Standard Practice for
Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Solid Phase Microextraction (SPME)[1]

This standard is issued under the fixed designation E2154; 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 small quantities of ignitable liquid residues from samples of fire debris. An adsorbent material is used to extract the residue from the static headspace above the sample. Then, analytes are thermally desorbed in the injection port of the gas chromatograph (GC).

1.2 This practice is best suited for screening fire debris samples to assess relative ignitable liquid concentration and for extracting ignitable liquid from aqueous samples.

1.3 This practice is suitable for extracting ignitable liquid residues when a high level of sensitivity is required due to a very low concentration of ignitable liquid residues in the sample.

1.3.1 Unlike other methods of separation and concentration, this method recovers a minimal amount of the ignitable residues present in the evidence, leaving residues that are suitable for subsequent resampling.

1.4 Alternate separation and concentration procedures are listed in Section 2.

1.5 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.

1.6 *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
E1388 Practice for Sampling of Headspace 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 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration
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

3. Summary of Practice

3.1 A fiber coated with a polydimethylsiloxane stationary phase is exposed to the headspace of the fire debris sample container to extract ignitable liquid residues. The fiber, which is housed in a needle similar to a syringe needle, is introduced directly in the injection port of a gas chromatograph to thermally desorb the analytes.

4. Significance and Use

4.1 This practice is suited ideally for screening samples for the presence, relative concentration, and potential class of ignitable liquid residues in fire debris.

4.2 This is a very sensitive separation procedure, capable of isolating small quantities of ignitable liquid residues from a

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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.

sample, that is, a 0.1 µL spike of gasoline on a cellulose wipe inside of a 1-gal can is detectable.

4.3 Actual recovery will vary, depending on several factors, including adsorption temperature, container size, competition from the sample matrix, ignitable liquid class and relative ignitable liquid concentration.

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, therefore, may be possible. Since the extraction is nonexhaustive, the technique permits reanalysis of samples.

4.5 This practice is intended for use in conjunction with other extraction techniques described in Practices E1386, E1388, E1412, and E1413.

4.6 The extract is consumed in the analysis. If a more permanent extract is desired, one of the separation practices described in Practices E1386, E1412, or E1413 should be used.

5. Apparatus

5.1 *Heating System,* such as, an oven or heating mantle to fit the evidence container (or a hot plate).

5.2 *Temperature Measuring Device,* such as, a thermometer or thermocouple capable of measuring temperatures in the range of 40 to 110°C.

5.3 *SPME Apparatus,* such as, a solid phase microextraction fiber holder.

5.4 *SPME Fiber*—Several polymer coatings can be used to extract volatiles such as ignitable liquid residues. A100 µm polydimethylsiloxane (PDMS) coating has been shown to perform well for most C_{10}-C_{25} compounds while a 85 µm Polyacrylate (PA) and a combined 75 µm Carboxen/PDMS has been shown to perform well for C_1-C_{10} compounds (**1**).[3]

5.5 *Punch.*

5.6 *Rubber Sleeve Septum.*

6. Sample Preparation/Analysis Procedure

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

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

6.1.1.1 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.

6.2 Verify that the fiber is free of ignitable residue components and other contaminants.

6.2.1 Introducing the fiber into the GC under the injector desorption and chromatographic conditions for ignitable residue analysis. This fiber residue "blank" always should be evaluated before residue sampling when performing a manual injection and desorption.

6.2.2 When performing batch analysis of fiber blanks for autosampler apparatus, all fibers determined to be clean by analysis should be promptly sealed in a vapor-tight container(s) until ready for use.

6.3 If the sample container has a metal lid, make a hole in the lid and install a rubber sleeve septum in the opening.

6.4 Heat the container until the sample reaches the desired temperature (approximately 20-30 min, but longer equilibration times may be needed depending on the heating method or the amount and type of matrix present in the can, or both).

6.4.1 Temperatures lower than 60°C may be insufficient to volatilize compounds above C_{16}.

6.4.2 Temperatures above 80°C may result in a significant discrimination against high volatility compounds when the 100 µm PDMS fiber is used.

6.5 Remove the container from the heating apparatus.

6.6 Immediately puncture the rubber sleeve septum or plastic evidence bag with the needle of the SPME apparatus.

6.7 Expose the SPME fiber to the headspace of the container for the desired sampling duration.

6.7.1 The optimum exposure time for maximum sensitivity will depend on the temperature and the concentration and composition of the volatile compound present in the sample headspace.

6.7.2 Exposure times for routine screening of samples typically are in the range of 5-15 min.

6.7.3 Residue extracts that provide off-scale or poorly resolved chromatographic responses should be resampled at lower temperatures or shorter exposure times.

6.8 Secure the fiber in the needle by withdrawing it into the SPME apparatus. Remove the apparatus from the sample container.

6.8.1 Rubber sleeve septa are self-sealing. Puncture holes in evidence bags should be sealed after sampling.

6.9 Analyze the adsorbate by exposing the fiber in the injection port of a gas chromatograph. Desorb the fiber for approximately 1.5 to 4 min between 200°C and 260°C. Analysis is performed as described in Test Method E1618.

6.9.1 Some instruments can desorb fibers using an autosampler apparatus. The acceptability of the autosampler parameters shall be verified using known ignitable liquids and fiber blanks within a sequence.

6.10 Repeat each step in Section 6 as necessary for each remaining fire debris sample.

6.10.1 Previously analyzed fiber blanks are analyzed during an autosampler sequence. At least one fiber blank must be analyzed, preferably near the end of the sequence, to verify that fibers were not contaminated during storage or sample processing.

6.11 Record the adsorption parameters including the exposure time and temperature in the analytical case notes.

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

7. Blanks and Standards

7.1 Analysis of a fiber blank (6.2) is required immediately before each manual sample extraction and batch analysis of fiber blanks prior to sample extractions when using autosampler apparatus.

7.1.1 If an internal standard is routinely used, include the internal standard in the blank run by placing the internal standard in an empty container and exposing the SPME fiber as described in Section 6.

7.2 Periodically check the adsorption efficiency of the fiber by running this procedure on a sample containing a known volume of an ignitable liquid standard.

7.3 An ignitable liquid standards library should be maintained with neat and evaporated commonly ignitable liquids recovered from sample containers by this technique at various concentrations.

8. Keywords

8.1 fire debris samples; passive headspace concentration; solid phase microextraction (SPME)

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