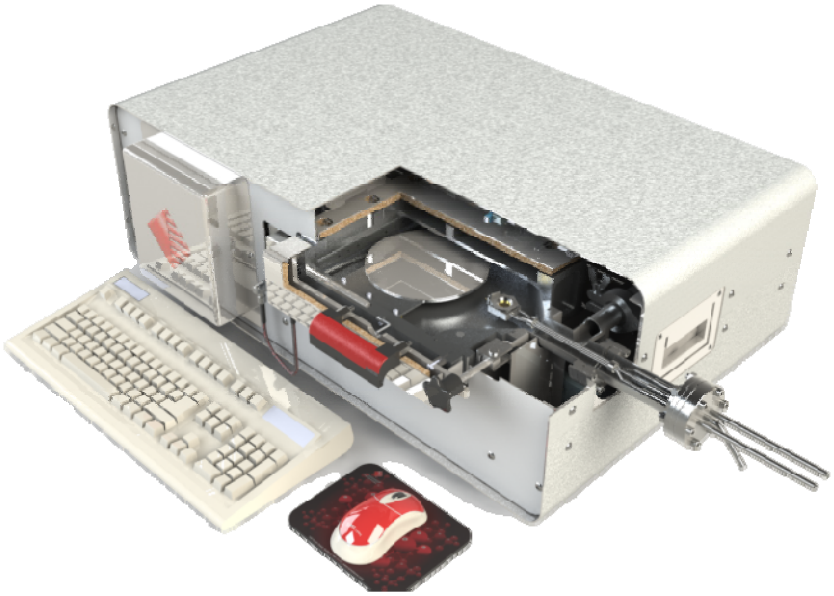
ARRADIANCE®

GEMSTAR™
BENCHTOP ALD SYSTEM

GEMSTAR Operation Manual



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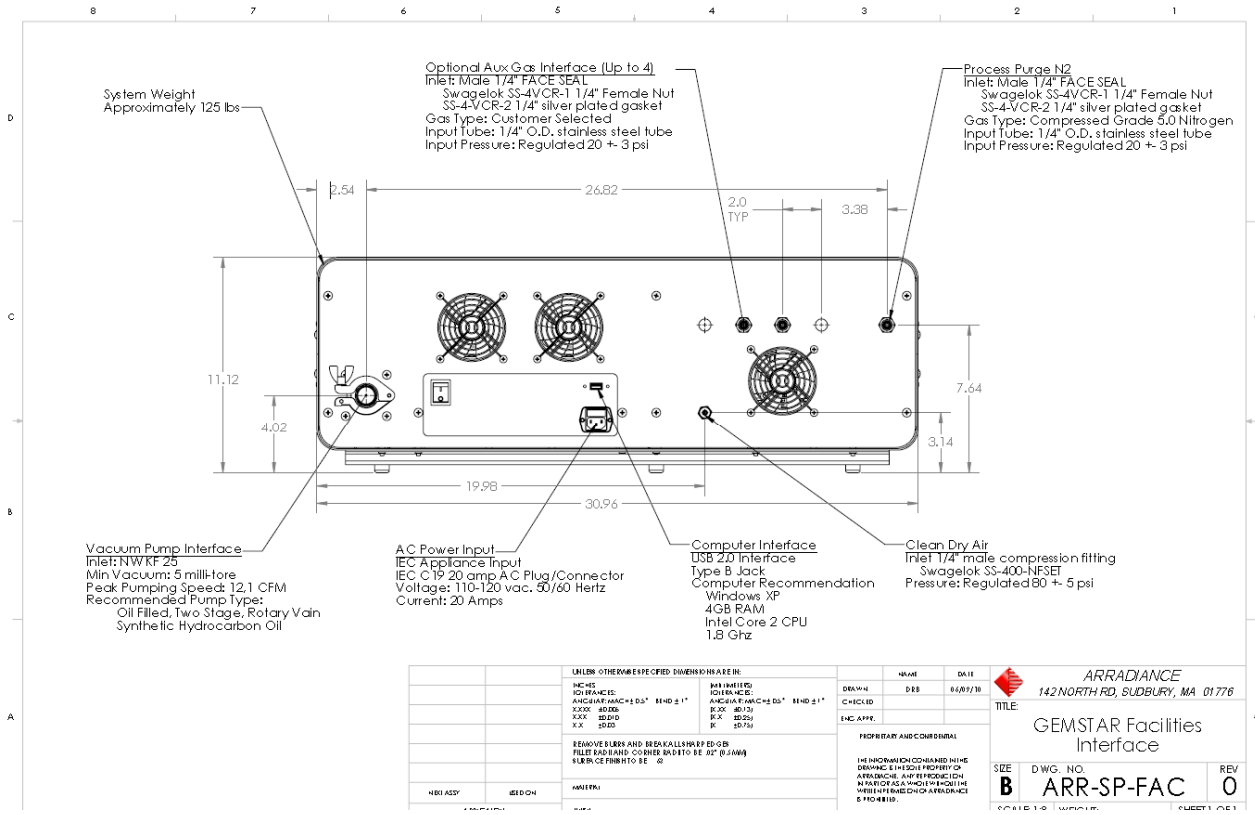
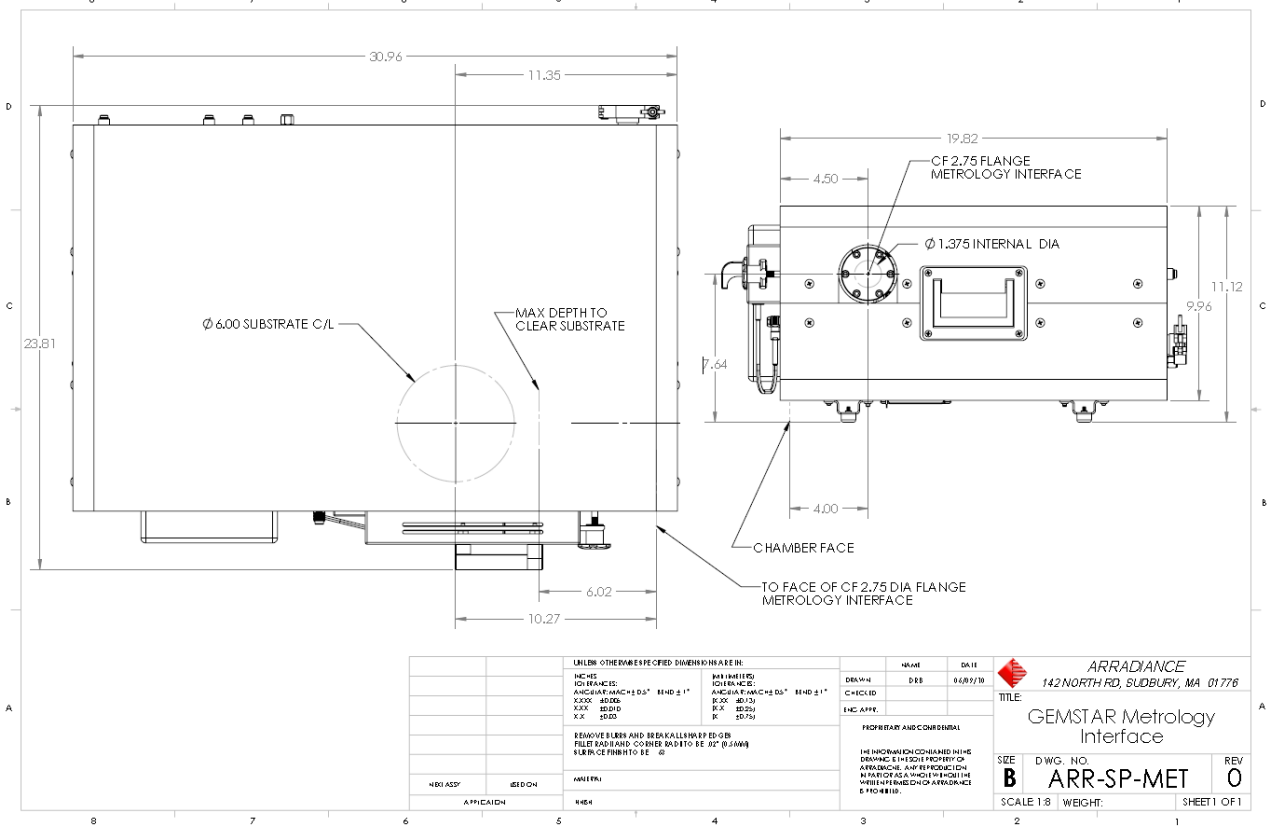
What Comes in the Box

1. GemStar Benchtop ALD system
2. Dell Vostro laptop with Windows 7 and GEMFlow Software
3. Dual power cable
4. USB cable
5. 1 KF 25 cap ,centering ring and clamp
6. 1 ¼" fitting with ferrule for CDA line
7. GemStar manual

Facilities Interface Drawings





How to Set up the System

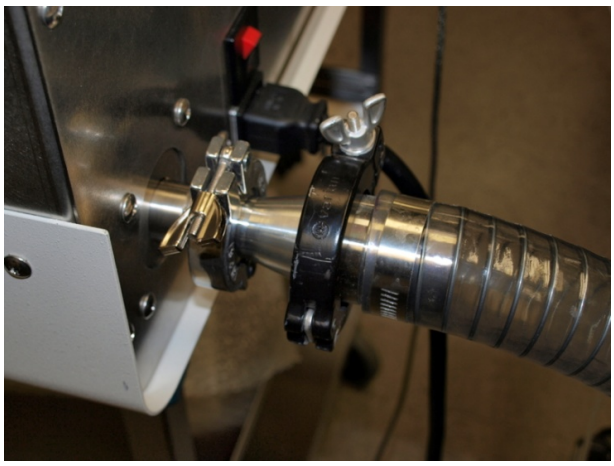
1. Using two people carefully lift the GemStar system out of the crate and onto the bench where it will be used.

Note: The system weighs ~125 lbs, so use appropriate two-person lifting techniques to extract from crate and position.

Note 2: The system will be shipped under partial vacuum. Assuming the following set-up procedures are followed than the system will remain unexposed to ambient air, which is preferable.

2. Begin installation by connecting the vacuum pump inlet to the chamber vacuum port at the back of the system.

Connection size is NW-25. The pump which should be rated 12 cfm or higher can be connected to the system using NW-25 (1") or NW-40 tubing (1.5"). The use of NW-40 tubing will require the addition of an NW-40 to NW-25 reducer in order to be compatible with the GemStar vacuum port (see photo below).

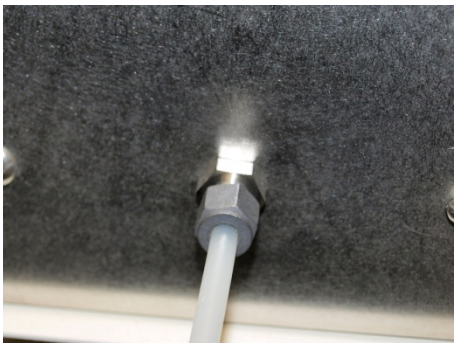


3. Install a compressed dry air (CDA) source near rear of chamber. Turn air off until connection with system is complete

a. Connect a ¼" Teflon or stainless steel line to source using appropriate fittings

b. Connect line to back of chamber using Swagelok fitting present on the rear connection. Tighten connection.

c. Turn CDA on and check for leaks at newly formed connections.



4. Bring nitrogen gas to back left (when facing) of system using ¼" stainless steel line equipped with 1/4" female VCR fitting.

   a. Nitrogen should be ultra high purity (>99.999%)

   b. Nitrogen should be regulated to 20 ± 3 psi

5. Using provided plug, connect GemStar system to a 20amp rated outlet

6. Turn on main power of GemStar system

7. Turn on laptop computer but do not start GemStar software

8. Connect the provided USB cable into GemStar system and then into an available port on the computer.

9. Start the GemStar software.

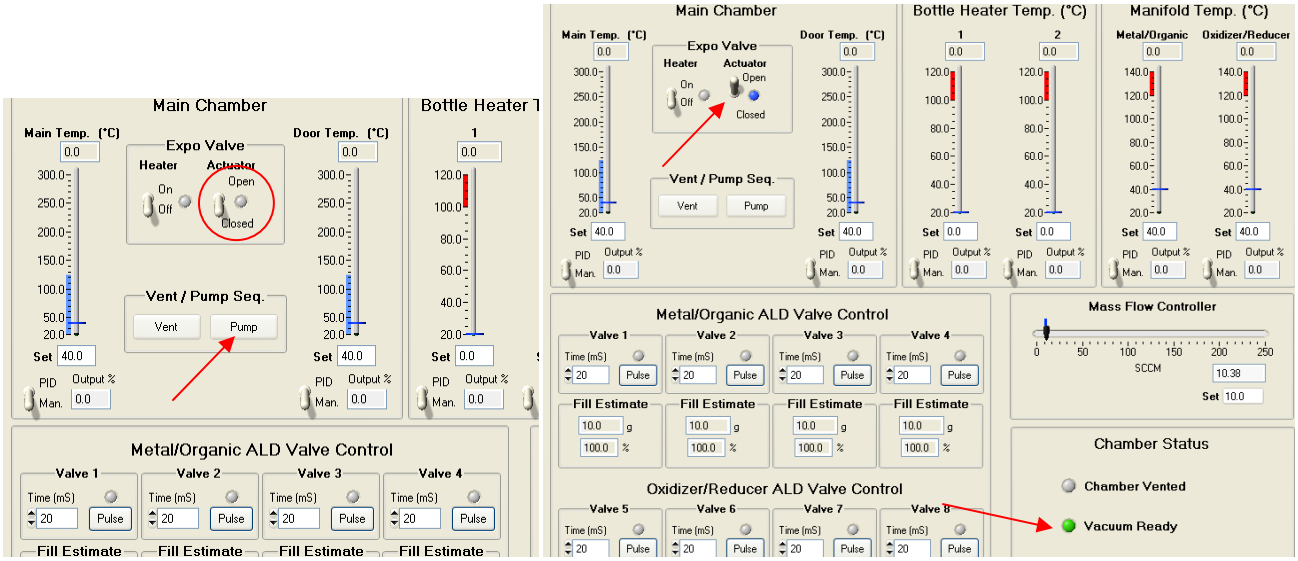
10. Confirm, once again, that all VCR and Swagelok connections that have been made are secure and tight.

11. At this point the system is ready to be pumped down.

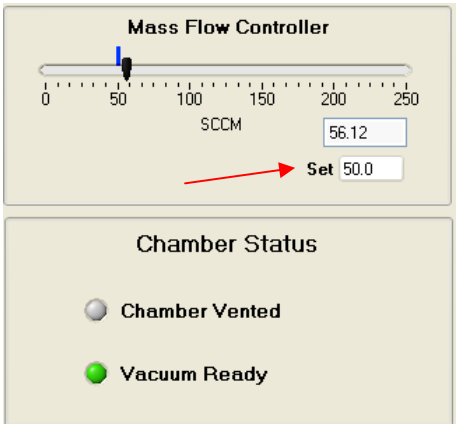
12. The door will be closed and ready for system pump down. Assuming it was not opened at some point. Please close door if open.

13. Turn on pump

14. Click "pump" to pump down system. After a successful pump down, actuator will be open and vacuum ready light will be lit.



15. Set MFC flow to 50sccm

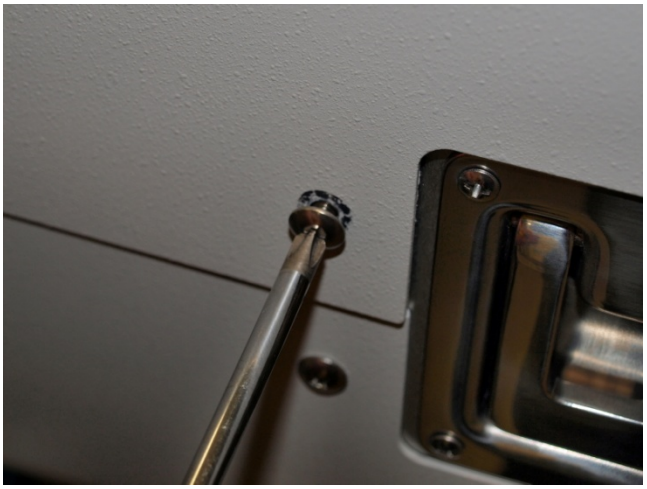


16. If the chamber is at room temperature and under vacuum, the door will not open after running the vent sequence. Please note, in order to vent chamber the system temperature needs to be greater than 60°C.

Precursor Bottle Removal/Installation

1. To begin installation of the precursor bottles you need to remove the top cover to expose the two gas manifolds.

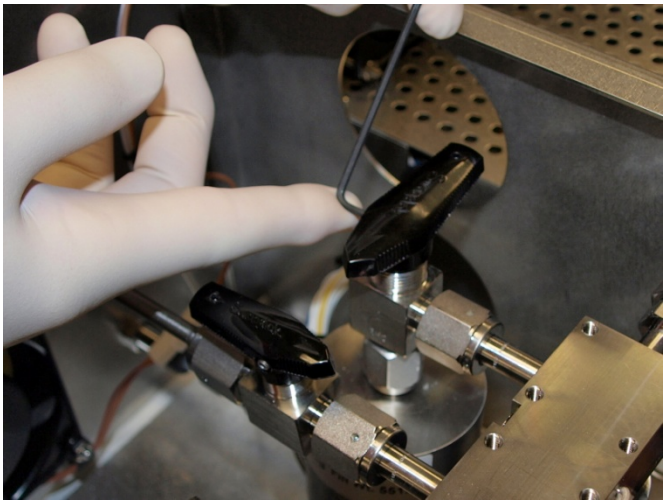
a. To do this, remove the 6 Philips head screws from the upper section of the enclosure



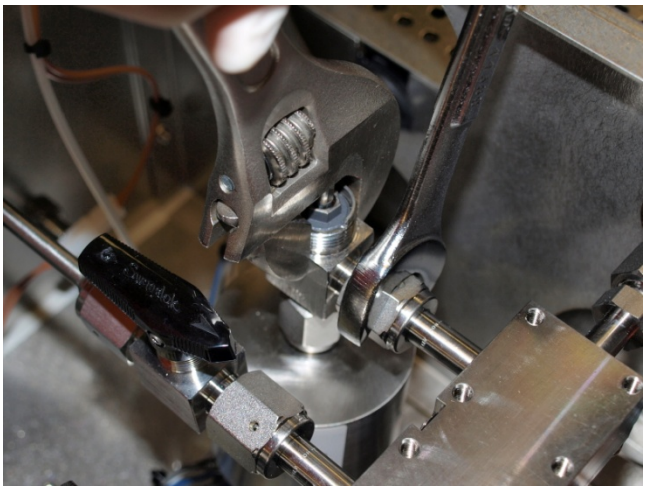
2. On the left side of the system you will see the gas manifold with the correct number of ALD valves, precursor bottles, and gas lines that you ordered with the system.

3. To remove a bottle in order to be filled with precursor follow this procedure:

a. Using a 5/64" allen key, remove the quarter turn valve handle.



b. Using an adjustable wrench and a ¾" wrench, turn (counter clockwise) the VCR fitting connecting the bottle valve with the manifold.



c. Remove the bottle from the system.

d. Repeat for other bottles installed on system.

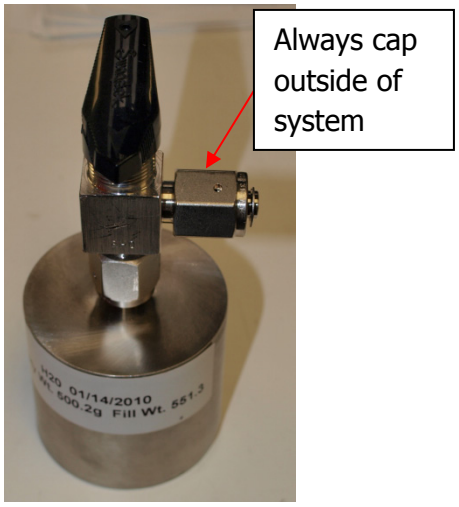
4. To fill precursor bottles, do one of the two following options:

a. Send bottles to Arradiance or reputable chemical company to be filled.

b. or, obtain precursor and fill bottles within an inert gas atmosphere enclosure such as a glove box.

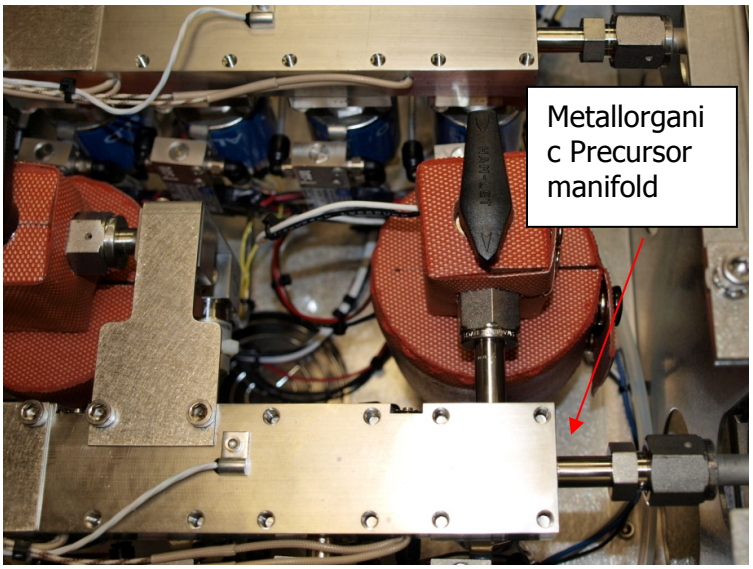
*Note: Be aware that most ALD organometallic precursors are at best air sensitive and at worst can be both **pyrophoric and toxic**, so please exercise extreme caution when filling the bottles.*

5. When precursor bottles are being transferred or otherwise contain precursor when not attached to the system, they should be capped with a female VCR cap and SS gasket. This will protect both the precursor and the user from accidental air exposure should the valve be opened inadvertently.



6. Install bottles with a new SS ¼" VCR fitting using the same procedure outlined above but turning clockwise to tighten the connection between the bottle and the manifold.

7. Metallorganic precursors should be installed on the metallorganic manifold shown in the picture below.



8. Once the bottles are installed the headspace between the valve on the bottle and the manifold should be purged.

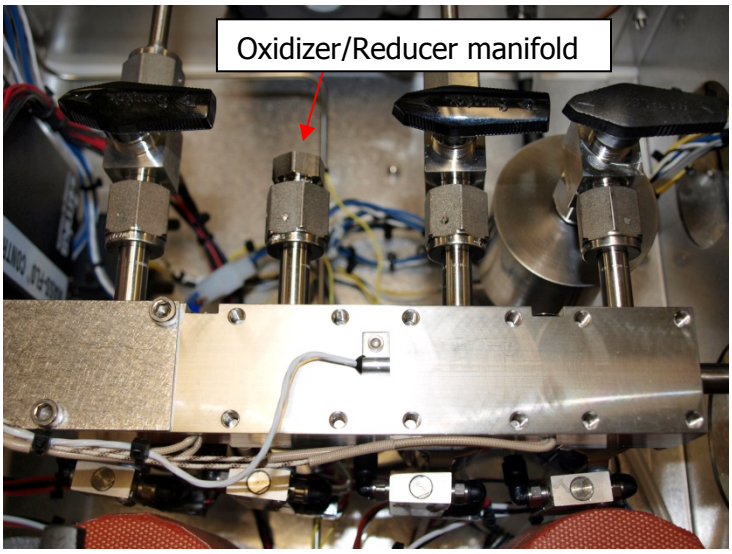
a. To purge lines before opening the hand valves please load the line purge program from the recipe file folder called "maintenance".

b. Run the recipe for each valve or gas line that is installed or run the recipe that degasses all precursor lines.

For example: To manually purge each line, start with the single line recipe in the maintenance folder. Click on run cycles to purge valve 1. When that is finished, change line 14 to the next actuator number that you would like to purge and run cycles. Continue until all newly connected precursor lines are evacuated.

9. Once the bottle line purges are finished, replace the handles on the quarter turn bottle valves and open all bottles and lines.

10. Oxidizers and reducer precursors should be installed on the opposite manifold these include, but are not limited to: H_2O, NH_3, O_2, H_2O_2 ozone, H_2, and NO_2

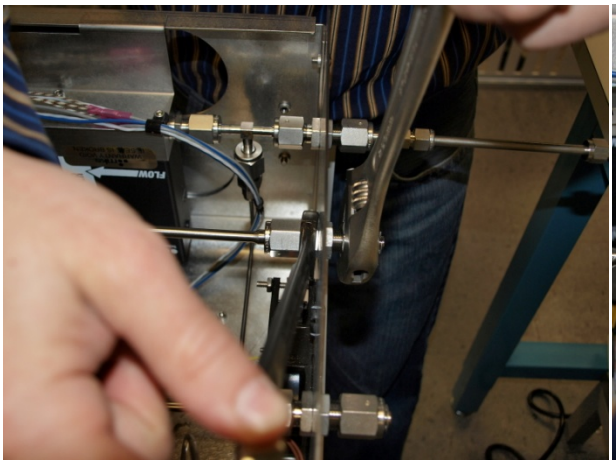


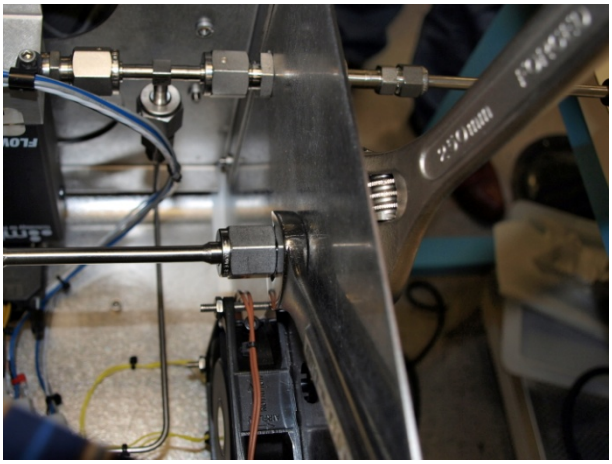
11. Gas and water (water vapor) sources can be installed exterior to the system provided the system that was purchased came with exterior gas connections.

a. To install a high pressure cylinder of O_2 (as an example), install an appropriate back pressure regulator on the gas tank and position it close to the system (make sure the cylinder is appropriately strapped).

b. Using either ¼" or 1/8" SS tubing, create a line extending from the regulator to the back of the GemStar system. The line should terminate in a ¼" female VCR fitting.

c. To connect the gas line to the system. First begin by removing the female cap on the exterior. To remove the cap, get two ¾" wrenches and position as shown in the two photos below.





d. Rotate the exterior wrench counter-clockwise until cap is loose.

e. Connect gas line using a new SS gasket placing the wrenches in the same configuration (1 inside and 1 outside of the system)

f. Open the valve on the gas line on the inside of the system then run the line purge program in the maintenance folder to remove all residual air from the line before opening the gas bottle.

g. Set the gas pressure to ~ 1-5psi. This can be adjusted based on process response.

12. The system is now ready for processing. (see section 4 and 5)

Safety Considerations

Qualified Personnel

Processing on the Arradiance GemStar shall be performed by those trained to follow all safety guidelines in addition to being trained in the creation of process recipes and the execution of said recipes.

Maintenance of tool shall only be performed by those trained to perform such operations. Persons should be familiar with working with high temperature components, high voltage electronics, and potentially dangerous chemical compounds.

Proper Use of System

GemStar Desktop ALD system should be used only in accordance with the procedures and methods described in this manual or by Arradiance employees. Any use other than those specified may cause injury or damage to system or components attached to system such as pump and computer.

When installing

a. Exercise caution and utilize best practices when moving system. Never lift or attempt to move system without assistance.

b. When handling and installing high pressure gases please follow gas suppliers stated warnings and procedures.

i. Always secure gas cylinders properly.

When operating

a. Do not override safety interlocks.

b. Do not attempt to pulse any valve (other than the expo valve) or run any process when the chamber is vented.

Note: The interlock is set such that only process and pulsing can be done when it is under vacuum, however, if this is disabled for any reason please exercise caution when the chamber is not under vacuum.

c. Each process recipe begins with the 'pump' script. This is to be retained in all recipes run on the GemStar to insure that the system only starts a process when proper vacuum levels have been detected.

d. Under no circumstances should the USB cable or the computer be removed or otherwise disconnected from the system when the software is controlling the system. Interruptions to communications between the computer and the system can damage the system.

i. If the computer needs to be disconnected from the GemStar, shutdown the GemStar software using the exit button. This will properly shutdown all valves and heaters.

When servicing or changing chemicals

a. When precursor bottles are being transferred or otherwise contain precursor when not attached to the system, they should be capped with a female VCR cap and SS gasket. This will protect both the precursor and the user from accidental air exposure should the valve be opened inadvertently.

*Note: Be aware that most ALD organometallic precursors are at best air sensitive and at worst can be both **pyrophoric and toxic**, so please exercise extreme caution when filling the bottles.*

b. Cool both manifolds to <40°C when working with the precursor bottles

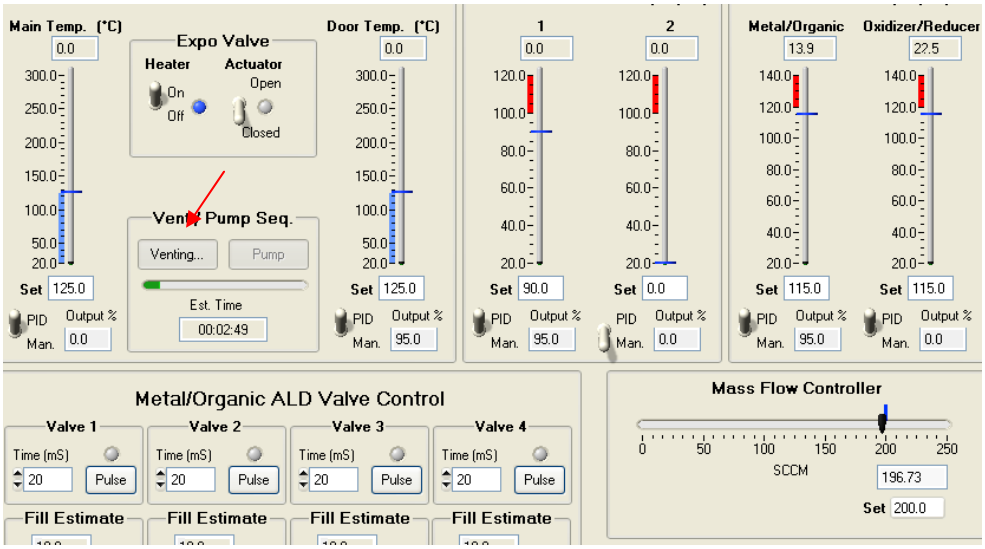
c. Turn off all heaters when servicing any part of the chamber assembly

d. MFC flow should be set to zero if removing any of the chamber and manifold assemblies during maintenance

e. Only Arradiance bottles and Arradiance sourced elastomer O-rings should be used.

Loading and Unloading Samples

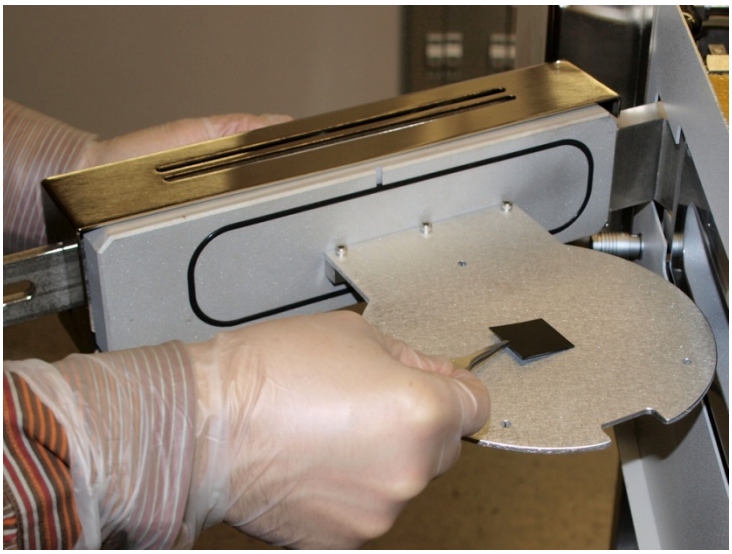
1. To load a sample, click 'Vent'. This will lower the chamber and door temperatures to 125°C, set the flow to 200sccm and will eventually close the expo actuator once the system cools. Once the system is cool, the chamber will reach atmosphere in ~ 4min.



Note: If vent time increases to >5min, you will need to replace the seal on the expo valve or replace the expo valve actuator (maintenance manual).

2. Unscrew the door clamp

4. Place sample on endefector.
   Note: If sample height is > 1.5cm, the endefector will have to be removed (see maintenance manual) and the sample loaded manually into chamber with a set of long tweezers



5. Close and clamp door.

6. Click on 'pump'. When chamber has successfully pumped down, the 'vacuum ready' light will be illuminated.
   Note: If chamber fails to pump, close 'expo valve actuator' and check to make sure the door is pressed uniformly against the chamber and the pump is functioning correctly.

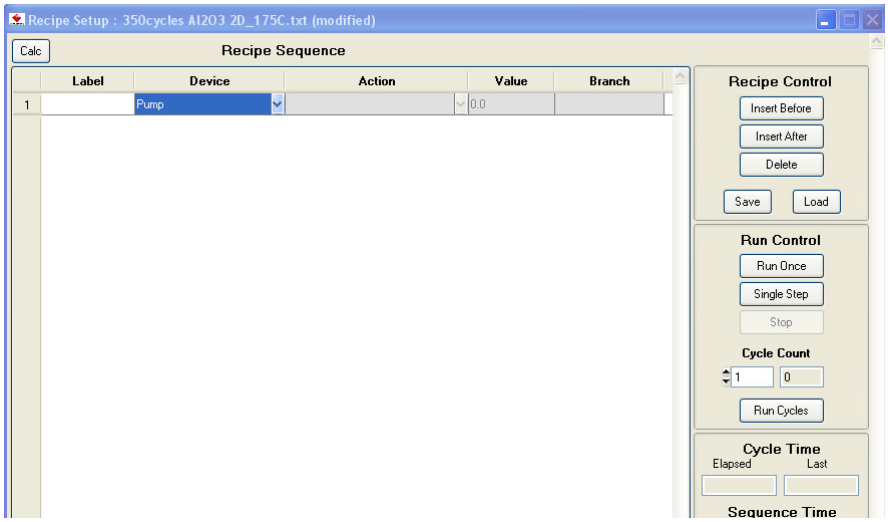
7. Run your desired process.

Loading and Running Process Recipes

Al2O3 for 2D Surface

Before running this recipe please confirm that the precursor bottle with trimethylaluminum is installed on valve 2 and that H_2O is installed on valve 5. If these precursors are installed in different positions, please make note of those.

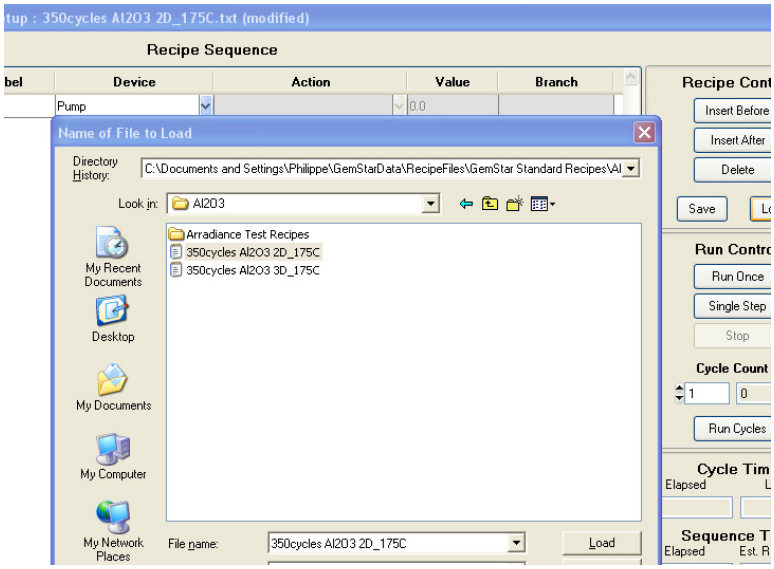
1. Maneuver your cursor to the process recipe screen.



2. Click on load process recipe and go to directory with the desired process.

3. As an example, go to the directory :GemStar Standard Recipes

4. Click on Al2O3 and then load the recipe "350cycles Al2O3 2D_175C"



5. The recipe is arranged by lines starting from the top and going down.

6. The first 7 lines are used to set the parameters that will hold for the rest of the process.

   a. The first three lines are used to verify that the system is in the proper initial state

      i. The pump script is engaged. To confirm the system is under vacuum before processing.

      ii. The 'expo valve heater' is set to on.

      iii. The 'MFC flow' is set to 200sccm. The high flow setting is used to bring the sample temperature up at the same rate as the chamber temperature.

Recipe Setup : 350cycles Al2O3 2D_175C.txt (modified)

Calc

Recipe Sequence

	Label	Device	Action	Value	Branch
1		Pump		0.0	
2		EXPO Heater	Set to On	0.0	
3		MFC Flow	Set to Value	200.0	

   b. The next 6 lines of the recipe set all of the process temperatures for the run.

      i. The lines 4 and 5 set the manifold temperatures to the standard 115°C.

      ii. Line 6 sets the temperature of the first heated bottle.

      iii. Line 7 set the temperature of the second heated bottle if present.

      Note: If no heated bottles are present simply set these values to 0.

      iv. The next two lines set the temperatures of the chamber and the chamber door.

4		Manifold 1 Heat	Set to Value	115.0
5		Manifold 2 Heat	Set to Value	115.0
6		Precursor 1 Heat	Set to Value	0.0
7		Precursor 2 Heat	Set to Value	0.0
8		Chamber Door Heat	Set to Value	175.0
9		Chamber Heat	Set to Value	175.0

      v. Line 10 is a conditional that makes sure the chamber and door are at the set point before setting the bakeout time

      vi. The last line sets the bakeout time which for a standard recipe is a default 3600 seconds.

7. The following 13 lines of this standard recipe are set in a loop that will repeat any number of times to grow the film cycle by cycle. The installed recipe is for 350 cycles which will yield 38nm of Al2O3 on a 2D surface.

a. To change the number of cycles go to line 24 and input the total number of desired cycles in the value column. The recipe shows the line that the recipe will branch to (step 12, 350 times).

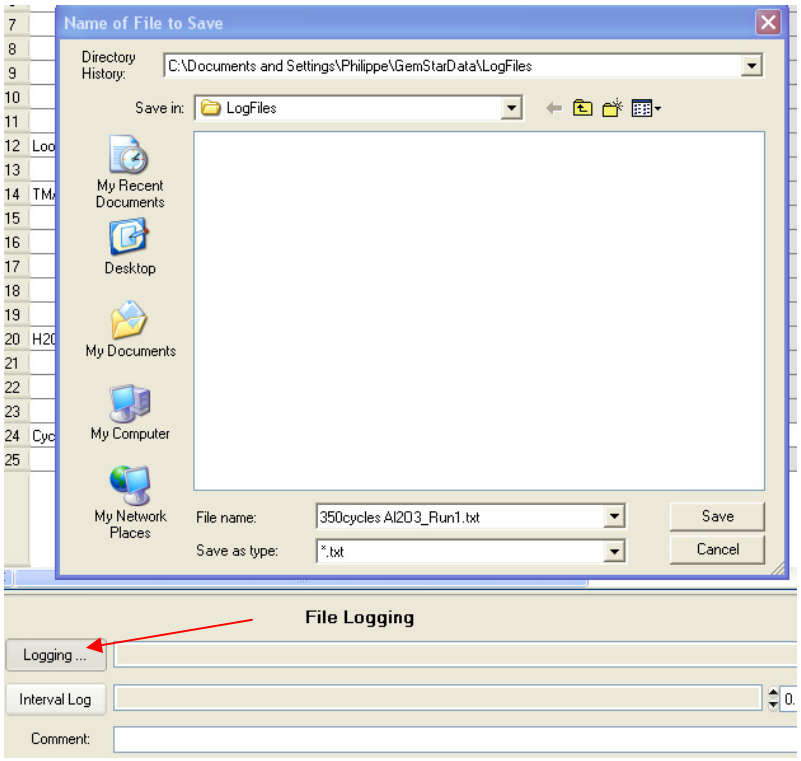
12	LoopStrt	MFC Flow	Set to Value	40.0		
13		Delay (Sec.)		1.5		
14	TMA	ALD 2 Actuator	Pulse (mSec.)	20		
15		Delay (Sec.)		0.5		
16		MFC Flow	Set to Value	90.0		
17		Delay (Sec.)		18.0		
18		MFC Flow	Set to Value	40.0		
19		Delay (Sec.)		1.5		
20	H2O	ALD 5 Actuator	Pulse (mSec.)	20		
21		Delay (Sec.)		1.0		
22		MFC Flow	Set to Value	90.0		
23		Delay (Sec.)		19.0		
24	Cycles	Loop n Times	Number	350	LoopStrt	
25		Vent		0.0		

b. When creating a new process recipe (see section 6, 'Creating new Recipes') it may be necessary to change the pulse time duration among other things.

i. To change the pulse time go to line 14 where the note indicates the TMA pulse, change the time in ms to the desired time (the default is 20ms).

ii. Go to line 20 to change the H_2O pulse time (default is 20ms).

8. To log the process and various parameters click on the log and create a file name for the process.



9. To run the process click on run cycles.

Note: Though there is an option to change the overall cycle numbers in the Run Control box,

the normal mode to operate is with Cycle Count set to 1 and run cycles or run once will complete the recipe as designed.

Creating new Recipes

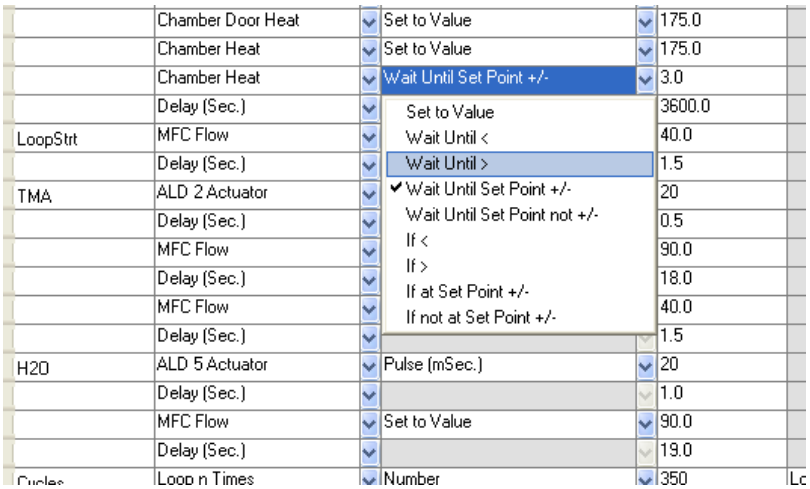
Before creating a new recipe, confirm the location of each precursor that will be used in the recipe.

It may be helpful to modify an existing recipe.

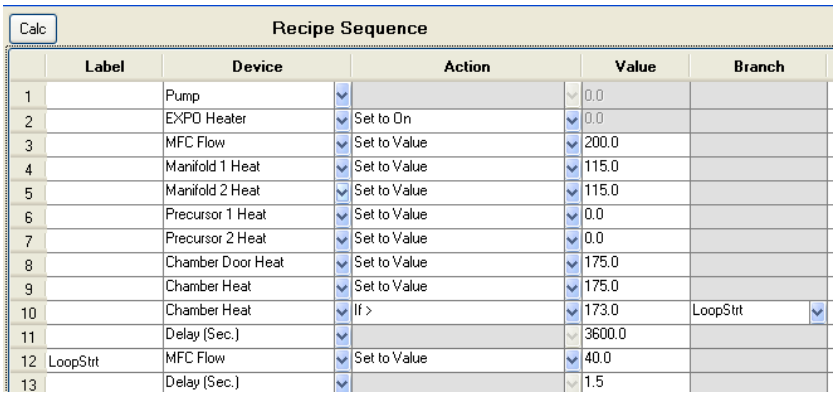
The following few examples begin with an existing pre-installed recipe and show which steps are ones that can be changed to accommodate differing precursor chemistries as well as device/film requirements.

1		Pump			0.0	
2		EXPO Heater	Set to On		0.0	
3		MFC Flow	Set to Value		200.0	
4		Manifold 1 Heat	Set to Value		115.0	
5		Manifold 2 Heat	Set to Value		115.0	
6		Precursor 1 Heat	Set to Value		0.0	
7		Precursor 2 Heat	Set to Value		0.0	
8		Chamber Door Heat	Set to Value		175.0	
9		Chamber Heat	Set to Value		175.0	
10		Chamber Heat	Wait Until Set Point +/-		3.0	
11		Delay (Sec.)			3600.0	
12	LoopStrt	MFC Flow	Set to Value		40.0	
13		Delay (Sec.)			1.5	
14	TMA	ALD 2 Actuator	Pulse (mSec.)		20	
15		Delay (Sec.)			0.5	
16		MFC Flow	Set to Value		90.0	
17		Delay (Sec.)			18.0	
18		MFC Flow	Set to Value		40.0	
19		Delay (Sec.)			1.5	
20	H2O	ALD 5 Actuator	Pulse (mSec.)		20	
21		Delay (Sec.)			1.0	
22		MFC Flow	Set to Value		90.0	
23		Delay (Sec.)			19.0	
24	Cycles	Loop n Times	Number		350	LoopStrt
25		Vent			0.0	

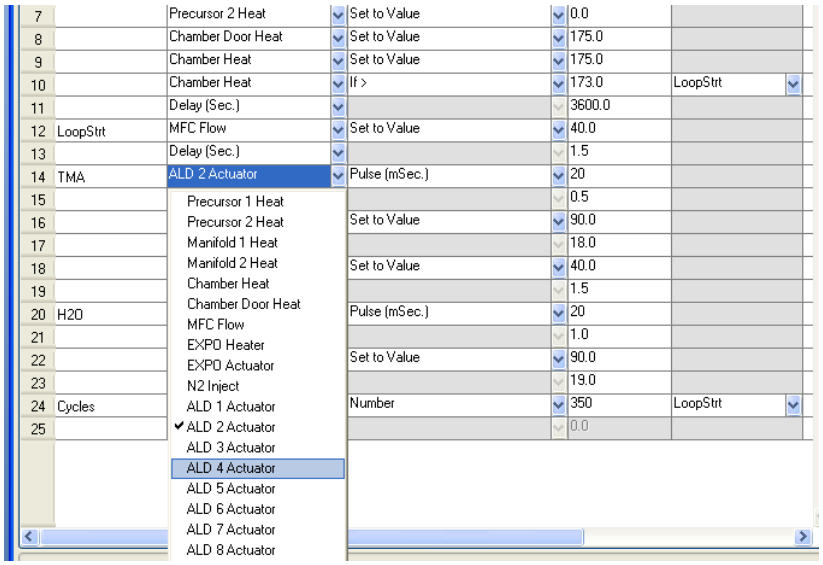
- Steps 6 and 7 can be used to set the temperature of precursors installed on actuators 1 and 3 on the metallorganic manifold if equipped with GemStar heating jackets. The temperature range for the precursor heating jackets is the same range as the manifold, 40-125°C.
    - If heating a precursor above 115°C, adjust manifold temperature to a few degrees above the precursor temperature to avoid condensation.
- If desired one can change the logic associated with the temperature conditional in line 10.



- Any of the thermal controls can be assigned a conditional attribute such as the following:
  - 'Set to value' → sets the heater and RTD to control at the value in °C
  - 'Wait until <' or '>' → recipe does not proceed until RTD reads a value less than the one provided in the value column or greater than the one provided
  - 'Wait until Set Point +-' or 'not +-' → The process will proceed to the following step once the RTD reads a temperature within + or – the value of the set point for that particular heater.
  - All 'if' logic controls provide a branch option, so if 'if >' is called then the recipe will check if the temperature is above the value that the user sets and will jump to the recipe line indicated in the branch column to the right of the value (see picture below).

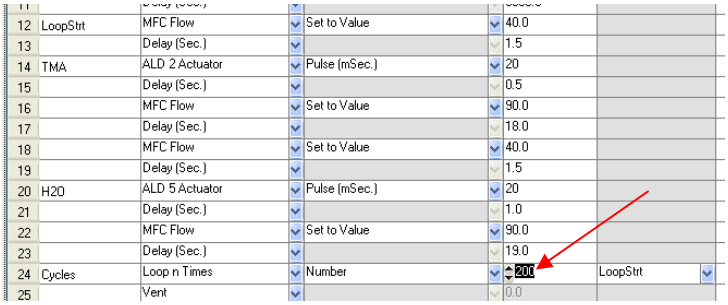


- To change the precursor bottle that you are using for a particular pulse, pick the correct actuator for that bottle from the drop down list under device:



- It is recommended to change the label (first column of recipe) to an abbreviation of the precursor installed on that line. Correct labeling of these will help ensure mistake free processing and recipe creation.

- Introducing loops into the recipe are important for changing the overall number of cycles as well as pulsing precursors multiple times and creating multicomponent films.
  - The following example shows how to change the overall number of cycles for a simple single component film:



The recipe was changed from 350 to 200 cycles

  - The next example shows the introduction of a loop to pulse a precursor more than once
    - This can be a useful way to achieve saturation without resorting to an increase in precursor temperature

TMA	ALD 2 Actuator	Pulse (mSec.)	20		
	Delay (Sec.)		0.5		
	MFC Flow	Set to Value	90.0		
	Delay (Sec.)		18.0		
	MFC Flow	Set to Value	40.0		
	Delay (Sec.)		1.5		
H2O	ALD 5 Actuator	Pulse (mSec.)	20		
	Delay (Sec.)		2.0		
	Loop n Times	Number	3	H2O	
	MFC Flow	Set to Value	90.0		
	Delay (Sec.)		19.0		
Cycles	Loop n Times	Number	200	LoopStrt	

- The next example shows the introduction of a second material (TiO_2) into the overall process sequence. This example is 200cycles of (1 cycle Al_2O_3/1cycle TiO_2) with the Ti precursor pulsed twice before introducing water.

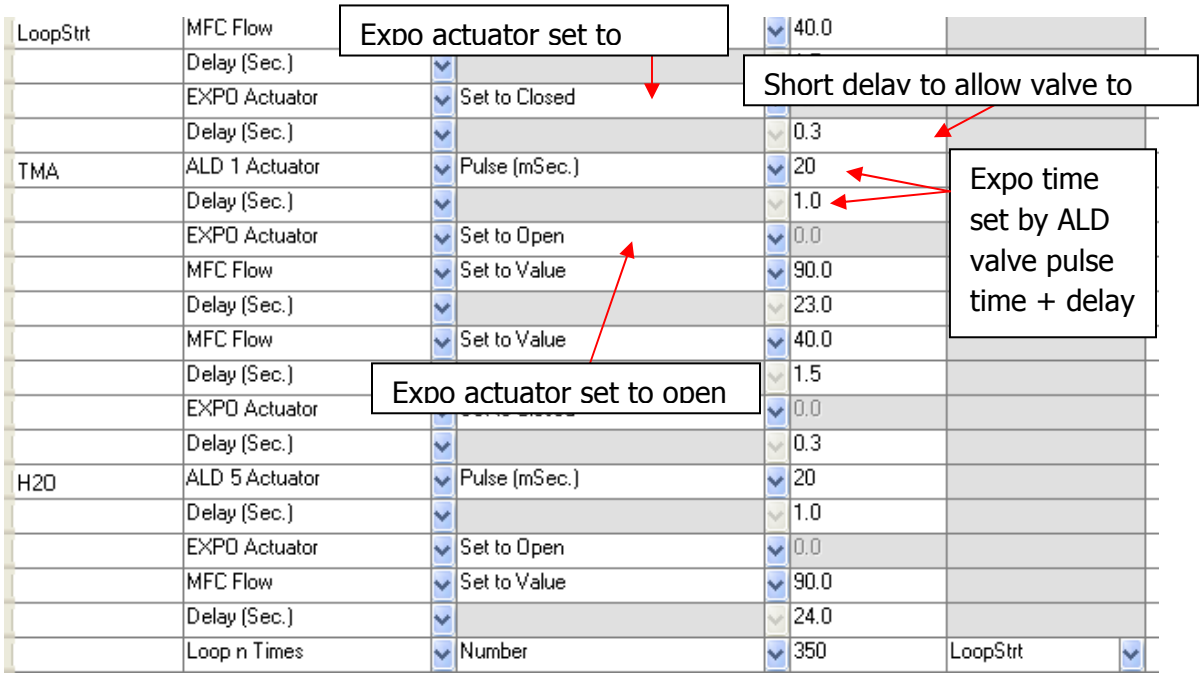
LoopStrt	MFC Flow	Set to Value	40.0		
	Delay (Sec.)		1.5		
TMA	ALD 2 Actuator	Pulse (mSec.)	20		
	Delay (Sec.)		0.5		
	MFC Flow	Set to Value	90.0		
	Delay (Sec.)		18.0		
	MFC Flow	Set to Value	40.0		
	Delay (Sec.)		1.5		
H2O-Al	ALD 5 Actuator	Pulse (mSec.)	20		
	MFC Flow	Set to Value	90.0		
	Delay (Sec.)		19.0		
	MFC Flow	Set to Value	40.0		
	Delay (Sec.)		1.5		
Tiamide	ALD 3 Actuator	Pulse (mSec.)	50		
	Delay (Sec.)		2.0		
	Loop n Times	Number	2	Tiamide	
	MFC Flow	Set to Value	90.0		
	Delay (Sec.)		18.0		
	MFC Flow	Set to Value	40.0		
	Delay (Sec.)		1.5		
H2O-Ti	ALD 5 Actuator	Pulse (mSec.)	20		
	MFC Flow	Set to Value	90.0		
	Delay (Sec.)		19.0		
Cycles	Loop n Times	Number	200	LoopStrt	

Added Ti precursor pulse with appropriate label

Ti pulsed twice before H2O

- The last two examples will introduce the concept of a static flow or high-exposure process and of utilizing the inert gas vapor pressure assist should that option be installed on the system

- By closing the valve actuator prior to introducing a precursor and then waiting for a certain amount of time before opening the actuator, you can greatly increase the exposure of the precursor to your device or surface.
  - Increasing the expo can be critical to achieving conformal films over high aspect ratio features.
  - A high expo process can in some cases increase the nucleation density which can have the effect of increasing the per cycle film growth rate, make the film denser, and potentially improve certain properties like lower leakage currents for insulators.
  - Please note that in some cases increasing the residence time(introducing an expo step) for precursors that are close to their decomposition temperature will increase the probability for precursor decomposition and subsequent CVD and contaminant effects.



LoopStrt	MFC Flow		40.0	
	Delay (Sec.)			
	EXPO Actuator	Set to Closed		
	Delay (Sec.)		0.3	
TMA	ALD 1 Actuator	Pulse (mSec.)	20	
	Delay (Sec.)		1.0	
	EXPO Actuator	Set to Open	0.0	
	MFC Flow	Set to Value	90.0	
	Delay (Sec.)		23.0	
	MFC Flow	Set to Value	40.0	
	Delay (Sec.)		1.5	
	EXPO Actuator		0.0	
	Delay (Sec.)		0.3	
H2O	ALD 5 Actuator	Pulse (mSec.)	20	
	Delay (Sec.)		1.0	
	EXPO Actuator	Set to Open	0.0	
	MFC Flow	Set to Value	90.0	
	Delay (Sec.)		24.0	
	Loop n Times	Number	350	LoopStrt

- Introducing the high pressure inert gas assist in the process sequence for ultra low vapor pressure precursors is straightforward

10		EXPO Actuator	Set to Closed	0.0
11		N2 Inject		0.0
12		Delay (Sec.)		0.3
13	Y(AMD)3	ALD 3 Actuator	Pulse (mSec.)	250
14		Delay (Sec.)		1.0
15		EXPO Actuator	Set to Open	0.0
16		MFC Flow	Set to Value	90.0
17		Delay (Sec.)		23.0

- Add the N2 Inject script from the pull down menu under device right after or before the expo actuator close (if using)
- Everything else remains unchanged

[END OF DOCUMENT]