Designation: E1388 – 17

Standard Practice for Static Headspace Sampling of Vapors from Fire Debris Samples[1]

This standard is issued under the fixed designation E1388; 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 removing vapor from the headspace of a fire debris container for the purpose of detecting or identifying ignitable liquid residues.

1.2 Separation and concentration procedures are listed in the referenced documents. (See Practices E1386, E1412, E1413, and E2154.)

1.3 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.

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

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.*

1.6 *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:*[2]

E1386 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction

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

E1413 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration

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 headspace of an item is sampled, preferably in its original container, for analysis by an appropriate technique (for example, gas chromatography-mass spectrometry). The container may be heated prior to sampling to increase the volatility of any ignitable liquid residues.

4. Significance and Use

4.1 This practice is useful for sampling fire debris to screen for the presence of ignitable liquid residues prior to extraction with other techniques. It is most appropriate for sampling light to medium range ignitable liquids (such as light oxygenates, lacquer thinners, and other similar volatile compounds or products), and less appropriate for sampling ignitable liquids that have compounds in the heavy range.[3, 4, 5]

4.1.1 When sampled for screening purposes, the instrumentation typically utilized is a gas chromatograph with either a

[1] This practice in under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics

Current edition approved July 15, 2017. Published July 2017. Originally approved in 1990. Last previous edition approved in 2012 as E1388 – 12. DOI: 10.1520/E1388-17.

[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] Ettling, B. V., and Adams, M. F., "The Study of Accelerant Residues in Fire Remains," *Journal of Forensic Sciences*, Vol 13, No. 1, 1968, pp. 76–89.

[4] Stauffer, E., Dolan, J. A., and Newman, R., *Fire Debris Analysis*, Boston, MA: Academic Press, 2008.

[5] Hinshaw, J. V., "Headspace Sampling," *LCGC Europe*, Vol 24, No. 10, 2011.

mass spectrometer (GC-MS, refer to Test Method E1618) or flame ionization detector (GC-FID).

4.2 This practice is generally less efficient at recovering limited quantities of ignitable liquids than Practices E1386, E1412, E1413, and E2154, particularly for higher boiling compounds.

4.3 The separation takes place in a closed container and the sample remains in approximately the same condition in which it was submitted. Since only a small aliquot of the sample headspace is removed for analysis, sample reanalysis may be possible.

4.4 High concentrations of highly volatile compounds can saturate the headspace, inhibiting the recovery of less volatile components and leading to the detection or identification of only the more volatile compounds in the sample.

4.5 This practice is intended for use in conjunction with other extraction techniques, such as those described in Practices E1386, E1412, E1413, and E2154, when analysis of a sample for all classes of ignitable liquids is required or desired.

NOTE 1—The headspace specimen (the portion in the syringe) is consumed in the analysis. Preserve an extract for potential reanalysis (see Practice E2451) using an alternative separation and concentration practice, such as those described in Practices E1386, E1412, and E1413, if preservation is required per laboratory policies.

5. Apparatus

5.1 *Heating System*—An oven or equivalent, capable of reproducible temperature operation for the desired range that can accommodate the evidence container.

5.1.1 An oven is recommended to achieve a constant temperature throughout the system.

5.2 *Temperature Measuring Device*—A thermometer or thermocouple capable of measuring temperatures in the required range of operation.

5.3 *Disposable or reusable gas-tight syringes,* capable of reproducibly sampling in the range from 0.5 to 2.0 mL.

5.4 *Disposable or reusable syringe needles.*

5.5 *Gas-tight vials and caps.*

5.6 A tool or device capable of creating holes in evidence containers (for example, drill, hole punch, hammer and nail).

5.7 *Tape, rubber sleeve stoppers, or equivalent,* capable of sealing holes in evidence containers.

6. Sample Preparation

6.1 Observe the appropriate procedures for handling and documentation of all submitted samples as described in Guide E1459 and Practice E1492.

6.1.1 Examine the fire debris sample to determine that it is consistent with its description prior to reporting. The examination may be performed prior to or after the sampling procedure in Section 7.

6.1.1.1 Resolve any discrepancies between the submitting agency's description of the evidence and the analyst's observation prior to the completion of the report.

6.2 Prepare the sample or evidence container for headspace sampling.

6.2.1 Create a hole in the container lid such that a syringe needle can be introduced. Seal the hole with tape, a rubber sleeve stopper, or as otherwise required. Alternatively, crack the container lid to allow a syringe needle to be introduced and close following sampling.

NOTE 2—Some cans designed for fire debris samples contain lids having rubber sleeve stoppers already in place.

6.2.2 Some samples, such as liquids, may be transferred and sealed into vials for analysis without any further preparation.

6.2.3 Prepare control samples and blanks for sampling using the same procedures used for questioned samples as required per laboratory policies.

6.3 If heating is required, place the container in the heating system until the container reaches the desired temperature and remove immediately prior to sampling. Record the temperature and duration of heating.

6.3.1 Sampling at room temperature is appropriate to recover the more volatile compounds. Sampling at elevated temperatures, typically between 60°C and 90°C, is necessary to recover compounds with lower volatility.

NOTE 3—When other evidentiary considerations arise (such as DNA or latent print examinations), consult with other relevant analyst(s) prior to heating samples.

7. Sampling Procedure

7.1 *Headspace Sampling for Manual Injection:*

7.1.1 Assemble a syringe and needle (sampling syringe) as needed. The syringe should be heated to the sampling temperature prior to sampling.

7.1.2 Withdraw between 0.5 to 2.0 mL of headspace from the evidence container into a sampling syringe.

7.1.2.1 Sample from an area above the fire debris in the evidence container.

7.1.3 Immediately analyze the specimen using an appropriate instrumental technique (for example, gas chromatography-mass spectrometry).

7.1.4 After sampling, reseal the evidence container as needed or prepare as necessary for any additional analyses to include other fire debris extraction techniques.

7.1.5 Perform sampling on control samples and blanks following the same procedures used for questioned samples as required in accordance with laboratory policies.

7.2 *Headspace Sampling for Automated Injection System:*

7.2.1 Some sample injection systems utilize headspace vials for delivery of a sample headspace into the gas chromatograph.

7.2.2 Assemble and seal (for example, crimp or screw) cap onto headspace vial.

7.2.3 Assemble a syringe and needle (sampling syringe) as needed to be used for evacuating the sealed headspace vial and removing headspace samples from an evidence container.

7.2.3.1 Syringe size (volume capacity) may vary but the sampling syringe should have approximately the same or greater volume capacity as the vials.

7.2.4 Evacuate a portion of the sealed vial using the sampling syringe. Repeat this procedure until the equivalent of at least twice the volume capacity of the vial has been evacuated.

NOTE 4—A needle assembly attached directly to a vacuum source may be used to evacuate the vial as an alternative to using a sampling syringe.

7.2.5 Withdraw a sample of the headspace above the fire debris from the evidence container using a sampling syringe.

7.2.6 Immediately transfer the contents of the sampling syringe into the evacuated vial. Repeat this process until the injected volume of headspace from the evidence container approximates the volume previously evacuated from the vial.

NOTE 5—If the vial was properly sealed and evacuated, the vacuum created should pull the contents of the sampling syringe of the initial headspace sample into the vial. Any subsequent sampling and transfer could require some effort to push the contents of the sampling syringe into the vial.

7.2.7 Immediately analyze specimens, using an appropriate instrumental technique (for example, gas chromatography-mass spectrometry).

7.2.8 After sampling, reseal the container as needed or prepare as necessary for any additional analyses to include other fire debris extraction techniques.

7.2.9 Perform sampling on control samples and blanks following the same procedures used for questioned samples as required in accordance with laboratory policies.

8. Keywords

8.1 fire debris samples; headspace