Agnitron Agilis MOCVD SOP

Purpose and Scope

This document provides job breakdowns and reference information for MOCVD using the Agnitron Agilis.

Table of Contents

Reference Documents

Reference Documents	SOP Number or link
Nanofab Lab User Guide	HTTPS://WWW.NANOFAB.UTAH.EDU/DOCUMENTS/2016/02/SMBB-USER-GUIDE.PDF/

Acronyms, Abbreviations and Definitions

Term	Description
SOP	Standard Operating Procedure
GC	Growth Chamber
JB	Job Breakdown
JR	Job Reference
MFC	Mass Flow Controller
UPW	Ultra Purified Water

Equipment and Supplies

Description	
Agnitron Agilis	Located in the MOCVD bay
IMPERIUM software	In the computer for the Agilis.
Water from an APW source	Container in the MOCVD bay
Sample substrate	Member brings
Sample holder	Located in the glove box or on the table
Sample loading tool	Located in the glove box
Sample holder pedestal	Located in the glove box
Mirror	Located in the glove box

Safety

Follow all Nanofab safety procedures.

Safety alert symbol

The Safety Alert Symbol is used in conjunction with signal words to convey a personal injury hazard is present.

Signal words

DANGER	Indicates an imminently hazardous situation, which if not avoided, will result in death or serious injury. The Safety Alert Symbol should always be used.
WARNING	Indicates a potentially hazardous situation, which if not avoided, may result in death or serious injury. If the safety alert symbol is NOT used in conjunction with this signal word, then the hazard conveyed is severe equipment or material damage.
CAUTION	Indicates a potentially hazardous situation, which if not avoided, may result in minor or moderate injury. If the safety alert symbol is NOT used in conjunction with this signal word, then the hazard conveyed is minor equipment or material damage.

Forms

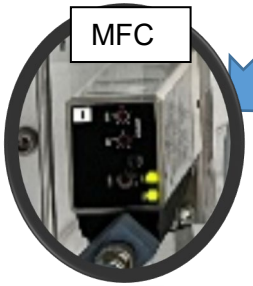
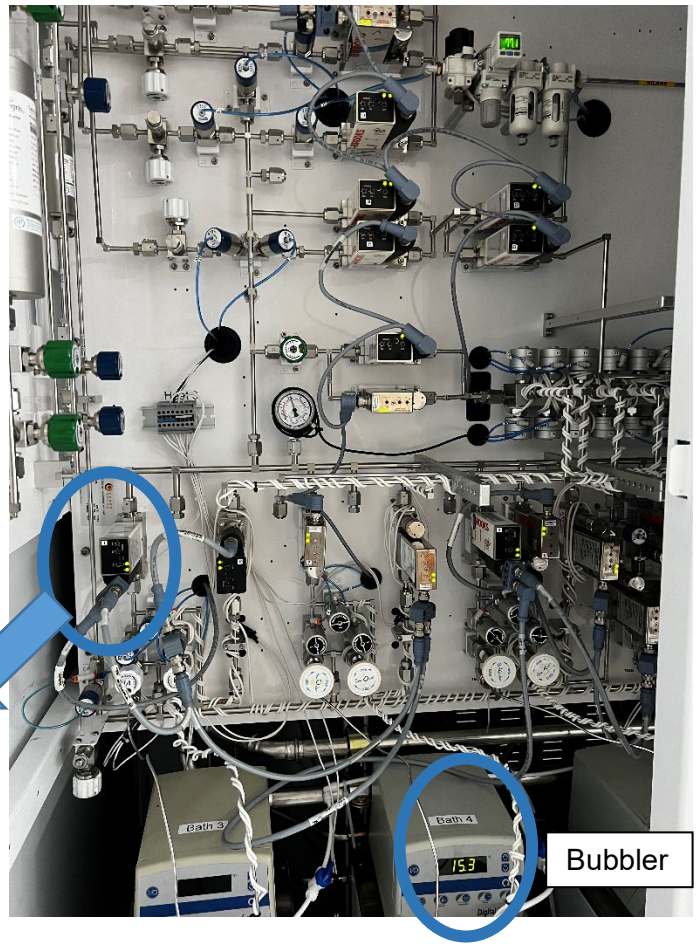
Training Form

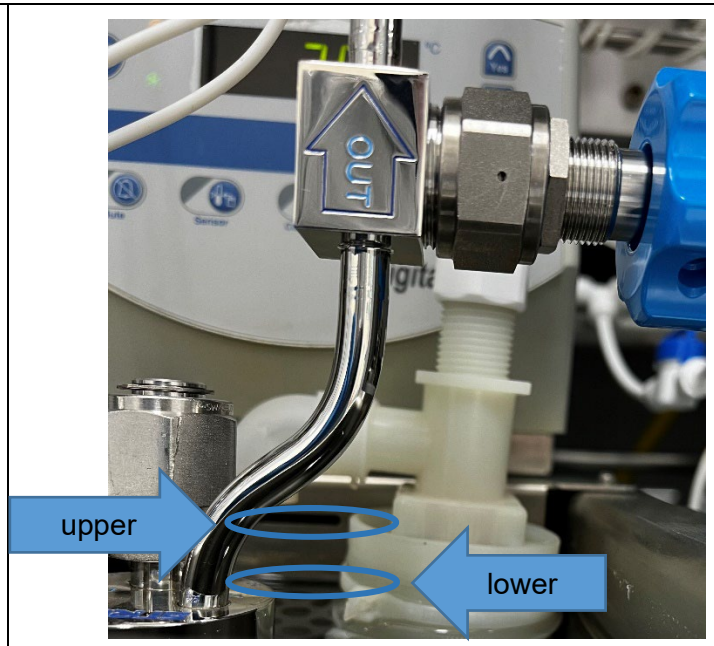
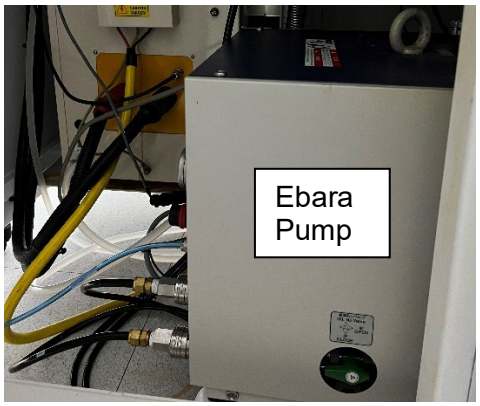
Trainee:			
Area:			
Item #	Task	Date Training Completed	Trainer
1	JB1 Pre-Process Check		
2	JB2 Reserve and Unlock in HSC.		
3	JB3 Open Growth Chamber and Load Samples		
4	JB4 Return lid and Pump Down Chamber		
5	JB5 Select Recipe		
6	JB6 Change Recipe Parameters		
7	JB7 Run Recipe		
8	JB8 Fill Unattended Work in Progress Sheet		
9	JB9 Once Complete, Restore Growth Chamber Pressure		
10	JB10 Remove Sample		
11	JB11 Restore N2 Purge		
12	JB12 Restore Screenlock		
13	JB13 Locking in HSC and Data Collection		
Training Notes (Optional)			

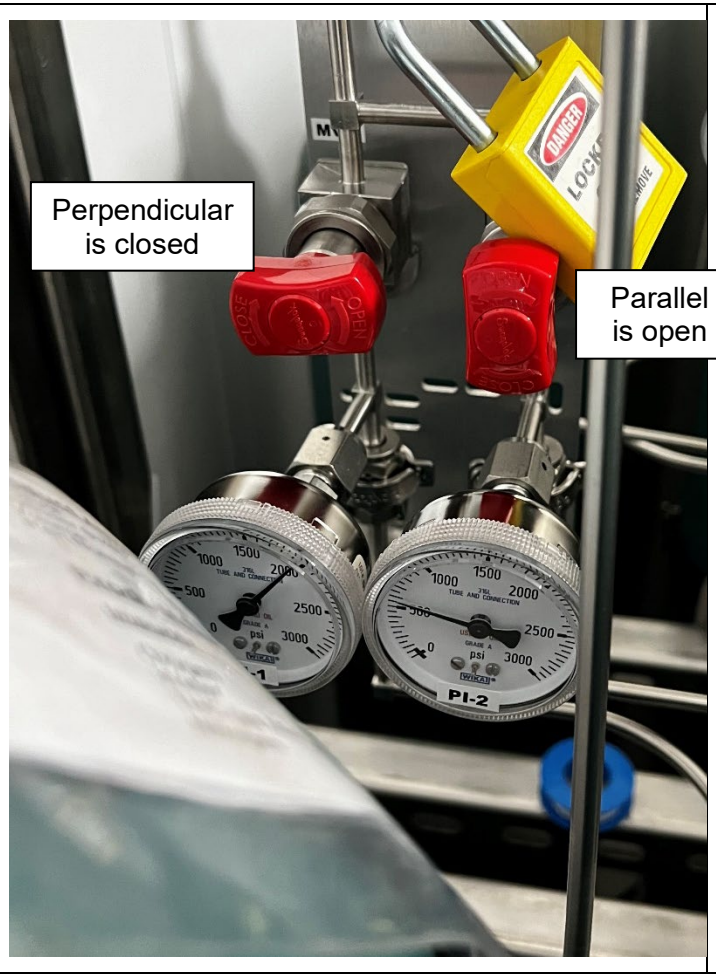
Run Data Collection Form

Date	Member	Precursor	Precursor Flow sccm	Bubbler Temp °C	Deposition Time min	Deposition Temp °C	GC Pressure torr	Spin Speed RPM	O_2 Flow sccm	Ar Flow sccm	Film Thickness nm

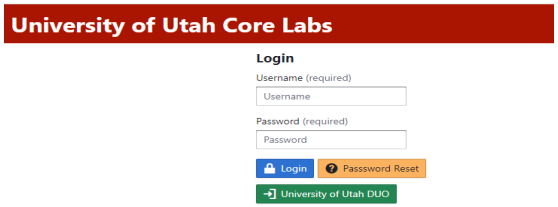
JB 1 – Pre-Process Check

A	**Verify** 1. **Experiment is planned** considering previous results. The impacts of Process variable changes on measured outcomes can only be distinguished if using a statistically designed experiment or changing one variable at a time between runs. Make an informed decision using results of past run data. A form to record run data is included in this SOP. More information on how to design an experiment can be found at What is design of experiments (DOE)? (nist.gov) 2. **Mass Flow Controllers (MFCs) are showing solid green lights**. This indicates they are communicating effectively. If lights are blinking or red, there is a communication error. Contact staff to correct the error before processing samples.	 	☐

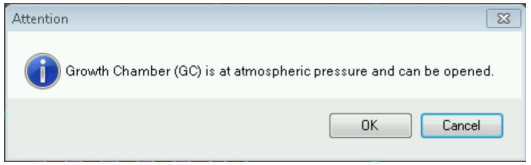
B	3. **Bubbler to be used is powered on and circulating water**. If it is off, press the power button. The display screen should display the target, then actual temp and the pump should begin circulating water. 4. **Bubbler to be used has enough water**. This is determined by checking that the water surface is between the upper and lower level marks as well as whether the bubbler display is showing a water low fault. If bubbler water level is low, use the container of UPW sourced water to pour to bring it up to between the lower and upper levels. If more water is needed, ask Nanofab staff to refill the water container from the UPW source. 5. **Temp of bubbler to be used is on target**. Temp is displayed digitally. A table matching bubbler precursors to target temperature is in Job Reference 1 of this spec. Note: It takes a few minutes right after turning on the bubbler for the temp to reach target. Wait for the temp to stabilize at target before proceeding. Caution: If bubbler target temp change is needed, request Nanofab Staff member enter the new value into the IMPERIUM software and set the bubbler to the new target. Bubbler consumption values displayed by IMPERIUM software are only correct if bubbler target and settings in software match. 6. No audible alarms are sounding on the system. 7. Scroll pump (small pump next to bubblers) and Ebara pump (loud pump accessible from behind the tool) are on.	 	☐

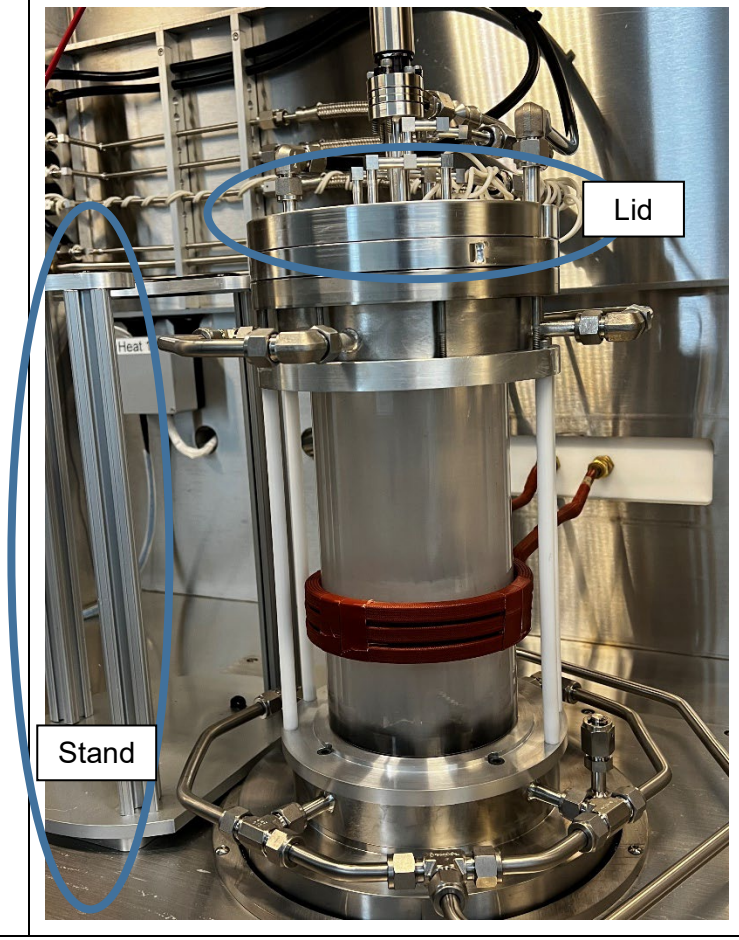
B	8. Argon supply is greater than 200 psi. When Argon supply is 200 psi or less, notify Staff. We will evaluate the length of your next run and determine how many runs worth of Argon remain. In the image shown, the canister in service is a little over 500psi and the canister next in line for service is at 2000 psi. Red valve knobs show open and closed positions. Caution: Risk of backflow and Argon pressure waste during crossover is the reason cross-over from using one canister to the next is done by Nanofab staff.		☐

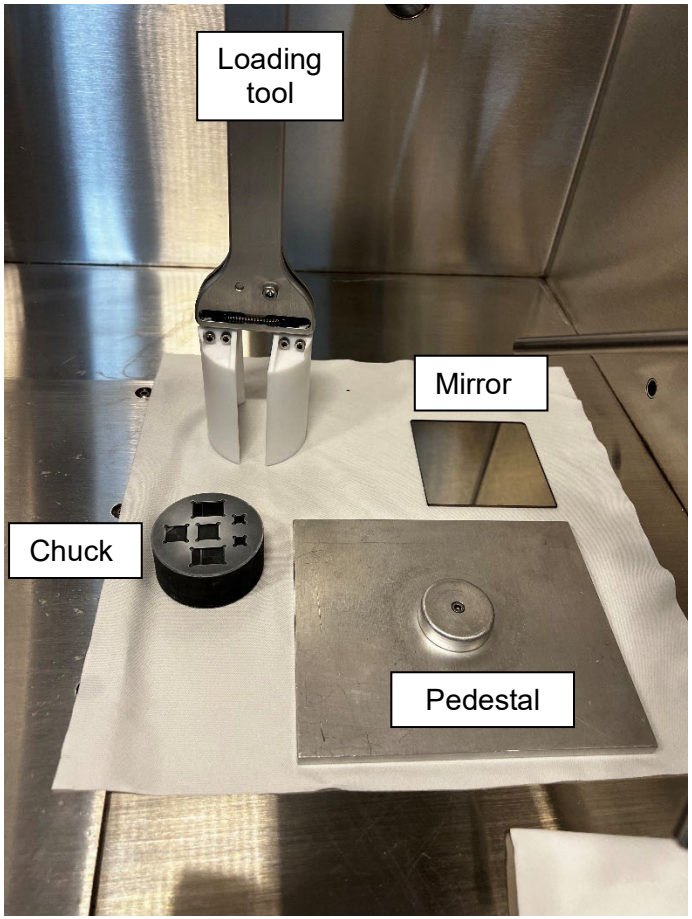
JB 2 – Reserve and Unlock in HSC

A	**Enabling Tool** 1. Log into HSC and reserve the MOCVD tool for the desired time. 2. Unlock the tool in HSC to enable the Main Control Panel. *Note: The tool can only be enabled if there is an active reservation.*		☐

JB 3 – Open Growth Chamber and Load Samples

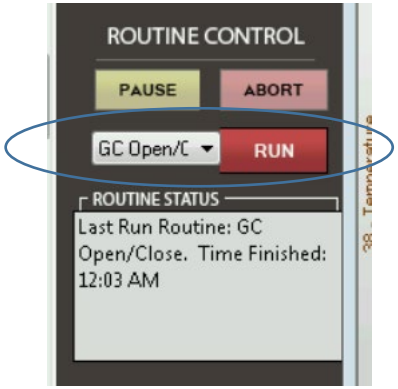
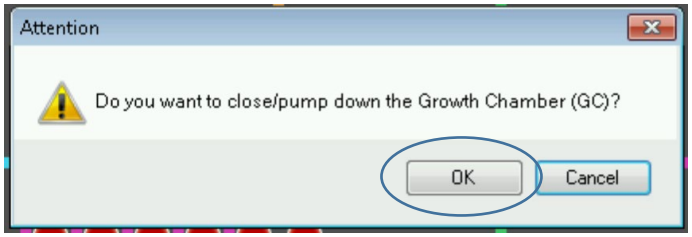
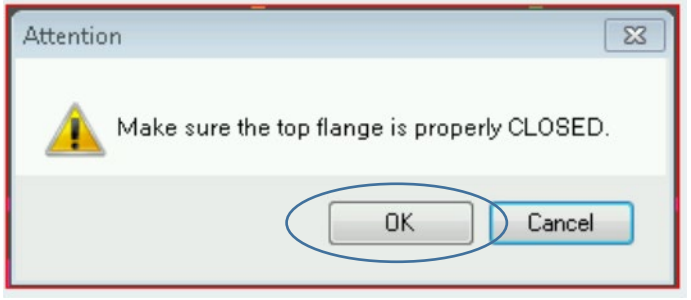
A	**Bring Chamber Pressure to Atmosphere** 1. From the Routine Control menu dropdown, select the GC open close routine and press RUN. This will begin a series of automated purge and fill steps to change the current state away from N2 idle and bring the growth chamber up to atmospheric pressure. 2. Wait until a window appears notifying that the growth chamber is at atmospheric pressure.	 	☐

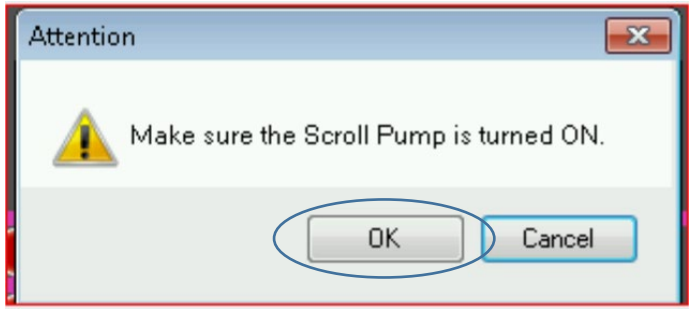
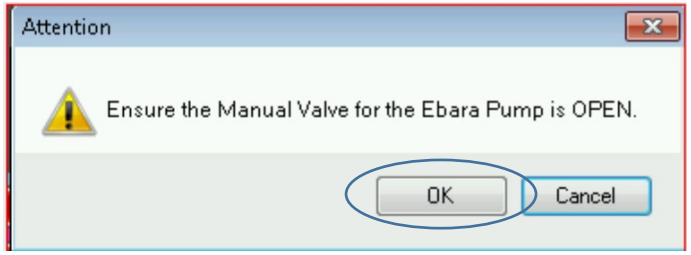
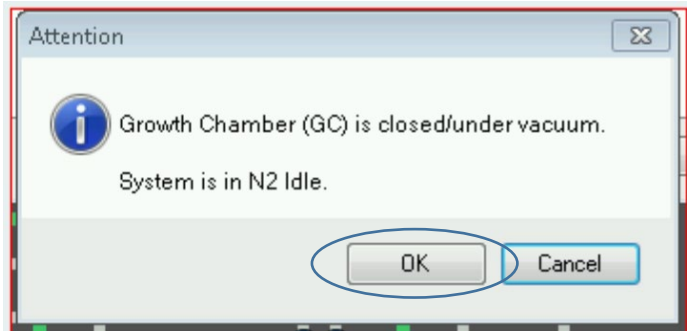
B	**<u>Open Chamber</u>** 1. Lift lid from growth chamber and place on stand. Avoid clipping, dinging or otherwise damaging lid edge or o-rings during placement.		☐

C	**Load Samples into Chamber** 1. Load samples onto chuck. 2. Transfer chuck into Antechamber 3. Close Antechamber 4. From inside glove box, open antechamber. 5. Position chuck pedestal to convenient position. 6. Remove sample from antechamber and place centered on chuck pedestal. 7. Use chuck loading tool to pick up chuck. 8. Use chuck loading tool to move chuck above growth chamber. 9. Lower loading tool with chuck vertically and place chuck to be centered on spindle. Caution: The chuck will not balance on the spindle if off center. This will cause a wobble during rotation. 10. Use lever on chuck loading tool to release chuck. 11. Carefully extract empty loader in vertical rise motion from the growth chamber. This must be done without bumping the chuck otherwise the chuck will be knocked off center and placement must be redone.		☐

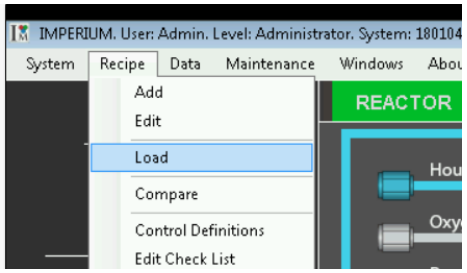
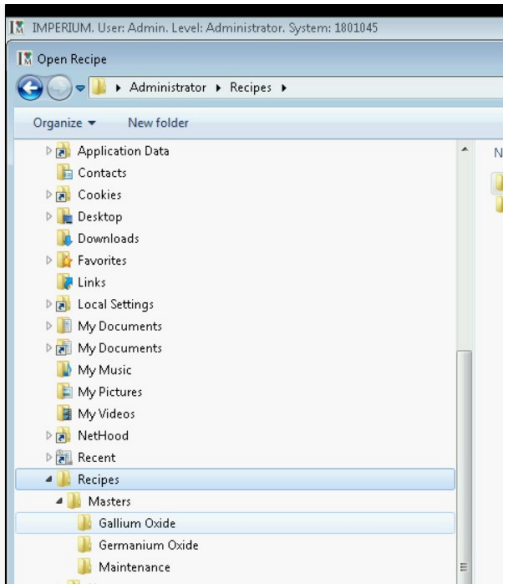
D	**Verify Chuck is Centered** 1. Use mirror to Verify chuck is level. if it is not, reposition. 2. If chuck appears level, run spindle rotation routine. To run the spindle rotation routine, select rotation from the drop down beneath GC Open/Close in the Routine Control menu. Select RUN. If wobble is observed during rotation, stop the rotation and use the chuck placement tool to recenter. If on center, run faster spindle routine when prompted. Confirm no wobble is present.	Use Dropdown Menu to select rotation routine 	☐

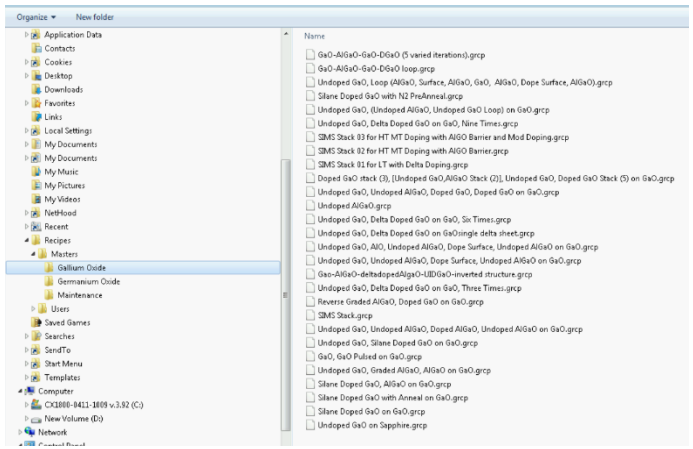
JB 4 – Return lid and Pump Down Chamber

A	**<u>Pump Down Chamber</u>** 1. Return lid to growth chamber 2. Run GC Open/Close routine		☐
B	**<u>Respond to Prompts – Pump Down</u>** 1. Confirm you want to pump down by selecting OK at the prompt.		☐
C	**<u>Respond to Prompts - Flange</u>** 1. If top flange is not yet closed, close it. 2. If top flange is closed, press OK		☐

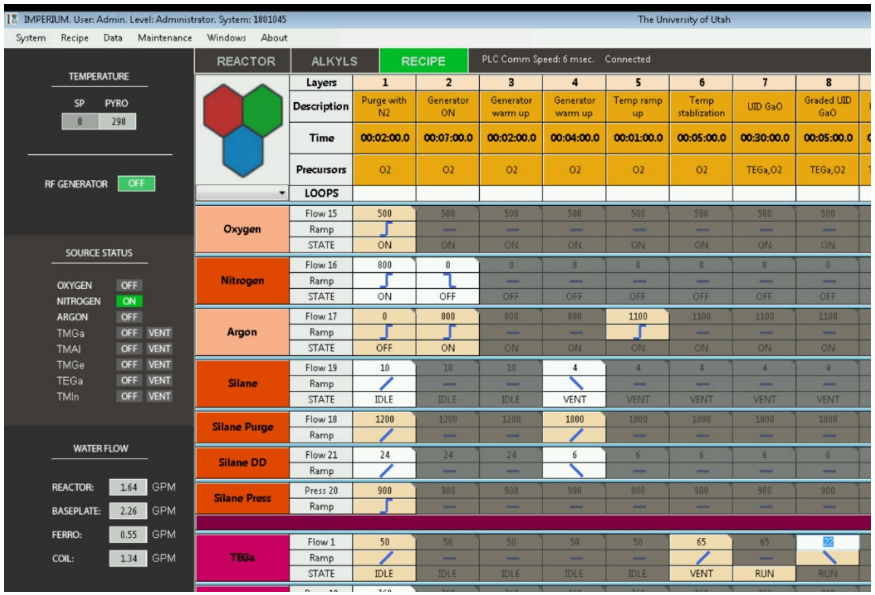
D	**Respond to Prompts – Scroll Pump** 1. If the Scroll Pump is off, turn it on. 2. If the Scroll Pump is on, press OK		☐
E	**Respond to Prompts – Ebara Pump** 1. If the Ebara Pump manual valve is closed, open it. 2. If the Ebara Pump manual valve is open, press OK		☐
F	**Respond to Prompts – GC Closed** 1. Wait until the prompt states that the growth chamber is under vacuum, then press OK.		

JB 5 – Select Recipe

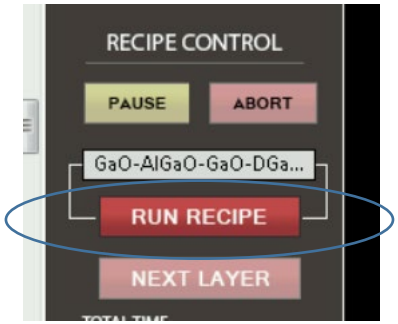
A	**Navigate to Recipes** 1. From the top menu bar, select Recipe 2. Select Load.		☐
B	**Navigate to Folder and Select Recipe** 1. Select Recipes 2. Select Masters 3. Select appropriate folder for recipe type. 4. Select recipe. The recipe screen will appear.		☐

C	**Select Recipe** 1. Select desired recipe. The recipe screen will appear.		☐

JB 6 – Change Recipe Parameters

A	**Select and Change Recipe Parameters as Needed** 1. Identify critical outcome to measure. 2. View recipe configurations of previous runs and compare to results to determine primary factors that affect that critical outcome. 3. Select parameter to vary 4. In the IMPERIUM recipe screen, change the value displayed for that parameter in each relevant step to the value to be tested.		☐

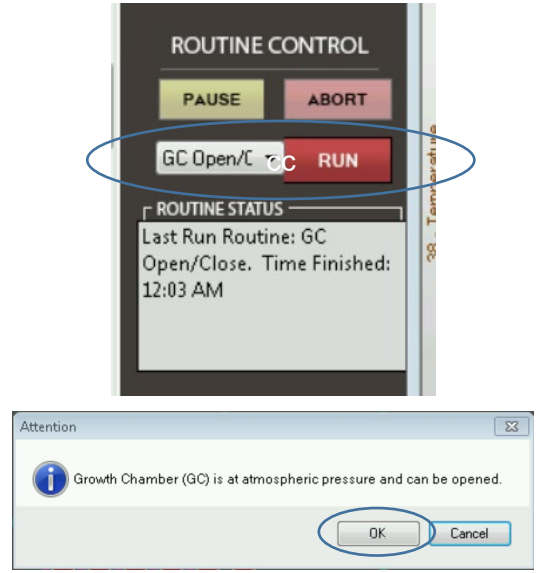
JB 7 – Run Recipe

A	1. On the IMPERIUM screen, in the Recipe Control menu, select the RUN RECIPE button.		☐

JB 8 – Fill Unattended Work in Progress Sheet

A	1. Fill out the Unattended Work In Progress Form		☐

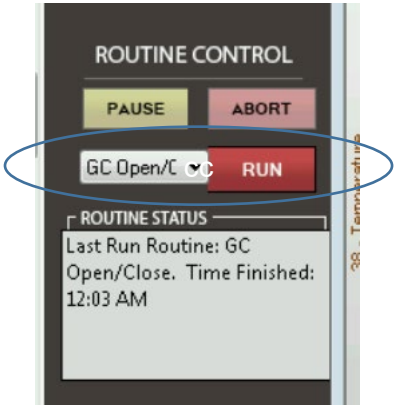
JB 9 –Return and Restore Growth Chamber Pressure

A	**<u>Restore Growth Chamber Pressure</u>** 1. Return once recipe is complete. 2. Run G/C open/Close Routine. 3. Wait until the prompt indicates the growth chamber is back to atmospheric pressure. Then press OK.		☐

JB 10 – Remove Sample

A	**Remove Lid** 1. Remove Lid and place on pedestal. *NOTE: This motion will give resistance. Apply force.*		☐
B	**Remove Sample** 1. Pick up chuck loader tool and engage the lever to enable it to pass over the edge of the chuck. 2. Carefully lower empty chuck loader in vertical motion into the growth chamber until it is low enough to grasp the chuck. This must be done without bumping the chuck otherwise the chuck will be knocked off balance. 3. Release lever to ensure chuck is secure in the loader. 4. Raise loading tool with chuck vertically and out of the growth chamber. Take care not to clip or bump the growth chamber. 5. Hover chuck over chuck pedestal. 6. Use lever on chuck loading tool to release chuck.		☐

JB 11 – Restore N2 Purge

A	**Pump Down Chamber** 1. Return lid to growth chamber 2. Run GC Open/Close routine 3. Wait for prompt to indicate the growth chamber is under vacuum. Select OK.		☐
B	**Select N2 IDLE** 1. From the Current State menu, select N2 Idle		

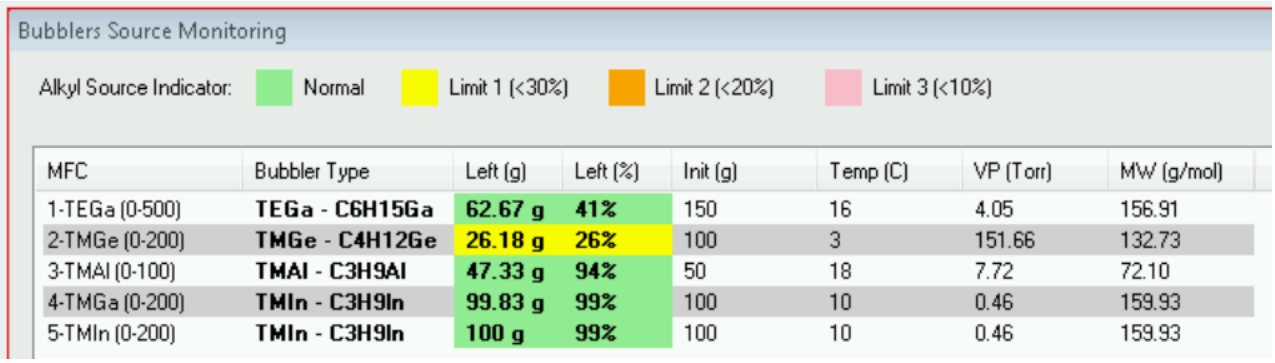
JB 12 – Restore Screenlock

A	**Select Windows Icon** 1. From bottom left hand corner of the screen, select the Windows icon 2. Select Screen Lock		☐

JB 13 – Locking in HSC and Data Collection

A	**Locate Computer with HSC access** 1. Log in to HSC 2. Select MOCVD session. 3. Select Lock 4. Enter results data as prompted. 5. If time is greater than MOCVD process time due to unattended processing, contact Nanofab Staff member to adjust to actual time.		☐

Job Reference 1 – Bubbler Temperature



MFC	Bubbler Type	Left (g)	Left (%)	Init (g)	Temp (C)	VP (Torr)	MW (g/mol)
1-TEGa (0-500)	**TEGa - C6H15Ga**	**62.67 g**	**41%**	150	16	4.05	156.91
2-TMGe (0-200)	**TMGe - C4H12Ge**	**26.18 g**	**26%**	100	3	151.66	132.73
3-TMAl (0-100)	**TMAl - C3H9Al**	**47.33 g**	**94%**	50	18	7.72	72.10
4-TMGa (0-200)	**TMIn - C3H9In**	**99.83 g**	**99%**	100	10	0.46	159.93
5-TMIn (0-200)	**TMIn - C3H9In**	**100 g**	**99%**	100	10	0.46	159.93

Bubblers, precursors and their target temperatures are listed above.

Job Reference 2 – Analog IDs

Analog IDs are helpful when troubleshooting errors displayed by the IMPERIUM software. Often the errors are listed in the alarms and errors panel by ID number and not name. Search by ID to find the name.

JR2 (1 of 4)

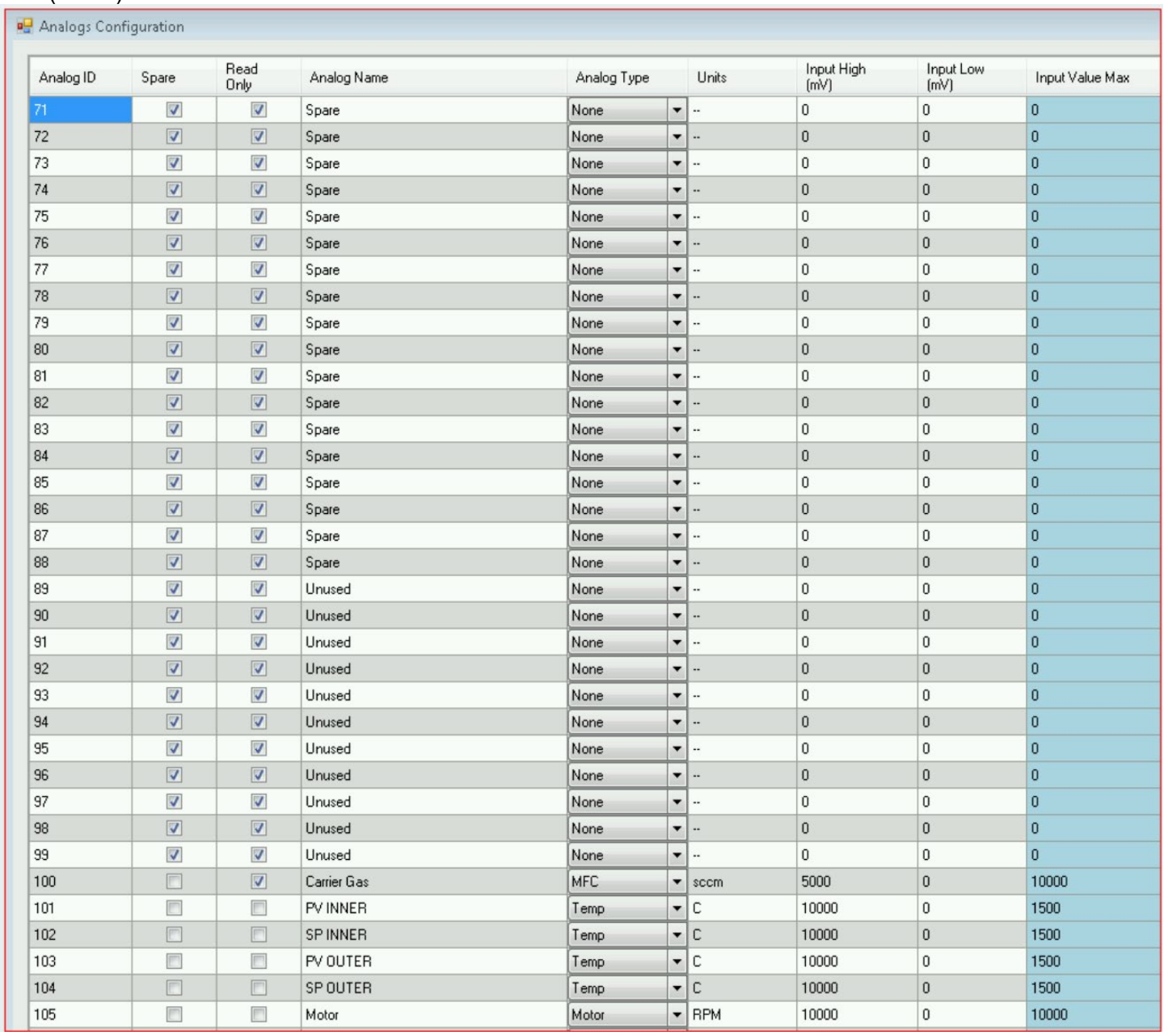
Analog ID	Spare	Read Only	Analog Name	Analog Type	Units	Input High (mV)	Input Low (mV)	Input Value Max
1	☐	☐	TEGa	MFC	sccm	5000	0	500
2	☐	☐	TMGe	MFC	sccm	5000	0	200
3	☐	☐	TMAl	MFC	sccm	5000	0	100
4	☐	☐	TMGa	MFC	sccm	5000	0	200
5	☐	☐	TMIn	MFC	sccm	5000	0	200
6	☐	☐	TMAl Push	MFC	sccm	5000	0	1000
7	☐	☐	TMGa Push	MFC	sccm	5000	0	1000
8	☐	☐	TMAl DD	MFC	sccm	5000	0	200
9	☐	☐	TMGa DD	MFC	sccm	5000	0	500
10	☐	☐	TEGa Press	PCU	Torr	5000	0	1000
11	☐	☐	TMGe Press	PCU	Torr	5000	0	1000
12	☐	☐	TMAl Press	PCU	Torr	5000	0	1000
13	☐	☐	TMGa Press	PCU	Torr	5000	0	1000
14	☐	☐	TMIn Press	PCU	Torr	5000	0	1000
15	☐	☐	Oxygen	MFC	sccm	5000	0	2000
16	☐	☐	Nitrogen	MFC	sccm	5000	0	5000
17	☐	☐	Argon	MFC	sccm	5000	0	2000
18	☐	☐	Silane Purge	MFC	sccm	5000	0	2000
19	☐	☐	Silane	MFC	sccm	5000	0	50
20	☐	☐	Silane Press	PCU	Torr	5000	0	1000
21	☐	☐	Silane DD	MFC	sccm	5000	0	100
22	☐	☐	Inj Blk Run	MFC	sccm	5000	0	10000
23	☐	☐	Inj Blk Vent	MFC	sccm	5000	0	2000
24	☐	☐	Shroud	MFC	sccm	5000	0	10000
25	☑	☑	Spare	None	--	0	0	0
26	☑	☑	Spare	None	--	0	0	0
27	☑	☑	Spare	None	--	0	0	0
28	☑	☑	Spare	None	--	0	0	0
29	☑	☑	Spare	None	--	0	0	0
30	☑	☑	Spare	None	--	0	0	0
31	☑	☑	Spare	None	--	0	0	0
32	☑	☑	Spare	None	--	0	0	0
33	☐	☐	GC Press	rBaratron	Torr	1000	0	1000
34	☑	☑	Spare	Press	Torr	10000	0	1000
35	☐	☐	Baratron Purge	MFC	sccm	5000	0	1000

JR2 (2 of 4)

Analogs Configuration

Analog ID	Spare	Read Only	Analog Name	Analog Type	Units	Input High (mV)	Input Low (mV)	Input Value Max
36	☑	☑	Spare	Motor	RPM	10000	0	1000
37	☐	☐	TV Position	TV Pressure	Torr	10000	0	100
38	☐	☐	Temperature	Temp	C	10000	0	1500
39	☐	☑	Antechamber Exhaust Press	None	--	10000	0	10000
40	☑	☑	Spare	None	--	0	0	0
41	☑	☑	Spare	None	--	0	0	0
42	☐	☑	Baseplate Temp	K-type	C	10000	0	1372
43	☐	☑	Reactor Wall Temp	K-type	C	10000	0	1372
44	☐	☑	Coil Temp	K-type	C	10000	0	1372
45	☐	☑	Shower Head Temp	K-type	C	10000	0	1372
46	☑	☑	Spare	None	--	0	0	0
47	☑	☑	Spare	None	--	0	0	0
48	☑	☑	Spare	None	--	0	0	0
49	☑	☑	Spare	None	--	0	0	0
50	☑	☑	Spare	None	--	0	0	0
51	☑	☑	Spare	None	--	0	0	0
52	☑	☑	Spare	None	--	0	0	0
53	☑	☑	Spare	None	--	0	0	0
54	☑	☑	Spare	None	--	0	0	0
55	☐	☐	New MFC	MFC	sccm	5000	0	1000
56	☑	☑	Spare	None	--	0	0	0
57	☑	☑	Spare	None	--	0	0	0
58	☑	☑	Spare	None	--	0	0	0
59	☑	☑	Spare	None	--	0	-6	0
60	☑	☑	Spare	None	--	0	-5	0
61	☑	☑	Spare	None	--	0	0	0
62	☑	☑	Spare	None	--	0	0	0
63	☑	☑	Spare	None	--	0	0	0
64	☑	☑	Spare	None	--	0	0	0
65	☑	☑	Spare	None	--	0	0	0
66	☑	☑	Spare	None	--	0	0	0
67	☑	☑	Spare	None	--	0	0	0
68	☑	☑	Spare	None	--	0	0	0
69	☑	☑	Spare	None	--	0	0	0
70	☑	☑	Spare	None	--	0	0	0

JR2 (3 of 4)



Analogs Configuration

Analog ID	Spare	Read Only	Analog Name	Analog Type	Units	Input High (mV)	Input Low (mV)	Input Value Max
71	☑	☑	Spare	None	--	0	0	0
72	☑	☑	Spare	None	--	0	0	0
73	☑	☑	Spare	None	--	0	0	0
74	☑	☑	Spare	None	--	0	0	0
75	☑	☑	Spare	None	--	0	0	0
76	☑	☑	Spare	None	--	0	0	0
77	☑	☑	Spare	None	--	0	0	0
78	☑	☑	Spare	None	--	0	0	0
79	☑	☑	Spare	None	--	0	0	0
80	☑	☑	Spare	None	--	0	0	0
81	☑	☑	Spare	None	--	0	0	0
82	☑	☑	Spare	None	--	0	0	0
83	☑	☑	Spare	None	--	0	0	0
84	☑	☑	Spare	None	--	0	0	0
85	☑	☑	Spare	None	--	0	0	0
86	☑	☑	Spare	None	--	0	0	0
87	☑	☑	Spare	None	--	0	0	0
88	☑	☑	Spare	None	--	0	0	0
89	☑	☑	Unused	None	--	0	0	0
90	☑	☑	Unused	None	--	0	0	0
91	☑	☑	Unused	None	--	0	0	0
92	☑	☑	Unused	None	--	0	0	0
93	☑	☑	Unused	None	--	0	0	0
94	☑	☑	Unused	None	--	0	0	0
95	☑	☑	Unused	None	--	0	0	0
96	☑	☑	Unused	None	--	0	0	0
97	☑	☑	Unused	None	--	0	0	0
98	☑	☑	Unused	None	--	0	0	0
99	☑	☑	Unused	None	--	0	0	0
100	☐	☑	Carrier Gas	MFC	sccm	5000	0	10000
101	☐	☐	PV INNER	Temp	C	10000	0	1500
102	☐	☐	SP INNER	Temp	C	10000	0	1500
103	☐	☐	PV OUTER	Temp	C	10000	0	1500
104	☐	☐	SP OUTER	Temp	C	10000	0	1500
105	☐	☐	Motor	Motor	RPM	10000	0	10000

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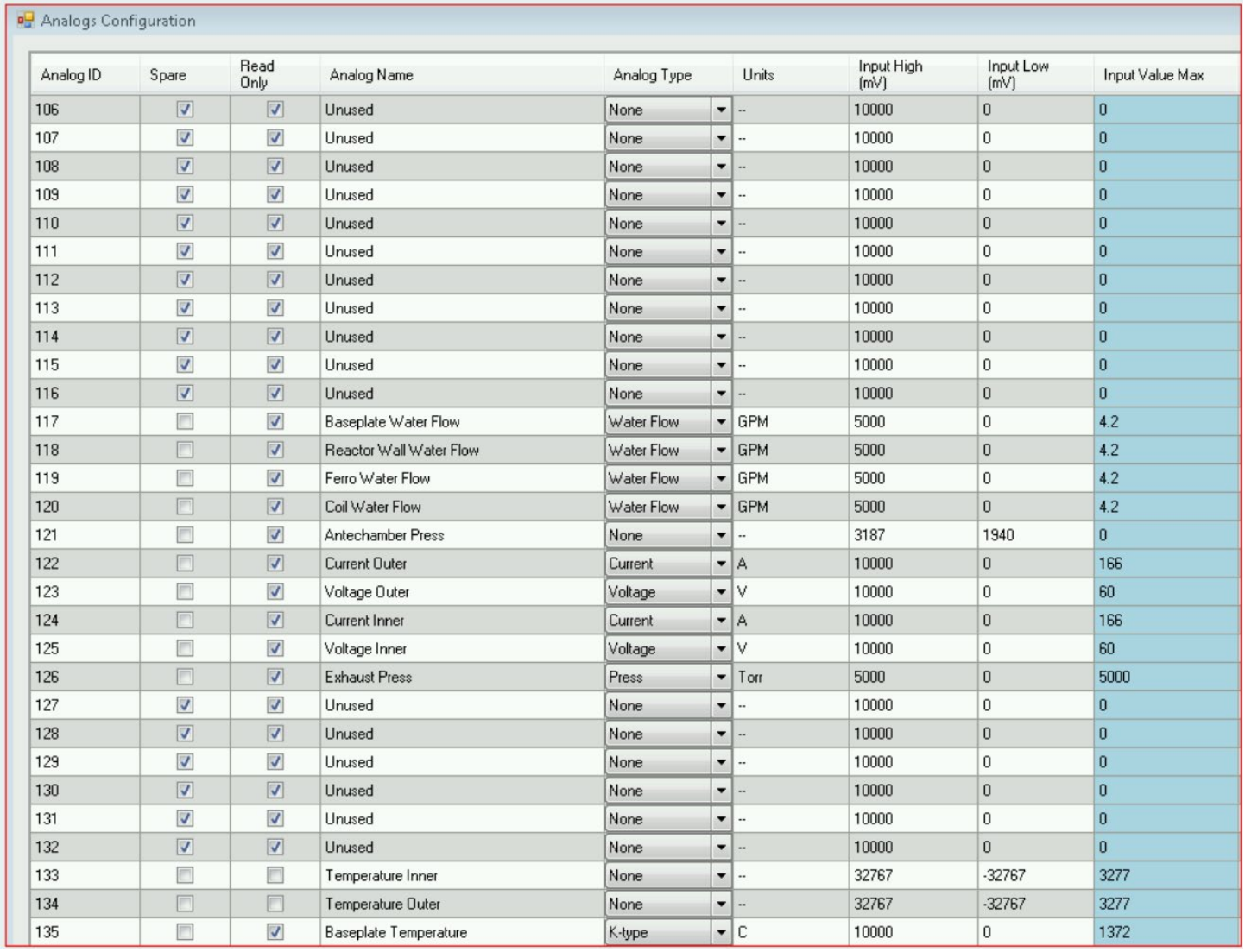

Analogs Configuration

Analog ID	Spare	Read Only	Analog Name	Analog Type	Units	Input High (mV)	Input Low (mV)	Input Value Max
106	☑	☑	Unused	None	--	10000	0	0
107	☑	☑	Unused	None	--	10000	0	0
108	☑	☑	Unused	None	--	10000	0	0
109	☑	☑	Unused	None	--	10000	0	0
110	☑	☑	Unused	None	--	10000	0	0
111	☑	☑	Unused	None	--	10000	0	0
112	☑	☑	Unused	None	--	10000	0	0
113	☑	☑	Unused	None	--	10000	0	0
114	☑	☑	Unused	None	--	10000	0	0
115	☑	☑	Unused	None	--	10000	0	0
116	☑	☑	Unused	None	--	10000	0	0
117	☐	☑	Baseplate Water Flow	Water Flow	GPM	5000	0	4.2
118	☐	☑	Reactor Wall Water Flow	Water Flow	GPM	5000	0	4.2
119	☐	☑	Ferro Water Flow	Water Flow	GPM	5000	0	4.2
120	☐	☑	Coil Water Flow	Water Flow	GPM	5000	0	4.2
121	☐	☑	Antechamber Press	None	--	3187	1940	0
122	☐	☑	Current Outer	Current	A	10000	0	166
123	☐	☑	Voltage Outer	Voltage	V	10000	0	60
124	☐	☑	Current Inner	Current	A	10000	0	166
125	☐	☑	Voltage Inner	Voltage	V	10000	0	60
126	☐	☑	Exhaust Press	Press	Torr	5000	0	5000
127	☑	☑	Unused	None	--	10000	0	0
128	☑	☑	Unused	None	--	10000	0	0
129	☑	☑	Unused	None	--	10000	0	0
130	☑	☑	Unused	None	--	10000	0	0
131	☑	☑	Unused	None	--	10000	0	0
132	☑	☑	Unused	None	--	10000	0	0
133	☐	☐	Temperature Inner	None	--	32767	-32767	3277
134	☐	☐	Temperature Outer	None	--	32767	-32767	3277
135	☐	☑	Baseplate Temperature	K-type	C	10000	0	1372

Revision History

Rev	Date	Originator	Description of Changes
1	2/06/2024	Kathy Anderson	Initial Release