	Standard Operating Procedure Agilent 7890 GC Operation Maintenance and Qualification		SOP Number D-801	Revision 1
			Effective Date 01/03/23	Page 1 of 20
Written by/ Date SSR 12/20/22		Reviewed by/ Date SAS 12/20/22		Approved by/ Date K. Brunner 12/20/22
Title: Quality Control Director		Title: Analytical Development Scientist		Title: Quality Assurance Director

1.0 Purpose

This procedure provides guidelines for general use, maintenance and qualification of GC systems.

2.0 Scope

This procedure applies to all GC systems used in the QC Laboratory at Ion Labs.

3.0 Responsibility

- 3.1 It is the responsibility of QC Laboratory analysts to follow the guidelines for general use of GC systems.
- 3.2 QC Laboratory Management and/or AD personnel are responsible for ensuring analysts follow the guidelines set forth herein.
- 3.3 It is the responsibility of QC Laboratory Management, AD personnel, and/or outside contractors to perform maintenance and qualification of GC instrumentation.
- 3.4 It is the responsibility of QC Laboratory Management and/or AD personnel to keep this SOP current with the latest Ion Labs Practices.

4.0 Definitions

- 4.1 **QC** – Quality Control
- 4.2 **AD** – Analytical Development
- 4.3 **GC** – Gas Chromatography
- 4.4 **FID** – Flame Ionization Detector
- 4.5 **ALS** – Automatic Liquid Sampler
- 4.6 **IQ** – Installation Qualification

4.7 **OQ** – Operational Qualification

4.8 **PQ** – Performance Qualification

5.0 References

5.1 D-807-F1, Form, HPLC/ GC Data Review Checklist

5.2 D-808, SOP, Use of OpenLab for HPLC and GC Data Acquisition and Reporting

5.3 D-603, SOP, Chemical Waste Disposal

6.0 Equipment

6.1 Agilent 7890B Gas Chromatograph

6.2 Agilent 7697A Headspace Sampler

6.3 Agilent 7693 Autosampler

7.0 Safety Precautions

7.1 Solvents used for sample preparation are toxic and flammable. The minimum required personal protective equipment includes safety glasses, gloves, and enclosed shoes.

7.2 Compressed gases may cause injury or suffocation if a sudden leak occurs. Always stop the flow of gases before changing the column, inlet septum, inlet liner, transfer line, or performing instrument maintenance. Use two stage regulators to ensure consistent gas supply to the instrument.

7.3 Decrease the temperature of heated zones and allow them to cool before performing operations.

7.4 Gas cylinders can be hazardous due to the high pressure of the gas contained within as well as the flammability of some gases. Always store gas cylinders in an upright position and secured from tipping by a cylinder cage, strap, chain, or guard. Never remove the cap of a cylinder unless it is properly secured. Always use a cylinder cart to move gas cylinders, and never move a cylinder without the cap on. Never use a gas cylinder or regulator that has visible damage.

- 7.5 Take care not to mix gas cylinder regulators. Connecting a cylinder to the wrong gas line could cause fire or explosion.
- 7.6 Electrical faults could cause electrocution, explosion or fire. If an electrical fault is suspected, disconnect power from the instrument and have it serviced by a qualified individual.

8.0 Waste Handling and Disposal

- 8.1 Waste handling and disposal procedures are outlined in SOP D-603 Chemical Waste Disposal.

9.0 General Guidelines

9.1 Documentation

- 9.1.1 The following parameters should be recorded in the laboratory notebook:

- 9.1.1.1 Instrument Ion Number
- 9.1.1.2 Instrument Cal Due Date
- 9.1.1.3 Column identifier

9.1.2 HPLC/ GC Data Review Checklist

- 9.1.2.1 Each sequence generated from the use of HPLC and/ or GC should include Form D-807-F1 HPLC/ GC Data Review Checklist.

- 9.1.2.1.1 In the event a sequence was rejected for any reason, this checklist is still required to be completed, listing the rationale for the rejection.

- 9.1.2.2 This checklist is completed by both the analyst and the data reviewer to ensure all essential information was captured and is present in the data packet.

- 9.1.2.3 Additionally, both the analyst and the data reviewer use the checklist to show assessment of all method suitability requirements.

9.2 Sample Filtration and/or Centrifugation

- 9.2.1 Samples to be injected into the GC system must be free of all particulates.
- 9.2.2 Filtration is the preferred method for removal of particulates. Use a filter with 0.45 µm membrane for sample filtration.
- 9.2.3 Alternatively, centrifugation may be used to remove particulates provided that the resulting solution is clear.

9.3 Sample pH

- 9.3.1 Do not inject samples containing strong acids or bases as these can damage a GC column.

9.4 Gas Purity

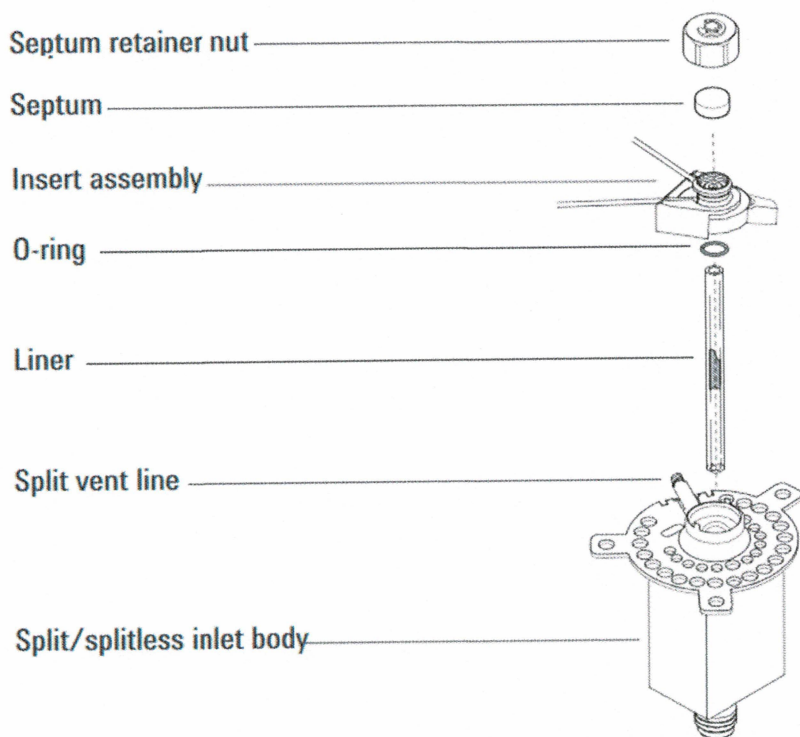
- 9.4.1 Use Ultra High Purity (UHP) grade compressed helium, nitrogen, and hydrogen.
- 9.4.2 Use Ultra Zero (UZ) grade compressed air.

10.0 Procedure for General Use

- 10.1 The following instructions outline how to prepare the GC to run samples. Refer to SOP D-808 Use of OpenLab for HPLC and GC Data Acquisition and Reporting for instructions on how to run samples, process the results, and generate a report.
- 10.2 Ensure that all gas cylinders are connected and that the supply valve for each cylinder is open.
- 10.3 Ensure that all gas cylinders have supply pressure (right gauge on the regulator) greater than 200 psi.
- 10.4 Ensure that gas cylinder line pressures (left gauge on the regulator) are within the recommended ranges:
 - 10.4.1 Helium (carrier): minimum is 20 psi greater than the maximum pressure used in the method, maximum is 120 psi.
 - 10.4.2 Hydrogen (fuel): 35 – 100 psi.
 - 10.4.3 Nitrogen (makeup): 55 – 100 psi.

- 10.4.4 Air (oxidant): 55 – 100 psi.
- 10.5 Turn on the power to the gas chromatograph (7890B) and headspace sampler (7697A).
- 10.6 If performing direct injection with the 7693 Automatic Liquid Sampler (ALS):
 - 10.6.1 Open the door of the ALS tower, and remove the syringe taking care not to bend the needle or plunger.
 - 10.6.2 Pull the plunger back and then push it down. If any resistance is felt, clean the syringe using an appropriate solvent. Most often, rinsing the syringe with water and then isopropanol is sufficient. If resistance is still encountered after cleaning, replace with a new syringe.
 - 10.6.3 Inspect the syringe tip. If damage is observed, replace with a new syringe.
 - 10.6.4 Re-install the syringe into the ALS tower.
 - 10.6.5 Fill the rinse vials in positions A and B of the ALS rotary turret with the diluent used in the method. If the diluent contains additives (e.g. internal standard), do not include the additives in the rinse solvent.
- 10.7 To change the inlet liner or septum (change after every 50 – 100 injections):
 - 10.7.1 Turn off heating to the inlet:
 - 10.7.1.1 On the instrument keypad, select Front Inlet or Back Inlet depending on which one you are using.
 - 10.7.1.2 Use the up/down arrows to navigate to Temperature.
 - 10.7.1.3 Press the Off button.
 - 10.7.2 Turn off flow to the inlet.
 - 10.7.2.1 On the instrument keypad, select Front Inlet or Back Inlet depending on which one you are using.
 - 10.7.2.2 Use the up/down arrows to navigate to Pressure.
 - 10.7.2.3 Press the Off button.
 - 10.7.3 When inlet and oven temperatures are $< 50\text{ }^{\circ}\text{C}$, proceed to the next step.

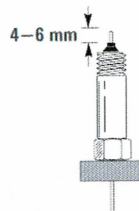
- 10.7.4 Carefully lift the ALS injection tower, and unplug the electrical connection from the bottom of the unit.
- 10.7.5 Place the ALS injection tower in a location where it will not be knocked over.
- 10.7.6 Remove the septum retainer nut and septum. Replace septum after every 50 – 100 injections.
- 10.7.7 Slide the retaining mechanism for the septum assembly toward you (counterclockwise) to release the lock.
- 10.7.8 Wear gloves to avoid contamination of the inlet liner.
- 10.7.9 Lift the septum assembly straight up to avoid chipping or breaking the liner.
- 10.7.10 Lift the inlet liner straight up to remove it from the inlet. A pair of tweezers may be helpful if the liner o-ring is stuck to the inlet body.



- 10.7.11 Replace the liner if contaminated or damaged.
- 10.7.12 Inspect the o-ring for wear. If necessary, replace the o-ring. The o-ring should be installed on the inlet liner approximately 3-4 mm from the top of the liner.

- 10.7.13 Carefully lower the inlet liner back into the inlet body.
 - 10.7.14 Line up the tab on the bottom of the septum assembly with the slot on the septum assembly and push down to connect. Slide the retaining mechanism away from you (clockwise) to engage the lock.
 - 10.7.15 Tighten the septum retainer nut until the C-ring is about 1 mm above the nut. Do not overtighten the septum retainer nut.
- 10.8 To change the column:
- 10.8.1 Turn off heating to the inlet, oven, and detector:
 - 10.8.1.1 On the instrument keypad, select Front Inlet or Back Inlet depending on which one you are using.
 - 10.8.1.2 Use the up/down arrows to navigate to Temperature.
 - 10.8.1.3 Press the Off button.
 - 10.8.1.4 Select Oven.
 - 10.8.1.5 Use the up/down arrows to navigate to Temperature.
 - 10.8.1.6 Press the Off button.
 - 10.8.1.7 Select Back Detector.
 - 10.8.1.8 Use the up/down arrows to navigate to Temperature.
 - 10.8.1.9 Press the Off button.
 - 10.8.2 Turn off flow to the inlet and detector.
 - 10.8.2.1 On the instrument keypad, select Front Inlet or Back Inlet depending on which one you are using.
 - 10.8.2.2 Use the up/down arrows to navigate to Pressure.
 - 10.8.2.3 Press the Off button.
 - 10.8.3 When inlet, oven, and detector temperatures are < 50 °C, proceed to the next step.

- 10.8.4 Open the door to the oven compartment by pressing the button at the bottom right side of the door.
- 10.8.5 Remove the old column using a wrench to loosen the column nuts.
- 10.8.6 Remove column nuts and ferrules from the old column.
- 10.8.7 Discard the used ferrules, and cap the column using an old septum.
- 10.8.8 Place the new column in the oven on the hanger.
- 10.8.9 Remove the septa used to plug the column inlet and outlet.
- 10.8.10 Place a septum, column nut, and ferrule on the inlet side of the column.
- 10.8.11 Score the column about an inch from the inlet end using a glass scribing tool.
- 10.8.12 Break off the end of the column by gently pulling and bending near the score.
- 10.8.13 Inspect the end with a magnifying loupe to ensure there are no burrs or jagged edges. If the cut is not clean and square, cut again.
- 10.8.14 Wipe the outside of the column with a tissue dampened with isopropanol to remove fingerprints and dust.
- 10.8.15 Position the column so it extends 4 – 6 mm above the end of the ferrule. Slide the septum up the column to hold the column nut at this position.
- 10.8.16 Thread the column nut into the inlet, but do not tighten.
- 10.8.17 Adjust the column position so that the septum contacts the bottom of the column nut.



- 10.8.18 Finger-tighten the column nut until it begins to grip the column.
- 10.8.19 Tighten the column nut an additional $\frac{1}{4}$ to $\frac{1}{2}$ turn with a wrench so that the column cannot be pulled from the fitting with gentle pressure.

10.8.20 Configure the new column:

10.8.20.1 On the instrument keypad, select Config.

10.8.20.2 Use the up/down arrows to navigate to Column #, and press Enter.

10.8.20.3 Press 1.

10.8.20.4 Use the up/down arrows to navigate to Length.

10.8.20.5 Enter the column length in m, and press Enter.

10.8.20.6 Use the up/down arrows to navigate to Diameter.

10.8.20.7 Enter the column internal diameter in μm , and press Enter.

10.8.20.8 Use the up/down arrows to navigate to Film Thickness.

10.8.20.9 Enter the column film thickness in μm , and press Enter.

10.8.20.10 Use the up/down arrows to navigate to Inlet.

10.8.20.11 Press the Mode key.

10.8.20.12 Use the up/down arrows to select the Inlet that the column is connected to, and press Enter.

10.8.20.13 Use the up/down arrows to navigate to Outlet.

10.8.20.14 Press the Mode key.

10.8.20.15 Use the up/down arrows to select the Outlet that the column is connected to, and press Enter.

10.8.20.16 Use the up/down arrows to navigate to Thermal Zone.

10.8.20.17 Press the Mode key.

10.8.20.18 Use the up/down arrows to select GC Oven, and press Enter.

10.8.21 Place a septum, column nut, and ferrule on the outlet side of the column.

10.8.22 Score the column about an inch from the end using a glass scribing tool.

10.8.23 Break off the end of the column by gently pulling and bending near the score.

- 10.8.24 Inspect the end with a magnifying loupe to ensure there are no burrs or jagged edges. If the cut is not clean and square, cut again.
- 10.8.25 Adjust the column so that the end extends about 7 cm beyond the ferrule.
- 10.8.26 Wipe the outside of the column with a tissue dampened with isopropanol to remove fingerprints and dust.
- 10.8.27 Initiate column flow:
 - 10.8.27.1 On the instrument keypad, select Coll.
 - 10.8.27.2 Use the up/down arrows to navigate to Velocity.
 - 10.8.27.3 Type 25, and press Enter.
- 10.8.28 Gently insert the outlet side of the column into the detector until it bottoms; do not attempt to force it further.
- 10.8.29 Gently slide the septum upward until it contacts the column nut.
- 10.8.30 Tighten the column nut until it just begins to grab the column.
- 10.8.31 Loosen the nut very slightly, and withdraw the column about 1 mm.
- 10.8.32 Tighten the nut an additional ¼ turn with a wrench to secure the column.

11.0 Instrument Shutdown

- 11.1 Shutdown for less than one week:
 - 11.1.1 Turn off all gasses, except for helium (carrier gas), at their sources.
 - 11.1.2 Reduce the temperature of the inlet, detector, and oven:
 - 11.1.2.1 On the instrument keypad, select Front Inlet or Back Inlet depending on which one you are using.
 - 11.1.2.2 Use the up/down arrows to navigate to Temperature.
 - 11.1.2.3 Type 150, and press Enter.
 - 11.1.2.4 Select Back Detector.
 - 11.1.2.5 Use the up/down arrows to navigate to Temperature.

11.1.2.6 Type 150, and press Enter.

11.1.2.7 Select Oven.

11.1.2.8 Use the up/down arrows to navigate to Temperature.

11.1.2.9 Type 100, and press Enter.

11.1.3 Set the column flow:

11.1.3.1 Press the Col1 button.

11.1.3.2 Use the up/down arrows to navigate to Velocity.

11.1.3.3 Type 25, and press Enter.

11.1.3.4 Press the Front Inlet or Back Inlet button depending on which is in use.

11.1.3.5 Use the up/down arrows to navigate to Split Ratio.

11.1.3.6 Type 2, and press Enter.

11.1.3.7 Use the up/down arrows to navigate to Septum Purge.

11.1.3.8 Press the Off button.

11.1.3.9 Press the Back Detector button.

11.1.3.10 Use the up/down arrows to navigate to Makeup.

11.1.3.11 Press the Off button.

11.1.3.12 Use the up/down arrows to navigate to Flame.

11.1.3.13 Press the Off button.

11.1.4 Put Headspace to Sleep

11.1.4.1 On the headspace keypad, select Method.

11.1.4.2 Use the up/down arrows to navigate to Sleep.

11.1.4.3 Press Enter.

11.1.4.4 Press Yes.

11.2 Shutdown for more than one week:

11.2.1 Turn off heat to the inlet, oven, and detector:

11.2.1.1 On the instrument keypad, select Front Inlet or Back Inlet depending on which one you are using.

11.2.1.2 Use the up/down arrows to navigate to Temperature.

11.2.1.3 Press Off.

11.2.1.4 Select Back Detector.

11.2.1.5 Use the up/down arrows to navigate to Temperature.

11.2.1.6 Press Off.

11.2.1.7 Select Oven.

11.2.1.8 Use the up/down arrows to navigate to Temperature.

11.2.1.9 Press Off.

11.2.2 When the GC is cool, shut off all gasses at their sources.

11.2.3 Remove the column from the oven and cap both ends with a used septum to keep out contaminants.

11.2.4 Turn off power to the GC and Headspace.

12.0 Preventative Maintenance

12.1 The most common GC maintenance procedures are listed here. For less common maintenance procedures, consult the instrument manual.

12.2 Septum Seat Cleaning

12.2.1 Reduce the temperature of the inlet and oven:

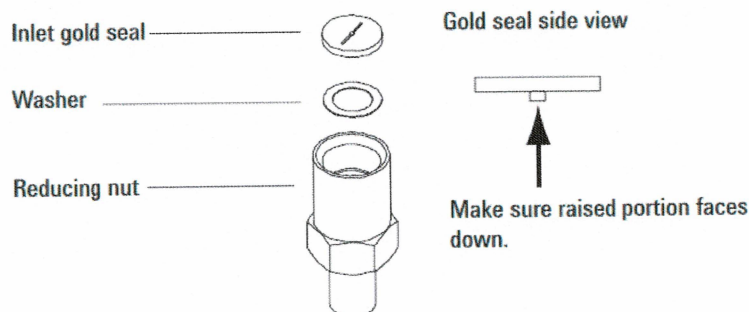
12.2.1.1 On the instrument keypad, select Front Inlet or Back Inlet depending on which one you are using.

12.2.1.2 Use the up/down arrows to navigate to Temperature.

12.2.1.3 Press Off.

- 12.2.1.4 Select Oven.
- 12.2.1.5 Use the up/down arrows to navigate to Temperature.
- 12.2.1.6 Press Off.
- 12.2.2 Slide the locking tab of the septum assembly forward (counterclockwise).
- 12.2.3 Remove the septum retainer nut.
- 12.2.4 Remove the septum from the septum assembly.
- 12.2.5 Lift the septum assembly straight up to avoid chipping or breaking the liner.
- 12.2.6 Scrub the residue from the retainer nut and septum holder with a small piece of rolled up steel wool and tweezers. Do not do this over the inlet to avoid any residue falling into the inlet.
- 12.2.7 Use compressed air to blow away the pieces of steel wool and septum.
- 12.2.8 Line up the tab on the bottom of the septum assembly with the slot on the septum assembly and push down to connect. Slide the retaining mechanism backward (clockwise).
- 12.2.9 Firmly press the new septum into the fitting.
- 12.2.10 Re-install the septum retainer nut and septum. Do not overtighten the septum retainer nut. Tighten the nut until the C-ring is about 1 mm above the nut.
- 12.3 Gold Seal Replacement
 - 12.3.1 Reduce the temperature of the inlet and oven:
 - 12.3.1.1 On the instrument keypad, select Front Inlet or Back Inlet depending on which one you are using.
 - 12.3.1.2 Use the up/down arrows to navigate to Temperature.
 - 12.3.1.3 Press Off.
 - 12.3.1.4 Select Oven.
 - 12.3.1.5 Use the up/down arrows to navigate to Temperature.

- 12.3.1.6 Press Off.
- 12.3.2 Turn off heating and flow to the inlet as outlined in Section 10.7.
- 12.3.3 Remove the inlet liner as outlined in Section 10.7.
- 12.3.4 Remove the column from the inlet, and cap the open end of the column using an old septum.
- 12.3.5 Remove the insulation cup around the base of the inlet using a Torx screwdriver.
- 12.3.6 Loosen and remove the reducing nut at the bottom of the inlet.
- 12.3.7 Remove the washer and seal inside the reducing nut.
- 12.3.8 Put on gloves to protect the new gold seal and washer from contamination.
- 12.3.9 Put a new washer in the reducing nut, and place the new gold seal on top of it with the raised portion facing down.



- 12.3.10 Replace the reducing nut and tighten securely with a wrench.
- 12.3.11 Replace the inlet liner as outlined in Section 10.7.
- 12.3.12 Install the column as outlined in Section 10.8.
- 12.3.13 Install the insulation cup.

13.0 Consumable Parts

- 13.1 Other part numbers may be used provided they are equivalent
- 13.2 Inlet Septa (11 mm) Restek Part # 23864
- 13.3 Inlet Liner (2 mm for headspace) Restek Part # 23313

- 13.4 Inlet Liner (4 mm for direct injection) Restek Part # 23300
- 13.5 Gold Inlet Seal Restek Part # 21317
- 13.6 Graphite Ferrule (for 0.53 mm column) Restek Part # 20252
- 13.7 Graphite Ferrule (for 0.32 mm column) Restek Part # 21007
- 13.8 Graphite Ferrule (for 0.25 mm column) Restek Part # 20250
- 13.9 Syringe (5 μ L) Restek Part # 24781
- 13.10 Syringe (10 μ L) Restek Part # 24785
- 13.11 Transfer Line (0.53 mm) Restek Part # 10054
- 13.12 UltiMetal Ferrule for Transfer Line Agilent Part # G3188-27503
- 13.13 Polyimide Ferrule for Transfer Line Restek Part # 20141

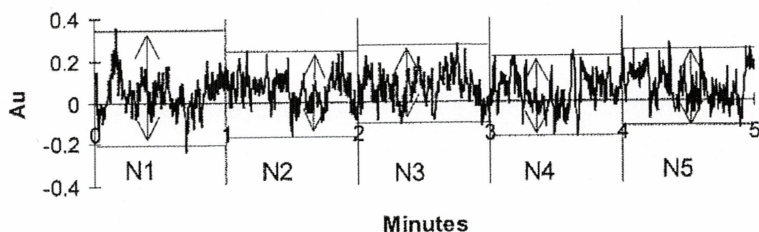
14.0 Performance Qualification

- 14.1 The following tests are recommended for annual performance qualification (PQ) of GC systems. Installation qualification (IQ) and operational qualification (OQ) are typically performed by the instrument vendor.
- 14.2 Supplied needed
 - 14.2.1 G1 column (30 m length and 0.32 mm id)
 - 14.2.2 Toluene
 - 14.2.3 Methanol
 - 14.2.4 Dimethylsulfoxide
 - 14.2.5 Ruler with mm gradations
 - 14.2.6 Calibrated temperature sensor
- 14.3 Oven temperature accuracy
 - 14.3.1 Place a calibrated thermometer in the column oven with the probe near the instruments oven thermocouple.

- 14.3.2 Set the oven to each of the following temperatures, allowing the reading to stabilize prior to recording the result: 50, 100, 150, 200, and 250 °C.
- 14.3.3 Acceptance criteria: nominal temperature ± 3 °C.
- 14.4 Injector Precision, Carrier Flow Precision, Carryover, Noise, and Drift for direct injection
- 14.4.1 Perform the test for each injector that will be used (e.g. front injector and back injector).
- 14.4.2 Prepare a solution of 10 µg/mL toluene in methanol.
- 14.4.2.1 Transfer 577 µL of toluene to a 50-mL volumetric flask containing about 25 mL of methanol, and dilute to volume with methanol. This solution contains 10,000 µg/mL toluene.
- 14.4.2.2 Transfer 1.0 mL of the resulting solution to a 50-mL volumetric flask, and dilute to volume with methanol. This solution contains 200 µg/mL toluene.
- 14.4.2.3 Transfer 5.0 mL of the resulting solution to a 100-mL volumetric flask, and dilute to volume with methanol. This solution contains 10 µg/mL toluene.
- 14.4.3 Install a G1 column with dimensions of 30 m length x 0.32 mm I.D.
- 14.4.4 Set the following GC parameters:
- 14.4.4.1 Injector temperature 250 °C
- 14.4.4.2 Septum Purge Flow 3 mL/min
- 14.4.4.3 Column Pressure 10 psi
- 14.4.4.4 Split Flow 50 mL/min
- 14.4.4.5 Oven Temperature 100 °C (isocratic)
- 14.4.4.6 Run Time 10 min
- 14.4.4.7 Detector Temperature 300 °C
- 14.4.4.8 Air Flow 400 mL/min

- 14.4.4.9 Fuel Flow 40 mL/min.
- 14.4.4.10 Makeup Flow 25 mL/min (constant col+makeup)
- 14.4.5 Ignite the detector.
- 14.4.6 Condition the column for at least 30 min.
- 14.4.7 Perform eight 0.5 μ L injections of the toluene solution followed by a blank (methanol) injection and then a null injection (set Injection Source to No Injection in the sequence table).
- 14.4.8 Record the peak areas for each injection.
- 14.4.9 Injector precision acceptance criteria: NMT 2.0% RSD for six consecutive replicate injections of toluene solution (choose six of eight).
- 14.4.10 Carrier flow precision acceptance criteria: NMT 0.1 min difference in retention time between the six consecutive replicate injections of toluene solution (choose six of eight).
- 14.4.11 Carryover acceptance criteria is NMT 0.1% for the blank injection.
- 14.4.12 Calculate the system noise using the null injection.
- 14.4.13 Measure the noise level for each 2 min interval as shown below.
- 14.4.14 Calculate the average noise level for the five 2 min intervals.

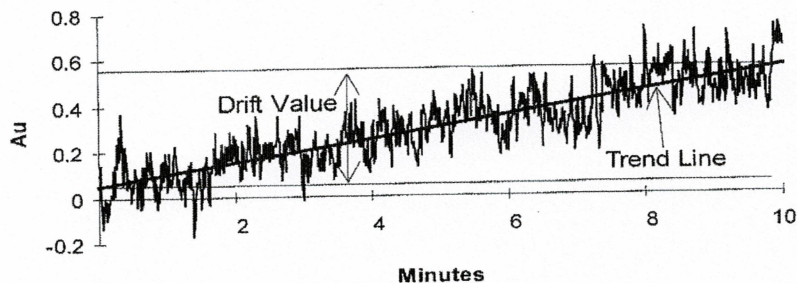
Noise calculation example



- 14.4.15 Noise acceptance criteria is NMT 0.1 pA.
- 14.4.16 Calculate the system drift using all 10 min of data from the null injection.
- 14.4.17 Multiply the drift value by 6 to obtain the drift per hour.

14.4.18 Drift acceptance criteria is NMT 0.5 (pA/hr).

Drift calculation example



14.4.19 Repeat the Injector Precision and Carryover test for direct injection in Splitless mode with Purge Flow to Split Vent set to 40 mL/min at 0.5 min.

14.5 Injector Precision, Carrier Flow Precision, and Carryover for headspace injection

14.5.1 Prepare a solution of 10 µg/mL toluene in water.

14.5.1.1 Transfer 577 µL of toluene to a 50-mL volumetric flask containing about 25 mL of DMSO, and dilute to volume with DMSO. This solution contains 10,000 µg/mL toluene.

14.5.1.2 Transfer 2.0 mL of the resulting solution to a 100-mL volumetric flask, and dilute to volume with water. This solution contains 200 µg/mL toluene.

14.5.1.3 Transfer 5.0 mL of the resulting solution to a 100-mL volumetric flask, and dilute to volume with water. This solution contains 10 µg/mL toluene.

14.5.2 Install a G1 column with dimensions of 30 m length x 0.32 mm I.D.

14.5.3 Set the following GC parameters:

14.5.3.1 Injector temperature 250 °C

14.5.3.2 Septum Purge Flow 3 mL/min

14.5.3.3 Column Pressure 10 psi

14.5.3.4 Oven Temperature 100 °C (isocratic)

- 14.5.3.5 Run Time 30 min
- 14.5.3.6 Detector Temperature 300 °C
- 14.5.3.7 Air Flow 400 mL/min
- 14.5.3.8 Fuel Flow 40 mL/min.
- 14.5.3.9 Makeup Flow 25 mL/min (constant col+makeup)
- 14.5.4 Set the following headspace parameters
 - 14.5.4.1 Oven temp 80 °C.
 - 14.5.4.2 Loop temp 90 °C.
 - 14.5.4.3 Transfer line temp 105 °C.
 - 14.5.4.4 Equilibration time 30 min
 - 14.5.4.5 Injection time 1 minute
 - 14.5.4.6 GC cycle time 30 min
 - 14.5.4.7 Fill pressure 15 psi.
 - 14.5.4.8 Loop ramp 40 psi/min
 - 14.5.4.9 Loop final pressure 10 psi.
 - 14.5.4.10 Loop equilibration time 0.2 minutes.
- 14.5.5 Ignite the detector.
- 14.5.6 Condition the column for at least 30 min.
- 14.5.7 Perform eight injections of the toluene solution followed by a blank (DI water) injection.
- 14.5.8 Record the peak areas for each injection.
- 14.5.9 Evaluate the Injector Precision, Carrier Flow Precision, and Carryover as outlined in 14.4.

15.0 Revision History

Revision	Date	Description of Changes	CCR #	By
0	02/11/20	New	N/A	S. Sassman
1	12/20/22	Added reference and section for HPLC/ GC Checklist	CC-22-0476	J. Sassman