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An American National Standard

Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Static Headspace Concentration onto an Adsorbent Tube[1]

This standard is issued under the fixed designation E3189; 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 static headspace concentration onto an adsorbent tube, for subsequent solvent elution or thermal desorption.

1.2 Static headspace concentration onto an adsorbent tube involves removal of a headspace extract from a sample container (typically a jar, can, or bag), through a small hole punctured in the container, using a syringe or pump.

1.3 Static headspace concentration systems for adsorption 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, E1413, 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.*

1.9 *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

E1413 Separation of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration onto an Adsorbent Tube

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

3. Terminology

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

[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.

[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.

3.2 *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, n*—an extraction technique in which a portion of the headspace vapors is removed from the sample container.

3.2.3 *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 sample container are collected and concentrated onto an adsorbent tube through a needle or plastic tip connected to the inlet of the adsorbent tube and inserted into the container by means of a small hole punctured in the container, and using a syringe or pump connected to the outlet of the adsorbent tube. The adsorbent tube is subsequently desorbed thermally or eluted with solvent prior to instrumental analysis (typically by gas chromatography-mass spectrometry (GC-MS)).

4.2 The technique of static headspace concentration onto an adsorbent tube is illustrated in Fig. 1. The sample container may be heated before sampling, as outlined in Section 8, but the adsorbent tube is not.

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 This practice is capable of removing a portion of the headspace vapors, containing quantities smaller than 0.1 µL/L of ignitable liquid residues, from a sample container and concentrating the ignitable liquid residues onto an adsorbent medium (**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 volume or sampling time and flow rate, and adsorptive competition from the sample matrix (**2**).

5.3 The principal concepts of static headspace concentration are similar to those of static headspace (Practice E1388) and dynamic headspace concentration (Practice E1413). The static headspace concentration technique can be more sensitive than the static headspace technique and less sensitive than the dynamic. The static techniques do however leave the sample in a condition suitable for resampling, as only a portion, typically less than 10 %, of the headspace is withdrawn from a sample container (**3**).

5.3.1 Re-sampling and analysis is possible with static headspace concentration onto an adsorbent tube, because only a portion of the headspace from the container is removed (**3**). Taking multiple headspace samples will continuously reduce the concentration of ignitable liquid vapors present, which can result in a change in relative composition of components and eventually non-recovery when the questioned headspace originally contained very low quantities of ignitable liquid residues (less than 0.1 µL/L).

5.4 Common solid adsorbent/desorption procedure combinations in use are activated carbon/solvent elution and Tenax[4] TA/thermal desorption.

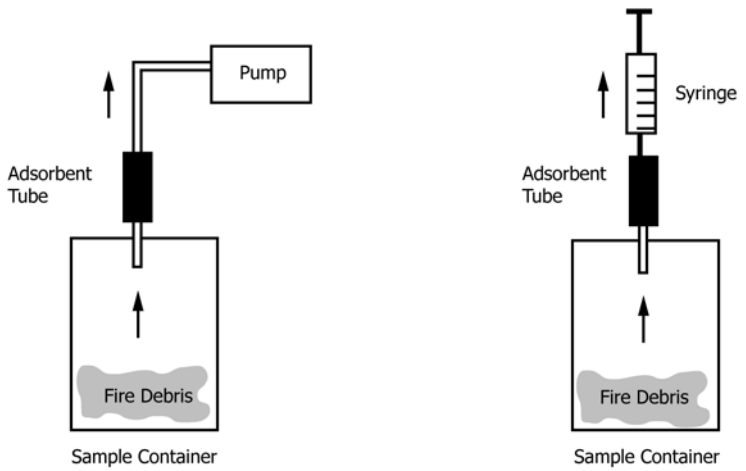
5.5 Solid adsorbent/desorption procedures not specifically described in this standard can be used as long as the practice has been validated as outlined in Section 11.

6. Apparatus

6.1 *Static Headspace Concentration Sampling Apparatus:*

6.1.1 *Sampling Device*—A device capable of concentrating volatile compounds in air, consisting of an adsorbent tube with the outlet connected to an air-tight syringe or pump and the inlet connected to a needle or plastic tip.

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



Left: Sampling by means of a pump. Right: Sampling by means of a syringe.

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

6.1.2 *Puncturing Device*—A device such as a pin or a nail that is capable of puncturing a small hole in the sample container. The size of the hole is such that a syringe needle or plastic pipette tip can fit (loosely) into the opening.

6.1.3 *Sampling System*—A system that consists of a sampling and puncturing device, capable of withdrawing a portion of headspace through a hole in the container and concentrating volatile compounds from this headspace portion onto an adsorbent tube.

6.1.3.1 The sampling system must be designed and used 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.

6.2 *Thermal Desorption System*—A system capable of desorbing the 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.

6.3 *Pump*—A vacuum or hand pump capable of drawing air at a flow rate of at least 2 mL/min to 80 mL/min.

6.4 *Oven*—An oven large enough to accommodate the sample container, and capable of maintaining the required temperature uniformly throughout.

6.5 *Temperature Measuring Device*—A thermometer or thermocouple capable of measuring oven temperatures in the required range of operation, to within approximately 5°C.

7. Reagents and Materials

7.1 *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.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficient 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 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 *Disposable Syringes*—1 mL to 100 mL.

7.8 *Disposable syringe needles, tips, or equivalent.*

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

7.10 *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 static headspace concentration onto an adsorbent tube.

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 sample container such as a jar, can, or bag suitable for static headspace concentration onto an adsorbent tube.

8.2.1.2 Verify the cleanliness of the transfer container prior to sample transfer. Cleanliness is determined by means of analysis of a static 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 a hole in the container with the puncturing device such that a syringe needle or plastic tip can be introduced. The hole is created in the lid of a can or jar, or in the headspace area of a bag.

8.2.2.1 Seal the hole 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.

8.3 When necessary, heat the container of fire debris sample at the selected temperature prior to the headspace sampling, in order to release any ignitable liquid residues as a vapor into the headspace.

8.3.1 Heating is not recommended if there are other evidentiary considerations such as preservation of DNA or latent fingerprints.

[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.