STANDARD OPERATING PROCEDURE
Gas Chromatography-Mass Spectrometry

HOW TO CHANGE COLUMN:

A) FOR SWITCHING FROM GC TO GC-MS:

1. All gas valve and mains switches of instrument must be **OFF**.
2. Remove columns from oven with the help of wrench kept in Tray.
3. Take column hanger for GC-MS and insert its end in two holes at the upper wall of oven and select a GC-MS column to be inserted.

4. Locate the column end with the **shorter length protruding beyond the ferrule and nut** and connect it to the **outlet of the autosampler**.
5. Locate the column end with the **longer length protruding beyond the ferrule and nut** and connect it to the **inlet of the MS detector**.
6. Secure all connections by tightening all fittings carefully to ensure leak-free connections, avoiding over-tightening.
7. Confirm the column is installed in the correct flow direction and that all connections are secure & positioned properly without strain or sharp bends.
8. Now Switch ON instrument and required gases. SWITCH ON pc and open GC-MS software and load a GC-MS method after vacuum is achieved.

B) FOR SWITCHING FROM GC-MS to GC:

1. All gas valve and mains switches of instrument must be **OFF**.
2. Remove column hanger and columns from oven with the help of wrench kept in Tray.
3. Take GC columns and Hold the "column 1" so that the printed details on the label face toward you and place it on the hooks at the back side wall of oven.

4. Connect the back end of the column to the port labeled **"Column 1"** on the **left** side.
5. Connect the opposite end of the column to the port labeled **"Column 1"** on the **right** side.

6. Do the same for "column 2" in the port labeled for **"Column 2"** and place it on the hooks of column 1.
7. Ensure all connections are tighten & secure and that the column is positioned properly without strain or sharp bends.
8. Now Switch ON instrument and required gases. SWITCH ON pc and open GC software and load a GC.

FOR GC-MS RUN

1. Open **He gas** valve and monitor pressure (75psi).
2. Switch on the power button of instrument and PC.
3. Power on GC by front switch.
4. Power on MS by front switch (red light appears). MS vacuum pump starts.
5. In PC, Double click on GC-MS. Acquisition window open.
6. Wait till proper vacuum condition (as per instrument parameter) or MS button appears green from red.
7. After some time a window appears to increase MS QUAD and MS SOURCE temperature. CLICK on apply.
8. CLICK on CLOSE window if temp of MS quad (150°C), MS source (230°C) is increased.
9. Now go to LOAD METHOD and select GC-MS SCAN method →OPEN. Temperature of MSD interface (250°C), ALS (250°C), increases.

Here **CONDITIONING OF COLUMN** can be done after every **40-50 sample** run by following steps.

　　　SKIP this process if conditioning is not to be done and go to **step 10**.

1. After instrument reaches vacuum , Go to GC screen → METHOD → OVEN → insert values of initial temperature and hold time . Slowly increase temp of oven with different ramp rate. (Temp must be below column's highest temperature)
2. Insert post run temp 70 or 80 degreee.
3. Click on PLAY button at the bottom of the screen.

4. Conditioning is done if temp of oven reaches equal to post run temperature.

10. Go to VIEW → TUNE AND VACUUM CONTROL for Tuning of MS. (To be done daily before sample running).
11. Go to TUNE--- many tune program appears → click on EXTRACTION SOURCE TUNE. Tuning starts and tune file will be generated. (takes 2 – 5 min)
12. After completion a window pop up to save the current tune file. Save it there with current date.

Tune report can be checked by going to DESKTOP → ETUNE → click on Tune file of current date (HEALTH REPORT OF MS).

If BASE PEAK, No of PEAKS, AIR/WATER CHECK and EM VOLTS is OK then we can run the sample.

OTHERWISE, **BAKING of MS** is to be done.

For Baking , Go to VIEW → TUNE → EXECUTE → BAKE OUT MSD → Fill Temp (less than limit) and Time (1/2 to 6 hours) and wait.

Sample Run procedure

13. Upload prepared sample vials in ALS locations.
14. Go to LOAD METHOD → select required METHOD for user (OR we can create new method according to user) → click OPEN.

   FOR SINGLE SAMPLE RUN
15. Click on SINGAL RUN (green arrow). A window appears to fill sample details and its location in ALS.
16. Fill FILE NAME and SAMPLE NAME and vial LOCATION.
17. Click on OK AND RUN METHOD.
    Run time appears on screen, sample running starts.

    STOP the run if desired peak appears by clicking on **RED STOP** button.

FOR MULTIPLE SAMPLE RUN

1. Click on NEW SEQUENCE → Make sequence of all samples by inserting next row and filling their details (sample name, method path, method file, data path) and exact location.
2. Click on OK AND RUN METHOD. Each sample runs one by one.

DATA ANALYSIS

1. Go to **DATA ANALYSIS** icon on desktop.
2. Left side panel , click on BROWSE DATA.
3. Search data file by its path and name and click on the file to open.
4. Go to FILE → PRINT → TIC AND SPECTRUM → OK. pdf file is saved.
5. To get spectrum of specific peak, right click on peak and follow step -4.
6. For Library search, Go to SPECTRUM → NIST Search. Library for particular peak appears.

SHUT DOWN METHOD FOR GC-MS

1. Go to METHOD → LOAD cooling method and wait until temp of ALS and MS interface becomes less than 100°C.
2. Now click on MS vacuum control → VENT.
3. WAIT until it shows VENT CYCLE IS COMPLETED. (Takes 45-60 min).
4. After that switch off MS and GC from front switch and shut down PC.
5. Shut down He gas valve and switch off all main switches.

FOR GC RUN

1. Open **Argon, Nitrogen, Air and Hydrogen gas** valve and monitor pressure (75psi) in each.
2. Switch on GC and PC from main switches.
3. Switch on GC from front switch.

4. OPEN GC software in PC.
5. Go to METHOD → load method. Select GAS MIX METHOD and load it. Wait until flame ignites.
   Now system is ready to run sample.
6. Click on SINGAL RUN (green arrow). Fill FILE NAME and SAMPLE NAME and vial LOCATION.
7. Click on OK AND RUN METHOD.
8. Now INJECT the sample with gas tight syringe or gas sample bag through the gas sample inlet.
9. CLICK **RUN** symbol on GC screen.

SHUT DOWN METHOD FOR GC

1. Go to METHOD and load STANDBY METHOD.
2. After 10 min switch off GC and SHUT DOWN PC.
3. SHUT DOWN all gases valve and switch off all main switches.