



Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration¹

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1. Scope

1.1 This practice describes the procedure for separation of small quantities of ignitable liquid residues from fire debris samples using the absorption/elution method of headspace concentration.

1.2 Both positive and negative pressure systems are described.

1.3 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.

1.4 Alternate separation and concentration procedures are listed in Section 2.

1.5 *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 Regulations Relating to Occupational Exposure to Carbon Disulfide³

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

E 1412 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal

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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.

³ Withdrawn.

3. Summary of Practice

3.1 The sample, preferably in its original container, is heated, forcing volatile compounds to vaporize. At the same time, the headspace in the sample is drawn or pushed through a tube containing activated charcoal which adsorbs the vaporized compounds.

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 0.1 µL of ignitable liquid residue from a sample.

4.2.1 Actual recovery will vary, depending on several factors, including adsorption temperature, container size, and competition from the sample matrix.

4.3 “This is a potentially destructive technique.” Portions of the sample subjected to this procedure may not be suitable for re-sampling. Therefore, a portion of the sample extract should be saved for potential future analysis. Consider using passive headspace concentration as described in Practice **E 1412**.

5. Apparatus

5.1 Positive Pressure Apparatus:

5.1.1 *Sample Pressurization Device*—A system capable of delivering pressurized dry nitrogen (or other inert gas) at up to 40 psi (276 kPa) to an orifice to be inserted into the bottom of the sample container.

5.1.1.1 A needle valve capable of fine control of the flow rate of the dry nitrogen, at up to 1500 cc/min.

5.1.1.2 A flow meter capable of measuring the flow of dry nitrogen through the end of the charcoal adsorption tube at the rate of 0 to 1500 cc/min.

5.1.1.3 *Container Closure*—A device suitable for sealing the container and directing the effluent nitrogen and vapors to the charcoal tube.

5.2 Negative Pressure Apparatus:

5.2.1 *Inlet and outlet system*—A tube containing 1 cm of charcoal is fitted into the lid of the original container with a

sleeve stopper. This serves as a filter for incoming room air. A vacuum is pulled on an adsorption tube also fitted into the lid of the original container with a sleeve stopper.

5.2.2 A vacuum system capable of pulling between 200 and 1500 cc/min on the sample collection tube.

5.2.3 A flow meter capable of measuring the flow of air through the end of the charcoal tube at the rate of 200 to 1500 cc/min.

5.3 *Adsorption Tubes*—Suitable absorption tubes may be made by inserting a small (approximately 1 cm) plug of glass wool or cotton in the bottom of a Pasteur pipette (approximately 5 mm diameter), then adding approximately 5 cm of activated charcoal, and finally, holding the charcoal in place with an additional 1 cm of glass wool or cotton.

5.3.1 Alternatively, charcoal tubes are available from commercial sources.

5.3.2 The use of a different adsorbing media such as Tenax is possible, but this results in complications in the desorbing phase because certain solvents cause the dissolution of the Tenax. Tenax is best employed when desorption is to be performed using a thermal operation.

5.4 *Heating System*—A heating mantel designed to fit the evidence container or an oven or a hot plate.

5.4.1 An oven may be set up with any number of stations to allow for multiple sample preparation.

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

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.

6.1.1 *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 *Activated Charcoal* (coconut).

6.2.1 Test each new batch of charcoal for residual hydrocarbons by analyzing a concentrated extract of blank adsorption media.

6.3 *Glass Wool*, or cotton, free of extractable hydrocarbons.

6.4 *Carbon Disulfide*, GC grade.

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

7. Sample Preparation

7.1 Observe the appropriate procedures for handling and documentation of all submitted samples.

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 Place the sample in an appropriate, clean sampling container, which is designed to be flushed by positive or negative pressure.

7.2.1 Ideally, the system will be designed so that the majority of samples can be extracted directly from the container in which they are delivered to the laboratory.

8. Adsorption Procedure

8.1 Positive Pressure:

8.1.1 Place the sample container in the heating system and connect to the nitrogen source which will introduce nitrogen into the bottom of the container, and to the exhaust line connected to the charcoal adsorbing tube.

8.1.1.1 Direct connection of the charcoal tube to the sample container is possible, unless an oven is used. Placement of the charcoal tube outside the oven requires the use of an intervening line which must be carefully cleaned between uses to remove any adsorbed or condensed residues.

8.1.2 *Pressurization*—Deliver nitrogen to the sample through a 1/8 in. (3.2 mm) line on the inlet side of the container at a pressure of 40 psig (276 kPa) upstream of the needle valve controlling the flow rate. Connect a 1/4 in. (6.35 mm) line on the downstream side of the container to the charcoal tube. Attach the flow meter to the outlet side of the charcoal tube, and use the needle valve to adjust the flow to the rate determined to be optimum for this system.

8.1.2.1 Determine the optimum flow rate by placing known quantities of gasoline, kerosene, mineral spirits, and diesel fuel onto standard substrate material such as squares of carpeting or filter paper. Determine the percent recovery by gas chromatographic analysis. The flow rate resulting in the highest percent recovery is the optimum.

8.1.2.2 Remove the flow meter once the flow is adjusted. Check the flow at least once after the sample has reached the final temperature.

8.1.3 Turn on the heating system and increase the temperature to no more than 150°C. At this temperature, most petroleum distillates can be volatilized in less than one hour, and there will be a minimum of pyrolysis of common polymers.

8.1.3.1 Allow 20 min for the sample to reach temperature, and an additional one hour flushing the sample out. At this point, turn off the gas. The charcoal adsorption tube is now ready for elution.

8.1.3.2 The adsorption time may be reduced if it can be shown that equal or superior recovery rates are achieved in the reduced time period.

8.2 Negative Pressure:

8.2.1 Place the inlet and outlet adsorption tubes in the lid of the evidence container.

8.2.2 Place the temperature measuring device in the lid of the container.

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.

8.2.3 Apply a vacuum to the sample adsorption tube. The flow through the tube should be between 200 and 1500 cc/min.

8.2.4 Heat the evidence container to 90°C and hold at that temperature for 5 min. After 5 min at 90°C, turn off the vacuum, and the sample adsorption tube is ready to be eluted.

9. Elution Procedure

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

9.2 Remove the charcoal tube from the sample outlet line and insert into the properly labeled test tube. Allow to cool to room temperature. Trickle one mL of elution solvent through the charcoal tube and into the test tube. An aspirator bulb may be necessary in order to push the solvent through the charcoal.

9.2.1 Alternatively, the charcoal may be removed from the tube and agitated in a container with elution solvent.

9.2.2 Any method of elution may be used if it can be shown that adsorbed residues are effectively eluted.

10. Sealing

10.1 A septum seal vial may be used for collecting and sealing the elution solvent extract.

10.2 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.

10.3 Preserve the extract so that it may be re-analyzed. Guidance on extract preservation may be found in Test Method **E 1387**.

11. Blanks and Standards

11.1 Frequent blanks must be run on every apparatus used for this procedure.

11.1.1 Prepare a blank by connecting an empty container to the adsorption apparatus, and running the adsorption procedure described in Section 8 for the normal length of time. Elute the charcoal tube according to Section 9, and analyze the extract according to Test Method **E 1387**.

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

11.2 Charcoal may be activated and cleaned by heating in a 400°C oven for 4 h and cooling in a desiccator.

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

12. Keywords

12.1 dynamic headspace concentration; fire debris samples

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