

GC/ECD Performance Monitoring and Maintenance

Table of Contents

1	INTRODUCTION	2
2	SCOPE	2
3	EQUIPMENT	2
4	STANDARDS AND CONTROLS	2
5	PROCEDURE	3
5.1	Daily Checks	3
5.2	As Needed Checks	3
6	INSTRUMENTAL CONDITIONS.....	4
6.1	Gas Chromatograph	4
7	ACCEPTANCE CRITERIA.....	4
7.1	Testmix.....	4
8	LIMITATIONS	4
9	SAFETY	5
10	REVISION HISTORY	5

GC/ECD Performance Monitoring and Maintenance

1 INTRODUCTION

This document addresses the performance monitoring and maintenance of the GC/ECD system. The Agilent GC/ECD is a gas chromatograph (GC) with an Electron Capture Detector (ECD). A sample is introduced onto the column by either manual or autosampler injection through an injection port. Definitions and guidelines are outlined in IOSS-701.

2 SCOPE

This document applies to personnel using the associated instrument(s)/equipment in the Explosives Chemistry subdiscipline.

3 EQUIPMENT

- Instrumentation
 - Agilent 7890 GC, ECD, and ChemStation Software (or equivalent)
 - Agilent ALS, accessories, and software (or equivalent)
- Materials
 - Agilent DB-5MS GC column, 6 m (approximate), 0.25 mm i.d., 0.25 μ m film (or equivalent)
 - Helium, 99.99% (high purity)
 - Nitrogen Gas, 99.99% (from gas generator, tank, or equivalent)
 - Redacted

Redacted

- 2 mL GC vials, crimp or screw top, with or without 100-500 μ L inserts (Thermo or equivalent)
- Acetone (reagent grade)
- 4 mm split-splitless, tapered GC inlet liners, with or without glass wool (Agilent or equivalent)
- Low-bleed 11 mm injection port septa (Agilent or equivalent)
- Hamilton 701ASN 10 μ L autosampler syringes (or equivalent)
- General laboratory supplies

4 STANDARDS AND CONTROLS

- GC/ECD Testmix

The GC/ECD testmix is a 10 ppm solution of Redacted in acetone. Individual 100 ppm or 1000 ppm standard solutions of most components are available from Accustandard, or Redacted s and can be synthesized as needed. The following preparations may be scaled appropriately and

modified based on the concentration of the starting materials and quantity required. If needed, 1000 ppm stock solutions can be prepared from solids by adding 100 mg of solid material to a 100-mL volumetric flask and diluting to volume with acetone.

The 10 ppm testmix is prepared by adding 100 μ L of the 1000 ppm purchased or stock solutions to a 10-mL volumetric flask and diluting to volume with acetone. The testmix and stock solutions will be maintained in amber vials in a refrigerator. The testmix is used to assess daily operating performance and continued integrity of the system.

5 PROCEDURE

5.1 Daily Checks

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

- A. 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. remaining.
- D. 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.
- E. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the chromatogram.
- F. 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 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).

6 INSTRUMENTAL CONDITIONS

6.1 Gas Chromatograph

Inlet/Injector	
Injection Volume:	1.0 µL
Mode:	Split
Inlet Temperature:	225°C
Carrier Gas:	Helium
Initial Inlet Pressure:	9.5 p.s.i
Split Ratio:	5:1
Flow Mode:	Constant Makeup Flow
Initial Flow:	3.7 mL/min
Oven	
Initial Temperature:	50°C
Initial Time:	1.5 min
Ramp:	25°C/min
Final Temperature:	250°C
Hold Time:	0.5 min
Final Time:	10.0 min
Column	
Type:	DB-5MS (or equivalent)
Length:	6 m (approximate)
Diameter:	0.25 mm
Film Thickness:	0.25 µm
Detector	
Temperature:	275°C
Makeup Flow:	25.0 mL/min
Makeup Gas:	Nitrogen

7 ACCEPTANCE CRITERIA

7.1 Testmix

- A. In order for the instrument to be considered in good operating condition, Redacted should generate well resolved, Gaussian-shaped peaks with baseline separation.
- B. An SNR of 3:1 will be the minimum response necessary to consider a response a peak.
- C. The retention times of the components should not deviate by $\pm 3\%$ compared to the previous run (unless GC maintenance has been performed, such as column clipping or replacement).

8 LIMITATIONS

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

9 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 will perform duties involved in the radiation leak testing of this instrument.

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
04	07/01/2022	Revised to match new format requirements. No other substantive changes to content.
05	11/15/2022	Section 2- Updated scope to remove location. Section 5.2- Expanded as needed checks. No other substantive changes to content.