
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.

- 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 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 2.5 mL 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 Unit [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 or dispenser bottle, 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 For neat samples with light solvent odors, such as alcohols and acetone, and DFLX extractions when light solvents are suspected, the extract shall be analyzed by the Trace Unit [Technical Procedure for Gas Chromatography–Mass Spectrometry](#) utilizing a method with no solvent delay.

5.3.2 For Heated Headspace samples, the extract shall be analyzed by the Trace Unit [Technical Procedure for Gas Chromatography–Mass Spectrometry](#) using the Alcohol method.

5.3.3 All other samples shall be analyzed by the Trace Unit [Technical Procedure for Gas Chromatography](#). If the sample can be identified based on **5.4 – Guidelines for Classification** or if the sample is negative, the examination may be concluded and report written. If additional analysis is required due to the GC-FID results, the classes indicated under **5.3.3.1**, or based on the Forensic Scientist’s training and experience, the extract shall be analyzed further using the Trace Unit [Technical Procedure for Gas Chromatography–Mass Spectrometry](#). After GC-MS analysis, the examination shall be concluded and report written.

5.3.3.1 The sample shall be analyzed using the Trace Unit [Technical Procedure for Gas Chromatography–Mass Spectrometry](#) when reporting results that include an Aromatic product (**5.4.3.8**), Normal Alkane product (**5.4.3.9**), Isoparaffinic product (**5.4.3.10**), Naphthenic-Paraffinic product (**5.4.3.11**), single-component sample (**5.4.3.12**), motor oil (**5.4.3.13**) or paraffin product (**5.4.3.14**).

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

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

5.4.3.12 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 Positive

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

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

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

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

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

5.5.2 Negative

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

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

5.5.3 Not Examined

5.5.3.1 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**.

5.5.3.1.1 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.

5.5.3.2 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.

5.5.3.2.1 Example: Item A was not examined.

5.6 Standards and Controls

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- 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** 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.
- 5.6.3** 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 Carbon disulfide may be toxic. Consult Material Safety Data Sheets for information on safe use.

7.2 Glass pipettes 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.

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
08/29/2014	1	Original ISO Document Supersedes “Technical Procedure for the Examination of Arson Evidence, Version 3”
03/20/2015	2	Added ethylene glycol to 2.0 and 5.4.3.12 Added acetone to 4.3 Added acetone option to 5.2.4.7 Reworded 5.6.1.2 Added “Performance Check Standard” to 5.6.3 Added “and recorded” to 6.2 Added validation study to list of references under 8.0
12/11/2015	3	Added “vial” and “reference material” and changed 100 to 150 in 4.2 Clarified steps in 5.2.2 Corrected heading reference in 5.2.4.1 Changed 60 to 70 in 5.2.4.3 Reworded 5.2.4.8 Indented previous sections 5.4.3.6 and 5.4.3.7 (now 5.4.3.5.1 and 5.4.3.5.2) Add reference to 5.9.2.2 to 5.5.3.2 Changed 60 to 70 in 6.2