GC SAMPLE PREPARATION:

- **Sample Components to Avoid Completely:**
  The following should never be injected: metals, strong acids or bases, salts, oligomeric and polymeric material. These classes of compounds are unsuitable for gas chromatography, and can damage the GC column.
- **Solvents:**
  Hexane, acetone, and methanol are the recommended solvents for sample preparation. Other acceptable options are benzene, ethers, and methylene chloride. Do not dissolve samples in water, DMSO, or DMF.

TURNING ON THE INSTRUMENT:

- Turn the Nitrogen (80 psi on regulator), compressed air (60 psi) and hydrogen (40 psi) gas tanks on. **Note:** If the gas in any of the tanks fall below 300 psi, the cylinder should be replaced.
- Turn on the Gas chromatograph. The power button is on the top left side of the instrument.
- Make sure the computer controlling the instrument is turned on and open the **Galaxie** software.
- Use the following user name: **User1**.

METHOD DEVELOPING AND EDITING:

- Under the File tab click on **Open a Method**. If a method is not already developed begin by opening **FID.METH** and proceed to **save method as** desired method name.
- Select the method on interest and click on the **Open** button.
- Once the Method of interest appears under the Data Files dialog box proceed to alter the method by following the **Data, System** and **Calibration** tabs at the bottom left of the control page.
- Click on **control** icon in the **Data** tab. This will open a dialog box in the split screen (Fig. 1) in which many parameters can be altered.
- Click on the **Autosampler (8400)** icon and make sure the **ENABLED** box is checked. The **Syringe volume** should be set to **10 µL**, the **Injection mode** to **Std (Split/Splitless)**, the sample and solvent penetration set to 90%. Default and Clean Mode can be altered to desired number of washes pre and post injection. **Make sure the solvent source is filled with the desired solvent!**

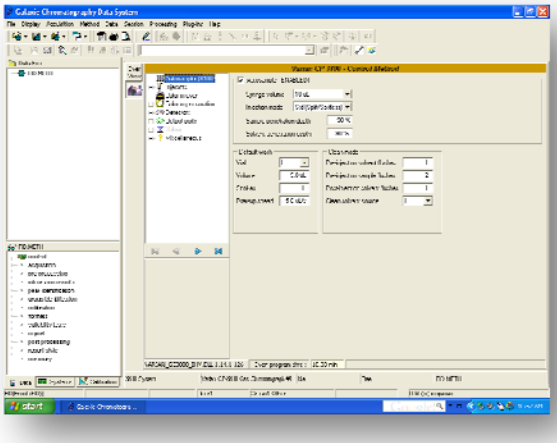

Figure 1. Autosampler- Control Method.

- Click on the **Injectors** icon. Only the front is installed and active. Make sure the **Heater** is set to ON and input the **Setpoint** to desired temperature. The set point should be set to 25°C warmer than the final column temperature or about 50°C warmer than the boiling point of the analyte. The split event table can be used to add or subtract events at injections. In order to increase injection volume without overloading the injector the following is a common event table for Split/Splitless injection:

	Time (min)	Split Rate	Split Ratio
1	Initial	ON	20-100
2	0.01	OFF	20-100
3	0.75	ON	20-100

  If the concentration of the analyte is not too dilute, simply setting one event to slit rate On and split ration between 20-100 will be sufficient.
- Click on **Column Oven** icon. This will allow controlling of the temperature ramp and the length of the experiment. Adjusting the **Rate** (°C/min temperature will increase), the **Temperature** to be reached and the length of **Time** for each step will allow to optimize experimental conditions.

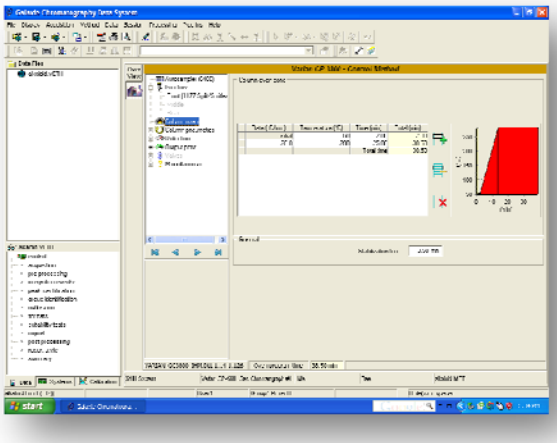

Figure 2. Column Oven- Control Method.

- Click on **Column Pneumatics** icon. For most experiments, constant flow should be enabled and the column flow set to 1.0 mL/min. However it is possible to also to run pressure pulse experiments by disabling **Constant Flow** and setting up Rate, **Pressure** and **Time** events.
- Click on the **Detectors** icon. Both the **Heater** and the **Electronics** should be set to **ON**. The set point of the FID heater should be similar to the final column temperature (usually 250-300 °C). The **Time constant** and **Range** can be altered to modify the data collected. A fast time constant and a range of 12 (10^{-12} Amp/mV) is amenable to most experiments. **Autozero** should be set to YES. The **Type 11 EFC** window will show the set points for the individual gas flows.

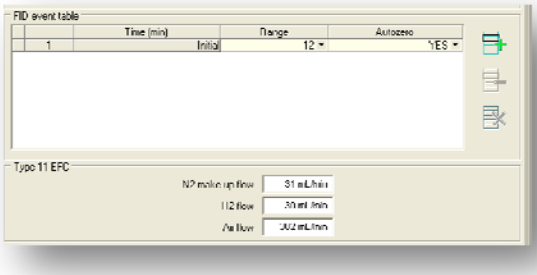

Figure 3. Detector Gas Setpoints.

- Once all the parameters are set, proceed to save the method and make sure the changes are loaded to the Gas Chromatograph by clicking on the **Overview** button under control in the **Data** tab, and press on the first icon with a red arrow. This will transfer all settings to the instrument to the default **Method 8**.

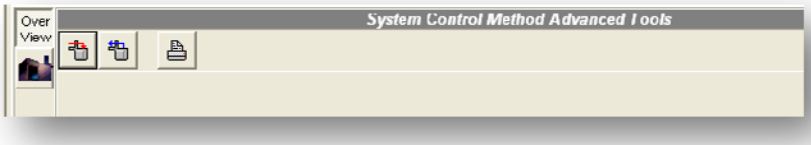

Figure 4. Communication to and from the GC.

- If the FID is not on or you experience a flame out, assure the parameters have been sent to the instrument and check the detector is on by putting the provided stopper and metal column on the detector, and check for vapor against a shiny object.
- Click on **Acquisition** in the data tab at the bottom left of the screen and proceed to enter the experiments parameters. **File prefix** will be applied to all runs carried out under the same conditions, **Sample properties** and **Column parameters** are optional, but **Acquisition parameters** should reflect the correct **Vial #** and **Acquisition length**. Check **Autoscale** or set up parameters for the data collected in the **Working Scale** portion.

SEQUENCE DEVELOPING AND EDITING:

- Under **File** select **Open Sequence** and proceed to modify and save settings from Start-up sequence.

- In the sequence control window it is possible to add and remove events, set up runs of multiple samples with the same method or changing methods in the analysis of the same samples:
- In the event table, the **Enabled** column will assure the sample will be run, the **Method** must be entered as well as the **Vial #** and the **Inj. Volume**. Other parameters are optional.

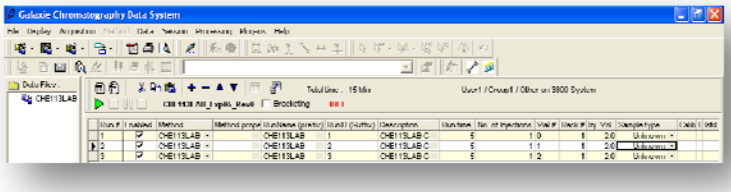

Figure 5. Sequence Editing.

- Once the sequence has been edited, proceed to **Save** the settings and press the **green arrow button** to begin the desired runs. It is possible at any point to stop the run by pressing the **red stop button**.
- It is possible to monitor the status of the GC and the progress of the run by clicking on the Systems tab and expanding both the Chromatogram and Status tab under the **3800 System semaphore**. Toggling between the **Overview** and the **GC icon** on the bottom panel will allow for monitoring of different settings on the GC.

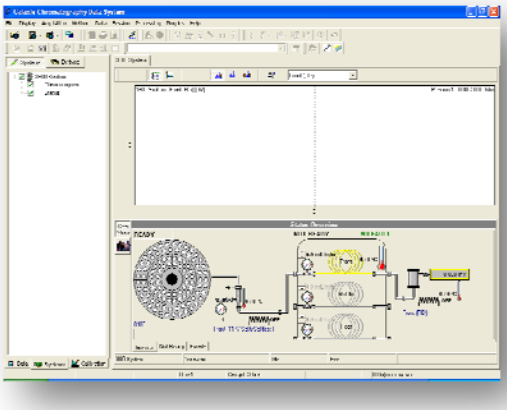

Figure 6. System Monitoring.

ANALYSIS:

- Once the chromatogram has been collected it is possible to reprocess the data at any time. To do so click on the Integration event under the data tab to **Set Peak Width** and **Threshold** and to determine when to **Turn integration on**, in order to ignore solvent peaks during integration. This can be altered post run and reprocess the chromatogram as needed.
- Other setting can be accessed by using the right mouse button to add as many integration events as needed.

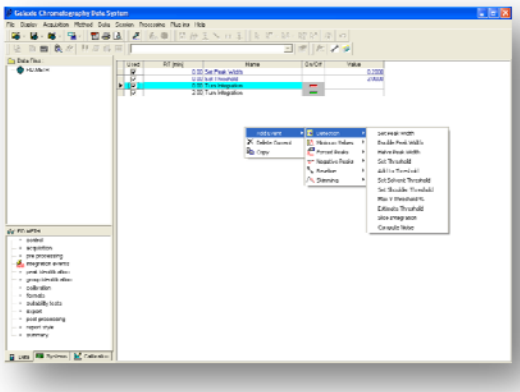

Figure 7. Integration Events.

- In order to view result, under **File** click on **Open Chromatogram** and select the data sets of interest. In the top left window, the data sets will appear with three possible views: **Data** to solely view the chromatogram, **Method** to view the integration events as well and the **Results** in order to view the result table as well as the chromatogram.
- In order to zoom in, hold the left mouse button and drag over the area of interest from top left to right bottom. To zoom out, invert the action from right bottom to top left.
- Click on **Print** under the **File** tab to print a standard report.
- A series of icons in the top toolbar allow to reprocess the chromatogram by altering the **Peak Markers**, **Labels**, **Add Peaks**, and **Split Peaks** among others options. Once these settings are altered, it is necessary to **Reprocess** the data. Under the **Processing** tab it is possible to **Reintegrate** and **Reprocess** the data. After doing so proceed to save changes and print new results.

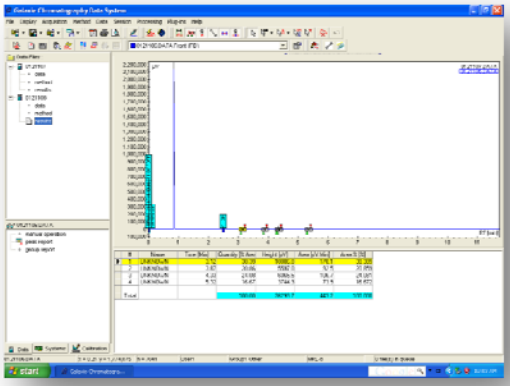

Figure 8. Data and Results.

SHUTTING DOWN THE INSTRUMENT:

- Proceed to load the Cool Off Method. This will turn down gas flows and turn off the detector.
- Once the instrument has been cooled for about 30 minutes, proceed to turn off the GC with the main switch on top of the instrument, and close all three gas tanks: H_2, Air and N_2.