

High Temperature GC/FID Performance Monitoring and Maintenance

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High Temperature GC/FID Performance Monitoring and Maintenance

1 INTRODUCTION

This document addresses the performance monitoring and maintenance of the Agilent High Temperature GC/FID. The Agilent High Temperature GC/FID is a gas chromatograph (GC) with a Multi-Mode inlet and a Flame Ionization Detector (FID). 'High Temperature' refers to the fact that the inlet is programmable to increase during a run and attain a higher temperature than an inlet is normally set, and the capillary column used is specifically designed to allow the temperature to be raised to a higher level than is typically applied to GC columns. 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 following subdisciplines: Explosives Chemistry and General Chemistry.

3 EQUIPMENT

- Instrumentation
 - Agilent 7890 Gas Chromatograph, FID, Multi-Mode inlet, and ChemStation Software (or equivalent)
 - Agilent ALS, accessories, and software (or equivalent)
- Materials
 - Zebron "Inferno" ZB-1HT Capillary Column, 15 m x 0.25 mm, 0.1 μ m film thickness (Phenomenex or equivalent)
 - 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)
 - Hexane (B&J UV grade or equivalent)
 - n-Paraffin Mix ~C16-C44 at 0.01% (w/w) in cyclohexane (Supelco 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)
 - Hamilton 701ASN 10 μ L autosampler syringes (or equivalent)
 - General laboratory supplies

4 STANDARDS AND CONTROLS

- Hi-Temp GC Testmix

The testmix is a commercially available hexane or cyclohexane solution of n-alkanes made up of ~C16-C44. This performance standard should have a concentration of approximately 0.001% (v/v) paraffin in hexane and be stored at room temperature in an amber vial. This solution does not expire but should be re-evaluated for

continued use after three years. The testmix is used to verify daily operating performance and continued integrity of the gas chromatograph-detector system.

5 PROCEDURE

5.1 Daily Checks

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

- 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. Ensure that the hydrogen generator is on and supplying gas to the FID. Verify that the FID is lit.
- E. Load the testmix method (see 'Instrumental Conditions' section). Program the autosampler to inject 1 μ L of the testmix. It is also acceptable to manually inject 1 μ L of the testmix and press Start on the GC.
- F. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the chromatogram for the performance verification standard.
- 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 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).
- F. Replace the autosampler bands (if applicable).
- G. Check the FID jet. Clean or replace if needed.

6 INSTRUMENTAL CONDITIONS

6.1 Gas Chromatograph

Inlet/Injector	
Injection Volume:	1.0 µL
Mode:	Splitless
Initial Temperature:	55°C
Ramp Rate:	500°C/min
Ramp Time:	10 min
Final Temperature:	400°C
Oven	
Initial Temperature:	55°C
Initial Time:	2.0 min
Ramp1:	30°C/min
Final Temp1:	100°C
Ramp1 Time:	0.0 min
Ramp2:	15°C/min
Final Temp2:	400°C
Ramp2 Time:	3.5 min
Run Time:	27.0 min
Equilibration Time:	0.5 min
Column	
Type:	Zebtron ZB-1HT
Length:	15 m
Diameter:	0.25 mm
Film Thickness:	0.1 µm
Flow Mode:	Constant Flow
Flow Rate:	1 mL/min
Carrier Gas:	Helium
Detector	
Temperature:	420°C
Mode:	Constant makeup flow
Hydrogen Flow:	40.0 mL/min
Air Flow:	450.0 mL/min
Makeup Gas:	Nitrogen
Makeup Flow:	30.0 mL/min

7 ACCEPTANCE CRITERIA

7.1 Testmix

- A. In order for the instrument to be considered in good operating condition, all components should generate well-resolved, Gaussian-shaped peaks with baseline separation.
- B. An SNR of 3:1 is the minimum response necessary to consider a response a peak.
- C. There should be no extraneous peaks in the testmix chromatogram greater than 5% of the tallest peak.

- D. The retention times of components should not deviate by $\pm 3\%$ compared to previous runs of the testmix (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

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
06	07/01/2022	Revised to match new format requirements. No other substantive changes to content.
07	11/15/2022	Section 2- Updated Scope to remove location. Section 5.2- Expanded as needed checks. No other substantive changes to content.