Deviation Request Form (DRF)

Directions: The Initiator will complete Sections A through C. Additional continuation pages can be included if necessary.

Initiator	Kristen Crawford	**Date**	3/3/2021

A. Requested deviation applies to (Technical Procedure – include specific section):

Technical Procedure for Gas Chromatography-Mass Spectrometry (Version 9, Effective date: 11/22/2019) section 5.3 and Appendix B.

B. Requested deviation:

5.3.3: Reword to state that the wash bottles shall be filled with carbon disulfide unless acetone was used as the extraction solvent.

Appendix B: Change three pre-inject solvent washes to one and change eight post-inject solvent washes to five, update column flow from 2.0 to 1.2 mL/min. Remove note in the Mass Spectrometer column of the table about solvent delay and update with "The MS turns off during the solvent peak. The timeframe that the MS is off depends on the individual instrument's retention time for the start and end of the solvent peak".

C. Necessity for the deviation:

New methods were validated for the new 7890/5977 instruments. The new methods begin data collection immediately, shut off during the solvent peak and then collect data again until the end of the run. Once the new methods were validated, the ignitable liquid library was run under those conditions for both 7890/5977 instruments. No changes were made to the methods for the 6890/5973 instrument (Appendix A). Carbon disulfide is the extraction solvent used in the majority of fire debris cases. Changing the default wash vial solvent to carbon disulfide keeps the solvent peak uniform from the extraction process to the instrumental analysis allowing the new methods to successfully "skip" the solvent peak.

D. Technical review and Authorization (to be completed by the Quality Manager and/or Technical Leader)
Comments(to include merits and impacts):

This deviation will allow the two new GC-MS instruments to analyze fire debris casework using the new method that skips the solvent peak.

Approved	☑	Yes	☐	No	Duration	one year
Signature	Kristen Crawford Digitally signed by Kristen Crawford DN: C=US, O=NC State Crime Laboratory, CN=Kristen Crawford, E=krawford@ncdoj.gov Reason: I have reviewed this document Location: Date: 2021-03-16 13:06:21 Foxit PhantomPDF Version: 9.3.0				Date	3/16/2021

E. Quality Assurance Authorization (to be completed by the Quality Manager, Forensic Scientist Manager or designee)

Acceptable within general QA guidelines and good laboratory practice?	☑	Yes	☐	No
Significant negative impact to Crime Laboratory Quality System?	☐	Yes	☑	No

Restrictions/limitations:

This DRF is for the use of the Trace Evidence Section Technical Procedure for Gas-Chromatography-Mass Spectrometry (GC-MS) (Version 9, Effective date: 11/22/2019).

☑	Authorized	☐	Rejected	Signature	Amy Brewer-Slish Digitally signed by Amy Brewer-Slish DN: C=US, OU=Trace Evidence Section, O=NC State Crime Laboratory, CN=Amy Brewer-Slish, E=abrewerslish@ncdoj.gov Reason: I am the author of this document Location: Date: 2021-03-16 14:01:40 Foxit PhantomPDF Version: 9.1.0	Date	03/16/2021

Form approved for use by: Timothy G. Suggs
Digitally signed by Timothy G. Suggs
DN: cn=Timothy G. Suggs, o=North Carolina State Crime Laboratory, ou=Quality Manager, email=tsuggs@ncdoj.gov, c=US
Date: 2014.12.19 09:01:52 -05'00'

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 7890 Gas Chromatograph with Agilent 5977 Mass Selective Detector
- Hydrogen Generator

4.2 Materials

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

4.3 Reagents

- Acetone
- Acetone – Reagent A.C.S. grade
- Carbon disulfide - Reagent A.C.S. grade
- Petroleum ether – Optima Grade
- 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 greater than 40 %.

5.1.2.3.3.2 Relative to the peak at 69 amu, the peak at 502 amu shall be greater than 2.4 %.

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 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

5.1.3.2 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 **5.1.2**.

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 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-MS 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. 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. When a sample is examined by GC-MS only, the Library Search Report is not required for the solvent blank, control blank (charcoal strip) or Performance Check Standard (100% diesel fuel standard).

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 Standards and Controls

5.3.1 If the sample was not previously analyzed on the GC-FID, there shall be a minimum of one blank solvent run for each case.

5.3.1.1 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.

5.3.1.3 If the extraction being performed is a heated headspace extraction, an air blank shall be analyzed.

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 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.

5.4 Maintenance

5.4.1 The septum 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.

5.4.3 The mechanical pump oil shall be changed twice a year.

5.4.4 The MS source shall be cleaned as needed based on tune acceptance criteria.

5.5 Sampling – N/A

5.6 Calculations – N/A

5.7 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 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 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
- Performance Check Chromatographs
- Autotune reports
- Request for Instrumental Examination of Evidence

10.0 Attachments

- Appendix A – Instrument Program Parameters for Agilent 6890 Gas Chromatograph and Agilent 5973 Mass Selective Detector
- Appendix B – Instrument Program Parameters for Agilent 7890 Gas Chromatograph and Agilent 5977 Mass Selective 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. 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.
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 5.1.3.1 Added "when in use" to 5.5.1
12/11/2015	7	4.2 – reworded for consistency between GC-FID and GC-MS procedures, changed 100 to 150 Added reagent grade acetone to 4.3 Removed extra periods and "#2 fuel oil" in 5.1.3.1 Referenced 5.1.2 in 5.1.4.2
09/22/2017	8	Updated methods in Section 5.3: Oven (5.3.2.4 through 5.3.9.4) - temperature ramp - 9.0 degrees C/min Column (5.3.1.5 through 5.3.9.5) - flow rate - 0.7 mL/min Mass spectrometer (5.3.1.6 through 5.3.9.6) - electron multiplier offset - 0 (zero) eV
11/22/2019	9	Changed name of section in header and 2.0 Removed Western Laboratory instrument (6890/5975) from 4.1 Added new instrument (7890/5977) and hydrogen generator to 4.1 Removed suggested columns and hypodermic syringe from 4.2 Removed "chloroform" from 4.3 5.1.2.3.3 – updated acceptance criteria for 219 and 502 Changed reference in 5.2.2 to Attachments Clarified requirements for Library Search Reports in 5.2.3 Relocated and updated GC/MS Program Parameters for 6890/5973 in 5.3 to Appendix A, removed DB-5MS column option Removed "chloroform" from 5.4.1.2 Added GC/MS Program Parameters for 7890/5977 to Appendix B Removed "Material" from 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 and Agilent 5973 Mass Selective Detector

Injection – Using the autosampler with a 10 µL syringe injection, perform three pre-injection and three post-injection solvent washes from each wash bottle. Inject the volume of sample indicated below with a fast plunger speed, no sample washes, and 3 sample pumps. The ALCOHOL method uses a manual injection.

Inlet – Run in split mode at 200 °C with the split ratio indicated below.

Carrier Gas – Helium

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 0.7 mL/min.

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 0 eV. The mass scan range is 20.0 amu to 400.0 amu with a threshold abundance of 150. Include a solvent delay as indicated below.

Method Name	Injection Volume	Split Ratio	Oven	Mass Spectrometer
ARSONL	0.2 µL	100:1	Run a temperature program starting at 50.0 °C for 2.50 minutes. Then ramp at 9.0 °C per minute until 300 °C is obtained. Hold at 300 °C for 5.00 minutes.	The mass spectrometer shall have a 2.50 minute solvent delay.
ARSON100S	1.0 µL	100:1		
ARSON20S	1.0 µL	20:1		
ARSON2UL20S	2.0 µL	20:1		
ARSONLNSD	0.2 µL	100:1		There is no solvent delay.
ARSON100SNSD	1.0 µL	100:1		
ARSON20SNSD	1.0 µL	20:1		
ALCOHOL	Manual injection of 1 mL using a 2.5 mL airtight syringe.	20:1	Run at an isothermal temperature program set at 80 °C for 15.00 minutes.	There is no solvent delay.

Appendix B

Instrument Program Parameters

Agilent 7890 Gas Chromatograph and Agilent 5977 Mass Selective Detector

Injection – Using the autosampler with a 10 µL syringe injection, perform three pre-injection and eight post-injection solvent washes from each wash bottle. Inject the volume of sample indicated below with a fast plunger speed, no sample washes, and 3 sample pumps.

Inlet – Run in split mode at 200 °C with the split ratio indicated below.

Carrier Gas – Hydrogen

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 2.0 mL/min.

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 0 eV. The mass scan range is 20.0 amu to 400.0 amu with a threshold abundance of 150. Include a solvent delay as indicated below.

Method Name	Injection Volume	Split Ratio	Oven	Mass Spectrometer
FIREL	0.2 µL	100:1	Run a temperature program starting at 50.0 °C for 1.50 minutes. Then ramp at 20.0 °C per minute until 300 °C is obtained. Hold at 300 °C for 3.00 minutes.	The mass spectrometer shall have a 0.95 minute solvent delay.
FIRE100S	1.0 µL	100:1		
FIRE20S	1.0 µL	20:1		
FIRE2UL20S	2.0 µL	20:1		
FIRELNSD	0.2 µL	100:1		There is no solvent delay.
FIRE100SNSD	1.0 µL	100:1		
FIRE20SNSD	1.0 µL	20:1		
FIRE2UL20SNSD	2.0 µL	20:1		