Standard Operation Procedure Agilent 7890B GC-MS system



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1. PC Workstation Access.

Username = admin

Password = 3000hanover

2. Hardwares.

The hardware of the GCMS system includes three major parts, the GC, the MSD, and PAL3 autosampler. Gases connected to the system are, He as the carrier gas, H2 and Air for FID detector.

- 3. Gases (Air, H2, and He) must pressurize the line otherwise flows, pressures, and temperatures will need to be turned back on as prompted on the screen of the GCMS.
- 4. Precautions
 - a. DO NOT turn off the power button on the MS detector without venting the system.
 - b. DO NOT expose the end of the column in air without a stopper.
- 5. Sample preparation.
 - a. The autosampler supports standard size 2ml GC vials and 1ml glass wells in 96 well plates.
 - b. Samples can be prepared in most organic solvents. Water, inorganic acid, inorganic base, and involatile salts should be avoid in GCMS samples.
 - c. Too much Chloride in the sample should be avoided
 - d. Samples should be pre-filtered before bring in for analysis.
 - e. Recommended sample concentration range is from 15ug/ml to 10mg/ml.
- 6. Method preparation.
 - a. Load a template method with "Method" -> "Load Method".
 - b. Modify the template method with "Instruments" options. The setup of the method includes "Inlet", "Autosampler", "GC" and "MS". Go through these setups and save the method as a new method.
 - c. On first Method parameters page edit:
 - i. Sample volume (based on the volume you want to inject for analysis)

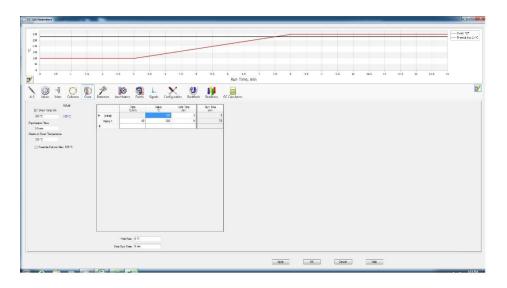
ii. GC cycle time based on calculated sample times (ramps+holds)

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Basic					pt Modified, Filday, Sept	ember 26. 2014
Tool	L51	1		Se	ript Description tot for standard GC Upp	id injection.
Sample Volume		2	ut.	w	Fuse 'Standard Wash' f	or all washes.
Wait for System Ready	After solvent wash					
Wash Vial Depth		10	mm			
Waste Port Depth		10	mm			
Bottom Sense	Off					
Height From Bottom of Sample Vial		0.5	mm			
Cooled Stack	none					
Cooled Stack Temperature		20	NC .			
GC Cycle Time		5	mn			
Pre Injection						
Syringe Fill For Wash		80	5			
Wash Aspirate Flow Rate		5	uL/s			
Washes with Solvent 1		2				
Washes with Solvent 2		0		-		
Washes with Solvent 3		0		De	(aut Parameters	
Washes with Solvent 4		0			Information	
 Sample 				Nat US1	Head	Type 10
Sample Vial Penetration Depth		30	mm			
Sample Vial Penetration Speed		50	mm/s			
Sample Washes		1				
Sample Wash Volume		3	uL			
Filing Strakes		4				
Fill Strokes Volume		4	ui			
Filling Strokes Aspirate Flow Rate		5	uL/s	4		

d. On the single Quadrupole MS Method Editor, under timed event be sure there is solvent delay to protect the filaments. Please use default unless you see that your solvent is evaporating after the delay then add a period where the detector is off then back on.

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Tune Typ	pe	EI			Solvent Delay		3.00	0 min		*		Time	Туре	-		-
Tune EM	VN	1200			Detector Setting									1.		
CI Gas V	/alve				Trace Ion D											
CI Flow				%	EM Setting	Gain Facto	or	-								
			stpoint		Gain Factor	1.	000									
MS Soun		Offline	230	Apply	Applied EM Volt	age (V) 50	00									
MS Quad	d	Offline	150	Leev .	EM Saver											
cquisition	Time	Scan			Limit	Sum Limit	1e8 (Defaul	t)	Ŧ							
an Time Se		Concentration		•												
					0 0 11 13	Freque	ncy C	Cycle Time	Step Size							
		Start Mass	End Mass	Threshold	Scan Speed (u/s)	(acdita.	/sec) (r	Cycle Time ins)	Step Size (m/z)							
	3.00	50.00	End Mass 500.00	Threshold 150			ncy C /sec) (r 3.2	Cycle Time ins) 310.63	Step Size (m/z) 0.1							
M Time Se	3.00 egments	50.00	500.00	150	1,562 [N=2	Cucle	/sec) (r 3.2	ms) 310.63	(m/z) 0.1							
M Time Se	3.00 egments Time	50.00	500.00	150	Total Dwell Time (ms)	Cycle Time (Hz)	/sec) (r 3.2 Resolu	me) 310.63 tion Gain Factor	(m/z) 0.1							
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e. On the GC Parameters page under "Oven", specify your ramp rates and hold times. All other parameters should be standard.



- 7. Edit sequence.
 - a. Use "Sequence" >"Load sequence" to load an existing sequence, then use "Sequence">"Edit sequence" to open the sequence setup page.
 - b. Add the sample information into the sequence table. Click "OK" to quit the sequence editing.
 - c. Use saved Method.
 - d. Specify data location MassHunter/GCMS/1/data folder
 - e. Use "Run sequence" button to submit the sequence to run the sequence from the first sample, or use "Sequence" I "Position and Run sequence" to starting running the sequence from the middle of a sequence.
 - f. When a sequence is running, use the "Edit" button in the status bar to get into the sequence setup page and add new samples or insert samples into the running sequence.
- 8. Data analysis.
 - a. All the raw data should be saved under D:/MassHunter/GCMS/1/data folder. This folder will be uploaded to the server and the data will be available online.
 - b. Use "MassHunter WorkStation" for data analysis. Use "File" "Load" to open the raw data. The TIC trace will be displayed in the "Chromatogram" window. Double click on a time point, and the MassSpec of this time point will be displayed in the "Spectrum" window.

- c. Right click and use "integrate chromatogram" for automatic integration, or use the "Manual Integration" tool to add peaks manually. Right click and select "Integration Peak List" to view the integration result.
- d. Use "File" ⊕ ③ "Report" ⊕ ③ "Print Report" to generate a report file in .pdf or .csv.
- e. Right click on the chromatogram or spectrum window, use "Export" to create the data table file.
- 9. Instrument reservation.
 - a. Users will get access to the instrument calendar after taking the instrument training. The schedule of the instrument will be displayed on the calendar, and users can book the instrument time on the calendar.
 - b. The maximum time for one reservation is for one whole 96 well plate. Between two plates, users must leave at least 8 hours for other users.
- 10. Data server information.

To connect to the catalysis center data server:

In the explorer window, display "this PC", then choose "add a network location", Input "\\128.32.246.30\catalytic center data" when you are asked for the network address. (If you are using a Mac, there is a similar setup in the Finder.)

Username: catcenter Password: c@t@lys1s

Trouble shooting:

If PALS system is acting up:

- 1. Shutdown mass hunter
- 2. Turn PALS system off (unplug)
- 3. Disconnect and reconnect all PALS connections x 2-3
- Disconnect and reconnect all PALS remote connections x 2-3
- 5. Then plug back in the PALS and start software
- 6. If there is an error message after restarting record message