Performance Monitoring Protocol (QA/QC) for the Agilent 7890 GC/ECD/NPD

1 Scope

This document addresses the performance monitoring (QA/QC) of the Agilent 7890 GC/ECD/NPD System. This document applies to personnel using the associated instrument(s)/equipment in Quantico, VA in the following disciplines/categories of testing: Drug chemistry, toxicology, and Chemistry Unit general physical and chemical analysis.

2 Principle

The Agilent 7890 GC/ECD/NPD is a gas chromatograph (GC) with two injectors, two columns, and two internal detectors. The instrument is configured with two capillary columns, each leading to their respective detectors. The column in the front position is connected to an electron capture detector (ECD), and the column in the back leads to a nitrogen-phosphorus detector (NPD). Samples are introduced into either column through the use of standard GC injection ports. The system may also be referred to as the 'ECD/NPD.'

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."

3 Equipment/Materials/Reagents

a. Instrumentation - Agilent 7890 Gas Chromatograph, Electron Capture Detector, Nitrogen-Phosphorus Detector, and Chemstation Software (or equivalent)

b. Autosampler - Agilent or CTC "Pal" Series automated sampler, accessories, and software (or equivalent)

c. ECD GC Column - Rtx-ClPest (Restek or equivalent)

d. NPD GC Column - Rtx-1701 (Restek or equivalent)

e. Carrier Gas - Helium, 99.99% (high purity)

f. Detector Makeup Gas - Nitrogen, 99.99% (high purity)

g. Hydrogen gas - high purity or equivalent

h. Compressed air

i. Autosampler vials - 2 mL GC vials, crimp or screw top, with or without 200 µL

inserts (HP or equivalent)

j. Injection port liners - 4 mm split-splitless, tapered, with or without glass wool (HP or equivalent)

k. Injection port septa - low-bleed 11 mm (HP or equivalent)

l. Autosampler syringes - Hamilton 701ASN 10 µL (or equivalent)

4 Standards and Controls

4.1 Performance Verification Standards

a. Organochlorine (OC) Pesticides Stock Solution:
A hexane:toluene (1:1) solution approximately 1 mg/mL each of aldrin, 4,4'-DDT, endrin, endrin aldehyde, and lindane. Purchased as a special order item from Chemservice, Inc. Store refrigerated in glass. Stable for at least two years, or as determined by manufacturer.

b. ECD Pesticides Testmix Solution:
Dilute 25 µL of the OC pesticides stock solution to 50 mL with hexane, yielding a solution approximately 0.5 µg/mL in each component. Store refrigerated in glass. Stable for at least two years. A portion of this testmix is analyzed prior to each batch of GC-ECD analyses.

c. Organophospahte (OP) Pesticides Stock Solution:
A hexane solution approximately 1 mg/mL each of chlorpyrifos, diazinon, fenchlorphos, parathion (ethyl), and prophos. Purchased as a special order item from Chemservice, Inc. Store refrigerated in glass. Stable for at least two years, or as determined by manufacturer.

d. NPD Pesticides Testmix Solution:
Dilute 500 µL of the OP pesticides stock solution to 25 mL in hexane, yielding a solution approximately 20 µg/mL in each component. Store refrigerated in glass. Stable for at least two years. A portion of this testmix is analyzed prior to each batch of GC-NPD analyses.

e. The performance verification standard is used to assess daily operating performance and continued integrity of the gas chromatography-detector system. The positive control standard for the applicable discipline-specific procedure and detector will be analyzed and evaluated as the ECD or NPD Performance Standard prior to the analysis of evidence.

5 Sampling or Sample Selection

Not applicable.

6 Procedures

6.1 Daily Checks

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

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.

d. Record the line pressure of the building nitrogen supply (makeup 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.

e. Conduct a performance verification standard analysis and evaluate prior to the analysis of evidence.

f. Evaluate the results using the 'Decision Criteria' section of this SOP. If the results are acceptable, print the chromatogram for the performance verification standard.

g. Prepare specific documentation as outlined in the "General Instrument Maintenance Protocol."

6.2 As Needed Checks

The following steps will be performed as needed based on system performance. Record the appropriate information on the QA/QC 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.

d. Check the internal bungee cords in the autosampler. Replace if needed.

e. Check the plungers in each autosampler syringe. Replace if needed.

7 Instrumental Conditions

7.1 Gas Chromatograph/Nitrogen-Phosphorus Detector

Oven

Temp 1:	125°C
Hold 1:	1 min
Ramp 1:	7°C/min
Temp 2:	280°C
Hold 2:	22 min

Column

Type:	Rtx-1701
Length:	30 m
Inner diameter:	0.32 mm
Film thickness:	0.5 µm

Inlet and Carrier

Inlet temp:	250°C
Injection mode:	Split
Carrier gas:	Helium
Carrier mode:	Constant pressure
Carrier pressure:	13.39 psi
Split ratio:	15:1

NPD

Temperature:	250°C
Offset:	10
Makeup flow: (nitrogen)	30 mL/min
Air flow:	60 mL/min
Hydrogen flow:	2 mL/min

7.2 Gas Chromatograph/Electron Capture Detector

Oven

Temp 1	125°C
Hold 1	1 min
Ramp 1	7°C/min
Temp 2	280°C
Hold 2	22 min

Column	
Type	Rtx-ClPest
Length	30 m
Inner diameter	0.32 mm
Film thickness	0.5 µm

Inlet and Carrier	
Inlet temp	230°C
Injection mode	Splitless
Carrier gas	Helium
Carrier mode	Constant pressure
Carrier pressure	16.85 psi
Splitless time	0.5 min

ECD	
Temp	300°C
Makeup gas	Nitrogen
Makeup flow	30 mL/min

8 Decision Criteria

8.1 Performance Verification Standard

The peaks of the analytes in the performance standard should show good chromatographic fidelity, with reasonable peak shape, width, and resolution. Peak areas and retention times should compare favorably to previous analyses of the performance standard.

9 Calculations

Not applicable.

10 Measurement Uncertainty

Not applicable.

11 Limitations

Only properly trained personnel will perform such duties involved in the 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 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.*

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
0	06/21/06	New document which replaces original titled "Performance Monitoring Protocol (QA/QC) for the HP 6890 GC/ECD/NPD"
1	06/08/09	Changed model number in Title and Sections 1, 2, and 3.a. Removed references to Hewlett Packard and HP in the Title and Sections 1 and 2. Clarified statement in Section 7.2 and added c and d. Changed 'slit' to 'split' in Section 8.1. Made changes to oven parameters in Sections 8.1 and 8.2. Made changes to solutions in Section 4.1 to reflect Tox Subunit modifications.
2	xxxx/18	Updated Section 1 Scope to include appliacable disciplines/categories of testing. Deleted Calibration Section and renumbered. Updated heading in Section 5. Changed ‘subunit’ to ‘discipline’ in Section 4.1 e. Added ‘appropriate instrument support personnel’ to Section 7.1 c & d. Updated ‘Instrument Operation and Systems Support’ in Section 14 and header.

Approval

Redacted - Signatures on File

Drug Chemistry/
General Chemistry
Technical Leader: Date: 09/28/2018

Toxicology
Technical Leader: Date: 09/28/2018

IOSS Manager: Date: 09/28/2018

Chemistry Unit Chief: Date: 09/28/2018

QA Approval

Quality Manager: Date: 09/28/2018