



Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples¹

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1. Scope

1.1 This practice describes the procedure for removing small quantities of ignitable liquid vapor from samples of fire debris by sampling the headspace of the debris container.

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

1.3 *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

[E1412](#) Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal

[E1413](#) Practice for Separation and Concentration 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)

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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. Summary of Practice

3.1 The sample, preferably in its original container, is heated in order to volatilize any ignitable liquid residues present in the debris. After heating, the headspace is sampled and analyzed by gas chromatography-mass spectrometry.

4. Significance and Use

4.1 This procedure is particularly useful for screening fire debris samples to determine relative ignitable liquid concentrations and possible ignitable liquid class prior to extraction with other techniques.

4.2 This practice is useful when volatile oxygenated products such as alcohols or lacquer thinners are suspected.

4.3 This practice is the least sensitive of the sample preparation techniques and may not detect quantities of less than 10 μL of an ignitable liquid.

4.4 Because this separation takes place in a closed container, the sample remains in approximately the same condition in which it was submitted. Repeat and interlaboratory analyses are therefore possible.

4.5 Due to variables in the debris sample condition prior to headspace sampling, complete reproducibility of chromatograms may be difficult to obtain.

4.5.1 To obtain greater reproducibility, use one of the separation procedures which results in a solution of the ignitable liquid residue (see Practices [E1386](#), [E1412](#), and [E1413](#)).

4.5.2 To obtain greater sensitivity for petroleum distillates and oxygenated products, use solid phase microextraction. (See Practice [E2154](#).)

4.6 High concentrations of highly volatile compounds may swamp the headspace, inhibiting the recovery of less volatile components.

5. Apparatus

5.1 *Heating System*—An oven, heating mantle or hot plate designed to fit the evidence container.

5.2 *Temperature Measuring Device*—A thermometer or thermocouple capable of measuring temperatures in the range from 40 to 150°C.

5.3 A gas-tight syringe capable of reproducibility introducing sample sizes in the range from 0.5 to 5 mL.

5.4 Disposable 20 cc syringes.

5.5 Disposable 16–20 gauge syringe needles.

5.6 A drill, punch, or other device to puncture holes in sample containers.

5.7 Headspace sample vials.

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 Open and examine the fire debris sample in order to determine that it is consistent with its description.

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 Punch or drill a small hole in the container lid and cover the hole with tape.

6.2.1 Alternatively, a rubber sleeve stopper may be placed in the hole.

6.3 If the sample is going to be heated, place the container in the heating system until the container reaches the desired temperature (not to exceed 90°C).

6.4 The syringe may be heated prior to sampling

7. Sampling Procedure

7.1 Direct Headspace Sampling:

7.1.1 Remove the container from the heating apparatus then push the syringe through the tape or rubber sleeve stopper into the hole in the container lid. Slowly pump the syringe several times, withdraw from the lid, and inject 0.5 to 2.0 mL of sample into the gas chromatograph. Analyze as described in Test Method [E1618](#).

7.1.2 After the headspace sample has been removed, reseal the hole.

7.2 Indirect Headspace Sampling Using Headspace Sample Vials:

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

7.2.2 Assemble and seal (crimp or screw) lid onto headspace sample vial. Using a 20 cc syringe and needle, puncture the headspace vial septum and evacuate the contents of the vial. Repeat this procedure 3 to 4 times.

7.2.3 Remove the container from the heating apparatus then push the 20 cc syringe through the tape or rubber sleeve stopper into the hole in the container lid. Slowly pump the syringe several times, withdraw 20 cc of vapor and inject into headspace sample vial. Repeat this process 2 to 4 times.

7.2.3.1 If the headspace sample vial was properly sealed and evacuated, the vacuum created should pull the initial injection into the vial. The latter injections into the vial will require forcibly injecting the 20 cc sample.

7.2.3.2 The sequence method on the gas chromatograph should be setup to inject 0.5 to 2.0 mL of the sample into the gas chromatograph. Analyze as described in Test Method [E1618](#).

7.2.4 After the headspace samples have been removed, reseal the hole in the container.

8. Keywords

8.1 fire debris samples; headspace

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