Title: Examination of Fire Debris Evidence

1 Introduction

The analysis of fire debris evidence for the presence of ignitable liquids (whether residual or bulk) can play a large role in law enforcement investigations. The detection of such compounds at a fire scene does not necessarily lead to a conclusion that a fire was incendiary in nature. Certain chemicals and types of mixtures such as gasoline, fuel oils, charcoal lighter fluids, and paint thinners can be used to accelerate or help ignite and propagate fires.

The term ignitable liquid (IL) refers to a set of substances which fall within certain commonly accepted classification groups of chemicals and/or mixtures. While certain liquids may, in fact, be ignited under certain parameters or within particular environments (e.g., candle wax, motor oils), they are outside the ASTM E1618 ignitable liquid classification scheme used within this discipline and will not be included within the ignitable liquid definition. Ignitable liquids are often mixtures that contain large numbers of hydrocarbon constituents. Gasoline, for example, has over 400 chemical constituents and can be considered one of the more complex ignitable liquids. The collection and concentration of ignitable liquids takes advantage of the volatility of their constituents. Charcoal (carbon) adsorbent material is typically used in conjunction with a heated container to capture such chemical constituents. Volatile chemicals are adsorbed onto the charcoal and then eluted with a solvent for further analysis.

Detection limits, volatility of liquid residues, and variations of sampling techniques/methods can influence detectability. Difficulties in identifying ignitable liquids arise from their complex composition, from matrix interferences caused by materials which contain ignitable liquids, and from the loss of volatile constituents due to weathering or other factors. Because most evidence is post-burn, very small amounts of ignitable liquids are trying to be detected – hence the term ignitable liquid residue (ILR). Analysis of materials for ILR is generally performed using gas chromatography-mass spectrometry (GC/MS). A technique within mass spectrometry called extracted ion profile analysis can be used to compare chemical data to reference materials and standards for identification purposes. Such an identification can be considered a characterization of a material based upon physical or chemical properties that sufficiently differentiates that material from similar substances.

2 Scope

The following procedure provides general guidelines for the examination, extraction, and detection of ignitable liquids (ILs) or ignitable liquid residues (ILRs) within evidentiary materials. There are seven major classes of ignitable liquids (and one miscellaneous/other class) that chemicals may be identified as being part of by using gas chromatography, mass spectrometry, extracted ion profiling, or a combination thereof within recovered fire debris evidence (or related materials). The scope of this procedure will focus on the following classifications: gasoline, petroleum distillates, isoparaffinic products, aromatic products, naphthenic-paraffinic products, normal-alkane products,

oxygenated solvents, and miscellaneous (others-miscellaneous). Authorized analysts who conduct such examinations within the fire debris category of testing are responsible for ensuring this procedure is applied to applicable evidence and that results are placed within final reports

Certain low molecular weight chemicals (e.g., alcohols) may only be detected when in sufficient quantity and not at the residue level using this procedure.

3 Principle

The techniques employed within this procedure utilize physical examination, extraction, and instrument analysis to determine whether ILs or ILRs are present within evidentiary material. In general, analytes are often captured and isolated by heating evidence within a container's headspace, trapped onto highly adsorbent material, extracted using an elution solvent, and then analyzed using instrumentation (e.g., GC/MS). Additionally, evidence may also be examined without the use of an adsorbent material and may warrant dilution prior to analysis (e.g., static headspace sampling, liquid dilution, etc.). When necessary, and if appropriate, alternate instrumental techniques can be used for analysis (e.g., infrared spectrophotometry) even though they may not be specifically described in this procedure. Use of other unit/section procedures is allowed with reasonable modifications and if approved by the unit's FSE2 or higher.

4 Samples

Any type of material related to fire debris evidence should be able to be analyzed using this procedure. In order for heated headspace-type analyses to be performed, materials need to be able to fit into containers that can be properly sealed and heated (e.g., metal cans or pails). Factory sealed containers of suspected ignitable liquids do not necessarily need to be submitted within an airtight evidence container for analyses to be performed. Evidence submitted which has not been stored within airtight/appropriate containers may need to be returned to submitting agencies due to the potential for contamination or loss of sample.

5 Equipment/Materials/Reagents

a. General laboratory glassware (reusable and disposable) and equipment (e.g. pipet bulbs)

b. Gas chromatograph/mass spectrometer (GC/MS) (Agilent or equivalent)

c. Autosampler (Agilent or equivalent)

d. GC column – Agilent DB-5(MS), 30 m, 0.25 mm i.d., 0.25 μm film (or equivalent)

e. Heating mantles (capable of continuously heating pint, quart, and gallon cans within the range of 60-80°C)

f. Oven (capable of continuously heating multiple cans within the range of 60-80°C)

g. Thermometers (capable of measuring ~60-80°C, NIST Traceable or verified)

h. Vacuum source (mechanical pump or equivalent)

i. Cans (unused metal pint, quart, or gallon cans, and five gallon pails)

j. Modified can/pail lids and fittings (e.g., Swagelok) to accommodate charcoal tubes

k. Pentane (reagent grade or equivalent)

l. Acetone (reagent grade or equivalent)

m. Isopropanol (reagent grade or equivalent)

n. ASTM E1618 test mixture (Restek or equivalent)

o. Reference standards/products (e.g., certified reference materials or consumer/commercial products)

p. Activated charcoal (carbon) tubes (SKC or equivalent)

q. Activated charcoal (carbon) strips (Albrayco or equivalent)

r. Gases (e.g., nitrogen, helium, air)

s. Syringes (disposable or reusable, gas-tight, capable of sampling/injecting at least 0.5 cc (0.5 mL) of headspace)

t. Autosampler vials, caps, and racks

u. Glass vials (e.g., 4 mL) and caps (air-tight)

v. Adhesive tape

w. Paperclips, tweezers, and scissors

x. Nylon string (e.g., un-waxed nylon dental floss)

y. Paint can key (or flat-head screwdriver), diagonal cutter (or similar pliers), hammer (or mallet), awl (or punch), and any other appropriate tools

z. Nylon bags

6 Standards and Controls

The stability of any reference standards/products should be determined by the manufacturer (e.g., expirations dates). If stability information is not available, then reference standards/products, when stored appropriately, will be valid for five years. Note: reference standards/products older than five years and with unknown stability information may still be used if their ignitable liquid classification, upon reanalysis, remains consistent/unchanged. Once diluted or prepared beyond their manufactured state, any solutions derived from reference standards/products are valid for one year from the date of solution preparation. All reagents, reference standards/products, and solutions thereof will be verified prior to use in casework analysis. Note: verification will consist of a successful analysis displaying expected instrumental data (e.g., chromatogram(s), mass spectral data, etc.) and exhibiting no unacceptable extraneous compounds. Appropriate positive and negative controls will

be used with each sample or batch of samples. Appropriate reference standards/products will be used for casework comparison and/or ignitable liquid class identification.

a. Positive Control

b. A positive control is used to demonstrate that the technique(s) selected for casework analysis is able to extract and recover the expected compounds/analytes of interest. Typically, a neat (undiluted) aliquot of a reference standard/product is used when preparing a positive control. A positive control is performed using the same apparatus/equipment/materials specific to the technique selected for casework analysis.

c. Negative Control

A negative control is used to demonstrate that the technique(s) selected for casework analysis has not contributed any contamination or interfering compounds. A negative control is performed using the same apparatus/equipment/materials specific to the technique selected for casework analysis. A negative control will also be subjected to any additional preparation/dilution used for casework analysis (e.g., if sample extracts are concentrated under nitrogen, then the negative control extract associated with those samples will be concentrated and analyzed in the same manner).

d. Reference Standards/Products

A reference standard/product is analyzed to make a comparison or ignitable liquid class identification. In order to make an ignitable liquid class identification a reference standard/product of the same class as the casework sample will be analyzed. A class identification does not require that the reference standard/product have the exact same pattern/profiles/ratios as the casework, but every attempt should be made to analyze and/or obtain a comparable reference standard/product. Typically, a reference standard/product is diluted prior to analysis with the same solvent used during elution/extraction (e.g., pentane), but may differ depending on the technique used (e.g., a single aliquot of acetone used as a positive control and standard, manually injected during static headspace sampling). Reference standards/products will be verified and deemed acceptable prior to being included with casework.

e. Blanks

A blank is used to demonstrate that there is no carry-over contamination in-between samples (or negative control and sample pairings) during the instrumental analysis. Typically, a blank is simply a clean representative aliquot of the extraction/elution solvent (e.g., pentane), but may differ depending on the technique used (e.g., room air manually injected during static headspace sampling). A blank is analyzed before and between each sample. A blank will also precede a reference standard/product that is analyzed for comparison or identification and will bracket it if another sample is to follow. Note: for some instrumentation a blank may not be necessary (e.g., FTIR).

7 Sampling

All submitted evidence will typically be analyzed and sampling is not applicable. However, if multiple items from a single case are submitted and a positive IL/ILR result is obtained from an item/items, the analyst may inform the FSE2 or higher and contact the submitter to possibly adjust the number of remaining items to be analyzed. Additionally, at any point in the process, the analyst, in conjunction with the FSE2 or higher, may elect to contact the submitter for further clarification regarding the items submitted and/or the requested examination.

8 Procedure

a. Initial Examination

i. Take custody of any evidence to be examined and create any appropriate sub-items within LIMS. During examination, if evidence is to be removed from its packaging, it is not to come into contact with surfaces that have not been previously cleaned with isopropanol and allowed to fully dry and also covered with an appropriate barrier layer (e.g. craft paper) to prevent contamination. When handling evidence directly, use only clean tools/materials and clean disposable gloves. Clean any reusable tools (e.g., paint can key, tweezers, scissors, etc.) with acetone and allow to fully dry between uses. Collect any waste acetone from cleaning and dispose of properly. Change out the barrier layer and clean the examination surface with isopropanol between cases. Change disposable gloves and use clean disposable glassware/materials when working directly with different evidence/samples, controls, and reference standards/products, and when the potential for cross-contamination exists.

ii. Perform a visual examination of the evidence, taking photographs to capture the condition of the evidence, its packaging, and any relevant markings or labels. Initial photographs of the exterior of evidence packaging should be taken prior to the evidence being opened or unsealed. Minimize the time that evidence containers are open and unsealed in order to limit the loss of volatile analytes.

iii. Presence of odors within evidence:

- Always work in well ventilated area, utilizing fume hoods as much as possible.
- Wafting evidence to evaluate the presence of odor is not recommended due to the nature of potentially hazardous chemicals within fire debris samples. However, ignitable liquid-like odors may be observed and noted.
- The observation of ignitable liquid-like odors may influence the recovery method choice.

iv. Record in LIMS or on worksheets a description of the evidence and any other appropriate observations or characteristics.

v. If evidence requires additional forensic analyses (e.g., latent prints, DNA, etc.), the nature of the evidence and the type of analyses will be considered before examination. Typically,

fire debris examination is performed once initial examination/collection for any additional forensic analyses is complete to prevent any potential deleterious effects.

b. Recovery & Analysis

- Record in LIMS or on worksheets the technique(s) used along with any additional preparation (dilution, concentration, etc.) performed and any parameters used outside of those listed in this procedure. Create any appropriate sub-items (e.g., extracts) within LIMS.
- All instrumentation will be successfully evaluated for use and deemed acceptable for analysis prior to being used for casework.
- Passive headspace concentration is utilized for routine examination. If an analyst is unable to make a determination or identification regarding the presence of an ignitable liquid residue following passive headspace concentration, then that item will be examined via dynamic headspace concentration.
- Static headspace sampling is utilized when alcohols or other ignitable liquids comprised primarily of compounds with retention times occurring before the GC/MS solvent delay are suspected.

i. Passive Headspace Concentration - Extraction and Elution:

A. Control cans (or appropriate containers) will be used with each group (batch) of evidence cans extracted on a given day. Prepare negative control cans first and positive control cans last. Prepare a negative control can by lining the bottom of a clean can with tissue paper or other appropriate contamination-free media (e.g., Kimwipes, barrier layer, craft paper). Prepare a positive control can by lining the bottom of a clean can with the same media used in the negative control can and then adding ~1 drop of an appropriate ignitable liquid product (e.g., gasoline). Finish preparing the control cans in the same manner as any evidence cans.

B. Open only one evidence can at a time.

C. Secure a piece of nylon string across the opening of the can, suspending a paperclip holding (2) activated charcoal strips. Adhesive tape should be used to secure the ends of the nylon string to the outside of the can. Suspend the paperclip and strips within the headspace of the can, above the can contents, and ~1 inch from the underside of the can lid. Do not allow the charcoal strip to contact the contents or surfaces of the can. Note: if there is insufficient headspace to properly suspend the activated charcoal strips, it may be necessary to split a portion of the can contents into a clean, unused can that will also be extracted along with the primary can and in the same manner. An analyst may elect to perform this technique with less than (2) activated charcoal strips.

D. For evidence in nylon bags, cut open the bag and transfer the contents to a clean and unused metal can. Prepare the can as described above.

E. Using a hammer, fully secure the lid of the can so that it is flush with the can body. Do not strike or damage the protruding nylon string.

F. Heat the cans in an oven at ~60ºC for 16-24 hours. Record in LIMS or on worksheets the heating temperature used along with the beginning and ending date and time of the extraction. Note: an analyst may elect to perform this technique for a shorter duration or without using an oven (i.e., at room temperature) if ignitable liquid-like odors are observed upon opening the evidence or other conditions exist.

G. After heating, turn off the oven and allow the cans to cool to the touch (approximately 15-30 minutes).

H. Remove the cans from the oven, and one at a time, open a can within a fume hood, remove its activated charcoal strips, and reseal the can.

I. Place one of the activated charcoal strips in a properly labeled glass vial for archiving/preservation (i.e., return to submitter), cap, and seal. Place the second activated charcoal strip in another properly labeled glass vial for elution.

J. Using a disposable glass pipet, add ~1mL of pentane to the elution vial. Slowly rinse the activated charcoal strip at least (3) times with the pentane aliquot.

K. Transfer an appropriate aliquot of the pentane extract to an insert within a properly labeled GC/MS autosampler vial and then cap the vials. Note: an analyst may opt to initially dilute an aliquot of the pentane extract with additional pentane, in which case a vial insert may not be needed. Record in LIMS or on worksheets any initial dilutions performed.

L. Pentane extracts are analyzed along with all controls, blanks, and reference standards/products via GC/MS and an appropriate method.

ii. Static Headspace Sampling:

A. Control cans will be used with each group of evidence cans sampled on a given day. Prepare and sample negative control cans first and positive control cans last. Prepare a negative control can by lining the bottom of a clean can with tissue paper or other appropriate contamination-free media. Prepare a positive control can by lining the bottom of a clean can with the same media used in the negative control can and then adding ~1 drop of an appropriate ignitable liquid product. Finish preparing the control cans in the same manner as any evidence cans.

B. Prepare and sample only one evidence can at a time. Note: if there in insufficient headspace, it may be necessary to split a portion of the can contents into a clean, unused can that will also be extracted along with the primary can and in the same manner.

C. Using an awl, along with a hammer (if needed), punch an appropriately sized hole in the lid of the can. Immediately cover the hole with a piece of adhesive tape.

D. Heat the can for an appropriate duration (e.g., 1-10 minutes) utilizing either an oven or heating mantle. Record in LIMS or on worksheets the duration and the temperature of the heating device. Note: an analyst may elect to perform this sampling technique without using a heating device (i.e., at room temperature) if ignitable liquid-like odors are observed upon opening the evidence or other conditions exist.

E. Remove the can from the heating device and immediately sample an appropriate volume (e.g., 0.5 mL (0.5 cc)) of the interior headspace of the can using a syringe (disposable or clean). Note: it is suggested that the sampling be performed as close to the GC/MS instrument used for analysis as possible.

F. Utilizing an appropriate manual injection method, inject the sampled headspace into the GC/MS and immediately start the analysis.

G. Reseal the hole used for sampling using a piece of evidence tape.

H. Analyze all controls, blanks, and reference standards/products via GC/MS using the same manual injection method.

iii. Dynamic Headspace Concentration – Extraction and Elution:

A. Control cans will be used with each group of evidence cans sampled on a given day. Prepare and sample negative control cans first and positive control cans last. Each evidence can to be extracted will have its own negative control can directly preceding it (i.e., a negative control will be extracted prior to the extraction of each evidence can). The analysis of a negative can prior to the extraction of each evidence can demonstrates that the reusable can lid and fittings are contamination free and that the process and extraction environment did not introduce contamination. Prepare a negative control can by lining the bottom of a clean can with tissue paper or other appropriate contamination-free media. Analyze each negative control and only extract its associated evidence can once the negative control is deemed acceptable. Note: the reusable can lid and fittings need not be cleaned after the negative control, assuming that the negative control is acceptable upon analysis. Prepare a positive control can by lining the bottom of a clean can with the same media used in the negative control can and then adding ~1 drop of an appropriate ignitable liquid product. Finish preparing the control cans in the same manner as any evidence cans.

B. Check the outside of the can for dirt, debris, or other potential sources of contamination and wipe down the can if necessary using tissue paper or other appropriate contamination-free media.

C. Open only one evidence can at a time.

D. Using an awl, along with a hammer (if needed), punch 2-4 appropriately sized holes around the base of the can. Note: if standing liquid is present inside the can, punch the holes ~1 inch above the level of the liquid.

E. Replace the original can lid with a clean compatible lid modified with appropriate

fittings to hold an activated charcoal tube. Note: if there is insufficient headspace to properly secure the modified lid and fitting assembly to the can, it may be necessary to split a portion of the can contents into a clean, unused can that will also be extracted along with the primary can and in the same manner.

F. Using a diagonal cutter, snip the ends off of an activated charcoal tube and place it into the fitting (oriented correctly based upon the flow direction indicated on the activated charcoal tube). Note: air will flow in through the can and be drawn towards the vacuum pump.

G. Connect the vacuum hose to the exposed end of the activated charcoal tube and turn on the vacuum pump.

H. Place the can into the appropriately sized heating mantle at ~80°C. Heat the can for an appropriate amount of time or until the lid of the can is hot to the touch. Note: liquid condensation appearing within the charcoal tube may be indicative of sufficient heating of the can contents. Record in LIMS and on worksheets the heating temperature and duration. Note: an analyst may elect to perform this technique for a shorter duration or without using a heating mantle (i.e., at room temperature) if ignitable liquid-like odors are observed upon opening the evidence or other conditions exist.

I. Remove the can from the heating mantle and disconnect the vacuum hose from the activated charcoal tube. Note: leave the vacuum pump on until after elution to allow the pump internals to completely dry.

J. Remove the activated charcoal tube from the fitting and place it into a clean glass vial in the same orientation as in the fitting (i.e., flow direction pointing up).

K. Using a glass pipette (disposable or clean), add ~1 mL of pentane through the top of the activated charcoal tube, allowing the pentane to flow through and collect in the glass vial. Note: a small amount of pressure may be needed to force the pentane through the activated charcoal tube and this can be achieved by squeezing a pipet bulb directly on the top of the activated charcoal tube.

L. Transfer an appropriate aliquot of the pentane extract to an insert within a properly labeled GC/MS autosampler vial and then cap the vials. Note: an analyst may opt to initially dilute an aliquot of the pentane extract with additional pentane, in which case a vial insert may not be needed. Record any initial dilutions performed.

M. Pentane extracts are analyzed along with all controls, blanks, and reference standards/products via GC/MS and an appropriate method.

N. Disassemble the modified can lid and fittings placing the fitting components in a glass beaker. Thoroughly clean the fitting components by adding enough acetone to the beaker to fully submerge the fitting components, swirl to clean. Thoroughly clean the bottom of the modified can lid by rinsing it with acetone. Allow the

modified can lid and fittings to fully dry between uses.

iv. Solvent Extraction

A. Controls will be used with each group of evidence cans sampled on a given day. Prepare and sample negative controls first and positive controls last. Each evidence can to be sampled from will have its own negative control associated with it (i.e., a negative control will be extracted prior to the extraction of the evidence sample). Prepare a negative control by selecting a clean, unused container of sufficient volume to accommodate the evidence to be sampled (e.g., can, pail, glass test tube, etc.). Analyze each negative control and only sample its associated evidence can once the negative control is deemed acceptable. Note: the same container used for the negative control will be used for its associated evidence sample, assuming that the negative control is acceptable upon analysis. Prepare a positive control by adding ~1 drop of an appropriate ignitable liquid product to the bottom of a clean container. Finish preparing the controls in the same manner as any evidence cans.

B. Open only one evidence can at a time.

C. Using a clean pair of gloves, or appropriate tools (e.g., tweezers), sample an appropriate and representative portion of the contents of the evidence can and place this portion into the same container previously used for the associated negative control.

D. Using glass pipet (disposable or clean), rinse the portion of evidence with an appropriate amount of pentane, allowing the pentane to collect within the can.

E. Transfer the pentane extract to a properly labeled vial. Note: an analyst may opt to filter the pentane extract due to the presence of sediment/turbidity.

F. Transfer an appropriate aliquot of the pentane extract to an insert within a properly labeled GC/MS autosampler vial and then cap the vials. Note: an analyst may opt to initially dilute an aliquot of the pentane extract with additional pentane, in which case a vial insert may not be needed. Record any initial dilutions performed.

G. Pentane extracts are analyzed along with all controls, blanks, and reference standards/products via GC/MS and an appropriate method.

v. Liquids

A. A negative control will be used with each group of evidence samples on a given day. Prepare a negative control by adding an aliquot of the same lot of pentane that will be used for evidence sample dilution to a properly labeled GC/MS autosampler vial and cap.

B. Using a glass pipet (disposable or clean), add ~1 drop of the liquid evidence sample to a properly labeled vial and dilute to ~4 mL with pentane. Cap and invert to mix several times. Note: an analyst may opt to alter the initial dilution factor.

C. Transfer an appropriate aliquot of the diluted liquid sample to an insert within a properly labeled GC/MS autosampler vial and cap. Note: a vial insert may not be needed depending on the initial dilution factor used.

D. Pentane dilutions are analyzed along with all controls, blanks, and reference standards/products via GC/MS and an appropriate method.

E. Other instrumentation (e.g., FTIR) may be utilized in addition to GC/MS. Typically, an undiluted (neat) aliquot of a liquid evidence sample is analyzed directly on an FTIR (refer to any applicable SOPs within the section for FTIR analysis).

c. Record in LIMS and/or on worksheets any additional preparatory information not captured within this procedure.

i. Reagents and steps not explicitly stated in this procedure will be recorded in LIMS and/or on worksheets.

ii. Lot numbers of solvents, standards/products, and other appropriate materials will be recorded in LIMS and/or on worksheets.

d. Sample Archival/Preservation

i. Passive Headspace Concentration:

Any un-eluted activated charcoal strips associated with evidence will be returned to the submitter. Note: in the absence of un-eluted activated charcoal strips, the eluted charcoal strips will be returned to the submitter. In the absence of charcoal strips, any pentane extracts will be returned to the submitter. Aliquots analyzed by GC/MS do not need to be returned to the submitter unless they are the only remaining volume of the extract.

ii. Dynamic Headspace Concentration:

Any pentane extracts associated with evidence will be returned to the submitter. Note: aliquots analyzed by GC/MS do not need to be returned to the submitter unless they are the only remaining volume of the extract.

iii. Any vials/containers to be returned to the submitter will be properly capped and sealed.

9 Instrumental Parameters

The following are the typical GC/MS operating parameters for this procedure. With approval from the FSE2 or higher, these instrument conditions may be modified to adjust or improve the

procedure. Documentation of these parameters should be included with sequence data so that any instrumental parameter changes may be recorded in case files.

GC Oven:

Initial Temp	40°C
Initial Time	2.0 min.
Ramp	10°C/min.
Final Temp	300°C
Hold Time	12.5 min.
Equilibration Time	0.5 min.

GC Inlet/Injector:

Injector Volume	1 µL
Mode	Splitless
Inlet Temp.	250°C

GC Column:

Type	HP-5MS (or equivalent)	Mode	Constant Flow
Length	30 m	Flow	0.9 mL/min.
Diameter	0.25 mm	Carrier Gas	Helium (He)
Film Thickness	0.25 µm		

Mass Spectrometer:

Transfer Line	300°C
Source	230°C
Quadrupole	150°C
Low Mass	40
High Mass	400

10 Decision Criteria

a. Evaluation of Data

i. The total ion chromatogram (TIC) and associated spectra for each sample are reviewed using appropriate GC/MS software. Each significant chromatographic peak should be reviewed to determine if it is an analyte of interest.

ii. Reference standards/products that have been previously analyzed (i.e., not contemporaneously with sample analysis) may still be used for comparison purposes if no significant instrument/method changes (e.g., column clipping, column change, temperature program change, etc.) have occurred between analyses. Significant changes would be those that may cause complete loss of analyte recovery or retention time shift beyond acceptability.

iii. Preliminary identification of an analyte or classification can be achieved by operator recognition of chromatographic patterns, profiles, abundancies, and retention times, by utilizing instrumental library mass spectral information, by the use of accepted methods of evaluation (e.g., ASTM guidelines), or a combination thereof.

iv. A full identification occurs when the sample analytes are compared to a reference standard/product and the following criteria are met:

A. Chromatography

Retention Times: Analyte peak retention times are within +/- 5% between the sample and the reference standard/product.

Profile: The overall chromatographic profile pattern of the sample should compare favorably to the chromatographic profile pattern of the reference standard/product. While pattern matching of extracted ion profiles rarely gives perfect correlation with reference standards/products, some correlation should be present.

Ratios: Based on accepted/published criteria (e.g., ASTM guidelines), certain analyte and analyte group peak abundance ratios within samples should be present and compare favorably with those within reference standards/products.

B. Mass Spectrometry

1. Mass spectra of samples will compare favorably to mass spectra from reference standards/products. Spectra may also compare favorably to known library spectra, although library spectra may have been produced under different mass spectral parameters.

2. Mass spectral reference libraries (e.g., Wiley, NIST, etc.) will be used during the evaluation process. An internal library may be made of substances analyzed in-house and may not be in published libraries. Entries within an in-house library should be compared to published spectra from reliable sources prior to use in casework.

v. Acceptance of GC/MS Data:

A. Blanks

1. The blank prior to the injection of the sample must not contain significant unexplainable peaks. However, due the nature of the type of samples analyzed, certain substances may remain within a GC flow path even after the method has completed, and thereby elute after multiple injections have been performed. If retention times differ significantly, then column carryover is likely the issue (as opposed to injection carryover) and data may still be acceptable. Consultation with an FSE2 or higher should occur in these situations when considering acceptance of data.

2. If the analytes being identified in a sample are also present in the proceeding blank, then the peak abundancy of the analytes in the sample should be ten-times (10x) greater in height than the peak abundancy of the same analytes in the proceeding blank.
3. When it is known that there is a carry-over issue with a sample, then multiple blanks may be injected to minimize the carryover and only the blank directly proceeding the next sample needs to be evaluated.

B. Controls

1. Negative controls should be free of analytes of interest and no significant unexpected peaks should be present. Explainable peaks will be acceptable. If the analyte(s) being identified in a sample is also present in the negative control, then the peak abundancy of the analyte(s) in the sample should be ten-times (10x) greater in height than the peak abundancy of the same analyte(s) in the proceeding blank.
2. Positive control analytes must have acceptable chromatographic and mass spectral data.
3. Reference standard/product analytes must have acceptable chromatographic and mass spectral data.
4. Unexpected results within positive or negative control data should be reported to the FSE2 or higher for evaluation.

vi. In cases where identifications cannot be made due to the lack of obtaining a suitable reference standard/ product, then indications can be made, if appropriate (e.g., based on mass spectral library data). Consultation with the FSE2 or higher will occur in such situations.

vii. Documentation

A. Appropriate instrumental printouts, including reagent blanks and controls, will be included in the case file such that an independent reviewer would be able to readily interpret analyst conclusions.

B. All appropriate chromatograms will be retained in case files. Label the chromatographic peaks for all analytes being identified.

C. All identified analytes will have documented spectra and appropriate comparative reference spectra (e.g., from reference standards/products and/or spectral libraries) within the case files.

D. In samples where no analytes are identified, the analyst should include the TIC along with any appropriate extracted ion chromatograms (EICs). If there are significant peaks that cannot be identified, or attributed to common artifacts, then such spectra should be included in the case file.

E. Instrumental data is considered significant if, by not including such data in a case file, it would be detrimental to the quality of the examination/results.

Exceptions may be made based on the analyte/mixture in question. For example, some weathered gasoline samples may not exactly compare in all aspects to pure gasoline and analyst experience/knowledge may factor in to decision making.

11 Calibration

Not applicable.

12 Uncertainty

Not applicable – this is a qualitative procedure only.

13 Safety

Appropriate standard safety procedures will be followed, including the use of proper personal protective equipment (e.g., gloves, safety glasses, lab coats, etc.). Biological specimens will be handled using universal precautions and will be treated as biohazardous. Potentially contaminated items and surfaces will be cleaned and/or disinfected prior to use.

14 References

a. ASTM Standards (e.g., E1618, E1412, E1413, E1388, E1386, E2451, etc.)

b. Hendrikse, J., Grutters, M., Schäfer, F. *Identifying Ignitable Liquids in Fire Debris. A Guideline for Forensic Experts*. Academic Press, 2016

c. Stauffer, E., Dolan, J.A, Newman, R., *Fire Debris Analysis*, Academic Press, Burlington, MA, 2008.