



Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal¹

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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 E 1385, E 1386, E 1388, and E 1413).

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:*²

D 1193 Specification for Reagent Water

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

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

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

E 1387 Test Method for Ignitable Liquid Residues in Extracts from Samples of Fire Debris by Gas Chromatography

E 1388 Practice for Sampling of Headspace Vapors from Fire Debris Samples

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

E 1459 Guide for Physical Evidence Labeling and Related Documentation

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

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

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 a suitable elution solvent as listed in Section 6.3.

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).

5.1.1 An oven is recommended to achieve a constant temperature throughout the system.

¹ This practice is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics. Current edition approved April 15, 2007. Published June 2007. Originally approved in 1991. Last previous edition approved in 2005 as E 1412 – 05.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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.

5.3.1.1 The minimum recommended polymer strip size is 10 mm by 10 mm, or 100 mm².

5.3.2 Non-Commercial Adsorption Packages.

5.3.2.1 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.³

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.⁴ 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 C-Bags:

6.2.1 4 by 5 in. (approximate) high strength, light weight, high porosity filter paper.

6.2.2 Activated Charcoal.

³ Dietz, W. R., “Improved Charcoal Packaging for Accelerant Recovery by Passive Diffusion,” *Journal of Forensic Sciences*, Vol 35, 1991, pp. 111–121. (Unk)

⁴ *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 *Analar 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.

6.3 Elution Solvent—Suitable elution solvents are carbon disulfide, *n*-pentane, or diethyl ether. Use of a heavier solvent, such as toluene or tetrachloroethylene, is sometimes necessary when the compounds of interest have low molecular weights.

6.3.1 Check solvent purity by evaporating to at least twice the extent used in the analysis and analyzing the evaporated solvent in accordance with Test Method E 1387 or Test Method E 1618.

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

6.3.3 Diethyl Ether, GC or HPLC grade.

6.3.3.1 Pentane and diethyl ether produce relatively large signals when passing through a flame ionization detector, possibly obscuring the signal from lighter hydrocarbons.

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 Heat the container to a temperature of 50 to 80°C, for 2 to 24 h. The longer times or higher temperatures, or both, are required for the adsorption of higher boiling point compounds or for the adsorption of very small quantities of volatile hydrocarbons. The adsorption temperature and duration may vary based on the sample.^{5, 3}

7.3.1 When other evidentiary considerations arise (such as document or latent print examinations) it may be appropriate to conduct the adsorption at ambient temperature (approximately 20°C) for extended periods (24 h or longer) to minimize damage.

7.3.2 Room temperature adsorption may also be appropriate to detect low molecular weight compounds.

⁵ Newman, R, et.al., “The Use of Activated Charcoal Strips for Fire Debris Extractions by Passive Diffusion. Part 1: The Effects of Time, Temperature, Strip Size and Sample Concentration,” *Journal of Forensic Sciences*, Vol 41, 1996, pp. 351-370.

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7.3.3 The optimum adsorption time for maximum sensitivity will depend on the adsorption package and temperature selected. Temperatures lower than 60°C may be insufficient to volatilize compounds heavier than C16.

7.3.4 Temperatures in excess of 80°C may result in disproportionate recovery of higher molecular weight compounds with the displacement of lower molecular weight compounds.

7.3.5 The optimum adsorption time for representative sampling or maximum sensitivity, or both, will depend on the adsorption package, the adsorption temperature, and the ignitable liquid composition and concentration.

7.3.5.1 Adsorption times for routine screening of samples are typically in the range of 8 to 24 h.

7.3.5.2 Data that appears overloaded or excessively displaced may be corrected by resampling at ambient temperatures, or with shortened adsorption times (1 to 4 h).

7.4 0.1 µL or 0.5 µL 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.

7.5 Document the adsorption parameters including the type and amount of adsorbent used, adsorption temperature and adsorption time.

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 required for instrumental analysis (typically 50 to 1000 µL).

8.4 Document the type and volume of solvent used to elute the adsorbent.

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, the eluate may be placed back in the vial or test tube with the adsorbent package. Hydrocarbons present in the eluate will be readсорbed onto the charcoal.

10.2 Alternately, a portion of the adsorbed charcoal may be separated and stored prior to elution and analysis of the remaining adsorbent.

11. Blanks and Standards

11.1 Run frequent blanks on each lot of adsorbent packages.

11.1.1 Prepare a blank by placing an adsorbent package into a clean dry evidence container and running the adsorption procedure as described in Section 7. Elute the adsorption package according to Section 8, and analyze the extract according to Test Method E 1387 or Guide E 1618.

11.1.1.1 If an internal standard is routinely used, include an internal standard in the blank.

11.2 When necessary, charcoal may be activated and cleaned by heating in a 400°C oven for 4 h, then cooling in a dessicator.

11.3 Periodically check the adsorption efficiency by running this procedure on a sample containing a known volume of standard ignitable liquid.

12. Keywords

12.1 fire debris samples; passive headspace concentration

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