



OPERATING INSTRUCTIONS FOR THE HP 6890 PLUS GC WITH FID AND 5973 MSD

OVERVIEW

The instrument is no more than a typical single column GC with four inlet modes, electronic pneumatic controls and, thus, both temperature and flow rate programming capabilities, and is equipped with two detectors, an FID and an MSD.

The column effluent exits to a permanently installed splitter which diverts the flow in a 50/50 ratio to the two detectors. The MSD can and should be turned off with only the FID utilized in the development of the specific method parameters necessary to produce the desired separation of a given sample into its components. As part of this method development, the FID signal output can be programmed to ignore the large peak associated with the solvent front.

The next step in the process is to incorporate the MSD into the system, also programmed to ignore the solvent front. The chromatogram produced by the MSD will be quite similar to that of the FID with some difference in sensitivity. The additional information afforded is the MS, produced simply by clicking twice on a chromatographic peak from the MSD response for a particular sample component. The resulting MS can then be interpreted by the user or a search conducted in the NIST library for identification.

The instrument might be considered 95% GC, 5% MS in operation. With respect to utility, however, these two percentages may well be reversed.

OPERATING INSTRUCTIONS

SET-UP

It is quite likely that the instrument will be already turned on as you begin your analysis but in a "cold" condition so that you will have only to set the various operating parameters that you believe will effect an adequate GC separation. If this is the case, go directly to the Method Development section. If this is not the case, the instructions in this introduction will allow you to get started properly from a complete instrument shutdown.

1. Turn on the He carrier gas and adjust the outlet pressure to 45 psi.
2. Power the GC. The power switch is located beneath the oven door.
3. Power the Kayak PC, monitor, and printer.
4. Select Windows NT Workstation Version 4.00.
5. Log on as prompted by the monitor.

6. (a) If necessary, change User name to user.
(b) Enter the Password, user.
7. Once the GC has completed its diagnostics, double click on the GC-MS Instrument #1 icon.
8. From the View menu, select Diagnostics/Vacuum Control.
9. From the Vacuum menu, select Pump Down.
DO NOT RESPOND WITH AN OK AT THIS POINT!
10. Remove the cover from the MSD.
11. As prompted, determine that the Vent Valve is closed.
12. As prompted, power the MSD. The power switch is located on the front of the MSD, near the bottom.
13. Click on OK.

It will take the pumping system approximately 10 minutes to develop a vacuum sufficient to continue, a Foreline pressure of approximately 50 mTorr. If the foreline pressure does not decrease, gently adjust the thumb screw on the slide plate with the screwdriver located near the instrument.

14. When prompted, click on OK.
15. Click on OK in response to the Recommendation message.
16. If the MS Source Setpoint is 230 and the MS Quad Setpoint is 150, click on OK.
If not at these two Setpoints, make the necessary changes before clicking on OK.
DO NOT EXCEED 250°C FOR THE SOURCE TEMPERATURE OR 200°C FOR THE QUADRUPOLE TEMPERATURE.
17. From the View menu, select Instrument Control.
18. In response to the Download setpoint? message, click on Yes.
19. Via Aux, turn on the MS Transfer Line (interface). The Setpoint should be 280.
If not, change the Setpoint to 280.
20. Replace the MSD cover.

The MSD needs two to three hours to come to thermal equilibrium in order to provide reproducible results. At this point, however, the various GC parameters can be set and the GC allowed to come to thermal equilibrium so that you can begin the method development utilizing the FID.

METHOD DEVELOPMENT

This section will lead you through the basic setup of the GC utilizing only the FID. The various parameters that must be set in order for the GC to function properly will be presented. To that end, typical values will be suggested as "starters." These values, of course, can be changed in accordance with your knowledge, wisdom, and experience as part of the general routine associated with method development.

It is very important at this stage that the MSD not be in the data collecting mode, so:

From the Instrument menu, select Inlet/Injection Types. The resulting window allows you to observe the Use MS box. If an "x" appears in this box, remove it; otherwise, the MSD will collect data, something that must not be done until the MSD has reached thermal equilibrium and the method development has been completed.

Clicking on Inlets will gain you access to an edit mode that allows you to set the various parameters.

Inlet

1. Click on Inlets.
2. Select the desired inlet mode. Split is typical.
3. Change the Heater, °C Setpoint. A value of 200 is typical.
4. Turn on Heater, °C by clicking in the box.

The Pressure and Total Flow values should be satisfactory as set.

5. Set the Split Ratio. A value of 100:1 is typical.
6. If necessary, turn on the Gas Saver. A value of 20.0 mL/min is typical as is 2.00 min.
7. Click on Apply.

Column

1. Click on Columns.

Many parameters with respect to the column configuration are entered permanently into the system software. The only parameter you need to set is the Flow rate.

2. Set the Flow. A value of 1.0 mL/min is typical.
3. If you change the value for Flow, click on Apply.

Oven

Click on Oven.

Isothermal Mode

1. Set the Setpoint °C to the desired temperature. A value within the range 125 to 225 is typical.
2. To program the oven for isothermal mode, set the value for Ramp1 °C/min to 0.00.
3. Turn on the Oven heater.

A run time of 999.99 min can be used initially until the method development is complete with the run being stopped manually. Once the desired length of the run time has been determined, it can be programmed into the oven software so that the GC automatically terminates a run.

4. Click on Apply.

Program Mode

Enter the desired temperature program information.

Detector

At this point, only the FID will be utilized.

1. Click on Detectors.
2. Set the Heater, °C to the desired Setpoint. A value of 200 is typical. (Note: The flame will not ignite if the FID is less than 150 °C.)
3. Turn on Heater, °C.
4. Click on Apply.
5. If necessary, turn on the air supply and adjust the regulator pressure to 75 psi.
6. If necessary, turn on the hydrogen cylinder and adjust the outlet pressure to 40 psi.
7. If necessary, change the Setpoint value for H₂ Flow, mL/min to 40.0.
8. If necessary, change the Setpoint value for Air Flow, mL/min to 450.
9. If necessary, turn on Const Col + Makeup, mL/min. A Setpoint of 45.0 is typical.
10. If necessary, turn on the Electrometer.

11. If necessary, set the Lit Offset value to 2.0.
12. After the FID temperature has reached at least 150 °C, click on the Flame box, then Apply.

You should be able to hear the ignition of the flame. When the hydrogen and air flows have reached their Setpoint values, "On" will appear in the Actual column.

Signal

1. Click on Signals.
2. Check to be certain that Signal 1 is Det. Change if necessary.

Start and Stop times for the FID signal can be set as desired after the method development is complete.

Aux

1. Click on Aux.
2. If necessary, under Aux Channel, select MSD Transfer Line.
3. If necessary, under Type, select MSD.
4. If necessary, set the Heater to 280 °C and turn on the Heater.
5. Click on OK.

Turning Off The MSD Signal

1. Open the Instrument menu and select Inlet/Injection Types.
2. If necessary, click in the Use MS box to turn off the MSD signal.

The MSD signal can be turned on after the method development is complete.

3. Click on OK.

STANDARD/SAMPLE INJECTION

1. Click on the large green arrow near the top of the window.
2. Enter: Operator Name
Data File Name (This is your choice.)
Sample Name
Misc. Info (If so desired.)

Vial Number 1 is appropriate because the instrument is not equipped with an autosampler.

3. Click on Start Run.

4. As prompted, press the Prep Run key on the GC panel.
5. Press the Status key on the GC panel.

Within a very short period of time, the Pre Run indicator will be lit and steady, and a Ready for injection message will appear in the GC window. When these two conditions have been met:

6. Inject the sample and press the Start key on the GC panel.
7. When the sample run is complete, click on the STOP sign in the window and respond with a Yes.

DATA ANALYSIS

To view the chromatogram:

1. Enter the View menu and select Data Analysis (offline).

The chromatogram that appears on the monitor is from the run previous to the one just completed.

2. From the File menu, select Load Data File.
3. Select the data file for your run.

The chromatogram from your run will now appear in the window. There are a number of ways you may want to change the appearance of the chromatogram or produce data reports. You are left to your own devices to explore these numerous possibilities under the Chromatogram, Quantitate, and Tools menus.

UTILIZING THE MSD

It is assumed at this point that the method development is complete and that the MSD has come to thermal equilibrium.

To use the MSD in tandem with the FID:

1. Enter the Instrument menu and select Inlet/Injection Types.
2. Click in the Use MS box to turn on the MSD.
3. Click on OK.
4. Click on the MS icon and select Edit MS SIM/Scan.
5. For Start Time, enter a value that is greater than the time associated with the solvent peak exiting the system but not greater than the retention time(s) of the peak(s) of interest.
6. Click on OK.

You will at this point, likely, want to enter Oven, select Edit GC Oven, and change the Hold min value to correspond with the desired run end time. After this change, click on Apply.

You may also want to enter Signals and edit the Start and Stop values to correspond with the desired response and end time values.

If you make either or both of the modifications suggested in the previous two paragraphs, click on OK.

You are now ready to inject the sample. Follow the directions as before under Sample Injection.

In response to the Override solvent delay? message that will appear, click on No.

If you have programmed a stop run value under Oven, the instrument will stop collecting data automatically at the appropriate time, i.e. you will not have to stop the run manually.

VIEWING THE DATA/MS

1. From the View menu, select Data Analysis (offline).
2. From the File menu, select Load Data File.
3. Select the Data File for your run and click on OK.

Both the MSD (as TIC) and FID chromatograms will appear on the screen.

4. To see the MS associated with a particular MSD response peak, move the cursor to that peak and double click the right hand mouse button.

IDENTIFICATION VIA THE NIST LIBRARY

1. From the Spectrum menu, select NIST Output.
2. Select Create\Search Integd. Spectra File and click on OK.

The Clipboard will display all of the chromatographic peaks of the sample by retention time.

3. To identify a particular peak, select the appropriate retention time.
4. Double click the left hand mouse button.

The NIST program will attempt to match the MS of the peak selected to that of a library entry and report the results of this search.

Each of the three data screens can be maximized for easier viewing. The Expanded Plot screen has both scrolling and sensitivity change capabilities.