

Performance Monitoring Protocol (QA/QC) for the Agilent 7890/5977 Headspace GC/FID/MS Prior to the Analysis of Toxicological Samples

1 Scope

This document addresses the performance monitoring (QA/QC) of the Agilent 7890/5977 GC/FID/MS System with a headspace autosampler. This document applies to toxicology personnel using the associated instrument(s)/equipment in Quantico, VA.

2 Principle

The Agilent 7890/5977 Headspace GC/FID/MS is a gas chromatograph (GC) with a headspace autosampler, two injectors, two columns, and two internal detectors. The headspace autosampler is a device used to sample the gas phase volatile analytes within a sealed vial. This sampling is transferred to the inlet of the GC and onto a column where the components are separated and sent to the detector. There are two columns in the Agilent 7890 GC (labeled front and back), each leading to a respective detector. The front is a capillary column and leads to a mass selective detector (MSD). The back is a capillary column which leads to a flame ionization detector (FID). The headspace autosampler can be configured to inject into either inlet.

This performance monitoring protocol is based upon the manufacturer's recommendations. Definitions and guidelines for following this protocol are outlined in the *General Instrument Maintenance Protocol* (Inst_001).

3 Equipment/Materials/Reagents

- a. Instrumentation - Agilent 7890 Gas Chromatograph, Flame Ionization Detector, Agilent 5977 Mass Selective Detector with EI Source, and Chemstation/Masshunter Software (or equivalent)
- b. Autosampler - Gerstel MPS2, CTC PAL, or MPS Robotic automated sampler, accessories, and Gerstel Master/Gerstel Maestro Software (or equivalent)
- c. Capillary GC Columns:
Restek RTX BAC Plus 2, 30 m, 0.32 mm i.d., 0.6 μm film (or equivalent) (FID)
Restek RTX BAC Plus 1, 30 m, 0.32 mm i.d., 1.8 μm film (or equivalent) (MSD)
- d. Helium, 99.99% (high purity)
- e. Perfluorotributylamine (PFTBA, FC-43) (Agilent or equivalent)

- f. Hydrogen gas (high purity)
- g. Compressed air
- h. Methanol (Reagent Grade)
- i. Ethanol (200 Proof)
- j. Isopropanol (HPLC Grade)
- k. Acetone (HPLC Grade)
- l. Deionized Water, 18 M Ω ·cm (Milli-Q or equivalent)
- m. t-Butanol (Reagent grade)
- n. Autosampler vials - 10 or 20 mL crimp-top headspace autosampler vials and caps for Gerstel autosamplers (Gerstel or equivalent)
- o. Injection port septa - low-bleed 11 mm (Agilent or equivalent)
- p. Injection port liners - 1 mm split-splitless, (Restek or equivalent)
- q. Autosampler syringes - 2.5 mL headspace and 1 mL liquid syringes (Gerstel or equivalent)
- r. Nitrogen (high purity)

4 Standards and Controls

4.1 System Suitability Standard

See Section 6.3 of *Common Volatiles Analysis by Headspace GC-MSD/FID* (Tox 200) for guidance on preparation of the system suitability standard.

4.2 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. Under normal conditions, this should not need to be refilled.

5 Sampling

Not applicable.

6 Procedure

6.1 Daily Checks

The following steps will be performed daily. Enter the appropriate information in the QA/QC log.

- a. Record the remaining disk space on the hard drive. Use Windows Explorer program to verify that the hard disk has at least 100 MB of free disk space. Do not use if less than 100 MB remain.
- b. Record the line pressure of the building helium supply (carrier gas). The regulator should read 70 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.
- c. Ensure that the autosampler injects into the appropriate GC inlet.
- d. If using the FID, ensure that the FID flame is lit.
- e. If using the MSD, perform an autotune (ATUNE or Low Mass Autotune) of the instrument. The mass spectrometer will tune itself using PFTBA. Use Low Mass Autotune for TOX200 MSD analysis. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, save and print the tune file when completed.
- f. Analyze the System Suitability Standard. Evaluate the results using the 'Decision Criteria' section of this SOP prior to the analysis of evidence. If the results are acceptable, print the chromatogram(s) and associated mass spectra (if applicable).
- g. If all requirements are within specification, prepare the documentation as outlined in the *General Instrument Maintenance Protocol*. If any requirements fail, contact appropriate instrument support personnel.

6.2 As Needed Maintenance/Checks

The following steps are to be performed as needed. Record the appropriate information on the QA/QC log.

- a. Replace the septum in the GC injection port(s).
- b. Replace the liner within the GC injection port(s).
- c. Check the GC syringe in the autosampler. Replace if needed.
- d. Check the bungee cords in the autosampler (if equipped). Replace if needed.

7 Instrumental Conditions

See Section 5 of *Common Volatiles Analysis by Headspace GC-MSD/FID* (Tox 200).

8 Decision Criteria

8.1 System Suitability Standard

See Section 8 of *Common Volatiles Analysis by Headspace GC-MSD/FID* (Tox 200) for guidance on system suitability standard decision criteria.

8.2 MSD Tune

Compare the results of the Autotune or Low Mass Autotune to previous tune results. Significant voltage increases or changes in the isotope ratios indicate the need to initiate corrective maintenance procedures. The following are typical ATUNE and Low Mass Autotune values for the MSD:

- a. PFTBA Tune: Mass ± 0.4 for m/z 69, 219, and 502 (69, 131, and 219 for Low Mass Autotune)
- b. Peak width: 0.45 - 0.65 m/z
- c. Relative abundance: 69 m/z, greater than 50% (~100% for Low Mass Autotune)
131 m/z, greater than 50% (Low Mass Autotune only)
219 m/z, greater than 50% (~100% for Low Mass Autotune)
502 m/z, greater than 1%

9 Calculations

Not applicable.

10 Measurement Uncertainty

Not applicable.

11 Limitations

Not applicable.

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 (Inst_001), Instrument Operation and Systems Support SOP Manual.

Gas Chromatograph General Maintenance Protocol (Inst_002), Instrument Operation and Systems Support SOP Manual.

Mass Spectrometer General Maintenance Protocol (Inst_004), Instrument Operation and Systems Support SOP Manual.

Common Volatiles Analysis by Headspace GC-MSD/FID (TOX200), Toxicology SOP Manual.

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
1	10/13/11	Updated column details in Section 3c, deleted nitrogen from Section 3d, changed internal standard from acetonitrile to t-butanol internal in Section 3m, replaced CTC “Pal” Series vials with Gerstel vials in Section 3.n, and changed 4mm liners to 1mm liners in Section 3p. Updated numbering of Section 4. Updated Section 4.1a to use t-butanol internal standard. Added reference to Tox SOP in Sections 4.1b and 14. Added new Section 4.2 regarding PFTBA tuning solution. Changed carrier gas from nitrogen to helium in Section 7.1b. Added new Section 7.1e and renumbered subsequent sections. Removed references to internal standard solution in Sections 7.1f and 9.1. Deleted cycle time and updated fill volume, strokes, and injection rate in Section 8.1; run time, column details, inlet temp, carrier gas, and split ratio in Section 8.2; oven temp, run time, column details, inlet temp, and flow rate in Section 8.4; and transfer line temp and solvent delay in Section 8.5. Reformatted Section 8.3 to match 8.1 layout.
2	10/04/18	Updated Section 1 Scope to include applicable disciplines/categories of testing. Deleted Calibration section and renumbered. Updated heading in Section 5. Changed to ‘appropriate instrument support personnel’ in Section 7.1 b & h. Updated ‘Instrument Operation and Systems Support’ in Section 14 and header.
3	12/15/20	Updated instrument model throughout. Removed general chemical analysis from the Scope. Updated software in 3.a. Updated GC columns in 3.c. Updated System Suitability Standard in 4.1. Updated 7 and 8.1 to reflect current toxicology procedures. Added Low Mass Autotune in Section 8.2. Updated Tox Volatile SOP name in Section 13.

Approval

Redacted - Signatures on File

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