Performance Monitoring Protocol (QA/QC) for the Agilent GC/MS

1 Scope

This document addresses the performance monitoring (QA/QC) of the Agilent Gas Chromatograph/ Mass Spectrometer (GC/MS) system with a Mass Selective Detector (MSD). This document applies to personnel using the associated instrument(s)/equipment in the following discipline/category of testing: Explosives (chemistry) examinations performed at the Huntsville facility.

2 Principle

The Agilent GC/MS system consists of an Agilent Gas Chromatograph (GC) with a single quadrupole Mass Selective Detector (MSD) Mass Spectrometer (MS). Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

The mass spectrometer will be configured to perform specific modes of ionization depending on which type of ion source is installed. If the electron impact (EI) ionization source is installed, only positive ion EI ionization analysis may be performed.

3 Equipment/Materials/Reagents

a. Instrumentation - Agilent 7890 GC, 5975 or 5977 MSD with EI, and MSD Chemstation software (or equivalent)

b. Autosampler - Agilent ALS automated sampler, accessories, and software (or equivalent)

c. GC Column (MSD) – Agilent DB-5 MS, 30 m, 0.25 mm i.d., 0.25 μm film (or equivalent)

d. Carrier Gas - Helium, 99.99% (high purity or equivalent)

e. Hydrogen Gas, 99.99% (high purity or equivalent)

f. Chloroform (GC grade)

g. Methanol (Optima grade)

h. Lidocaine HCl (Sigma or equivalent)

i. Tributoxyethyl Phosphate (TBEP) (Chem Service or equivalent)

j. Perfluorotributylamine (PFTBA, FC-43) (Agilent or equivalent)

k. Analytical balance

l. Volumetric flask

m. Autosampler vials - 2 mL GC vials, crimp or screw top, with or without 100-500 µL inserts (Thermo or equivalent)

n. Injection port liners - 4 mm split/splitless, tapered, with or without glass wool (Agilent or equivalent)

o. Injection port septa - standard low-bleed 11 mm (Agilent or equivalent)

p. Autosampler syringes –10 µL syringe (Agilent or equivalent)

q. Wash vials – 4 mL screw top without insert (Agilent or equivalent)

4 Standards and Controls

4.1 PFTBA Tuning Solution

The PFTBA tuning solution is used for tuning the mass spectrometer and verifying mass assignment and accuracy when the EI source is installed. It is supplied by the instrument manufacturer and does not expire. It is stored in a glass container attached to the MSD. Refill as needed.

4.2 Testmix (0.05 mg/mL each of Lidocaine and TBEP)

The Testmix is used to assess daily operating performance, mass assignment, and continued integrity of the system. To prepare, weigh 5.8 mg Lidocaine HCl and 5 mg TBEP into a 100-mL volumetric flask. Bring to the mark with chloroform and mix well. Store the solution in the refrigerator. It has a shelf-life of two years. This preparation may be appropriately scaled up. Record stock solution preparations in the Reagent Log.

5 Sampling or Sample Selection

Not applicable.

6 Procedures

6.1 Daily Checks

The following steps are to be performed daily, regardless of the ion source installed, mode of ionization, or the detector to be used. Enter the appropriate information in the QA/QC log for tracking purposes.

a. Check to ensure that the GC wash vials are filled with methanol, the waste vials are empty, and all are in the appropriate positions.

b. Record the remaining disk space on the hard drive. Use Windows to verify that the hard disk has at least 100 MB of free disk space. Do not use if less than 100 MB remain.

c. Record the line pressure of the building helium supply (carrier gas). The regulator should read 50 psi or above. If it cannot maintain this pressure, contact the appropriate instrument support personnel. If the helium is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100 psi. remain.

d. Perform a tune of the instrument. If Autotune (ATUNE) is selected, the mass spectrometer will tune itself using PFTBA. Evaluate the results using the 'Decision Criteria' section of this protocol. Ensure N_2 and H_2O values do not exceed 5%. If the results are acceptable, print the tune report (ATUNE) when completed.

e. Check the Ion Gauge to ensure that there are no significant leaks in the system. Do not use if the source pressure is higher than 6×10^{-5} torr.

f. Perform an analysis of the Testmix. Open the appropriate Testmix instrument method, and verify the parameters as listed in the 'Instrumental Conditions' section of this protocol. Set up a sequence, load the autosampler with a vial containing the Testmix, and start the analysis. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the TIC and spectra for both TBEP and Lidocaine.

g. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact the appropriate instrument support personnel.

6.2 As Needed Checks

The following steps are to be performed as needed based on system performance. Indicate completion in the appropriate log.

a. Replace the septum in the GC injection port.

b. Replace the liner within the GC injection port.

c. Check the GC syringe in the autosampler. Replace if needed.

7 Instrumental Conditions

Use the instrumental conditions listed below based on the detector needed. Refer to the "General Instrument Maintenance Protocol" for procedures on minor deviations.

7.1 Gas Chromatograph/Mass Spectrometer

7.1.1 Gas Chromatograph

Oven

Initial Temp:	60°C
Initial Time:	2.0 min
Ramp:	35°C/min
Final Temp:	250°C
Hold Time:	10.0 min
Equilibration Time:	0.5 min

Inlet/Injector

Inj Vol:	1.0 µL
Mode:	Splitless
Inlet Temp:	220°C

Column

Type:	DB-5 MS
Length:	30 m
Diameter:	0.25 mm
Film Thickness:	0.25 µm
Mode:	Constant Flow
Init Flow:	1.2 mL/min
Average Lin Velocity:	40 cm/sec
Carrier Gas:	Helium

7.1.2 Mass Spectrometer

Solvent Delay:	3.0 min
Scan Mode:	Full Scan
Scan Range:	50-500 m/z

Temperatures

Transfer Line:	280°C
Source:	200°C – 230 °C
Quad:	150°C

8 Decision Criteria

8.1 Autotune

If an autotune of the mass spectrometer has been performed, verify the results below. Compare the results of the autotune to previous autotune results. Significant voltage increases or changes in the isotope ratios indicate the need to initiate corrective maintenance procedures.

The following are typical EI ion autotune values for the MSD:

a.	PFTBA Tune:	Mass ± 0.4 for m/z 69, 219, and 502
b.	Peak width:	0.45-0.65
c.	Relative abundance:	69 greater than 50% 219 greater than 50% 502 greater than 1%

8.2 Testmix

8.2.1 Gas Chromatograph

Verify the results of the Testmix.

a. In order for the instrument to be considered in good operating condition, both Lidocaine and TBEP should generate well-resolved, Gaussian- shaped peaks with baseline separation.

b. A SNR of 3:1 will be the minimum response necessary to consider a response a peak.

c. There should be no extraneous peaks in the chromatogram/TIC greater than 5% of the height of the tallest Testmix peak.

d. The retention times of Lidocaine and TBEP should not deviate by ± 3% compared to previous runs of the Testmix.

8.2.2 Mass Spectrometer

In addition to the criteria in section 8.2.1, check the following criteria when using the mass spectrometer:

a. Check for the correct mass assignments for the mass spectra for EI:

- Lidocaine ions 86 and 234
- TBEP ions 57, 199, and 299

9 Calculations

Not applicable.

10 Measurement Uncertainty

Not applicable.

11 Limitations

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

12 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis. Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

13 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (IOG 001) *Instrument Operations Group SOP Manual.*

"Gas Chromatograph General Maintenance Protocol" (IOG 002) *Instrument Operations Group SOP Manual.*

"Mass Spectrometer General Maintenance Protocol" (IOG 004) *Instrument Operations Group SOP Manual.*

FBI Laboratory Safety Manual.

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0	10/04/18	New document which specifies instrument protocol for the Huntsville facility.

Approval

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