



ATF-LS-FD6 Solvent Extraction	Published Online: March 2018
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I. Scope: This policy and procedure guideline establishes a standard method for separating small quantities of ignitable liquid residues from fire debris samples or aqueous liquids. This method is also used for further characterizing heavy petroleum distillates or for certain evidence requiring fingerprint analysis. This method is best suited for analyzing nonporous matrices; however, porous matrices can also be analyzed using this technique. Co-extraction of interfering compounds may be a concern with some porous matrices using this technique.

II. References:

ASTM E 1386 - Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction

ASTM E 1618 - Guide for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry

ASTM E 752 - Practice for Safety and Health Requirements Relating to Occupational Exposure to Carbon Disulfide

Occupational Health Guidelines for Carbon Disulfide, U.S. Department of Labor and Department of Health, September 1978.

Safety Data Sheet for Carbon Disulfide

Safety Data Sheet for Pentane

"Sample preparation for the chemical analysis of debris in suspect arson cases", W. Bertsch and Q. Zhang, *Analytica Chimica Acta*, 236 (1990) 183-195.

"Methods of fire debris preparation for detection of accelerants", B. Caddy, F. P. Smith, and J. Macy, *Forensic Science Review*, 3 (1), June 1991, 58-69.

III. Apparatus/Reagents:

- A. Solvent – Carbon disulfide (CS₂), pentane or other appropriate solvent.
- B. Filter apparatus – free of extractable hydrocarbons.
- C. Beakers or similar containers

IV. Safety Precautions:

Personal protective equipment including but not limited to safety glasses, gloves, and lab coat should be worn.

Carbon disulfide is a hazardous chemical with respect to both health and fire safety, and should be handled with extreme care. Use of carbon disulfide should be confined to a properly operating ventilation hood. Avoid physical contact with carbon disulfide. Carbon disulfide should be kept from heat, heat sources and sources of ignition.

Pentane should be used in a fume hood and kept away from open flames and sparks.

V. Procedures – Fire Debris

Open and examine the fire debris sample to determine that it is consistent with its description.

1. The sample may be extracted in its original container, or placed in a disposable or new container for extraction.
2. Add sufficient solvent to thoroughly moisten the sample.
3. Mix the solvent and debris thoroughly. Simple rinsing of nonporous surfaces may not adequately separate residues.
4. Filter the solvent if necessary.
5. Evaporate (concentrate) the solvent, if necessary (examiner may monitor by taking sample aliquots for injection into the GC-MS during the evaporation process).
6. Inject sample into GC-MS.

Procedure - Liquids

1. Place a sample of unknown aqueous liquid in a container and add pentane, CS₂, or other appropriate solvent.
2. Mix the liquids then allow the layers to separate.
3. Remove the pentane or CS₂ layer.
4. Filter if necessary.
5. Evaporate (concentrate) the solvent, if necessary (examiner may monitor by taking sample aliquots for injection into the GC-MS during the evaporation process).
6. Analyze by GC-MS.

Concentration – If the sample needs to be concentrated, perform the concentration in containers free of extractable hydrocarbons (disposable or new containers). Place the container in a chemical fume hood. Evaporate at room temperature. Compressed dry nitrogen can be used to accelerate evaporation. Heating should be done with caution to prevent excessive concentration resulting in substantive changes to the chemical profile or loss of sample due to complete evaporation or boiling over. Heating mantles are not recommended, however, a steam bath may be used to accelerate evaporation.

VI. Quality Control:

1. The materials control will be prepared the same way as the sample(s). If a liquid is diluted in a solvent or extracted using a simple liquid-liquid extraction technique, the solvent blank prior to the sample may serve as the materials control. Additionally, if the sample is filtered or concentrated, the materials control shall be treated the same way.
2. A copy of the materials control will be maintained in the appropriate case jacket.

Extract Storage - All fire debris extracts will be preserved with charcoal and returned with evidence to the submitting agency. If the sample is a liquid that did not burn or if a portion of the original liquid will be returned, it may not be necessary to preserve the extract or a sample of the liquid with charcoal.