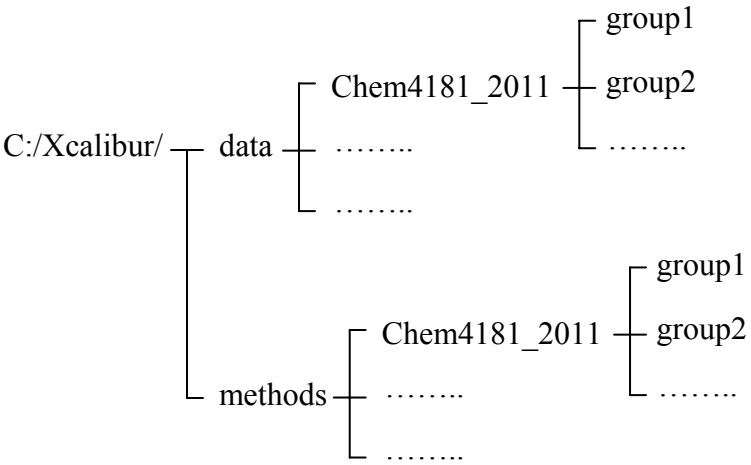
GC/MS Lab – Instructions - Central Analytical Lab – Ekeley E266

*** **<u>Most important things to follow</u>** ***

1. Set the **Start Time** to 2.5 min or longer (Page 2).
2. The **inlet and column temperatures** must be 320°C or lower (Page 2). A practical maximum temperature is 300°C.
3. **Avoid too high concentrations of samples**. Be conservative (Page 2).
4. Follow the **finish-up** procedure (Page 5).
5. Remember to **log off** (Page 5).

*** Your location on the computer:***



*** There are two programs to run the GC/MS --- "**Polaris Q Tune**" for initial instrument tuning and "**Xcalibur**" for data acquisition and processing. You should always run Polaris Q Tune before running Xcalibur.***

<u>(1) Polaris Q Tune</u>

Before running Polaris Q Tune, make sure Oven at **100°C** and Right Inlet at **150°C**.

(i) Check three parameters at bottom of display
- Source **200°C**
- Fore pressure **38-45 mTorr**
- Ion Gauge ≈ **1.5 E(-05) Torr**

(ii) Go to "**Experiment**"

→ Select "Air Water" → Filament ON → Ion Time ~ **25 msec**
→ TIC ~ **a few times E(4)**

→ Select "Full Scan" (mass range 50-650) → Ion Time ~**25 msec**
→ TIC ~ **1-3 E(4)**

→ Turn OFF Filament

> Ion Time = 25 msec is the default maximum, indicating low backgrounds. TIC (total ion current) is a measure for the detector sensitivity.

(2) Xcalibur

Xcalibur has three menus of interest on the **Road Map** homepage --- "**Instrument Setup**", "**Sequence Setup**", and "**Qual Browser**".

(i) "**Instrument Setup"** → File → Open your instrument method file (→ Modify the file if necessary.)

VERY IMPORTANT: Always use your existing method file as a template when creating a new method. **Never** create a new method from scratch!

a) **Polaris Q set up** (MS part)

Not many places to modify but…

"Start time"
- **2.5 min or longer** (for MeOH solvent)
- **3.5 min or longer** (for other solvents such as CH_2Cl_2, hexanes)

VERY IMPORTANT: Filament and MS must turn on only **after** the Start Time, i.e., after air and the solvent have mostly eluted!

"Full Scan" → Parameter → Setup mass range (up to 1,000 amu)
Low mass must be **≥ 40 amu** to exclude H_2O^+, N_2^+, O_2^+, …

b) **TRACE GC 2000 set up** (GC part)

"Oven" → Final temperature **≤ 320°C** (practically **≤ 300°C** for most applications)

VERY IMPORTANT: Column **breaks** at 340°C.

"Right SSL" → Inlet temperature **≤ 320°C** (practically **≤ 300°C** for most applications)

> a tip from the Manufacturer manual
> --- "The inlet temperature is usually about 50°C higher than the boiling point of the least volatile component in the sample".

"Split" injection: (for samples of **~100 ng/µL**)
- Split Flow = 10 mL/min
- Split Ratio = 10
  (He column flow = 1.0 mL/min)

"Splitless" injection:
(for samples of
<< 100 ng/µL)

- Split Flow = 10 mL/min
- Split Ratio = 10
- "**Split Time**" = **2.00 min**
  (He column flow = 1.0 mL/min)

> In the Splitless mode, a solenoid valve directs all the analyte into the column at 1 mL/min He during the Split Time (i.e., the first 2 minutes after sample injection). To avoid a stress on the valve, the inlet system comes back to the normal Split mode after this initial period.

"Right Carrier"

- Flow = ON. 1.0mL/min
- Constant Flow
- "Vacuum Compensation" = ON

"Aux Zone" → • MS Transfer line = 300°C (or lower)

→ Save your setup file ("___.meth") in your "methods" folder (C:\Xcalibur\methods\YourFolder)

(ii) "**Sequence Setup"** (main program to run the sample and acquire data)

<u>a) Set up parameters</u> (if you do not see some of these, just pay attention to what you see.)

"Sample Type" → Single click and pick "**Std Bracket**" from the pull down menu

"File Name" → Press **F2 twice** and open the editor

"Sample ID" Really doesn't matter. Don't change.

"Path" (data save) → C:\Xcalibur\Data\YourFolder

"Inst. Meth." → Pick your ".meth" file
(Instrument Setup)

"Proc Meth" (Ignore)
"Position" (Ignore)

"Injection Vol" → **1.0µL**

<u>b) Run the instrument</u>

Click on the row number on the Sequence Setup page → Highlight the row
→ "Actions" → "**Run Sequence…**"

At this point, the inst. set data and sequence are sent to the GC/MS instrument

On the left hand side pane, watch until:

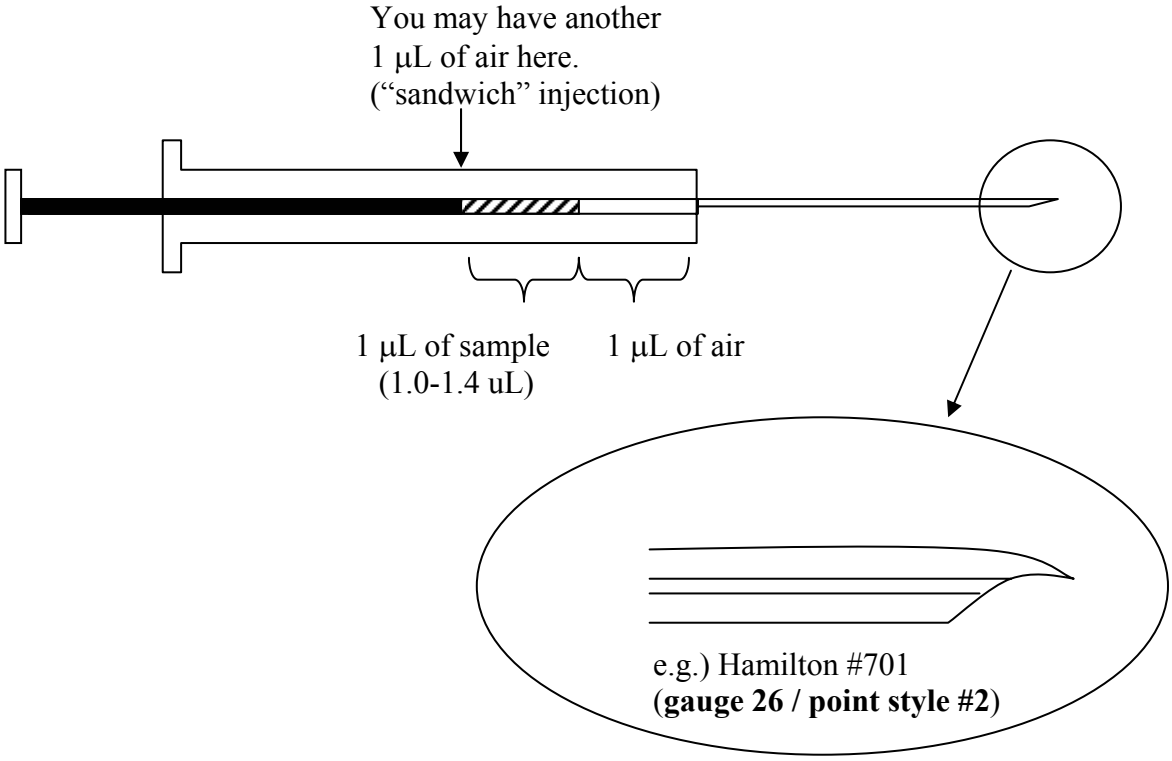
PolarisQ	→ "**Waiting for Contact Closure**"
TRACE GC 2000	→ "**Waiting for Contact Closure**"
Acquisition Queue	→ The current queue must show.

Now the sample is ready to be injected.

VERY IMPORTANT: If no injection occurs for 8 - 10 minutes, Waiting for Contact Closure disappears and the sequence gets stuck. You must inject the sample within this time period.

See Manual 3-88, 89, 91, and 92.

c) Inject 1 μL of your sample solution



- Push down the needle → Wait 2 seconds → Push down the plunger → Press "Start" button → Pull off the syringe

- To watch the data while the sequence is running, click on "Real Time Plot" icon

  Each time you work on a real-time display (chromatogram or mass spectrum), the real time plot stops.

  To resume, click on the  icon and the plot will start (either immediately or with a delay).

(This delay is annoying. As shown next, it is easier to work on an ongoing data by returning to the Road Map homepage then **Qual Browser → Right click**)

(iii) **"Qual Browser"** (on-the-run/post-run data analysis)

Manual 3-94 – 3-105 is most useful.

- To print out chromatogram(s) and mass spectra:
  File → Page Setup → Portrait or Landscape
  → Printer → Properties

- To print out instrument settings:
  Go back to "Instrument Setup" on the Road map Homepage
  → File → Print
- **NIST Library Search** → Manual 3-100 – 3-102

- **Peak Height & Area calculations** → Manual 3-106 – 3-107

(3) File manipulation

- To create a spreadsheet file for Excel:
  Qual Browser → Open Data → stay on **Chromatogram** or **Mass Spectrum**
  → Right click → Export → Clipboard
  → (PC Computer) → All Programs → Accessory → Notepad → Paste → Save as → .txt
  (→ Excel)

- To create a .tif file of a graph:
  Qual Browser → Open Data → Print
  → Microsoft Office Document Image Writer → Properties
  → Orientation (Portrait or Landscape) → Advanced → **.MDF** or **.TIFF** → Default Folder
  → OK → OK → Save

(4) Finish-up procedure

***** When done for the day, never forget to do the following things *****

(i) On the GC manual control panel:

- Go to "Oven" → Set oven temperature to **100°C**
- Go to "Right Inlet" → Set inlet temperature to **150°C**
  → Set mode to "**Split**"

  (Also set back these parameters if you have modified them:
  Split Flow → 10, Split Ratio → 10, Right Carrier/Col. Flow → 1.0)

(ii) Log off your account --- **VERY IMPORTANT**: Otherwise, you will continue to be charged.