



Designation: **E1413—07 E1413 – 13**

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

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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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 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 ~~are described~~ 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 archival 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

~~D1193~~[E1386 Specification for Reagent Water](#)[Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction](#)

~~E752~~[E1388 Practice for Safety and Health Requirements Relating to Occupational Exposure to Carbon Disulfide](#)[Sampling of Headspace Vapors from Fire Debris Samples](#) (Withdrawn 1995)

[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](#)

~~E1387~~[E1618 Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography](#)[Chromatography-Mass Spectrometry](#) (Withdrawn 2010)

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; August 15, 2013. Published June 2007; September 2013. Originally approved in 1991. Last previous edition approved in 2006 as E1413—06; E1413—07. DOI: 10.1520/E1413-07; 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.

[E1412](#)[E2154](#) Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal 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. ~~At the same time, the~~ 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, GC/MS, or GC/IR chromatography-mass spectrometry (GC-MS), see Test Method [E1618](#).

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~~ 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~~ This is a potentially destructive ~~technique.”~~ 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 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 (or other inert gas) and vapors to the ~~charcoal~~ adsorption tube.

5.2 Negative Pressure Apparatus:

5.2.1 ~~Inlet and outlet system~~—Outlet System—A tube containing approximately 1 cm of charcoal is fitted into the lid of the original container with a ~~sleeve stopper~~ 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 ~~sleeve stopper~~ 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*—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.

5.4.1 *Charcoal Tubes*—Alternatively, charcoal tubes are available from commercial sources. 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*—~~The use of a different adsorbing media such as Tenax is possible, but this~~ 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 is best should be employed when thermal desorption is to be performed using a thermal operation 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.

6.1.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification [D1193](#).

6.2 *Activated Charcoal/Solid Adsorbent: (coconut):*

6.2.1 *Activated Charcoal* ~~Test each new batch of charcoal for residual hydrocarbons by analyzing a concentrated extract of blank adsorption media.~~(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 *Carbon Disulfide, Extraction solvent* ~~GC~~ grade (for example, carbon disulfide, pentane, ethyl ether).

6.4.1 Read and follow the safety precautions described in ~~Practice~~ [E752](#): 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](#).

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.~~ samples (Guide [E1459](#) and Practice [E1492](#)).

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

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.