

Technical Procedure for Gas Chromatography–Mass Spectrometry (GC-MS)

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

2.0 Scope – This procedure applies to all gas chromatograph-mass spectrometers 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 Agilent 5973 Mass Selective Detector
- Agilent 6890 Gas Chromatograph with Agilent 5975 Mass Selective Detector

4.2 Materials

- Auto sampler vials, 100 µL inserts, and crimp seals
- Vial crimper
- Vial de-crimper
- 2.5 mL airtight syringe or 3 mL disposable hypodermic syringe
- 10 micro-liter autosampler syringe
- A non-polar capillary column with a (5 %-Phenyl)-methylpolysiloxane stationary phase such as a DB-5MS or HP-5MS
- Perfluorotributylamine
- 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
- Chloroform
- Helium – UHP

5.0 Procedure

5.1 Start-up and Performance Check

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

5.1.2 Daily GC-MS Performance Check using the Autotune program

5.1.2.1 Each day that the instrument is used, the performance of the MS shall be checked. The performance check only needs to be performed once per day. If the instrument is not being used, the daily Autotune shall not be required.

5.1.2.2 This procedure uses Perfluorotributylamine (PFTBA) as a tuning standard and the resulting tune report shall be electronically archived.

5.1.2.3 The parameters for passing an Autotune are as follows:

5.1.2.3.1 The three tuning masses in the upper profile part of the report shall be within +/- 0.2 amu of 69.00, 219.00, and 502.00.

5.1.2.3.2 The peak widths of these three peaks shall be 0.60 +/- 0.1 amu.

5.1.2.3.3 The peak at 69 amu shall be set to 100 % relative abundance

5.1.2.3.3.1 Relative to the peak at 69 amu, the peak at 219 amu shall be in the range of 70-250 %.

5.1.2.3.3.2 Relative to the peak at 69 amu, the peak at 502 amu shall be greater than 3 %.

5.1.2.3.3.3 Relative to the peak at 69 amu, the peak at 70 amu shall be in the range of 0.5-1.6 %.

5.1.2.3.4 Relative to the peak at 219 amu, the peak at 220 amu shall be in the range of 3.2-5.4 %.

5.1.2.3.5 Relative to the peak at 502 amu, the peak at 503 amu shall be in the range of 7.9-12.3 %.

5.1.2.3.6 M/z 28 greater than m/z 18 may indicate an air leak somewhere in the system.

5.1.2.3.6.1 If an air leak is detected, the air leak shall be isolated and corrected.

5.1.2.3.6.2 The instrument shall be tuned again.

5.1.2.4 If any of the above stated parameters are out of specification, the instrument shall be tuned again. If the problem persists, a service engineer shall be contacted or maintenance performed. Once the tune parameters are within specifications, the instrument may be used for casework.

5.1.3 Monthly GC-MS Performance Check

5.1.3.1 To ensure performance, the following samples shall be analyzed and the Total Ion Chromatograms (TICs) electronically archived.

- Petroleum ether blank.
- Carbon disulfide adsorption elution blank.
- 50 % residual gasoline reference material.
- 100 % # 2 fuel oil/Diesel fuel reference material.

- 5.1.3.2 If there is a shift in peak retention time by 0.3 minute or more or a change in peak resolution, maintenance shall be performed or a service engineer shall be called. Once maintenance has been performed, the samples shall be re-examined. If the resulting chromatograms are within the limits, the instrument may be used for casework.

5.1.4 Performance Verification for New Instrument Set Up

- 5.1.4.1 New GC-MS instruments shall be installed by a certified engineer according to manufacturer guidelines.
- 5.1.4.2 The Autotune shall be run on the instrument and checked according to the above criteria.
- 5.1.4.3 The samples used in the monthly GC-MS performance check shall be analyzed on the instrument and the resulting chromatographs are 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-MS programs located in 5.3.
- 5.2.3 Once data has been collected the resulting data file shall be loaded in the Data Analysis program for examination. At a minimum, the Total Ion chromatograph and Library Search Report shall be included in the case record. Additional items such as Extracted Ion Chromatographs and Library searches shall be included if performed.
- 5.2.4 After the method has run, allow the instrument to return to the set program and leave the GC-MS on.

5.3 Agilent 6890 Gas Chromatograph and Agilent 5973 Mass Selective Detector - Program Parameters

5.3.1 ALCOHOL.M

- 5.3.1.1 Injection – Manual with either a 2.5 mL airtight syringe or 3mL hypodermic syringe
- 5.3.1.2 Inlet – Run in split mode at 200 °C with a 20:1 split ratio.
- 5.3.1.3 Carrier gas – Helium.

- 5.3.1.4 Oven – Run at an isothermal temperature program set at 80 °C for 15.00 minutes.
- 5.3.1.5 Column – Use a (5 %-Phenyl)-methylpolysiloxane (such as 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 0.6 mL/min.
- 5.3.1.6 Mass spectrometer – The transfer line temperature shall be set at 300 °C. The MS Quad temperature is set at 150 °C and the MS source is set at 230 °C. The MS shall be run in scan mode using the settings from the atune.u file with an electron multiplier offset of 200 eV. The mass scan range is 20.0 amu to 400.0 amu with a threshold abundance of 150.

5.3.2 ARSONL.M

- 5.3.2.1 Injection – Using the autosampler with a 10 µL syringe injection, perform three solvent washes from each wash bottle. Inject a 0.2 µL sample with a fast plunger speed, no sample washes, and 3 sample pumps.
- 5.3.2.2 Inlet – Run in split mode at 200 °C with a 100:1 split ratio.
- 5.3.2.3 Carrier gas – Helium.
- 5.3.2.4 Oven – Run a temperature program starting at 50.0 °C for 2.50 minutes. Then ramp at 10.0 °C per minute until 300 °C is obtained. Hold at 300 °C for 5.00 minutes.
- 5.3.2.5 Column – Use a (5 %-Phenyl)-methylpolysiloxane (such as 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 0.6 mL/min.
- 5.3.2.6 Mass spectrometer – The transfer line temperature shall be set at 300 °C. The MS Quad temperature is set at 150 °C and the MS source is set at 230 °C. The MS shall be run in scan mode using the settings from the atune.u file with an electron multiplier offset of 200 eV. The mass scan range is 20.0 amu to 400.0 amu with a threshold abundance of 150. The mass spectrometer shall have a 2.50 minute solvent delay.

5.3.3 ARSONLNSD.M

- 5.3.3.1 Injection – Using the autosampler with a 10 µL syringe injection, perform three solvent washes from each wash bottle. Inject a 0.2 µL sample with a fast plunger speed, no sample washes, and 3 sample pumps.
- 5.3.3.2 Inlet – Run in split mode at 200 °C with a 100:1 split ratio.
- 5.3.3.3 Carrier gas – Helium.

- 5.3.3.4 Oven – Run a temperature program starting at 50.0 °C for 2.50 minutes. Then ramp at 10.0 °C per minute until 300 °C is obtained. Hold at 300 °C for 5.00 minutes.
- 5.3.3.5 Column – Use a (5 %-Phenyl)-methylpolysiloxane (such as 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 0.6 mL/min.
- 5.3.3.6 Mass spectrometer – The transfer line temperature shall be set at 300 °C. The MS Quad temperature is set at 150 °C and the MS source is set at 230 °C. The MS shall be run in scan mode using the settings from the atune.u file with an electron multiplier offset of 200 eV. The mass scan range shall be 20.0 amu to 400.0 amu with a threshold abundance of 150.

5.3.4 ARSON100S.M

- 5.3.4.1 Injection – Using the autosampler with a 10 µL syringe injection, perform three solvent washes from each wash bottle. Inject a 1.0 µL sample with a fast plunger speed, no sample washes, and 3 sample pumps.
- 5.3.4.2 Inlet – Run in split mode at 200 °C with a 100:1 split ratio.
- 5.3.4.3 Carrier gas – Helium.
- 5.3.4.4 Oven – Run a temperature program starting at 50.0 °C for 2.50 minutes. Then ramp at 10.0 °C per minute until 300 °C is obtained. Hold at 300 °C for 5.00 minutes.
- 5.3.4.5 Column – Use a (5 %-Phenyl)-methylpolysiloxane (such as 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 0.6 mL/min.
- 5.3.4.6 Mass spectrometer – The transfer line temperature shall be set at 300 °C. The MS Quad temperature is set at 150 °C and the MS source is set at 230 °C. The MS shall be run in scan mode using the settings from the atune.u file with an electron multiplier offset of 200 eV. The mass scan range shall be 20.0 amu to 400.0 amu with a threshold abundance of 150. The mass spectrometer shall have a 2.50 minute solvent delay.

5.3.5 ARSON100SNSD.M

- 5.3.5.1 Injection – Using the autosampler with a 10 µL syringe injection, perform three solvent washes from each wash bottle. Inject a 1.0 µL sample with a fast plunger speed, no sample washes, and 3 sample pumps.
- 5.3.5.2 Inlet – Run in split mode at 200 °C with a 100:1 split ratio.

5.3.5.3 Carrier gas – Helium.

5.3.5.4 Oven – Run a temperature program starting at 50.0 °C for 2.50 minutes. Ramp at 10.0 °C per minute until 300 °C is obtained. Hold at 300 °C for 5.00 minutes.

5.3.5.5 Column – Use a (5 %-Phenyl)-methylpolysiloxane (such as 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 0.6 mL/min.

5.3.5.6 Mass spectrometer – The transfer line temperature shall be set at 300 °C. The MS Quad temperature is set at 150 °C and the MS source is set at 230 °C. The MS shall be run in scan mode using the settings from the atune.u file with an electron multiplier offset of 200 eV. The mass scan range shall be 20.0 amu to 400.0 amu with a threshold abundance of 150.

5.3.6 ARSON20S.M

5.3.6.1 Injection – Using the autosampler with a 10 µL syringe injection, perform three solvent washes from each wash bottle. Inject a 1.0 µL sample with a fast plunger speed, no sample washes, and 3 sample pumps.

5.3.6.2 Inlet – Run in split mode at 200 °C with a 20:1 split ratio.

5.3.6.3 Carrier gas – Helium.

5.3.6.4 Oven – Run a temperature program starting at 50.0°C for 2.50 minutes. Then ramp at 10.0 °C per minute until 300 °C is obtained. Hold at 300 °C for 5.00 minutes.

5.3.6.5 Column – Use a (5 %-Phenyl)-methylpolysiloxane (such as 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 0.6 mL/min.

5.3.6.6 Mass spectrometer – The transfer line temperature shall be set at 300 °C. The MS Quad temperature is set at 150 °C and the MS source is set at 230 °C. The MS shall be run in scan mode using the settings from the atune.u file with an electron multiplier offset of 200 eV. The mass scan range shall be 20.0 amu to 400.0 amu with a threshold abundance of 150. The mass spectrometer shall have a 2.50 minute solvent delay.

5.3.7 ARSON20SNSD.M

5.3.7.1 Injection – Using the autosampler with a 10 µL syringe injection, perform three solvent washes from each wash bottle. Inject a 1.0 µL sample with a fast plunger speed, no sample washes, and 3 sample pumps.

5.3.7.2 Inlet – Run in split mode at 200 °C with a 20:1 split ratio.

5.3.7.3 Carrier gas – Helium.

5.3.7.4 Oven – Run a temperature program starting at 50.0 °C for 2.50 minutes. Then ramp at 10.0 °C per minute until 300 °C is obtained. Hold at 300 °C for 5.00 minutes.

5.3.7.5 Column – Use a (5 %-Phenyl)-methylpolysiloxane (such as 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 0.6 mL/min.

5.3.7.6 Mass spectrometer – The transfer line temperature shall be set at 300 °C. The MS Quad temperature is set at 150 °C and the MS source is set at 230 °C. The MS shall be run in scan mode using the settings from the atune.u file with an electron multiplier offset of 200 eV. The mass scan range shall be 20.0 amu to 400.0 amu with a threshold abundance of 150.

5.3.8 ARSON2UL100S.M

5.3.8.1 Injection – Using the autosampler with a 10 µL syringe injection, perform three solvent washes from each wash bottle. Inject a 2.0 µL sample with a fast plunger speed, no sample washes, and 3 sample pumps.

5.3.8.2 Inlet – Run in split mode at 200 °C with a 100:1 split ratio.

5.3.8.3 Carrier gas – Helium.

5.3.8.4 Oven – Run a temperature program starting at 50.0 °C for 2.50 minutes. Then ramp at 10.0 °C per minute until 300 °C is obtained. Hold at 300 °C for 5.00 minutes.

5.3.8.5 Column – Use a (5 %-Phenyl)-methylpolysiloxane (such as 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 0.6 mL/min.

5.3.8.6 Mass spectrometer – The transfer line temperature shall be set at 300 °C. The MS Quad temperature is set at 150 °C and the MS source is set at 230 °C. The MS shall be run in scan mode using the settings from the atune.u file with an electron multiplier offset of 200 eV. The mass scan range shall be 20.0 amu to 400.0 amu with a threshold abundance of 150. The mass spectrometer shall have a 2.50 minute solvent delay.

5.3.9 ARSON2UL20S.M

5.3.9.1 Injection – Using the autosampler with a 10 µL syringe injection, perform three solvent washes from each wash bottle. Inject a 2.0 µL sample with a fast plunger speed, no sample washes, and 3 sample pumps.

- 5.3.9.2 Inlet – Run in split mode at 200 °C with a 20:1 split ratio.
- 5.3.9.3 Carrier gas – Helium.
- 5.3.9.4 Oven – Run a temperature program starting at 50.0 °C for 2.50 minutes. Then ramp at 10.0 °C per minute until 300 °C is obtained. Hold at 300 °C for 5.00 minutes.
- 5.3.9.5 Column – Use a (5 %-Phenyl)-methylpolysiloxane (such as 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 0.6 mL/min.
- 5.3.9.6 Mass spectrometer – The transfer line temperature shall be set at 300 °C. The MS Quad temperature is set at 150 °C and the MS source is set at 230 °C. The MS shall be run in scan mode using the settings from the atune.u file with an electron multiplier offset of 200 eV. The mass scan range shall be 20.0 amu to 400.0 amu with a threshold abundance of 150. The mass spectrometer shall have a 2.50 minute solvent delay.

5.4 Standards and Controls

- 5.4.1 If the sample was not previously analyzed on the GC-FID, there shall be a minimum of one blank solvent run for each case.
 - 5.4.1.1 If the sample being analyzed is a neat sample, petroleum ether or carbon disulfide shall be used for the blank.
 - 5.4.1.2 If an extraction is performed using acetone, chloroform, carbon disulfide, petroleum ether, etc., the same solvent shall be used for the blank.
 - 5.4.1.3 If the extraction being performed is a heated headspace extraction, an air blank shall be analyzed.
- 5.4.2 The resulting data from the blanks shall not have peaks that interfere with the analysis.
- 5.4.3 The wash bottles on the autosampler shall be filled with acetone unless it is thought that acetone or another light solvent is present. In that case, the wash bottles shall be changed and carbon disulfide used as the wash solvent.

5.5 Maintenance

- 5.5.1 The septum for the injection port shall be changed at least monthly.
- 5.5.2 The injection port liner shall be changed at least yearly.
- 5.5.3 The mechanical pump oil shall be changed twice a year.
- 5.5.4 The MS source shall be cleaned as needed based on tune acceptance criteria.

5.6 Sampling – N/A

5.7 Calculations – N/A

5.8 Uncertainty of Measurement – N/A

6.0 Limitations - Mass Spectrometry alone cannot differentiate isomers.

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 3: Forensic Applications of Mass Spectrometry, p. 117.

Agilent GC-MSD Chemstation and Instrument Operation Volume 1 G1701DA Version D.00.00 Student Manual. Printed March 2002, Agilent Technologies.

ASTM Guidelines

ASTM Standard E1618, 2006, “Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography–Mass Spectrometry.” ASTM International, West Conshohocken, PA, 2006, www.astm.org.

9.0 Records

- Maintenance log
- Use log
- Quality Control Check Chromatographs
- Autotune reports
- 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. Agilent 6890 Gas Chromatograph with Agilent 5975 Mass Selective Detector was added to equipment. All column references were updated to reflect a (5 %-Phenyl)-methylpolysiloxane (such as a HP-5MS or DB-5MS) column.
09/30/2013	3	5.3.1.6, 5.3.2.6, 5.3.3.6, 5.3.4.6, 5.3.5.6, 5.3.6.6, 5.3.7.6, 5.3.8.6 and 5.3.9.6 units regarding threshold were updated. Quality Control Check log was removed from 9.0; Quality Control Check Chromatograms are stored electronically.