Gas Chromatograph Mass Spectrometer Standard Operating Procedures

Last modified on: July 13, 2018 by Mark Redd

- **NOTICE: Lab policy requires that any person performing GCMS measurements must have done the following before performing any experimental work:**
  - **Complete pertinent laboratory safety training**
  - **Read this SOP in its entirety**
  - **Become familiar with all the experimental steps outlined in this SOP**
  - **Sign and Date the GCMS SOP Signatures Sheet**

Contents

1 Start up Procedures

- Ensure that HiVac is not lower than $1.0 * 10^{-6}$ and not higher than $3.5 * 10^{-5}$

2 Tuning the GCMS

2.1 Tune MSD should be done weekly

2.2 Quick Tune should be done daily

- Save as pdf $\Longrightarrow$ TuneCreationDate

- Creation Date is listed, just copy and paste it in File Name
  - Ex: Tune20151103091307
  - Save on the Desktop in Folder "MS Tune Reports"
  - In pdf files, ensure Relative Abundance for Mass 502 is above 3% and roughly the same as the day before

3 Edit Method $\Longrightarrow$ This is where you tell the GCMS how to run (flow rates, temperatures, ramp rates, sample size, etc.)

- This process is where the "art" of GCMS work comes in. Use p. 33-37 of black binder for MSD to determine how to change the method to get smoother/more resolved peaks.
- Click pencil icon underneath "method"
- Everything on the left hand side should read "Ready" except GC Ready State
- Oven Icon
  - Choose duration of temperature holding a ramp rate
- CFT Icon
  - Choose Control method (flow rate or pressure) and specify
- Inlets Icon
  - Use the Back Inlet Tab
  - Total Flow can **never** be above 1250 mL/min
  - Split ratio can be adjusted as long as Total Flow is not too large
- ALS Icon
  - Choose injection volume
    - The large the molecule, the smaller injection volume
    - Solvent Washes
      - Always want at least one wash for Solvent A (Acetone) both Pre and Post Injection to keep the needle clean. If necessary, use Solvent B (Methanol).
    - Click **OK** until you reach **Save Method**
    - Save method as **CompoundName.M** in Compound Folder

4 Single Runs

- Start the Run
  - Click the big green arrow
  - Change the operator intiials
  - Ensure correct data path
  - File Name

        * If you have changed something in the method, include that in the file name
            · Ex: NOV 2 HEXYLCYCLOHEXANE 5 – 190c, 25KMIN, SPLIT.D

- To Do the Next Run
    - Repeat Procedure 5
    - If you are changing the method in any way, you will also need to repeat Procedure 4
- Run Several **BLANK** runs between each sample run to flush out any residual sample (simply use an empty vial)
    - When running blanks, label the runs around the sample runs:
        * [Date] Blank 1aa (the first "a" means "before the sample run", the second "a" means "the first blank done")
        * [Date] Blank lab
        * [Date] [Sample] 1
        * [Date] Blank 1ba (the "b" means "after the sample run")
        * [Date] Blank 1bb
        * [Date] Acetone 1 (to clean everything out)
        * [Date] Blank 2aa
        * [Date] Blank 2ab
        * [Date] [Sample] 2...

5 Multiple Runs (Sequence)

- Edit Sequence
    - Click the pencil icon underneath "Sequence"
    - Keep data path the same as the method path (same folder)
    - Choose Type (Sample or Blank), Vial position, and Sample Name
    - Method should be spelled the exact same as the **.M** file used (CompoundName)
- Running Sequences
    - Include the desired number of runs in the Edit Sequence step
    - To run, click the running man underneath "Sequence" instead of the big green arrow, then click "Run Sequence"

6 Analysis

- Purity
    - Tmplibrp.txt file includes acetone in purity, so use rteres.txt file to compute actual purity
        * Add up the corrected peak areas excluding acetone and divide each peak area by the total
- Compound identification
    - Open AMDIS from the Desktop

* Open the file you wish to analyze
* Click Run near the top of the window
* In the top graph, zoom in on a peak
* Click on the top of the peak
* Analyze $\Longrightarrow$ Go to NIST MS Program
* Right center graph compares MS data (red) to known compound MS (blue)
    - Use tabs below to select different comparisons
* Lower left list gives compounds that are similar to collected MS
    - Prob. Column gives probability of a match
* On the bar graph, the farther to the left the red bar is, the better the math
* Bottom graph gives structure of selected compound