



Laboratory Services

Fire Debris

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1. Scope

- 1.1. The policy and procedure establishes a standard approach for both debris and liquid samples submitted for ignitable liquid analysis.
- 1.2. The standard approach serves as a general outline for a sequence of examinations for the analysis of fire debris.
- 1.3. Many factors, including the observations of the debris samples submitted, allow the skilled ATF Fire Debris Examiner to enter the various protocols at alternate points. Not all of the tests listed in the scheme will be required for every examination and other unlisted tests may be applicable.
- 1.4. The methods of analysis listed in the standard approach are the standard for all ATF Examiners performing Fire Debris Analysis.

2. Procedure

- 2.1. Refer to Appendix A for a flow chart on liquid sample analysis and Appendix B for a flow chart on debris analysis.
- 2.2. Initial Evidence Examination
 - 2.2.1. Perform macroscopic/visual inspection of the debris or liquid sample.
 - 2.2.2. Confirm that the contents match the container labeling and/or transmittal documentation. Document any discrepancies.
 - 2.2.3. Liquids
 - 2.2.3.1. Note and document the physical properties of the sample such as color, clarity, viscosity, and obvious odors.
 - 2.2.3.2. If only one layer is present, the layer is assumed to be homogeneous and shall be screened.
 - 2.2.3.3. If multiple layers are present, each individual layer is assumed to be homogeneous, and, if possible, shall be screened.
 - 2.2.4. Debris
 - 2.2.4.1. Note any obvious odors.



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2.2.4.2. Based on these observations, select an appropriate extraction procedure (*ATF-LS-FD3*).

2.3. Screening Techniques

2.3.1. Liquids

2.3.1.1. When sufficient volume is available, at least one screening technique (ignition test or miscibility) shall be performed at the examiner's discretion.

2.3.1.2. Refer to *ATF-LS-FD12* for ignition susceptibility.

2.3.1.3. Miscibility is determined by adding a portion of the liquid into either an organic solvent or an aqueous liquid. Examples of suitable organic solvents include pentane and carbon disulfide. The aqueous liquid shall be water.

2.3.2. Debris

2.3.2.1. Static headspace sampling can be performed as an initial screening technique for debris samples or for pursuing indications of the presence of oxygenated products. If the initial screening headspace analysis indicates a high level of an ignitable liquid, then choose appropriate extraction technique and parameters. Refer to the static headspace procedure in *ATF-LS-FD3*.

2.4. Sample Preparation and Extraction

2.4.1. Liquids

2.4.1.1. Solvent Dilution – dilute small aliquot of sample with an appropriate organic solvent.

2.4.1.2. Wet Needle – wet an injection syringe needle with some sample.

2.4.1.3. Refer to static headspace, passive headspace concentration and solvent extraction in *ATF-LS-FD3*.

2.4.2. Debris

2.4.2.1. Refer to static headspace, passive headspace concentration and solvent extraction in *ATF-LS-FD3*.



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2.5. Instrumental Analysis

- 2.5.1. Refer to *ATF-LS-FD1* for ignitable liquid analysis.
- 2.5.2. Refer to *ATF-LS-FD10* for vegetable oil analysis.
- 2.5.3. Refer to *ATF-LS-FD2*, *ATF-LS-FD14*, and *ATF-LS-FD15* for analysis of lubricating oils, waxes, and petrolatum.

2.6. Data Interpretation

- 2.6.1. Refer to *ATF-LS-FD1* and *ATF-LS-FD8* for ignitable liquid analysis.
- 2.6.2. Refer to *ATF-LS-FD10* for vegetable oil analysis.
- 2.6.3. Refer to *ATF-LS-FD2*, *ATF-LS-FD14*, and *ATF-LS-FD15* for analysis of lubricating oils, waxes, and petrolatum.

2.7. Report Wording

- 2.7.1. Use all appropriate policies and procedures to clearly document exhibits and report all analyses and findings
- 2.7.2. Refer to *ATF-LS-FD8* and *ATF-LS-FD9*.

2.8. Sample Preservation

- 2.8.1. Refer to *ATF-LS-FD3* for extract storage and sample preservation requirements.

2.9. Other Instrumentation

- 2.9.1. For debris and liquid cases, examiners may need to utilize other instruments in order to identify incendiary mixtures and device components. Refer to the following protocols as needed:
 - SEM-EDS (*ATF-LS-E3*)
 - XRF (*ATF-LS-E4*)
 - XRD (*ATF-LS-E5*)
 - FTIR (*ATF-LS-E6*)
 - Raman (*ATF-LS-TE07*)

3. References



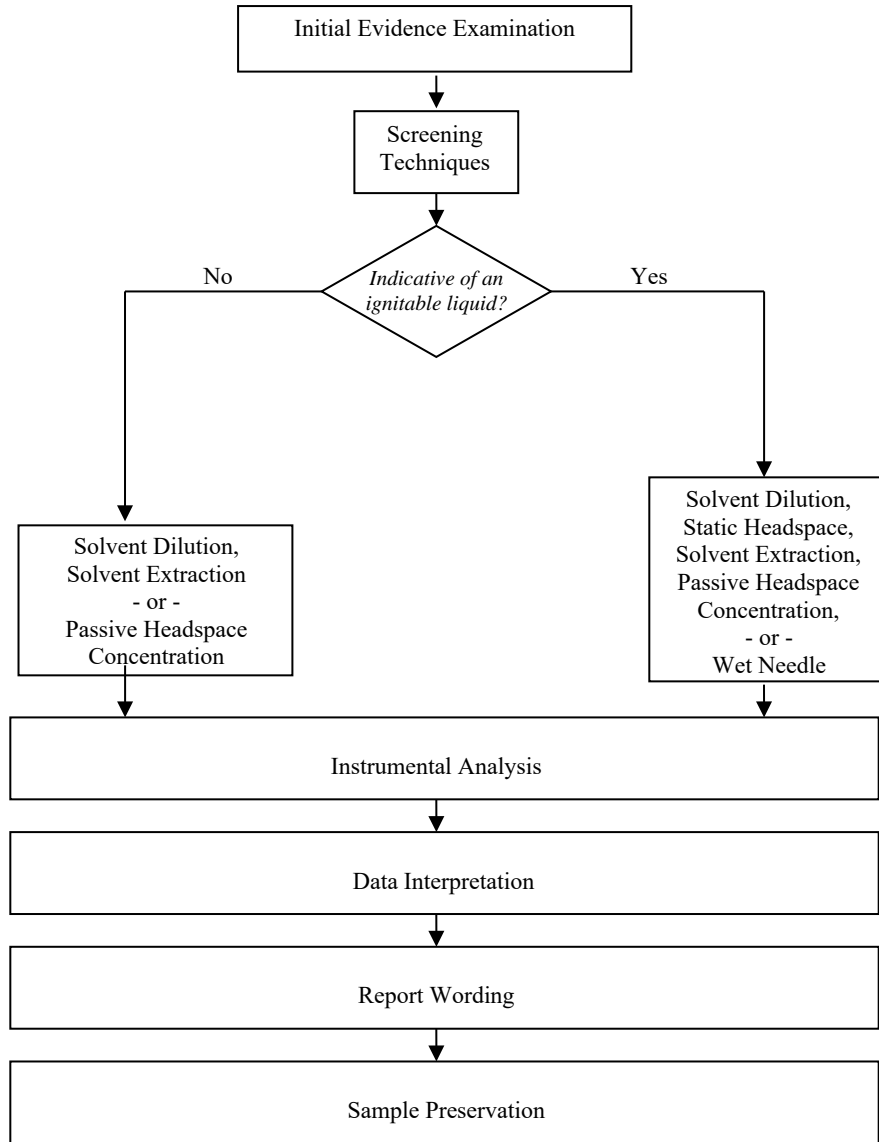
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- 3.1. *ASTM E3245, Guide for Systematic Approach to the Extraction, Analysis, and Classification of Ignitable Liquids and Ignitable Liquid Residues in Fire Debris Samples*
- 3.2. *ASTM E1618, Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.*
- 3.3. *ASTM E1386, Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction*
- 3.4. *ASTM E1388, Practice for Static Headspace Sampling of Vapors from Fire Debris.*
- 3.5. *ASTM E1412, Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris by Passive Headspace Concentration.*
- 3.6. *ASTM E 2881, Test Method for Extraction and Derivatization of Vegetable Oils and Fats from Fire Debris and Liquid Samples with Analysis by Gas Chromatography-Mass Spectrometry.*
- 3.7. *ASTM E2997, Test Method for Analysis of Biodiesel Products by Gas Chromatography-Mass Spectrometry.*



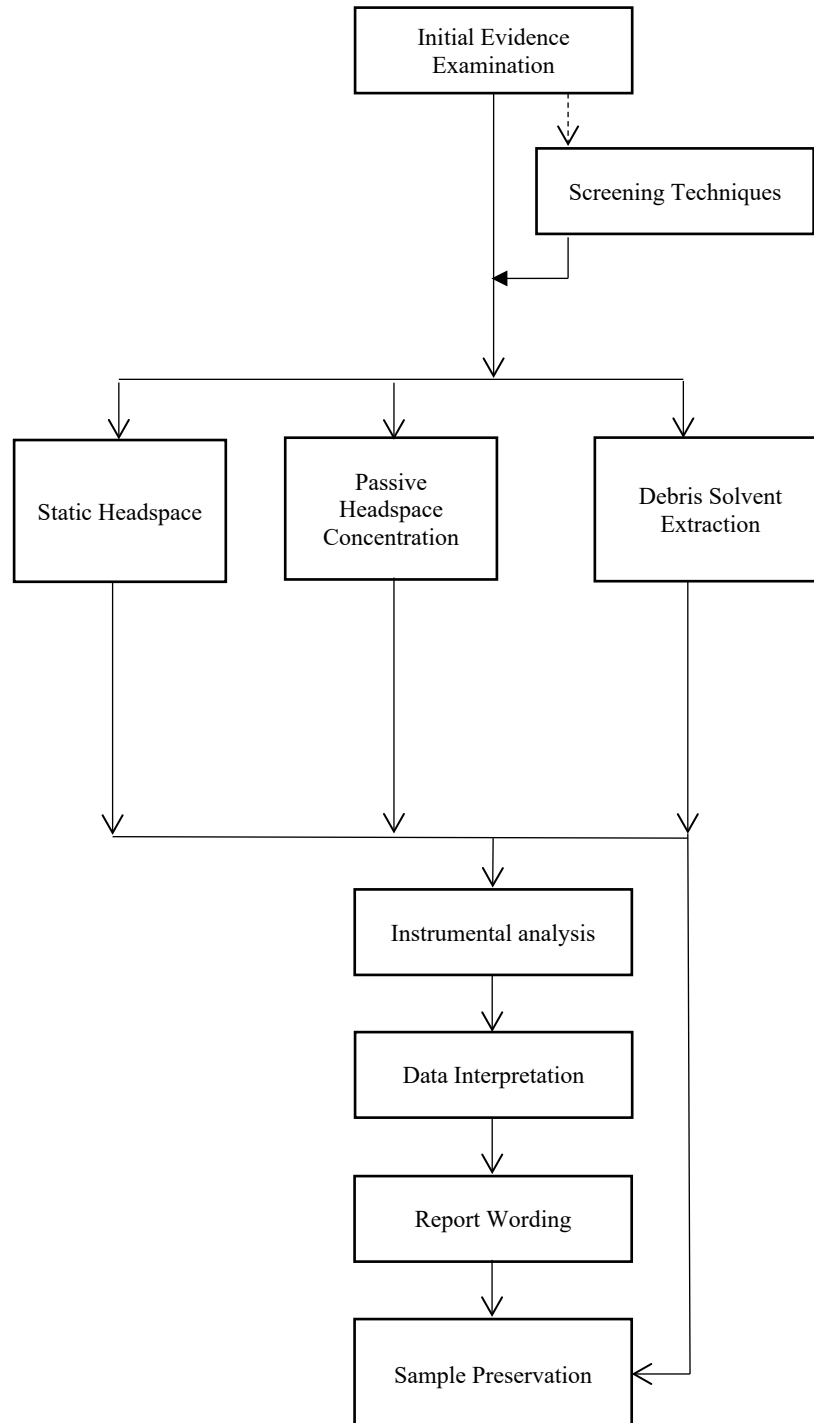
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APPENDIX A LIQUID SAMPLE ANALYSIS





APPENDIX B DEBRIS ANALYSIS





ATF-LS-FD 1 Gas Chromatography-Mass Spectrometry (GC-MS)	ID: 1615 Revision: 17
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1. Scope

1.1. This policy and procedure establishes a standard method for identification and classification of ignitable liquids and their residues extracted from fire debris by Gas Chromatography-Mass Spectrometry (GC-MS).

2. Instrumentation/Reagents

2.1. Gas Chromatograph

2.1.1. Capable of using capillary columns and being interfaced to a mass spectrometer.

2.1.2. Sample inlet system that can be operated in either split or splitless mode with capillary columns.

2.1.3. A capillary, bonded phase, methylsilicone column or equivalent column.

2.1.4. A column oven capable of reproducible temperature program operation in the range from 30 to 300°C.

2.2. Mass Spectrometer

2.2.1. Capable of scanning between 20 and 600 m/z with unit resolution or better, with continuous data output.

2.3. Computerized data station

2.3.1. Capable of storing chromatographic and mass spectral data from sample runs.

2.3.2. Capable of performing, either through its operating system or by user programming, various data handling functions, including input and storage of sample data files, generation of selected ion profiles, searching data files for selected compounds, and qualitative and semi-quantitative compound analysis.

2.3.3. Capable of retrieving a specified mass spectral scan from a data file and comparing it against a library of mass spectra available to the data system.

2.4. Syringes capable of introducing a sample size in the range from 0.1 to 10.0 µL.

2.5. Quality Control and References



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- 2.5.1. A commercially available test mixture, such as Restek (E1387), consisting of equal parts by volume of the even-numbered n-alkanes (ranging from n-hexane through n-eicosane), toluene, p-xylene, o-ethyltoluene, m-ethyltoluene, and 1,2,4-trimethylbenzene.
- 2.5.2. Commercially available reagent grade acetone, methyl ethyl ketone, ethanol and isopropanol.
- 2.5.3. A reference collection including evaporated samples of a broad variety of ignitable liquids including but not limited to gasoline, petroleum distillates, isoparaffinic products, normal alkane products, naphthenic/paraffinic products, aromatic products, oxygenated products and other miscellaneous ignitable liquids.

3. Safety Considerations

- 3.1. Whenever facilities permit, vacuum pumps, mechanical or turbo, should be properly vented to a fume hood or filtered prior to start-up of the GC-MS.
- 3.2. Used pump oil should be disposed of in an environmentally safe manner (i.e. Safety Kleen or other vendor). This oil contains both known and unknown chemicals that could pose potential health hazards.
- 3.3. Personal protective equipment such as lab coat, safety glasses and gloves should be available and used, if necessary, when preparing samples and conducting GC-MS analyses.
- 3.4. All gases should be properly secured/stored.
- 3.5. Proper precautions and care should be exercised when performing manual injections, performing maintenance on the injector or column (hot surfaces), and performing diagnostics (electrical hazards).
- 3.6. SDS references for solvents should be available and read by user.

4. Procedure

- 4.1. The identification of ignitable liquids in fire debris is done using pattern recognition techniques and mass spectral identification techniques when deemed necessary. The data shall be interpreted using the current version of *ATF-LS-FD8* on Ignitable Liquid Classification. Documentation of recognized petroleum patterns and comparison of patterns to a reference liquid shall be included in the case jacket. Pattern recognition techniques include examining both the Total Ion Chromatogram (TIC) and Extracted Ion



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Profiles (EIP). Computer macros exist which produce hard copies of these extracted ion profiles. The following extracted ion profiles are used for routine fire debris analysis:

Alkanes	57	71	85	99
Simple aromatics	91	105	119	133
Indanes	117	131	145	
Olefins/Cycloparaffins	55	69	83	
Polynuclear aromatics	128	142	156	

- 4.2. If a broad range heavy petroleum distillate is detected, then an extracted ion profile that includes m/z 74 shall be generated to screen for the presence of fatty acid methyl esters (FAMES). If deemed necessary, the analyst may utilize other ion profiles to aid in the interpretation of the data. It should be clearly documented in the case jacket if profiles other than those listed above are used in data interpretation.
 - 4.3. In cases where identifying and reporting a unique chemical compound occur, the retention time (RT) and spectral data shall be compared with a known reference liquid. The retention times of the questioned (Q) and known (K) peaks shall be within 2%. This will be determined using the following equation: $[RT(K) - RT(Q)]/RT(K) * 100\%$.
 - 4.4. The relative abundances of ions in the spectra of the unknown sample and the reference liquid will have general agreement across the majority of the ions. The unknown and known samples should have the same base peak and the same molecular ion, where the molecular ion is present. Isotopic ions present in the reference spectrum shall be present in similar proportions in the unknown sample spectrum; low abundance ions (less than 5% of the total spectral abundance) may be absent without precluding an identification. Background subtraction may be necessary to remove any background contribution to the sample, such as air/water. There shall be no unexplained extraneous ions above 30% relative abundance.
 - 4.5. Classify ignitable liquids as defined in the Ignitable Liquid Classification System (*ATF-LS-FD8*). *ASTM E 1618* may offer further guidance in correctly classifying the ignitable liquid.
5. Quality Assurance and Controls
 - 5.1. Tune
 - 5.1.1. The instrument will be tuned using perfluorotributylamine (PFTBA) no more than one week prior to casework analysis. The tune report shall be examined to ensure that appropriate parameters are within their normal expected range specified by the manufacturer and initialed by the examiner performing the tune.



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Documentation that the values were verified as meeting manufacturer's specifications will be maintained in the instrument log. Tune reports are maintained near the instrument. Record the tune date in the case file.

5.2. Quality Control Mix

- 5.2.1. Each day the instrument is used for analysis, a quality control (QC) sample shall be run. This QC may be either a Restek (E1387) or equivalent standard (25 μ L quality control solution in 1 mL of solvent) and will include ethanol, acetone, isopropanol (IPA), and methyl ethyl ketone (MEK) added by the laboratory. The QC will be prepared in similar proportions to the following: 625 μ L Restek (or equivalent), 5 μ L ethanol, 3 μ L acetone, 2 μ L IPA, and 3 μ L MEK diluted to approximately 25 mL with carbon disulfide. In cases where sequences carry over into the next day, the quality control sample will be considered still valid for the sequence. Additional "passing" quality control samples must be run for sequences continuing beyond the next day and each day thereafter.
- 5.2.2. The QC data shall be assessed each time it is run to ensure overall good chromatography. The retention times of the following compounds will be monitored: ethanol, 1,2,4-trimethylbenzene, and eicosane (C20). Retention times shall be within 2% of the QC analyzed after the last instrument maintenance that affects chromatography (refer to the Procedures section above for the calculation).
- 5.2.3. The retention times will be recorded in a log and maintained near the instrument. When the retention times fall outside of the specified range (i.e. "fail"), the error shall be corrected and documented in the logbook. Sample data collected after a QC fails will be re-analyzed after the issue(s) have been resolved. Evidence examination may proceed after it has been demonstrated that the instrument is functioning properly, according to the specifications defined by the instrument manufacturer and this procedure, and that the system and solvent(s) are free of contamination (i.e. "pass"). A hard copy of the QC data will be printed and initialed by the person evaluating the data. Documentation that the data was verified as meeting the criteria listed above will be maintained in the instrument log. The initialed QC printout will be maintained near the instrument and its use (including, at a minimum, the QC name and date prepared) will be documented in the case record.

5.3. System and Solvent Blanks

- 5.3.1. Run a system blank or materials control (defined in each method of analysis) with case samples to ensure that the instrument, solvent and method of analysis are free of contamination. Run a solvent blank before the QC and between each case



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sample. These blanks ensure that no carryover from the previous sample exists and that the system is not contaminated. If extraneous peaks are noted in either the system blank or the solvent blanks, an attempt will be made to determine the source of these and will be documented in the case notes. When solvent blanks indicate potential carryover or contamination from ignitable liquids, the affected exhibits will be re-analyzed. Maintain the data for the system blank(s) and solvent blanks in the case record.

5.4. Maintenance

- 5.4.1. Changes to existing data acquisition methods and data analysis macros shall be performance checked to verify the appropriate response is given.
- 5.4.2. When changes outside the acceptable parameters occur in the tune or the quality control sample check the following items:
 - Septum
 - Column nut
 - GC/MS interface nut
 - Injection Port Liner
 - Capillary column
 - Mass Spectrometer Source
- 5.4.3. When GC-MS maintenance is performed that affects chromatography, a QC will be analyzed and the new retention times will be recorded to ensure criteria are met. All maintenance performed on the instrument will be recorded in a logbook.
- 5.4.4. Change the carrier gas cylinder as necessary.

6. References

- 6.1. *ASTM E1618, Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.*
- 6.2. *Safety Data Sheets for applicable solvents and chemicals*
- 6.3. *Newman, R., Gilbert, M. and Lothridge, K. "GC-MS Guide to Ignitable Liquids," CRC Press 1998.*
- 6.4. *Smith, R. M., Analytical Chemistry, Vol. 54, No. 13, November 1982, pp 1399A-1409A.*



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- 6.5. *Nowicki, J., "An Accelerant Classification Scheme based on Analysis by Gas Chromatography-Mass Spectrometry (GC-MS)," Journal of Forensic Science, Volume 35(5) pp. 1064-1086.*
- 6.6. *Smith, R. M., "Mass Chromatographic Analysis of Arson Accelerants," Journal of Forensic Science Vol. 28(2), pp 318-329.*
- 6.7. *Gas Chromatograph-Mass Spectrometer Maintenance and Troubleshooting Manual*
- 6.8. *Instrument Hardware Manual*



ATF-LS-FD2 High-Temperature Gas Chromatography-Mass Spectrometry Analysis	ID: 764 Revision: 8
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1. Scope

- 1.1. This policy and procedure guideline establishes a standard method for identification and classification of materials by High Temperature Gas Chromatography-Mass Spectrometry (HTGC-MS). This method is useful for the analysis of oils, waxes, lubricants (such as petroleum jelly), and plasticizers.
- 1.2. High temperature GC uses injector and column temperatures above 330°C to separate high boiling compounds and the mass spectrometer to detect the components with electron ionization.
- 1.3. Oils, waxes, and lubricants may be analyzed at lower, “normal”, temperatures (below 330°C), but higher boiling hydrocarbons above C44 may not be eluted from the column. Examiners who use normal temperature GC-MS for the analysis of these materials should follow *ATF-LS-FDI*.

2. Instrumentation/Reagents

2.1. Gas Chromatograph

- 2.1.1. Capable of using capillary columns and being interfaced to a mass spectrometer.
- 2.1.2. Programmable Temperature Vaporizer (PTV) injection system that can be operated in either split or splitless mode and ramped up to at least 400°C.
- 2.1.3. A cross-linked or bonded phase capillary column (5% phenyl or equivalent), rated to handle temperature limits of at least 375°C.
- 2.1.4. A column oven capable of reproducible temperature program up to at least 375°C.

2.2. Mass Spectrometer

- 2.2.1. Capable of scanning between 20 and 600 m/z with unit resolution or better, with continuous data output.

2.3. Computerized data station

- 2.3.1. Capable of storing chromatographic and mass spectral data from sample runs.
- 2.3.2. Capable of performing, either through its operating system or by user programming, various data handling functions, including input and storage of



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sample data files, generating selected ion profiles, searching data files for selected compounds, and qualitative and semi-quantitative compound analysis.

2.3.3. Capable of retrieving a specified mass spectral scan from a data file and comparing it against a library of mass spectra available to the data system.

2.4. Syringes capable of introducing a sample size in the range from 0.1 to 10.0 μL .

2.5. Quality Control and References

2.5.1. A quality control hydrocarbon mixture, such as Restek D2887 mix or equivalent, containing n-alkanes C20, C24, C28, C32, C36, and C44.

2.5.2. A reference collection including of a broad variety of materials expected to be analyzed.

3. Safety Considerations

3.1. Whenever facilities permit, vacuum pumps, mechanical or turbo, should be properly vented to a fume hood or filtered prior to start-up of the GC-MS.

3.2. Used pump oil should be disposed of in an environmentally safe manner (i.e. Safety Kleen or other vendor). This oil contains both known and unknown chemicals that could pose potential health hazards.

3.3. Personal protective equipment such as lab coat, safety glasses and gloves should be available and used, if necessary, when preparing samples and conducting GC-MS analyses.

3.4. All gases should be properly secured/stored.

3.5. Proper precautions and care should be exercised when performing manual injections, performing maintenance on the injector or column (hot surfaces), and performing diagnostics (electrical hazards).

3.6. Safety Data Sheet (SDS) references for solvents should be available and read by user.

4. Procedure

4.1. Refer to *ATF-LS-FD14* and *ATF-LS-FD15* for sample preparation guidelines. For other samples, such as plasticizers, which are not covered in these protocols, analysts should extract samples in trichloroethylene (TCE), pentane, or other solvent appropriate for the



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sample and the chosen GC capillary column at a recommended concentration of 0.1% by weight. Sample preparation is critical to avoid overload and carryover.

5. Quality Assurance and Controls

5.1. Tune

5.1.1. Each day that the instrument is used for analysis it will be tuned using perfluorotributylamine (PFTBA). The tune report shall be examined to ensure that all parameters are within their normal expected range specified by the manufacturer and initialed by the examiner performing the tune. The tune shall monitor ions 69, 219, and 502. If the 502 ion falls below 0.1% of the 69 ion, appropriate instrument maintenance should be performed. Tune reports are maintained near the instrument. Documentation that the values were verified as meeting manufacturer's specifications will be maintained in the instrument log. In cases where sequences carry over into the next day, the tune will be considered still valid for the sequence.

5.2. Quality Control Mix

5.2.1. Each day the instrument is used for analysis, a quality control (QC) sample shall be run. The QC shall contain at least C20, C24, C28, C32, C36, and C44 in trichloroethylene (TCE) or other solvent appropriate for the chosen GC capillary column. Restek Calibration Mix D2887 or equivalent is recommended to be diluted at least 1:200. Each day the QC is run, the signal to noise ratio of the C44 peak will be monitored and shall be no less than 100:1. If the C44 peak falls below this, instrument maintenance is required. In cases where sequences carry over into the next day, the QC will be considered still valid for the sequence. Additional quality control samples must be run for sequences continuing beyond the next day and each day thereafter. A hard copy of the QC data will be printed and initialed by the person evaluating the data. Documentation that the data was verified as meeting the criteria listed above will be maintained in the instrument log. The initialed QC printout will be maintained near the instrument and its use (including, at a minimum, the QC name and date prepared) will be documented in the case record.

5.3. System and Solvent Blanks

5.3.1. Run a materials control with case samples to ensure that the instrument, solvent and method of analysis are free of contamination. Run a solvent blank before the QC and between each case sample. These blanks ensure that no carryover of the target material from the previous sample exists and that the system is not



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contaminated with this target material. If extraneous peaks are noted in either the system blank or the solvent blanks, an attempt will be made to determine the source of these and will be documented in the case notes. When solvent blanks indicate potential carryover or contamination from the target material, the affected exhibits will be re-analyzed. Maintain the data for the system blank(s) and solvent blanks in the case record.

5.4. Sample Identification

5.4.1. The identification of materials is performed using retention time and spectral data compared with a known reference material and/or pattern recognition techniques, which involve examining the sample chromatograms and comparing them to applicable references. For comparative purposes, run a reference similar to the sample data contemporaneously.

5.4.2. In cases where identifying and reporting a unique chemical compound occur, compare the retention time (RT) and spectral data with a known reference liquid. The retention times of the questioned (Q) and known (K) peaks shall be within 2%. This will be determined using the following equation: $[\text{RT}(\text{K}) - \text{RT}(\text{Q})] / \text{RT}(\text{K}) * 100\%$.

5.4.3. The relative abundances of ions in the spectra of the unknown sample and the reference liquid will have general agreement across the majority of the ions. The unknown and known samples should have the same base peak and the same molecular ion, where the molecular ion is present. Isotopic ions present in the reference spectrum shall be present in similar proportions in the unknown sample spectrum; low abundance ions (less than 5% of the total spectral abundance) may be absent without precluding an identification. Background subtraction may be necessary to remove any background contribution to the sample, such as air/water. There shall be no unexplained extraneous ions above 30% relative abundance.

5.5. Maintenance

5.5.1. Maintain logs as to operator use and service/maintenance by vendors or primary operators.

5.5.2. When changes outside the acceptable parameters (as defined by manufacturer) occur in the tune or the quality control sample (i.e. air leaks, decreased resolution, decreased sensitivity) check the following items:

- Septum
- Column nut



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- GC-MS interface nut
- Injection port liner
- Capillary column
- Mass spectrometer source

5.6. Sources of Error

- 5.6.1. If a large volume of sample is injected and/or the sample is too concentrated, peaks may broaden and become distorted.
- 5.6.2. Extraneous peaks may result from impurities in the solvent, carryover from previous runs or other materials used in the method of analysis.
- 5.6.3. If no peaks are observed, check sample concentration and the mass spectrometer. The sample may need to be concentrated or the MS source may need to be cleaned.

6. References

- 6.1. *ASTM D5442, Test Method for Analysis of Petroleum Waxes by Gas Chromatography.*
- 6.2. *Safety data sheets for Pentane, Trichloroethylene, and other applicable solvents and chemicals*
- 6.3. *Barker, A, The chromatographic analysis of refined and synthetic waxes. In: Adlard, ER (ed.), Chromatography in the petroleum industry, Elsevier, 1995, pp 55-93.*
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- 6.12. Reardon MR and Bender EC, *Differentiation of Composition C-4 based on the analysis of the process oil*, *Journal of Forensic Sciences*, 50(3):564-570, 2005.
- 6.13. Roehner, RM, Fletcher, JV, and Hanson, FV, *Comparative Compositional Study of Crude Oil Solids from the Trans Alaska Pipeline System Using High-Temperature Gas Chromatography*, *Energy and Fuels*, Vol 16, 2002, pp 211-17.
- 6.14. U.S. Customs Laboratory Methods (USCL Method 34-07), *Quantitation of paraffin in beeswax and other waxes by high temperature capillary gas chromatography*, March 2003.



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1. Scope

1.1. This policy and procedure guideline describes the standard methods for the extraction of ignitable liquids and their residues from fire debris. These methods include static headspace sampling, passive headspace concentration, and solvent extraction.

1.2. Static Headspace Sampling of Vapors

1.2.1. This method is used to recover light oxygenated products.

1.2.2. This method is not suitable for low concentrations of ignitable liquids or heavy petroleum products. Due to the limitations of this technique, it may be necessary to perform additional extractions such as passive headspace concentration or dynamic headspace concentration.

1.3. Passive Headspace Concentration

1.3.1. This method, under certain gas chromatograph-mass spectrometer (GC-MS) parameters, is used to recover both light oxygenated products and hydrocarbon products. Therefore, this technique may be used to identify light oxygenated products without the use of static headspace sampling.

1.3.1.1. Note that this method may not be efficient for the extraction of methanol. If instrumental conditions are not adjusted to recover light oxygenated products, static headspace sampling must be used to identify a light oxygenated product.

1.3.2. This method may not recover hydrocarbons with a boiling point above heptadecane (C_{17}), and therefore may not be able to fully recover some heavy petroleum distillates. If appropriate, solvent extraction may also be used for the separation and concentration of ignitable liquid residues from fire debris samples.

1.4. Solvent Extraction

1.4.1. This method is used to extract ignitable liquids from aqueous samples and from items that may not be suitable for passive headspace concentration.

1.4.2. This method is also used for further characterizing heavy petroleum distillates.

1.4.3. This method is best suited for analyzing nonporous matrices; however, porous matrices can also be analyzed using this technique. Co-extraction of interfering compounds may be a concern with some porous matrices using this technique.



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2. Instrumentation/Reagents

2.1. Static Headspace Sampling

- 2.1.1. New disposable syringe, approximately 3cc volume or airtight syringes
- 2.1.2. Tape
- 2.1.3. Rubber sleeve stopper
- 2.1.4. Punch
- 2.1.5. Analytical Oven

2.2. Passive Headspace Concentration

- 2.2.1. Activated charcoal strips
- 2.2.2. Carbon disulfide (CS₂) with automatic volume pipette
- 2.2.3. New, unused paperclips, safety pins, fishing line or similar device
- 2.2.4. Magnets
- 2.2.5. Vials, autosampler vials, autosampler vial inserts
- 2.2.6. Analytical oven

2.3. Solvent Extraction

- 2.3.1. CS₂, pentane or other appropriate solvent.
- 2.3.2. Filter apparatus free of extractable hydrocarbons.
- 2.3.3. Beakers or similar containers

3. Safety Considerations

- 3.1. Personal protective equipment such as lab coat, safety glasses and gloves should be available and used, if necessary, when preparing samples.
- 3.2. Exercise caution when removing cans from ovens. All cans should be allowed to cool prior to opening or puncturing.
- 3.3. CS₂ and pentane are hazardous chemicals with respect to both health and fire safety and should be handled with extreme care. Use CS₂ and pentane in a properly operating ventilation hood. Avoid physical contact with CS₂ and pentane. Keep CS₂ and pentane from heat and sources of ignition.

4. Procedure

4.1. Static Headspace Sampling



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4.1.1. Cans

- 4.1.1.1. Punch a small hole in the container lid. Cover the hole with tape. Alternatively, a rubber sleeve stopper may be placed in the hole. If deemed necessary, place the sample container in the oven at approximately 65°C until an estimated thermal equilibrium is reached.
- 4.1.1.2. Remove the container from the oven. Push the syringe needle through the tape or rubber sleeve stopper into the hole in the container lid and slowly pump the syringe three times, withdraw sample from the container, and inject up to 2 mL of sample vapor directly into the injection port of the gas chromatograph/mass spectrometer. Seal the hole with tape to prevent vapors from escaping the container.

4.1.2. Fire Debris Suitable Bags

- 4.1.2.1. If possible, transfer debris to a can, otherwise heat the bag in an oven at 65°C until approximate thermal equilibrium is reached.
- 4.1.2.2. Remove the bag from the oven and push the syringe needle in to the bag. Slowly pump the syringe three times. Inject up to 2 mL of sample vapors into the injection port of the GC-MS.

4.1.3. Vial containing Liquid

- 4.1.3.1. Open vial. Place syringe over the headspace and slowly pump the syringe three times, withdraw sample from vial, and inject up to 2 mL of sample vapors into the injection port of the GC-MS.

4.2. Passive Headspace Concentration

- 4.2.1. Open the container and determine if the sample appears to agree with the description on the transmittal documents and can labeling. Suspend pre-cut activated charcoal strips inside the evidence container using a paperclip, fishing line or similar device. Close the container.
- 4.2.2. Samples are generally extracted for approximately 16 hours at a temperature between 60 - 70°C. An ambient temperature extraction may be necessary due to sample size or condition. In these cases, samples will be extracted for a minimum of 16 hours. In cases in which there is a significant discernible odor indicative of a petroleum product, the extraction time may be shortened to 4 hours. If this shorter



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extraction time is used and the data indicates a product other than gasoline, then a follow up extraction that is more representative of the heavier components, such as a 16-hour extraction at 60-70°C or a solvent extraction, shall be done.

- 4.2.3. Monitor the oven's temperature during extractions in order to ensure consistent temperatures. Document deviations from the set temperature in the case notes. Based on the circumstances of the case and the degree of temperature deviation, the examiner will determine if an additional extraction is necessary. The temperature monitoring chart shall be retained in a file near the oven. If a laboratory has more than one oven for extractions, then the oven used shall be noted in the case record.
- 4.2.4. Once containers have cooled, remove the pre-cut activated charcoal strips. Place one of the strips in a vial labeled with case and exhibit numbers. Extract and analyze the other strip. To extract the strip for analysis, add, at a minimum, enough CS₂ to the vial to wet the strip. The actual volume used is sample dependent and is at the discretion of the analyst. Further dilution may be necessary due to the concentration of the sample. Agitate the vial and transfer the CS₂ solution using a clean disposable pipette to a new vial.
- 4.2.5. Analyze by GC-MS (refer to *ATF-LS-FD1*).

4.3. Solvent Extraction

4.3.1. Fire Debris

- 4.3.1.1. Open and examine the fire debris sample to determine that it is consistent with its description.
- 4.3.1.2. The sample may be extracted in its original container or placed in a disposable or new container for extraction.
- 4.3.1.3. Add sufficient solvent to thoroughly moisten the sample.
- 4.3.1.4. Mix the solvent and debris thoroughly. Simple rinsing of nonporous surfaces may not adequately separate residues.
- 4.3.1.5. Filter the solvent if necessary, using a filter that is compatible with the extraction solvent.



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4.3.1.6. A portion of the extract shall be preserved with a charcoal strip or portion thereof. Place the preserved extract in a vial labeled with case and exhibit numbers.

4.3.1.7. Analyze by GC-MS (refer to *ATF-LS-FDI*).

4.3.2. Liquids

4.3.2.1. Place a sample of unknown aqueous liquid in a container and add pentane, CS₂, or other appropriate solvent.

4.3.2.2. Mix the liquids then allow the layers to separate.

4.3.2.3. Remove the pentane or CS₂ layer.

4.3.2.4. Filter if necessary, using a filter that is compatible with the extraction solvent.

4.3.2.5. A portion of the extract shall be preserved with a charcoal strip or portion thereof. Place the preserved extract in a vial labeled with case and exhibit numbers,

4.3.2.5.1. If the sample is a liquid that did not burn or if a portion of the original liquid will be returned, it may not be necessary to preserve the extract or a sample of the liquid with a charcoal strip or portion thereof.

4.3.2.6. Analyze by GC-MS (refer to *ATF-LS-FDI*).

4.3.3. Concentration

4.3.3.1. If the sample needs to be concentrated, perform the concentration in containers free of extractable hydrocarbons (disposable or new containers).

4.3.3.2. Place the container in a chemical fume hood and evaporate at room temperature. Compressed dry nitrogen can be used to accelerate evaporation.

4.3.3.3. Heating should be done with caution to prevent excessive concentration resulting in substantive changes to the chemical profile or loss of sample due to complete evaporation or boiling over. Heating mantles are not recommended; however, a steam bath may be used to accelerate evaporation.

5. Quality Assurance and Controls



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5.1. Static Headspace Sampling

- 5.1.1. Analyze a positive control using an appropriate ignitable liquid prior to sampling any exhibits.
- 5.1.2. Analyze a syringe/air blank prior to sampling each exhibit. The syringe to be used for sampling an exhibit will be pumped three times and a sample of room air collected and injected into the injection port of the GC-MS. Documentation of air blanks will be maintained in the appropriate case jacket.
- 5.1.3. Preserved Extracts
 - 5.1.3.1. Sample preservation is not feasible when using this technique.

5.2. Passive Headspace Concentration

- 5.2.1. Test each new batch/lot number of charcoal strips prior to being implemented for casework to determine that it is free of contaminants and that it qualitatively performs as expected for passive headspace.
 - 5.2.1.1. Place a charcoal strip or portion thereof in two separate vapor-tight containers: one will contain 5 – 10 μ L of a 50:50 gasoline:diesel mixture on a Kimwipe (or equivalent), and the other will contain a Kimwipe (or equivalent).
 - 5.2.1.2. Extract each container using the first set of conditions listed in 4.2.2.
 - 5.2.1.3. Both gasoline and the heavy petroleum distillate shall be identifiable as per the (*ATF-LS-FD8*) in the positive (spiked) container. The results of these tests will be noted in a log, and the data will be maintained with the log.
 - 5.2.1.4. Note the lot number of the charcoal strips used in casework in the case record.
- 5.2.2. For each day that samples are prepared using this method, a system blank will be prepared and extracted.
 - 5.2.2.1. To create a system blank, a charcoal strip or portion thereof will be extracted using the same procedure as the sample.



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5.2.2.2. If a sample extract is filtered or concentrated, the system blank shall be treated the same way.

5.2.2.3. A copy of the system blank will be maintained in the appropriate case record.

5.2.3. Preserved Extracts

5.2.3.1. All preserved extracts shall be returned with the evidence to the submitting agency.

5.2.3.2. When fire debris evidence may need DNA and/or print examinations, the duplicate charcoal strip(s) will be sub-designated and returned with the evidence in a separate convenience container.

5.2.4. Oven Performance Check

5.2.4.1. The oven temperature reading and the temperature monitoring device shall be checked on an annual basis using a NIST traceable thermometer. This thermometer will be placed in the oven at 65°C for a minimum of two hours. Record the temperature of the thermometer, the oven and the monitoring device. The tolerance will be $\pm 15^{\circ}\text{C}$.

5.3. Solvent Extraction

5.3.1. Prepare the materials control the same way as the sample(s). If a liquid is diluted in a solvent or extracted using a simple liquid-liquid extraction technique, the solvent blank prior to the sample may serve as the materials control. Additionally, if the sample is filtered or concentrated, the materials control shall be treated the same way.

5.3.2. A copy of the materials control will be maintained in the appropriate case jacket.

5.3.3. Preserved Extracts

5.3.3.1. All preserved extracts shall be returned with the evidence to the submitting agency.

5.3.3.2. When the extracted evidence may need DNA and/or print examinations, the preserved extract will be sub-designated and returned with the evidence in a separate convenience container.



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6. References

- 6.1. *ASTM E752, Practice for Safety & Health Requirement relating to Occupational Exposure to Carbon Disulfide.*
- 6.2. *ASTM E1386, Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction*
- 6.3. *ASTM E1388, Practice for Static Headspace Sampling of Vapors from Fire Debris.*
- 6.4. *ASTM E1412, Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris by Passive Headspace Concentration.*
- 6.5. *ASTM E1618, Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.*
- 6.6. *Occupational Health Guidelines for Carbon Disulfide, U.S. Department of Labor and Department of Health, September 1978.*
- 6.7. *Safety Data Sheets for Carbon Disulfide, Pentane, and other applicable solvents*
- 6.8. *Bertsch, W. and Q. Zhang, Sample Preparation for the Chemical Analysis of Debris in Suspect Arson Cases, Analytica Chimica Acta, 236 (1990) 183-195.*
- 6.9. *Caddy, B. et al., Methods of Fire Debris Preparation for Detection of Accelerants, Forensic Science Review, 3 (1), June 1991, 58-69.*
- 6.10. *Chrostowski, J.E. and R.M. Holmes: Collection and Determination of Accelerant Vapors from Arson Debris, Arson Analysis Newsletter, 3, No. 5, 1 (1979).*
- 6.11. *Dietz, W. R. Improved Charcoal Packaging for Accelerant Recovery by Passive Diffusion, Journal of Forensic Sciences 36(1):111-121.*
- 6.12. *Levadie, G.J. and S. MacAskill: Analysis of Organic Solvents Taken On Charcoal Tube Samplers by a Simplified Technique. Analytical Chemistry, 48, 76 (1976).*
- 6.13. *Newman, R.T., Lothridge, Kevin and Dietz, W. "The Use of Activated Charcoal Strips for Fire Debris Extractions by Passive Diffusion Part 1: The Effects of Time, Temperature, Strip Size, and Sample Concentration" Journal of Forensic Sciences, Vol. 41, No. 3, May, 1996, pp. 167-176*



ATF-LS-FD7 Ignitable Liquid Reference Library	ID: 1621 Revision: 4
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1. Scope

- 1.1. This policy and procedure establishes the maintenance of an Ignitable Liquid Reference Library.
- 1.2. An ignitable liquid reference library is necessary for the identification of ignitable liquids or ignitable liquid residues recovered from fire debris. Each ATF Forensic Science Laboratory will maintain its own ignitable liquid reference library. In addition, each laboratory may use published references to assist in identifying additional products for addition to their reference library.
- 1.3. The liquids maintained in the ignitable liquid library are used for classification purposes only. A liquid selected for comparison in casework may be one of several liquids representative of an ignitable liquid class. The selection of a particular liquid does not constitute the identification of a specific product due to the marketing practices of the petroleum industry.
- 1.4. Most of the liquids obtained are commercially available. Therefore, certified reference liquids are not specifically required. Given the practices of the petroleum industry, each commercially obtained liquid is considered unique and there is no expiration date associated with these liquids.

2. Instrumentation/Reagents

- 2.1. Refer to *ATF-LS-FDI*.

3. Safety Considerations

- 3.1. No specific safety requirements. Use appropriate care in handling and storing each product.

4. Procedure

4.1. Library collection

- 4.1.1. The library will consist of samples of ignitable liquids from each class identified in the ATF Ignitable Liquid Classification System. Samples will be run on the GC-MS under the same instrumental parameters used for sample analysis. In addition to original unevaporated samples, samples in various stages of evaporation will be included as appropriate. If instrumental analysis parameters are significantly changed, the reference liquid should be rerun to match the new instrumental parameters.



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4.2. Library database

- 4.2.1. Each laboratory will maintain a database containing all the samples in the library. The database will contain a unique identifier for each sample, the commercial name, manufacturer/distributor, classification and date acquired (if known). Photos of containers may be placed in the database.

4.3. Database availability

- 4.3.1. Each forensic laboratory may review the database of the other laboratories and should request a sample from another laboratory's library as needed. All non-routine samples encountered should be sent to other laboratories.

4.4. Library maintenance

- 4.4.1. It is highly likely that formulations of commercial products will change. New commercially available products or formulations should be added as they become available or as appropriate. The library will be reviewed periodically to ensure it encompasses the types of products routinely encountered in casework.

5. Quality Assurance and Controls

- 5.1. Reference ignitable liquids used for comparison in casework will be run under the same conditions as the unknown sample and compared to the chromatogram maintained in the paper and/or electronic library.
- 5.2. Section Chiefs are responsible for ensuring that the Ignitable Liquid Reference Library is maintained.

6. References

- 6.1. *ASTM E1618, Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.*
- 6.2. *Newman, R., Gilbert, M. and Lothridge, K. "GC-MS Guide to Ignitable Liquids," CRC Press 1998.*



ATF-LS-8 Ignitable Liquid Classification System	ID: 1622 Revision: 13
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1. Scope

- 1.1. This policy and procedure guideline establishes a standard Ignitable Liquid Classification System.
- 1.2. Ignitable liquids recovered in fire debris analysis will generally fall into the following classifications:
 - Distillates (Light, Medium, Heavy)
 - Aromatic Products
 - Isoparaffinic Products
 - Normal Alkane Products
 - Naphthenic/Paraffinic Products
 - Oxygenated Products
- 1.3. The distillate category must be further classified based on boiling point range as Light, Medium or Heavy. The remaining categories may be sub-classified as Light, Medium or Heavy at the discretion of the analyst.
- 1.4. Gasoline is a unique product and shall be identified and reported as such.
- 1.5. The fact that some petroleum products do not fall clearly into any of these categories, will not necessarily preclude their identification.

2. Procedure or Analysis

- 2.1. When the descriptor Light, Medium or Heavy is used, the following general guidelines for peak spread shall be used:

Light C₄ – C₉
Medium C₈ – C₁₃
Heavy C₈ – C₂₃₊

- 2.1.1. When the carbon range does not fit into one of the above categories, it may be necessary to characterize a product as “light to medium” or “medium to heavy”.

2.2. Gasoline

- 2.2.1. The predominant feature of gasoline is its aromatic content. Gasoline will also have aliphatic content that can vary among gasolines. Naphthalene and substituted naphthalenes are typically present but are not necessary for identification. The peak spread for gasoline will typically be from C₄ – C₁₃. Microbial degradation of



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gasoline in soil has been well documented in scientific literature. Degraded gasoline may be identified as gasoline, if the substrate is soil and data agrees with published references or with activity observed in a comparison sample.

2.3. Distillates

- 2.3.1. The predominant feature of distillates is a Gaussian distribution of straight-chain alkanes. Distillates will also contain branched-chain alkanes, cycloalkanes and aromatic compounds at abundance less than that of the predominant normal alkane structure. Some of these compounds present at lower levels may not be readily visible in a total ion chromatogram, however a recognizable pattern should be present in the appropriate extracted ion profiles.
- 2.3.2. Light Petroleum Distillates are an exception to the above criteria in that they typically will not contain significant amounts of aromatic compounds, yet will still be classified as a distillate. Based on the boiling point range of LPDs and the instrumental technique used, it is recognized that the entire Gaussian distribution of the alkane pattern will not be visible. Additionally, due to the nature of these products, branched-chain and cyclic alkanes may be higher in abundance in light petroleum distillates (possibly similar or greater abundance than the n-alkanes) than those seen in medium or heavy petroleum distillates.
- 2.3.3. Medium Petroleum Distillates typically have four consecutive n-alkanes and therefore appear as a narrow Gaussian distribution, with a fairly sharp beginning and end.
- 2.3.4. Heavy Petroleum Distillates are typically broad fractions and must have at least five consecutive n-alkanes. When C₁₇ and C₁₈ are present, pristane and phytane must also be present in appropriate ratios. Also included in this classification are narrow range (encompassing fewer than five consecutive n-alkanes) ignitable liquid products starting above C₁₁.
- 2.3.5. It is recognized that certain distillates will have greatly reduced or non-existent aromatic content. These products will not be separately classified.

2.4. Aromatic Products

- 2.4.1. Aromatic products consist essentially of aromatic compounds with virtually no aliphatic content. Typically aromatic products will have a narrow peak spread. Depending on boiling point range, polynuclear aromatics may be present.

2.5. Isoparaffinic Products



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2.5.1. Isoparaffinic products consist essentially of branched-chain alkanes. There will be no significant presence of straight-chain alkanes, aromatic compounds, or cycloalkanes. Typically isoparaffinic products will have a narrow peak spread.

2.6. Normal Alkane Products

2.6.1. Normal alkane products consist essentially of straight-chain alkanes. There will be no significant presence of branched-chain alkanes, aromatic compounds, or cycloalkanes. Typically these products will consist of 3 to 5 normal alkanes.

2.7. Naphthenic/Paraffinic Products

2.7.1. Naphthenic/Paraffinic products consist essentially of branched-chain alkanes and cycloalkanes. Straight-chain alkanes can be absent or present at diminished levels compared to petroleum distillates. There will be no significant presence of aromatic compounds. The total ion chromatogram of this type of product will typically appear similar to that of a distillate without the predominant n-alkanes. Typically naphthenic/paraffinic products will have a broad peak spread.

2.8. Oxygenated Products

2.8.1. The defining characteristic of an oxygenated product is the significant presence of one or more oxygen-containing compounds, such as an alcohol, ester, or ketone. These commonly appear in blends with petroleum products. At examiner discretion the oxygenated compounds and the petroleum product may be reported separately. If only distinct oxygenated compounds are present, they should be identified and reported as such.

2.9. Petroleum Products

2.9.1. If a pattern is observed which meets some, but not all of the criteria for classification within the previously defined categories, it may be classified as a petroleum product. Differences from existing categories must be minor, such as a difference in peak spread or slight differences in ratios of components. These minor differences could be explained by evaporation, sample preparation technique, the presence of a mixture, overlapping patterns, or bacterial degradation associated with soil samples. It should be emphasized that a petroleum product shall not be identified without the presence of a suitable reference ignitable liquid for comparison. More than one reference can be used to account for portions of the pattern.



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2.10. Refer to chart in Appendix A for classification examples.

3. References

- 3.1. *ASTM E-1618, Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography Mass Spectrometry.*
- 3.2. *Mann DC, Gresham WR. Microbial degradation of gasoline in soil. J Forensic Sci. 35:913–21, 1990.*
- 3.3. *Kirkbride K, et al., Microbial degradation of petroleum hydrocarbons: implications for arson residue analysis, J Forensic Sci. 37:1585–1599, 1992.*
- 3.4. *Turner DA, Goodpaster JV. The effects of microbial degradation on ignitable liquids. Anal Bioanal Chem. 394(1):363–72, 2009.*
- 3.5. *Turner DA, Goodpaster JV. Comparing the Effects of Weathering and Microbial Degradation on Gasoline Using Principal Components Analysis. J Forensic Sci. 57(1):64-9, 2012.*



Appendix

CLASSIFICATION	n-Alkane Range (Unevaporated Liquid)	EXAMPLES
<i>Gasoline</i>	C ₄ - C ₁₃	All brands & grades of automotive gasoline, including gasohol
<i>Distillates</i>	Light C ₄ - C ₉	Petroleum ethers, pocket "cigarette" lighter fuels, some rubber cement products, VM & P Naphtha, Skelly solvent, some camping fuels
	Medium C ₈ - C ₁₃	Some mineral spirits, some paint thinners, some charcoal starters, some dry-cleaning solvents, some torch fuels, and some lamp oils, some insecticide vehicles, some polishes
	Heavy C ₈ - C ₁₆	Kerosene, No. 1 fuel oil, Jet-A fuel, some insecticide vehicles, some polishes, some paint thinners, some lamp oils
	C ₁₀ - C ₂₃₊	Diesel fuel, No. 2 fuel oil (home heating oil), highly evaporated gasoline
<i>Aromatic products</i>	Variable	Some paint/varnish removers, some automotive parts cleaners, Xylenes, Toluene based products. Some automotive parts cleaners, specialty cleaning solvents, some insecticide vehicles, some fuel additives Some insecticide vehicles, some cleaning solvents, some adhesive solvents
<i>Isoparaffinic products</i>	Variable	Specialty solvents, some aviation gases Some charcoal starters, some paint thinners, some camping fuels, some copier toners, some lamp oils Some commercial specialty solvents, some insecticide vehicles, some polishes, some adhesive solvents
<i>Normal Alkane products</i>	Variable	Solvents (pentane, hexane) Some candle oils, some copier toners, some insecticide vehicles, some polishes Some candle oils, some copier toners, NCR papers, some insecticide vehicles, some polishes
<i>Naphthenic/Paraffinic products</i>	Variable	Cyclohexane based solvents/products Some charcoal starters, some insecticide vehicles, some lamp oils Some insecticide vehicles, some lamp oils, some industrial solvents
<i>Oxygenated products</i>	Variable	Some lacquer thinners, some fuel additives, some surface preparation solvents, some industrial solvents, some metal cleaners & gloss removers

Notes: Peak spread may vary based on commercial products



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1. Scope

1.1. This policy and procedure provides guidelines for reporting analyses and comparisons from fire debris examinations. Report wording will accurately reflect what was identified in the sample and clearly communicate those findings to the reader. The wordings listed below are examples only and may be used as a guideline.

2. Procedure

2.1. Liquids that ignite

2.1.1. The liquid in Exhibit 1 contained a/an (classification), which is an ignitable liquid. Some examples of (classification) are (list appropriate examples from *ATF-LS-FD8*).

2.1.2. A/an (classification), which is an ignitable liquid, was identified in the liquid in Exhibit 1. This classification is consistent with the (product label / Safety Data Sheet (SDS)).

2.2. Liquid that did not ignite

2.2.1. Exhibit 1 contained a/an (classification), which is an ignitable liquid. Some examples of (classification) are (list appropriate examples from *ATF-LS-FD8*). It should be noted that Exhibit 1 did not burn when exposed to an open flame.

2.3. Debris

2.3.1. Exhibit 1 contained a/an (classification), which is an ignitable liquid. Some examples of (classification) are (list appropriate examples from *ATF-LS-FD8*).

2.3.2. A/An (classification) was identified in Exhibit 1. Some examples of (classification) are (list appropriate examples from *ATF-LS-FD8*). This (classification) is an ignitable liquid.

2.4. Terpenes

2.4.1. Terpenes were identified in Exhibit 1. Terpenes consistent with those detected are essential components of turpentine and are naturally occurring in some types of wood.

2.5. Single Components



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2.5.1. Exhibit 1 contained (chemical name), commonly referred to as (common name). (Common name) may be found in some (product types).

2.6. Mixtures

2.6.1. Analysis of a liquid from a commercially labeled product (i.e. paint thinner)

2.6.1.1. The liquid in Exhibit 1 contained a medium petroleum distillate (MPD) and a naphthenic/paraffinic product. Examples of MPD's are some paint thinners, mineral spirits, and some charcoal starters. Examples of naphthenic/paraffinic products include some charcoal starters, some lamp oils, and some industrial solvents. Certain brands of paint thinners contain a blend of a MPD and a naphthenic/paraffinic product.

2.6.2. Analysis of fire debris from the same case

2.6.2.1. Exhibit 1 contained a medium petroleum distillate (MPD) and a naphthenic/paraffinic product. There are some commercial products, such as some brands of paint thinners, which contain such a mixture. It could not be determined whether Exhibit 1 contained a single commercial product or a mixture of two individual products.

2.7. Alternative Fuels

2.7.1. Biodiesels

2.7.1.1. Fatty acid methyl esters (FAMEs), which are indicative of a biodiesel product, were identified in Exhibit 1. Biodiesel products are ignitable liquids.

2.7.1.2. A heavy petroleum distillate and fatty acid methyl esters (FAMEs) were identified in Exhibit 1. This combination is indicative of a biodiesel product. Biodiesel products are ignitable liquids.

2.7.2. E85

2.7.2.1. Ethanol (ethyl alcohol) and gasoline were identified in exhibit 1. Ethanol is commonly used in alcoholic beverages. Additionally, ethanol is used as an additive to gasoline and is the primary constituent of E85 fuel, which is a blend of 85% ethanol and 15% gasoline. Ethanol and gasoline are both ignitable liquids.



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2.8. Vegetable Oils and Animal Fats

2.8.1. Liquid and solid samples

2.8.1.1. Fatty acids, characteristic components of vegetable oils (or animal fats), were identified in Exhibit 1.

2.8.2. Debris samples

2.8.2.1. Fatty acids were identified in Exhibit 1. Fatty acids are naturally occurring and are found in animal fats and vegetable oils. The original source of these fatty acids could not be determined.

2.8.2.2. Fatty acids were identified in Exhibit 1. Fatty acids are naturally occurring and are found in animal fats and vegetable oils. The fatty acids present are consistent with known vegetable oil compositions; however the original source could not be determined.

2.8.3. Negative

2.8.3.1. No fatty acids were identified in Exhibit 1.

2.9. Lubricating Oils

2.9.1. Liquid samples

2.9.1.1. The liquid in Exhibit 1 contained a lubricating oil. Examples of lubricating oils include mineral oils and motor oils.

2.9.2. Debris samples

2.9.2.1. A lubricating oil was identified in Exhibit 1. Examples of lubricating oils include mineral oils and motor oils.

2.9.2.2. Exhibit 1 contained a lubricating oil. Examples of lubricating oils include mineral oils and motor oils.

2.9.3. Negative

2.9.3.1. No lubricating oils were identified in Exhibit 1.

2.10. Waxes and Petrolatum



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2.10.1. Solid samples (waxes)

2.10.1.1. Exhibit 1 contained a/an (color) (type) wax.

2.10.1.2. A/an (color) (type) wax was identified in Exhibit 1.

2.10.2. Semi-solid substances (petrolatum)

2.10.2.1. Exhibit 1 contained a semi-solid paraffin- (and/or) lubricating oil-based material. Examples of this type of product include petroleum-based greases such as petroleum jelly and hair grooming products.

2.10.3. Negative

2.10.3.1. No waxes were identified in Exhibit 1.

2.10.3.2. No semi-solid paraffin- (and/or) lubricating oil-based materials were identified in Exhibit 1.

2.10.4. No material present

2.10.4.1. Exhibit 1 contained chemical components indicative of (or consistent with) a (type) wax.

2.10.4.2. Exhibit 1 contained chemical components indicative of (or consistent with) a paraffin wax and a lubricating oil. It could not be determined if the exhibit contained two individual products or a single commercial product. Examples of lubricating oils include motor oils and mineral oils. Examples of a single commercial product containing components consistent with a paraffin wax and lubricating oil include petroleum-based greases such as petroleum jelly and hair-grooming products.

2.10.5. Examiners may identify the type of wax (paraffin, beeswax, vegetable-based, etc.) at their discretion and with supporting analytical data.

2.11. Non-Identifications

2.11.1. Non-identification of some exhibits from one case

2.11.1.1. No ignitable liquids were identified in Exhibits 2, 6 or 7.



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2.11.2. Or, if all exhibits produced negative results

2.11.2.1. Exhibits 2 through 8 were examined for the presence of ignitable liquids with negative results.

2.12. Disclaimers

2.12.1. Some situations warrant the use of a disclaimer. Examples of such situations include when the submitted material is known to potentially contain a petroleum product or when a submitted comparison sample contains a petroleum product. Refer to table in Appendix A for a list of materials which may inherently contain petroleum products. An example of appropriate disclaimer wording is:

2.12.1.1. Some (material) may inherently contain (classification).

2.12.2. Products generally attributed to pyrolysis will not be reported except under unusual circumstances or when a large amount of unexplainable product is detected.

2.13. Disposition

2.13.1. Disposition of evidence statements shall include information on extracts and liquids being returned with the evidence. Examples of these statements include:

2.13.1.1. Exhibits 1 through 5 along with their preserved extracts will be returned to the submitter.

2.13.1.2. A portion of the Exhibit 1 liquid was preserved in its original container, and the remaining liquid was destroyed.

2.14. Incendiary Device Descriptors

2.14.1. At a minimum, suspected device casework reports must contain a brief description of device components.

2.15. Test Methods and Methods of Analysis

2.15.1. The following techniques will be listed in reports, if used during the examination process:

- derivatization
- dynamic headspace concentration



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- Fourier transform infrared spectroscopy
- gas chromatography-mass spectrometry
- ignition susceptibility test
- microscopical examination
- passive headspace concentration
- physical and chemical examination
- Raman spectroscopy
- scanning electron microscopy-energy dispersive spectroscopy
- solvent extraction
- static headspace sampling
- visual examination
- X-ray diffraction
- X-ray fluorescence spectroscopy

2.15.2. All details of the methods and detectors are not required. Approved names for techniques not listed here can be found in *ATF-LS-E25*.

3. Comparative Results and the Interpretation Scale

3.1. When reporting the results of a comparative examination, the Interpretation Scale in Section 3.3 will be used to provide additional context and significance to the results, except as described below.

3.1.1. Due to the potential for heat induced chemical changes, only unburned, intact materials are typically compared.

3.1.2. In the absence of physical characteristics or when a case involves an analysis with the potential for matrix contributions, an uncompromised analytical comparison may not be possible. Therefore, the use of the scale will be up to the discretion of the examiner.

3.2. Device Components

3.2.1. Refer to *ATF-LS-E25* and *ATF-LS-TE16* for component comparison guidance.

3.2.2. Refer to *ATF-LS-TE16* for physical fit (source identification) guidance.

3.3. The following interpretation scale, in italics below, will be added as an appendix to comparative reports as described above, except when only an “exclusion” conclusion is reached. In this instance, only the “exclusion” wording is necessary in the report.



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The following descriptions are meant to provide context to the opinions reached in this report. Not every type of conclusion may be applicable in every case or for every material type.

Type I Inclusion: Source Identification – *Source Identification is the highest degree of association between items. This association provides the strongest support that the items originated from the same source as opposed to different sources. Source Identification, which includes a physical fit, is reached when the items display physical features that correspond/re-align in a manner that is not expected to be replicated.*

Type II Inclusion: Inclusion with Highly Discriminating Characteristics – *This is the highest degree of association that can be determined in the absence of a Source Identification. This type of association provides strong support that the items originated from the same source as opposed to different sources. The items correspond in all measured physical properties, chemical composition and/or microscopic characteristics and share highly discriminating characteristic(s) that would rarely be expected to occur in the relevant types of materials examined.*

Type III Inclusion: Inclusion with Discriminating Characteristics – *This type of association provides support that the items originated from the same source as opposed to different sources. The items correspond in all measured physical properties, chemical composition and/or microscopic characteristics; however, other items have been manufactured or could occur in nature that would also be indistinguishable from the examined materials.*

Type IV Inclusion: Inclusion with Limitations – *This type of association provides limited support that the items originated from the same source as opposed to different sources. Therefore, the possibility that the items came from the same source cannot be eliminated. As compared to the categories above, this type of association has decreased evidential value due to limiting factors such as the items are more commonly encountered, a limited analytical scheme was conducted, or minor variations were observed in the data.*

Inconclusive – *No conclusion could be reached regarding an inclusion or an exclusion between the items.*

Exclusion with Limitations – *This conclusion provides support that the items originated from different sources as opposed to the same source due to observed differences; however, an Exclusion conclusion was not reached due to limiting factors such as possible natural or manufactured source variations, damage or contamination that cannot be removed or avoided.*



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Exclusion – *The items display differences that support that the two items did not originate from the same source.*

3.4. Interpretation of Evidence

3.4.1. The following are examples of how the interpretation scale may be applied to specific types of evidence. Comparisons of some materials may fit into different areas of the scale depending on the circumstances of the case, the material itself, and the resulting data.

Type I Inclusion: Source Identification

- Not applicable to comparisons addressed in this document

Type II Inclusion: Inclusion with Highly Discriminating Characteristics

- Not likely to be applicable to comparisons addressed in this document

Type III Inclusion: Inclusion with Discriminating Characteristics

- Not likely to be applicable to comparisons addressed in this document

Type IV Inclusion: Inclusion with Limitations

- Vegetable oils (or animal fats) consistent in physical characteristics (appearance including color, opacity, apparent viscosity, etc.) and chemically consistent using appropriate GC-MS
- Lubricating oils consistent in physical characteristics (appearance including color, opacity, apparent viscosity, etc.) and chemically consistent using appropriate GC-MS
- Waxes and petrolatum consistent in physical characteristics (appearance, meltability, etc.) and chemically consistent using FTIR or appropriate GC-MS

Inconclusive

- Vegetable oils with minor differences in appearance, but consistent in chemical composition
- Lubricating oils with minor differences in GC-MS patterns



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- Example: Slight shift in maxima for unresolved envelope
- Waxes and petrolatum with minor differences in FTIR or GC-MS patterns or insufficient sample to run replicates
 - Example: Slight shift in maxima for paraffin waxes
- Insufficient sample available to conduct a full comparison

Exclusion with Limitations

- Not likely to be applicable to comparisons addressed in this document

Exclusion

- Vegetable oils (or animal fats) with different physical characteristics (appearance including color, opacity, apparent viscosity, etc.) or chemical compositions using appropriate GC-MS
- Lubricating oils with different physical characteristics (appearance including color, opacity, apparent viscosity, etc.) or chemical compositions using appropriate GC-MS
- Waxes and petrolatum with different physical characteristics (appearance, meltability, etc.) or chemical compositions using FTIR or appropriate GC-MS

3.5. Report Wording Examples

- 3.5.1. The following examples are meant to provide guidance for wording of comparative examinations within fire debris laboratory reports; however, exact wording is left to the discretion of the examiner.

Type I Inclusion: Source Identification

- Not applicable to comparisons addressed in this document

Type II Inclusion: Inclusion with Highly Discriminating Characteristics

- Not likely to be applicable to comparisons addressed in this document



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Type III Inclusion: Inclusion with Discriminating Characteristics

- Not likely to be applicable to comparisons addressed in this document

Type IV Inclusion: Inclusion with Limitations

- Vegetable oils
 - The clear, yellow, viscous liquid in Exhibit 1 was consistent in appearance and chemical composition to the clear, yellow, viscous liquid in Exhibit 2. The liquid in Exhibit 1 could have originated from Exhibit 2 or from any other source with the same physical characteristics and chemical composition (**Type IV Inclusion**). This type of conclusion was reached due to the commonness of this type of material.
- Lubricating oils
 - Exhibits 1 and 2 contained liquids that were consistent in physical characteristics and chemical composition. Therefore, Exhibit 2 cannot be eliminated as the source of the Exhibit 1 liquid (**Type IV Inclusion**). This type of conclusion was reached due to the limited characteristics available for comparison. Additionally, other lubricating oils manufactured to the same specifications would be indistinguishable from these liquids.
- Waxes and petrolatum
 - The red vegetable oil-based waxes in Exhibits 1 and 2 were physically and chemically consistent and could have come from the same source or any other source with the same physical and chemical characteristics (**Type IV Inclusion**). This association was limited because other waxes manufactured to the same specifications would be indistinguishable from the submitted evidence.

Inconclusive

- Vegetable oils
 - The questioned liquid in Exhibit 1 and the known liquid in Exhibit 2 differed slightly in their physical appearance (color) but were



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consistent in chemical composition. Due to the questioned liquid's exposure to (heat, the elements, etc.) no conclusion can be reached regarding an association or exclusion between the questioned and known liquids (**Inconclusive**).

- Lubricating oils
 - The lubricating oils in Exhibits 1 and 2 differed slightly in chemical composition. Therefore, due to the limitations of the analytical scheme, it could not be determined if they originated from the same source (**Inconclusive**).
- Waxes and petrolatum
 - Exhibits 1 and 2 contained white paraffin waxes that were consistent in appearance but differed slightly in chemical composition. Because of the small sample size of Exhibit 1 and the limitations of the analytical technique utilized, no conclusion could be reached regarding the association of these waxes (**Inconclusive**).

Exclusion with Limitations

- Not likely to be applicable to comparisons addressed in this document

Exclusion

- Vegetable oils
 - The Exhibit 1 clear, yellow, viscous liquid and the Exhibit 2 clear, pale yellow, viscous liquid differed in color and chemical composition. The Exhibit 1 and 2 liquids could not have originated from the same source (**Exclusion**).
- Lubricating oils
 - The lubricating oils in Exhibits 1 and 2 were consistent in physical characteristics, but different in chemical composition. Therefore, Exhibit 2 is excluded as the source of Exhibit 1 (**Exclusion**).
- Waxes and petrolatum



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- The semi-solid paraffin-based material in Exhibit 1 differed chemically from the semi-solid paraffin-based material in the Exhibit 2 container. Therefore, Exhibit 1 could not have originated from the container in Exhibit 2 (**Exclusion**).

4. References

- 4.1. *ASTM E1618, Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.*
- 4.2. *ASTM 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.*
- 4.3. *ASTM E2997, Test Method for Analysis of Biodiesel Products by Gas Chromatography-Mass Spectrometry.*
- 4.4. *Lentini JJ, et al., The petroleum-laced background. J Forensic Sci. 45(5):968-989, 2000.*
- 4.5. *Baerncopf J, Hutches K. A review of modern challenges in fire debris analysis. Forensic Sci. Int. 2014.*
- 4.6. *Baerncopf J. Prevalence of ignitable liquids in clothing with printing. Forensic Sci. Int. 2020.*



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Appendix A

SUBSTRATE	INHERENT PETROLEUM PRODUCTS
Adhesives	Aromatic products, Petroleum distillates, Isoparaffinic products
Bubble wrap	Isoparaffinic products
Carbonless forms	Medium n-alkane products
Cardboard	Limonene, 2-butoxyethanol
Clothing with printing	Aromatic products
Denim clothing	Heavy petroleum distillates
Magazines	Light aromatic products, Heavy petroleum distillates
Newspapers	Heavy petroleum distillates
Plastic bags	Isoparaffinic products, HPDs
Ropes	Heavy petroleum distillates
Shoes	Light aromatic products, Heavy petroleum distillates, Gasoline
Shop rags	Heavy petroleum distillates
Tires	Limonene
Vinyl flooring	Medium isoparaffinic products

Note: This is not an exhaustive list.



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1. Scope

- 1.1. This policy and procedure guideline establishes a standard method for extracting, derivatizing, and analyzing vegetable oils encountered in neat liquids and fire debris samples.
- 1.2. Vegetable oils contain triglycerides, which are fatty acids attached to a glycerol backbone. These are not volatile and will not be detected using typical fire debris headspace concentration techniques. However, if vegetable oils are suspected, a solvent extraction should be performed after routine ignitable liquid analysis. The extract is derivatized, which causes the triglyceride to break apart into fatty acids that are transesterified into fatty acid methyl esters (FAMES). The derivatized extract is analyzed on a specific GC column to identify the presence of FAMES.
- 1.3. Co-extraction of interfering compounds may be a concern with some porous matrices using this technique.
- 1.4. It should be noted that this procedure will also work for animal fats, such as butter, as these are similar in chemical composition to vegetable oils.

2. Instrumentation/Reagents

2.1. Materials

- 2.1.1. Pentane
- 2.1.2. Derivatization reagent, approximately 2N KOH in methanol
- 2.1.3. Drying agent, anhydrous sodium sulfate or equivalent
- 2.1.4. Filter apparatus free of extractable hydrocarbons
- 2.1.5. Beakers or similar containers

2.2. GC-MS

- 2.2.1. Refer to *ATF-LS-FD1*.
- 2.2.2. A capillary, bonded phase polar column suitable for FAME analysis, such as a DB-23 or equivalent.

2.3. Quality Control and References



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2.3.1. Commercially available test mixture, such as NuChek Prep #17AA', or equivalent containing the following FAMES: C8:0, C10:0, C12:0, C14:0, C16:0, C16:1, C17:0, C18:0, C18:1, C18:2, C18:3, C20:0, C22:0, C22:1, and C24:0.

2.3.2. Reference collection of various vegetable oils

3. Safety Considerations

3.1. Personal protective equipment such as lab coat, safety glasses and gloves should be available and used, if necessary, when preparing samples and conducting analyses.

3.2. Pentane should be used in a fume hood and kept away from open flames and sparks.

4. Procedure

4.1. If ignitable liquid analysis is required, it shall be performed prior to analysis for oils and fats as the methods discussed below are destructive techniques. A fire debris sample (or sub-sample) or an aliquot of a liquid will be initially analyzed for ignitable liquid residues following ATF Fire Debris protocols. The same sample of fire debris (or a different sub-sample) or an additional aliquot of liquid is then extracted with an organic solvent, derivatized and analyzed on a GC-MS.

4.2. The following procedures are suggested for the extraction of neat liquids and debris samples and may be modified depending on sample material and size.

4.2.1. Neat liquids

4.2.1.1. Place approximately one drop of liquid into a suitable container and add approximately 10 mL pentane.

4.2.1.2 A portion of the pentane extract, prior to derivatization, shall be preserved with a charcoal strip or portion thereof. Place the preserved extract in a vial labeled with case and exhibit numbers.

4.2.1.2.1 When a portion of an original liquid sample will be preserved and returned, it may not be necessary to preserve the extract.

4.2.1.3. Add approximately 0.5 mL of derivatization agent.

4.2.1.4. Seal container, shake, and allow layers to separate.



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4.2.1.5. Remove the top layer and filter as deemed necessary.

4.2.1.6. Analyze filtered extract on GC-MS.

4.2.2. Debris samples

4.2.2.3. Open and examine the fire debris sample to determine that it is consistent with its description and perform routine ignitable liquid analysis, if required.

4.2.2.4. Extract sample (or suitable sub-sample) in its original container or in a disposable or new container by adding sufficient pentane to moisten the sample and then thoroughly mixing the pentane and debris. Simple rinsing of nonporous surfaces may not adequately separate residues.

4.2.2.5. Remove the extract from the debris. NOTE: If water is present, after removal of the extract from the debris, allow the pentane and water to separate. Decant the solvent layer into a clean beaker and add a drying agent (such as anhydrous sodium sulfate) to the sample extract until the drying agent flows freely without clumping. The amount of drying agent added will vary depending on the amount of water present.

4.2.2.6. A portion of the pentane extract, prior to derivatization, shall be preserved with a charcoal strip or portion thereof. Place the preserved extract in a vial labeled with case and exhibit numbers.

4.2.2.7. Transfer at least a 2 mL portion of the extract to a suitable container and add approximately 0.5 mL of derivatization agent.

4.2.2.8. Seal container, shake, and allow layers to separate.

4.2.2.9. Remove the top layer and filter as deemed necessary.

4.2.2.10. Analyze filtered extract on GC-MS.

4.3. Concentration

4.3.1. If the initial GC-MS results indicate that the sample needs to be concentrated, perform the concentration in containers free of extractable hydrocarbons (disposable or new containers). Place the container in a chemical fume hood and evaporate at room temperature.



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4.3.2. Compressed dry nitrogen can be used to accelerate evaporation. Extreme caution should be used if applying heat. Heating mantles are not recommended; however, a steam bath may be used to accelerate evaporation. Use of heat for concentration will cause the loss of the more volatile compounds present in the sample.

5. Quality Assurance and Controls

5.1. Tune

5.1.1. The instrument will be tuned using perfluorotributylamine (PFTBA) no more than one week prior to casework analysis. The tune report shall be examined to ensure that appropriate parameters are within their normal expected range specified by the manufacturer and initialed by the examiner performing the tune. Documentation that the values were verified as meeting manufacturer's specifications will be maintained in the instrument log. Maintain tune reports near the instrument.

5.2. Quality Control Mix

5.2.1. Each day the instrument is used for analysis, a quality control (QC) sample shall be run. This QC shall be a commercially available test mixture, such as NuChek Prep #17AA', or equivalent containing the following FAMES: C8:0, C10:0, C12:0, C14:0, C16:0, C16:1, C17:0, C18:0, C18:1, C18:2, C18:3, C20:0, C22:0, C22:1, and C24:0. The suggested preparation is 100 mg of the test mixture in approximately 200 mL of methanol. However, this can be adjusted based on the sensitivity of the instrument. In cases where sequences carry over into the next day, the quality control sample will be considered still valid for the sequence. Additional quality control samples must be run for sequences continuing beyond the next day and each day thereafter.

5.2.2. When using a standard fire debris column and method to identify FAMES, follow the QC procedures in *ATF-LS-FDI*.

5.2.3. When using a FAME-specific column and method to identify and compare FAMES, follow these procedures for the FAME QC. Due to the infrequent use of this method, QC acceptance criteria procedures in *ATF-LS-FDI* are not applicable. The QC data shall be assessed each time it is run to ensure overall good chromatography. All peaks in the QC will be present with a S/N of at least 5:1 and eluting in an order appropriate for the selected column chemistry. The FAMES C18:0, C18:1, C18:2, and C18:3 will be baseline resolved. Mass spectral data for C16:0, C18:0, C18:1, C18:2, and C18:3 will be evaluated to ensure the molecular ion is present and a library search hit provides a quality score of at least 85. When the criteria mentioned above are not met (i.e. "fail"), the error shall be corrected



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and documented in the logbook. Sample data collected after a QC fails will be re-analyzed after the issue(s) have been resolved. Evidence examination may proceed after it has been demonstrated that the instrument is functioning properly, according to the specifications defined by the instrument manufacturer and this procedure, and that the system and solvent(s) are free of contamination (i.e. “pass”).

5.2.4. A hard copy of the QC data (TIC and library search) will be printed and initialed by the person evaluating the data. Documentation that the data was verified as meeting the criteria listed above will be maintained in the instrument log. The initialed QC printout will be maintained near the instrument and its use (including, at a minimum, the QC name and date prepared) will be documented in the case record.

5.3. Materials Control and Solvent Blanks

5.3.1. Run a materials control with case samples to ensure that the method of analysis is free of contamination. Prepare the materials control the same way as the sample(s), using the same materials (solvent, disposable beakers, filters, drying agent, etc.).

5.4.1.1 If the sample extract is concentrated, the materials control will also be concentrated. If the sample extract is diluted, the materials control does not need to be diluted further.

5.3.2. Run a solvent blank before the QC and between each case sample. These blanks ensure that no carryover of FAMES from the previous sample exists and that the system is not contaminated with FAMES.

5.3.3. If extraneous peaks are noted in either the materials control or the solvent blanks, an attempt will be made to determine the source of these and will be documented in the case notes. When solvent blanks indicate potential carryover or contamination from FAMES, the affected exhibits will be re-analyzed. The data for the materials control and solvent blanks will be maintained in the case record.

5.4. Reference Liquids and FAME Identification

5.4.1. Run a reference liquid containing fatty acids contemporaneously with the case samples in order to verify the performance of the derivatization agent. If FAMES are not observed in the reference liquid, prepare a new derivatization agent and reanalyze all case samples once the agent’s performance is verified.



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- 5.4.2. Identify FAMEs by comparing retention time (RT) and spectral data to a known reference liquid or the FAME QC. The retention times of the questioned (Q) and known (K) peaks shall be within 2%. This will be determined using the following equation: $[RT(K) - RT(Q)]/RT(K) * 100\%$.
- 5.4.3. The relative abundances of ions in the spectra of the unknown sample and the reference liquid or FAME QC will have general agreement across the majority of the ions. The unknown and known samples should have the same base peak and the same molecular ion, where the molecular ion is present. Isotopic ions present in the reference spectrum shall be present in similar proportions in the unknown sample spectrum; low abundance ions (less than 5% of the total spectral abundance) may be absent without precluding an identification. Background subtraction may be necessary to remove any background contribution to the sample, such as air/water. There shall be no unexplained extraneous ions above 30% relative abundance.
- 5.4.4. A standard fire debris column and method may be used for general identification (refer to *ATF-LS-FDI*). However, peak resolution is not sufficient for comparative purposes. If further information (such as possible type(s) of vegetable oil/fat or tendency to self-heat) or comparison is requested, the samples will be analyzed using a FAME-specific column. If possible, the compared vegetable oil samples should be analyzed in triplicate to assess any minor instrumental and concentration variations.

5.5. Maintenance

- 5.5.1. Refer to *ATF-LS-FDI*.

5.6. Preserved Extracts

- 5.6.1. Each new batch/lot number of charcoal strips shall be tested prior to being implemented for casework as per *ATF-LS-FD3*.
- 5.6.2. Note the lot number of the charcoal strips used in casework in the case record.
- 5.6.3. All preserved extracts shall be returned with the evidence to the submitting agency.
- 5.6.4. When fire debris evidence may need DNA and/or print examinations, the preserved charcoal strip(s) will be sub-designated and returned with the evidence in a separate convenience container.



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6. References

- 6.1. *ASTM E 2881, Test Method for Extraction and Derivatization of Vegetable Oils and Fats from Fire Debris and Liquid Samples with Analysis by Gas Chromatography-Mass Spectrometry.*
- 6.2. *ASTM E1618, Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.*
- 6.3. *ASTM E1386, Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction.*
- 6.4. *Safety Data Sheets for Pentane, Methanol, Potassium Hydroxide (KOH), and anhydrous sodium sulfate*
- 6.5. *L Schwenk and M Reardon, "Practical aspects of analyzing vegetable oils in fire debris," Journal of Forensic Sciences, 54(4): 874-80, 2009.*
- 6.6. *A Gambrel and M Reardon, "Extraction, derivatization, and analysis of vegetable oils from fire debris," Journal of Forensic Sciences, 53(6):1372-80, 2008.*
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- 6.8. *E Stauffer, "A review of the analysis of vegetable oil residues from fire debris samples: Spontaneous ignition, vegetable oils, and the forensic approach," Journal of Forensic Sciences, 50:1091-1100, 2005.*
- 6.9. *E Stauffer, D Byron, and D Carpenter, Vegetable Oil Workshop, <http://www.swissforensic.org/publications/aafsvor/aafsvor.html>, American Academy of Forensic Sciences Annual Meeting, 2007.*



ATF-LS-FD12 Ignition Susceptibility	ID: 1626 Revision: 5
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1. Scope

- 1.1. This policy and procedure establishes a method for performing ignition susceptibility.
- 1.2. Ignition is the act of kindling or setting on fire any combustible substance. Ignitable liquids all exhibit burning properties.
- 1.3. Initial testing/screening of unknown liquids is performed to determine if they will support combustion. Additional testing is required for positive identification of the suspect material. The physical condition of the sample needs to be taken into consideration if the sample does not readily burn as expected.

2. Instrumentation/Reagents

- 2.1. Pipette, cotton-tipped swab, crucible, or other suitable instrument
- 2.2. Tongs (used for holding burned matrix)
- 2.3. Ignition source (burner, match, etc.)
- 2.4. Reference ignitable liquids – 1-2 drops of a known ignitable liquid that is representative of the suspected ignitable liquid sample may be used for comparative purposes.

3. Safety Considerations

- 3.1. Use as small a sample as possible.
- 3.2. Make sure the area is clear of any flammable or explosive materials (solvents, containers of explosives, etc.).
- 3.3. Perform test in a hood, if practical, and wear eye protection.
- 3.4. Extinguishment of matrices will be performed in a safe and practical manner.

4. Procedure

- 4.1. Remove one to two drops of suspected ignitable liquid with a pipette, cotton-tipped swab or other sampling device.
- 4.2. Place sample over ignition source (flame) and observe resulting effect.

5. Quality Assurance and Controls



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5.1. The physical condition of the sample, along with the ignition technique, need to be taken into consideration if the sample does not readily burn as expected. Aqueous liquids may affect the ignition susceptibility of a liquid.

6. References

6.1. Speight A., Taylor and Frances Group, "Petroleum Chemistry and Refining," 1977

6.2. Speight A., John Wiley & Sons Inc, "Handbook of Petroleum Analysis," 2001

6.3. Speight J. "The Chemistry and Technology of Petroleum," Chemical Industries, Vol. 76

6.4. Wiley John & Sons Inc. Kirk and Othmer, "Encyclopedia of Chemical Technology," Vol.26, 2001.

6.5. Wiley John & Sons Inc., "Petroleum (Refinery Process, Survey), and Petroleum Products," 2008



ATF-LS-FD13 Fire Debris Glossary	ID: 1973 Revision: 4
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1. Scope

1.1. This glossary is intended for use as a guide for ATF Forensic Science Laboratory Fire Debris Chemists. It is not intended to be all inclusive. Commonly accepted abbreviations are acceptable and do not need to be defined. Abbreviations included in other discipline glossaries can be used and will not be duplicated in this glossary.

1,2,4-TMB	1,2,4-trimethylbenzene
ALK	Alkane
ARO	Aromatic
C _x	hydrocarbon or hydrocarbon chain containing x number of carbons
C ₄ -abs	C ₄ -alkylbenzenes or tetramethylbenzenes
C-strip	charcoal strip
EIP	extracted ion profile
ER	evidence room
ET	evidence tape
Ex	exhibit
Evap	evaporated
FA	fatty acid
FAME	fatty acid methyl ester
FD	fire debris
HHS	heated headspace
HPD	heavy petroleum distillate
HPP	heavy petroleum product
ID	identify
IL	ignitable liquid
IND	indane
ISO	isoparaffinic
IST	ignition susceptibility test
LPD	light petroleum distillate
LPP	light petroleum product
MC	materials control
MPD	medium petroleum distillate
MPP	medium petroleum product
n-alk	normal alkane
N/P	naphthenic/paraffinic
OCP	olefin/cycloparaffin
o/n	overnight
PCA	passive charcoal adsorption
PCE	passive charcoal extraction
PHC	passive headspace concentration



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PHS	passive headspace
PNA	polynuclear aromatic hydrocarbons
QC	quality control
Ref	reference
SB	system blank
SE	solvent extraction
TIC	total ion chromatogram
μscope	microscope



ATF-LS-FD14 Analysis of Waxes and Petrolatum	ID: 2162 Revision: 5
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1. Scope

- 1.1. This policy and procedure guideline establishes a standard method for the analysis and identification of waxes and petrolatum encountered in evidentiary samples.
- 1.2. Petroleum-derived, paraffin waxes contain normal alkanes above C₁₇ with a lesser abundance of branched alkanes. Some waxes, including naturally-derived waxes such as beeswax and soybean wax, may contain a variety of esters, acids, and other oxygenated compounds instead of or in addition to alkanes, the distribution of which may differ from petroleum-derived waxes. Petrolatum, also known as petroleum jelly, is generally comprised of wax and lubricating oil components. Since petrolatum is a semi-solid substance, the analysis procedures are similar to waxes.

2. Instrumentation/Reagents

- 2.1. Appropriate solvents, such as trichloroethylene (TCE) or pentane
- 2.2. Instruments capable of identifying organic chemical unknowns, including Fourier transform Infrared Spectroscopy (FTIR) and high temperature gas chromatography-mass spectrometry (HTGC-MS).

3. Safety Considerations

- 3.1. Personal protective equipment such as lab coat, safety glasses and gloves should be available and used, if necessary, when preparing samples and conducting analyses.
- 3.2. When using solvents, such as trichloroethylene (TCE) and pentane, examiners should utilize laboratory hoods to minimize exposure to solvent vapors.
- 3.3. Safety Data Sheet references for solvents should be available and read by the user.
- 3.4. Pentane should be kept away from open flames and sparks.

4. Procedure

- 4.1. Wax and petrolatum may be *identified* using physical characteristics (e.g. appearance, texture, solubility, pliability, clarity, melting point) and/or optical properties of the wax crystals using polarized light microscopy (PLM) in conjunction with at least one of the following techniques:
 - Fourier transform infrared spectroscopy (FTIR): *ATF-LS-E6*
 - High-temperature gas chromatography-mass spectrometry (HTGC-MS): *ATF-LS-*



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FD2

- Sample derivatization and analysis of fatty acid methyl esters (FAMES) using GC-MS: *ATF-LS-FD10* (Note: may not apply for petrolatum.)
- Pyrolysis gas chromatography-mass spectrometry (PyGC-MS): *ATF-LS-TE4*

- 4.2. Samples to be analyzed by HTGC-MS will be dissolved in a solvent at an appropriate concentration, such as approximately 0.1% by weight. Heat may be necessary for complete dissolution of the solid sample. This preparation may be modified at examiner discretion.
- 4.3. Samples to be analyzed by the FAME GC-MS must be extracted and derivatized according to the *ATF-LS-FD10*.
- 4.4. Paraffin waxes and petrolatum may be extracted during routine ignitable liquid testing using passive charcoal adsorption (*ATF-LS-FD3*) or solvent extraction (*ATF-LS-FD3*). If a physical examination of the debris does not result in finding solid material to further analyze, the wax or petrolatum should be considered indicated only and an identification should not be made. If petrolatum is suspected, examiners should also refer to the *ATF-LS-FD15* for further guidance on sample preparation.
- 4.5. Wax and petrolatum comparisons must be performed with extreme caution on solid/semi-solid samples or visible residues only and shall include analysis by HTGC-MS, PyGC-MS, or FAME GC-MS (if applicable). The identification and comparison of waxes and petrolatum by GC-MS is done using overall pattern comparison and the retention time and spectral data compared with a known reference. Minor differences in GC-MS patterns do not necessarily constitute a conclusion of “chemically different”. If possible, at least three replicates of the questioned and known samples shall be prepared in a similar manner and analyzed to determine if differences seen can be attributed to the samples or the instrument. While minor heating for sample dissolution is acceptable, it should be noted that waxes can also be affected by excessive heat, resulting in changes in the chromatographic pattern. Refer to *ATF-LS-FD9* for suggested wording for comparisons. Note that sample concentration may affect the resulting data. When feasible, samples should be prepared so that the extract concentrations are as similar as possible.

5. Quality Assurance and Controls

- 5.1. Examiners should follow the appropriate instrument protocols for quality control for each instrument.
- 5.2. Error potentials for each instrumental technique are referred to in the instrument protocols.



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5.3. Sample concentration may adversely alter the chromatographic data and affect the resulting conclusion. Care should be taken to ensure that samples being compared are prepared in a similar manner and at a similar concentration.

6. References

- 6.1. *ASTM D5442, Test Method for Analysis of Petroleum Waxes by Gas Chromatography.*
- 6.2. *ASTM 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.*
- 6.3. *Safety Data Sheets for appropriate solvents and chemicals*
- 6.4. *Barker, A, The chromatographic analysis of refined and synthetic waxes. In: Adlard, ER (ed.), Chromatography in the petroleum industry, Elsevier, 1995, pp 55-93.*
- 6.5. *Giles, JJ, The analysis of waxes and greases using high resolution gas chromatography, Forensic Science Society, Vol 27, 1987, pp 231-9.*
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- 6.7. *Jimenez, JJ, Bernal, JL, Aumente, S, del Nozal, MJ, Martin, MT, and Bernal Jr., J, Quality assurance of commercial beeswax Part I. Gas chromatography-electron impact ionization mass spectrometry of hydrocarbons and monoesters, Journal of Chromatography A, Vol 1024, 2004, pp 147-154.*
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- 6.9. *Kuk, RJ, Analysis of artificial fireplace logs by high temperature gas chromatography, Journal of Forensic Science, Vol 47, 2002, pp 1288-93.*
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- 6.12. *Blackledge, R, Cabiness, L., Examination for Petrolatum Based Lubricants in Evidence from Rapes and Sodomies. Journal of Forensic Science, Vol 28(2), April 1983, pp 451-462.*



ATF-LS-FD15 Analysis of Lubricating Oils	ID: 2179 Revision: 4
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1. Scope

- 1.1. This policy and procedure establishes a standard method for extracting and analyzing lubricating oils encountered in neat liquids and debris samples.
- 1.2. Lubricating oils are derived from a heavy boiling fraction of crude oil. They generally range from C₁₈ to C₄₀₊ and contain an unresolved envelope of hydrocarbons. There are several types of lubricating oils which include conventional lubricating oils (from crude oil), synthetic lubricating oils (modified petroleum components), and synthetic blends (mixture of conventional and synthetic). Lubricating oils may also contain additional components such as glycols and esters that are added to alter the properties of the oil.
- 1.3. Lubricating oils are not volatile and may not be detected using typical fire debris headspace concentration techniques. If lubricating oils are suspected, a solvent extraction should be performed after routine ignitable liquid or explosive analysis.
- 1.4. Though some components of lubricating oils can be observed using a typical gas chromatograph-mass spectrometer (GC-MS), a high temperature instrument (HTGC-MS) may be necessary for identification of heavier oils. For comparisons of lubricating oils, HTGC-MS analysis is required.

2. Instrumentation/Reagents

2.1. Materials

- 2.1.1. Pentane or other appropriate organic solvent
- 2.1.2. Drying agent, anhydrous sodium sulfate or equivalent
- 2.1.3. Filter apparatus free of extractable hydrocarbons
- 2.1.4. Beakers or similar containers

2.2. GC-MS

- 2.2.1. Refer to *ATF-LS-FD1* and *ATF-LS-FD2*.

2.3. Quality Control and References

- 2.3.1. Refer to *ATF-LS-FD1* and *ATF-LS-FD2* for the appropriate quality control procedures.



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2.3.2. Reference collection of various lubricating oils.

3. Safety Considerations

3.1. Personal protective equipment including but not limited to safety glasses, gloves, and lab coat should be worn.

3.2. Pentane should be used in a fume hood and kept away from open flames and sparks.

4. Procedure

4.1. The following procedures are suggested for the extraction of neat liquids and debris samples and may be modified depending on sample material and size. HTGC-MS is required for comparisons of lubricating oils (if requested by the submitter).

4.1.1. Neat liquids:

4.1.1.1. Dilute the sample in an appropriate volume of solvent, such as pentane, to obtain acceptable instrument signal.

4.1.1.2. Filter as necessary and analyze the extract by GC-MS and/or HTGC-MS.

4.1.2. Debris Samples:

4.1.2.1. Lubricating oils may be extracted during routine ignitable liquid testing using passive charcoal adsorption (*ATF-LS-FD3*); however, solvent extraction (*ATF-LS-FD3*) may be necessary to recover heavier oils. Solvent extraction of debris should be performed after routine ignitable liquid or explosives analysis.

4.1.2.2. Co-extraction of interfering compounds may be a concern with some porous matrices using this technique. Clean-up procedures using silica gel cartridges or similar products may be needed to separate lubricating oils from interfering products.

4.2. For identification of lubricating oils, samples should be compared to a reference lubricating oil with similar characteristics such as peak distribution and chemical content. Comparisons of lubricating oils must be performed with extreme caution and must include analysis by HTGC-MS. Comparisons should be based on overall chromatographic patterns such as shape of the unresolved envelope and apex position. Minor differences do not necessarily indicate that the two samples are chemically different and may be due to instrumental variation. Additionally, comparisons of



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lubricating oils may be affected by sample concentration. Oils that are too concentrated will skew the pattern and potentially affect the comparison conclusion. If possible, the compared lubricating oils should be analyzed in triplicate to assess any minor instrumental and concentration variations.

5. Quality Assurance and Controls

5.1. Examiners should follow the Quality Control/Quality Assurance procedures outlined in the *ATF-LS-FD1* and *ATF-LS-FD2*.

5.2. Sources of Error

5.2.1. Error potentials for GC-MS and HTGC-MS are referred to in the instrument protocols.

5.2.2. Concentration differences may affect conclusions; thus every effort should be made to ensure that the oils being compared are similar in abundance/concentration.

5.3. Extract Storage

5.3.1. All solvent extracts will be preserved with charcoal and returned with the evidence to the submitting agency.

6. References

6.1. *ASTM E1386, Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction*

6.2. *ASTM E1618, Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry*

6.3. *Safety Data Sheet for Pentane and other applicable solvents and chemicals*

6.4. Gary JH, Handwerk GE. *Petroleum Refining Technology and Economics*. 4th edition. New York: Marcel Dekker, 2001.

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