

Designated E xxxx-xx

Standard Guide for a Systematic Approach to the Analysis of Fire-Related Samples for Ignitable Liquids and their Residues

Version 1.0

Developed by the

Laboratory Fire Standards and Protocols Committee Scientific Working Group for Fire and Explosions

1. Scope

- 1.1. This guide describes a systematic approach for the analyses of fire debris and liquid samples for the presence of ignitable liquids and their residues.
- 1.2. This guide does not address the specific methodologies necessary for the extraction of debris, the techniques of instrumental analysis, or the interpretation of analytical data.
- 1.3. This guide does not attempt to address all the issues regarding sample analyses. There may be additional tests or analyses that can be performed to provide further discrimination and characterization of samples.
- 1.4. This guide does not address the necessity for laboratory notes and the proper documentation required for all forensic examinations.
- 1.5. This guide 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 guide to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1. ASTM Standards:

- E 1386 Practice for the Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction
- E 1387 Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography
- E 1388 Practice for Sampling of Headspace Vapors from Fire Debris Samples
- E 1412 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal
- E 1413 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration
- E 1459 Guide for Physical Evidence Labeling and Related Documentation
- E 1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory

- E 1618 Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry
- E 2154 Practice for the Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Solid Phase Microextraction (SPME)
- E xxxx Standard Practice for Archiving Ignitable Liquid Residues in Extracts from Fire Debris Samples and Questioned Liquid Samples Submitted for Ignitable Liquid Analysis

3. Summary of Guide

- 3.1. The analysis of fire debris samples should be conducted systematically. There are a variety of established methods for the extraction of fire debris samples. This guide provides an organized approach for the examination and analysis of this type of evidence.

4. Significance and Use

- 4.1. This guide provides an efficient and comprehensive analytical scheme for the analysis of fire debris samples.
- 4.2. This guide is to be used in conjunction with the referenced documents. The analyst must be familiar with the limitations and applicability of each technique.

5. Evidence handling

- 5.1. Ensure that chain of custody is intact and properly identifies the evidence. Resolve deficiencies as necessary.
- 5.2. Ensure that the evidence is properly packaged and resolve deficiencies as necessary.
- 5.3. Ensure that accompanying documentation properly describes the evidence. Resolve deficiencies as necessary.

6. Ensure proper storage

- 6.1. Items believed to contain substrate materials likely to contribute to the degradation of petroleum products, such as vegetation or soil should be refrigerated or frozen until the time of analysis if the analysis cannot be expedited.
- 6.2. Items of evidence shall be stored in a manner that will avoid contamination.
- 6.3. Following analysis, the original sample and extracts generated during sample preparation should be preserved.

7. Investigative Background

- 7.1. Information from scene investigators can aid the analyst in understanding the events of the incident, can assist in evaluating the evidence submitted, and can help in determining which tests or test methods might be appropriate.
- 7.2. Information from the investigator that may affect the analytical scheme should be obtained prior to analysis when possible. This type of information may include sample location, suppression techniques, knowledge of ignitable liquids inherent to the scene, the types of examinations requested, and the suspected presence of materials that may require specialized testing.
- 7.3. Identify special considerations that may affect the initial approach to the evidence.
- 7.4. To ensure adherence to universal precautions, attempt to determine if any biological or chemical hazards are present. Determine if other evidence of value could be potentially derived from the evidence, such as DNA, latent prints, or other trace evidence.
- 7.5. When multiple forensic disciplines will be involved, coordinate the overall approach to the analysis with the investigator and other analysts. Determining the sequence of multiple examinations will depend upon the priorities of the investigation, the nature of the evidence, and the requested examinations.

8. Initial Assessment of Evidence

- 8.1. A brief visual assessment of each item should be conducted. Preliminary identification of contents can provide direction to the selection of an analytical scheme and suggest complexities that may be encountered in the analytical data obtained.
 - 8.1.1. If the evidence is determined to contain one or more of the following types of materials, additional examinations will likely be required.
 - 8.1.1.1. Flare residues.
 - 8.1.1.2. Incendiary residues.
 - 8.1.1.3. Device remnants.
 - 8.1.2. Different substrates may produce a variety of interfering products; therefore, the substrate material should be noted when possible.
 - 8.1.3. If the matrix is excessively charred or if excessive water is present, it may affect the recovery of ignitable liquid residues.
 - 8.1.4. Note the presence of extraneous materials in samples such as additional layers of packaging or disposable gloves used for collection. Such items shall be separated from the evidence prior to processing.
 - 8.1.5. Given the unknown nature and origin of most samples, olfactory analysis may pose a health risk and is not recommended. Obvious odors should be noted because they may provide an indication of the type of ignitable liquid residues that are present in the sample and may aid in the selection of an extraction scheme.

9. Comparison Samples

- 9.1. Comparison samples may provide information that can aid in the interpretation of data obtained from fire debris samples. Examples of such information include the effects of a soil on the degradation of a petroleum product, the presence of incidental ignitable liquids or background products in the substrate, and the nature of pyrolysis and combustion products released by the material being tested.

10. Sample Preparation

10.1. *Liquid Sample Assessment Techniques:*

- 10.1.1. Liquid samples may be submitted by fire investigators for identification or comparison. These samples may not always be submitted in their original containers. Ignitable liquids, if present, may be in their original state or mixed with other liquids. The ideal sampling procedure will provide a representative sample of the liquid in its entirety. There is no sampling procedure that will perform ideally for all samples. Preliminary assessment of the sample can aid the analyst in determining the best sampling procedure to use. These assessment techniques may include many common chemical and physical tests.
- 10.1.2. Ignitability: Observing a sample's ignition and burning characteristics is a valuable assessment technique. Some of the ignition and burning characteristics that may be of value include ease of ignition, flame color, and smoke characteristics. While flash point (flammability or combustibility) may sometimes be determined using a flash point apparatus, such analyses are not generally required.
- 10.1.3. Miscibility: Miscibility testing can be used for preliminary assessment of liquids. Most ignitable petroleum products are immiscible with water. Many common oxygenates are miscible with water. Therefore, if a sample contains more than one phase, individual sampling of each phase should be considered.
- 10.1.4. Other tests: Additional types of testing may be useful in characterizing an unknown liquid. These may include pH, density, freezing point, boiling point, refractive index and other tests deemed necessary.

10.2. *Liquid Sample Preparation Techniques for Gas Chromatographic Analysis:*

- 10.2.1. Headspace Sampling (ASTM E 1388): Headspace sampling is particularly useful for highly volatile liquid samples.
- 10.2.2. Direct Injection: This method can provide the best representation of a liquid's components, however, it is not generally recommended for aqueous samples. This technique is not typically amenable to automated analyses.
- 10.2.3. Simple Dilution: Simple dilution is the most common technique for preparation of liquid samples. A 1% solution in a miscible solvent is generally recommended. One disadvantage of this technique is that the solvent peak may mask components of interest.

- 10.2.4. Liquid-Liquid Extraction: Liquid-Liquid extraction is a technique used to extract ignitable liquid residues from aqueous samples. An appropriate organic solvent is added to the aqueous sample and the sample is agitated. Petroleum products and other organic components will be extracted into the organic phase. The organic layer is then isolated, concentrated if necessary, and analyzed.
- 10.2.5. Passive Headspace Concentration (ASTM E-1412): Passive headspace concentration with activated charcoal can be used to extract liquid samples. It should be noted that in the presence of high concentrations of ignitable liquids, the chromatographic data may be distorted.
- 10.2.6. Solid Phase Microextraction (SPME) (ASTM E-2154): SPME can be used for headspace sampling of liquids. It should be noted that in the presence of high concentrations of ignitable liquids, the chromatographic data may be distorted.

10.3. *Debris Assessment Techniques:*

- 10.3.1. Assessment Considerations: Most samples submitted for analysis by fire investigators are in the form of fire debris collected in the course of scene investigations. These samples may be in various states of charring, destruction, or decomposition due to fire damage, fire suppression, and packaging. Ignitable liquid residues, if present, may range from very low to very high concentrations. The ideal extraction procedure will provide a representative sample of the volatile components with minimal contributions from matrix materials. There is no single extraction technique or set of parameters within a technique that is ideal for all samples. Some samples may require more than one extraction or analysis before a conclusion can be made. For this reason, the least destructive procedures should be used first. Preliminary assessment of the samples to determine relative concentration can aid the analyst in determining the best extraction procedure and parameters for a given sample.
 - 10.3.1.1. The analysis procedures for simple matrices including non-porous materials and small samples may differ from that of more complex matrices (e.g. carpet, pad, plastics, clothing, printed paper, floor tile, wood, and “unidentifiable” charred debris).
- 10.3.2. Headspace Sampling (ASTM E-1388): Headspace analysis is simple, quick, and non-destructive. Headspace sampling is an ideal alternative for olfactory techniques in screening debris samples. The headspace technique can provide the analyst with preliminary information concerning the presence of an ignitable liquid, its classification, and its relative concentration. Headspace sampling is also appropriate for the analysis of very volatile components such as ethers or alcohols. The disadvantages of this technique include limited sensitivity and the inability to recover less volatile components.
- 10.3.3. Solid Phase Microextraction (SPME) (ASTM E-2154): SPME sampling is an ideal alternative for olfactory techniques in screening debris samples. SPME is a simple technique that can provide the analyst with preliminary

information concerning the presence of an ignitable liquid, its classification, and its relative concentration.

10.4. Debris Extraction Techniques for Gas Chromatographic Analysis:

- 10.4.1. Passive Headspace Concentration (ASTM E-1412): Passive headspace concentration using activated charcoal is a versatile, non-destructive technique offering numerous advantages and is, therefore, the initial method of extraction for most analyses. This is an efficient technique that is highly sensitive and can recover a broad range of compounds. It requires minimal sample manipulation, has very low potential for sample contamination, and is amenable to sample preservation and resampling. It should be noted that recovery might be less than ideal in the presence of a highly adsorbent matrix. Determining the optimum parameters is critical for obtaining data representative of any ignitable liquid residue.
- 10.4.2. Dynamic Headspace Concentration (ASTM E-1413). Dynamic headspace concentration using activated charcoal is highly sensitive and can recover a broad range of compounds. Total extraction time is relatively short however, it is a labor-intensive technique. Special consideration must be given to minimizing the potential for sample contamination. This technique may be considered destructive, although, it is amenable to sample preservation. It should be noted that recovery might be less than ideal in the presence of a highly adsorbent matrix. Determining the optimum parameters is critical for obtaining data representative of any ignitable liquid residue.
- 10.4.3. Solvent Extraction (ASTM E-1386): Solvent extraction is a technique capable of extracting both volatile and nonvolatile compounds across a broad range with minimal sample discrimination. This method is efficient in the recovery of compounds of interest from a highly adsorbent matrix. Solvent extraction is an efficient technique for isolating all soluble compounds. It might result in the extraction of extraneous compounds and cause complexities in the analytical data. Solvent extraction may be warranted when definitive information is required for semi-volatile components. A loss of low boiling point compounds may occur during sample concentration. This is a destructive technique therefore, only a portion of the sample should be used for solvent extractions.

11. Instrumental Analysis

- 11.1. In general, analytical techniques based upon separation and detection of individual components are required.
- 11.2. Gas Chromatography (Flame Ionization Detector): Gas chromatography is an instrumental technique capable of resolving a wide range of organic compounds. A flame ionization detector (FID) is sensitive, universal and non-specific. This technique may be used for the identification and classification of complex ignitable liquids and their residues. It is not sufficient for the identification of all ignitable liquids or in cases of high concentrations of interfering compounds.

- 11.3. Gas Chromatography-Mass Spectrometry: Gas chromatography-mass spectrometry (GC-MS) is an instrumental technique capable of resolving a wide range of organic compounds and providing structural information about individual components. This technique may be used for the identification of single component ignitable liquids and for samples that contain high concentrations of interfering compounds.
- 11.4. Other Instrumental Methods: Other instrumental methods may be useful for the characterization or identification of ignitable liquids. The use of other methods will be dictated by the investigative needs of the case and the unique characteristics of the sample.

12. Sample Preservation

- 12.1. Whenever possible, samples should be preserved for potential reanalysis.