Designation: E1413 – 13

Standard Practice for
Separation of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration[1]

This standard is issued under the fixed designation E1413; 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 fire debris samples using the method of dynamic headspace concentration.

1.2 Dynamic headspace concentration uses adsorption and subsequent solvent elution or thermal desorption.

1.3 Both positive and negative pressure systems for adsorption are described, as well as a thermal desorption system.

1.4 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.

1.5 Alternate separation and concentration procedures are listed in Section 2. If archival of the extract is of importance, then this practice's thermal desorption procedure, SPME (Practice E2154), and headspace (Practice E1388) sample separation techniques are not recommended unless a portion of the extract can be split and retained. In order to have an archivable extract, then this practice's sample collection on charcoal, solvent extraction (Practice E1386), or passive headspace concentration (Practice E1412) is recommended.

1.6 This practice offers a set of instructions for performing one or more specific operations. This standard cannot replace knowledge, skill, or ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgment.

1.7 *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

E1412 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal

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 The sample, preferably in its original container, is heated, forcing volatile compounds to vaporize. The headspace in the sample is then drawn or pushed through a tube containing an adsorption media (typically activated charcoal or Tenax) which adsorbs the vaporized compounds.

3.2 Other solid adsorbents and collection systems can be used as long as the method has been validated with a wide variety of ignitable liquids with a range of both flash points and polarity.

4. Significance and Use

4.1 This practice is useful for preparing extracts from fire debris for later analysis by gas chromatography-mass spectrometry (GC-MS), see Test Method E1618.

---

[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 Aug. 15, 2013. Published September 2013. Originally approved in 1991. Last previous edition approved in 2007 as E1413 – 07. DOI: 10.1520/E1413-13.

[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.

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 E1412.

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 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 (or other inert gas) and vapors to the adsorption tube.

5.2 *Negative Pressure Apparatus:*

5.2.1 *Inlet and Outlet System*—A tube containing approximately 1 cm of charcoal is fitted into the lid of the original container with a suitable penetrable seal. 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 suitable penetrable seal.

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 *Thermal Desorption Apparatus:*

5.3.1 *Sampling System*—An air-tight syringe is connected to the back-end of a Tenax tube. The front end of the Tenax tube is fitted into the heated container.

5.3.2 In order to prevent leakage and contamination, the system should be designed to minimize loss of vapors from the container. For example by means of a septum mounted on top of the lid of the container.

5.3.3 *Thermal Desorption Device*—A system capable of desorbing the volatiles by means of elevated temperature and trapping the volatiles in a cold-trap. This apparatus is directly coupled to a GC-MS.

5.4 *Adsorption Tubes:*

5.4.1 *Charcoal Tubes*—Suitable charcoal filter and sample adsorption 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 2.5 to 5 cm of activated charcoal, and finally, holding the charcoal in place with an additional plug of glass wool or cotton.

5.4.1.1 Alternatively, charcoal tubes are available from commercial sources.

5.4.2 *Tenax Tubes*—Suitable Tenax tubes are commercially available. Note that solvent extraction of these tubes results in complications in the desorbing phase because certain solvents cause the dissolution of the Tenax. Tenax should be employed when thermal desorption is to be performed.

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

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

5.6 *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.[3] 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 *Solid Adsorbent:*

6.2.1 *Activated Charcoal* (coconut).

6.2.1.1 Charcoal may be activated and cleaned by heating in a 400°C oven for approximately 4 hours and cooling in a desiccator.

6.2.1.2 Test each new or reconditioned batch of charcoal for residual hydrocarbons by analyzing a concentrated extract of blank adsorption media according to Test Method E1618.

6.2.2 *Tenax:*

6.2.2.1 Tenax may be cleaned and conditioned according to the manufacturer's instructions.

6.2.2.2 Test each new or reconditioned batch of Tenax for residual hydrocarbons by analyzing a concentrated extract of blank adsorption media according to Test Method E1618.

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

6.4 *Extraction solvent* (for example, carbon disulfide, pentane, ethyl ether).

6.4.1 Read and follow the safety precautions described in the Safety Data Sheets (SDS) of the extraction solvent that is used.

6.4.2 Test each new lot number of the extraction solvent by analyzing a concentrated portion according to Test Method E1618.

---

[3] *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.

6.5 Pasteur pipettes and glass vials free of extractable hydrocarbons.

7. Sample Preparation

7.1 Observe the appropriate procedures for handling and documentation of all submitted samples (Guide E1459 and Practice E1492).

7.1.1 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 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 E1412 which is essentially nondestructive.

7.3 The system should be designed so that the majority of the samples can be extracted from the container in which they are delivered to the laboratory.

7.3.1 Alternatively, the sample or a portion of the sample can be placed in an appropriate, clean sampling container which is designed to be flushed by positive or negative pressure.

8. Adsorption Procedure

8.1 A laboratory typically has one method of adsorption and elution that is employed. The most common combinations in use are charcoal adsorption followed by solvent elution, and Tenax adsorption followed by thermal desorption. Both adsorbents can be collected using either a positive or a negative pressure system. Other solid adsorbents and collection systems can be used as long as the method has been validated with a wide variety of ignitable liquids with a range of both flash points and polarity.

8.1.1 The volume of air sampled is generally less when thermal desorption is employed as the entire collected sample is commonly injected into the GC-MS system for analysis.

8.1.2 With thermal desorption there is typically no material that can be archived once GC-MS analysis has been performed. If archival of a portion of the sample extract is needed, then a different adsorbent and elution combination should be chosen either following this practice, or following one of the other sample collection methods for fire debris analysis (Practices E1386 or E1412). Some instrument manufacturers now have the facility to collect the unused sample from the split; if your instrument has such capability, then there will be material available for archival when thermal desorption is used.

8.2 *Positive Pressure:*

8.2.1 Place the sample container in the heating system and connect to the inert gas source (commonly nitrogen) which will introduce the gas into the bottom of the container, then connect the exhaust line to the adsorbing tube.

8.2.1.1 Direct connection of the adsorption tube to the sample container is possible, unless an oven is used. Placement of the adsorption 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.

8.2.2 *Pressurization*—Deliver the inert gas to the sample through a ⅛ 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 ¼ 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.

8.2.2.1 Determine the optimum system conditions (flow rate, temperature and time) by placing known quantities of gasoline, kerosene, mineral spirits, and diesel fuel onto exemplar substrate material such as squares of carpeting or filter paper. Determine the percent recovery by gas chromatographic analysis. The conditions resulting in the highest percent recovery is the optimum.

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

8.2.3 Place the temperature measuring device in an appropriate location for the system used in order to monitor the temperature of the sample. 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.

8.2.3.1 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.

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

8.3 *Negative Pressure:*

8.3.1 Place the inlet air filtering adsorption tube and the outlet adsorption tubes in the lid of the evidence container.

8.3.2 Place the temperature measuring device in an appropriate location for the system used in order to monitor the temperature of the sample.

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

8.3.4 Determine the optimum system conditions (flow rate, temperature and time) for this system by placing known quantities of gasoline, kerosene, mineral spirits, and diesel fuel onto exemplar substrate material such as squares of carpeting or filter paper. Determine the percent recovery by gas chromatographic analysis. The conditions resulting in the highest percent recovery is the optimum.

8.3.4.1 An example of system conditions could be to heat the evidence container to 90 to 95°C and hold at that temperature for 15 to 25 minutes with the flow at 500 cc/min. Longer times may be necessary for heavily loaded cans. Turn off the vacuum, and the sample adsorption tube is ready to be desorbed.

8.4 *Thermal Desorption:*

8.4.1 Heat the sample to the optimum temperature for your system.

8.4.2 Place the temperature measuring device in an appropriate location for the system used in order to monitor the temperature of the sample.

8.4.3 Remove the caps from the Tenax tube.

8.4.4 Connect the syringe to the back-end of the Tenax tube. Connect the front-end of the Tenax tube to the heated container.

8.4.5 Pull the plunger of the syringe and sample the headspace vapors onto the Tenax adsorption tube. Remove the Tenax system from the container and close the container. Cap the Tenax tube. The Tenax tube is ready for thermal desorption.

8.4.6 Determine the optimum system conditions (temperature, time and sample size) for this system by placing known quantities of gasoline, kerosene, mineral spirits, and diesel fuel onto exemplar substrate material such as squares of carpeting or filter paper. Determine the percent recovery by gas chromatographic analysis. The temperature-volume combination resulting in the highest percent recovery is the optimum.

9. Desorption Procedure

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

9.2 *Desorption:*

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

9.2.2 *Charcoal Tube (Solvent Elution)*—Remove the charcoal tube (or other solid adsorbent) from the sample outlet line and insert into the properly labeled test tube. Allow to cool to room temperature. Trickle approximately 1 mL of elution solvent into the charcoal tube. If possible, allow the solvent to remain in the charcoal for approximately 5 minutes. An aspirator bulb may be necessary in order to push the solvent through the charcoal. Collect the solvent into a GC vial or clean test tube.

9.2.2.1 Concentration of the solvent extract can be performed using a stream of gas (such as hydrocarbon free air or nitrogen) over the solvent.

9.2.2.2 Alternatively, the charcoal (or other solid adsorbent) may be removed from the tube and agitated in a container with elution solvent.

9.2.3 *Tenax Tube (Thermal Desorption)*—The thermal desorption device coupled to a GC-MS is used for the desorption and direct analysis of the sampled volatiles. The Tenax tube is heated and the sample is collected in a cold-trap. The collected sample is directly injected in the GC-MS.

10. Sealing

10.1 A septum seal vial or screw cap glass vial with PTFE-lined seals may be used for collecting and sealing the elution solvent extract.

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

11. Blanks and Standards

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

11.1.1 Analyze an absorbent package blank by desorbing a unit according to the same method employed with samples and analyze according to Test Method E1618.

11.1.2 Prepare a system 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. Desorb the adsorbent tube according to Section 9 (including concentration, if used), 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 *Cleaning and Regeneration:*

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

11.2.2 Tenax may be cleaned and conditioned according to the manufacturer's instructions.

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 activated charcoal; dynamic headspace concentration; elution; fire debris samples; forensic science; ignitable liquid residues; Tenax; thermal desorption