BUCK SCIENTIFIC MODEL 910
GAS CHROMATOGRAPH
OPERATION GUIDELINES
Chem. 231 Spring, 2013

Both of the Buck Scientific gas chromatographs (GCs) in Room 448 are available for this class. Both instruments have a capillary column attached to the heated split/splitless injector on the inlet side and a flame ionization detector (FID) on the exit side. The column to be used is a megabore (0.53 mm inner diameter) 15 m MXT-5 column. Because of the wide internal diameter of the megabore column, the split/splitless injector will always direct the entire injected volume onto the column (it only will work in the split mode with narrower diameter columns).

The Buck Scientific GCs are attached to a PC for instrument control and data acquisition using PeakSimple software. An operation manual is located in a shelf to the right of the atomic absorption spectrometer in Room 448. For operation of the GCs using the FID, three gas sources are needed: helium for the carrier gas and hydrogen and air for the FID's flame. Injections will need to be made manually using a 5 or 10 μL syringe for liquids and with larger syringes (typically 50 μL to 1 mL) for gases. GC number two also has fixed loop injector for gas samples (although the plumbing in the instrument would need to be re-routed to use it).

Changes to the instrument parameters can be accomplished by changing settings on the computer (e.g. for temperature programs or time of chromatogram) or by changing the set screws on the GC (e.g. FID temperature) using a small screwdriver which comes with the instrument. The detector response is recorded on the computer only, while other parameters (such as inlet pressures, oven, inlet, or detector temperatures) can be read by pushing the black buttons on the GC (when the switch on the right side of the instrument is in the "display setpoint" position. These buttons also can be used to find the "local" set point and "total" set point. The local set point refers to the setting from the screws on the GC itself, while the total also includes the addition provided from the computer. The switch for the display also may be in the "display column oven" to read the temperature of the column oven.

1. Basic Software Operation

PeakSimple is the software on the PC needed to control the instrument. If the instrument is not turned on, you will see an error message when the computer tries to interface with the GC. It is possible to use the Peak Simple software for data processing without the instrument on. You also can run the Peak Simple software in two windows, although only the first one started will control the computer and you will get an error message when opening the second window. This may be useful for doing data processing on past chromatograms while collecting data.

The software is started by double clicking on the PeakSimple icon. When the software starts up, the settings called up will come from the default settings. Since this instrument is most heavily used for other classes, it is best to start by opening a control file in which the instrument use is similar. The control files are opened under FILE => OPEN CONTROL FILE. You can start with a demo control file in the Chem 231 folder as the capillary column/FID has been operated under these conditions. If making any changes in many of the operating parameters, it is best to save the

parameters by using FILE => SAVE CONTROL FILE and **renaming the file** in your own folder within the Chem. 231 folder.

Most of the changes in operating conditions are done using the EDIT => CHANNELS menu. When opening this menu, you will see 4 separate channels that correspond to up to 4 different detectors that can be run with this instrument. The Channel 1 should read FID and be the only one we need to be concerned with (the boxes for active, display and integrate should be checked). The TCD normally is on Channel 2. Also note that the temperature on Channel 1 is the only temperature setting which can actually control the oven temperature. The sampling rate, default display range (Y-Axis), and run times can be changed in the "Details" submenu. Note that the run time may be automatically changed to correspond to changes in the temperature program. For capillary chromatography, I recommend using 5 Hz for the collection rate. The instrument should be set up in the mode to be controlled by temperature (as indicated in the right box in the details submenu).

2. Oven Temperature Control

The oven temperature is set under the EDIT => CHANNELS "temperature" submenu. BOTH the set screw (also called "local setting") and the computer program (which ADDS TO the base temperature) control the oven temperature. Currently, the local setting will be 0° C so that the computer program temperature will be the actual temperature. In the temperature submenu, the initial temperature and ramp rates are added. If you wish to change the ramp rate at a later time in the program or have a hold time at the end of the ramp, this can be done by adding a second line to the temperature program. The temperature program also will give a plot of the temperature vs. the time. Temperature programs can be loaded from and saved as with the control files. The oven temperature should not exceed 280° C, as this will damage the other column in the oven.

The following temperature program can be used as an example:

1st Line: Start T = 80°C
Ramp from start at 10°C/min until reaching 200°C
2nd Line: Remain at T = 200°C for 3 min. (15 minute run)

3. Setting Other Parameters

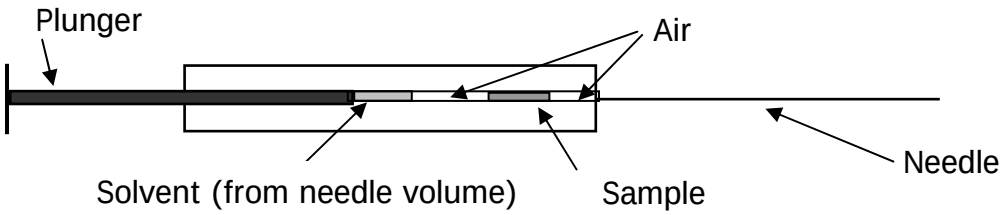
Other submenus on the EDIT => CHANNELS also will need to be changed. The "integration" submenu is used to set parameters to differentiate between noise and peaks. A larger peak number is needed to detect smaller peaks. A higher baseline number will tend to draw the baseline to valleys between peaks as opposed to dropping a line at the valley to a baseline on overlapping peaks. The area rejection sets the minimum area for retaining the information from the peak integration. The parameters can be reset after data collection. In which case you will need to use EDIT => RE-INTEGRATE.

The "components" submenu is used for naming the integrated peaks. The menu asks for the peak number (normally given in order of retention times), the name, and the "window" (earliest to latest retention times for the peak). Peaks need to come out within a component window in order to be integrated and reported. This submenu also allows for calibration and use of an internal standard. If you are interested in using Peak Simple for this type of data processing, read the section in the operation manual. The alternative procedure is to record the peak areas (or heights) and use Excel for calculations. Finally, you should use the "postrun" submenu to name the files that correspond to the chromatograms. Be sure to put these into your folder. If you want to save any of the work that you have done, you can rename temperature, component, integration, etc. files. You also can save all changes by saving the control file (make sure that this is your own file before saving).

4. Injections:

Use a 10 μL syringe (for liquid injections), preferable one with a replaceable needle. Be careful not to bend needle. Injection must be made quickly, but worry more about inserting the needle properly first. To fill the syringe using the "sandwich injection technique", pull the plunger back to the 2 μL line, place the tip of the syringe in the solution, pull the plunger back further to the 3 μL line, remove the syringe and pull the syringe further back to about to the 5 μL line. The syringe should look like that shown in the figure below. An alternative technique is to simply put the syringe into the liquid with the plunger on 0 and pull back to the 1 μL line line. The second technique seems to be more susceptible to variations in amount injected. Push the syringe needle slowly and carefully until it has pierced the septum in the inlet. Once this is completed, the needle should slide in easily and you can push the needle in the rest of the way in a faster motion. Then depress the plunger which makes the actual injection.

Syringe filled with sample using sandwich injection technique.



5. Start of chromatograph:

Hit space bar to start acquisition. This needs to be done near the time of the actual injection to get accurate retention times.

6. Additional Instructions:

Additional specific instructions will probably be needed on the operation of the GC and will be given by the instructor.

Getting Started

The instrument power normally will be left on. ALL the gasses EXCEPT the He carrier gas should be turned off at the end of the lab period. DO NOT CHANGE THE OUTPUT PRESSURE VALVES, JUST TURN THE MAIN TANK VALVE(S) OFF (CLOCKWISE) SO THE PRESSURE WILL BLEED OFF. LEAVE the He GAS ON. The air and hydrogen flows for the FID should not need to be changed.

1. Open the main valves on all the gas tanks. The He cylinder should already be open.

2. The He pressure should be already adjusted correctly. It is possible to modify the flow rate. If this is requested, the instructor will demonstrate how to adjust and measure the flow rates.

3. The H_2, and air gases for the FID should be opened at the cylinder main valve without changing the pressure setting! Note that these valves need to be opened about 30 minutes before instrument use (it takes time to flush air out of the H_2 lines).

4. Ignite the FID: lift the ignition button. MAKE SURE NOT TO ACCIDENTALLY PUSH THE CARRIER FILTER BUTTON WHICH LOOKS SIMILAR. Check to see that the FID

changes to a non-zero response after lighting. Alternatively, it may be possible to lift the instrument cover, and use a mirror to check for condensation forming on the exhaust gas (DO NOT TOUCH THE EXHAUST TUBE WITH THE MIRROR).

5. Before running, make sure that all of the temperatures are stable. This will be indicated by flickering LED indicator lights (lights are on when heating) or by checking the set temperatures against the actual temperatures.

Conditioning the Column:

The column should be "purged" before doing any serious work. This can be done by raising the oven temperature to a high value (e.g. 200° C or higher). There is a temperature program (colbake.tem) which can be loaded to bake the column. Remember not to go above 270° C.

Running a Blank

A small portion of the makeup solvent (e.g. hexane) should be subjected to the same chromatographic conditions as your samples. High grade (expensive) solvents are used because the technique is so sensitive, only very low levels of impurities are tolerable. NOTE! This means you must be very careful not to contaminate your samples. Be sure to rinse the syringe with pure solvent several times before using it to withdraw a different sample. Glassware should be rinsed with a small portion of solvent before using.

The temperature program should be the same as for samples and can be reloaded by reopening the control file after the column baking program.

To make an injection, first hit the "Z" button on the display to zero the baseline, and make the injection according to the instructions given above. The space bar on the keyboard should be depressed to start the data acquisition as soon as possible after the injection. If you wish to stop the injection early, you can use ACQUISITION =>STOP. You will need to go to FILE =>SAVE to save the file if the run ends early.

Running Samples

Samples will normally be run in the same manner as blanks. Normally, you start by running standards and making sure that all of the standard compound elute during the chromatogram. Method development involves adjusting the temperature program to make sure that peaks elute far enough away from the solvent peak, that peaks are well resolved, and that all peaks are eluted. Besides adjusting the temperature program, the injector temperature can have an effect on the size and interference from the solvent peak.

Shutting the Instrument Down

Depending on the time scale it may be desired to shut the whole instrument down or to put it into a "sleep" mode. You should first make sure that injected compounds have been eluted. It is also better to have carrier flow through the columns while the oven is still hot. If it is desired to put the instrument into a "sleep" mode, a sleep temperature program can be loaded. Then the gases for the FID can be turned off at the cylinder valves. Peak simple then can be shut down. If the instrument is not needed for several days, the carrier flow can be turned off at the He cylinder and the main power button for the GC can be turned off.