

GC/ECD/NPD Performance Monitoring and Maintenance

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GC/ECD/NPD Performance Monitoring and Maintenance

1 INTRODUCTION

This document addresses the performance monitoring and maintenance of the Agilent GC/ECD/NPD system. The Agilent 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. Definitions and guidelines are outlined in IOSS-701.

2 SCOPE

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/subdisciplines: General Chemistry and Seized Drugs.

3 EQUIPMENT

- Instrumentation
 - Agilent 7890 Gas Chromatograph, ECD, NPD, and Chemstation Software (or equivalent)
 - Agilent or CTC "Pal" Series automated sampler, accessories, and software (or equivalent)
- Materials
 - Restek Rtx-CLPest GC column, 30m, 0.32 i.d., 0.5 µm film (or equivalent) (ECD)
 - Restek Rtx-1701 GC column, 30m, 0.32 i.d., 0.5 µm film (or equivalent) (NPD)
 - Helium, 99.99% (high purity)
 - Compressed air (from air purifier, compressor, tank, or equivalent)
 - Hydrogen Gas, 99.9% (from gas generator, tank, or equivalent)
 - Nitrogen Gas, 99.99% (from gas generator, tank, or equivalent)
 - 4 mm split-splitless, tapered GC inlet liners, with or without glass wool (Agilent or equivalent)
 - Low-bleed 11 mm injection port septa (Restek or equivalent)
 - 701ASN 10 µL autosampler syringes (Hamilton or equivalent)
 - Deionized Water, 18 MΩ·cm (Milli-Q or equivalent)
 - Autosampler vials - 2 mL GC vials, crimp or screw top, with or without 200 µL inserts (Agilent or equivalent)
 - Organochlorine (OC) Pesticides Stock Solution:
Hexane:toluene (1:1) solution approximately 1 mg/mL each of aldrin, 4,4'-DDT, endrin, endrin aldehyde, and lindane (ChemService or equivalent)
 - Organophosphate (OP) Pesticides Stock Solution:
A hexane solution approximately 1 mg/mL each of chlorpyrifos, diazinon, fenchlorphos, parathion (ethyl), and prophos (ChemService or equivalent)
 - General laboratory supplies

4 STANDARDS AND CONTROLS

The testmix is used to assess daily operating performance and continued integrity of the gas chromatography-detector system prior to the analysis of evidence. A positive control standard for the applicable discipline/subdiscipline-specific procedure and detector can be substituted for the ECD or NPD testmix provided it is documented in the appropriate log.

- **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.
- **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.

5 PROCEDURE

5.1 Daily Checks

- Ensure that the GC wash vials are filled, the waste vials are empty, and all are in the appropriate positions.
- 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.
- 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.
- 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. remaining.
- If using the NPD, verify that the hydrogen generator water level is sufficient and is generating the proper hydrogen gas pressure.
- Perform an analysis of the appropriate testmix or discipline/subdiscipline-specific positive control.
- Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the chromatogram.
- 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 will be performed as needed based on system performance. Indicate completion in the appropriate log. 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 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).
- F. Check the overall column performance. Clip the inlet side of the GC column(s) if needed.
- G. Check the internal bungee cords in the autosampler (if applicable). Replace if needed.
- H. Check the water level in the hydrogen generator. Fill with 18 MΩ·cm deionized water if needed.
- I. Check the NPD bead (if applicable). Replace if needed.

6 INSTRUMENTAL CONDITIONS

6.1 GC/ECD

Inlet/Injector	
Injection Volume:	1.0 µL
Mode:	Splitless
Splitless time:	0.5 min
Temperature:	230°C
Oven	
Initial Temperature:	125°C
Initial Time:	1.0 min
Ramp:	7°C/min
Final Temp:	280°C
Final Temp Hold Time:	22.0 min
Column	
Type:	Rtx-CLPest
Length:	30 m
Diameter:	0.32 mm
Film Thickness:	0.5 µm
Flow Mode:	Constant Pressure
Carrier Pressure:	16.85 psi
Carrier Gas:	Helium
Detector	
Temperature:	300°C
Makeup Gas:	Nitrogen
Makeup Flow:	30.0 mL/min

6.2 GC/NPD

Inlet/Injector	
Injection Volume:	1.0 µL
Mode:	Split
Split Ratio:	15:1
Temperature:	250°C
Oven	
Initial Temperature:	125°C
Initial Time:	1.0 min
Ramp:	7°C/min
Final Temp:	280°C
Final Temp Hold Time:	22.0 min
Column	
Type:	Rtx-1701
Length:	30 m
Diameter:	0.32 mm
Film Thickness:	0.5 µm
Flow Mode:	Constant Pressure
Carrier Pressure:	13.39 psi
Carrier Gas:	Helium
Detector	
Temperature:	250°C
Offset:	10
Makeup Gas:	Nitrogen
Makeup Flow:	30.0 mL/min
Air Flow:	60 mL/min
Hydrogen Flow:	2 mL/min

7 ACCEPTANCE CRITERIA

The peaks of the analytes in the testmix or discipline/subdiscipline positive control should show good chromatographic fidelity, with reasonable peak shape, width, and resolution. If applicable, peak areas and retention times should compare favorably to previous analyses of the performance standard.

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
02	10/04/2018	Section 1- Updated Scope. Section 4- Changed 'subunit' to 'discipline'. Section 7- Added 'appropriate instrument support personnel' Section 6- Added 'appropriate instrument support personnel'. Section 14- Updated 'Instrument Operation and Systems Support'.
03	09/30/2022	Revised to match new format requirements. Section 5- Expanded as needed checks. No other substantive changes to content.
04	02/18/2025	Section 2 – Updated scope to remove Toxicology.