



ATF-LS-FD1 Gas Chromatography-Mass Spectrometry (GC-MS)	Published Online: March 2018
Authority: Technical Leader	
Unofficial Copy; May Not Be Most Current Version	Page: 1 of 5

- I. **Scope:** This policy and procedure guideline 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).

II. **References:**

1. ASTM E1618 Standard Guide for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.
2. Newman, R., Gilbert, M. and Lothridge, K. "GC-MS Guide to Ignitable Liquids," CRC Press 1998.
3. Smith, R. M., Analytical Chemistry, Vol. 54, No. 13, November 1982, pp 1399A-1409A.
4. 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.
5. Smith, R. M., "Mass Chromatographic Analysis of Arson Accelerants," Journal of Forensic Science Vol. 28(2), pp 318-329.
6. Gas Chromatograph-Mass Spectrometer Maintenance and Troubleshooting Manual
7. Instrument Hardware Manual

III. **Apparatus/Reagents:**

A gas chromatograph-mass spectrometer with the following features:

Gas Chromatograph:

- Capable of using capillary columns and being interfaced to a mass spectrometer.
- Sample Inlet System that can be operated in either split or splitless mode with capillary columns
- A capillary, bonded phase, methylsilicone column or equivalent column
- A column oven capable of reproducible temperature program operation in the range from 30 to 300°C

Mass Spectrometer:

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

Computerized data station:

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

- The system must be 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.
- Syringes capable of introducing a sample size in the range from 0.1 to 10.0 μL .
- 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.
- Commercially available reagent grade acetone, methyl ethyl ketone, ethanol and isopropanol.
- 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.

IV. Safety Precautions:

1. Whenever facilities permit, vacuum pumps, mechanical and/or turbo, should be properly vented to a fume hood or filtered prior to start-up of the GC-MS.
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. Personal protective equipment such as lab coat, safety glasses and gloves should be available and used when preparing samples and conducting GC-MS analyses.
4. All gases should be properly secured/stored. Pressure regulators should be inspected whenever the bottles are replaced.
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).
6. MSDS references for solvents should be available and read by user.

V. Procedures:

Quality Control procedures listed for sample preparation methods should be followed.

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 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

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.

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: $[RT(K) - RT(Q)]/RT(K) * 100\%$.

Ignitable liquids will be classified as defined in the Ignitable Liquid Classification System (ATF Fire Debris protocol). ASTM E 1618 may offer guidance in correctly classifying the ignitable liquid.

VI. Quality Control:

Autotune

The instrument will be autotuned 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. Initials on the tune printout indicate that the values were verified as meeting manufacturer's specifications. Tune reports are maintained near the instrument.

Quality Control Mix

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 µL 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 µL Restek (or equivalent), 5 µL ethanol, 3 µL acetone, 2 µL IPA, and 3 µL 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.

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 0.03 minutes of the QC analyzed after the last instrument maintenance that affects chromatography.

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 source of 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. Initials on the QC printout indicate that the data was verified as meeting the criteria listed above. 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.

System and Solvent Blanks

A system blank or materials control (defined in each method of analysis) will be run for cases to insure that the instrument, solvent and method of analysis are free of contamination. A solvent blank will be run before the QC and between each case sample and shall be free of extraneous peaks. 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, the source of these shall be determined and documented in the case notes. When solvent blanks indicate potential carryover or contamination, the affected samples and/or references will be re-analyzed. The data for the system blank(s) and solvent blanks will be maintained in the case record.

Maintenance

Changes to existing data acquisition methods and data analysis macros shall be performance checked to verify the appropriate response is given.

When changes outside the acceptable parameters occur in the “autotune” or the quality control sample check the following items:

- Septum
- Column nut
- GC/MS interface nut
- Injection Port Liner
- Capillary column
- Mass Spectrometer Source

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.

The carrier gas cylinder will be changed as necessary.