Agilent 6890N Gas Chromatograph (GC) with FID Detector

Updated May 29, 2008

Instrument instructions can be found at:
http://academic.bowdoin.edu/chemistry/resources/instructions.shtml

If you have any problems with the instrument or would like to get trained, please contact Celeste Moody (725-3756 / cmoody@bowdoin.edu / Druckenmiller 256)

1. Protocol

a. **Read instructions carefully before using instrument**. Reading the bold sentences in each category will tell you what you need to know to run the instrument.
   i. Bullets are under the bold sentences when more detail is required.
   ii. At the end of the instructions is a frequently asked questions/troubleshooting section.

2. Startup Procedure

a. **Turn on computer and login** (using your Bowdoin account).
   i. First time users only.
      1. Create folders to store your data, method, and sequence.
         a. Open Windows Explorer.
            i. (Start > All Programs > Accessories > Windows Explorers).
         b. Go to Desktop > My Computer > Local Disk (C:) > HPCHEM > 1.
         c. Create a data, methods, and sequence folder.
            ii. Click once on the data folder to highlight it.
            iii. Go to (File > New > Folder).
            iv. Type in your name or initials to name that folder.
            v. Repeat for the methods and sequence folder.
      2. Configure a network printer and set it as default.
         a. Make sure you are connected to the Bowdoin network.
         b. Go to Start > Run.
         c. Type in the location of the printer.
            i. First floor – "\\madison\werner".
            ii. Second floor – "\\madison\dahlia".
         d. Click OK.
         e. Set printer as default.
            i. Start > Printers and Faxes.
            ii. Right click on printer you just added.
            iii. In the menu, select "Set as Default Printer".
b. **Open "Instrument 1 Online".**
   i. (Start > All Programs > ChemStations > Instrument 1 Online).
c. **Check solvent and waste bottles in the autosampler.**

d. **Check gases.**
   i. Check the He, H2 and air pressure. They should all have at least 500 psi. If not, call Celeste x3756.
e. **Enter sample information into GC log sheet.**

3. Create/Edit Method

a. **Program should be in <u>Method and Run Control</u> view for the following steps**.
   i. Go to View > Method and Run Control.
b. **Load method** (File > Load > Method).
   i. To create a new method, use the default method (C:\HPCHEM\1\METHODS\default.m) as a starting point.
c. **Edit instrument parameters** (Instrument > Edit Parameters). Click on each of these icons to edit those parameters.
   i. 

– adjust injection volume and needle wash parameters. You should have at least two Pre-injection and two Post-Injection washes with solvent A and B.
   ii. 

– record information listed in "Manufacturer's Specifications" section in your notebook.
   iii. 

– adjust oven temperature parameters and develop a ramping method.
   iv. 

– turn detector on by selecting the boxes for H2 Flow, Air Flow, and Makeup Flow. Select Flame and hit Apply. Under the "Actual" column, "On" will be displayed when the flame is lit (takes a few seconds). If detector doesn't light, see Celeste x3756.
   v. Click OK.
d. **Save method** (File > Save > Method).

4. Create/Edit Sequence

a. **Program should be in <u>Method and Run Control</u> view for the following steps**.
   i. Go to View > Method and Run Control.
b. **Select Sequence Task icon**  .
c. **Load sequence** (Sequence > Load Sequence) or create a new sequence (Sequence > New Sequence).
d. **Edit Sequence Table** (Sequence > Sequence Table).
   i. If you have several samples to enter, use the Insert/FillDown Wizard. If you have only a few samples, skip to step ii and enter information manually.
      1. Click the Insert/FillDown Wizard button in the Sequence Table window.
      2. Select Insert; enter the number of lines to insert, starting location of the vials, method name, and injection/location. In step ii, fill in any information the wizard did not do automatically.
      3. In "List of Detected Ranges", select the line that is present.
      4. Click OK.
   ii. Enter information for your first vial in the sample table.
      1. Location – location of sample in autosampler tray.

2. Sample Name – name of sample.
3. Method Name – select method.
4. Inj/Volume – number of injections per vial.
5. Sample Type – Sample/Calibration/Control - typically set to "Sample".

iii. To enter more information, highlight "Line 1" and click the Append Line button.
iv. Click OK.

e. **Edit Sequence Parameters** (Sequence > Sequence Parameters).
   i. Enter your name.
   ii. Specify how you want the data file to be stored.
      1. Prefix/Counter – enter a prefix and number and the program use that prefix for every sample, but increment the number.
      2. Auto – program will create a number (i.e. 003-0102). The "003" is vial #, "01" is sequence line #, and "02" is injection #.
   iii. Type in a subdirectory. If you did not create a directory, one will be created.
   iv. Click OK.

f. **Save sequence** (Sequence > Save Sequence As).

5. Start Sequence

a. **Program should be in <u>Method and Run Control</u> view for the following steps**.
   i. Go to View > Method and Run Control.

b. **Configure monitor analysis window.**
   i. Open signal window – if Online Plot is not already open (View > Online Signals > Signal Window 1).
   ii. Change signal (if necessary).
      1. Click "Change" to display Edit Signal Plot screen.
      2. "Front Detector" is probably the only signal you'll need to monitor.
      3. Click OK.

c. **Run sequence** (Run Control > Run Sequence).
   i. A report will be generated after the run is complete, close this if you don't want to print it. You can generate a report in a later section.

6. Integrate

a. **Program should be in <u>Data Analysis</u> view for the following steps.**
   i. Go to View > Data Analysis.

b. **Select Integration task icon**  .

c. **Adjust x and y scale** (Graphics > Signal Options).
   i. In Ranges section, enter minimum and maximum values.
   ii. Click OK.

d. **Integrate spectrum.**
   i. Auto integrate (Integration > Auto Integrate) – this will integrate everything.

e. **Remove unwanted peaks from integration table by selecting icon**  .
   i. Click any labeled peaks in chromatogram you do not want integrated.

7. Generate Report

a. **Program should be in <u>Data Analysis</u> view for the following steps.**
    i. Go to View > Data Analysis.
b. **Select report destination and style** (Report > Specify Report).
c. **Print report** (File > Print > Report).

8. Shutdown Procedure

a. **Program should be in Method and Run Control for the following steps.**
    i. Go to View > Method and Run Control.
b. **Load method "Off.m".**
c. **Turn off detector.**
    i. Open Instrument Parameters (Instrument > Edit Parameters).
    ii. Click on Detector icon.
    iii. Deselect H_2 Flow, Air Flow, Makeup Flow and Flame. Click Apply then OK.
d. **Log off computer.**
e. **Leave the instrument and computer on.**
f. **Remove your samples from autosampler tray.**

Agilent 6890N GC Frequently Asked Questions

1. How do I run a single sample?

Follow the instructions, but skip Create/Edit Sequence and replace the Start Sequence section with the following steps:

a. **Program should be in <u>Method and Run Control</u> view for the following steps**.
   i. Go to View > Method and Run Control.
b. **Enter Sample Information** (Run Control > Sample Info).
   i. Enter Operator Name, Subdirectory (where the data will be stored), Filename, Location (where the vial is in the autosampler), Sample Name, and Comments.
c. **Run sample** (RunControl > Run Method).

2. How do I take a snapshot of the chromatogram?

a. **Program should be in <u>Data Analysis</u> view for the following steps**.
   i. Go to View > Data Analysis.
b. **Collect snapshot** (File > Snapshot).

3. How do I increase or decrease the number of peaks integrated?

Only peaks that are integrated will show up in your report. Also, the report will only show spectra of the peaks that are integrated.

a. **Program should be in <u>Data Analysis</u> view for the following steps**.
   i. Go to View > Data Analysis.
b. **Select the Integration Task icon.**
c. **Open Integration Events** (Integration > Integration Events).
d. **Adjust the Area Reject and Height Reject.**
e. **Hit the "Exit and Save Events to Method" icon.** This will show you the results of the new integration values. If the number of peaks is still not correct, repeat this process.

4. Configure screen layout so all windows and options are present.

a. **Program should be in <u>Method and Run Control</u> view for the following steps**.
   i. Go to View > Method and Run Control.
b. **View > Short Menu** ("Short Menu" should be displayed, meaning Full Menu is selected).
c. **View > Show Top Toolbar** (shortcut icons).
d. **View > Show Status Toolbar** (status of instrument and method and sequence loaded).
e. **View > Sampling Diagram** (ALS tray).
f. **View > Instrument Diagram** (system layout).
g. **If "Online Plot" is not present, go to View > Online Signals > Signal Window 1.**
h. **View > Command Line** (allows you to run macros).

Agilent 6890N GC Troubleshooting

1. The desktop doesn't have the shortcuts/icons to ChemStation.

a. **Go to the desktop and right click anywhere on the desktop to get the shortcut menu.**
b. **Go to New > Shortcut.**
c. **Type in the location of the shortcut.** Do both an offline and online shortcut to ChemStation.
    i. Online – c:\hpchem\core\hpcore.exe 2 /a
    ii. Offline - c:\hpchem\core\hpcore.exe 2
d. **Type in the name of the shortcut and then click finish.**