# 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 or carbon disulfide.

**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** Refer to Appendix C for chemical hygiene and safety precautions for extremely hazardous and particularly hazardous substances.

**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.

**7.5** Use extreme caution when dismantling, installing, or transporting compressed gas cylinders. Cylinders shall only be moved with the cylinder cap securely in place.

**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
- Appendix C – Chemical Hygiene and Safety Precautions for High Risk and Particularly Hazardous Substances

| **Revision History** | | |
|---|---|---|
| Effective Date | Version Number | Reason |
| 07/16/2021 | 9 | Added option of carbon disulfide for wash vials to 5.3.3<br>Appendix A - retired ARSON and ARSONL methods<br>7.1 – changed wording away from carbon disulfide only to all high risk or particularly hazardous substances<br>7.5 – new<br>10 – added appendix C<br>Appendix B – retired ARSON and ARSONL methods, added FIREFIDL and FIREFID method names, updated plunger speed, updated pre-inject and post-inject solvent washes<br>Appendix C - new |

**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 |
|---|---|---|
| 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 one pre-inject and five 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

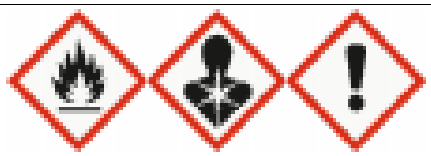
Oven – Run a temperature program starting at 80.0 °C for 0.00 minutes. Then ramp at 20.0 °C per minute until 280 °C is obtained. Hold at 280 °C for 2.00 minutes.

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 |
|---|---|---|
| FIREL / FIREFIDL | Inject a 1.0 µL sample with a fast plunger speed, no sample washes, and three sample pumps. | 75:1 |
| FIRE / FIREFID | Inject a 1.0 µL sample with a fast plunger speed, no sample washes, and three sample pumps. | 20:1 |

## Appendix C – Chemical Hygiene and Safety Precautions for Extremely Hazardous and Particularly Hazardous Substances

| **Gasoline**<br>**DANGER: PARTICULARLY HAZARDOUS SUBSTANCE*** | |
|---|---|
|  | **HEALTH 2**<br>**FLAMMABILITY 3**<br>**REACTIVITY 0** |
| **Detection of Release** | Translucent, straw-colored or light yellow liquid. Strong, characteristic hydrocarbon odor. Sweet-ether like. |
| **Signs/Symptoms of Exposure** | • Skin irritation with prolonged or repeated contact.<br>• Eye contact with liquid or vapor may cause irritation.<br>• Ingestion may cause gastrointestinal disturbances, including nausea, vomiting, and diarrhea, and CNS effects similar to alcohol intoxication. In severe cases, tremors, convulsions, loss of consciousness, coma, respiratory arrest, and death may occur.<br>• Inhalation may cause irritation to the nose, throat, lungs, and respiratory tract. CNS effects may include headache, dizziness, loss of balance and coordination, unconsciousness, coma, respiratory failure, and death. |
| **PEL (8-hr TWA)** | • Gasoline: 300ppm (ACGIH)<br>• Toluene: 20 ppm (ACGIH), 200 ppm (OSHA); 100ppm (NIOSH)<br>• Butane: 1000 ppm (ACGIH), 800 ppm (OSHA, NIOSH)<br>• Xylenes: 100 ppm (ACGIH, OSHA)<br>• Benzene, 1,2,4 trimethyl: 25ppm (NIOSH)<br>• Ethyl alcohol: 1000 ppm (ACGIH, OSHA, NIOSH)<br>• Ethylbenzene: 20 ppm (ACGIH), 100 ppm (OSHA, NIOSH)<br>• Benzene: 0.5 ppm (ACGIH), 1ppm (OSHA), 0.1 ppm (NIOSH)<br>• Hexane: 50 ppm (ACGIH, NIOSH), 500 ppm (OSHA) |
| **Associated Hazards** | Highly flammable liquid and vapor. May cause genetic defects. May cause cancer. May damage fertility or the unborn child. May cause respiratory irritation. May cause drowsiness or dizziness. May be fatal if swallowed and enters airways. Harmful to aquatic life. |
| **Controls** | Use adequate ventilation to keep vapor concentrations below exposure and flammability limits. PPE: Gloves – nitrile, neoprene, or PVC are recommended; safety glasses/goggles |
| **Safe handling, storage, disposal** | DO NOT SIPHON BY MOUTH. Handle as a flammable liquid. Keep away from heat, sparks, excessive temperature, and open flame! Bond and ground containers during product transfer to reduce the possibility of static-initiated fire or explosion. Use approved vented containers. Keep containers closed and clearly labeled. Empty product containers or vessels may contain explosive vapors. Store in a well-ventilated area. Keep cool. |
| **Emergency Procedures** | **Eye Contact:** Immediately flush with clean, low-pressure water for at least 15 min. Hold eyelids open to ensure adequate flushing. Seek medical attention.<br>**Inhalation Exposure:** Remove person to fresh air. If person is not breathing, provide artificial respiration. If necessary, provide additional oxygen once breathing is restored if trained to do so. Seek medical attention immediately.<br>**Ingestion:** DO NOT INDUCE VOMITING. Do not give liquids. Obtain immediate medical attention. If spontaneous vomiting occurs, lean victim forward to reduce the risk of aspiration. Monitor for breathing difficulties. Small amounts of material which enter the mouth should be rinsed out until the taste is dissipated.<br>**Skin Contact:** Remove contaminated clothing. Wash contaminated areas thoroughly with soap and water or with waterless hand cleanser. Obtain medical attention if irritation or redness develops. |

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|---|---|
| | **Spills:**<br>Small spills: Carefully contain and stop the source of the spill, if safe to do so. Take up with sand or other oil-absorbing materials. Carefully shovel, scoop, or sweep up into a waste container for disposal. Caution, flammable vapors may accumulate in closed containers.<br>Large spills: Evacuate non-essential personnel and remove or secure all ignition sources. Call 911 with hazmat request. |