# Chapter 6 **Operation**

This chapter outlines the checks and cleaning procedures of the Q Exactive GC or Exactive GC system that should be performed every day to ensure the proper operation of your system.

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# **Safety Guidelines for Operation**

When operating the Q Exactive or Exactive GC system, pay attention to the following general safety guidelines.

- **WARNING** Suffocation Hazard. Accumulation of nitrogen gas could displace sufficient oxygen to suffocate personnel in the laboratory. Ensure that the laboratory is well ventilated. Local regulations may require a risk assessment for the workplace.
- **A** CAUTION Hazardous Chemicals. Samples and solvents might contain toxic, carcinogenic, mutagenic, or corrosive/irritant chemicals. Avoid exposure to potentially harmful materials. Always wear protective clothing, gloves, and safety glasses when you handle solvents or samples. Also contain waste streams and use proper ventilation. Refer to your supplier's Material Safety Data Sheet (MSDS) for proper handling of a particular compound.
- **A CAUTION** Hot Surface. The external surface of the ion source housing can become hot enough to burn your skin. Avoid touching the ion source housing when the mass spectrometer is in operation. Allow the ion source to cool to room temperature (approximately 60 minutes) before touching the ExtractaBrite removable ion source.
- ★ CAUTION
  Hot Surface. Touching hot parts of the ion source interface might cause severe burns. During operation of the mass spectrometer, the ExtractaBrite removable ion source might reach temperatures of up to 350 °C, while the GC column transfer line might reach temperatures of up to 400 °C. To let the ion source interface cool down, set the ion source and transfer line temperatures to 25 °C. Wait until the ion source has cooled down to room temperature (for approximately 60 minutes) before you begin working on it. Do not leave the instrument unattended when the covers and front door are removed, exposing the hot source parts. Hot ion source parts might ignite combustible material. Keep combustible materials away from the ion source mount.

## **Before Operating the System**

Every day before starting analyses, verify that the instrument is ready for operation by doing the following:

- Checking the Forepump
- Checking the System Vacuum Levels
- Checking the Disk Space on the Data System
- Checking the Mass Accuracy of the Instrument

**NOTICE** Accurate results can be obtained only if the system is properly calibrated. ▲

## **Checking the Forepump**

#### \* To check the forepump before use

Make sure that the following conditions are met:

- The forepump is filled with oil.
- It is connected to the power supply.
- The gas ballast is shut.

## **Checking the Nitrogen Supply**

Check the nitrogen supply on the regulator of the nitrogen gas tank or tanks. Make sure that you have sufficient gas for your analysis. Based on 24 hour per day operation, typical nitrogen consumption is 1 L (0.035 ft<sup>3</sup>) per day. If necessary, replace the tank. Verify that the pressure of nitrogen reaching the mass spectrometer is at 800 ± 30 kPa (8 ± 0.3 bar, 116 ± 4 psi). If necessary, adjust the pressure with the tank pressure regulator.

Continue with "Checking the System Vacuum Levels".

## **Checking the System Vacuum Levels**

For proper performance, your Q Exactive GC or Exactive GC system must operate at the acceptable vacuum levels. Operating the system at poor vacuum levels can cause reduced sensitivity and tuning problems. Check your system for air leaks by checking the system vacuum levels before you begin your first acquisition.



You can check the current pressure values in the Vacuum / Bakeout window of the Tune software. To display the Tune software window, choose **Start > Programs > Thermo Exactive Series > Tune**. The Instrument status LED in the toolbar (see image in margin) turns green when the vacuum levels are sufficient for operating your Exactive Series system. If the LED is not green, you might have an air leak; see "Vacuum Leak" on page 7-5 for instructions.

Continue with "Checking the Disk Space on the Data System".

## Checking the Disk Space on the Data System

Periodically verify that your hard disk drive has enough free space for data acquisition. The amount of available disk space is shown in the Disk Space dialog box.

- To determine the amount of available disk space
- From the Home Page window (which is available by choosing Start > Programs > Thermo Xcalibur > Xcalibur), choose Actions > Check Disk Space to open the Disk Space dialog box. The Disk Space dialog box lists the following:
  - Current drive and directory (for example, C:\Xcalibur\system\programs)
  - Number of Mb that are available (free) on the current drive
  - Percentage of the current drive that is available
  - Total capacity of the current drive
- 2. To select another disk drive so that you can determine its disk space, click **Directory**.
- 3. When you have completed this procedure, choose **OK** to close the dialog box.

If necessary, you can free space on the hard disk by deleting obsolete files and by moving files from the hard disk drive to a backup medium. First, copy files to the backup medium. After you have copied the files, you can delete them from the hard disk.

Continue with "Checking the Mass Accuracy of the Instrument".

## **Checking the Mass Accuracy of the Instrument**

Thermo Fisher Scientific recommends checking the mass accuracy before you start working. Follow the procedure described in the QuickStart Guide for your instrument. If the instrument indicates that the tune parameters and calibration parameters are not optimal, Thermo Fisher Scientific recommends tuning and calibrating the Q Exactive GC or Exactive GC mass spectrometer. Follow the procedure described in the QuickStart Guide or Software Manual.

**NOTICE** *Calibration parameters* are instrument parameters that affect the mass accuracy and resolution. *Tune parameters* are instrument parameters that affect the intensity of the ion signal. ▲

You need to optimize the tune parameters (or change the Tune Method) whenever you change the type of experiment. Refer to the QuickStart Guide for a procedure for optimizing the tune parameters for your experiment.

# Setting the System in Standby Condition

The Q Exactive GC or Exactive GC system should not be shut down completely if you are not going to use it for a short period of time, such as overnight or over the weekend. In that case, you can leave the system in Standby condition.

**NOTICE** Thermo Fisher Scientific recommends leaving the mass spectrometer in Standby overnight to provide the best mass accuracy next day. ▲

#### To place the Q Exactive GC or Exactive GC system in the Standby condition

1. Wait until data acquisition, if any, is complete.



- 2. In the Tune software window, click the **On/Standby** button to put the instrument in Standby condition. The System LED on the front panel of the mass spectrometer turns yellow when the system is in Standby condition.
- 3. Leave the GC power On.
- 4. Leave the autosampler power On.
- 5. Leave the data system power On.
- 6. Leave the Q Exactive GC or Exactive GC main power circuit breaker switch in the On position.

## **Shutting Down the System**

The Q Exactive GC or Exactive GC system does not need to be shut down completely if you are not going to use it for a short period of time, such as overnight or over a weekend. See "Setting the System in Standby Condition" on page 6-6. This section describes how to shut down the system for a maintenance or service procedure.

**NOTICE** Although removing power abruptly by placing the *main power circuit breaker switch* (see Figure 3-5 on page 3-9) in the Off (O) position will not harm any component within the system, this not the recommended shutdown procedure to follow. ▲

#### To shut down the Q Exactive GC or Exactive GC system

- 1. Wait until data acquisition, if any, is complete.
- 2. Set heated zones to room temperature on the GC transfer line and the MS transfer line.



- 3. In the Tune software window, click the **On/Standby** button to put the instrument in Off condition. (See image in margin.) All high voltages are shut off, as is the C-Trap and HCD gas.
- 4. Place the electronics service switch, located on the power panel (See Figure 3-5 on page 3-9.), in the Service Mode position.
- 5. Put the main power circuit breaker switch of the mass spectrometer in the Off position.
- 6. Shut down the TRACE 1310 GC. See the GC user documentation for instructions.

**WARNING** High Voltage. Hazardous electric voltage capable of causing an electric shock is used in the instrument. To ensure that the instrument is free from all electric current, always disconnect the power cords of *mass spectrometer and forepump* before attempting any type of maintenance.

If you are planning to perform routine or preventive system maintenance on the mass spectrometer only, you do not need to turn off the autosampler or data system. In this case, the shutdown procedure is completed. However, if you do not plan to operate your system for an extended period of time, you might want to turn off the autosampler and data system. **NOTICE** An instrument that is shut down still consumes nitrogen because the vent valve is connected to the nitrogen supply of the laboratory. Keeping the nitrogen flow on prevents humidity from contaminating the vacuum system of the mass spectrometer. You may however switch off the nitrogen if your supply is limited. ▲

## Starting Up the System after a Shutdown

To start up the Q Exactive GC or Exactive GC mass spectrometer after it has been shut down (and vented), you need to do the following:

- 1. Start up the instrument
- 2. Set up conditions for operation

## **Starting Up the Instrument**

#### \* To start up the Q Exactive GC or Exactive GC system

- 1. Start up the GC and autosampler as is described in the manual that came with the GC and autosampler.
- 2. Switch on data system and monitor as described in the manuals that came with them. Wait until the operating system of the computer is completely loaded.
- 3. To start the Tune software on the data system computer, choose **Start > Programs > Thermo Exactive Series > Tune**.

**NOTICE** The data system must be running before you start up the instrument. The instrument will not operate until software is received from the data system. ▲

- 4. Turn on the nitrogen flow at the tank, if it is off.
- 5. Make sure that the main power circuit breaker switch is in the Off (O) position and that the electronics service switch is in the Service Mode position.
- Place the main power circuit breaker switch in the On (|) position.
   When you place the main power circuit breaker switch in the On (|) position, the forepump and the turbomolecular pumps are started. All LEDs on the mass spectrometer front panel are off.
- 7. Set the power to the GC to On after the mass spectrometer. This allows the GC to properly identify the auxiliary temperature module controlling the GC transfer line.

When the vacuum system is switched on, the following occurs:

a. After the main switch is switched On, the pumps of the mass spectrometer are run up. To monitor the vacuum readings in the Tune software, it is necessary to switch on the electronics switch, too. The Pirani gauge (see "Vacuum Gauges" on page 3-13) monitors the pressure at the forepump. Within a short time, a significant pressure decrease must be observed. The quality of the vacuum can be estimated by means of the rotation speed of the TMPs (for example, 80% after 15 minutes).

b. If the working pressure is not reached after a preset time, the complete system is switched off.

**NOTICE** The vacuum control board triggers an alert in the Tune software when a vacuum failure has occurred. ▲

c. The Ion Gauge 1 (IKR 251) is switched on only after the source TMP has exceeded 90% of its maximum rotation speed for five minutes. To extend its life time, this ion gauge is switched off automatically after 30 minutes.

**NOTICE** For diagnostic purposes, the ion gauge can be switched on manually in the instrument status view of the Tune software. ▲

If the pressure exceeds 1E-4 mbar for more than 10 seconds, the ion gauge is switched off. After five minutes, the ion gauge is switched on again. After three failed attempts, it is only possible to switch on Ion Gauge 1 manually in the Tune software.

d. The Ion Gauge 2 (IKR 270) is switched on only after both TMPs have exceeded 90% of their maximum rotation speed for five minutes. It is then used to monitor the vacuum in the Orbitrap analyzer.

If the pressure exceeds 1E-4 mbar for more than 10 seconds, the ion gauge is switched off. After five minutes, the ion gauge is switched on again. After three failed attempts, it is only possible to switch on Ion Gauge 2 manually in the Tune software.

- e. The Vacuum LED on the system panel turns green when all the following conditions are met:
  - In the Tune software, all LEDs are green. (The Ion Gauge 1 is allowed to be off).
  - Analyzer temperature is below 45 °C.
  - Both TMP frequencies have exceeded 90% of their maximum rotation speed.
- f. When the vacuum measured by the Ion Gauge 2 is better than 1E-8 mbar, the power supplies of the high voltage electronics and the capillary heater are switched on.

**NOTICE** If both ion gauges are defective, it is not possible to switch on the RF voltage and the high voltages. ▲

- 8. Allow the mass spectrometer to pump down for 5 minutes.
- 9. Place the electronics service switch in the Operating Mode position. See Figure 3-5 on page 3-9. When the electronics service switch is in the Operating Mode position, the following occurs:
  - a. Power is provided to all electronic boards. (The main RF voltage and transfer multipole RF voltage remain off.)
  - b. The internal computer reboots. After several seconds, the Status LED on the front panel turns yellow to indicate that the data system has started to establish a communication link.
  - c. After several more seconds, the Status LED turns green to indicate that the data system has established a communication link. Software for the operation of the instrument is then transferred from the data system to the instrument.
  - d. After approximately 3 minutes, the System LED turns yellow to indicate that the software transfer from the data system is complete.

Start the GC or autosampler (if present) it as is described in the manual that came with the GC or autosampler. After the GC and autosampler are powered on, go to "Setting Up Conditions for Operation" below.

## **Setting Up Conditions for Operation**

#### To set up your Q Exactive GC or Exactive GC mass spectrometer for operation

- Operation of the system with excessive air and water in the vacuum manifold can cause reduced sensitivity and tuning problems. Before you begin data acquisition with your Q Exactive GC or Exactive GC system, you need to bake out the system for at least twelve hours plus a cooling period of three hours. See "Baking Out the System" on page 8-11.
- After having performed a system bakeout, make sure in the Vacuum/Bakeout window of the Tune software that the UHV pressure is < 1E-8 mbar. Also make sure that the FV pressure is in the < 6E-2 mbar region. Compare the values of the other parameters in the instrument status window with values that you recorded previously.
- 3. Make sure that the gas pressure is within the operational limits:

Nitrogen: 800 ± 30 kPa (8 ± 0.3 bar, 116 ± 4 psi)

See also "Before Operating the System" on page 6-3 for additional information.

#### Introducing Samples into the Q Exactive GC or Exactive GC Mass Spectrometer

The Q Exactive GC or Exactive GC mass spectrometer, the GC, and the autosampler (if present) have been installed by a Thermo Fisher Scientific field service engineer.

The QuickStart Guide for your instrument gives an introduction on setting up and using the Q Exactive GC or Exactive GC mass spectrometer.

Consider the following safety guideline when introducing sample into the Q Exactive GC or Exactive GC mass spectrometer.

**A CAUTION Hazardous Chemicals.** Samples and solvents might contain toxic, carcinogenic, mutagenic, or corrosive/irritant chemicals. Avoid exposure to potentially harmful materials. Always wear protective clothing, gloves, and safety glasses when you handle solvents or samples. Also contain waste streams and use proper ventilation. Refer to your supplier's Material Safety Data Sheet (MSDS) for proper handling of a particular compound.

#### **Performing Mass Spectrum Measurements**

The QuickStart Guide gives an introduction on performing mass spectrum measurements with the Q Exactive GC or Exactive GC mass spectrometer.

Consider the following safety guidelines when performing measurements with the Q Exactive GC or Exactive GC mass spectrometer.

**WARNING** Suffocation Hazard. Accumulation of nitrogen gas could displace sufficient oxygen to suffocate personnel in the laboratory. Ensure that the laboratory is well ventilated. Local regulations may require a risk assessment for the workplace.

## **Resetting the System**

If communication between the mass spectrometer and data system computer is lost, it may be necessary to reset the system using the Reset button of the Q Exactive GC or Exactive GC mass spectrometer.

#### \* To reset the system

- 1. Ensure that mass spectrometer and data system computer are both powered on and the mass spectrometer is in Standby condition. See page 6-6 for instructions about placing the instrument to Standby condition.
- 2. To reset the mass spectrometer, press the Reset button located on the power panel. See Figure 3-5 on page 3-9. Make sure the Status LED is extinguished before releasing the Reset button. When you press the Reset button, the following occurs:
  - a. An interruption of the embedded computer causes the CPU to reboot. All LEDs on the front panel are off except the Power LED.
  - b. After several seconds, the Status LED turns yellow to indicate that the data system and the instrument are starting to establish a communication link.
  - c. After several more seconds, the Status LED turns green to indicate that the data system and the instrument have established a communication link. Software for the operation of the instrument is then transferred from the data system to the instrument.

After 3 minutes, the software transfer is complete. The System LED turns either green to indicate that the instrument is functional and the high voltages are on, or yellow to indicate that the instrument is functional and it is in Standby condition.

**NOTICE** If resetting the system does not resolve the failure: Put the electronics switch in the Service Mode position for about one minute. Afterwards, place the electronics service switch back in the Operating Mode position. ▲

# **Installing the Column**

Check the column for leaks and perform a column evaluation if needed before inserting it into the MS transfer line.

#### \* To check the column for leaks

- On the TRACE 1310, select the Leak Check icon in the Maintenance menu. Otherwise, perform the leak check through the Chromatography Data System. Refer to the *TRACE 1300 and TRACE 1310 Series GC User Guide* for instructions.
- 2. Start the leak check:

The split and purge valves of the selected channel are automatically closed, and the channel is pressurized with carrier gas to the leak check set point.

The system monitors the pressure for one minute. If the pressure does not drop more than the maximum allowed sensitivity value, then the leak check will pass. If the leak check does not pass, use the leak detector to find and fix any leaks.

**NOTICE** Leaks can be caused by not tightening the fitting on the column flowmeter connector. Check the fitting before looking for the leak elsewhere. ▲

**NOTICE INSTRUMENT DAMAGE:** Do not allow the column flowmeter connector to exceed 80 °C (176 °F). Otherwise, it will melt and damage the instrument. ▲

- 3. Repeat the leak check until no leaks are indicated.
- 4. Calibrate the carrier gas flow (column evaluation):

a. Carefully push the capillary column end into the flowmeter section of the column flowmeter connector.



Figure 6-1. Connecting the Column to the Flowmeter

- b. Connect the flowmeter to the dedicated fitting on the column flowmeter connector.
- c. If you have a TRACE 1310, select the Back or Front Column icon in the Configuration menu. Otherwise, perform the column evaluation through the Chromatography Data System. See the TRACE 1300 and TRACE 1310 User Guide for instructions.
- d. Select Column and input the column's physical characteristics.
- e. If a pre-/post column is present, set the length and nominal internal diameter of the pre-/post column in the same valid ranges for the column. The following two lines are added to the menu.

**NOTICE** For the most reproducible results, you should conduct a more detailed column evaluation. However, the following steps, while recommended, are not required. ▲

- f. Start the column evaluation. According to the physical characteristics of the column, the system calculates and displays the relevant column K-factor. At the end of the routine, a message will indicate that the evaluation was successful.
- g. Expect a K-factor of approximately 0.7–0.9 for a 15 m, 0.25 mm i.d. column (1.3–2.0 for a 30 m, 0.25 mm i.d. column). If the column does not report a K-factor within this range or within 0.1 units of the previous stored value, check for a leak or broken column using the leak detector. The K-factor is a

measured resistance for the column. A K-factor that is too low may indicate a leak in the system, while a K-factor that is too high may indicate a blockage.

Fix any issues found and rerun column evaluation until an appropriate K-factor is achieved.

- 5. Disconnect the column flowmeter.
- 6. Disconnect the column from the column flowmeter connector.
- 7. Remove the clear plastic component, including its fittings, from the oven and set them aside.
- \* To install the column in the Q Exactive GC or Exactive GC system
- 1. In Tune, place the instrument in the Off state.
- 2. Cool the oven, transfer line and source:
  - a. On the GC, set the Oven to **Off**.
  - b. On the GC, in the Auxiliary Temperature Control, set Transfer Line 1 and Transfer Line 2 to **Off**.
  - c. In Tune Instrument Control EI/CI source, set the:
    - i. MS transfer line temp to 40 °C.
    - ii. Ion source temp to 175 °C (to avoid excessive oxidation of source parts or contamination from the source plug).
- 3. Using the source removal tool and vacuum interlock, remove the ion source. (See "Removing the Ion Source Cartridge from the Q Exactive GC or Exactive GC System" on page 8-15 for correct use of the vacuum interlock and source insertion/removal tool.)

4. Place the ion source cartridge on the source holder and set aside.



Figure 6-2. Storing the Ion Source Cartridge in the Source Holder

5. Place the source plug in the source plug holder.



Figure 6-3. Attaching the Source Plug to the Source Plug Holder

6. Attach the source exchange tool to the source plug in the source plug holder.





7. Twist the plug until it aligns securely in the grooves in the source exchange tool and remove the plug from the holder



**Figure 6-5.** Attaching the Source Plug to the Source Exchange Tool

**NOTICE IMPORTANT** Use compressed air to blow all the dust off the source plug before inserting it into the mass spectrometer.

8. Once the ion source temperature has dropped below 200 °C, insert the barrel end of the source exchange tool into the vacuum interlock

and twist it clockwise to lock it into position. Be sure the black handle remains fully extended and locked.

Figure 6-6. Inserting the Source Plug into the Vacuum Interlock

- 9. Evacuate the VPI.
  - a. Confirm that the source removal tool is properly engaged in the VPI.
  - b. Press the blue Evacuate button on the front of the instrument.
  - c. The Evacuate light will begin to flash green, and should continue to flash green for approximately 20 seconds.
  - d. If the pressure has returned to an acceptable value after the 20-30 second wait, the evacuate light will turn off and the Ready to Open light will be solid green. At that point, the air has been evacuated from the instrument and it is safe to open the vacuum interlock value.
- 10. Pull the vacuum interlock handle up when the Ready to Open light is a solid green.
- 11. Twist the handle of the tool slightly to the left until it is feels like it is lodged into the left-most track.
- 12. Push the handle toward the instrument until the end of the handle aligns with the engraved line at the end of the barrel. When you reach this line, the tool is all the way in and the source plug is securely placed in the instrument.

**NOTICE IMPORTANT** The source plug should remain attached to the source exchange tool. Do not rotate the source exchange tool handle to attempt to disengage the source plug.



Figure 6-7. Raising the Vacuum Interlock Handle

- 13. Wait for oven and transfer line temperatures to drop below 50 °C to avoid burns before proceeding to touch the column and nut.
- 14. Use the GC transfer line handle to slide the GC transfer line away from the MS transfer line.



Figure 6-8. Separating the GC and MS Transfer Lines

15. Remove the current column and nut if present.

**NOTICE** Monitor the foreline pressure when removing the column and nut to confirm that the source plug is properly sealing the transfer line. If the pressure exceeds 3 mbar, the leak is excessive, and the source plug should be reseated or inspected for damage. If the turbomolecular pump is forced off by the vacuum protection and the pump speed drops below 80% speed, cycling power will not restart the turbomolecular pump. The pump speed must further drop below 25% before cycling the power to reset the pump and allow it to spin up to 100%. Be careful not to have the circuit breaker off for more than 5 seconds or there is a risk that the system will vent and must be pumped down overnight.  $\blacktriangle$ 

16. Unwind an appropriate column length to insert into the transfer line along the front of the instrument. Leaving about an inch gap between the column and the left side of the front panel will usually give you an appropriate length of column for installation.

Column Gap



Figure 6-9. Measuring the Column Length



17. Insert the new column through the GC transfer line.

Figure 6-10. Inserting the New Column through the GC Transfer Line

18. Carefully extend the column out the front to allow application of the nut and ferrule.



Figure 6-11. Extending the Column to the Front of the Instrument

- 19. Insert the column through the nut and ferrule (flat side of the nut faces the MS).

Figure 6-12. Inserting the Column through the Nut and Ferrule

- 20. Use a scoring wafer to score and then remove the last 10 mm of column to provide a clean, well-cut end.
- 21. Wipe the column with an alcohol soaked-wipe after inserting through the ferrule.



Figure 6-13. Scoring and Cutting the Column

22. Carefully push the column back into the GC transfer line while keeping the nut and ferrule on the column.



Figure 6-14. Proper Placement of the Column in the GC Transfer Line

23. Insert the column into the MS transfer line and tighten the nut until the column just resists sliding through the ferrule.



Figure 6-15. Inserting the Column into the MS Transfer Line

- 24. Loosen the nut ¼ turn and push the column into the MS transfer line until it just touches the source plug.
- 25. Pull the column  $\frac{1}{2}$  to 1 mm away from the source plug and tighten the nut  $\frac{1}{2}$  turn.

**NOTICE** To avoid forcing the column into the source plug when tightening the nut, the column can be pulled back approximately 1 mm before tightening the nut. ▲



Figure 6-16. Tightening the Nut

26. Ensure the column and nut are correctly installed in the MS transfer line.



Figure 6-17. Correctly Installed Column in MS Transfer Line

27. Pull the GC transfer line handle to the left to close it.



Figure 6-18. Closed MS Transfer Line

- 28. Remove the source plug with the insertion/removal tool.
- 29. Lower the handle to close the vacuum interlock.
- 30. Remove the source exchange tool.
- 31. Allow the source plug to cool.
- 32. Carefully remove the source plug from the insertion/removal tool using the source plug holder.

**NOTICE Tip** To avoid collecting dust on the source plug, store it in a closed container when not in use.  $\blacktriangle$ 

- 33. Using the source holder, place the ion source on the insertion/removal tool and install the ion source using the vacuum interlock.
- 34. In Tune Instrument Control > EI/CI Source, set the:
  - a. MS transfer line temp to the method temperature.
  - b. Ion source temp to the method temperature.
- 35. In Tune, place the instrument in the On state.
- 36. Leak check at the nut with tetrafluoroethane spray and tighten with a 1/4" wrench as needed. Perform leak checking by scanning in full scan and looking for +CF<sub>3</sub> (68.99466) and +CH<sub>2</sub>CF<sub>3</sub> (83.01031) peaks while spraying potential leak spots with tetrafluoroethane (dusting spray or refrigerant R-134A).)

- 37. Confirm the GC transfer line is covering the nut to prevent a cold zone on the column.
- 38. If vacuum compensation was set to **Off** for any reason (such as conditioning a new column), set vacuum compensation to **On**.
- 39. On the GC, set the Oven to **On**.
- 40. On the GC, in the Auxiliary Temperature Control, set Transfer Line 1 and Transfer Line 2 to **On**.
- 41. If necessary, reinsert the ion source to assure good electrical contacts using the insertion/removal tool and vacuum interlock once all temperatures have stabilized for 30 minutes and the source has thermally expanded.
- 42. Replace the vacuum interlock knob and evacuate the inlet valve chamber to remove the remaining air between the ball valve and interlock plug.
- 43. Confirm there are no leaks before beginning any tuning or analyses.