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| ATF-LS-FD5 Dynamic Charcoal Adsorption | Published Online: March 2018 |
| Authority: Technical Leader | |
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I. **Scope:** This policy and procedure guideline establishes a standard procedure for extracting fire debris evidence by the dynamic headspace concentration method (dynamic charcoal adsorption). This method may not recover hydrocarbons with a boiling point above heptadecane (C₁₇) and therefore may not be able to fully recover some heavy petroleum distillates. If appropriate, Solvent Extraction (FD-6) may also be used for the separation and concentration of ignitable liquid residues from fire debris samples.

II. **References:**

ASTM E 1413 Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration

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

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

Material Safety Data Sheet for Carbon Disulfide

Chrostowski, J.E. and R.M. Holmes: Collection and Determination of Accelerant Vapors from Arson Debris, Arson Analysis Newsletter, 3, No. 5, 1 (1979).

Levadie, G.J. and S. MacAskill: Analysis of Organic Solvents Taken On Charcoal Tube Samplers by a Simplified Technique. Analytical Chemistry, 48, 76 (1976).

"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. **Charcoal (Inlet/Outlet) Tubes**

1. Disposable Pasteur pipets approximately 5mm in diameter

2. Approximately 1cm of glass wool or cotton for insertion into the bottom of the pipet
3. Approximately 1-2 cm of charcoal (50-200 mesh activated coconut)
4. 1 cm of wool or cotton to place on top of the charcoal

B. Negative Pressure Apparatus

1. Vacuum system
2. Rubber sleeve stopper
3. Heating mantel
4. Thermometer capable of measuring temperatures up to 150° C
5. Carbon Disulfide

IV. Safety Precautions:

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

V. Procedures:

Open and examine the fire debris sample to determine that it is consistent with its description. If the sample is not in a metal can, transfer the debris to an appropriate sized can, or use the passive charcoal strip method.

1. Punch three holes in the lid of the can.
2. Cover each hole with a rubber sleeve or similar device.
3. Insert a charcoal tube into two of the covered holes.
4. Place a thermometer into the third hole.
5. Attach a vacuum system to one of the adsorption tubes.
6. Place the can into a heating mantel.
7. Turn on vacuum.
8. While maintaining vacuum, heat the can to 90°C. Hold temperature for 5 minutes.
9. Turn off vacuum and remove adsorption tube.
10. Allow adsorption tube to cool.

Elution Procedure

1. Slowly introduce or flush enough carbon disulfide to thoroughly wet the charcoal in the tube (approximately 500 μ L). An aspirator bulb may be necessary in order to flush the solvent through the charcoal.
2. Alternately, the charcoal may be removed from the tube and agitated in a container with carbon disulfide.

VI. Quality Control:

Prior to a new batch/lot number of activated charcoal being implemented for casework it will be tested to determine that it is free of contaminants and that it qualitatively performs as expected for passive headspace analyses. Two metal cans will be prepared using the above procedure. One can will contain 5 – 10 mL of a 50:50 gasoline:diesel mixture on a Kimwipe (or equivalent). The other can will contain a Kimwipe (or equivalent). Each can shall then be extracted as per the above procedure. Both gasoline and the heavy petroleum distillate shall be identifiable as per ATF-LS-FD8 in the positive (spiked) can. The results of these tests will be noted in a log and the data will be maintained with the log.

A system blank will be run each day that this method is used. A system blank consists of a prepared charcoal sampling tube inserted into a clean, unused paint can and sampled as outlined above. Elute with carbon disulfide in the same manner as for the samples. A copy of the system blank data will be placed in the appropriate case jacket.

Extract storage - All carbon disulfide extracts will be preserved with charcoal and returned to submitting agent.

Thermometer Performance Check: The thermometers used for this procedure shall be checked on an annual basis using a NIST certified thermometer.