Agilent GC/MSD Instructions

GC/MS 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.
- **Concentration:**
  Mass spectrometry is several orders of magnitude more sensitive than NMR, so please do not use the same samples you have prepared for NMR analysis for MS analysis. The upper limit of concentration needed is 100 ppm. If you introduce a more concentrated sample, you will damage the EI filament. **WHEN IN DOUBT, DILUTE!**

STARTUP PROCEDURE:

- Open the Agilent software that controls the GC, by clicking on the **Instrument #1** icon. When prompted, enter: **manager** as both the login name and password.
- Check solvent bottle is filled with the appropriate solvent and the waste bottle is in the appropriate location on the autosampler carousel.
- Check the He pressure is at least 500 psi on the main gauge. If not call Massimo X58016.

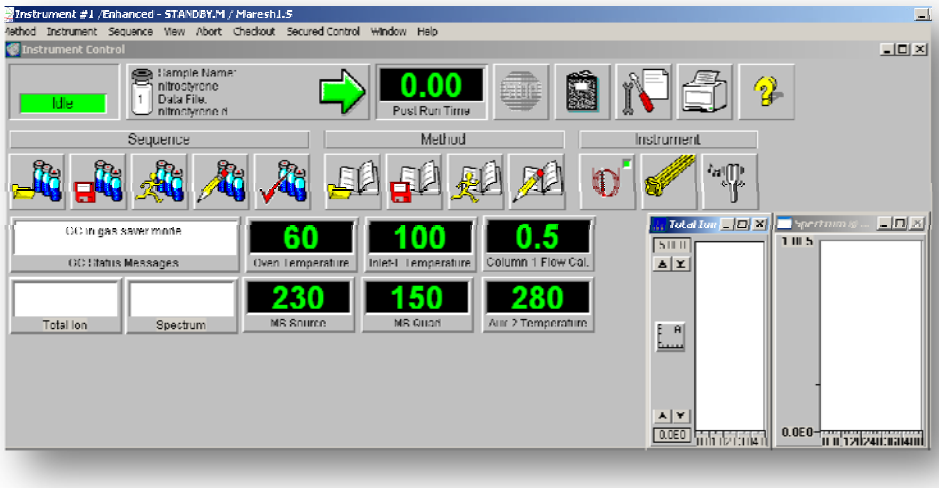

Figure 1. Instrument Control View.

METHOD DEVELOPING AND EDITING:

- Under the **View** tab make sure the software is in the **Instrument Control** view.
- Under the **File** tab, click on **Load Method.** In order to create a new method use the following default method: **MPEDIT**.
- Edit the instrument parameters by clicking on the individual icon to vary specific experimental conditions.

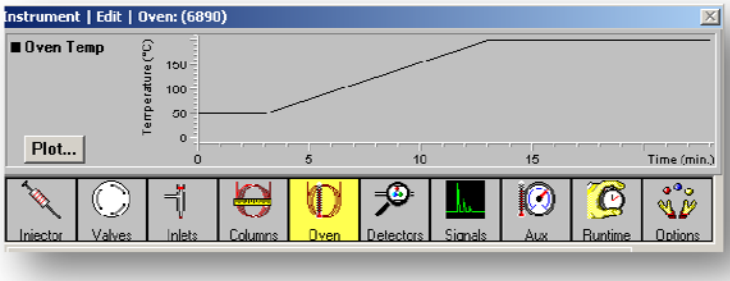

Figure 2. Method Editing.

- Under the **Injector** icon, set the injection volume (**1.0 µL**) and set the conditions for pre and post injection syringe washes.
- Under the **Inlet** icon, set the **Split** or **Splitless Modes**, the **Split Ratio** and **Flow**, make sure the **Heater**, **Pressure** and **Total flow** are checked and input the setpoints. Make sure the **GasSaver** is turned on at 20.0 mL/min @ 2.00 min.

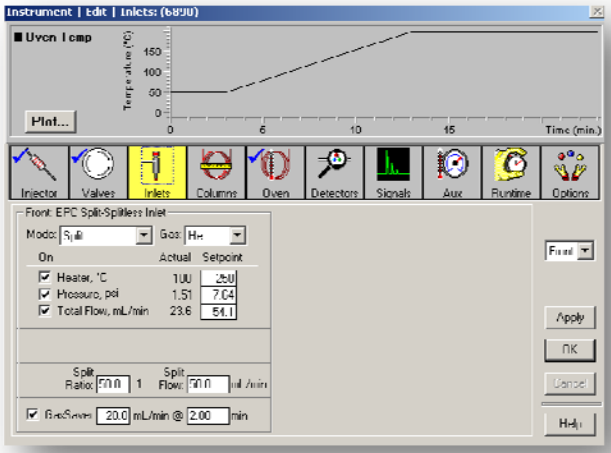

Figure 3. Inlet Editing.

- Under the **Column** icon note the manufacturer's specifications. Only **Column 1** is installed in the front inlet. **Constant flow** mode is appropriate for most experiments, but the mode can be altered to other settings as desired. The **Detector** used is **MSD**. Helium **flow** should be set to 7.64 psi and **Average velocity** to 36 cm/sec.

- Under the **Oven** icon, the temperature can be set by setting up temperature ramps by inputting the Rate (°C/min), the **Temperature** desired and **Time** to hold the set temperature. The **Run time** will be automatically calculated.
- Under the **Signal** icon, the **Data Rate** and **Minimum Peak Width** can be set for detection.
- Under the **Aux** icon, check the **Thermal Aux #2** and **Heater** and set point to 280 °C. **Type** is **MSD**.

SEQUENCE DEVELOPING AND EDITING:

- Select Sequence Task icon. Either **Load Sequence** or create a new sequence by editing the default sequence: **MPEDIT**.
- Enter information for your first vial in the sample table. Enter the sample **Type** (Sample/Calibration/Control), the **Vial** number based on its position in the autosampler, the **Sample name**, the **Method**, **Data file** and **Comments**.
- Consider including in the sequence a solvent blank run in the sequence. Also you should always include a last item in the sequence, which will load the **Standy method**.
- Click OK when all changes have been made.
- Save the sequence in **Save Sequence As**.

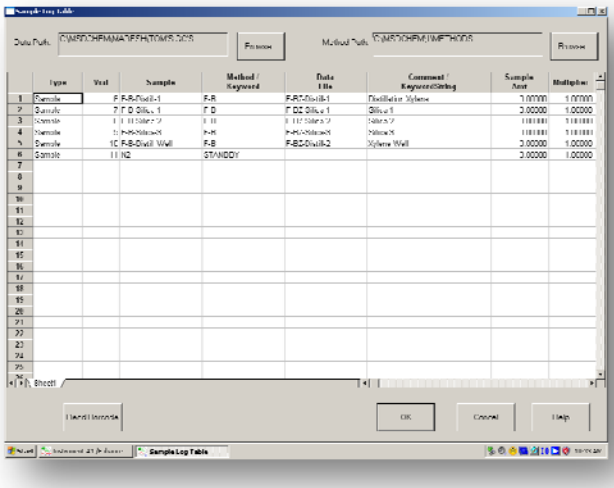

Figure 4. Sequence Editing.

- In order to start the sequence click on the **Run Sequence** icon, or access the command from the toolbar. Enter the sequence **Comment**, **Operator name** and **Data file directory**, then click on **Run Sequence**.

- In order to run a single sample:
  - **Load** the desired method.
  - Under file click on the **Run Method** and input the **Data** information, **Operator Name**, **Vial #** and **Sample name** and **Information**. Click on **OK**.
  - Click on **Run Method** to begin the run.

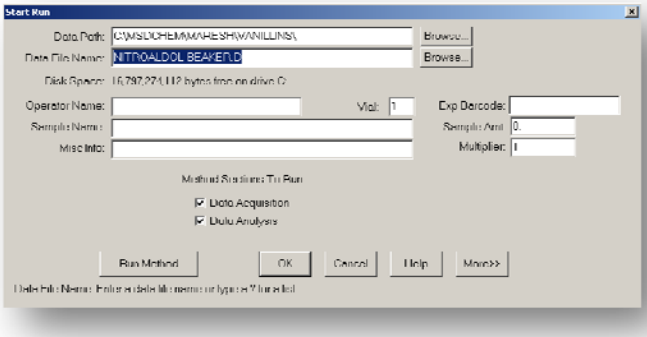

Figure 5. Start Run Window.

- You can monitor the data collection on the screen in the **Total Ion** and **Spectrum** windows.
- A standard report will print automatically once the run is complete.

DATA ANALYSIS:

- Under the **View** tab, switch to **Instrument Data and Analysis**, when prompted input **manager** as the login name and password.
- Under the **File** tab, click on **Open Chromatogram** and select the data set of interest. The chromatogram will be displayed and available for reprocessing.
- In order to integrate peaks, click on the **Integration** icon and adjust the **x and y scale**. Alternatively, click on **Autointegrate** in order to integrate all peaks above set threshold.
- The toolbar will allow to alter **Integration events** by adjusting **Area Reject** and **Height Reject**.

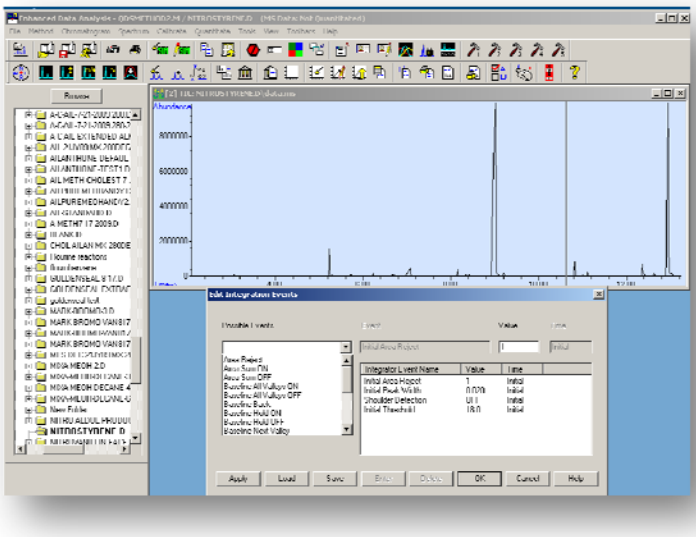

Figure 6. Integration Event Set-up.

- In order to obtain the **Mass Spectrum** for a certain peak, just right-click on the desired peak.

- In order to print a report, under **File** click on **Print** and select the desired report. The **TIC & Spectrum** option will generate a standard report with the Mass Spectrum and Chromatogram as well as peak integration information.

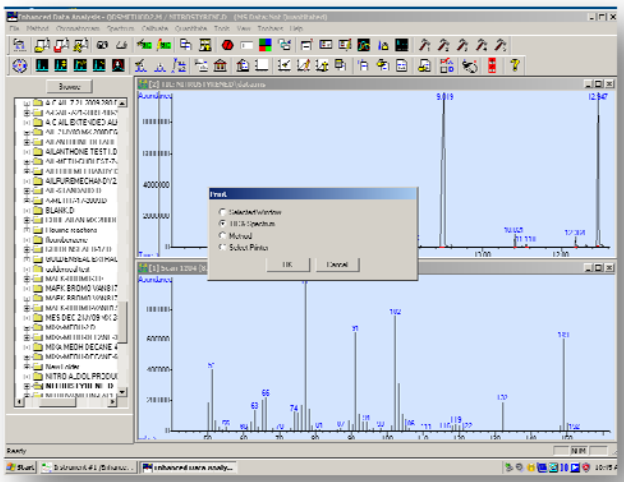

Figure 7. Chromatogram and Mass Spectrum.

FINISH:

- Once done with the experiment, always load the **Standby Method**.
- Go back to the **View** tab and select **Instrument Control** view.
- Under **Method**, click on **Load Method** and select **Standby**. This will minimize gas consumption and instrument wear will the GC/MS is inactive.