Designation: E1412 – 16

Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal[1]

This standard is issued under the fixed designation E1412; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes the procedure for separation of small quantities of ignitable liquid residues 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 E1386, E1388, E1413, and E2154).

1.4 This practice does not replace knowledge, skill, ability, experience, education, or training and should be used in conjunction with professional judgment.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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]

E1386 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction

E1388 Practice for Sampling of Headspace Vapors from Fire Debris Samples

E1413 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration

E1459 Guide for Physical Evidence Labeling and Related Documentation

E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory

E1618 Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry

E2154 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Solid Phase Microextraction (SPME)

E2451 Practice for Preserving Ignitable Liquids and Ignitable Liquid Residue Extracts from Fire Debris Samples

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 6.3.

4. Significance and Use

4.1 This practice is useful for preparing extracts from fire debris for later analysis by gas chromatography mass spectrometry.

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.

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[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 100°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 mm^2.

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 sampling.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade or better 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.

6.2 *Adsorption Package:*

6.2.1 *C-Bags:*

6.2.1.1 4 by 5 in. (approximate) high strength, light weight, high porosity filter paper.

6.2.1.2 Activated charcoal.

6.2.2 Commercial charcoal adsorption package.

6.2.3 Check charcoal media purity by, at a minimum, desorbing a representative unit using the same elution solvent as will be used for questioned samples and analyzing in accordance with Test Method E1618.

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 E1618.

6.3.2 Read and follow the safety precautions described in the safety data sheet (SDS) of the extraction solvent that is used.

7. Sample Preparation/Adsorption Procedure

7.1 Observe the appropriate procedures for handling and documentation of all submitted samples as described in Practice E1492.

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 agency's description of the evidence and the analyst's observation prior to the completion of the report.

7.2 Place an adsorbent package in the evidence container according to laboratory protocols and reseal the container. Suspend the adsorbent package above the sample whenever possible.

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.[3, 5]

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.

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), or by performing a solvent extract on a portion of the debris in accordance with Practice E1386.

7.4 A known amount (typically, 0.1 µL to 0.5 µL) of an internal standard may be added to the sample in order to evaluate the efficiency of the procedure.

7.4.1 Internal standards are typically prepared using a single compound that is easily identified (such as 3-phenyltoluene or diphenylmethane) dissolved in the eluting solvent.

7.4.2 If an internal standard is added, the compound and the quantity added shall be documented along with the manufacturer, grade, and lot number used.

[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.

[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.

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 Follow the evidence documentation and handling procedures described in Guide E1459 and Practice E1492.

8.3 Place the adsorption substrate in a properly labeled container and desorb with a minimal amount of eluting solvent required for instrumental analysis (typically 50 to 1000 µL).

8.4 Document the type and approximate volume of solvent used to desorb the analyte(s) from the adsorbent.

9. Sealing

9.1 A septum seal or screw cap glass vial may be used for collecting and sealing the extract for analysis.

10. Extract and Adsorbent Storage

10.1 Refer to Practice E2451 for short term and long term storage of ignitable liquid extracts.

11. Blanks and Standards

11.1 Run frequent blanks on each lot of adsorbent packages.

11.1.1 Analyze an absorbent package blank by eluting the adsorbent with 50 to 1000 µL of solvent and analyze according to Test Method E1618.

11.1.2 Prepare a system blank by placing an adsorbent package into a clean dry evidence container and running the adsorbent procedure as described in Section 7. Elute the adsorption package according to Section 8, and analyze the extract according to Test Method E1618.

11.1.2.1 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 desiccator.

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 activated charcoal; fire debris samples; passive headspace concentration