GC/MS Performance Monitoring and Maintenance

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1 INTRODUCTION

This document addresses the performance monitoring and maintenance of the Agilent GC/MS. The Agilent GC/MS system consists of an Agilent Gas Chromatograph (GC) with a single quadrupole Mass Selective Detector (MSD) Mass Spectrometer (MS). The system may also be equipped with an additional detector, such as an FID. Definitions and guidelines are outlined in IOSS-701.

The mass spectrometer will be configured to perform specific modes of ionization depending on which of the two types of ion sources is installed. If the electron impact (EI) ionization source is installed, only positive ion EI ionization analysis will be performed. However, if the chemical impact (CI) ionization source is installed, then either positive ion CI (PICI) or negative ion CI (NICI) analyses may be performed.

2 SCOPE

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/subdisciplines: General Chemistry, Seized Drugs, Explosives Chemistry, and Fire Debris.

3 EQUIPMENT

- Instrumentation
    - Agilent 7890 GC, 5975 or 5977 MSD with EI or CI Source, FID (if equipped) and Chemstation/Masshunter Software (or equivalent)
    - Agilent ALS, CTC PAL, or Gerstel MPS automated sampler, accessories, and software (or equivalent)
- Materials
    - Agilent J&W DB-5 MS GC column, 30 m, 0.25 mm i.d., 0.25 µm film (or equivalent) (MSD)
    - Agilent J&W DB-5, 15 m, 0.25 mm i.d., 0.25 µm film (or equivalent) (FID)
    - Helium, 99.99% (high purity)
    - Methane (CI reagent gas), 99.99% (high purity)
    - Hydrogen gas (high purity)
    - Compressed air
    - Nitrogen (high purity)
    - Perfluorotributylamine (PFTBA, FC-43) (Agilent or equivalent)
    - Perfluoro-5,8-dimethyl-3,6,9-trioxidodecane (PFDTD) Tuning Solution (Agilent or equivalent)
    - Chloroform, GC grade
    - Lidocaine HCl (Sigma or equivalent)
    - Tributoxyethyl Phosphate (TBEP) (Chem Service or equivalent)
    - 2 mL GC vials, crimp or screw top, with or without 100-500 µL inserts (Agilent or equivalent)
    - Low-bleed 11 mm injection port septa (Agilent or equivalent)

- 4 mm split-splitless tapered injection port liners and o-rings (Agilent or equivalent)
- Hamilton 701ASN 10 µL autosampler syringes (or equivalent)
- General laboratory supplies

4 STANDARDS AND CONTROLS

- PFTBA Tuning Solution (MSD)
  The PFTBA tuning solution is used for tuning the mass spectrometer and verifying mass calibration. It is supplied by the instrument manufacturer and does not expire. It is stored in a glass container attached to the MSD.
- PFDTD Tuning Solution
  The PFDTD tuning solution is used for tuning the mass spectrometer and verifying mass assignment and accuracy when the CI 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. Under normal conditions, this should not need to be refilled.
- 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 three years. This preparation may be appropriately scaled up.

5 PROCEDURE

5.1 Daily Checks

The following steps will be performed daily, regardless of the ion source installed, mode of ionization, or the detector to be used. Enter the appropriate information in the instrument log.

A. Check to ensure that the GC wash vials are filled, the waste vials are empty, and all are in the appropriate positions.
B. Record the remaining disk space on the hard drive. 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 p.s.i. or above. If it cannot maintain this pressure, contact appropriate instrument support personnel. If the instrument is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100 p.s.i. is remaining.

5.1.1 EI Source Daily Checks

If using the MSD with the EI source installed, perform the following steps. Enter the appropriate information in the instrument log.

A. 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.
B. Perform a tune of the instrument. If Autotune (ATUNE) is selected, the mass spectrometer will tune itself using PFTBA. Evaluate the results using the 'Acceptance

Criteria' section. If the results are acceptable, save and print the tune file (ATUNE) when completed.

C. Perform an analysis of the testmix. Open the appropriate testmix instrument method, and verify the parameters as listed in the 'Instrumental Conditions' section. Set up a sequence, load the autosampler with a vial containing the testmix, and start the analysis. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the TIC and mass spectra for both TBEP and Lidocaine.
D. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

5.1.2 CI Source Daily Checks

If using the MSD with the CI source installed, perform the following steps. Enter the appropriate information in the instrument log.

A. Record the tank pressure of the methane tank (reagent gas). Change the tank if less than 100 p.s.i. remaining.
B. 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 the following:
    - PICI: 6×10^{-4} torr with reagent gas on at approximately 20%
    - NICI: 6×10^{-4} torr with reagent gas on at approximately 40%
C. Perform an analysis of the testmix. Open the appropriate testmix instrument method, and verify the parameters as listed in the 'Instrumental Conditions' section. Set up a sequence, load the autosampler with a vial containing the testmix, and start the analysis. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the TIC and mass spectra for both TBEP and Lidocaine.
D. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

5.1.3 FID Daily Checks

If using the FID, perform the following steps. Enter the appropriate information in the instrument log.

A. Record the line pressure of the hydrogen supply from the generator. The value should read 50 p.s.i. or above. If it cannot maintain this pressure, contact appropriate instrument support personnel.
B. Ensure that the FID flame is lit and functioning properly.
C. Perform an analysis of the testmix. Open the appropriate testmix instrument method, and verify the parameters as listed in the 'Instrumental Conditions' section. Set up a sequence, load the autosampler with a vial containing the testmix, and start the analysis. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the chromatogram.
D. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

5.2 As Needed Maintenance/Checks

The following steps are to be performed as needed. Enter the information in the appropriate instrument log to indicate completion. Refer to IOSS-701 for more information on instrument maintenance and documentation.

A. Replace the septum in the GC injection port(s).
B. Replace the liner and o-ring within the GC injection port(s).
C. Replace the gold seal within the GC injection port(s).
D. Check the GC syringe in the autosampler. Replace if needed.
E. Replace the GC split vent trap(s).
F. Replace the autosampler bands (if applicable).
G. Check the MSD calibrant level. Fill if needed.
H. Check the MSD filaments and overall MSD performance. Replace blown filaments and clean source if needed.
I. Perform positive and/or negative CI autotune.
J. Check the FID jet (if applicable). Clean or replace if needed.

6 INSTRUMENTAL CONDITIONS

6.1 GC/MS

6.1.1 Gas Chromatograph

Oven	
Initial Temperature:	60°C
Initial Time:	2.0 min
Ramp:	35°C/min
Final Temperature:	250°C
Hold Time:	10.0 min
Equilibration Time:	0.5 min
Inlet/Injector	
Injection Volume:	1 µL
Inlet Temperature:	220°C
Mode:	Splitless
Flow Mode:	Constant flow
Initial Flow:	1.2 mL/min
Average Linear Velocity:	40 cm/sec
Carrier Gas:	Helium
Column	
Type:	DB-5 MS
Length:	30 m
Diameter:	0.25 mm
Film Thickness:	0.25 µm

6.1.2 Mass Spectrometer

Scan Mode:	Full Scan
Scan Range:	50-500 m/z
Transfer Line Temp:	280°C
Quad Temperature:	150°C
Source Temp:	200°C - 230°C
Solvent Delay:	3.0 min

6.2 GC/FID

6.2.1 Gas Chromatograph

Oven	
Initial Temperature:	100°C
Initial Time:	1.0 min
Ramp:	30°C/min
Final Temperature:	260°C
Hold Time:	4.0 min
Equilibration Time:	0.5 min
Inlet/Injector	
Injection Volume:	1 µL
Inlet Temperature:	220°C
Mode:	Split
Split Ratio:	50:1
Flow Mode:	Constant flow
Initial Flow:	1.0 mL/min
Carrier Gas:	Helium
Column	
Type:	DB-5
Length:	15 m
Diameter:	0.25 mm
Film Thickness:	0.25 µm

6.2.2 FID

Temperature:	280°C
Mode:	Constant makeup flow
Hydrogen Flow:	40.0 mL/min
Air Flow:	450.0 mL/min
Makeup Flow:	30.0 mL/min
Makeup Gas:	Nitrogen

7 ACCEPTANCE CRITERIA

7.1 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.

7.1.1 Electron Impact (EI) Ionization Mode

The following are typical EI autotune values for the MSD:

A. PFTBA tune:
   - m/z ±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% (~100% for Low Mass Autotune)
   - 502 greater than 1%
   - 18 and 28 (water & nitrogen) each 5% or below

7.1.2 Positive Ion Chemical Ionization (PICI) Mode

The following are typical PICI autotune values for the MSD:

A. PFDTD tune:
   - m/z ±0.4 for m/z 41, 267, 599

B. Peak width:
   - 0.45-0.65

C. Relative abundance:
   - 69 present
   - 267 present
   - 599 present

7.1.3 Negative Ion Chemical Ionization (NICI) Mode

The following are typical NICI autotune values for the MSD:

A. PFDTD tune:
   - m/z ±0.4 for m/z 185, 283, 351

B. Peak width:
   - 0.45-0.65

C. Relative abundance:
   - 185 present
   - 283 present
   - 351 present

7.2 Testmix

7.2.1 Gas Chromatograph (regardless of detector type)

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, symmetrical 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 significant extraneous peaks in the chromatogram.
D. The retention times of each component should be similar as compared to previous analyses (unless GC maintenance has been performed, such as column clipping or replacement).

7.2.2 Mass Spectrometer

In addition to the criteria in section 7.2.1, check the following criteria when using the mass spectrometer:

A. Check for the correct testmix mass assignments (EI):
   - Lidocaine ions 86 and 234
   - TBEP ions 57, 199, and 299
B. Check for the correct testmix mass assignments (PICI):
   - Lidocaine ion 235
   - TBEP ions 199, 299, 399

8 LIMITATIONS

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

9 SAFETY

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.

10 REVISION HISTORY

Revision	Issued	Changes
02	10/04/2018	Section 1- Updated scope to include applicable disciplines/categories of testing. Section 6- Added 'appropriate instrument support personnel' throughout. Updated Section 8- Updated to account for instrument variation and maintenance. Section 13- Updated to 'Instrument Operation and Systems Support'
03	09/15/2022	Revised to match new format requirements. Section 5- Expanded as-needed maintenance.