



Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration onto an Adsorbent Tube¹

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 ignitable liquid residues from fire debris samples using dynamic headspace concentration onto an adsorbent tube, with subsequent solvent elution or thermal desorption.

1.2 Dynamic headspace concentration onto an adsorbent tube takes place from a closed, rigid sample container (typically a metal can), using a source of dry inert gas or a vacuum system.

1.3 Both positive and negative applied pressure systems for dynamic headspace concentration onto an adsorbent tube are illustrated and described.

1.4 This practice is suitable for preparing extracts from fire debris samples containing a range of volumes (μL to mL) of ignitable liquid residues, with sufficient recovery for subsequent qualitative analysis (1).²

1.5 Alternative headspace concentration methods are listed in Section 2 (see Practices E1388, E1412, E3189, and E2154).

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

1.7 *This standard cannot replace knowledge, skills, or abilities acquired through education, training, and experience (Practice E2917) and is to be used in conjunction with professional judgment by individuals with such discipline-specific knowledge, skills, and abilities.*

1.8 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

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 The boldface numbers in parentheses refer to a list of references at the end of this standard.

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:³

- E1388 Practice for Static Headspace Sampling of 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
- E1732 Terminology Relating to Forensic Science
- 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
- E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Programs
- E3189 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Static Headspace Concentration onto an Adsorbent Tube

³ 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. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology E1732.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *dynamic headspace concentration, n*—an extraction technique in which a portion of the headspace vapors is exchanged from the sample container and concentrated onto an adsorbent medium through applied positive or negative pressure.

3.2.2 *static headspace concentration, n*—an extraction technique in which a portion of the headspace vapors is removed from the sample container and concentrated onto an adsorbent medium.

4. Summary of Practice

4.1 Headspace vapors from the closed sample container are collected and concentrated onto an adsorbent tube by means of headspace exchange performed through the use of a system based on either positive or negative applied pressure. The adsorbent tube is subsequently eluted with solvent or desorbed thermally prior to instrumental analysis (typically by gas chromatography-mass spectrometry (GC-MS)).

4.2 The technique of dynamic headspace concentration onto an adsorbent tube is illustrated in Fig. 1. The sample container, and the adsorbent tube when necessary, can be heated during sampling, as outlined in Section 9.

5. Significance and Use

5.1 This practice is useful for preparing extracts from fire debris for subsequent qualitative analysis by gas chromatography mass spectrometry, see Test Method E1618.

5.2 The sensitivity of this practice is such that a sample consisting of a laboratory tissue onto which as little as 0.1 μL of ignitable liquid has been deposited, in an otherwise empty sample container, will result in an extract that is sufficient for identification and classification using Test Method E1618 (1).

5.2.1 Recovery from fire debris samples will vary, depending on factors including debris temperature, adsorbent temperature, container size, adsorptive material, headspace volume, sampling time and flow rate, and adsorptive competition from the sample matrix (2).

5.3 The principal concepts of dynamic headspace concentration are similar to those of static headspace concentration (Practice E3189). The dynamic headspace concentration technique can be more sensitive than the static headspace concentration technique. However, sample containers subjected to dynamic headspace concentration could be unsuitable for re-sampling.

5.3.1 Dynamic headspace concentration alters the original composition of the test sample because a portion of the original headspace from the sample container is removed and exchanged with dry inert gas or air. A portion of the concentrated headspace sample should be preserved for potential future analysis, if possible and if required, in accordance with Practice E2451.

5.4 Common solid adsorbent/desorption procedure combinations in use are activated carbon/solvent elution, and Tenax⁴ TA/thermal desorption.

5.5 Solid adsorbent/desorption procedure combinations not specifically described in this standard can be used as long as the practice has been validated as outlined in Section 11.

6. Apparatus

6.1 *Positive Applied Pressure-Based Sampling System*—A system capable of increasing the pressure inside the sample container by applying dry inert gas at the sample container inlet, thereby pushing the headspace vapors toward and through the outlet.

6.1.1 *Sample Pressurization Device*—A system capable of delivering pressurized dry inert gas at up to approximately 276 kPa (40 psi) to the inlet.

6.1.1.1 A needle valve capable of fine control of the flow rate of the dry inert gas, at up to approximately 400 mL/min.

6.1.2 A *filtering tube*, consisting of an adsorbent tube as described in 7.5, attached to the sample container inlet, for the purpose of filtering incoming dry inert gas.

6.1.3 A *flow meter* capable of measuring the flow of the dry inert gas through the end of the adsorption tube at the rate of approximately 40 to 400 mL/min.

6.1.4 Design and use the sampling system in a manner that prevents the loss of volatile compounds from the container and

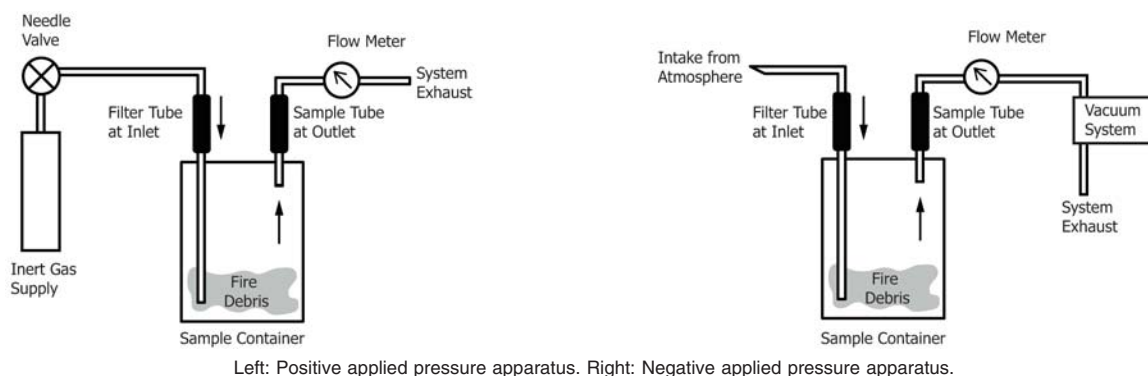


FIG. 1 Illustration of Dynamic Headspace Concentration onto an Adsorbent Tube

⁴ Tenax is a trademark of Teijin Carbon America, Inc., Rockwood, TN.

contamination of the sample from the apparatus itself or the laboratory environment.

6.2 *Negative Applied Pressure-Based Sampling System*—A system capable of creating partial vacuum inside the sample container by connecting a vacuum system to the outlet, thereby pulling the headspace vapors toward and through the outlet.

6.2.1 A *vacuum system* capable of pulling between approximately 40 and 400 mL/min at the outlet.

6.2.2 A *filtering tube*, consisting of an adsorbent tube as described in 7.5, attached to the sample container inlet, for the purpose of filtering incoming air.

6.2.3 A *flow meter* capable of measuring the flow of filtered air and headspace vapors through the outlet at the rate of approximately 40 to 400 mL/min.

6.2.4 Design and use the sampling system in a manner that prevents the loss of volatile compounds from the container and contamination of the sample from the apparatus itself or the laboratory environment.

6.3 *Heating System*—An oven large enough to accommodate the sample container, or a heating mantel designed to fit the sample container, capable of maintaining the required temperature range uniformly throughout.

6.4 *Temperature Measuring Device*—A thermometer or thermocouple capable of measuring the temperature of the heating system in the required range of operation, to within approximately 5°C.

6.5 *Thermal Desorption System*—A system capable of desorbing trapped volatile compounds from an adsorbent tube by means of elevated temperature, refocusing them on a cold-trap, and subsequently introducing them to a capillary GC column by flash heating. The desorption apparatus is directly coupled to a GC-MS.

6.6 *Puncturing Device*—A device, such as a nail, that is capable of puncturing small holes in the lid of the sample container. The size of the holes is such that the adsorbent tubes can be introduced.

7. Reagents and Materials

7.1 *Purity of Reagents*—Use reagent grade or better chemicals 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.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity through evaluation of appropriate blank(s) to permit its use without lessening the accuracy of the determination.

7.2 *Filter*—Permeable material such as silanized glass wool, wire mesh or cotton held in place with a frit.

7.3 *Solid Adsorbent*—Activated carbon, Tenax TA or equivalent.

7.4 *Sampling Tubes*—Glass Pasteur pipettes or equivalent glass tubes for solvent elution, and stainless steel or glass tubes for thermal desorption.

7.5 *Adsorbent Tubes*—Sampling tubes packed with a solid adsorbent.

7.5.1 Pre-packed adsorbent tubes are commercially available. Empty sampling tubes that can be packed by the purchaser are also available.

7.5.1.1 Activated carbon tubes and equivalent for solvent elution can be made by inserting a filter into the bottom of a sampling tube (approximately 5-mm diameter), then adding 2.5 cm to 5 cm of activated carbon or equivalent, which is held in place with a second filter.

7.5.1.2 Tenax TA tubes and equivalent for thermal desorption can be made by inserting a filter into one end of a sampling tube, adding approximately 80 mg of pre-conditioned Tenax TA or equivalent, and then packing tightly with a second filter.

NOTE 1—Tenax TA or equivalent is conditioned by heating, in accordance with instrument manufacturer or supplier instructions.

7.6 *Screw cap or crimp-top glass vials* with polytetrafluoroethylene (PTFE) lined seals.

7.7 *Tape, rubber sleeve stopper*, or equivalent.

7.8 *Elution Solvent*—Suitable elution solvents include carbon disulfide, *n*-pentane, dichloromethane and diethyl ether.

8. Sample Preparation

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

8.2 Prepare the fire debris sample container for sampling by dynamic headspace concentration.

8.2.1 The sampling system is designed so that the headspace sample can be extracted from the container in which it was received at the laboratory.

8.2.1.1 Alternatively, the fire debris sample, or a portion of it, can be transferred to a clean, rigid sample container suitable for dynamic headspace sampling, such as a metal can with a friction-seal lid.

8.2.1.2 Verify the cleanliness of the transfer container prior to sample transfer. Cleanliness is determined by means of analysis of a dynamic headspace concentration sample obtained from the empty transfer container using the same conditions as will be used for the questioned sample.

8.2.1.3 Allow the transfer container with sample to equilibrate for at least one hour before sampling.

8.2.2 Create two holes in the lid of the rigid sample container using a puncturing device, such that the adsorbent tubes can be introduced.

8.2.2.1 Seal the holes with tape, rubber sleeve stopper, or equivalent.

NOTE 2—Cans that are designed for fire debris samples, with a hole pre-fitted with a rubber sleeve stopper, are commercially available.

⁵ *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.