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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration¹

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1. Scope

1.1 This practice covers the procedure for removing small quantities of ignitable liquid residue from samples of fire debris using an adsorbent material to extract the residue from the static headspace above the sample, then eluting the adsorbent with a solvent.

1.2 While this practice is suitable for successfully extracting ignitable liquid residues over the entire range of concentration, the headspace concentration methods are best used when a high level of sensitivity is required due to a very low concentration of ignitable liquid residues in the sample.

1.2.1 Unlike other methods of separation and concentration, this practice is essentially nondestructive.

1.3 Alternate separation and concentration procedures are listed in the referenced documents. (see Practices).

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 752 Practice for Safety and Health Requirements Relating to Occupational Exposure to Carbon Disulfide³

E 1385 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Steam Distillation⁴

E 1386 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction⁴

E 1387 Test Method for Ignitable Liquid Residues in Ex-

tracts from Samples of Fire Debris by Gas Chromatography⁴

E 1413 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration⁴

E 1459 Guide for Physical Evidence Labeling and Related Documentation⁴

E 1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory⁴

E 1618 Guide for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry⁴

3. Summary of Practice

3.1 Charcoal in some form of an adsorption package is placed in the sample container to adsorb ignitable liquid residues. The container may be heated or left at ambient temperature. The charcoal is removed and eluted with CS₂ or diethyl ether.

4. Significance and Use

4.1 This practice is useful for preparing extracts from fire debris for later analysis by gas chromatography, GC/MS, or GC/IR.

4.2 This is a very sensitive separation procedure, capable of isolating quantities smaller than 1/10 µL of ignitable liquid residue from a sample.

5. Apparatus

5.1 *Heating System*—An oven, or a heating mantle to fit the evidence container (or a hot plate).

5.2 *Temperature Measuring Device*—A thermometer or thermocouple capable of measuring temperatures in the range of 40 to 150°C.

5.3 Adsorption Package.

5.3.1 Commercial charcoal adsorption packages are available from several companies. These packages, in the form of polymer strips or small charcoal canisters or “C bags,” are used to adsorb organic vapors.

5.3.2 Non-Commercial Adsorption Packages.

5.3.2.1 *Coated Wire*—Flatten a piece of copper wire, 22 ga. by 4 cm on one end for 5 mm. Dip the flattened end in water

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² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 11.03.

⁴ Annual Book of ASTM Standards, Vol 13.01.

glass (sodium silicate) and then into activated charcoal (50 to 200 mesh, or equivalent) and dry in an oven at 70°C.^{5,6}

5.3.2.2 *Coated Beads*—Coat plexiglass beads, $\frac{3}{8}$ in. diameter, with 50 to 200 mesh charcoal, or equivalent, by placing a bead and small amount of charcoal in a 2 mL flat bottomed disposable beaker and agitating in an amalgamator for 30 s.⁷

5.3.2.3 *C-Bags*—Prepare C-bags by encapsulating 0.2 g of activated charcoal within a folded sheet of high strength, light weight, high porosity tissue paper, such as that commonly used for making tea bags.⁸

5.3.3 *Storage of Adsorption Packages*—To prevent contamination, store all adsorption packages away from any sources of organic vapors prior to and after use for sampling.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁹ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.1.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

6.2 Copper Wire Adsorption:

6.2.1 *Activated Charcoal*, 50 to 200 mesh, or equivalent.

6.2.2 *22 Gage Copper Wire*.

6.2.3 *Water Glass* (sodium silicate).

6.3 Coated Beads:

6.3.1 *Activated Charcoal*, 50 to 200 mesh, or equivalent.

6.3.2 *Plexiglass Poly(Methyl Methacrylate) Beads*, $\frac{3}{8}$ in. diameter.

6.3.3 2 mL flat bottomed disposable beaker.

6.4 C-Bags:

6.4.1 4 by 5 in. (approximate) high strength, light weight, high porosity filter paper.

6.4.2 *Activated Charcoal*.

6.5 Elution Solvent:

6.5.1 *Carbon Disulfide*, GC grade.

6.5.1.1 Read and follow the safety precautions described in Practice E 752.

6.5.2 *Diethyl Ether*, GC or HPLC grade.

7. Sample Preparation/Adsorption Procedure

7.1 Observe the appropriate procedures for handling and documentation of all submitted samples as described in Practice E 1492.

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

7.1.1.1 Resolve any discrepancies between the submitting agent's description of the evidence and the analyst's observation with the submitting agent prior to the completion of the report.

7.2 Remove an adsorbent package from its sealed packaging and place it in the evidence container, then reseal the container.

7.3 Allow the evidence container to sit overnight. Alternatively, heat the container to a temperature of 50 to 80°C, for a minimum of two h. Longer times are required for the adsorption of higher boiling point compounds or for the adsorption of very small quantities of volatile hydrocarbons.

7.3.1 The optimum adsorption time for maximum sensitivity will depend on the adsorption package and temperature selected.

7.3.2 If the ignitable liquid residue is present at sufficiently high concentration, selective adsorption of higher molecular weight components may occur under high temperature conditions or longer adsorption times.

7.4 0.1 μ L or 0.5 μ L of an internal standard may be added to the sample in order to check on the efficiency of the procedure. Internal standards are typically prepared using a single compound that is easily identified (such as 3-phenyltoluene or diphenylmethane) dissolved in the eluting solvent.

8. Elution Procedure

8.1 Remove the adsorption package from the evidence container. If it is not to be eluted immediately, store the adsorption package in a clean, vapor-tight container.

8.2 Code or label an appropriately sized vial or test tube with the case number, sample number, date, separation method, and analyst's initials.

8.2.1 Follow the evidence documentation and handling procedures described in Guide E 1459 and Practice E 1492.

8.3 Place the adsorption substrate in a properly labeled container and rinse with a minimal amount of eluting solvent.

9. Sealing

9.1 A septum seal vial may be used for collecting and sealing the extract.

9.1.1 A test tube containing CS₂ may be sealed by the addition of water which will float on top of the CS₂ and reduce its evaporation.

10. Extract and Adsorbent Storage

10.1 After analysis, place the eluate back in the vial or test tube with the adsorbent package. Hydrocarbons present in the eluate will be readsorbed onto the charcoal and should remain stable indefinitely.

11. Blanks and Standards

11.1 Run frequent blanks on each lot of adsorbent packages.

⁵ Twibell, J. D., and Home, J. M., "Novel Methods for Direct Analysis of Hydrocarbons in Crime Investigation and Air Pollution Studies," *Nature*, Vol 268, 1977, pp. 711–713.

⁶ Trantham-Fryer, D. J., "The Application of a Simple and Inexpensive Modified Carbon Wire Adsorption/Solvent Extraction Technique to the Analysis of Accelerants and Volatile Organic Compounds in Arson Debris," *Journal of Forensic Sciences*, Vol 35, 1990, pp. 271–280.

⁷ Juhala, J. A., "A Method for Adsorption of Flammable Vapors by Direct Insertion of Activated Charcoal into the Debris Samples," *Arson Analysis Newsletter*, Vol 6, 1982, pp. 32–36.

⁸ Dietz, W. R., "Improved Charcoal Packaging for Accelerant Recovery by Passive Diffusion," *Journal of Forensic Sciences*, Vol 35, 1991, pp. 111–121.

⁹ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Anal. Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.