

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

4.2 Materials

- Auto sampler vials, 100 µL inserts, and crimp seals
- Vial crimper
- Vial de-crimper
- 10 micro-liter autosampler syringe
- A non-polar capillary column with a (5 %-Phenyl)-methylpolysiloxane stationary phase such as a DB-5 or HP-5
- 50 % Residual Gasoline reference material
- 100 % #2 fuel oil/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 Calibration

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.

- Petroleum ether blank.

- Carbon disulfide adsorption elution blank.
- 50 % residual gasoline reference material.
- 100 % # 2 fuel oil/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 case work.

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 **5.3 and 5.4**.

5.2.3 Once data has been collected, the resulting data file shall be loaded in the Data Analysis program. 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 Agilent 6890 Gas Chromatograph (Raleigh Laboratory) – Program parameters

5.3.1 ARSONL.M

5.3.1.1 Injection – Using the autosampler with a 10 µL syringe injection, perform eight solvent washes from each wash bottle. Inject a 1.0 µL sample with a fast plunger speed, three sample washes, and three sample pumps.

5.3.1.2 Inlet – Run in split mode at 240 °C with a 50:1 split ratio.

5.3.1.3 Carrier gas – Hydrogen.

5.3.1.4 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.

5.3.1.5 Column – Use a HP-5MS or DB-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.

5.3.1.6 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.

5.3.2 ARSON.M

5.3.2.1 Injection – Using the autosampler with a 10 µL syringe injection, perform eight solvent washes from each wash bottle. Inject a 5.0 µL sample with a fast plunger speed, no sample washes, and three sample pumps.

5.3.2.2 Inlet – Run in split mode at 240 °C with a 50:1 split ratio.

5.3.2.3 Carrier gas – Hydrogen.

5.3.2.4 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.

5.3.2.5 Column – Use a HP-5MS or DB-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.

5.3.2.6 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.

5.4 Agilent 7890 Gas Chromatograph (Raleigh Laboratory) – Program Parameters

5.4.1 ARSONL.M

5.4.1.1 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 a 1.0 µL sample with a 6000 µL/min plunger speed, three sample washes, and three sample pumps.

5.4.1.2 Inlet – Run in split mode at 240 °C with a 50:1 split ratio.

5.4.1.3 Carrier gas – Hydrogen.

5.4.1.4 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.

5.4.1.5 Column – Use a HP-5 or DB-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.

5.4.1.6 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.

5.4.2 ARSON.M

5.4.2.1 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 a 5.0 µL sample with a 6000 µL/min plunger speed, no sample washes, and three sample pumps.

5.4.2.2 Inlet – Run in split mode at 240 °C with a 50:1 split ratio.

5.4.2.3 Carrier gas – Hydrogen.

5.4.2.4 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.

5.4.2.5 Column – Use a HP-5 or DB-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.

5.4.2.6 Flame Ionization Detector – The detector temperature is 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.

5.5 Agilent 6890 Gas Chromatograph (Western Regional Laboratory) – Program parameters

5.5.1 ARSONL.M

5.5.1.1 Injection – Using the autosampler with a 10 µL syringe injection, perform eight solvent washes from each wash bottle. Inject a 1.0 µL sample with a fast plunger speed, three sample washes, and three sample pumps.

5.5.1.2 Inlet – Run in split mode at 240 °C with a 50:1 split ratio.

5.5.1.3 Carrier gas – Hydrogen.

5.5.1.4 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.

5.5.1.5 Column – Use a (5 %-Phenyl)-methylpolysiloxane (such as HP-5MS or DB-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.

- 5.5.1.6** 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 helium is 25.0 mL/min.

5.5.2 ARSON.M

- 5.5.2.1** Injection – Using the autosampler with a 10 μL syringe injection, perform eight solvent washes from each wash bottle. Inject a 5.0 μL sample with a fast plunger speed, no sample washes, and three sample pumps.
- 5.5.2.2** Inlet – Run in split mode at 240 °C with a 50:1 split ratio.
- 5.5.2.3** Carrier gas – Hydrogen.
- 5.5.2.4** 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.
- 5.5.2.5** Column – Use a (5 %-Phenyl)-methylpolysiloxane (such as HP-5MS or DB-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.
- 5.5.2.6** 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 helium is 25.0 mL/min.

5.6 Standards and Controls

- 5.6.1** There shall be at least one solvent blank run for each case.
- 5.6.1.1** If the sample being analyzed is a neat sample, petroleum ether or carbon disulfide shall be used for the blank.
- 5.6.1.2** If an extraction is performed using acetone, chloroform, carbon disulfide, petroleum ether, etc., the same solvent shall be used for the blank.
- 5.6.2** The resulting data from the blanks shall not have peaks that interfere with the analysis.
- 5.6.3** The wash bottles on the autosampler shall be filled with acetone.

5.7 Maintenance

- 5.7.1** The septa for the injection port shall be changed at least monthly.
- 5.7.2** The injection port liner shall be changed at least yearly.

5.8 Sampling and Sample Selection – N/A

5.9 Calculations – N/A

5.10 Uncertainty of Measurement – N/A

6.0 Limitations – N/A

7.0 Safety

7.1 Carbon disulfide may be toxic. Consult Material Safety Data Sheets for information on safe use.

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
- Quality Control Check Chromatographs
- Request for Instrumental Examination of Evidence

10.0 Attachments – N/A

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.