

# 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

<b>1</b>	<b>Start up Procedures</b>	<b>1</b>
<b>2</b>	<b>Tuning the GCMS</b>	<b>1</b>
2.1	Tune MSD should be done weekly . . . . .	1
2.2	Quick Tune should be done daily . . . . .	1
<b>3</b>	<b>Edit Method <math>\Rightarrow</math> This is where you tell the GCMS how to run (flow rates, temperatures, ramp rates, sample size, etc.)</b>	<b>2</b>
<b>4</b>	<b>Single Runs</b>	<b>2</b>
<b>5</b>	<b>Multiple Runs (Sequence)</b>	<b>3</b>
<b>6</b>	<b>Analysis</b>	<b>3</b>

## 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  $\Rightarrow$  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 $\implies$ 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 larger 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 initials
  - 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 1ab
    - \* [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  $\implies$  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 match
- \* Bottom graph gives structure of selected compound