Technical Procedure for Gas Chromatography (GC-FID)

1.0 Purpose – This technical procedure shall be followed for the operation of the gas chromatograph (GC-FID).

2.0 Scope – This procedure applies to all gas chromatograph-flame ionization detectors set up for liquid samples in the Trace Evidence Section.

3.0 Definitions – N/A

4.0 Equipment, Materials, and Reagents

4.1 Equipment

- Agilent 6890 Gas Chromatograph with a Flame Ionization Detector
- Agilent 7890 Gas Chromatograph with a Flame Ionization Detector
- Hydrogen generators

4.2 Materials

- Auto sampler vials, 150 µL vial inserts, and crimp seals
- Vial crimper and decrimper
- 10 micro-liter autosampler syringe
- A non-polar capillary column with a (5 %-Phenyl)-methylpolysiloxane stationary phase
- 50 % residual gasoline reference material
- 100 % diesel fuel reference material

4.3 Reagents

- Acetone
- Carbon disulfide - Reagent A.C.S. grade
- Petroleum ether – Optima Grade
- Hydrogen gas
- Air –Zero grade
- Nitrogen – UHP grade

5.0 Procedure

5.1 Start-up and Performance Check

5.1.1 The GC-FID shall be kept on at all times.

5.1.2 Monthly GC-FID Performance Check

5.1.2.1 To ensure performance, the following samples shall be analyzed and the chromatographs electronically archived each month the instrument is in use.

If the instrument was not in use the previous month, the performance check shall be performed prior to casework.

- Petroleum ether blank
- Carbon disulfide adsorption elution blank
- 50 % residual gasoline reference material
- 100 % diesel fuel reference material

5.1.2.2 If there is a shift in peak retention time by 0.2 minute or more or a change in peak resolution, maintenance shall be performed or a service engineer must be called. Once maintenance has been performed, the samples shall be re-examined. If the resulting chromatographs are within the limits, the instrument may be used for casework.

5.1.3 New Instrument Setup and Performance Verification:

5.1.3.1 New GC-FID instruments shall be installed by a certified engineer according to the manufacturer's guidelines.

5.1.3.2 The samples used in the monthly GC-FID performance check shall be analyzed on the instrument and the resulting chromatographs compared to the same samples acquired on similar instruments. The resulting chromatographs shall have similar peak ratios and component separation in order for the new instrument to be utilized for casework.

5.2 Casework Analysis

5.2.1 Samples shall be prepared for examination according to the appropriate technical procedure.

5.2.2 Once the samples are prepared, they shall be analyzed using one of the GC-FID programs located in the **Attachments**.

5.2.3 Once data has been collected, the resulting data file shall be loaded in the Data Analysis program for examination. The resulting chromatograph shall be included in the Case Record.

5.2.4 After the method has run, allow the instrument to return to the set program and leave the GC-FID on.

5.3 Standards and Controls

5.3.1 There shall be at least one solvent blank run for each case.

5.3.1.1 If the sample being analyzed is a neat sample, petroleum ether or carbon disulfide shall be used for the blank.

5.3.1.2 If an extraction is performed using acetone, carbon disulfide, petroleum ether, etc., the same solvent shall be used for the blank.

5.3.2 The resulting data from the blanks shall not have peaks that interfere with the analysis.

5.3.3 The wash bottles on the autosampler shall be filled with acetone.

5.4 Maintenance

5.4.1 The septa for the injection port shall be changed at least monthly when in use.

5.4.2 The injection port liner shall be changed at least yearly.

5.5 Sampling and Sample Selection – N/A

5.6 Calculations – N/A

5.7 Uncertainty of Measurement – N/A

6.0 Limitations – N/A

7.0 Safety

7.1 Carbon disulfide may be toxic. Consult Safety Data Sheets for information on safe use of reagents listed in this procedure.

7.2 Burns may result from contact with hot items such as containers, syringes, liners, and septa nuts.

7.3 Syringe needles are sharp and can easily puncture skin.

7.4 Care shall be exercised when using the above-listed items or procedures.

8.0 References

ATF National Laboratory Center Class. "Laboratory Detection and Identification of Accelerants Found in Arson Debris."

Saferstein, R. *Forensic Science Handbook.* Volume I, 2nd edition. Upper Saddle River, NJ: Prentice Hall, 2002. Chapter 9: Arson and Explosive Investigation. pp. 479-524.

ASTM Guidelines

ASTM Standard E1387, 2001, "Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography." ASTM International, West Conshohocken, PA, 2001, www.astm.org.

9.0 Records

- Maintenance log
- Use log
- Performance Check Chromatographs
- Request for Instrumental Examination of Evidence

10.0 Attachments

- Appendix A – Instrument Program Parameters for Agilent 6890 Gas Chromatograph with a Flame Ionization Detector
- Appendix B – Instrument Program Parameters for Agilent 7890 Gas Chromatograph with a Flame Ionization Detector

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original ISO Document
02/01/2013	2	Request for Instrumental Examination of Evidence was added as a record. 5.5 Instrument parameters for the Western Regional Laboratory were added.
09/30/2013	3	Quality Control Check log was removed from 9.0; Quality Control Check Chromatograms are stored electronically.
10/18/2013	4	Added issuing authority to header
08/29/2014	5	Updated header to Physical Evidence Section – Trace Unit, issuing authority to Physical Evidence Section Forensic Scientist Manager. Updated all references in procedure from Trace Evidence Section to Trace Unit.
03/20/2015	6	Changed requirements for monthly performance check in 5.1.2.1 Added "when in use" to 5.7.1
12/11/2015	7	4.2 and 5.2.3 – reworded for consistency between GC-FID and GC-MS procedures; changed 100 to 150 Removed extra periods and "#2 fuel oil" in 5.1.2.1 Added "for examination" in 5.2.3
11/22/2019	8	Changed name of section in header and 2.0 Added hydrogen generators to 4.1 Removed suggested columns from 4.2 Replaced "calibration" with "performance check" in 5.1 Changed reference in 5.2.2 to Attachments Relocated and updated instrument program parameters in 5.3 and 5.4 to Attachments as Appendix A and Appendix B Removed Western Laboratory program parameters (5.5) Removed chloroform in 5.6.1.2 Removed "Material" in 7.1 and clarified its application to all reagents Changed Quality Control Check to Performance Check under 9.0 Added Appendix A and Appendix B under 10.0

Appendix A

Instrument Program Parameters

Agilent 6890 Gas Chromatograph with a Flame Ionization Detector

Injection – Using the autosampler with a 10 µL syringe injection, perform three pre-inject and eight post-inject solvent washes from each wash bottle. Inject the sample as indicated below.

Inlet – Run in split mode at 240 °C with the split ratio indicated below.

Carrier Gas – Hydrogen

Oven – Run a temperature program starting at 80.0 °C for 0.00 minutes. Then ramp at 20.0 °C per minute until 280 °C is obtained. Hold at 280 °C for 2.00 minutes.

Column –Use a (5 %-Phenyl)-methylpolysiloxane (such as an HP-5MS) column which is 0.25 mm in diameter that is approximately 30 m long with a 0.25 µm film thickness. The column shall be kept at a constant flow of 1.6 mL/min.

Flame Ionization Detector – The detector temperature shall be set at 300 °C. The flow rate for hydrogen is 30.0 mL/min, for air is 400.0 mL/min, and the makeup flow of nitrogen is 25.0 mL/min.

Method Name	Injection	Split Ratio
ARSONL	Inject a 1.0 µL sample with a fast plunger speed, three sample washes, and three sample pumps.	50:1
ARSON	Inject a 5.0 µL sample with a fast plunger speed, no sample washes, and three sample pumps.	50:1
FIREL	Inject a 1.0 µL sample with a fast plunger speed, no sample washes, and three sample pumps.	75:1
FIRE	Inject a 1.0 µL sample with a fast plunger speed, no sample washes, and three sample pumps.	20:1

Appendix B

Instrument Program Parameters

Agilent 7890 Gas Chromatograph with a Flame Ionization Detector

Injection – Using the autosampler with a 10 µL syringe injection, perform three pre-inject and eight post-inject solvent washes from each wash bottle. Inject the sample as indicated below.

Inlet – Run in split mode at 240 °C with the split ratio indicated below.

Carrier Gas – Hydrogen

Oven – Run a temperature program starting at 80.0 °C for 0.00 minutes. Then ramp at 20.0 °C per minute until 280 °C is obtained. Hold at 280 °C for 2.00 minutes.

Column –Use a (5 %-Phenyl)-methylpolysiloxane (such as an HP-5) column which is 0.32 mm in diameter that is approximately 30 m long with a 0.25 µm film thickness. The column shall be kept at a constant flow of 1.6 mL/min.

Flame Ionization Detector – The detector temperature shall be set at 300 °C. The flow rate for hydrogen is 30.0 mL/min, for air is 400.0 mL/min, and the makeup flow of nitrogen is 25.0 mL/min.

Method Name	Injection	Split Ratio
ARSONL	Inject a 1.0 µL sample with a 6000 µL/min plunger speed, three sample washes, and three sample pumps.	50:1
ARSON	Inject a 5.0 µL sample with a 6000 µL/min plunger speed, no sample washes, and three sample pumps.	50:1
FIREL	Inject a 1.0 µL sample with a 6000 µL/min plunger speed, no sample washes, and three sample pumps.	75:1
FIRE	Inject a 1.0 µL sample with a 6000 µL/min plunger speed, no sample washes, and three sample pumps.	20:1