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

## 1 Scope

This document addresses the performance monitoring (QA/QC) of the Agilent Gas Chromatograph with an Electron Capture Detector (GC/ECD) system. 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/ECD system consists of a gas chromatograph (GC) with an Electron Capture Detector (ECD). This performance monitoring protocol is generally 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 GC, Electron Capture Detector, and Chemstation software (or equivalent)

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

c. GC Column - Agilent DB-5 MS, 6 m, 0.25 mm i.d., 0.25 µm film (or equivalent)

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

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

Redacted

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

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

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

k. Autosampler syringes - 10 µL syringes (Agilent or equivalent)

l. Wash vials - 4 mL screw top without insert (Agilent or equivalent)

m. Volumetric flask

n. Acetone, Reagent grade

## 4 Standards and Controls

### 4.1 GC/ECD Testmix

The GC/ECD testmix is a 10 ppm solution Redacted in acetone. Individual 100 ppm or 1000 ppm standard solutions of most components are available from Cerilliant, or equivalent. Redacted

A 100 ppm intermediate stock solution is prepared by adding 2 mL of each 1000 ppm liquid component and 2 mg of each solid component to a 20-mL volumetric flask, and diluting to volume with acetone.

The 10 ppm testmix is prepared by adding 1 mL of the 100 ppm stock solution to a 10-mL volumetric flask and diluting to volume with acetone. The testmix and intermediate stock solutions will be maintained in colored or amber vials in a refrigerator.

The Testmix is used to assess daily operating performance and continued integrity of the system.

## 5 Sampling

Not applicable.

## 6 Procedures

### 6.1 Daily Checks

The following steps are to be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

a. Check to ensure that the GC wash vials are filled with acetone, 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 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 chromatogram.

e. 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 and Maintenance

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

Oven

| | |
|---|---|
| Initial Temp: | 50°C |
| Initial Time: | 1.5 min |
| Ramp: | 25°C/min |
| Final Temp: | 250°C |
| Hold Time: | 0.5 min |
| Equilibration Time: | 1.0 min |

Inlet/Injector

| | |
|---|---|
| Inj Vol: | 1.0 µL |
| Inlet temperature: | 225ºC |
| Injection mode: | Split |
| Carrier gas: | Helium, 99.99% (split) |
| Carrier mode: | Constant flow |
| Pressure: | 9.5 psi |
| Split ratio: | 5:1 |

Column

| | |
|---|---|
| Type: | DB-5 MS |
| Length: | 6 m (approximate) |
| Diameter: | 0.25 mm |
| Film Thickness: | 0.25 µm |

Detector

| | |
|---|---|
| Temperature: | 275°C |
| Mode: | Constant makeup flow |
| Makeup flow: | 25 mL/min |
| Makeup Gas: | Nitrogen |

## 8 Decision Criteria

Verify the results of the Testmix.

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

b. The retention times of the components should not deviate by ±3% compared to the previous run.

## 9 Calculations

Not applicable.

## 10 Measurement Uncertainty

Not applicable.

## 11 Limitations

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

## 12 Safety

The ECD uses a sealed Nickel 63 source that should not be opened or modified in any way. Radiation leak checks are performed approximately twice per year as a safety precaution. Only properly trained personnel shall perform duties involved in the radiation leak testing of this instrument.

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.*

*FBI Laboratory Safety Manual*
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| Rev. # | Issue Date | History |
|---|---|---|
| 0 | 10/04/18 | New document that specifies instrument protocol for the Huntsville facility. |
| 1 | 07/15/20 | Updated "Scientific and Biometrics Analysis Unit" in header and approval section. Added Section 4.1 heading. Removed Redacted Sections 3g, 4.1, and 8a. Added Redacted to Sections 3g, 4.1, and 8a. Updated heading in Section 6.2. Removed sentence about minor deviations from Section 7. Added unit name to title of each approver. |

**Approval** Redacted - Signatures on File

Scientific and Biometrics
Analysis Unit Chief Date: 07/14/2020

Explosives Unit-Chemistry
Technical Leader Date: 07/14/2020

Explosives Unit Chief Date: 07/14/2020