

SOP for the PerkinElmer HRMS (direct injection mode)

1. Check the nitrogen level, the pressure should be around 80 psi and valve should be open.
2. Open the software “TOF MS Drive”.
3. “File” --> “Open Manual Tune”. In the list, click “Modified” to list the tune files in timely order, choose the most recently saved Tune file (with most current date stamp).
4. Click on the “Apply” button to start the tune method.
5. Let the instrument warm up for 20-30 min (While nitrogen is turned on. The heaters and Ion source buttons are highlighted in green).
*While this is happening fill both the Calibration syringe (marked by green tape) and the sample syringe with Acetonitrile to purge. Also disconnect transfer line to inject and purge with Acetonitrile and reconnect.
6. Run Calibration.
 - a) Fill the syringe with standard sample. Fix the syringe on the syringe pump.
 - b) Turn on syringe pump both on the software and pump. In the “Manual Tune” control panel window, on “Syringe pump” page, click on the grey “PUMP IS OFF” button to turn on the pump. When the pump is on, the button will turn green and said “PUMP IS ON”. (Also check the syringe pump to see if the light is on.)
 - c) Turn on acquisition. In the “Manual Tune” control panel window, click the grey button with “Acquisition is off” button, then it will turn green as “Acquisition is on”. Then you will see the Chromatogram in the “TIC” window and the current Mass Spectrum in the “Spectrum” window.
 - d) Choose the standard ions for calibration. In the “calibration” menu, select “configure”, check the standard selection. Use Agilent 1-7 as the standard ion, select three standard ions for your calibration, then click “OK”.
 - e) Run calibration. In the manual tune window, click the “Calibrate” button, the calibration result will show up after 50 spectra were collected. Click “show status” button to check the residual value. Three zeroes will be good for an acceptable calibration. Then click the “Accept” button. The calibration is finished.
 - f) Turn off the pump and acquisition.
7. Run sample.
 - a) Load sample solution in the sample syringe. Connect the syringe to the tubing, and manually flush with solution. Fix the syringe on the syringe pump.
 - b) Turn on syringe pump. In the “Manual Tune” control panel window, on “Syringe pump” page, click on the grey “PUMP IS OFF” button to turn on the pump. When the pump is on, the button will turn green and said “PUMP IS ON”. (Tip: Also check the syringe pump to see if the light is on.)
 - c) Turn on acquisition. In the “Manual Tune” control panel window, click the grey button with “Acquisition is off” button, then it will turn green as “Acquisition is on”. Then you will see the Chromatogram in the “TIC” window and the current Mass Spectrum in the “Spectrum” window.
 - d) Save data. When you see the peak for the target molecule appear in the Spectrum window, click the grey button with “Spectra Saving is off” button in the “Manual Tune” control panel window,

then it will turn green as “Spectra Saving is on”. Then you will see the number after the “Saved Count” starting counting. Save 50-100 data points for each sample. Then click on the “Spectra Saving is on” button to stop the data saving. Then turn off the pump and turn off the acquisition. When you turn off the acquisition, there will be a punched-out window to set the directory of the data file. The file is named systematically and cannot be changed in this step. You can add the sample information in comments and use the comments to identify which file is for which sample. You can also change the file name afterwards.

- e) Purge the syringe and load next sample.

8. After the experiment:

- a) Turn off the acquisition and the syringe pump.
- b) Flush the syringe with clean solvent (Acetonitrile or methanol). Fill the syringe with pure solvent and turn on the syringe pump to flush the ESI probe.
- c) Turn off the syringe pump.
- d) Leave the manual tune page and the software open. DO NOT save the manual tune file.
- e) Clean the bench surface and record on the login book. If you are the last user of the day, you will also need to turn off the instrument as follow:
- f) Turn off the gas and heating by click the green button of “turn off the gas and heating” on the bottom of “ion source” panel.
- g) Turn off the voltage by click the green button of “turn off voltage” on the bottom of the “ion source” panel.
- h) Turn off the valve on the liquid nitrogen tank.

9. Data analysis:

- a) Click on the frame of the software to active the main menu.
- b) File -> Open Data File -> choose the file and open.
- c) There will be two windows opened for the data file, one for TIC and one for spectrum. It automatically opens the mass spectrum for the first data point in the data file. Right click on TIC window and choose “Mark Data Points”. Then all the points saved in this file will be displayed. Click on the selected point and the mass spectrum for this pointed will be displayed in the Spectrum window. Left click and select a range in the TIC window, click “average selected range”, a new spectrum window will be opened to display the average spectrum for the selected data points.
- d) Read the numbers in the Spectrum window and calculated the tolerance. If the error is larger than 5ppm, take a new calibration and rerun your sample.
- e) Print Screen to save the spectrum as a picture. Or right click and Export the XY data plots in Clipboard and save it as .txt file. The picture file and the .txt file should be saved under “Documents” folder, which is uploaded to the data server, and can be downloaded when you are using the network within the chemistry department.

Server address: 128.32.246.254 (shared data folder: catalytic center data)

Username: catcenter

Password: c@t@lys1s