Toxicology Headspace GC/FID/MS Performance Monitoring and Maintenance

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1 INTRODUCTION

This document addresses the performance monitoring and maintenance of the Agilent 7890/5977 GC/FID/MS System with a headspace autosampler. 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 that leads to a mass selective detector (MSD). The back is a capillary column that leads to a flame ionization detector (FID). The headspace autosampler can be configured to inject into either inlet. Definitions and guidelines are outlined in IOSS-701.

2 SCOPE

This document applies to personnel using the associated instrument(s)/equipment in the Toxicology discipline.

3 EQUIPMENT

- Instrumentation
    - Agilent 7890 Gas Chromatograph, Agilent Flame Ionization Detector, Agilent 5977 Mass Selective Detector with EI Source, and Chemstation/Masshunter Software (or equivalent)
    - Gerstel MPS2, CTC PAL, or MPS Robotic automated sampler, accessories, and Gerstel Master/Gerstel Maestro Software (or equivalent)
- Materials
    - Restek RTX BAC Plus 2 GC column, 30 m, 0.32 mm i.d., 0.6 µm film (or equivalent) (FID)
    - Restek RTX BAC Plus 1 GC column, 30 m, 0.32 mm i.d., 1.8 µm film (or equivalent) (MSD)
    - Helium, 99.99% (high purity)
    - Hydrogen gas (high purity)
    - Compressed air
    - Nitrogen (high purity)
    - Perfluorotributylamine (PFTBA, FC-43) (Agilent or equivalent)
    - Methanol (Reagent Grade)
    - Ethanol (200 Proof)
    - Isopropanol (HPLC Grade)
    - Acetone (HPLC Grade)
    - Deionized Water, 18 MΩ·cm (Milli-Q or equivalent)
    - t-Butanol (Reagent grade)
    - Low-bleed 11 mm injection port septa (Agilent or equivalent)
    - 1 mm split-splitless injection port liners and o-rings (Restek or equivalent)

- 2.5 mL headspace and 1 mL liquid syringes (Gerstel or equivalent)
- 10 or 20 mL crimp-top headspace autosampler vials and caps for Gerstel autosamplers (Gerstel or equivalent)
- General laboratory supplies

4 STANDARDS AND CONTROLS

- System Suitability Standard
  See Section 6.3 of TOX-200 for guidance on preparation of the system suitability standard.
- 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.

5 PROCEDURE

5.1 Daily Checks

The following steps will be performed daily. Enter the appropriate information in the instrument log.

A. 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.
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 is set to inject 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 TOX-200 MSD analysis. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, save and print the tune file when completed.
F. Analyze the system suitability sample. Evaluate the results using the 'Acceptance Criteria' section 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 IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

5.2 Yearly Scheduled Maintenance/Checks

The following steps will be performed once in a calendar year. 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 GC split vent trap(s).
B. Check the overall column performance. Clip the inlet side of the GC column(s) if needed.
C. Check the MSD calibrant level. Fill if needed.
D. Check the MSD filaments and overall MSD performance. Replace blown filaments and clean source if needed.

5.3 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. Check the water level in the hydrogen generator. Fill with 18 MΩ·cm deionized water if needed.
B. Replace the septum in the GC injection port(s).
C. Replace the liner and o-ring within the GC injection port(s).
D. Replace the gold seal within the GC injection port(s).
E. Check the GC syringe in the autosampler. Replace if needed.
F. Check the FID jet. Clean or replace if needed.

6 INSTRUMENTAL CONDITIONS

See Section 7 of TOX-200.

7 ACCEPTANCE CRITERIA

7.1 System Suitability Sample

See Section 8.1 TOX-200 for guidance on system suitability sample criteria.

7.2 MSD Tune

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

A. PFTBA tune:
    - m/z ±0.4 for m/z 69, 219, and 502 (69, 131, and 219 for Low Mass Autotune)
B. Peak width:
    - 0.45-0.65
C. Relative abundance:
    - 69 greater than 50% (~100% for Low Mass Autotune)
    - 131 greater than 50% (Low Mass Autotune only)
    - 219 greater than 50% (~100% for Low Mass Autotune)
    - 502 greater than 1%
    - 18 and 28 (water & nitrogen) each 5% or below

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
03	12/15/2020	Updated instrument model throughout. Added reference to TOX-200 throughout. Section 1- Removed General Chemical Analysis from Scope. Section 3- Updated software and GC columns. Section 4- Updated System Suitability Standard Section 6- Updated to 'appropriate instrument support personnel'. Section 8- Added Low Mass Autotune.
04	09/15/2022	Revised to match new format requirements. Section 5- Added annual maintenance and expanded as-needed maintenance.