

## Guideline for operation of GC instruments in lab 338 (ABBOTT HALL)

### IMPORTANT

- \* Replace He, H<sub>2</sub> or Air tank when the main pressure drops below 100 psi.
- \* Do NOT use matches or splints to light FID, use long-tip butane lighter.
- \* The HYDROGEN, AIR and AUX GAS knobs should always be either full OFF (clockwise) or full ON (c-clockwise). Turn them gently! Do not force to open further!!!
- \* The column should NEVER be heated without a flow of carrier gas!!

### Pressures & flow rates for HP5890 Series II split/splitless system with FID and capillary column<sup>a</sup>

<i>He (carrier gas)</i>	
<i>tank, 2<sup>nd</sup> stage</i>	80 psi
<i>inlet line</i>	60 psi
<i>column head</i>	~ 3-4 psi
<i>split vent</i>	30 mL/min
<i>purge vent (septum)</i>	3 mL/min
<i>column flow</i>	1 mL/min <sup>b</sup>

**All the flow rates are preset, you need to verify them but DO NOT modify the settings unless instructed by your instructor. If you have any concerns contact Dr. Kubatova AH 301 7-0348**

<i>FID gases</i>					
<i>Air</i>		<i>H<sub>2</sub></i>		<i>He</i>	
<i>tank, 2<sup>nd</sup> stage</i>	80 psi	<i>tank, 2<sup>nd</sup> stage</i>	40 psi		
<i>inlet line</i>	~ 40-50 psi	<i>inlet line</i>	~ 20-25 psi		
<i>FID flow</i>	400 mL/min <sup>d</sup>	<i>FID flow</i>	30 mL/min <sup>d</sup>	<i>aux. gas flow</i>	35 mL/min <sup>c</sup>

(a) This column head pressure is based on a 15 m x 0.32 mm ID capillary column.

(b) Control flow rate using column head-pressure valve.

(c) The AUX GAS control is an ON/OFF device combined with a needle valve for flow adjustment (center screw). The needle valve is VERY delicate and should not be forced to either end of its travel.

(d) Control flow rate using inlet line-pressure valve.

### Gas supplies:

Before starting make sure you have on all gas cylinders sufficient pressure. REPLACE HE, H<sub>2</sub> OR AIR TANK WHEN THE MAIN PRESSURE DROPS BELOW 100 PSI.

Carrier gas: Helium (carrier gas) ultra high purity.

Detector: Nitrogen, Hydrogen, Air (breathing quality)

**Pressure regulators:** the gas supplies are controlled with dual stage pressure regulators. The first stage shows the pressure in the gas tank (how much gas is still inside). Replace the gas tank when < 100 psi. The second stage shows pressure needed to supply gas to GC (note in this case enough for three GCs). Depending which GC will be operated, the on/off toggle valve has to be switched on.

## ***Operational Instructions for the HP 5890 Series II GC's with FID***

The following steps should be followed to start-up and shut-down each GC/FID system. In some instances consultation of the GC and integrator operation manuals may be needed.

### **STARTUP OF INSTRUMENT**

1. Turn ON the main valves for the He (UHP grade), H<sub>2</sub>, air, and N<sub>2</sub> cylinders. If the main pressure for any tank is less than 100 psi, it should be replaced.
2. OPEN the He, H<sub>2</sub>, & air manifold valves (toggle on/off) to the GC (#1, 2, or 3) you will be using.
3. Note which injector/detector ports have the column installed (A-front or B-back) and *confirm the pressures and flow rates as shown in table on 1<sup>st</sup> page*. Use the manual bubble meter to verify the flow rates (a stop watch on the instrument for determination is a convenient tool for the determination). Set flow rates from lowest to highest.

The flow rates are determined by connecting the flow meter to the outlets of detector (when detector is off and cold), split line, and septum purge. If the flow rates need to be reset, open completely the total flow control, set the column head pressure to achieve sufficient flow rate on the column; setup of the flow rate on the septum purge; Open the makeup gas on the detector; and decrease the flow rate on split line by the total flow control.

4. Turn on the GC main power and set the heated zones to nominal initial temperatures (oven 35 °C, injector 250 °C, and detector 300 °C).
5. Configure the injector for split operation (set Purge A (or B) ON) and ignite the FID:
  - a. Through the GC keypad, set DET A (or B) ON, set SIG1 to A (or B), and press SIG1 (twice). This displays the FID signal (in pA), *which should be less than 1 pA before ignition*.
  - b. *Use a long-tip butane lighter to ignite flame*. When the temperature of the detector reaches 150 °C, the detector can be ignited. To ignite the FID, turn the HYDROGEN fully ON, after 2-4 seconds proceed with AIR. Lower the butane flame to the collector opening, observe the FID signal to see response. Although the ignitor button is disabled, sometimes pushing it at the moment of igniting with butane lighter helps).
  - c. If the FID ignites, an audible pop may be heard. The signal will jump above 100 pA then quickly drop back to between 10 – 20 pA and remain stable to within  $\pm 0.1$  pA (you can use a glossy surface like mirror or spatula to verify there is a flame). Turn the AUX GAS fully ON, make sure that flame stays lit.

6. Turn ON the integrator. Consult the instruction manual for operational commands.

The system should be ready to operate at this point.

### **OVERNIGHT SHUTDOWN OF INSTRUMENT**

The instrument can be left ON overnight. However, students **must inform** the instructor. To save the carrier gas the overall flow rate (split line) should be reduced to 5 ml/min. When reducing total flow, the pressure on the column should be checked in 20 min.

## LONG TERM SHUTDOWN OF INSTRUMENT

1. Shut down GC in the following order:
  - a. Turn OFF the integrator.
  - b. For the FID, turn the AIR, HYDROGEN and AUX GAS fully OFF. The FID signal should drop to less than 1 pA.
  - c. Turn OFF the Injector and Detector heaters and set the oven temperature to ambient.
  - d. Wait for the entire system to reach room temperature and then turn off the GC main power.
2. CLOSE the He, H<sub>2</sub>, & air manifold valves to the GC (#1-3).
3. Turn OFF the main valves for the He (UHP grade), H<sub>2</sub>, and air cylinders. If the main pressure for any tank is less than 100 psi, it should be replaced.

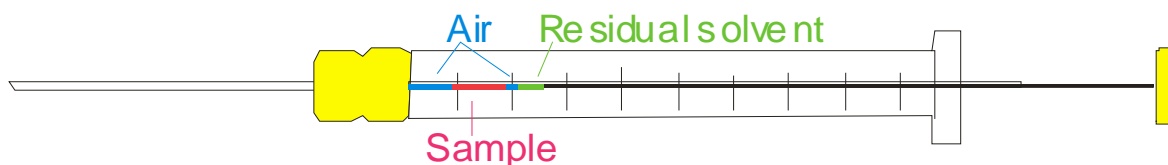
## OPERATION OF GC INSTRUMENT

(Detailed instructions are available in manual)

**Sample injection into a split/splitless injection port.** The suitable volume of sample injected depends on the volume of the injector liner. The injector liner with i.d 4 mm has a volume of ~900  $\mu\text{L}$ . Do not forget that liquid expands when converted to the gas phase. The expansion depends on the pressure, temperature, density and MW of solvent used (defined by the ideal gas law). See equations in examples.pdf, or you can use a vapor volume calculator to determine the volumes.

Generally the 4 mm i.d. injector liner allows for injection of 1  $\mu\text{L}$  for the majority of solvents. When making an injection, it is important to keep firm pressure on the syringe plunger or the injector pressure could cause it to blow off.

To ensure injection of the same volume, you need to preclean the syringe in the pure solvent, then draw 0.2  $\mu\text{L}$  of air, followed by 1  $\mu\text{L}$  of sample, and again air. This will result in a 1  $\mu\text{L}$  band of the sample visible in the syringe.

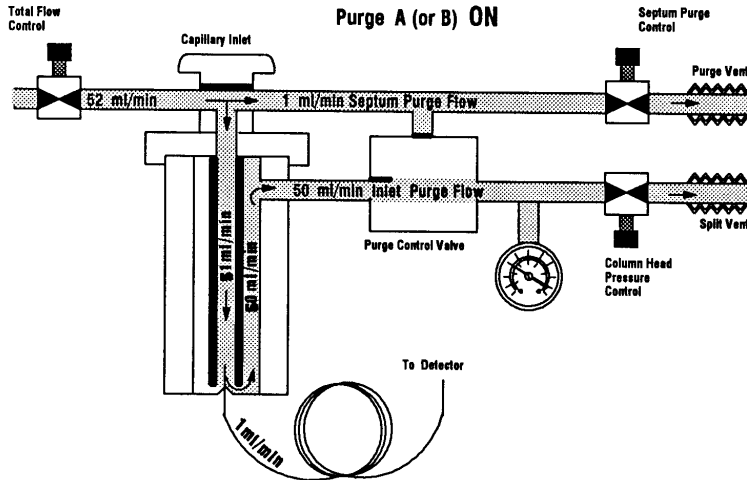


The syringe is introduced into the injector, the piston pushed quickly, then the syringe is quickly withdrawn from the injector in one smooth upward motion. To start the program, push the start button on GC.

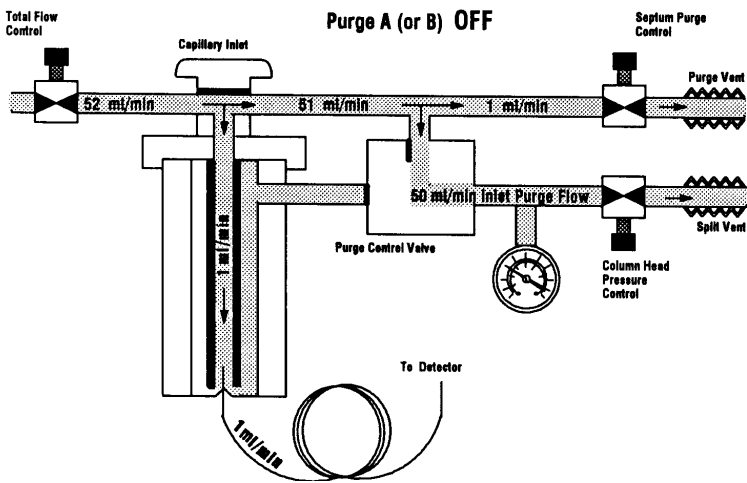
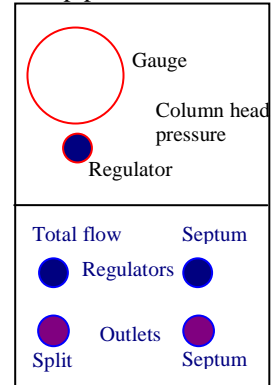
### Operating split/splitless injector port

Always make sure you are operating the injector port in which column is installed (A–front or B–back). The diagrams below show the split and splitless flow diagrams. Three regulators on the top panel are setting the correct flow rates on the system. On the top panel, we can also

observe gauge showing the head pressure on the column and two outlets one for split/splitless line and one for septum purge. Total flow control determines overall flow rate of Helium on the GC system (this include helium going through column, septum purge, split vent, and also makeup gas on detector).



Top panel of GC



Besides the to setup of flow rates, purge control valve needs to be programmed. This valve controls whether will be the injection in split or splitless mode.

For split injection the valve has to be continuously set to “Purge ON” (top figure). Go to the electronic board on the front of the GC (left side).

**Programming of split operation (turns purge valve A on)**

- PURGE/VALVE [A] [ON]
- PURGE/VALVE [A] [TIME] [ON] [0] [ENTER]
- PURGE/VALVE [A] [TIME] [OFF] [0] [ENTER]

**For splitless injection** the purge has to be off for the initial splitless time and than ON

PURGE/VALVE (A) TIME OFF 0 ENTER

PURGE/VALVE (A) TIME ON 1 ENTER

In this case the valve is off initially, and switched on at 1 min. Always make sure the valve is off just before the injection.

PURGE/VALVE (A) OFF

If you run more than one run, you can set purge time off just before the end of analysis.

### Temperature programming

Single temperature ramp or multiple can be programmed; the highest rate is ca. **35°C/min**. If the instrument starts blinking red during the run, and the temperature gradient is higher, it is possible that the instrument cannot increase the temperature linearly.

### ISOTHERMAL

OVEN TEMP 9 5 ENTER Sets the oven temperature to 95 °C.

INIT TIME 1 ENTER Sets initial time to 1 min. This defines how long will be the analysis.

RATE 0 ENTER Eliminates all possible temperature programs.

### 1 RAMP

OVEN TEMP INIT VALUE 3 5 ENTER Sets initial temperature to 35 °C.

INIT TIME 1 ENTER Sets initial time to 1 min.

RATE 3 5 ENTER Sets rate to 35 °C/min.

FINAL VALUE 9 0 ENTER Sets final temperature to 90 °C.

FINAL TIME 5 ENTER Sets final time to 5 min.

### MULTIRAMP

Input ramp 1 as shown above and then.

RATE (A) 5 ENTER Sets 2<sup>nd</sup> rate to 5 °C/min.

FINAL VALUE 2 8 0 ENTER

FINAL TIME 5 ENTER Sets 2<sup>nd</sup> final temperature and time to 280 °C for 5 min.

Note the instrument should show you this is “A” final value and time. To program third ramp use program “B”.

### Stopwatch/Time

Pushing button “Time” before run scrolls through a last run time, next run time, and stopwatch. During a run, it scrolls through the elapsed time, remaining time, and stopwatch. You operate stopwatch by pushing “ENTER” and “CLEAR”.

## OPERATION OF INTEGRATOR

(Detailed instructions are available in manual)

Turn the integrator ON.

To check the major parameters press [LIST] [LIST] (twice)

Those parameters can be changed by selecting the [parameter] [value] [ENTER]

**ZERO** sets the baseline zero position

**ATT2<sup>^</sup>** attenuation (-8 to 36). For the attenuation = 0 the plotter sensitivity is 1 mV full scale.

Each step lower doubles the heights of peaks (e.g. lower number = bigger peaks).

**CHT SP** = chart speed in cm/min.

**AR REJ** = area reject, is a minimum value of the reported peak area.

**THRSH** = peak detection threshold, with range -6 to 28, defines minimum peak height that will be accepted.

**PK WD** peak width (default 0.04 min) if peaks are wider integrator will not recognize them.

### Other parameters

Use [INTEG] and [number]

[INTEG][0] Set baseline now.

[INTEG][7] Turn off retention time labeling.

[INTEG][8] Turn on Start/stop marks ( where integration stops starts).

[INTEG][9] Turn off integration.

### Moving paper

[CTRL] [L] Advance paper to next top of form.

[CTRL] [A] Advance paper 1/8 of a line.

[ENTER] Advance paper one line.

[SHIFT]+[ENTER] Advances paper continuously.