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An American National Standard

Standard Guide for Systematic Approach to the Extraction, Analysis, and Classification of Ignitable Liquids and Ignitable Liquid Residues in Fire Debris Samples[1]

This standard is issued under the fixed designation E3245; 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.

ε[1] NOTE—Editorial corrections were made throughout in July 2020.

1. Scope

1.1 This guide describes a systematic approach to the extraction, analysis, and classification of ignitable liquids and their residues in solid (for example, fire debris) and liquid samples.

1.2 This guide addresses evidence handling, extraction methodologies, instrumental analysis techniques, and analytical data interpretation.

1.3 *This guide 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 *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.*

1.5 *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]

E620 Practice for Reporting Opinions of Scientific or Technical Experts

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

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 Practice for 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

E2881 Test Method for Extraction and Derivatization of Vegetable Oils and Fats from Fire Debris and Liquid Samples with Analysis by Gas Chromatography-Mass Spectrometry

E2997 Test Method for Analysis of Biodiesel Products by Gas Chromatography-Mass Spectrometry

E3189 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Static Headspace Concentration onto an Adsorbent Tube

E3197 Terminology Relating to Examination of Fire Debris

2.2 *NFPA Standards:*[3]

NFPA 921 Guide for Fire and Explosion Investigation

[1] This guide 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] 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] Available from National Fire Protection Association (NFPA), 1 Batterymarch Park, Quincy, MA 02169-7471, http://www.nfpa.org.

2.3 *Other Resources:*

National Center for Forensic Science Ignitable Liquids Reference Collection (NCFS-ILRC) and Substrates Database [4]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this guide, refer to Terminologies E1732 and E3197.

4. Summary of Guide

4.1 This guide describes a systematic approach for the analysis of ignitable liquids and their residues in fire debris or other samples.

4.2 This guide highlights some of the considerations associated with each step in the process: evidence assessment, preliminary screening, sample preparation, instrumental analysis techniques, data analysis and interpretation, reporting, and preservation of evidence.

5. Significance and Use

5.1 This guide is applicable for all ignitable liquids as defined by Test Method E1618.

5.2 This guide is applicable to solid and liquid samples (burned and unburned), which are packaged in a closed vapor-tight container (typically a can, fire debris bag, jar, or vial).

5.3 This guide is to be used in conjunction with the referenced documents by an analyst familiar with the limitations and applicability of each technique used.

5.4 This guide does not attempt to address all the issues regarding sample extraction and analysis. There could be additional laboratory validated or verified tests, extractions or analyses that can be performed to provide further discrimination and classification of ignitable liquids and ignitable liquid residues present in samples.

5.5 This guide is intended to be used in conjunction with generally accepted forensic accreditation requirements including documentation and records.

6. General Analytical Approach

6.1 A general approach for the analysis of ignitable liquids and their residues in fire debris and related samples is outlined in Fig. 1.

6.1.1 Within Fig. 1, reference is made to applicable ASTM documents.

6.2 General approaches to fire debris analysis are extensively covered in textbooks by Bertsch, Holzer, and Sellers (**1**);[5] Almirall and Furton (**2**); Stauffer, Dolan and Newman (**3**); Saferstein (**4**); DeHaan and Icove (**5**); Lentini (**6**); Siegel and Saukko (**7**); Hendrikse, Grutters, Schäfer (**8**); and Houck (**9**).

6.2.1 Review papers on fire debris analysis were compiled by Pert, Baron and Birkett (**10**), Zadora and Borusiewicz (**11**), Sandercock (**12**), Viitala and Kakko (**13**), Viitala and Hyyppä (**14**), Baerncopf and Hutches (**15**), and Stauffer (**16**).

6.2.2 A comprehensive list of references covering all aspects of ignitable liquid and ignitable liquid residue extraction and analysis is available on the Organization of Scientific Area Committees (OSAC) website (**17**).

7. Initial Assessment of Case and Evidence

7.1 Evaluate the case information and the requested examination type(s) to determine whether the evidence can be accepted (Practice E1492) and analyzed within the requested timeline.

7.2 Identify special considerations that can affect the initial approach to the evidence, including the need for multiple analyses, or special safety measures.

7.2.1 Materials submitted for analysis could contain chemical, biological, or physical hazards that require special safety measures.

7.2.2 Consider only relevant information about the case that pertains to the submitted samples and avoid basing interpretations and conclusions on cognitively biasing information, such as an investigator's hypotheses or contextual cues not related specifically to the analysis of ignitable liquids. Examples of contextual cues irrelevant to the analysis include a fatality, the insurance status of a building or the removal of valuable items prior to a fire (**18**).

7.3 Evaluate the evidence to determine whether it is suitable for analysis.

7.3.1 Specific suitability requirements for fire debris include proper vapor-tight packaging and storage (NFPA 921).

7.3.2 Store the evidence in a manner that will avoid contamination and minimize degradation.

7.3.2.1 Items believed to contain substrate materials likely to contribute to the degradation of ignitable liquids, such as vegetation or soil, should be refrigerated or frozen at the laboratory until the time of analysis if the analysis cannot be expedited (NFPA 921) (**19-23**).

7.3.3 Determine if the content of the evidence is in agreement with its description on the accompanying documentation. Document and resolve discrepancies as necessary.

7.3.3.1 Examine the content of the evidence prior to or after analysis.

8. Preliminary Evaluation of Evidence (Screening)

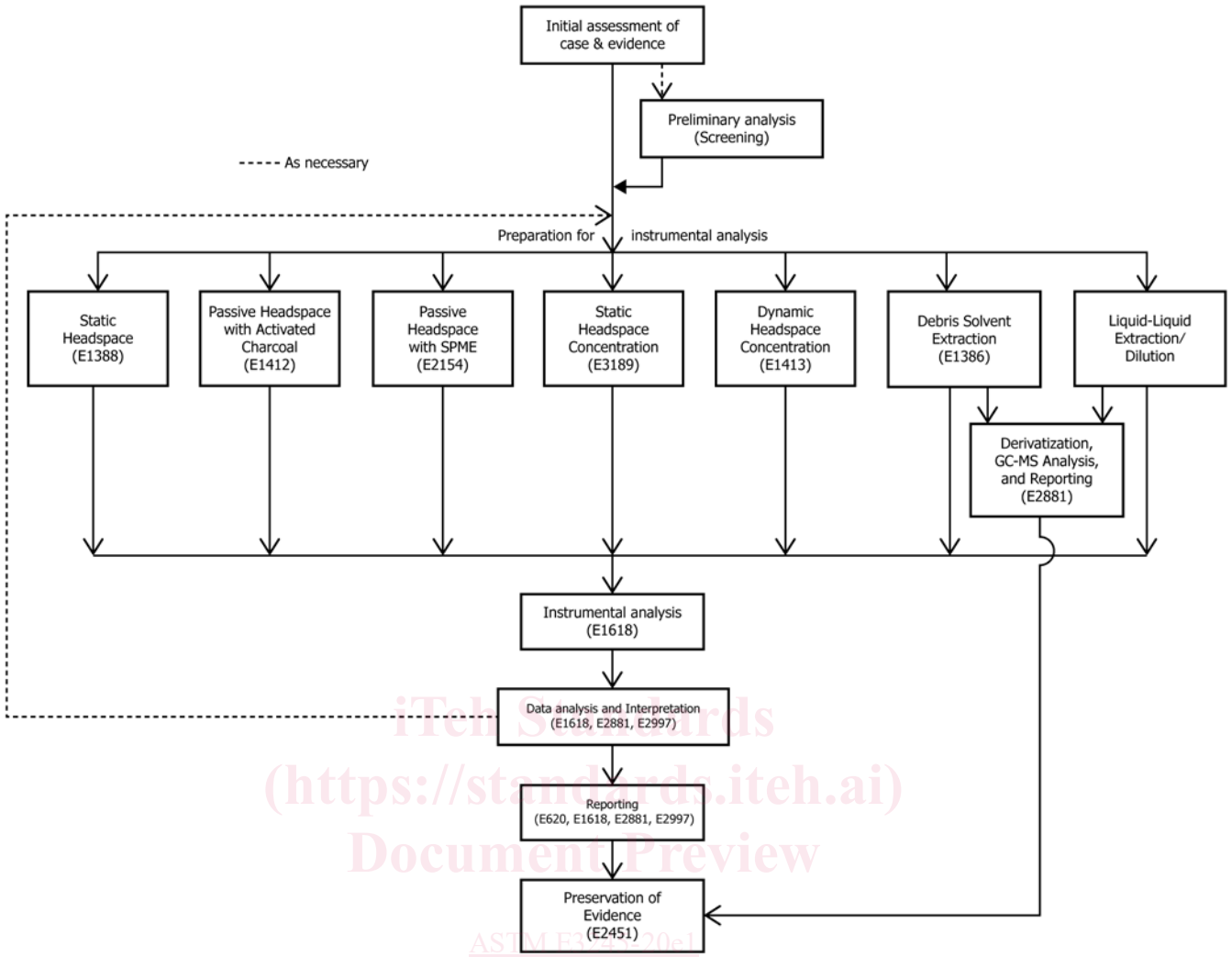
8.1 The preliminary tests listed below are considered optional but can provide an indication as to the presence of an ignitable liquid or ignitable liquid residue, as well as guidance in the selection of the best extraction method(s) for analysis. A preliminary evaluation shall not be used as the sole method to establish a conclusion. Preliminary test results shall be supported by additional analyses as detailed in Section 9.

8.2 *Liquid Samples:*

8.2.1 Liquid samples submitted by fire investigators for analysis or comparison could consist of one or more layers. A portion from each layer should be independently examined by performing one or more of the following tests:

[4] Available from http://ilrc.ucf.edu.

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



NOTE 1—Abbreviated titles for the ASTM documents are provided.

FIG. 1 General Analytical Approach for the Analysis of Ignitable Liquids.

8.2.1.1 *Ignitability*—Test the ignition and burning characteristics at ambient temperature using an open flame. Some of the ignition and burning characteristics that could be of value include the ability to ignite, flame color, and smoke characteristics.

NOTE 1—While flash point (flammability or combustibility) can sometimes be determined using a flash point apparatus, such detailed analyses are not generally required.

8.2.1.2 *Miscibility*—Test the miscibility of the sample with known liquids. Most organic liquids (which includes petroleum products) are immiscible with water. Many common oxygenated compounds are miscible with water.

8.2.1.3 *Other Tests*—Additional types of testing can be useful in characterizing an unknown liquid. These could include pH of aqueous liquids, physical properties, spectroscopic techniques, determination of the presence of water using anhydrous copper(II) sulfate or cobalt(II) chloride powder or test paper, and other tests deemed necessary.

8.2.2 Liquid samples can be screened for ignitable liquid residues using static headspace (Practice E1388) and gas chromatography with flame ionization or mass spectrometry detection.

8.2.2.1 Use of a medium polarity GC column provides the ability to screen for a wide range of compounds with different polarities (from polar to non-polar).

8.3 *Debris Samples (Burned and Unburned):*

8.3.1 Most samples submitted for analysis by fire investigators are in the form of fire debris collected in the course of scene investigations. These samples could exhibit various states of charring, destruction, or decomposition resulting from fire damage, fire suppression, and packaging. Ignitable liquid residues, if present, could be in very low or very high concentrations. Fire debris samples can be screened to detect ignitable liquid residues using static headspace (Practice E1388) and gas chromatography with flame ionization or mass spectrometry detection.

8.3.1.1 The initial use of a medium polarity GC column provides the ability to screen for a wide range of compounds with different polarities (from polar to non-polar).

8.4 Follow health and safety precautions when handling evidentiary materials.

8.4.1 Human olfactory analysis can pose a health risk and is not recommended. Unavoidable odors should be noted, as they could assist in the selection of an extraction scheme.

9. Sample Preparation for Instrumental Analysis

9.1 There is no single extraction technique or set of parameters within a technique that is ideal for all types of samples (**24, 25**). Some samples require more than one extraction or analytical evaluation before a conclusion about the classification of the ignitable liquid or ignitable liquid residue can be made. For this reason, select the least destructive procedures first.

9.2 *Liquid Samples:*

9.2.1 *Static Headspace Sampling (Practice E1388)*—Static headspace sampling is useful for highly volatile liquid samples, and for aqueous liquid samples with small quantities of ignitable liquid residues. This technique does not recover compounds of lower volatility.

9.2.2 *Direct Injection*—Direct injection can provide a more accurate representation of a liquid's components. (**Warning—** The volume and concentration of an ignitable liquid specimen injected and the instrumental conditions affects the GC-MS data.)

9.2.3 *Simple Dilution*—Simple dilution is the most common technique for preparation of organic liquid samples. A dilute solution in a miscible organic solvent is generally recommended. Select a solvent that will not mask the compounds of interest.

9.2.4 *Liquid-Liquid Extraction*—Liquid-liquid extraction is a technique used to extract ignitable liquid residues from aqueous samples. An appropriate organic solvent, such as pentane, carbon disulfide, diethyl ether or dichloromethane, is added to a portion of the aqueous layer of the sample and agitated. Organic products and other compounds preferentially soluble in the chosen solvent are extracted into the organic phase. The organic layer is then isolated, concentrated if necessary, and analyzed. Select a solvent that will not mask the compounds of interest.

9.2.4.1 Liquid-liquid extraction and derivatization are also used for preparation of specimens for the analysis of fatty acids commonly found in vegetable oils and animal fats (Test Method E2881).

9.2.5 *Passive Headspace Concentration (Practice E1412)*—Passive headspace concentration (PHC) using an adsorbent, such as activated carbon, can be used to extract portions of liquid samples with compounds having a broad range of boiling points. Depending on the adsorbent used, PHC is amenable to extract preservation. Disadvantages could include displacement of ignitable liquid compounds and limited recovery of compounds with carbon number greater than n-C_{16}.

9.2.6 *Static Headspace Concentration (Practice E3189)*—Static headspace concentration using an adsorbent, such as activated carbon or Tenax[6] TA, can recover compounds having a broad range of boiling points. One disadvantage could include limited recovery of compounds with carbon number less than n-C_6 and greater than n-C_{16}, depending on conditions.

9.2.7 *Dynamic Headspace Concentration (Practice E1413)*—Dynamic headspace concentration using an adsorbent, such as activated carbon, can recover compounds having a broad range of boiling points. Dynamic headspace concentration is amenable to extract preservation. Disadvantages could include displacement of ignitable liquid compounds and breakthrough.

9.2.8 *Solid Phase Microextraction (Practice E2154)*—Solid phase microextraction (SPME) can be used for headspace concentration and sampling compounds having a broad range of boiling points. SPME is not amenable to extract preservation. Disadvantages could include displacement of ignitable liquid compounds and limited recovery of compounds with carbon number greater than n-C_{16}.

9.3 *Debris Samples (Burned or Unburned):*

9.3.1 *Static Headspace Sampling (Practice E1388)*—Static headspace analysis can provide the analyst with preliminary information concerning the detection of an ignitable liquid or ignitable liquid residue, its classification, and its relative concentration. It is most appropriate for the analysis of very volatile compounds such as ethers or alcohols. The disadvantages of this technique include limited sensitivity and the inability to recover less volatile compounds. This technique provides no mechanism for extract preservation.

9.3.2 *Passive Headspace Concentration (Practice E1412)*—Passive headspace concentration (PHC) using an adsorbent, such as activated carbon, can be used to extract debris samples with compounds having a broad range of boiling points. Depending on the adsorbent used, PHC is amenable to extract preservation. Disadvantages could include displacement of ignitable liquid compounds, competitive adsorption of compounds of differing chemical properties with the substrate, and dependent on conditions, limited recovery of compounds with carbon number greater than n-C_{16}.

9.3.3 *Static Headspace Concentration (Practice E3189)*—Static headspace concentration using an adsorbent, such as activated carbon or Tenax TA, can recover compounds having a broad range of boiling points. Disadvantages could include competitive adsorption of compounds of differing chemical properties with the substrate and limited recovery of compounds with carbon number less than n-C_6 and greater than n-C_{16}, depending on conditions.

9.3.4 *Dynamic Headspace Concentration (Practice E1413)*—Dynamic headspace concentration using an adsorbent, such as activated carbon, can recover compounds having a broad range of boiling points. Dynamic headspace concentration is amenable to extract preservation. Disadvantages could include displacement of ignitable liquid compounds, breakthrough, and competitive adsorption of compounds of differing chemical properties with the substrate.

9.3.5 *Solid Phase Microextraction (Practice E2154)*—Solid phase microextraction (SPME) can be used for headspace

[6] A trademark of Teijin Carbon America, Inc., Rockwood, TN.