratory Center Class, "Laboratory Detection and Identification of Accelerants Found in Arson Debris."

Demers-Kohls J.F., Ouderkirk S.L., Buckle J.L., Norman W.E., Cartwright N.S., Dagenais C. "Evaluation of the DFLEX Device for Fire Debris Analysis." *Canadian Society of Forensic Sciences Journal* 27.3 (1994).

Saferstein, R. *Forensic Science Handbook*. Volume I. 2nd edition. Upper Saddle River, NJ: Prentice Hall, 2002. Chapter 9: Arson and Explosive Investigation. pp. 479-524.

ASTM Standard E1386, 2000, "Standard Practice for Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction." ASTM International, West Conshohocken, PA, 2001, www.astm.org

ASTM Standard E1387, 2001, "Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography." ASTM International, West Conshohocken, PA, 2001, www.astm.org.

ASTM Standard E1388, 2000, "Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples." ASTM International, West Conshohocken, PA, 2000, www.astm.org.

ASTM Standard E1412, 2000, "Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Activated Charcoal." ASTM International, West Conshohocken, PA, 2001, www.astm.org.

ASTM Standard E1618, 2006, "Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography – Mass Spectrometry." ASTM International, West Conshohocken, PA, 2006, www.astm.org.

Trace Unit validation study, "Validation of the Extraction of Ethylene Glycol by Passive-Adsorption Elution using Charcoal Strips Eluted with Acetone." Summer 2014.

9.0 Records

- Oven temperature log

10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
06/01/2020	5	Updated Section name in header and throughout procedure. Clarified DNA requests on fire debris evidence in 5.1.1 Specified “Pasteur pipette” in 5.2.1.1 Removed dispenser bottle from 5.2.3.1 Reworded and clarified that all samples in a case may be included in 5.3.1 Added 5.3.2 for samples with ignitable liquid odors Added 5.4.3.10 and 5.4.3.12 to clarify classification of oxygenated solvents, multi-component samples and mixtures Added methodology statement as 5.5.1 Added example for damaged packaging results as 5.5.4.1.2 Added 5.6.2 Edited 7.1 to reference Appendix 1. Added razor blades to 7.2 Added 7.6 Added chemical hygiene and safety precautions to Appendix 1.

Appendix 1

Carbon Disulfide
DANGER: HIGH RISK SUBSTANCE*

HEALTH	**2**
FLAMMABILITY	**3**
REACTIVITY	**0**

Detection of Release	Strong Odor/Stench
Signs/Symptoms of Exposure	Skin Irritation; serious eye irritation; acute exposure to high concentrations of carbon disulfide may result in rapid onset of both local irritation and concentration-dependent increased severity of neurological symptoms such as nausea, dizziness, headache, delusions, hallucinations, delirium, mania, psychosis, blurred vision, convulsions, and coma.
PEL	OSHA 20 ppm (TWA)/Peak 100 ppm (8 hr shift)
Associated Hazards	Flammable Liquid; Inhalation hazard/respiratory sensitizer; suspected of damaging fertility or the unborn child; skin irritant, chronic damage to organs if inhaled.
Controls	Use under fume hood. Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product. Use eye protection. Handle with gloves. Wear lab coat. Gloves: Fluorinated rubber 0.7 mm thickness (break through time = 480 minutes), nitrile (break through time = 2 minutes)
Safe handling, storage, disposal	Avoid contact with skin and eyes. Avoid inhalation of vapor or mist; Use explosion-proof equipment; Keep away from heat and sources of ignition; take measures to prevent the build-up of electrostatic charge. Dispose in Hazardous Chemical Waste.
Emergency Procedures	**Eye Contact:** Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician. **Inhalation Exposure:** If inhaled, move person into fresh air. If not breathing, give artificial respiration. Consult a physician. **Ingestion:** Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician. **Skin Contact:** Take off contaminated clothing and shoes immediately. Wash off with soap and plenty of water. Consult a physician. **Spills:** Avoid breathing vapors, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapors accumulating to form explosive concentrations. Vapors can accumulate in low areas. Small contained spill: wearing appropriate PPE, collect with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and place in container. Dispose in

	Hazardous Chemical Waste. Large spills: Evacuate area and call 911 (Haz Mat).

Petroleum Ether **DANGER: HIGH RISK SUBSTANCE**[*]	
	**HEALTH 2** **FLAMMABILITY 4** **REACTIVITY 0**
Detection of Release	Odor of petroleum distillates
Signs/Symptoms of Exposure	Breathing difficulties. Inhalation of high vapor concentrations may cause symptoms like headache, dizziness, tiredness, and nausea and vomiting. Acute neurotoxic effects of petroleum ether include anesthesia, euphoria, abuse, vertigo, and limb numbness. Chronic neurotoxic effects include motor polyneuropathy.
PEL	OSHA 300 ppm (TWA); NIOSH- IDLH TWA @ 350 mg/m^3, Ceiling @ 1800 mg/m^3
Associated Hazards	Highly flammable liquid and vapor. May be fatal if swallowed or enters airways. May cause genetic defects. May cause cancer.
Controls	Use under fume hood. Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product. Use eye protection. Handle with gloves. Wear lab coat. Gloves: nitrile (break through time = 29 minutes)
Safe handling, storage, disposal	Handling: Use protective equipment. Do not get in eyes, on skin, or on clothing. Use only under a chemical fume hood. Do not breathe vapors or spray mist. Do not ingest. Keep away from open flames, hot surfaces and sources of ignition. Use only non-sparking tools. Use explosion-proof equipment. Take precautionary measures against static discharges. To avoid ignition of vapors by static electricity discharge, all metal parts of the equipment must be grounded. Storage: Keep containers tightly closed in a dry, cool and well-ventilated place. Keep away from heat and sources of ignition. Dispose in Hazardous Chemical Waste.
Emergency Procedures	**<u>Eye Contact</u>:** Flush eyes with water as a precaution. **<u>Inhalation Exposure</u>:** If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician. **<u>Ingestion</u>:** Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician. **<u>Skin Contact</u>:** Wash off with soap and plenty of water. Consult a physician. **<u>Spills</u>:** Avoid breathing vapors, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapors accumulating to form explosive concentrations. Vapors can accumulate in low areas. Small contained spill: wearing appropriate PPE, collect with non-combustible absorbent material, (e.g. sand, earth,

	diatomaceous earth, vermiculite) and place in container. Dispose in Hazardous Chemical Waste. Large spills: Evacuate area and call 911 (Haz Mat).