1. **Purpose:** To determine the presence of flammable liquid(s) in fire debris.

2. **Responsibility:** Analysts who conduct examinations within the fire debris category of testing.

3. **Materials:**

   a) Heating mantles: capable of heating a pint, quart or gallon can
   b) Thermometer: 0°C – 100°C (reference only)
   c) Vacuum source (mechanical pump or equivalent)
   d) Cans: unused paint cans (e.g., pint, quart, one gallon, five gallon)
   e) Standard laboratory glassware
   f) Pentane (reagent grade or equivalent)
   g) Acetone (reagent grade or equivalent)
   h) Appropriate standards and controls (e.g., hydrocarbon mix)
   i) Activated charcoal tubes (SKC or equivalent)
   j) Gas chromatograph/Mass Spectrometer (GC/MS) (Agilent or equivalent)
   k) Nitrogen Gas

4. **Standards and Controls**

   a) Positive Controls
   One or more of the following will be analyzed with every case and at least daily to ensure that instrumentation are operating as expected.
      i. Hydrocarbon Mixture (C8 – C20 ; Prepared in-house from individual hydrocarbon components at approximately 1000ppm in appropriate solvent (e.g., pentane, hexane))
      ii. Gasoline Reference Standard (Prepared in-house at an appropriate concentration within pentane)
      iii. Other ignitable liquid standards (National Center for Forensic Science or equivalent)

   b) Negative Controls
   One or more of the following will be analyzed with every case and prior to each batch of extract solutions that are analyzed to ensure that extractions and instrumentation are free from

contamination. If samples are concentrated under $N_{2(g)}$, then a negative control (same solvent used for the evidence sample) will be concentrated and analyzed in the same manner at least once during the case in order to demonstrate that extractions are contamination-free.

i. Pentane
ii. Acetone
iii. Empty Can
iv. Activated Charcoal Tube

c) Blanks – An appropriate blank solvent (e.g., extraction solvent) should be analyzed in-between each sample to ensure the instrument is free from carryover and is ready for the next analysis. However, if a sample is analyzed and the result is negative for accelerant-related compounds, then a blank solvent analysis is not required before the next sample is analyzed.

5. Procedure

a) Initial Examination
   i. Observe the evidence (i.e., contents of the can/packaging)
   ii. If necessary, appropriately smell the item to determine if odors are present (e.g., flammable liquid odors)
   iii. Note on the worksheet a description of the material and any other appropriate observations or characteristics.
   iv. Write the lab case number, the item number, and the analyst's initials on the top or side of the can.
   v. If materials are present which may require additional forensic analyses (e.g., latent print or DNA), the nature of the materials and the type of testing should be considered before heating.

b) Recovery and Analysis of organic compounds (use one or more of the steps below)

- Analyze a negative control (e.g., blank air, extraction solvent, or a can treated in the same manner as evidence) to ensure that the procedure and/or instrument is contamination-free.
- Analyze blanks (e.g., blank air, appropriate solvent) in-between all samples to ensure

that there is no significant carryover. If a sample is analyzed and the result is negative for accelerant-related compounds, then a blank analysis is not required before the next sample is analyzed.

i. Heated headspace
   A. Punch a hole in the lid of the submitted can.
   B. Cover the hole with a piece of plastic adhesive tape.
   C. Heat the can until a bulge appears under the tape.
   D. Remove the can from the heat source.
   E. Using an appropriate disposable 3.0 mL syringe, inject 0.5 mL (0.5 cc) of headspace into the GC/MS

ii. Adsorption/elution (Dynamic)
   A. Check the outside of the can for dirt or debris. If present, clean the can to ensure that it is contamination-free.
   B. Punch 1-4 evenly spaced holes around the circumference of the can near the bottom. If there is water in the can, place the holes approximately 1.0 inch above the water level.
   C. Replace the original lid with a lid adapted to hold a charcoal tube. Snip the ends of the charcoal tube and place it into the Swagelok fitting. Insert the tube with the 'open' end in the holder.
   D. Insert the can into the heating mantle (the temperature is approximate 80 °C. In some cases it may be necessary to insert a dial thermometer through the lid until it touches the contents of the can (determined by analyst).
   E. Connect the end of the charcoal tube to the vacuum line, and turn on the vacuum pump.
   F. After an appropriate period of time (i.e., when condensation appears in the open area of the tube or when the temperature of the contents reaches 40-50 °C) remove the charcoal tube. CAUTION: Avoid sample temperatures above 80 °C to minimize the stripping of 'lighter' components of flammable liquids.
   G. Insert the 'full' end of the charcoal tube into a small (4.0 mL) glass vial.
   H. Using a Pasteur pipette add approximately 0.3 mL of pentane to the 'open' end of the

charcoal tube.

I. Elute the pentane through the charcoal tube. Repeat the procedure with a second ~0.3 mL of pentane.
J. If the sample extract cannot be analyzed the same day, store the extract in the refrigerator or freezer under proper seal.
K. If a petroleum distillate or gasoline odor was not discernible during initial observations, concentrate the sample by using a stream of nitrogen gas ($N_{2(g)}$) to approximately 100 µL.
L. Inject approximately 1 µL of the extract solution into the GC/MS and analyze. Clean the GC syringe appropriately.
M. Thoroughly clean the bottom of the modified can lid by rinsing it and the interior of the Swagelok fitting with acetone. This removes condensed water and any organics from the lid.

iii. Solvent Extraction

A. Fill a suitable clean beaker half way with all or a portion of the contents of the submitted item and place in the fume hood.
B. Add pentane (the amount needed will vary with the amount of debris that was submitted). Add the solvent slowly, washing the debris in the process. Swirl gently to make sure the solvent comes in contact with all of the debris.
C. Filter the pentane extract solution and collect in a second clean beaker.
D. Concentrate (either by heating or evaporation) until most of the pentane is removed.
E. Transfer to a 4.0 mL glass vial and concentrate as necessary using $N_{2(g)}$.
F. Inject 1.0 µL of extract solution into the GC/MS and analyze.

c) Record appropriate information within either case notes or worksheets

i. Reagents and steps not explicitly stated in this procedure will be recorded within examination documents (e.g., dilutions, alternate extraction solvents, appropriate standards)
ii. Lot numbers of solvents and other appropriate materials will be recorded within examination documents.

6. Sample Preservation

After each sample is analyzed, solvent is added to the sample vial and it is placed into the submitted can. The sample vial should be placed under proper seal prior to being placed into the can. The can is then secured with it's top, sealed with evidence tape, and initialed.

7. References

a) ASTM Standard Methods: Designation E 1387-95 (modified)
b) GC-MS Guide to Ignitable Liquids
c) ASTM Methods: E 1386, E 1388, E 1413

Revision #	Revision History
2	Slight format change. Replaced section with unit. Modified the 'Materials' section. Added a 'Standards and Controls' section specifically requiring controls to be analyzed. Re-worded the 'smell' portion of the procedure. Decreased injection volume in 'Heated Headspace' section from 1 mL to 0.5 mL. Fixed type from 180 °C to 80 °C within 'Absorption/Elution' section. Decreased amount of pentane used during extraction within 'Absorption/Elution' section from 0.5 mL to approximately 0.3 mL. Added requirement to record lot numbers and solvents within examination documents. Added a 'Revised History' section to the document.
3	Revised sections 4c and 5b to reflect that if samples are accelerant-free, the analysis of a blank after that particular sample is not required.