Agilent GCMS Operation Notes
Data Acquisition

The first experiment involves obtaining a series of chromatograms of a single analyte run under the same isothermal chromatographic parameters except changing the flow rate of the mobile phase from run to run.

Instrument set up (general remarks): Mobile phase is He and the He tank is always kept open with the inlet pressure set to 70 psi (tank pressure ~250psi).

Air is held at 60psi and Hydrogen is held at 40psi at the inlets of FIDs.

The instrument has two columns, two injectors (labeled front and back) and three detectors. Front injector eluate directed to the front FID only and the back injector eluate splits into the MS and the back FID.

The Program (ChemStation) that runs the instrument is invoked by double clicking, DC, the (FIDONLY) icon on the desktop for this experiment.

Next step is to load a generic method file, change parameters as needed and save it as a new method file with a new unique name.

Data Acqusition:

Load a generic method file (method files has an extension .M):
[Method] →[Load Method] →
<msdchem> → < 2 > → <METHODS> → <Chem480> → |Van Deemter Template.M| → [OK]

{Instrument Control} opens

Under Methods Section select [Edit Pencil]

(Edit Pencil) → {Edit Method} → check two boxes Method Information and Instrument/Acquisition → [OK]

{Method Information} opens → {OK}

{Inlet and Injection Parameters} – leave GC and GC ALS as is, SC [OK]

{GC Edit Parameters} opens at this point.

> Edit the current parameters in the memory using five icons starting from the left but skipping the second icon. Select the frontInjector and FID for this experiment (F). Every time a parameter is edited SC [APPLY] and at the end of all parameter selection SC [OK].
>
> Note: [Apply] makes the parameter effective right away (one parameter only).
> [OK] makes all the parameters edited thus far effective, returns to the previous menu and saves the method file; *make sure to assign a new method file name*.

SC (Injector): Select [Front Injector] tab, make sure injection volume is 1uL, leave the pre and post washes at 2 each (solvent A) and the number of sample pumps at 6, as is, SC [APPLY]

SC (Inlets): This controls the injection ports, select [SSL-Front] check (√) all boxes with Heater 225°C, Pressure ~~11.148psi~~, Total flow ~~105 mL/min~~, Septum purge flow 3mL/min, Gas saver - ON, Mode – split, Split ratio 50:1 @ ~~100mL/min~~. SC [APPLY]

SC (Column): Go down to the 4th field. Note 'flow' ⊙ is filled at some *x* mL/min in blue (only blue field is editable, everything else adjusts accordingly. *For this experiment from run to run, only x will be changed in this window*.

Verify Control Mode checked

Verify Oven Temp is 'On' and Post run temp = Oven Temperature

SC (Oven) 90°C, Hold time = 10, Run time = adjusts appropriate, SC [APPLY]

SC (Detector): [FID Front] tab, √ boxes, Heater 225°C, H_2 flow 30mL/min, Air flow 200mL/min, Makeup 19mL/mil, Const Col makeup 21ml?min, Flame, Electrometer, Subtract from signal – nothing. SC [APPLY] [OK]

{GC Detector Data}: In Signal 1 block √ Display, Attn – 4, Offset - 10%, Time - 10.0min SC [OK]

{Save Method As}: Select the proper directory ….\CHEM480\Van Deemter 2013
Give a File Name to the method just created, e.g. Van Deemter fl #.M SC [OK]

{Instrument Control} window opens

To run an analysis of a single sample with the current method file: either press ⇨ or press the (*Running Man Icon)*

{Start Run}: Check the following fields: Current method injection style (GC ALS), Inlet Location – Front, Data Path, Data File name (i.e. your output file – must be unique), Sample Name, Miscellaneous information (this is the most useful field to enter notes about the current run), Multiplier – 1, vial number (actually the position in the auto-sampler rack), √ Data Acquisition, √ Data Analysis → [OK and Run Method]

{Instrument Control} appears; note the information in the box with the arrow.

In general to run a single sample with a method file already saved:

Method →Load Method →
<msdchem> → <2> → <METHODS> → <Chem480> →<Van Deemter Flows> → |vde *INITIALS FLOWRATE*.M| → [OK and Run Method]

Notations:

{ } window;
( ) Icon;
| | file name
SC Single click left button once;
⊙ radio button
[ ] Tab on the menu ribbon, dropdown menu or button;
< > Directory;
DC Double click left button twice;
RC Single click right button once

Setting up a Sequence

From the menu bar select:

[Sequence] → [Load Sequence] → |Van Deemter.S|

Select in Sequence Section [Pencil] →Browse Method →|Van Deemter fr #.M|

Complete the spreadsheet with auto-sampler position, sample name, output file names

Save, Run

Agilent GCMS Operation Notes
Data Analysis

DC (FIDONLY Data Analysis) on the desktop.

SC [Method] → [Load Method] SC method file *xyzab*.M file in <METHODS\CHEM480\ …\>|*xyzab.***M**|→ [OK]

SC [File] → [Load Data File] → {Select Data File} → choose the Data file …|*xyzab*.**D**|→ [OK]

[File] → [Select Signals] → {Select Signals to Load} √ FIDA.ch only → [OK]

[Chromatogram] → [Select Integrator] →{⊙ ChemStation Integrator}→[OK]

[Chromatogram] → [Signal 1 Integration Parameters] → {Edit Integration Events} → select the field ' Integration ON' → type an appropriate time after solvent front elution in the time box → [Enter] → [Apply] →[OK]

{Edit Integration Events} →Save Changes? →[Yes]

[Chromatogram]→ [Integrate]

[Chromatogram]→ [Generate Percentage Report] RC on the bottom window the select [Print] →[OK]

To save the chromatogram as an Excel file: (Need a removable data storage device)
[File] → [Export Data to .CSV File] → {Select Export mode} → click ⊙ Current File – Select Destination → [OK]

{Select Data to Export} → click ⊙ Chromatogram in RO → [OK]

Save file(s) in ***your*** thumb drive with the same name as the data file in **xxx.csv** format. Change the drive to F (probably)

[File] → [Exit]