AGILENT 5973N GC/MS OPERATING INSTRUCTIONS

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This instrument is kept under operating conditions at all time. Do not turn off or change the carrier gas flow or change any switch settings on the GC/MS.

Samples for GC/MS analysis should be fully dissolved in an organic solvent at a concentration between 10 and 300ppm (Weight/Volume). Do not inject samples directly from an NMR tube. Typically, sample concentrations applicable for NMR experiments are far too concentrated. Excessive concentration will damage the filament and result in substantial instrument down time.

Suitable solvents to dissolve your samples are: ether, acetone, methanol, dichloromethane, chloroform, benzene, toluene, hexane, ethyl acetate, acetontritile or THF.

Do NOT add water or acid to your sample. Water or acid will damage the column.

1. Press "**Ctrl**", "**Alt**" and "**Del**" together to get the log in prompt. Log in using your assigned DCIF user ID. Using other user accounts is strictly forbidden.

2. Start GC/MS operation program:
   - Click the "**Instrument #1**" desktop icon.
   - Using the pull down menus, go to "**View**" and select "**Instrument control**" (verify the option is checked)

3. Load a method:
   - Using the pull down menus, go to "**Method**" and select "**Load**", select a method from the list. Note that all method files have a .M extension (i.e. XXXX.M).
   - By default, the software will prompt you to save changes to the existing method. Always select "**No**".
   - To edit Method:.
     - The Method has four out of six editable thumbnail options buttons:
       A. **Inlets**: The injection port heater temperature may be changed here. The maximum temperature is 300^{o}C. Do not exceed this temperature and do not change any other parameters.
       B. **Columns**: Typically, no user changes are required for routine analysis
       C. **Oven → Edit GC Oven…** The oven temperature ramp rate may be edited here. The maximum ramp rate is 70^{o}C/minute. Do not change the maximum oven temperature (325^{o}C).
       D. **MS → Edit MS SIM/Scan**: The solvent delay time may edited here. The solvent delay must not be set to less than 5 minutes. The mass range may be edited by pressing the "**Edit Scan Parameters**" button. The

instrument mass *upper limit* is 800 amu. Normal Scan Range should be 50-600 m/z

- When finished, select from the pull down menus: **Method → Save**

4. To run your sample:
   - Select from the **Method → Run** pull down menus.

Important!
Enter a data file name using the correct data path in the "**Start Run**" box. The data path will have the form… **D:\DATA\GROUP\USER-ID\filename.D**
All data sets by default have the .D extension. You must save your data in your *own* data directory. Any data not in a designated user directory will be deleted. Data will remain on the instrument for approximately 6 months, after which the data will be deleted.

- Click "**Run Method**" then follow the directions displayed on screen.
- If all is well, the "**Acquisition – Prepare to Inject**" window will open.
- Push the "**Prep Run**" on the GC key panel then wait for the red "**Not Ready**" light to go off.
- Inject 1 µl (no more no less) of sample and push the "**Start**" button on the GC key panel.

Important!
When prompted to override the solvent delay click "**No**"!

Never overwrite the solvent delay time, *unless* the vacuum has gone back down below ~5.0*10^{-5} Torr for over a minute.

- Click the "**Green Arrow"** to start the run for the second sample and thereafter.
- The run may be stopped at any time by clicking on the red "**Stop**" button in the "**Instrument Control Panel**".

5. To monitor your run:
   - Click on the "**Total Ion**" thumbnail window to enlarge the view.
   - Click the "**Instrument #1 Data Analysis**" desktop icon.
   - From the pull down menus select **File → Take Snapshot**

6. To display mass spectrum:
   - From the pull down menus select **File → Load** and select an existing data file.
   - Select a peak from TIC chromatogram by holding down the left mouse button and drag the mouse to frame it then release the button to zoom in on the peak.
   - From the pull down menu select **Tools → Do Scan** then click "**OK**". This will open a Mass Spectrum window.

- Move the vertical line to the center of the peak
- Double click the right mouse button to display mass spectrum of single scan *or*
- Use right mouse button to draw a line Crosse inside the peak at half peak height to display an average mass spectrum

7. To subtract a background (mass: 207, 281, and 355)
    - First obtain an average spectrum from the target peak.
    - Move the vertical cursor to the right side of the target peak and double click the right mouse button to obtain the background spectrum.
    - From the pull down menus select **Spectrum → Subtract**

8. To calculate the Molecular Weight:
    - In the command line window (at the top of the TIC window) type MW"molecular formula"

      Example: MW"C10H12NO"
    - The press the **execute** button.
    - The calculated result is displayed below the spectrum window

9. To print the report:
    - From the pull down menus select **File → Print**.

10. Exit all of the software, close the **CAG Bootp Server**, and **log out**.

11. To copy data files onto CD:
    - Place a blank CD-R in the CD drive (E:).
    - Close the Chemstation Window.
    - Click "**Easy CD Creator**" desktop icon, choose "**Make a data CD**", and then choose "**Data CD project**".
    - Select the files that you need to copy then click "**Record**" button.

12. To review data files from CD:
    - Place the CD in CD drive
    - Click the "**Instrument #1 Data Analysis**" desktop icon
    - Go to "**File**" and select the data files from E: drive
    - From the pull down menu select **Tools → Do Scan** then click "**OK**".
    - Repeat procedure "6." for each data set desired.