

Shimadzu GCMS-QP2020 w/ AOC-6000 Operating Procedure



Rochester Institute of Technology

Department of Chemistry and Material Science

SOP prepared by Robert Winter and William Charbonneau on September 14, 2018

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I. Purpose

To promote the effective use of the Shimadzu GCMS-QP2020 along with the Shimadzu AOC-6000 auto sampler to establish an instrumental method.

II. Scope

This SOP is intended for in-group use by trained and certified personnel in the Chemistry Department

III. Prerequisites

The experimenter must be trained in proper instrument techniques before using this SOP.

IV. Responsibilities

The responsibility for this instrument lies with Tom Allston

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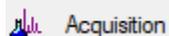
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V. Procedures and Information

1. Ensure that both the GCMS unit and vacuum pump are turned on and pumped down, if not or unsure, please seek assistance from Tom Allston. Be sure Optic 4 unit on right side of instrument is also switched on. (Disregard except for after known power outages or scheduled instrument maintenance)
2. Log onto computer using RIT login credentials.

3. Connecting and startup of the instrument
 - a. On the computer desktop, open both the “GCMS real time analysis” and “Evolution” programs. (Evolution is used to run the Autosampler)
 - i. The login credentials for “GCMS real time analysis” are:
 Username: Admin
 Password: *None*
 Then click enter

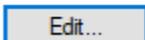
4. Editing instrument parameters/Building a method
 - a. Open “GCMS real time analysis” tab
 - b. Open “Acquisition” tab in the bottom left corner



- c. In “Sampler” tab, select the “Edit...” button underneath AOC-6000



AOC-6000



- i. Choose the “Methods” option (icon picture depicting 3 flasks) and then select the option for “Liquid Injection”. Then click “OK”
- ii. In the “Setup” settings:
 1. Select the “Value” for Gas Chromatograph as “GC1”
 2. Set both Pre and Post Wash Station to “Standard Wash 1”
- iii. In the “Analysis” settings:
 1. Set the “Value” for Syringe Tool as “LS1”
 2. Set the “Value” for Injector to “Injector 1”

NOTE: The number of Pre and Post Wash Cycles as well as the Pre and Post Wash solvents should be determined by the user and optimized based on his/her experiment. *Be sure the rinse bottles are MINIMUM half full*

- iv. Once all settings have been selected/optimized (no red warnings), hit “Apply” and close out of the tab.

- d. In “GC” tab:

Inj. Port : SPL1 Inj. Heat Port : INJ1
 Column Oven Temp. : 150.0 °C
 Injection Temp. : 250.0 °C
 Injection Mode : Split
 Sampling Time : 1.00 min
 Carrier Gas : He Prim. Press. : 500-900
 Flow Control Mode : Linear Velocity
 Pressure : 35.8 kPa
 Total Flow : 5.0 mL/min
 Column Flow : 0.50 mL/min
 Linear Velocity : 26.9 cm/sec
 Purge Flow : 2.0 mL/min
 Split Ratio : 5.0

Rate	Final Temperature	Hold Time
0	150.0	0.00
1	0.0	0.00
2	0.0	0.00
3	0.0	0.00

Total Program Time : 0.00 min
 Column Name : RTX-1MS Thickness : 0.25 um
 Length : 30.0 m Diameter : 0.25 mm

- i. “Column Oven Temp.” should be set to the desired starting temperature for the run
- ii. “Injection Temp.” should be set to 200decC minimum (or higher than highest temperature achieved during the run)
- iii. “Injection Mode” is set to “Split”
- iv. “Flow Control Mode” is recommended to use “Linear Velocity” (keeps the flow constant by changing pressure)
- v. Set “Split Ratio” at 20 to start (can be changed up or down to enhance the peak shape)
- vi. Edit the “Column Oven Temperature” program to fit desired experimental results
 1. Rate is an increase/decrease of temperature per minute (°C/min)
 2. Final Temp. is desired stopping temperature
 3. Hold Time is how long the Final Temp. is held

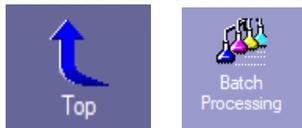
e. In “MS” tab:

GCMS-QP Series
 Ion Source Temp. : 200 °C Ionization Mode : NCI SCI SEI
 Interface Temp. : 250 °C
 Solvent Cut Time : 2 min Detector Voltage : Relative to the Tuning Result Absolute
 Micro Scan Width : 0 u 0 kV
 Use MS Program : Set... Threshold : 1000
 Group#1 - Event#1 GC Program Time : 0.00 min

	Start Time (min)	End Time (min)	Acq. Mode	Event Time(sec)	Scan Speed	Start m/z	End m/z	Ch1 m/z	Ch2 m/z	Ch3 m/z
1	3.00	10.00	Scan	0.50	666	40.00	350.00			
2	0.00	0.00	Scan	0.00	0	0.00	0.00			

- i. Set the “Start Time” for when the Mass Spec is wanted to start analyzing for ions (usually any time after approx. 2 minutes)
- ii. The “End Time” is determined in the “GC” tab based on the temperature program that is made, be sure to enter that same time (time given under the plot of temperature vs. time)
- iii. Adjust the Start and End m/z to ensure the ion(s) of interest is/are being analyzed by the Mass Spec
- iv. The “Event Time” is how fast/often data points are being collected to form the Mass Spec plot

- v. "Ionization Mode" should be set to "SEI" (scanning electron impact)
 - vi. For "Detector Voltage", select the option for "Relative to the Tuning Result"
- f. Once all parameters have been optimized for the given experiment, go to "File" and select "Save Method File As...". Be sure to save the method in the appropriate file location for your class/research group.
5. Building a batch/running an experiment
- a. Select the icon for "Top" from along the left-hand tool bar, then select the icon for "Batch Processing"



- i. Here is where the number of samples/how many times a given sample is to be run may be edited.
 - 1. Include the location of the sample in the Auto sampler (vial and tray number) and the name for the sample
 - 2. In the "Method File" cell for the sample, click on the blue arrow and locate the desired method file for the experiment that was made earlier or saved previously
 - 3. In the "Data file" cell for the sample, click on the blue arrow to specify where the data is to be saved to and under what naming convention
 - 4. Once batch table is created, go to "File" and select "Save Batch File As...". Be sure to save the batch file in the appropriate file location for your class/research group
- b. Before the run is started, re-open the "Evolution" program
 - i. Select "File", then "Open...", then open the "Standby" folder, then select the "Standby.mth" file
 - ii. After, select "Sequence", then "Execute", then select the "StandbyRun.seq" file
- c. Reenter the "GCMS Real Time Analysis" software and select the icon for "Start" to begin the run

