Manual

for

Metalorganic Chemical Vapor Deposition System

in

Room EP 240

at

South Dakota School of Mines & Technology

Nanoscience & Nanoengineering

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Overview of System Operation and Features

A MetalOrganic Chemical Vapor Deposition (MOCVD) system has been constructed at SDSM&T for the growth of epitaxial layers of semiconducting compounds formed from elements in groups III and group V of the periodic table. The alloys which can be grown include those containing indium, aluminum, gallium, arsenic and phosphorus. The potential exists for the grown layers to be doped with impurities to form either n-type or p-type layers. The system is housed in Room 240 of the Electrical Engineering/Physics Building.

Operational features

Sources

1. The group V sources are provided in the form of gases by the hydrides AsH_3 and PH_3. Quantities are limited to 0.21 cu. ft. cylinders to reduce the consequences of a catastrophic release of the contents. These sources may also be provided by alkyl bubblers of TertiaryButylArsine (TBAs) or TertiaryButylPhosphine (TBP)
2. Group III sources are provided by alkyl bubblers of EthylDiMethylIndium (EDMln), TriMethylGallium (TMGaa), and TriTethyAluminum (TMA).
3. N-dopants can be provided either by silane diluted to 10 percent in hydrogen or by a TertaEthylTin
4. (TESn) bubbler.
5. P-dopants can be provided either by DiMethylZinc (DMZ) diluted to 0.1 percent in hydrogen or by a carbon tetrachloride bubbler.
6. The dopant for growing semi-insulating material is iron provided by a ferrocene bubbler.

Carrier gases

1. Hydrogen from a palladium purifier is used as the carrier for all the alkyl sources and as the primary diluent for the reactor gases.
2. Nitrogen purified by a Nanochem purifier is used as an alternative diluent and purge gas for displacement of hydrogen in the reactor.

Input gas control

1. Gas flows are controlled by precision Mass Flow Controllers from Vacuum General.
2. Gas valving is with Nupro BNV series, low-dead space bellows valves. Normally closed valves provide fail-safe operation.
3. Vent/run valving to the chamber is performed with a Thomas Swann ten-port, low-dead-space manifold.
4. The vapor pressure of the alkyl sources is controlled by constant temperature Lauda/Brinkman cooling baths.

Gas delivery plumbing

1. All-welded, electropolished, pre-cleaned, semiconductor grade stainless steel tubing is used in the gas system.
2. All tubing connections are with Nupro VCR metal gasket fittings.
3. Some Nupro bellows valves have been modified by adding ports to eliminate dead space in critical gas lines. Low dead-space tee connections are within the valve bodies.

Reactor chamber

1. The circular cross section reaction chamber is part of a vertical flow system in which gases enter from the top and impinge on a horizontal substrate supported on a graphite susceptor which is heated by inductive coupling from an RF magnetic field.

2. A high velocity air stream flows over the walls of the quartz upper deposition chamber to keep the walls cool and prevent deposition on the quartz. The lower portion of the chamber which is constructed of stainless steel is water cooled.
3. The system is operated at pressures in the range 7.6 -76.0 Torr.

Exhaust gas control

1. The chamber exhaust passes through a Vacuum General AdapTorr pressure controlling valve before it enters the vacuum pump. This allows constant chamber pressure to be maintained regardless of the gas flow rate.
2. The source gas vent lines are connected downstream of the chamber at the inlet to the pressure control valve to provide the same pressure on both the "vent" and "run" gas routes.
3. A particle filter on the pump inlet and a recirculating oil filter system on the pump are used to remove solid and corrosive contaminants from the exhaust gas to promote long pump life.
4. Continuous nitrogen gas-ballast through the pump is used to control condensation of condensable vapors and to scrub dissolved toxic vapors from the pump oil between runs.
5. Exhaust gas from the pump is scrubbed by a charcoal filter to remove arsine and phosphine vapors before the exhaust is vented to air.

Operating controls

1. The reactor can be operated by manual controls on the reactor panel. All valving is done with switches, and the gas flow of the individual gas source is digitally displayed.
2. Alternatively, the reactor can be computer operated. Interfacing of the valve and flow controllers is provided via a Hewlett Packard Multiprogrammer controlled by a PC running LabView.

Safety features

1. Normally closed bellows valves are used for fail-safe protection from toxic gases.
2. Pneumatic valve operation assures positive, leak-tight shut-off.
3. All-welded stainless steel plumbing with Nupro VCR compression gasket fittings meets requirements for hazardous production gases.
4. A ventilated reactor cabinet with separate sections for the chamber, toxic gases, gas control, and the electronics contains all of the hazardous gas components.
5. Flow reducing orifices are used on all the gas cylinders.
6. The reactor will shut down for:
   a. Failure of cabinet exhaust.
   b. Detection of PH3 or AsH_3 leaks.
   c. Detection of hydrogen leaks.
   d. Loss of pneumatic valve air pressure.
   e. Low hydrogen pressure.
   f. Low nitrogen pressure.
   g. Power failure.
   h. Loss of chamber vacuum.

Valve switching features assuring safe operation

1. Computer operation of the valves is set individually for each valve; only those switched to remote operation can be computer activated.

2. All interlocking of valves to assure that certain combinations do not occur, or conversely that certain combinations occur together, is hard-wired and not dependent on operator performance or software. The interlocking operates the same for both manual and computer operation.
3. The interlock system, when in abort status, removes power from the solenoid valves to return them to their normally closed position. The abort mode overrides all valve settings under either manual or computer control for fail-safe shut down of the gas flows.
4. The system abort mode applies a valve close signal to the mass flow controllers and sounds an audio alarm.

Vacuum system operational features

1. The reactor exhaust system is vacuum tight.
2. The vacuum pump is sealed from the atmosphere to contain all process gases.
3. Nitrogen gas us used to provide ballast to the vacuum pump. This helps to purge the pump of gases trapped in the pump oil.
4. A solenoid valve introduces nitrogen only when the pump is on (this prevents pressurizing the pump case and forcing oil up the inlet).
5. The gas ballast flow is controlled by a manual metering valve and measured with a rotameter to limit the flow and provide the pressure drop from the 15 psi delivery pressure to the pump case.
6. The pump exhaust lines are gas tight to prevent leakage of toxic gas.
7. Continuous gas flow (from the combined gas ballast and the inlet flow) keep air out of the pump case and the exhaust lines to prevent explosive mixtures of H_2 and air.
8. Loss of N_2 supply pressure is interlocked to close the valve on the gas ballast input to the vacuum pump.
9. Catastrophic loss of vacuum during reactor operation closes vacuum exhaust valve EXPV2 to the pump inlet to prevent pumping of air.
10. Loss of vacuum during reactor operation puts the interlock system in abort mode which terminates all gas flows.

Interlock system

The system is shut down and latched-off if any of the conditions listed below arise.

1. Power failure (system power must be reset to operate any sources; only N_2 flow can turn on when power is resumed).
2. AsH_3/PH_3 monitor alarm (set to monitor for the TLV level in both the gas cabinet exhaust and in the adjacent lab space).
3. H_2 at 20% of the explosive limit alarm.
4. Exhaust ventilation failure alarm.
5. Low H_2 supply pressure alarm.
6. Low N_2 supply pressure alarm.
7. Low of vacuum in chamber alarm.
8. Loss of pneumatic valve air supply pressure.
9. Emergency stop button activated.

Control panel design criteria

1. Controls for each source input are organized in individual blocks on the reactor front panel. During manual operation, the operator's attention is thus focused on one block at a time minimizing the possibility of error.
2. The control layout for each source is in a left-to right sequence according to a normal sequence of operating steps. This keeps the operator aware of the gas circuit function.

3. The spatial positions of like controls are maintained from one gas source to another so that the operator can recognize the switch functions by the layout format.
4. No map layout of the gas control has been used because the complexity with such a large number of sources and dopant lines is too visually confusing.
5. All valve controls are hardware interlocked to prevent improper or hazardous valve combinations for operation by either manual or computer control.
6. All valves are electrical-switch operated for manual or computer control.
7. Computer control requires a two step enabling procedure before a source valve can be turned on. If a single switch for a control panel is turned on inadvertently, no valves will be activated.
8. All mass flow controllers can be operated from either the panel set-point dial or by a computer generated D/A signal.
9. All hazardous conditions cause the system to shut-down to an abort status. The response is as follows:
    1. All valves close.
    2. Heater power is shut off.
    3. A panel indicator identifies the problem.
    4. An audio signal is sounded.
    5. The system must be reset to continue.
    6. A system power failure will switch the system to abort status. The system must be reset to continue.

Gas system criteria

1. All stainless steel gas lines.
2. All-welded fittings with compression gasket seals.
3. Low dead-space vent/run manifold for gas delivery to the reactor (Thomas Swann "Epifold" inline manifold). Its features are:
4. Vent lines at same pressure as reactor to prevent gas switching pressure transients.
5. Small internal volume manifold to reduce residence time of gases and dead-space.
6. Compact in-line arrangement of 10 input valves, 5 each on opposite sides.
7. H_2 and/or N_2 diluent gas through the manifold to carry sources gases to the reactor.
8. Electronically controlled pressure balance between the vent and run manifolds.
9. N_2 diluent gas through the vent line manifold.
10. Each vent/run valve is backed up with a positive shut-off, normally closed, pneumatically operated bellows valve for fail-safe turn off of source gases in an emergency.
11. Hydride (AsH_3 and PH_3) source lines can be operated with either the hydride or H_2.
12. The hydride and H_2 alternate input valves are interlocked to prevent simultaneous operation.
13. The hydride and H_2 input valves are normally closed for fail-safe turn off in an emergency. This prevents any hydride back diffusion into the H_2 delivery line as would occur if a 3-way valve had been used.
14. The gas input valves cannot operate unless the outlet valve is also open.
15. A valve closure signal is applied to the mass flow controller when the outlet valve is closed.
16. Alkyl bubbler source lines operate with H_2 carrier gas. The bubbler pressure is controlled by mechanical pressure regulators upstream of the bubblers. The pressure differential between the bubblers and the deposition chamber is maintained by electronic mass flow controllers between the bubblers and the chamber. A constant source mole fraction is maintained regardless of the pressure in the deposition chamber.
17. H_2 carrier gas for each bubbler can bypass the bubbler and flow through the circuit when the alkyl source is in the off position.
18. The bubbler valves can open only if the output valve is open.

19. The bubbler valves can open only if the chamber pressure is below a preset limit.
20. The bubbler bypass valve closure is delayed for 2 seconds following opening of the bubbler valves to assure that input and output pressures equilibrate to prevent back flow of liquid.
21. Dopant sources are introduced in three separate circuits to allow sequential growth of n, p, and semi-insulating layers.
22. A H_2 diluent scheme is used to provide a four decade range of dopant gas concentration on each dopant circuit.
23. Each class of dopants accommodates two different sources which can be used one at a time without provision for their mixing or sequencing.
24. Valving on each dopant circuit is interlocked to prevent simultaneous operation of the two dopants and to assure safe operation.
25. Modified valves are used to eliminate any dead space in the circuits.
26. The three circuits (n, p, and semi-insulating dopant) can be used independently or together.
27. Any unused dopant line can function as an H_2 input to the manifold to balance total gas flows when switching from one source to another to prevent flow transients to the reactor.
28. Large-size (2 inch 0.D.) valves are used on the reactor exhaust to protect against plugging with solid residues.
29. A vacuum exhaust valve opens to the pump for growth at reduced pressures.
30. A pressure control valve on the pump inlet is controlled by a sensor on the reactor to maintain constant, preset reactor pressure.
31. A separate sample-entry chamber and load lock system allows samples to be inserted and removed from the deposition chamber without the necessity of letting air into the reaction chamber. This improves the purity of the grown material by eliminating water vapor absorption on the chamber walls. Hazardous H_2 and source gases are prevented from venting to atmosphere for operator safety.
32. The exhaust valves are interlocked to prevent simultaneous operation.
33. A 1 psig pressure relief valve prevents inadvertent over pressure of the reactor if the exhaust valve is closed.

Computer interface

The operations necessary for layer growth can be remotely programmed by a desktop computer.
The features of this system are;

1. Uses LabView for simplified operation.
2. Magnetic disk mass storage for retraining a library of operating programs.
3. Printout of process parameters.
4. All low level functions such as keeping track of relay numbers and programming commands for the D/A converters are performed in subroutine modules which have been previously debugged and tested. The programming statements used for a growth sequence are character strings which have a logical relationship to the function being performed. This simplifies the programming and greatly minimizes the possibility of error in writing programs for growth sequences. It is not possible to program the wrong valve to be open by entering an incorrect decimal or binary number in a growth program since numbers are not used in the high-level programming commands.
5. Each gas circuit must first be set in the REMOTE mode by computer control before any other circuit functions can be programmed by computer. This two-step sequence minimizes the possibility that a valve will be accidentally turned on under computer control.
6. The gas flows necessary for a growth sequence can be programmed by set-point voltages applied by D/A converters under computer control. The set-point voltages can also be applied from potentiometers on the front panels.

7. The actual flow rates through the mass flow controllers, the pressure in the deposition chamber, and the susceptor temperature can be measured under computer control to provide automatic data logging during a growth sequence.

Emergency Procedures

Background

The DOD ChemLogic CL8 toxic gas monitor which monitors the MOCVD cabinet and the laboratory space in Room 240 is connected to the building evacuation alarm. The system is connected so that if a level of toxic arsine gas in excess of the ACGIH TWA-TLV (Time Weighted Average - Threshold Limit Value) of 5ppb occurs in either the MOCVD system cabinet, the reactor cabinet, the toxic gas cabinet, the exhaust gas outlet, the fume hood exhaust, or 2ppb in the laboratory space in Room 240 then an audio alarm on the MOCVD system will sound and the interlock circuits for the MOCVD system will go into the abort mode. All of the pneumatic valves for the MOCVD system that carry hydrogen and the source gases will close automatically. These valves cannot reopen until the system is manually reset. These events are not life threatening and do not trigger the building alarm but the gas monitor light tower located in the hallway immediately outside Room 240 will light red and an audio alarm will sound.

If an arsine concentration that is in excess of the OSHA TWA-TLV (Time Weighted Average - Threshold Limit Value) of 50ppb is detected in either the MOCVD system cabinet, the reactor cabinet, the toxic gas cabinet, the exhaust gas outlet, the fume hood exhaust, or 10ppb in the laboratory space in Room 240; the gas monitor light tower located in the hallway immediately outside Room 240 will flash red and an audio alarm will sound the evacuation alarm for the building will sound and the fire department will be notified. The strobe light outside Room 118 by the door on the northeast corner of the building will come on and audible chime will sound. This chime is distinct from the building alarm horns to indicate that the evacuation is due to a toxic gas event and not a fire. The building fire alarm system latches on when triggered and stays on until it is reset, even if the triggering event is no longer active. The strobe outside room 118 does not latch and will only indicate an active gas event inside room 240.

Arsine and the hydrogen carrier gas are flammable. Arsine is a toxic colorless gas with a garlic-like odor. The odor threshold is 0.5 ppm. You cannot smell arsine at either the OSHA or ACGIH TWA-TLV ceiling level. The DOD ChemLogic CL8 toxic gas monitor is set to give an alarm at the TWA ceiling which is the same as the TWA-TLV. Pure arsine has a specific gravity of 2.695 (air=1).

Pure phosphine is pyrophoric. The hydrogen diluent gas is flammable. Phosphine is a toxic colorless gas with a disagreeable characteristic fishy, garlic-like, or acetylene-like odor. The odor threshold is 0.021 ppm. The OSHA TWA ceiling is 0.3 ppm so phosphine can be detected by smell at the TWA ceiling. The DOD ChemLogic CL8 toxic gas monitor is set to give an alarm at 0.005 ppm. Pure phosphine has a specific gravity 1.17 (air=1).

The liquid alkyl sources used in the MOCVD system are all pyrophoric (they burn spontaneously when exposed to air). If a spill of one of these liquids occurs, the material will slowly burn as it is oxidized by the air. Some of these liquids react violently when mixed with water which should never be applied to the burning material. Use either a dry chemical fire extinguisher or absorbent clay to smother the fire. The fumes from the burning material are in some cases toxic. Do not inhale them. If the burning material is presenting a hazard to personnel it is best to leave the area and let the material burn until it is consumed. If the smoke is escaping from Room 240 and the building evacuation alarm has not already sounded, then activate one of the building alarm stations to evacuate the building.

Action to be taken

If the building evacuation alarm sounds while the MOCVD system is in operation, the operator should immediately check the light bar outside room 240. If the lamp is flashing red, the evacuation is due to a toxic gas event. Since the MOCVD system will shut down automatically, no corrective action should be taken by the operator. The operator will immediately leave the laboratory space and alert others in the vicinity to also leave immediately. If the operator is also a member of the Emergency Response Team (ERT), they should proceed immediately to the stock room of the physics shop (EEP 232) and retrieve their SCBA before vacating the building. Once outside the building, check with facilities and campus safety to make sure no one is working on the penthouse roof of the EEP building as the vent stack from EEP 240 does not extend above the penthouse roof. The remote monitoring company (Knight Security, 343-3333 x3) will notify the Rapid City Fire Department (RCFD) that the alarm was initiated by the TGM and will inform the RCFD that the HAZard MATerials (HAZMAT) team should respond. The operator should then wait outside the building to provide assistance to the HAZMAT team as needed. The system operator is a valuable resource person since he/she knows better than anyone what the HAZMAT team will encounter. There are two possible scenarios at this point. Either the level of toxic gas in Room 240 will be above the TLV, or it will not.

Level of gas below TLV

It is likely that once the pneumatic valves on the cylinder outlets close, the gas level will drop below the TLV and it is safe to reenter the building. If this is the case, the building alarm will continue to sound but the strobe outside the power room (Room 118) on the first floor northeast corner will be out. Once the building alarm had been reset by Physical Plant personnel, it is safe to proceed to the laboratory space in Room 240. The valves on the high pressure cylinders for arsine and phosphine should be turned off until the cause or the release is determined, repaired, and the system is purged and leak tested. The individual channel for the point where the gas was detected should be examined on the monitor. Once the gas concentration has gone to zero, use the Matheson model 8057 portable hazardous gas detector (red leather case) kept in Room 240 to check for leaks in the system. It would be prudent to use the portable gas detector to detect hydrogen leaks in the system before flowing any toxic gasses.

Level of gas above TLV

If the level of toxic gas in Room 240 remains above the TLV, the red lamp outside Room 118 will stay on and the building evacuation alarm will continue to sound. Under these conditions it is unsafe to enter the building without the protection of a self contained breathing apparatus (SCBA). No fewer than two people, each wearing SCBA, may proceed to Room 240. The first step to secure the system is to close the valves (turn clockwise) on top of the high pressure cylinders for arsine and phosphine. The cylinders are located in the gas cabinet located at the far end of the MOCVD system in room 240. The valves can be accessed by opening the hinged plexiglass window. Check to see if the exhaust for the cabinet is drawing air and leave the building. If the exhaust is working, the level of gas in the room should fall below the TLV in a few minutes. This will be indicated then the strobe light outside room 118 goes out. At that point it is safe to enter Room 240 without emergency breathing apparatus.

The individual channel for the point where the gas was detected should be examined on the monitor. Once the gas concentration has gone to zero, use the Matheson model 8057 portable hazardous gas detector (red leather case) kept in Room 240 to check for leaks in the system. It would be prudent to use the portable gas detector to detect hydrogen leaks in the system before flowing any toxic gasses.

Visible flame in Toxic Gas Cabinet

One possible scenario is that gas may be leaving a portion of the plumbing system in sufficient quantity to cause a flame. If this is happening and the gas bottles cannot be approached without endangering the emergency team, then a reasonable course of action would be let the fire continue to burn until the gas bottle is empty. The total volume gas contained in these cylinders has been kept low (0.21 cu. ft.) to allow this option.

Brief summary of emergency procedures

If the building evacuation alarm is activated by a toxic gas release:

1. Leave the building immediately.
2. Check with Campus Safety to ensure no one is working on the EEP penthouse roof.
3. Stand by to provide information and assistance to the fire department.
4. If an alkyl spill occurs use only a dry chemical fire extinguisher or absorbent clay to extinguish the fire. Otherwise let it burn.

Interlock System

A series of system wide interlocks sense the states of a number of transducers which can detect conditions which require immediate intervention. Some of these conditions present immediate or potential operating hazards and some of the conditions jeopardize the integrity of the source materials. The interlocks have been connected to system controls in a manner such that appropriate action is taken without operator intervention. The system will be shut down, an alarm will sound, and a light on the ABORT panel will come on, indicating the nature of the malfunction. If a transducer, say for example the smoke detector, sends out an alarm which shuts down the system and at later time the smoke goes away, the system cannot restart unless it is manually reset. Also, the lamp on the ABORT panel will stay on indicating the nature of the fault even though it has cleared. The transducers are all wired in such a manner that their normal (or no alarm) state is a closed switch. If wires to the transducer are broken or become disconnected, an alarm will be indicated. The ABORT panel contains a key-operated switch that deactivates the alarm for servicing purposes. During normal operation the alarm should enabled and the key removed. The key can be removed only when the alarm is enabled.

Alarm Inputs

The transducer inputs and their functions are as follows:

AsH_3/PH_3 MONITOR	An output signal from the DOD Toxic Gas Monitor indicating the presence of toxic gas in the MOCVD system, the fume hood, or in room 240. If the abort panel activates and the building alarm does not, then the gas is contained within the system.
H_2 LEAK DETECTOR	An output signal from the Matheson hydrogen gas purifier indicating the presence of hydrogen gas above the level of 4000 ppm.
EXHAUST AIR FLOW	An output signal from a differential manometer checks for loss of air flow through the venting system.
LOW H_2 SOURCE PRESSURE	A pressure switch which checks for sufficient hydrogen pressure for proper operation of the bubbler sources.
LOW N_2 SOURCE PRESSURE	A pressure switch which checks for sufficient nitrogen pressure for the nitrogen diluent system.
CHAMBER VAC LOSS	A signal indicating that the vacuum deposition chamber has come up to atmospheric pressure in an abnormal fashion. In order for this circuit to be activated the exhaust valve between the chamber and the vacuum pump must be open and the system must have first been evacuated to a pressure of 100 Torr or less. This circuit would be activated if the quartz chamber were to break in the middle of a run or if the vacuum pump failed in the middle of a run. The three conditions necessary for a chamber vacuum loss signal are (1) the exhaust valve must be open, (2) the vacuum latch must be off, and (3) the chamber must be at atmospheric pressure. See the section on vacuum control for further details.
SMOKE DETECTOR	A combined signal from a smoke detector and a heat detector in the MOCVD cabinet. Both are located near the vent outlet for greatest sensitivity.
HIGH H_2 HUMIDITY	An output signal from the Panametrics hygrometer indicating high humidity in the hydrogen gas coming from the Matheson hydrogen purifier. This would probably be caused by a rupture in the internal membrane of the purifying cell. The system is shut down automatically so that the moisture in the hydrogen does not contaminate the bubbler sources.
HIGH N_2 HUMIDITY	An output signal from the Panametrics hygrometer indicating high humidity in the

	nitrogen gas coming from the dessicator NP1. This would indicate that the dessicator cartridge should be replaced.
EMERGENCY STOP	Indicates that the red EMERGENGY STOP button has been pressed. This is the preferred method for shutting down the system quickly since a nitrogen flow into the vacuum pump inlet is maintained.
POWER FAIL	Indicates that the power from the 24 VDC supply has been interrupted either by a failure of the power supply or an interruption of the electrical service.

Interlock Structure

The interlock system operates by disconnecting the +24 v power lead from circuits which are to be shut down. Since all of the pneumatic systems are designed to be in the closed state when power is removed, disconnecting the power provides a safe shutdown of the system. An alarm signal from any of the above interlocks de-energizes relay ABRTK12 which removes power from the A level interlock system. If no alarms are being received from any of the above sensors, then all of the A-level systems are powered up. If the A-level system is up and the vacuum valve (EXPV2) between the deposition chamber and the vacuum pump is open then all B-Ievel systems are powered up. If the B-Ievel system is up, the vacuum chamber is evacuated (pressure < 400 Torr), and the SCRUBBER OXIDATION switch on the VACUUM CONTROL panel is in the off position; then the all C-level systems are powered up. If the C-Ievel system is up and either the nitrogen or the hydrogen manifold diluent gas is turned on, then all D-Ievel systems are powered up.

ABORT Status

If the A-level system is powered down (ABORT status) then the following components are active:

1. If the vacuum pump is on, then EXSV5 is energized which turns on EXPV5. This feeds nitrogen into the inlet of the vacuum pump to maintain a pressure of 0.1 Torr and prevent the upstream plumbing from being coated with vacuum pump oil. The green DEP CHAMBER light for the NITROGEN FLUSH system will be on. The other NITROGEN FLUSH feeds will be disabled.
2. The N_2 CARRIER function on the HYDROGEN/NITROGEN control panel is enabled. The H_2 CARRIER function is disabled.

A-Level Status

If the A-level system is powered up then the following components are active:

1. The REMOTE/LOCAL function is activated on the ALUMINUM, INDIUM, ARSENIC LIQUID, PHOSPHORUS LIQUID, ARSINE, PHOSPHINE, IRON, GALLIUM, N DOPANT, and P DOPANT control panels.
2. The ACTIVE/INACTIVE function is activated on the ALUMINUM, INDIUM, ARSENIC LIQUID, PHOSPHORUS LIQUID, ARSINE, PHOSPHINE, IRON, GALLIUM, N DOPANT, and PDOPANT control panels.
3. The substrate spinner can be activated.
4. The substrate heater can be activated
5. The substrate can be raised or lowered.
6. The nitrogen manifold diluent can be turned on if there is vacuum in the deposition chamber.
7. Solenoid valve AUXSV1 is energized which opens ASGPV20, PGPV20, NDOPPV20, and PDOPPV20.

B-Level Status

If the B-Ievel system is powered up then all A-level components are active in addition to the following:

1. The nitrogen manifold diluent can be turned on regardless of whether or not there is vacuum in the deposition chamber.

C-Level Status

If the C-level system is powered up then all B-level components are active in addition to the following:

1. The hydrogen manifold diluent can be turned on.

D-Level Status

If the D-level system is powered up then all C-level components are active in addition to the following:

1. All pneumatic valves for the ALUMINUM system can be opened.
2. All pneumatic valves for the INDIUM system can be opened.
3. All pneumatic valves for the ARSENIC LIQUID system can be opened.
4. All pneumatic valves for the PHOSPHORUS LIQUID system can be opened.
5. All pneumatic valves for the IRON system can be opened.
6. All pneumatic valves for the GALLIUM system can be opened.
7. All pneumatic valves for the N-DOPANT system can be opened.
8. All pneumatic valves for the P-DOPANT system can be opened.

Hydrogen/Nitrogen

Gas Distribution

The hydrogen and nitrogen gas flows are controlled from a common panel on the electronics cabinet. Nitrogen and/or hydrogen gas must flow through the Swan fast switching manifold in order for the source gas controllers to be enabled. The flow rates for nitrogen and hydrogen manifold diluent gases can be controlled separately. In addition either nitrogen or hydrogen gas can be used for the transport gas through the source bubblers. Nitrogen is used only for purging purposes and hydrogen is used both for both source purging and vapor transport during growth of layers. The hydrogen and nitrogen flows for the vapor carrier system cannot be turned on simultaneously.

Hazards

1. Nitrogen is an asphyxiant. If the oxygen in the room were displaced with nitrogen, suffocation could occur. With the room ventilation at approximately 1100 cubic feet/minute, this poses a small hazard since the total volume of nitrogen in the high-pressure cylinder is 230 cubic feet. If a liquid source of nitrogen is used, then the room should be evacuated if a massive leak of nitrogen gas or liquid occurs which cannot be stopped by turning off the valve on the source cylinder.
2. Hydrogen is flammable and potentially explosive. Hydrogen mixtures between the limits of 4.0% and 75% by volume in air are flammable. It is very important that hydrogen-air mixtures not be present in the components of the reactor. For example, if hydrogen mixed with air were present in the deposition chamber with the substrate heater on, an explosion could result, shattering the quartz deposition chamber.
3. The Nanochem nitrogen purification cell contains a lithium resin which reacts with oxygen and water vapor. Excessive heating of the cell would occur if air were to flow through the cell. The lines should be carefully purged before opening the inlet and outlet valves.

Precautions

1. Each hydrogen cylinder is equipped with an excess flow shutoff valve which closes if the flow exceeds 5 liters/minute. This prevents a high volume release of hydrogen in the event of rupture of one of the gas lines. The excess flow valves must be manually reset once they are tripped.
2. The hydrogen line contains an over pressure release valve (HPRV1) to protect the components in the event of failure of one of the tank regulators. If the pressure in the line exceeds 150 psig, then the hydrogen is dumped directly into the exhaust vent.
3. The hydrogen supply is enabled by a pneumatic valve (HPV17) which closes if the interlock system is in Abort status.
4. A hydrogen sensor in the hydrogen purifier activates the interlock system and places the system in Abort status.
5. The air bleed system for scrubber oxidation cannot be turned if the hydrogen manifold diluent is on, or if any of the source gases are turned on.
6. System shutdown occurs if the pressure in the nitrogen supply line falls below 10 psi or the pressure in the hydrogen supply line falls below 50 psi.

Normal Operation

When the reactor is in an idle state a small quantity of nitrogen flows through the system to continuously purge the vacuum pump. There is no hydrogen flow in the idle state. At the end of the day the hydrogen supply should be turned off at the high pressure cylinders and the nitrogen supply should be left on.

Start of day

1. Check that the nitrogen supply is on and that there is sufficient nitrogen pressure in the cylinder for the days operation; about 400 psig for a 216 cu. ft. high pressure cylinder.
2. Turn on the valves for both hydrogen cylinders. The outputs of both hydrogen regulators feed into a common supply line for the MOCVD reactor. If the pressure regulators for the tanks are set correctly, when the main tank is empty, the backup tank will automatically come on. The procedure for doing this is in the section on cylinder replacement. The outlet pressure should be 120 psig.
3. During a normal growth run the carrier gas should be hydrogen. Nitrogen should be used in the carrier gas circuit only during purging of the lines and changing cylinders. On the HYDROGEN/NITROGEN control panel the switch which selects the carrier gas has no off position. Either hydrogen is selected or nitrogen is selected. When the switch is in the CARRIER N_2 position then valve NPV13 is open and HPV13 is closed. When the switch is in the CARRIER H_2 position then valve HPV13 is open and NPV13 is closed. Two panel lamps indicate the gas selected. The selection of the carrier gas cannot be implemented from the remote computer.
4. The manifold diluent gas may be either hydrogen or nitrogen or a combination of the two. The selection of the gases and the flow rates may be made remotely from the computer. When the MANIFOLD N_2 switch is turned on, NPV4 opens and nitrogen begins to flow through NMFC1, through the Swan manifold and into the deposition chamber. The MANIFOLD N_2 DVM on the control panel indicates the nitrogen flow through NMFC1 on the HYDROGEN/NITROGEN control panel. When the DVM1 switch is pressed in, the set point for NMFC1 can be adjusted using the FLOW SET 1 potentiometer. When the MANIFOLD H_2 switch is turned on, HPV4 opens and nitrogen begins to flow through HMFC1, through the Swan manifold and into the deposition chamber. The MANIFOLD H_2 DVM on the control panel indicates the hydrogen flow through HMFC1 on the HYDROGEN/NITROGEN control panel. When the DVM2 switch is pressed in, the set point for NMFC2 can be adjusted using the FLOW SET 2 potentiometer.

End of day

1. The MANIFOLD N_2 and the MANIFOLD H_2 switches should both be off.
2. The control panel switches for all of the source materials should be off.
3. Close the valves on both of the hydrogen cylinders.
4. Leave the nitrogen cylinder turned on unless the system is to be completely turned off with the vacuum pump off. If the vacuum pump is turned off, then the valve for the nitrogen cylinder may be turned off.

Interconnections

1. N_2 CARRIER controls NSV2 which drives NPV13.
2. H_2 CARRIER controls HSV2 which drives HPV13.
3. H_2 ON controls relay HNK1which controls HSV1 which drives HPV4.
4. N_2 ON controls relay HNK2 which controls NSV1 which drives NPV4.

Interlocks

1. All functions are disabled in Abort status with the exception of the N_2 CARRIER.
2. In Abort status the N_2 CARRIER function on the HYDROGEN/NITROGEN control panel is enabled. The H_2 CARRIER function is disabled.
3. If the system is not in Abort status and the exhaust valve EXPV2 is closed, the nitrogen manifold diluent can be turned on only if there is vacuum in the deposition chamber.
4. If the system is not in Abort status and the exhaust valve EXPV2 is open, then the nitrogen manifold diluent can be turned on regardless of whether or not there is a vacuum in the deposition chamber.
5. Only if the system in not in abort status and the exhaust valve EXPV2 is open and there is vacuum in the deposition chamber and the air bleed to the scrubber is in the off position, can the hydrogen manifold diluent can be turned on.
6. None of the source gases or bubbler systems can be turned on unless either the hydrogen manifold diluent or the nitrogen manifold diluent is turned on.
7. If any source gas or bubbler system is turned on, then neither the hydrogen manifold diluents nor the nitrogen manifold diluent can be turned off.

Hydrogen purification system

The hydrogen gas from the high pressure cylinder undergoes a three step purification process before it is used in the MOCVD reactor. First it passes through a Matheson model 64-1030A catalytic cell (HP1) which converts oxygen in the gas to water vapor. Next the gas passes through a water absorbing cartridge contained in the Matheson 460 gas purifier (HP2). As a final step the gas passes through a palladium diffusion cell in the Matheson model 8373V hydrogen purifier which reduces the total impurity level to less than 0.5 ppm. With an inlet pressure of 120 psig and an outlet pressure of 20 psig with a cell temperature of 400°C and a bleed flow of 250 sccm, the maximum flow through the purifier is 8 liters/min.
The operating principle of the purification cell is that palladium metal, when heated, allows the selective diffusion of hydrogen. The heated metal also undergoes expansion due to the absorbed hydrogen. If the metal is allowed to cool in the presence of hydrogen it is likely to crack since if becomes less ductile when cold. For this reason the purification cell should never be allow to cool when it contains hydrogen. An automatic switching arrangement connected to the purifier will automatically shut off the hydrogen and purge the cell with nitrogen in the event of a power failure. A low temperature condition inside the cell will trigger an alarm setting within the temperature controller that will also shut off the hydrogen. The system must be manually reset to start the hydrogen flow.

Hydrogen purifier start-up procedure

1. Insure that the tank valve for the nitrogen is open, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Turn the H_2 PURIFIER PURGE switch on the HYDROGEN/NITROGEN control panel to the on position. Under this condition, valves NPV14 and HPV15 will be open and valves HPV14 and HPV16 will be closed. Nitrogen will flow through NPV14, through the purifier, through HPV15 and out the exhaust. The nitrogen flow rate will

controlled by the metering valve on the front panel of the hydrogen purifier. Adjust this valve for a flow of 200 sccm (Note this valve is somewhat unconventional in that the off position is fully clockwise).

3. Make sure that the power to the hydrogen purifier is in the off position.
4. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is lit. If not, press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve.
5. On the HYDROGEN/NITROGEN control panel turn the MANIFOLD N_2 switch to the on position and adjust the FLOW SET 1 potentiometer to 0.30slm. Turn the MANIFOLD H_2 switch on and adjust the FLOW SET 2 potentiometer a reading of 1.00slm. Wait 10 minutes to evacuate the UHP outlet of the purifier.
6. Turn on the purifier. The power up configuration for the temperature controller is on, with a setpoint (SV) of 400°C. The setpoint can be adjusted with the up and down arrow keys on the front panel. If SV is flashing STBY or TUNE, hold down the ▤ key until the setpoint value is steady.
7. Wait 30 minutes for the temperature to stabilize at 400°C. The Manifold H_2 panel meter on the HYDROGEN/NITROGEN control panel should have reached a steady state value of 0.00slm.[1] This indicates that the UPH output of HP3 has been evacuated, which can be verified by checking PR1 for a reading of 25inHg. If these conditions are not reached, it may indicate a leak in the UHP line or a damaged purifier. Do not proceed further until this is repaired.
8. Reset the latched low temperature alarm on the temperature controller by pressing the ▤ key.
9. Move the H_2 PURIFIER PURGE switch on the HYDROGEN/NITROGEN control panel to the off position. This will close valves NPV14 and HPV15 and open valves HPV14 and HPV16. Hydrogen gas will flow through HPV14 into the purifier and the cell temperature (PV) will rise, fall, and rise again; eventually settling at 400°C. Some of the gas will leave the bleed port through HMV5 and HPV16. The remainder will leave through the UHP outlet. The gas flow through the bleed circuit is now controlled by the setting of HMV5, which is located to the left of the hydrogen purifier. Adjust this valve for a flow of approximately 100 sccm as indicated by the rotameter on the front panel of the purifier.
10. Observe the pressure on the output of pressure regulator PR1 (located on the back wall of the system cabinet). It will rise to its setpoint as hydrogen displaces nitrogen in the cell and pure hydrogen appears at the output. Allow hydrogen to purge both inlet and outlet for ≈10 minutes and check that the cell temperature has re-stabilized at 400°C.
11. Adjust regulator PR1 for an outlet pressure of 20 psig.
12. Turn off the MANIFOLD H_2 and MANIFOLD N_2 switches on the HYDROGEN/NITROGEN control panel and continue as appropriate.

Hydrogen purifier shut-down procedure

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is lit. If not, press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve.
3. On the HYDROGEN/NITROGEN control panel turn the MANIFOLD N_2 switch to the on position and adjust the FLOW SET 1 potentiometer to 300sccm. Turn the MANIFOLD H_2 switch on and adjust the FLOW SET 2 potentiometer a reading of 100 sccm. Wait 10 minutes for the H_2 to stabilize.

[1] Drift of the internal calibration circuitry of the MFC may cause readings to vary by ±0.05sccm. Rough recalibration of MFC zero may be performed when valve setting insure there is no flow through the MFC. Refer to the MFC manual for adjustment procedure.

4. Turn the H_2 PURIFIER PURGE switch on the HYDROGEN/NITROGEN control panel to the on position. Under this condition, valves NPV14 and HPV15 will be open and valves HPV14 and HPV16 will be closed. Nitrogen will flow through NPV14, through the purifier, through HPV15 and out the exhaust. The nitrogen flow is now controlled by the metering valve on the front panel of the hydrogen purifier. Adjust this valve for a flow of 200 sccm *(*Note: this valve is somewhat unconventional in that **the off position is fully clockwise***).* Allow the nitrogen flow to purge the hydrogen from the purifier cell for at least 10 minutes
5. Hold down the ▤ key down until the setpoint temperature flashes STBY. **It is very important that the cell not be allowed to cool with hydrogen in it. The cell can be damaged.**
6. When the cell temperature is < 100°C, which takes approximately one hour, reduce the nitrogen flow by turning the valve on the front panel of the purifier counterclockwise. Set for approximately 5 sccm to conserve nitrogen.
7. Turn off the H_2 MANIFOLD and N_2 MANIFOLD switches on the HYDROGEN/NITROGEN control panel. Close the vacuum exhaust valve.
8. Turn off the purifier.

Hydrogen purifier idling procedure

If there is going to be a period of 2 days or less in between use of the unit, it is suggested it be left on. This will aid in maintaining the operating life of the diffusion unit. In order to do this:

1. Perform steps 1-4 in the purifier shut-down procedure.
2. After 30 minutes, reduce the nitrogen flow by turning the valve on the front panel of the purifier counterclockwise. Set for approximately 5 sccm to conserve nitrogen. Note – the temperature will not cool down while idling but it is assumed that any hydrogen in the system will be displaced by nitrogen after one hour.
3. Leave the purifier power on.

Return from idle procedure

1. Adjust the metering valve on the front panel of the hydrogen purifier for a flow of 200 sccm (Note this valve is somewhat unconventional in that **the off position is fully clockwise**).
2. Perform steps 4 and 5 from the purifier start up procedure.
3. Perform steps 9-12 from the purifier start up procedure.

Hydrogen cylinder replacement

The hydrogen supply has two gas cylinders and regulators operating in parallel. They are connected so that when the bottle being used for the primary source becomes empty the second bottle can come on automatically. In order for this to happen the operator must adjust the outlet pressure of the backup regulator so that it is at least 20 psig less than the outlet pressure of the primary source. This is to account for worst case inaccuracies in the readings of the gauges on the pressure regulators. When flow from the primary cylinder stops, the pressure in the shared manifold will drop to the pressure setting of the second regulator. At this point gas will begin to flow through the backup cylinder. The pressure regulator of the primary cylinder will act as a check valve preventing it from being filled with gas from the backup cylinder. If the lines between the hydrogen cylinders and purifier HP3 are filled with air it will be necessary to purge them with hydrogen before HP3 is turned on. Oxygen in concentrations of over 1000 ppm should never be allowed to enter the cell when it contains hydrogen. The palladium alloy is an excellent catalyst for the reaction of hydrogen and oxygen to water vapor. This is an exothermic reaction and could result in overheating and damage to the cell

Hydrogen cylinder change with gas manifold already purged

The following assumes that the plumbing between the hydrogen cylinders and HP3 is already filled with hydrogen and that both hydrogen cylinders are connected; one cylinder being empty and the second partially full.

1. Close the tank valve on the cylinder being replaced (fully clockwise).
2. Close the plug valve with the green handle (either HMV2 or HMV4 depending on the cylinder being replaced).
3. Close the manual valve on the regulator output (either HMV1 or HMV3 depending on the cylinder being replaced).
4. Disconnect the VCR fitting between the plug valve and the manual valve on the outlet of the regulator.
5. Disconnect the fitting connecting the excess flow valve (either HEXFV1 or HEXFV2) to the hydrogen cylinder. Note; this connector has a left-hand thread so it should be turned clockwise (when facing the cylinder) to loosen it.
6. Replace the cap on the hydrogen cylinder and store it in a location where it is chained to the wall until picked up by the vendor.
7. Put the new tank in position, remove the cap on the cylinder, connect the regulator, and reconnect the VCR fitting. The self-centering gasket in the VCR fitting does not need to be replaced every time the tank is changed as long as it is in good condition. However, it probably should not be reused more than four or five times.
8. Turn the pressure control knob on the regulator counterclockwise two turns to reduce the pressure setting of the regulator.
9. Open the plug valve and the valve on the output of the regulator.
10. Set the knob on the excess flow valve to the reset or purge position. To do this, the black knob on the excess flow valve should be turned counterclockwise (when facing the body of the valve) to the stop.
11. Open the valve on the outlet of the hydrogen cylinder. The valve should be opened very slowly at first to avoid a sudden shock to the regulator.
12. Close the manual valve on the outlet of the other hydrogen gas regulator.
13. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
14. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve.
15. On the HYDROGEN/NITROGEN control panel turn the MANIFOLD N_2 switch to the on position and adjust the FLOW SET 1 potentiometer to give a flow rate of 1000 sccm. Turn the MANIFOLD H_2 switch on and adjust the FLOW SET 2 potentiometer to give a flow fate of 200 sccm.
16. The hydrogen pressure at the outlet of the regulators will begin to drop. When it drops below 140 psig, begin to adjust the pressure regulator on the cylinder just replaced slowly upward (clockwise) until its pressure setting is 140 psig. The outlet pressure of the regulator should now remain steady.
17. Re-open the manual valve on the outlet of the regulator for the other cylinder. Turn the pressure on this regulator up to 160 psig. With the regulators set in this manner, gas in the new cylinder will not be used until the older one is empty.
18. Set the excess flow valve on the cylinder that was just replaced to the RUN position. Do this by turning the knob clockwise (as viewed facing the valve) until it is tight.
19. Turn off the hydrogen and nitrogen manifold gas flows on the HYDROGEN/NITROGEN control paned.
20. Close the VAC EXHAUST switch on the VACUUM CONTROL panel.
21. Shut down the system as appropriate.

Initial connection of hydrogen cylinders.

For the initial startup of the system or if the hydrogen feed lines have been disconnected for repairs, it will be necessary to completely purge the lines of air.

1. Connect the cylinders.
2. Close HMV1 and HMV3.
3. Set the knobs on the excess flow valves HEXFV1 and HEXFV2to the reset or purge position. To do this, the black knob on the excess flow valve should be turned counterclockwise (when facing the body of the valve) to the stop.
4. Open the tank valves for both hydrogen cylinders.
5. Set the pressure on the regulator for the backup cylinder to 140 psig.
6. Set the pressure on the regulator for the 'in use' cylinder to 160 psig.
7. Open HMV1 and HMV3.
8. Set the excess flow valve for each cylinder to the RUN position. Do this by turning the knob clockwise (as viewed facing the valve) until it is tight.
9. Proceed with the purging *of* the hydrogen gas manifold as described below.

Purging the hydrogen gas manifold

If there is air in the feed lines from the hydrogen cylinders to the MOCVD cabinet or in the purifiers HP1 or HP2, it will be necessary to purge this with hydrogen before HP3 is turned on. This should be done as follows:

1. HP3 should be turned off and the purifier cell should be cold.
2. Adjust the setting on the temperature controller for the hydrogen purifier cell to 0° C. Make sure the purifier is plugged in. Press the red RESET button and flip the power switch to the on position. Adjust HMV5to give maximum flow.
3. Close HMV3. Open the cylinder valve on hydrogen cylinder #1 (the one connected to HEXFV2). Gas should flow from cylinder #1 into the inlet of HP3 and out the bleed port of HP3. Check to see that gas flow is indicated on the rotameter on the front panel of HP3. Allow the hydrogen to flow for 4 minutes.
4. Close HMV1. Open the cylinder valve on hydrogen cylinder #2. Open HMV3. Check for gas flow on the rotameter on the front panel of HP3. Allow the components to purge with hydrogen for 10 minutes.
5. Open HMV1. Turn the power switch on HP3 to the off position. This will close HPV14 and HPV16 and open NPV14 and HPV15. Nitrogen will begin to flow into HP3. Set the float on the front panel rotameter for a flow of 200 sccm and let the gas flow for 10 minutes. This will fill the purifier cell inlet with nitrogen. Reset the nitrogen flow to about 5 sccm if the purifier is to be left turned off.

Nitrogen purification system

Nitrogen gas for purging of the carrier gas system, manifold diluent, hydrogen purifier purging, and flushing of the deposition chamber and the sample entry chambers undergoes a two step purification process. It first passes through a desiccating element (NP1) to remove water vapor and then passes through a Nanochem purification cell, which removes oxygen. The Nanochem cartridge contains a lithium resin which is oxidized by the residual oxygen. The resin can become overheated and damaged if air passes through the cell so it is very important for the gas lines up to the Nanochem cell to be thoroughly flushed before its valves are opened. The cartridges are quite expensive so it is important not to use more of the purified nitrogen than necessary. The nitrogen supply to the vacuum pump inlet, the pump ballast port, and the vacuum eductors on the silane, arsine, phosphine, and trimethylzinc purge manifolds is not purified.

Purging the nitrogen purification system

1. Valves NMV6 and NMV7 should be closed. NMV8 should be open (lever should be pointing straight up).
2. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure *of* 20 psig, and that NMV1 is open.
3. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. *If* not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve.
4. On the HYDROGEN/NITROGEN control panel turn the MANIFOLD N_2 switch to the on position and adjust the FLOW SET 1 potentiometer to give a flow of 1000 sccm. Let the gas flow for 5 minutes.
5. Close the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel. Close the VAC EXHAUST switch on the HYDROGEN/NITROGEN control panel.
6. Open valves NMV6 and NMV7. Close valve NMV8 (lever should be horizontal)
7. Shut down the system as appropriate.

Nitrogen cylinder replacement

Since a continuous nitrogen supply is essential to many of the safety features and the presence of nitrogen pressure is sensed by the interlock system, the nitrogen cylinder should only be changed when the MOVCD reactor is in an idle state. The nitrogen can be changed in either of two manners. The first requires two people and does not require purging of the nitrogen purifier. The second requires only one person but the nitrogen lines should be purged afterward.

Nitrogen cylinder replacement without purge

1. Have the replacement cylinder ready for installation with the cap removed from the cylinder.
2. Close the manual valve, NMV1, on the outlet of the regulator to prevent the deposition system from pulling air through the regulator when it is removed from the cylinder.
3. Remove the regulator from the cylinder. Have one person hold the regulator while the second person rolls the old cylinder out of the way and rolls the new cylinder into position.
4. Reconnect the regulator to the new cylinder.
5. Attach the cap to the old cylinder and make sure it is chained to the wall until it is picked up by the vendor.
6. Open NMV1. Since a very small volume of air is introduced into the plumbing using this method it is not necessary to purge the nitrogen lines before continuing.

Nitrogen cylinder replacement with purge

1. Turn off the vacuum pump and bring the inlet of the pump up to atmospheric pressure.
2. Close NMV6 and NMV7, NMV1 and the valve on the nitrogen tank. Open NMV8.
3. Disconnect the VCR fitting below NMV1 and remove the regulator from the cylinder.
4. Replace the cap on the cylinder and make sure it is chained to the wall until it is picked up by the vendor.
5. Install the new cylinder in position, attach the regulator and reconnect the VCR fitting next to NMV1. The self-centering gasket in the VCR fitting does not need to be replaced every time the tank is changed as long as it is in good condition. However, it probably should not be reused more than four or five times.
6. Open the valve on the nitrogen tank and NMV1. Restart the vacuum pump.
7. Purge the nitrogen purification system.

Trimethylaluminum (TMA)

Gas Distribution

1. Trimethylaluminum is a liquid at room temperature with a vapor pressure of about 9 Torr. This means that the pressure of the vapor is 9/760 x 14.7 psia; very much less than atmospheric pressure. The vapor is transported from the stainless steel canister which contains the liquid, into the reaction chamber by bubbling hydrogen gas through the liquid.
2. The pressure of the hydrogen gas inside the liquid container is determined by the pressure setting of PR2. This is normally set at 10 psig.
3. The precise vapor pressure of the TMA is determined by the temperature of the liquid. This is controlled by immersing the liquid in a coolant bath which is controlled at a precise temperature by a small Brinkmann refrigeration unit.
4. Pressure drop to the reaction chamber occurs across the mass flow controller, ALMFC1.
5. The alkyl is exposed to the heated sensor element of the mass flow controller (110°C) thus it must be stable at this temperature.
6. Loss of liquid from the bubbler into the gas lines is unlikely because pressure in the gas line is the same as the bubbler. (Closure of the bypass valve, ALPV2, is delayed to allow bubbler inlet and outlet pressure to equilibrate.)
7. Upon power failure or other abort conditions, contamination of the upstream H_2lines is prevented by closure of ALPV12.
8. Adiabatic cooling could possibly cause alkyl condensation in the mass flow controller valve. It is advisable to keep the alkyl temperature below room temperature.
9. A purge mode overrides some interlocks to allow evacuation and backfill of the bubbler lines

Hazards

1. TMA is pyrophoric.
2. Aluminum oxide (from burning TMA) is highly toxic.
3. TMA reacts violently with water which should never be used to extinguish the burning alkyl. Use a dry chemical fire extinguisher only or else let it burn.

Precautions

1. Keep the manual valves on a newly installed bubbler closed until it is connected to the system, leak checked and purged.
2. Avoid gas pressure differentials that can force the alkyl from the bubbler back into the carrier gas piping.

Normal Operation

Start-of-day:

1. Since the pneumatic valves ALPV1 and ALPV2 seal off the bubbler from the rest of the system, the manual valves on the bubbler inlet and outlet can be left open except when the bubbler is being replaced or removed. At the start-of-day the TMA is therefore ready to operate from the control panel.
2. During a normal growth sequence the switches on the front panel are turned on in a left to right sequence and turned off in a right to left sequence. With all switches closed, ALMFC1 is commanded closed, ALPV1, ALPV2, ALPV3, and ALPV4 are closed and ALPV5 is in the vent position. When the ACTIVE switch is turned on, the

signal to command closure of ALMFC1 is removed, ALPV2, ALPV4-and ALPV12 open and the green ACTIVE light comes on. At this point hydrogen carrier gas will flow through the bypass valve ALPV2 and into the vent manifold at a rate determined by the current value of the voltage input to ALMFC1 as determined either by the front panel potentiometer or the D/A converter output for this flow controller. The red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve ALPV2 is open. When the BUBBLER IN switch is turned on, ALPV1 and ALPV3 open immediately and the green light BUBBLER IN turns on. After a two-second delay the red PURGE BUBBLER light goes out and ALPV2 closes. This insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when ALPV1 and ALPV3 open. When ALPV2 closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve. When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. During manual operation from the front panel, the flow through ALMFC1 is controlled by the setting of the 10-turn DVM SELECT potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black DVM SELECT button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to ALMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the DVM SELECT button is pushed in, the meter will read the input voltage to the flow controller from the *D/A* converter. Shutdown of the system should proceed by reversing the sequence of the above steps.

3. The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through ALMFC1.

End of day:

The alkyl vapors will accelerate the deterioration of some of the elastomers from which the o-rings are fabricated. Elastomer seals have been eliminated whenever possible in the construction of this system but current technology does not allow them to be completely eliminated. They are present in the mass flow controller and in the vent/run valve. For this reason it is good practice to thoroughly flush the alkyl vapor from ALMFC1 and ALPV5 before the circuit is shut down. This can be accomplished by allowing hydrogen to flow through the system for approximately 5 minutes after the BUBBLER IN switch is closed. After this, the ACTIVE switch can be closed, sealing off the circuit.

Interconnections

1. ACTIVE controls ALSV1 which drives ALPV4 and ALPV12.
2. BUBBLER IN controls ALSV2 which drives ALPV1 and ALPV3.
3. Relays ALK1 and ALK2 control ALSV3 which drives ALPV2.

Interlocks

1. All source valves are deactivated if the following conditions exist:
    1. System is in abort status.
    2. The vacuum exhaust valve, EXPV2, is closed.
    3. The deposition chamber is up to atmosphere.
    4. The manifold diluent gas is in the off position.
    5. ALPV1 and ALPV3 cannot be opened unless ALPV4 and ALPV12 are open.

2. With the PURGE switch on the front panel in the locked position (key removed), ALPV2 cannot be opened if ALPV1 and ALPV3 are open.
3. The bypass valve (ALPV2) is open if the BUBBLER IN switch is closed (handle down) and the ACTIVE switch is on (handle up).
4. The ACTIVE, BUBBLER IN, and RUN switches on the front panel are disabled when the computer switches the circuit into the REMOTE mode.
5. When the bubbler is connected to the circuit by opening the BUBBLER IN switch (handle up), valves ALPV1 and ALPV3 open immediately. Valve ALPV2 closes after a 2 second time delay to insure that the pressure on each side of the bubbler is equalized.
6. The manifold diluent gas cannot be turned off if the ACTIVE switch is on.

Programming commands for remote operation

LabView programming/remote operations info goes here

Removing and replacing source bubbler

Purging of the bubbler lines must be done through the outlet route. The output can be evacuated with the vacuum pump through valves ALPV4 and ALPV5. The limited conductance of the mass flow controller ALMFC1 requires that times of ≈1 minute be used to evacuate the lines to the bubbler. Purging is accomplished by alternately evacuating the lines and back filling them with nitrogen. During the evacuation cycle it is necessary to open ALPV4 and keep ALPV12 closed. Since the switching circuitry does not permit this, it is necessary to temporarily install a solenoid valve in the air line controlling ALPV12. The configuration of the solenoid valve keeps ALPV12 closed if the switch controlling the solenoid valve is closed. ALPV12 will be open only if both the switch controlling the solenoid valve is open and the ACTIVE switch on the ALUMINUM control panel is on.

Bubbler removal

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Connect the solenoid valve from the BUBBLER PURGE kit to the pneumatic control inlet of ALPV12. The connector on the 'A' port of the solenoid valve should be attached to the pneumatic inlet of ALPV12. The connector on the 'P' port of the solenoid valve should be attached to the 1/8" OD nylon tubing leading to ALPV12. Insure that the switch on the control box for the BUBBLER PURGE kit is closed and plug its line cord into a 120 VAC outlet.
3. Close the manual valves on the inlet and outlet of the TMA bubbler.
4. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
5. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the N_2 CARRIER position.
6. <u>Evacuation</u>: On the ALUMINUM control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for ALMFC1 to a flow of 100 sccm. As the gas in the line is evacuated, the flow rate will drop.

7. Backfill: When the flow rate for ALMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated. Turn the switch for the BUBBLER PURGE kit to the ON position for 10 seconds and then turn it off. This will allow the bubbler circuit to fill with nitrogen.
8. Repeat steps 6 and 7 twenty times to completely purge the circuit of alkyl vapor. Finish with a backfill step.
9. Turn the BUBBLER IN switch and the ACTIVE switch to the off position.
10. Disconnect and remove the bubbler. CAUTION: This is a potentially hazardous step if the manual bubbler valves have failed to seal. The pyrophoric liquid will ignite when exposed to air. The quantities are usually small enough to easily contain the burning alkyl and the oxidation is limited by the diffusion rate of oxygen into the tubing, which is rather slow. Avoid breathing the oxide fumes. Have a dry chemical or non-flammable dry absorbent medium available to smother the chemical fire should it be necessary. In most cases probably the best remedy is to attach the stainless steel VCR caps to seal the inlet and outlet of the bubbler. DO NOT USE WATER!!
11. Install the stainless steel VCR caps on the bubbler ports. Use new gaskets unless the gaskets are of the self centering type.
12. If a new bubbler is being installed immediately, proceed to the next section. Otherwise remove the bubbler purge kit, reconnect the pneumatic control line to ALPV12 and shut down the system as appropriate.

Bubbler installation

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Insure that one of the hydrogen cylinders is actively connected to the hydrogen supply line, that the pressure regulator is properly set and that the hydrogen purifier is turned on and up to temperature.
3. Insure that PR2 is at its normal setting of 10 psig.
4. Connect the solenoid valve from the BUBBLER PURGE kit to the pneumatic control inlet of ALPV12. The connector of the 'A' port of the solenoid valve should be attached to the pneumatic inlet of ALPV12. The connector on the 'P' port of the solenoid valve should be attached to the 1/8" OD nylon tubing leading to ALPV12. Insure that the switch on the control box for the BUBBLER PURGE kit is closed and plug its line cord into a 120 VAC outlet.
5. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
6. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the H_2 CARRIER position.
7. Insure that the manual valves on the bubbler are closed. Uncap the inlet and outlet ports on the bubbler and connect the bubbler to the circuit using new gaskets if the previously used gaskets were not of the self-centering type.
8. Evacuation: On the ALUMINUM control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for ALMFC1 to a flow of 100 sccm. As the gas in the line is evacuated, the flow rate will drop.
9. Backfill: When the flow rate for ALMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated. Turn the switch for the BUBBLER PURGE kit to the ON position for 10 seconds and then turn it off. This will allow the bubbler circuit to fill with hydrogen.

10. Repeat steps 6 and 7 twenty times to completely purge the circuit of air. Finish with a backfill step.
11. Open the manual valve on the outlet of the bubbler first. If the pressure in the bubbler should happen to be greater than 10 psig (the setting of PR2) and the inlet valve were opened first, the liquid alkyl would be forced back into the inlet plumbing.
12. Open the manual valve on the inlet of the bubbler.
13. Turn the BUBBLER IN switch and the ACTIVE switch to the off position. Turn the PURGE key lock switch off (counter-clockwise) and remove the key.
14. Remove the BUBBLER PURGE kit and reconnect the 1/8" OD nylon supply line to ALPV12.
15. Shut down the system as appropriate.

Trimethylindium (TMln)

Gas Distribution

1. Trimethylindium is a solid at room temperature with a vapor pressure of about 2.6 Torr. This means that the pressure of the vapor is 2.6/760 x 14.7 psia; very much less than atmospheric pressure. The vapor is transported from the stainless steel canister which contains the liquid, into the reaction chamber by bubbling hydrogen gas through the liquid.
2. The pressure of the hydrogen gas inside the liquid container is determined by the pressure setting of PR2. This is normally set at 10 psig.
3. The precise vapor pressure of the TMln is determined by the temperature of the liquid. This is controlled by immersing the liquid in a coolant bath which is controlled at a precise temperature by a small Brinkmann refrigeration unit.
4. Pressure drop to the reaction chamber occurs across the mass flow controller, INMFC1.
5. The alkyl is exposed to the heated sensor element of the mass flow controller (110° C); thus it must be stable at this temperature.
6. Loss of liquid from the bubbler into the gas lines is unlikely because pressure in the gas line is the same as the bubbler (closure of the bypass valve. INPV2, is delayed to allow bubbler inlet and outlet pressure to equilibrate).
7. Upon power failure or other abort conditions, contamination of the upstream H_2 lines is prevented by closure of INPV12.
8. Adiabatic cooling could possibly cause alkyl condensation in the mass flow controller valve. It is advisable to keep the alkyl temperature below room temperature.
9. A purge mode overrides some interlocks to allow evacuation and backfill of the bubbler lines.

Hazards

1. TMln is pyrophoric.
2. Indium oxide (from burning EDMln) is an irritant.
3. TMln reacts violently with water which should never be used to extinguish the burning alkyl. Use a dry chemical fire extinguisher only or else let it burn.

Precautions

1. Keep the manual valves on a newly installed bubbler closed until it is connected to the system, leak checked and purged.
2. Avoid gas pressure differentials that can force the alkyl from the bubbler back into the carrier gas piping.

Normal Operation

1. Start-of-day: Since the pneumatic valves INPV1 and INPV2 seal off the bubbler from the rest of the system, the manual valves on the bubbler inlet and outlet can be left open except when the bubbler is being replaced or removed. At the start-of-day the EDMln is therefore ready to operate from the control panel.
2. During a normal growth sequence the switches on the front panel are turned on in a left to right sequence and turned off in a right to left sequence. With all switches closed, INMFC1 is commanded closed, INPV1, INPV2, INPV3, and INPV4 are closed and INPV5 is in the vent position. When the ACTIVE switch is turned on, the signal to command closure of INMFC1 is removed, INPV2, INPV4 and INPV12 open and the green ACTIVE light comes

on. At this point hydrogen carrier gas will flow through the bypass valve INPV2 and into the vent manifold at a rate determined by the current value of the voltage input to INMFC1 as determined either by the front panel potentiometer or the D/A converter output for this flow controller. The red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve INPV2 is open. When the BUBBLER IN switch is turned on, INPV1 and INPV3 open immediately and the green light BUBBLER IN turns on. After a two-second delay the red PURGE BUBBLER light goes out and INPV2 closes. This insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when INPV1 and INPV3 open. When INPV2 closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve. When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. During manual operation from the front panel, the flow through INMFC1 is controlled by the setting of the 10-turn DVM SELECT potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black DVM SELECT button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to INMFC1 is supplied by a *DIA* converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the DVM SELECT button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. Shutdown of the system should proceed by reversing the sequence of the above steps.

3. The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through INMFC1.
4. <u>End of day</u>: The alkyl vapors will accelerate the deterioration of some of the elastomers from which the o-rings are fabricated. Elastomer seals have been eliminated whenever possible in the construction of this system but current technology does not allow them to be completely eliminated. They are present in the mass flow controller and in the vent/run valve. For this reason it is good practice to thoroughly flush the alkyl vapor from INMFC1 and INPV5 before the circuit is shut down. This can be accomplished by allowing hydrogen to flow through the system for approximately 5 minutes after the BUBBLER IN switch is closed. After this, the ACTIVE switch can be closed, sealing off the circuit.

Interconnections

1. ACTIVE controls INSV1 which drives INPV4 and INPV12.
2. BUBBLER IN controls INSV2 which drives INPV1 and INPV3.
3. Relays INK1 and INK2 control INSV3 which drives INPV2.

Interlocks

1. All source valves are deactivated if the following conditions exist:
    1. System is in abort status.
    2. The vacuum exhaust valve, EXPV2, is closed.
    3. The deposition chamber is up to atmosphere.
    4. The manifold diluent gas is in the off position.
2. INPV1 and INPV3 cannot be opened unless INPV4 and INPV12 are open.
3. With the PURGE switch on the front panel in the locked position (key removed), INPV2 cannot be opened if INPV1 and INPV3 are open.

4. The bypass valve (INPV2) is open if the BUBBLER IN switch is closed (handle down) and the ACTIVE switch is on (handle up).
5. The ACTIVE, BUBBLER IN and RUN switches on the front panel are disabled when the computer switches the circuit into the REMOTE mode.
6. When the bubbler is connected to the circuit by opening the BUBBLER IN switch (handle up), valves INPV1 and INPV3 open immediately. Valve INPV2 closes after a 2 second time delay to insure that the pressure on each side of the bubbler is equalized.
7. The manifold diluent gas cannot be turned off if the ACTIVE switch is on.

Programming commands for remote operation

INSERT LABVIEW DOCUMENTATION HERE

Removing and replacing source bubbler

Purging of the bubbler lines must be done through the outlet route. The output can be evacuated with the vacuum pump through valves INPV4 and INPV5. The limited conductance of the mass flow controller INMFC1 requires that times of ≈1 minute be used to evacuate the lines to the bubbler. Purging is accomplished by alternately evacuating the lines and back filling them with hydrogen. During the evacuation cycle it is necessary to open INPV4 and keep INPV12 closed. Since the switching circuitry does not permit this, it is necessary to temporarily install a solenoid valve in the air line controlling INPV12. The configuration of the solenoid valve keeps INPV12 closed if the switch controlling the solenoid valve is closed. INPV12 will be open only if both the switch controlling the solenoid valve is open and the ACTIVE switch on the INDIUM control panel is on.

Bubbler removal

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Connect the solenoid valve from the BUBBLER PURGE kit to the pneumatic control inlet of INPV12. The connector on the 'A' port of the solenoid valve should be attached to the pneumatic inlet of INPV12. The connector on the 'P' port of the solenoid valve should be attached to the 1/8" OD nylon tubing leading to INPV12. Insure that the switch on the control box for the BUBBLER PURGE kit is closed and plug its line cord into a 120VAC outlet.
3. Close the manual valves on the inlet and outlet of the EDMln bubbler.
4. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
5. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the N_2 CARRIER position.
6. <u>Evacuation</u>: On the INDIUM control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for INMFC1 to a flow of 100 sccm. As the gas in the line is evacuated, the flow rate will drop.

7. Backfill: When the flow rate for INMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated. Turn the switch for the BUBBLER PURGE kit to the ON position for 10 seconds and then turn it off. This will allow the bubbler circuit to fill with nitrogen.
8. Repeat steps 6 and 7 twenty times to completely purge the circuit of alkyl vapor. Finish with a backfill step.
9. Turn the BUBBLER IN switch and the ACTIVE switch to the off position.
10. Disconnect and remove the bubbler. CAUTION: This is a potentially hazardous step if the manual bubbler valves have failed to seal. The pyrophoric liquid will ignite when exposed to air. The quantities are usually small enough to easily contain the burning alkyl and the oxidation is limited by the diffusion rate of oxygen into the tubing, which is rather slow. Avoid breathing the oxide fumes. Have a dry chemical or non-flammable dry absorbent medium available to smother the chemical fire should it be necessary. In most cases probably the best remedy is to attach the stainless steel VCR caps to seal the inlet and outlet of the bubbler. DO NOT USE WATER!!
11. Install the stainless steel VCR caps on the bubbler ports. Use new gaskets unless the gaskets are of the self centering type.
12. If a new bubbler is being installed immediately, proceed to the next section. Otherwise remove the bubbler purge kit, reconnect the pneumatic control line to INPV12 and shut down the system as appropriate.

Bubbler installation

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Insure that one of the hydrogen cylinders is actively connected to the hydrogen supply line, that the pressure regulator is properly set and that the hydrogen purifier is turned on and up to temperature.
3. Insure that PR2 is at its normal setting of 10 psig.
4. Connect the solenoid valve from the BUBBLER PURGE kit to the pneumatic control inlet of INPV12. The connector of the 'A' port of the solenoid valve should be attached to the pneumatic inlet of INPV12. The connector on the 'P' port of the solenoid valve should be attached to the 1/8" OD nylon tubing leading to INPV12. Insure that the switch on the control box for the BUBBLER PURGE kit is closed and plug its line cord into a 120VAC outlet.
5. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
6. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the H_2 CARRIER position.
7. Insure that the manual valves on the bubbler are closed. Uncap the inlet and outlet ports on the bubbler and connect the bubbler to the circuit using new gaskets if the previously used gaskets were not of the self-centering type.
8. Evacuation: On the INDIUM control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for INMFC1 to a flow of 100 sccm. As the gas in the line is evacuated, the flow rate will drop.
9. Backfill: When the flow rate for INMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated. Turn the switch for the BUBBLER PURGE kit to the ON position for 10 seconds and then turn it off. This will allow the bubbler circuit to fill with hydrogen.

10. Repeat steps 6 and 7 twenty times to completely purge the circuit of air. Finish with a backfill step.
11. Open the manual valve on the outlet of the bubbler first. If the pressure in the bubbler should happen to be greater than 10 psig (the setting of PR2) and the inlet valve were opened first, the liquid alkyl would be forced back into the inlet plumbing.
12. Open the manual valve on the inlet of the bubbler.
13. Turn the BUBBLER IN switch and the ACTIVE switch to the off position. Turn the PURGE key lock switch off (counter-clockwise) and remove the key.
14. Remove the BUBBLER PURGE kit and reconnect the 1/8" OD nylon supply line to INPV12.
15. Shut down the system as appropriate.

Tertiarybutylarsine (TBAs)

Gas Distribution

1. Tertiarybutylarsine is a liquid at room temperature with a vapor pressure of 81 Torr at 10° C. This means that the pressure of the vapor is 81/760 x 14.7 psia; slightly more than 1/10 of atmospheric pressure. The vapor is transported from the stainless steel canister which contains the liquid, into the reaction chamber by bubbling hydrogen gas through the liquid.
2. The pressure of the hydrogen gas inside the liquid container is determined by the pressure setting of PR5. This is normally set at 10 psig.
3. The precise vapor pressure of the TBAs is determined by the temperature of the liquid. This is controlled by immersing the liquid in a coolant bath which is controlled at a precise temperature by a small Brinkmann refrigeration unit.
4. Pressure drop to the reaction chamber occurs across the mass flow controller, ASLMFC1.
5. The alkyl is exposed to the heated sensor element of the mass flow controller (110°C); it must be stable at this temperature.
6. Loss of liquid from the bubbler into the gas lines is unlikely because pressure in the gas line is the same as the bubbler. (Closure of the bypass valve, ASLPV2, is delayed to allow bubbler inlet and outlet pressure to equilibrate.)
7. Upon power failure or other abort conditions, contamination of the upstream H_2 lines is prevented by closure of ASLPV12.
8. Adiabatic cooling could possibly cause alkyl condensation in the mass flow controller valve. It is advisable to keep the alkyl temperature below room temperature.
9. A purge mode overrides some interlocks to allow evacuation and backfill of the bubbler lines

Hazards

1. TBAs is a colorless oil that is pyrophoric. In case of skin contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes.
2. Combustion may produce carbon monoxide and arsenic oxides.
3. The vapor has a very unpleasant odor suggesting rotten onions. The limits of olfactory detection are unknown. The OSHA TWA Ceiling for the vapor is 0.5 mg/m^3 (0.1 ppm). The DOD ChemLogic CL8 toxic gas monitor which is set to give an alarm at the lower TWA Ceiling of 0.05 ppm for arsine will detect TBAs at the above ceiling. In the event of a TBAs spill, all personnel not equipped with respirators should immediately leave the vicinity of the spill.
4. The most effective fire extinguishing agent is a dry chemical powder pressurized with nitrogen. Sand, absorbent clay or CO2 may be used. If the fire cannot be controlled with an extinguishing agent, keep a safe distance and allow the material to burn until extinguished.

Precautions

1. Keep the manual valves on a newly installed bubbler closed until it is connected to the system, leak checked and purged.
2. Avoid gas pressure differentials that can force the alkyl from the bubbler back into the carrier gas piping.

Normal Operation

1. Start-of-day: Since the pneumatic valves ASLPV1 and ASLPV2 seal off the bubbler from the rest of the system, the manual valves on the bubbler inlet and outlet can be left open except when the bubbler is being replaced or removed. At the start-of-day the TBAs is therefore ready to operate from the control panel.
2. During a normal growth sequence the switches on the front panel are turned on in a left to right sequence and turned off in a right to left sequence. With all switches closed, ASLMFC1 is commanded closed, ASLPV1, ASLPV2, ASLPV3, and ASLPV4 are closed and ASLPV5 is in the vent position. When the ACTIVE switch is turned on, the signal to command closure of ASLMFC1 is removed, ASLPV2, ASLPV4 and ASLPV12 open and the green ACTIVE light comes on. At this point hydrogen carrier gas will flow through the bypass valve ASLPV2 and into the vent manifold at a rate determined by the current value of the voltage input to ASLMFC1 as determined either by the front panel potentiometer or the *D/A* converter output for this flow controller. The red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve ASLPV2 is open. When the BUBBLER IN switch is turned on, ASLPV1 and ASLPV3 open immediately and the green light BUBBLER IN turns on. After a two-second delay the red PURGE BUBBLER light goes out and ASLPV2 closes. This insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when ASLPV1 and ASLPV3 open. When ASLPV2 closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve. When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. During manual operation from the front panel, the flow through ASLMFC1 is controlled by the setting of the 10-turn DVM SELECT potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black DVMSELECT button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to ASLMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the DVM SELECT button is pushed in, the meter will read the input voltage to the flow controller from the *D/A* converter. Shutdown of the system should proceed by reversing the sequence of the above steps.
3. The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through ASLMFC1.
4. End of day: The alkyl vapors will accelerate the deterioration of some of the elastomers from which the o-rings are fabricated. Elastomer seals have been eliminated whenever possible in the construction of this system but current technology does not allow them to be completely eliminated. They are present in the mass flow controller and in the vent/run valve. For this reason it is good practice to thoroughly flush the alkyl vapor from ASLMFC1 and ASLPV5 before the circuit is shut down. This can be accomplished by allowing hydrogen to flow through the system for approximately 5 minutes after the BUBBLER IN switch is closed. After this, the ACTIVE switch can be closed, sealing off the circuit.

Interconnections

1. ACTIVE controls ASLSV1 which drives ASLPV4 and ASLPV12.
2. BUBBLER IN controls ASLSV2 which drives ASLPV1 and ASLPV3.
3. Relays ASLK1 and ASLK2 control ASLSV3 which drives ASLPV2.

Interlocks

1. All source valves are deactivated if the following conditions exist:
2. System is in abort status.
3. The vacuum exhaust valve, EXPV2, is closed.
4. The deposition chamber is up to atmosphere.
5. The manifold diluent gas is in the off position.
6. ASLPV1 and ASLPV3 cannot be opened unless ASLPV4 and ASLPV12 are open.
7. With the PURGE switch on the front panel in the locked position (key removed), ASLPV2 cannot be opened if ASLPV1 and ASLPV3 are open.
8. The bypass valve (ASLPV2) is open if the BUBBLER IN switch is closed (handle down) and the ACTIVE switch is on (handle up).
9. The ACTIVE, BUBBLER IN, and RUN switches on the front panel are disabled when the computer switches the circuit into the REMOTE mode.
10. When the bubbler is connected to the circuit by opening the BUBBLER IN switch (handle up), valves ASLPV1 and ASLPV3 open immediately. Valve ASLPV2 closes after a 2 second time delay to insure that the pressure on each side of the bubbler is equalized.
11. The manifold diluent gas cannot be turned off if the ACTIVE switch is on.

Programming commands for remote operation

INSERT LABVIEW DOCUMENTATION HERE

Removing and replacing source bubbler

Purging of the bubbler lines must be done through the outlet route. The output can be evacuated with the vacuum pump through valves ASLPV4 and ASLPV5. The limited conductance of the mass flow controller ASLMFC1 requires that times of ",1 minute be used to evacuate the lines to the bubbler. Purging is accomplished by alternately evacuating the lines and backfilling them with hydrogen. During the evacuation cycle it is necessary to open ASLPV4 and keep ASLPV12 closed. Since the switching circuitry does not permit this, it is necessary to temporarily install a solenoid valve in the air line controlling ASLPV12. The configuration of the solenoid valve keeps ASLPV12 closed if the switch controlling the solenoid valve is closed. ASLPV12 will be open only if both the switch controlling the solenoid valve is open and the ACTIVE switch on the ARSENIC LIQUID control panel is on.

Bubbler removal

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Connect the solenoid valve from the BUBBLER PURGE kit to the pneumatic control inlet of ASLPV12. The connector on the 'A' port of the solenoid valve should be attached to the pneumatic inlet of ASLPV12. The connector on the 'P' port of the solenoid valve should be attached to the 1/8" OD nylon tubing leading to ASLPV12. Insure that the switch on the control box for the BUBBLER PURGE kit is closed and plug its line cord into a 120 VAC outlet.
3. Close the manual valves on the inlet and outlet of the TBAs bubbler.
4. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to

maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.

5. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the N_2 CARRIER position.
6. Evacuation: On the ARSENIC LIQUID control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for ASLMFC1 to a flow of 100 sccm. As the gas in the line is evacuated, the flow rate will drop.
7. Backfill: When the flow rate for ASLMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated. Turn the switch for the BUBBLER PURGE kit to the ON position for 10 seconds and then turn it off. This will allow the bubbler circuit to fill with nitrogen.
8. Repeat steps 6 and 7 twenty times to completely purge the circuit of alkyl vapor. Finish with a backfill step.
9. Turn the BUBBLER IN switch and the ACTIVE switch to the off position.
10. Disconnect and remove the bubbler. CAUTION: This is a potentially hazardous step if the manual bubbler valves have failed to seal. The pyrophoric liquid will ignite when exposed to air. The quantities are usually small enough to easily contain the burning alkyl and the oxidation is limited by the diffusion rate of oxygen into the tubing, which is rather slow. Avoid breathing the oxide fumes. Have a dry chemical or non-flammable dry absorbent medium available to smother the chemical fire should it be necessary. In most cases probably the best remedy is to attach the stainless steel VCR caps to seal the inlet and outlet of the bubbler. DO NOT USE WATER!!
11. Install the stainless steel VCR caps on the bubbler ports. Use new gaskets unless the gaskets are of the self centering type.
12. If a new bubbler is being installed immediately, proceed to the next section. Otherwise remove the bubbler purge kit, reconnect the pneumatic control line to ASLPV12and shut down the system as appropriate.

Bubbler installation

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Insure that one of the hydrogen cylinders is actively connected to the hydrogen supply line, that the pressure regulator is properly set and that the hydrogen purifier is turned on and up to temperature.
3. Insure that PR5 is at its normal setting of 10 psig.
4. Connect the solenoid valve from the BUBBLER PURGE kit to the pneumatic control inlet of ASLPV12. The connector of the 'A' port of the solenoid valve should be attached to the pneumatic inlet of ASLPV12. The connector on the 'P' port of the solenoid valve should be attached to the 1/8" OD nylon tubing leading to ASLPV12. Insure that the switch on the control box for the BUBBLER PURGE kit is closed and plug its line cord into a 120 VAC outlet.
5. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
6. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the H_2 CARRIER position.
7. Insure that the manual valves on the bubbler are closed. Uncap the inlet and outlet ports on the bubbler and connect the bubbler to the circuit using new gaskets if the previously used gaskets were not of the self-centering type.

8. Evacuation: On the ARSENIC LIQUID control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for ASLMFC1 to a flow of 100 sccm. As the gas in the line is evacuated, the flow rate will drop.
9. Backfill: When the flow rate for ASLMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated. Turn the switch for the BUBBLER PURGE kit to the ON position for 10 seconds and then turn it off. This will allow the bubbler circuit to fill with hydrogen.
10. Repeat steps 6 and 7 twenty times to completely purge the circuit of air. Finish with a backfill step.
11. Open the manual valve on the outlet of the bubbler first. If the pressure in the bubbler should happen to be greater than 10 psig (the setting of PR5) and the inlet valve were opened first, the liquid alkyl would be forced back into the inlet plumbing.
12. Open the manual valve on the inlet of the bubbler.
13. Turn the BUBBLER IN switch and the ACTIVE switch to the off position. Turn the PURGE key lock switch off (counter-clockwise) and remove the key.
14. Remove the BUBBLER PURGE kit and reconnect the 1/8" OD nylon supply line to ASLPV12.
15. Shut down the system as appropriate.

Tertiarybutylphosphine (TBP)

Gas Distribution

1. Tertiarybutylphosphine is a liquid at room temperature with a vapor pressure of 286 Torr at room temperature. This means that the pressure of the vapor is 286/760 x 14.7 psia; or about 1/3 atmospheric pressure. The vapor is transported from the stainless steel canister which contains the liquid, into the reaction chamber by bubbling hydrogen gas through the liquid.
2. The pressure of the hydrogen gas inside the liquid container is determined by the pressure setting of PR5. This is normally set at 10 psig.
3. The precise vapor pressure of the TBP is determined by the temperature of the liquid. This is controlled by immersing the liquid in a coolant bath which is controlled at a precise temperature by a small Brinkmann refrigeration unit.
4. Pressure drop to the reaction chamber occurs across the mass flow controller, PLMFC1.
5. The alkyl is exposed to the heated sensor element of the mass flow controller (110° C); thus it must be stable at this temperature.
6. Loss of liquid from the bubbler into the gas lines is unlikely because pressure in the gas line is the same as the bubbler. (Closure of the bypass valve, PLPV2, is delayed to allow bubbler inlet and outlet pressure to equilibrate.)
7. Upon power failure or other abort conditions, contamination of the upstream H_2 lines is prevented by closure of PLPV12.
8. Adiabatic cooling could possibly cause alkyl condensation in the mass flow controller valve. It is advisable to keep the alkyl temperature below room temperature.
9. A purge mode overrides some interlocks to allow evacuation and backfill of the bubbler lines

Hazards

1. TBP is a colorless oil that is pyrophoric. In case of skin contact, immediately flush skin with plenty of water for at least 1Sminutes while removing contaminated clothing and shoes.
2. Combustion may produce carbon monoxide, phosphine oxides, phosphinic acid, phosphonic acid, and P_20_5. The products formed by the thermal decomposition in an anaerobic atmosphere are elemental phosphorus, isobutane, isobutylene, and phosphine.
3. The vapor has a very unpleasant odor. The limits of olfactory detection are unknown. Animal studies have indicated that the toxicity is about 20 times less than for phosphine. The OSHA TWA (time-weighted average for an 8 hour shift) ceiling for phosphine is 0.3 ppm. The DOD ChemLogic CL8 toxic gas monitor which is set to give an alarm at the lower TWA ceiling of O.O5ppm for arsine will detect TBP at the above ceiling. In the event of a TBP spill, all personnel not equipped with respirators should immediately leave the vicinity of the spill.
4. The most effective fire extinguishing agent is a dry chemical powder pressurized with nitrogen. Sand, absorbent clay or CO2 may be used. If the fire cannot be controlled with an extinguishing agent, keep a safe distance and allow the material to burn until extinguished.

Precautions

1. Keep the manual valves on a newly installed bubbler closed until it is connected to the system, leak checked and purged.
2. Avoid gas pressure differentials that can force the alkyl from the bubbler back into the carrier gas piping.

Normal Operation

1. Start-of-day: Since the pneumatic valves PLPV1 and PLPV2 seal off the bubbler from the rest of the system, the manual valves on the bubbler inlet and outlet can be left open except when the bubbler is being replaced or removed. At the start-of-day the TBP is therefore ready to operate from the control panel.
2. During a normal growth sequence the switches on the front panel are turned on in a left to right sequence and turned off in a right to left sequence. With all switches closed, PLMFC1 is commanded closed, PLPV1, PLPV2, PLPV3, and PLPV4 are closed and PLPV5 is in the vent position. When the ACTIVE switch is turned on, the signal to command closure of PLMFC1 is removed, PLPV2, PLPV4 and PLPV12 open and the green ACTIVE light comes on. At this point hydrogen carrier gas will flow through the bypass valve PLPV2 and into the vent manifold at a rate determined by the current value of the voltage input to PLMFC1 as determined either by the front panel potentiometer or the D/A converter output for this flow controller. The red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve PLPV2 is open. When the BUBBLER IN switch is turned on, PLPV1and PLPV3 open immediately and the green light BUBBLER IN turns on. After a two-second delay the red PURGE BUBBLER light goes out and PLPV2 closes. This insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when PLPV1and PLPV3 open. When PLPV2closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve. When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. During manual operation from the front panel, the flow through PLMFC1 is controlled by the setting of the 10-turn DVM SELECT potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black DVM SELECT button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to PLMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the DVM SELECT button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. Shutdown of the system should proceed by reversing the sequence of the above steps.
3. The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through PLMFC1.
4. End of day: The alkyl vapors will accelerate the deterioration of some of the elastomers from which the o-rings are fabricated. Elastomer seals have been eliminated whenever possible in the construction of this system but current technology does not allow them to be completely eliminated. They are present in the mass flow controller and in the vent/run valve. For this reason it is good practice to thoroughly flush the alkyl vapor from PLMFC1 and PLPV5 before the circuit is shut down. This can be accomplished by allowing hydrogen to flow through the system for approximately 5 minutes after the BUBBLER IN switch is closed. After this, the ACTIVE switch can be closed, sealing off the circuit.

Interconnections

1. ACTIVE controls PLSV1 which drives PLPV4 and PLPV12.
2. BUBBLER IN controls PLSV2 that drives PLPV1and PLPV3.
3. Relays PLK1 and PLK2 control PLSV3 which drives PLPV2.

Interlocks

1. All source valves are deactivated if the following conditions exist:
    1. System is in abort status.
    2. The vacuum exhaust valve, EXPV2, is closed.
    3. The deposition chamber is up to atmosphere.
    4. The manifold diluent gas is in the off position.
2. PLPV1 and PLPV3 cannot be opened unless PLPV4 and PLPV12 are open.
3. With the PURGE switch on the front panel in the locked position (key removed), PLPV2 cannot be opened if PLPV1and PLPV3are open.
4. The bypass valve (PLPV2) is open if the BUBBLER IN switch is closed (handle down) and the ACTIVE switch is on (handle up).
5. The ACTIVE, BUBBLER IN, and RUN switches on the front panel are disabled when the computer switches the circuit into the REMOTE mode.
6. When the bubbler is connected to the circuit by opening the BUBBLER IN switch (handle up), valves PLPV1 and PLPV3 open immediately. Valve PLPV2closes after a 2 second time delay to insure that the pressure on each side of the bubbler is equalized.
7. The manifold diluent gas cannot be turned off if the ACTIVE switch is on.

Programming commands for remote operation

INSERT LABVIEW DOCUMENTAION HERE

Removing and replacing source bubbler

Purging of the bubbler lines must be done through the outlet route. The output can be evacuated with the vacuum pump through valves PLPV4 and PLPVS. The limited conductance of the mass flow controller PLMFC1 requires that times of ≈1minute be used to evacuate the lines to the bubbler. Purging is accomplished by alternately evacuating the lines and backfilling them with hydrogen. During the evacuation cycle it is necessary to open PLPV4 and keep PLPV12 closed. Since the switching circuitry does not permit this, it is necessary to temporarily install a solenoid valve in the air line controlling PLPV12. The configuration of the solenoid valve keeps PLPV12 closed if the switch controlling the solenoid valve is closed. PLPV12 will be open only if both the switch controlling the solenoid valve is open and the ACTIVE switch on the PHOSPHORUS LIQUID control panel is on.

Bubbler removal

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Connect the solenoid valve from the BUBBLER PURGE kit to the pneumatic control inlet of PLPV12. The connector on the 'A' port of the solenoid valve should be attached to the pneumatic inlet of PLPV12. The connector on the 'P' port of the solenoid valve should be attached to the 1/8" OD nylon tubing leading to PLPV12. Insure that the switch on the control box for the BUBBLER PURGE kit is closed and plug its line cord into a 120 VAC outlet.
3. Close the manual valves on the inlet and outlet of the TBP bubbler.
4. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on

the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.

5. On the HYDROGEN/NITROGEN panel set the N_2 CARRIERlH_2 CARRIER switch to the N_2 CARRIER position.
6. Evacuation: On the PHOSPHORUS LIQUID control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for PLMFC1to a flow of 100 sccm. As the gas in the line is evacuated, the flow rate will drop.
7. Backfill: When the flow rate for PLMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated. Turn the switch for the BUBBLER PURGE kit to the ON position for 10 seconds and then turn it off. This will allow the bubbler circuit to fill with nitrogen.
8. Repeat steps 6 and 7 twenty times to completely purge the circuit of alkyl vapor. Finish with a backfill step.
9. Turn the BUBBLER IN switch and the ACTIVE switch to the off position.
10. Disconnect and remove the bubbler. CAUTION: This is a potentially hazardous step if the manual bubbler valves have failed to seal. The pyrophoric liquid will ignite when exposed to air. The quantities are usually small enough to easily contain the burning alkyl and the oxidation is limited by the diffusion rate of oxygen into the tubing, which is rather slow. Avoid breathing the oxide fumes. Have a dry chemical or non-flammable dry absorbent medium available to smother the chemical fire should it be necessary. In most cases probably the best remedy is to attach the stainless steel VCR caps to seal the inlet and outlet of the bubbler. DO NOT USE WATER!!
11. Install the stainless steel VCR caps on the bubbler ports. Use new gaskets unless the gaskets are of the self centering type.
12. If a new bubbler is being installed immediately, proceed to the next section. Otherwise remove the bubbler purge kit, reconnect the pneumatic control line to PLPV12 and shut down the system as appropriate.

Bubbler installation

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Insure that one of the hydrogen cylinders is actively connected to the hydrogen supply line, that the pressure regulator is properly set and that the hydrogen purifier is turned on and up to temperature.
3. Insure that PR5 is at its normal setting of 10 psig.
4. Connect the solenoid valve from the BUBBLER PURGE kit to the pneumatic control inlet of PLPV12. The connector of the 'A' port of the solenoid valve should be attached to the pneumatic inlet of PLPV12. The connector on the 'P' port of the solenoid valve should be attached to the 1/8" OD nylon tubing leading to PLPV12. Insure that the switch on the control box for the BUBBLER PURGE kit is closed and plug its line cord into a 120VAC outlet.
5. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
6. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the H_2 CARRIER position.

7. Insure that the manual valves on the bubbler are closed. Uncap the inlet and outlet ports on the bubbler and connect the bubbler to the circuit using new gaskets if the previously used gaskets were not of the self-centering type.
8. Evacuation: On the PHOSPHORUS LIQUID control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for PLMFC1to a flow of 100 sccm. As the gas in the line is evacuated, the flow rate will drop.
9. Backfill: When the flow rate for PLMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated. Turn the switch for the BUBBLER PURGE kit to the ON position for 10 seconds and then turn it off. This will allow the bubbler circuit to fill with hydrogen.
10. Repeat steps 6 and 7 twenty times to completely purge the circuit of air. Finish with a backfill step.
11. Open the manual valve on the outlet of the bubbler first. If the pressure in the bubbler should happen to be greater than 10 psig (the setting of PR5) and the inlet valve were opened first, the liquid alkyl would be forced back into the inlet plumbing.
12. Open the manual valve on the inlet of the bubbler.
13. Turn the BUBBLER IN switch and the ACTIVE switch to the off position. Turn the PURGE key lock switch off (counter-clockwise) and remove the key.
14. Remove the BUBBLER PURGE kit and reconnect the 1/8" OD nylon supply line to PLPV12.
15. Shut down the system as appropriate.

Iron carbonyl (FE)

Gas Distribution

1. Iron carbonyl is a liquid at room temperature with a vapor pressure of about 178 Torr at room temperature. This means that the pressure of the vapor is 178/760 x 14.7 psia; about 1/5 atmospheric pressure. The vapor is transported from the stainless steel canister which contains the liquid, into the reaction chamber by bubbling hydrogen gas through the liquid.
2. The FE plumbing system is a more complex arrangement which allows the vapor stream passing through the bubbler to be diluted with additional hydrogen carrier gas. In addition, a portion of the total volume of gas may be dumped into the scrubber before it enters the vent/run valve. This arrangement permits a wider range of FE flow rates to be directed into the deposition chamber. The pressure of the hydrogen gas inside the liquid container is determined by the pressure setting of either PR1 or PR4 depending on the mode of operation. The proper functioning of the various components is discussed in detail in the section on operating procedures.
3. The precise vapor pressure of the FE is determined by the temperature of the liquid. This is controlled by immersing the liquid in a coolant bath which is controlled at a precise temperature by a small Brinkmann refrigeration unit.
4. Pressure drop to the reaction chamber occurs across the mass flow controller, FEMFC1.
5. The alkyl is exposed to the heated sensor element of the mass flow controller (=110° C); thus it must be stable at this temperature.
6. Loss of liquid from the bubbler into the gas lines is unlikely because pressure in the gas line is the same as the bubbler. (Closure of the bypass valve, FEPV2, is delayed to allow bubbler inlet and outlet pressure to equilibrate.)
7. Upon power failure or other abort conditions, contamination of the upstream H_2 lines is prevented by closure of the control valve for mass flow controller FEMFC2.
8. Adiabatic cooling could possibly cause alkyl condensation in the mass flow controller valve. It is advisable to keep the alkyl temperature below room temperature.
9. A purge mode overrides some interlocks to allow evacuation and backfill of the bubbler lines.

Hazards

1. FE is pyrophoric.
2. The oxides formed from the burning of FE are toxic.
3. FE reacts violently with water which should never be used to extinguish the burning alkyl. Use a dry chemical fire extinguisher only or else let it burn.

Precautions

1. Keep the manual valves on a newly installed bubbler closed until it is connected to the system, leak checked and purged.
2. Avoid gas pressure differentials that can force the alkyl from the bubbler back into the carrier gas piping.

Normal Operation

1. Start-of-day: Since the pneumatic valves FEPV1and FEPV2 seal off the bubbler from the rest of the system, the manual valves on the bubbler inlet and outlet can be left open except when the bubbler is being replaced or

removed. At the start-of-day the FE is therefore ready to operate from the control panel. The operation of the controls for the cases without hydrogen dilution and with hydrogen dilution will be discussed separately.

2. Normal growth sequence:

Operation without additional hydrogen dilution:

During a normal growth sequence the switches on the front panel are turned on in a left to right sequence and turned off in a right to left sequence. With all switches closed, FEMFC1, FEMFC2, and FEMFC3 are commanded closed, FEPV1, FEPV2, FEPV3, FEPV4, FEPV6, FEPV7, and FEPV12 are closed and FEPV5 is in the vent position. When the ACTIVE switch is turned on, the signals to command closure of FEMFC1 and FEMFC2 are removed, FEPV2, and FEPV4 open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control input of FEMFC2which programs its flow setting to 200 sccm. This insures that the flow setting through FEMFC2 will always be larger than the flow through FEMFC1 and the bubbler will not become evacuated; the control valve contained within FEMFC2 will remain wide open. With the switches in this configuration, the hydrogen carrier gas will flow through FEMFC2,through the bypass valve FEPV2, through FEMFC1, through FEPV4, through FEPV5 and into the vent manifold at a rate determined by the current value of the voltage input to FEMFC1 as determined either by the front panel potentiometer or the D/A converter output for this flow controllers. The bubbler pressure will be determined by the setting of PR1 and the pressure drop from the bubbler to the chamber will occur across FEMFC1. During manual operation from the front panel, the flow through FEMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to FEMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the *D/A* converter.

With the switches in the present configuration, the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve FEPV2 is open. When the BUBBLER IN switch is turned on, FEPV1 and FEPV3open immediately and the green light, BUBBLER IN, turns on. After a two-second delay the red PURGE BUBBLER light goes out and FEPV2 closes. This insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when FEPV1and FEPV3 open. When FEPV2 closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve. When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. Shutdown of the system should proceed by reversing the sequence of the above steps.

The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through FEMFC1.

Operation with hydrogen dilution:

With all switches closed, FEMFC1, FEMFC2, and FEMFC3 are commanded closed, FEPV1, FEPV2, FEPV3, FEPV4, FEPV6, FEPV7, and FEPV12 are closed and FEPV5 is in the vent position

When the ACTIVE switch is turned on, the signals to command closure of FEMFC1 and FEMFC2 are removed, FEPV2, and FEPV4 open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control input of FEMFC2which programs its flow setting to 200 sccm. This insures that the flow setting through FEMFC2 will always be larger than the flow through FEMFC1 and the bubbler will not become evacuated; the control valve contained within FEMFC2 will remain wide open. With the switches in this configuration, the hydrogen carrier gas will flow through FEMFC2, through the bypass valve FEPV2, through FEMFC1, through FEPV4, through FEPV5, and into the vent manifold at a rate determined by the current value of the voltage input to FEMFC1 as determined either by the front panel potentiometer or the D/A converter output for this flow controller. The bubbler pressure will be determined by the setting of PR1 and the pressure drop from the bubbler to the chamber will occur across FEMFC1. During manual operation from the front panel, the flow through FEMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to FEMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. With the switches in this configuration the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve FEPV2 is open.

The H_2 DILUTE switch becomes enabled when the ACTIVE switch is on. It may be turned at any time, however, in a normal growth sequence it would be turned on after the ACTIVE switch is turned on and before the BUBBLER IN switch is turned on. When the H_2 DILUTE switch is turned on, FEPV6, FEPV7, and FEPV12 open, the positive closure signal on FEMFC3 is removed, and the +10VDC control signal which had been keeping FEMFC2fully open is removed. The flow rates through FEMFC1, FEMFC2, and FEMFC3 can now be controlled independently. Hydrogen may be supplied to the circuit through FEMFC2 and/or FEPV12 and may leave through FEMFC1 and/or FEMFC3. If the flow meters are set correctly, then the flow through FEMFC2 is less than the sum of the flows through FEMFC1 and FEMFC3. The pressure in the circuit will then drop until it is less than the pressure at the outlet of PR4. At this point the check valve between FEPV6and FEPV12 will open and the circuit pressure will be maintained at the pressure of PR4. The difference between the exit rate of gas through FEMFC1 and FEMFC3 and the entrance rate through FEMFC2 will be maintained by the flow through the check valve. The equivalent flow through valve FEPV5 is

$$\frac{Flow_2}{Flow_1 + Flow_3} Flow_1$$

There is nothing to prevent the operator from setting the mass flow controllers such that the flow setting for FEMFC2 is greater than the combined flow through FEMFC1 and FEMFC3. In this case the control valve for FEMFC2 would remain fully open and the flow through the circuit would be controlled by the settings on FEMFC1 and FEMFC3. The pressure in the bubbler would remain at the outlet pressure of PR1 and flow through the check valve between FEPV6 and FEPV12 would not occur. Although no damage would occur from operating the system in this manner, there would be no advantage to doing it. In fact, the amount of alkyl flowing through FEMFC3 would be wasted with no improvement in the accuracy in controlling the flow of a small amount of alkyl through FEMFC1.

When the BUBBLER IN switch is turned on, FEPV1 and FEPV3 open immediately and the green light, BUBBLER IN, turns on. After a two-second delay the red PURGE BUBBLER light goes out and FEPV2 closes. The delay on closure insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when FEPV1and FEPV3open. When FEPV2 closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve.

When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. Shutdown of the system should proceed by turning off, in sequence, the RUN switch, the H_2 DILUTE switch, the BUBBLER IN switch, and the ACTIVE switch.

The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through FEMFC1 and/or FEMFC3.

3. End of day: The alkyl vapors will accelerate the deterioration of some of the elastomers from which the o-rings are fabricated. Elastomer seals have been eliminated whenever possible in the construction of this system but current technology does not allow them to be completely eliminated. They are present in the mass flow controller and in the vent/run valve. For this reason it is good practice to thoroughly flush the alkyl vapor from FEMFC1, FEMFC2, FEMFC3, and FEPV5 before the circuit is shut down. This can be accomplished by allowing hydrogen to flow through FEMFC1, FEMFC2, and FEMFC3for approximately 10 minutes after the BUBBLER IN switch is closed. During the purging step the ACTIVE and H_2 PURGE switches must be on and there should be a gas flow of at least 10 sccm through FEMFC1and 100 sccm through FEMFC2 and FEMFC3. After this, the ACTIVE switch can be closed, sealing off the circuit.

Interconnections

1. ACTIVE controls FESV1 which drives FEPV4.
2. BUBBLER IN controls FESV2 which drives FEPV1 and FEPV3.
3. Relays FEK1 and FEK2 control FESV3which drives FEPV2.
4. H_2 DILUTE controls FESV4 that drives FEPV6, FEPV7, and FEPV12.

Interlocks

1. All source valves are deactivated if the following conditions exist:
    1. System is in abort status.
    2. The vacuum exhaust valve, EXPV2, is closed.
    3. The deposition chamber is up to atmosphere.
    4. The manifold diluent gas is in the off position.
2. All switches are disabled if the ACTIVE switch is in the off position.
3. FEPV1 and FEPV3 cannot be opened unless FEPV4 is open.
4. With the PURGE switch on the front panel in the locked position (key removed), FEPV2 cannot be opened if FEPV1 and FEPV3 are open.
5. The bypass valve (FEPV2) is open if the BUBBLER IN switch is closed (handle down) and the ACTIVE switch is on (handle up).
6. The ACTIVE, BUBBLER IN, H_2 DILUTE, and RUN switches on the front panel are disabled when the computer switches the circuit into the REMOTE mode.
7. When the bubbler is connected to the circuit by opening the BUBBLER IN switch (handle up), valves FEPV1 and FEPV3 open immediately. Valve FEPV2closes after a 2 second time delay to insure that the pressure on each side of the bubbler is equalized.
8. FEMFC1, FEMFC2, and FEMFC3 are commanded closed if the ACTIVE switch is in the off position. The key lock switch CLOSE MFC2 when turned clockwise will close FEMFC2 under all conditions.
9. When the H_2 DILUTE switch is in the off position, FEMFC3 is commanded closed and the set point of FEMFC2 is at 200 sccm. The purpose of setting the FEMFC2flow to 200 sccm is to insure that the flow setting for FEMFC2 is always greater that the flow setting for FEMFC1 when H_2 DILUTE is in the off position. This prevents the bubbler from becoming evacuated. When the H_2 DILUTE switch in on, the closure signal is removed from FEMFC3 and the set point voltage for FEMFC2 is determined by either front panel potentiometer or remote D/A converter. A closure signal on a mass flow controller will over-ride the set point signal.
10. The manifold diluent gas cannot be turned off if the ACTIVE switch is on.

Programming commands for remote operation

INSERT LABVIEW DOCUMENTATION HERE

Removing and replacing source bubbler

Purging of the bubbler lines must be done through the outlet route. The output can be evacuated with the vacuum pump through valves FEPV4 and FEPV5. The limited conductance of the mass flow controller FEMFC1 requires that times of ≈1minute be used to evacuate the lines to the bubbler. Purging is accomplished by alternately evacuating the lines and backfilling them with hydrogen. During the evacuation cycle it is necessary to open FEPV4 and keep FEMFC2 closed. For this purpose, a key lock switch, CLOSE MFC2, is installed on the front panel. When the key position is fully clockwise, FEMFC2 will be closed regardless of the positions of the other switches.

Bubbler removal

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Close the manual valves on the inlet and outlet of the FE bubbler.
3. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
4. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the N_2 CARRIER position.
5. On the IRON control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for FEMFC1 to a flow of 100 sccm.
6. Evacuation: Insert a key in the CLOSE MFC2 switch and turn it clockwise. This will shut off the gas flow through FEMFC2. As the gas in the line is evacuated, the flow rate will drop. When the flow rate for FEMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated.
7. Backfill: Turn the key in the CLOSE MFC2 switch counter clockwise for 10 seconds. This will allow the bubbler circuit to fill with nitrogen.
8. Repeat steps 6 and 7 twenty times to completely purge the circuit of alkyl vapor. Finish with a backfill step.
9. Turn the BUBBLER IN switch and the ACTIVE switch to the off position.
10. Disconnect and remove the bubbler. CAUTION: This is a potentially hazardous step if the manual bubbler valves have failed to seal. The pyrophoric liquid will ignite when exposed to air. The quantities are usually small enough to easily contain the burning alkyl and the oxidation is limited by the diffusion rate of oxygen into the tubing, which is rather slow. Avoid breathing the oxide fumes. Have a dry chemical or non-flammable dry absorbent medium available to smother the chemical fire should it be necessary. In most cases probably the best remedy is to attach the stainless steel VCR caps to seal the inlet and outlet of the bubbler. DO NOT USE WATER!!
11. Install the stainless steel VCR caps on the bubbler ports. Use new gaskets unless the gaskets are of the self centering type.
12. If a new bubbler is being installed immediately, proceed to the next section. Otherwise, shut down the system as appropriate.

Bubbler installation

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Insure that one of the hydrogen cylinders is actively connected to the hydrogen supply line, that the pressure regulator is properly set and that the hydrogen purifier is turned on and up to temperature.
3. Insure that PR4 is set at 10 psig and PR1 is set at 20 psig.
4. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
5. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the H_2 CARRIER position.
6. Insure that the manual valves on the bubbler are closed. Uncap the inlet and outlet ports on the bubbler and connect the bubbler to the circuit using new gaskets if the previously used gaskets were not of the self-centering type.
7. On the IRON control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for FEMFC1 to a flow of 100 sccm.
8. <u>Evacuation</u>: Insert a key in the CLOSE MFC2 switch and turn it clockwise. This will shut off the gas flow through FEMFC2. As the gas in the line is evacuated, the flow rate will drop. When the flow rate for FEMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated.
9. <u>Backfill</u>: Turn the key in the CLOSE MFC2 switch counter clockwise for 10 seconds. This will allow the bubbler circuit to fill with nitrogen.
10. Repeat steps 8 and 9 twenty times to completely purge the circuit of air. Finish with a backfill step. Remove the key to the CLOSE MFC2 switch.
11. Open the manual valve on the outlet of the bubbler first. If the pressure in the bubbler should happen to be greater than 10 psig (the setting of PR4) and the inlet valve were opened first, the liquid alkyl would be forced back into the inlet plumbing.
12. Open the manual valve on the inlet of the bubbler.
13. Turn the BUBBLER IN switch and the ACTIVE switch to the off position. Turn the PURGE key lock switch off (counter-clockwise) and remove the key.
14. Shut down the system as appropriate.

TriMethylGallium (TMGa)

Gas Distribution

1. TriMethylGallium is a liquid at room temperature with a vapor pressure of about 178 Torr at room temperature. This means that the pressure of the vapor is 178/760 x 14.7 psia; about 1/5 atmospheric pressure. The vapor is transported from the stainless steel canister which contains the liquid, into the reaction chamber by bubbling hydrogen gas through the liquid.
2. The TMGa plumbing system is a more complex arrangement that allows the vapor stream passing through the bubbler to be diluted with additional hydrogen carrier gas. In addition, a portion of the total volume of gas may be dumped into the scrubber before it enters the vent/run valve. This arrangement permits a wider range of TMGa flow rates to be directed into the deposition chamber. The pressure of the hydrogen gas inside the liquid container is determined by the pressure setting of either PR1 or PR2 depending on the mode of operation. The proper functioning of the various components is discussed in detail in the section on operating procedures.
3. The precise vapor pressure of the TMGa is determined by the temperature of the liquid. This is controlled by immersing the liquid in a coolant bath which is controlled at a precise temperature by a small Brinkmann refrigeration unit.
4. Pressure drop to the reaction chamber occurs across the mass flow controller, GAMFC1.
5. The alkyl is exposed to the heated sensor element of the mass flow controller (110°C); thus it must be stable at this temperature.
6. Loss of liquid from the bubbler into the gas lines is unlikely because pressure in the gas line is the same as the bubbler. (Closure of the bypass valve, GAPV2, is delayed to allow bubbler inlet and outlet pressure to equilibrate.)
7. Upon power failure or other abort conditions, contamination of the upstream H_2 lines is prevented by closure of the control valve for mass flow controller GAMFC2.
8. Adiabatic cooling could possibly cause alkyl condensation in the mass flow controller valve. It is advisable to keep the alkyl temperature below room temperature.
9. A purge mode overrides some interlocks to allow evacuation and backfill of the bubbler lines.

Hazards

1. TMGa is pyrophoric.
2. The oxides formed from the burning of TMGa are toxic.
3. TMGa reacts violently with water, which should never be used to extinguish the burning alkyl. Use a dry chemical fire extinguisher only or else let it burn.

Precautions

1. Keep the manual valves on a newly installed bubbler closed until it is connected to the system, leak checked and purged.
2. Avoid gas pressure differentials that can force the alkyl from the bubbler back into the carrier gas piping.

Normal Operation

1. Start-of-day: Since the pneumatic valves GAPV1and GAPV2 seal off the bubbler from the rest of the system, the manual valves on the bubbler inlet and outlet can be left open except when the bubbler is being replaced

or removed. At the start-of-day the FE is therefore ready to operate from the control panel. The operation of the controls for the cases without hydrogen dilution and with hydrogen dilution will be discussed separately.

2. Normal growth sequence:

Operation without additional hydrogen dilution:

During a normal growth sequence the switches on the front panel are turned on in a left to right sequence and turned off in a right to left sequence. With all switches closed, GAMFC1, GAMFC2, and GAMFC3 are commanded closed, GAPV1, GAPV2, GAPV3, GAPV4, GAPV6, GAPV7, and GAPV12 are closed and GAPV5 is in the vent position. When the ACTIVE switch is turned on, the signals to command closure of GAMFC1 and GAMFC2 are removed, GAPV2, and GAPV4 open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control input of GAMFC2which programs its flow setting to 200 sccm. This insures that the flow setting through GAMFC2 will always be larger than the flow through GAMFC1 and the bubbler will not become evacuated; the control valve contained within GAMFC2 will remain wide open. With the switches in this configuration, the hydrogen carrier gas will flow through GAMFC2,through the bypass valve GAPV2, through GAMFC1, through GAPV4, through GAPV5 and into the vent manifold at a rate determined by the current value of the voltage input to GAMFC1 as determined either by the front panel potentiometer or the D/A converter output for this flow controllers. The bubbler pressure will be determined by the setting of PR1 and the pressure drop from the bubbler to the chamber will occur across GAMFC1. During manual operation from the front panel, the flow through GAMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to GAMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the *D/A* converter.

With the switches in the present configuration, the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve GAPV2 is open. When the BUBBLER IN switch is turned on, GAPV1 and GAPV3open immediately and the green light, BUBBLER IN, turns on. After a two-second delay the red PURGE BUBBLER light goes out and GAPV2 closes. This insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when GAPV1and GAPV3 open. When GAPV2 closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve. When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. Shutdown of the system should proceed by reversing the sequence of the above steps.

The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through GAMFC1.

Operation with hydrogen dilution:

With all switches closed, GAMFC1, GAMFC2, and GAMFC3 are commanded closed, GAPV1, GAPV2, GAPV3, GAPV4, GAPV6, GAPV7, and GAPV12 are closed and GAPV5 is in the vent position

When the ACTIVE switch is turned on, the signals to command closure of GAMFC1 and GAMFC2 are removed, GAPV2, and GAPV4 open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control input of GAMFC2which programs its flow setting to 200 sccm. This insures that the flow setting through GAMFC2 will always be larger than the flow through GAMFC1 and the bubbler will not become evacuated; the control valve contained within GAMFC2 will remain wide open. With the switches in this configuration, the hydrogen carrier gas will flow through GAMFC2, through the bypass valve GAPV2, through GAMFC1, through GAPV4, throughGAPV5andintotheventmanifoldata rate determined by the current value of the voltage input to GAMFC1as determined either by the front panel potentiometer or the D/A converter output for this flow controller. The bubbler pressure will be determined by the setting of PR1 and the pressure drop from the bubbler to the chamber will occur across GAMFC1. During manual operation from the front panel, the flow through GAMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to GAMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. With the switches in this configuration the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve GAPV2 is open.

The H_2 DILUTE switch becomes enabled when the ACTIVE switch is on. It may be turned at any time, however, in a normal growth sequence it would be turned on after the ACTIVE switch is turned on and before the BUBBLER IN switch is turned on. When the H_2 DILUTE switch is turned on, GAPV6, GAPV7, and GAPV12 open, the positive closure signal on GAMFC3 is removed, and the +10VDC control signal which had been keeping GAMFC2fully open is removed. The flow rates through GAMFC1, GAMFC2, and GAMFC3 can now be controlled independently. Hydrogen may be supplied to the circuit through GAMFC2 and/or GAPV12 and may leave through GAMFC1 and/or GAMFC3. If the flow meters are set correctly, then the flow through GAMFC2 is less than the sum of the flows through GAMFC1 and GAMFC3. The pressure in the circuit will then drop until it is less than the pressure at the outlet of PR4. At this point the check valve between GAPV6and GAPV12 will open and the circuit pressure will be maintained at the pressure of PR4. The difference between the exit rate of gas through GAMFC1 and GAMFC3 and the entrance rate through GAMFC2 will be maintained by the flow through the check valve. The equivalent flow through valve GAPV5 is

$$\frac{Flow_2}{Flow_1 + Flow_3} Flow_1$$

There is nothing to prevent the operator from setting the mass flow controllers such that the flow setting for GAMFC2 is greater than the combined flow through GAMFC1 and GAMFC3. In this case the control valve for GAMFC2 would remain fully open and the flow through the circuit would be controlled by the settings on GAMFC1 and GAMFC3. The pressure in the bubbler would remain at the outlet pressure of PR1 and flow through the check valve between GAPV6 and GAPV12 would not occur. Although no damage would occur from operating the system in this manner, there would be no advantage to doing it. In fact, the amount of alkyl flowing through GAMFC3 would be wasted with no improvement in the accuracy in controlling the flow of a small amount of alkyl through GAMFC1.

When the BUBBLER IN switch is turned on, GAPV1 and GAPV3 open immediately and the green light, BUBBLER IN, turns on. After a two-second delay the red PURGE BUBBLER light goes out and GAPV2 closes. The delay on closure insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when GAPV1and GAPV3open. When GAPV2 closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve.

When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. Shutdown of the system should proceed by turning off, in sequence, the RUN switch, the H_2 DILUTE switch, the BUBBLER IN switch, and the ACTIVE switch.

The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through GAMFC1 and/or GAMFC3.

3. End of day: The alkyl vapors will accelerate the deterioration of some of the elastomers from which the o-rings are fabricated. Elastomer seals have been eliminated whenever possible in the construction of this system but current technology does not allow them to be completely eliminated. They are present in the mass flow controller and in the vent/run valve. For this reason it is good practice to thoroughly flush the alkyl vapor from GAMFC1, GAMFC2, GAMFC3, and GAPV5 before the circuit is shut down. This can be accomplished by allowing hydrogen to flow through GAMFC1, GAMFC2, and GAMFC3for approximately 10 minutes after the BUBBLER IN switch is closed. During the purging step the ACTIVE and H_2 PURGE switches must be on and there should be a gas flow of at least 10 sccm through GAMFC1and 100 sccm through GAMFC2 and GAMFC3. After this, the ACTIVE switch can be closed, sealing off the circuit.

Interconnections

1. ACTIVE controls GASV1 which drives GAPV4.
2. BUBBLER IN controls GASV2 which drives GAPV1 and GAPV3.
3. Relays GAK1 and GAK2 control GASV3 which drives GAPV2.
4. H_2 DILUTE controls GASV4, which drives GAPV6, GAPV7, and GAPV12.

Interlocks

1. All source valves are deactivated if the following conditions exist:
    1. System is in abort status.
    2. The vacuum exhaust valve, EXPV2, is closed.
    3. The deposition chamber is up to atmosphere.
    4. The manifold diluent gas is in the off position.
2. All switches are disabled if the ACTIVE switch is in the off position.
3. GAPV1 and GAPV3 cannot be opened unless GAPV4 is open.
4. With the PURGE switch on the front panel in the locked position (key removed), GAPV2 cannot be opened if GAPV1 and GAPV3 are open.
5. The bypass valve (GAPV2) is open if the BUBBLER IN switch is closed (handle down) and the ACTIVE switch is on (handle up).
6. The ACTIVE, BUBBLER IN, H_2 DILUTE, and RUN switches on the front panel are disabled when the computer switches the circuit into the REMOTE mode.
7. When the bubbler is connected to the circuit by opening the BUBBLER IN switch (handle up), valves GAPV1and GAPV3 open immediately. Valve GAPV2 closes after a 2 second time delay to insure that the pressure on each side of the bubbler is equalized.
8. GAMFC1, GAMFC2, and GAMFC3 are commanded closed if the ACTIVE switch is in the off position. The key lock switch CLOSE MFC2 when turned clockwise will close GAMFC2 under all conditions.
9. When the H_2 DILUTE switch is in the off position, GAMFC3 is commanded closed and the set point of GAMFC2 is at 200 sccm. The purpose of setting the GAMFC2flow to 200 sccm is to insure that the flow setting for GAMFC2 is always greater that the flow setting for GAMFC1 when H_2 DILUTE is in the off position. This prevents the bubbler from becoming evacuated. When the H_2 DILUTE switch in on, the closure signal is removed from GAMFC3 and the set point voltage for GAMFC2 is determined by either the front panel

potentiometer or the remote D/A converter. A closure signal on a mass flow controller will over-ride the set point signal.

10. The manifold diluent gas cannot be turned off if the ACTIVE switch is on.

Programming commands for remote operation

Insert Labview Documantation Here

Removing and replacing source bubbler

Purging of the bubbler lines must be done through the outlet route. The output can be evacuated with the vacuum pump through valves GAPV4 and GAPV5. The limited conductance of the mass flow controller GAMFC1 requires that times of ≈1minute be used to evacuate the lines to the bubbler. Purging is accomplished by alternately evacuating the lines and backfilling them with hydrogen. During the evacuation cycle it is necessary to open GAPV4 and keep GAMFC2 closed. For this purpose, a key lock switch, CLOSE MFC2, is installed on the front panel. When the key position is fully clockwise, GAMFC2 will be closed regardless of the positions of the other switches.

Bubbler removal

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Close the manual valves on the inlet and outlet of the TMGa bubbler.
3. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
4. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the N_2 CARRIER position.
5. On the GALLIUM control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for GAMFC1 to a flow of 100 sccm.
6. <u>Evacuation</u>: Insert a key in the CLOSE MFC2 switch and turn it clockwise. This will shut off the gas flow through GAMFC2. As the gas in the line is evacuated, the flow rate will drop. When the flow rate for GAMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated.
7. <u>Backfill</u>: Turn the key in the CLOSEMFC2switch counter clockwise for 10 seconds. This will allow the bubbler circuit to fill with nitrogen.
8. Repeat steps 6 and 7 twenty times to completely purge the circuit of alkyl vapor. Finish with a backfill step.
9. Turn the BUBBLER IN switch and the ACTIVE switch to the off position.
10. Disconnect and remove the bubbler. CAUTION: This is a potentially hazardous step if the manual bubbler valves have failed to seal. The pyrophoric liquid will ignite when exposed to air. The quantities are usually small enough to easily contain the burning alkyl and the oxidation is limited by the diffusion rate of oxygen into the tubing, which is rather slow. Avoid breathing the oxide fumes. Have a dry chemical or non-flammable dry absorbent medium available to smother the chemical fire should it be necessary. In most cases probably the best remedy is to attach the stainless steel VCR caps to seal the inlet and outlet of the bubbler. DO NOT USE WATER!!

11. Install the stainless steel VCR caps on the bubbler ports. Use new gaskets unless the gaskets are of the self centering type.
12. If a new bubbler is being installed immediately, proceed to the next section. Otherwise, shut down the system as appropriate.

Bubbler installation

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Insure that one of the hydrogen cylinders is actively connected to the hydrogen supply line, that the pressure regulator is properly set and that the hydrogen purifier is turned on and up to temperature.
3. Insure that PR2 is set at 10 psig and PR1 is set at 15 psig.
4. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
5. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the H_2 CARRIER position.
6. Insure that the manual valves on the bubbler are closed. Uncap the inlet and outlet ports on the bubbler and connect the bubbler to the circuit using new gaskets if the previously used gaskets were not of the self-centering type.
7. On the GALLIUM control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for GAMFC1 to a flow of 100 sccm.
8. <u>Evacuation</u>: Insert a key in the CLOSE MFC2 switch and turn it clockwise. This will shut off the gas flow through GAMFC2. As the gas in the line is evacuated, the flow rate will drop. When the flow rate for GAMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated.
9. <u>Backfill:</u> Turn the key in the CLOSE MFC2 switch counter clockwise for 10 seconds. This will allow the bubbler circuit to fill with nitrogen.
10. Repeat steps 8 and 9 twenty times to completely purge the circuit of air. Finish with a backfill step. Remove the key to the CLOSE MFC2 switch.
11. Open the manual valve on the outlet of the bubbler first. If the pressure in the bubbler should happen to be greater than 10 psig (the setting of PR2) and the inlet valve were opened first, the liquid alkyl would be forced back into the inlet plumbing.
12. Open the manual valve on the inlet of the bubbler.
13. Turn the BUBBLER IN switch and the ACTIVE switch to the off position. Turn the PURGE key lock switch off (counter-clockwise) and remove the key.
14. Shut down the system as appropriate.

Arsine (AsH_3)

Gas Distribution

1. Arsine at 10 psig output pressure of the gas cylinder regulator is fed to a mass flow controller supplying this gas to the reactor vent/run manifold. The pressure differential between the source and the reactor chamber is accommodated across the flow controller. Alternatively, the AsH_3 can be valved off and hydrogen passed through the flow controller to purge the toxic arsine.
2. A purge-mode switchable from the front panel allows evacuation and backfill of the delivery lines with H_2 purge gas. Valving at the regulator allows purging of the cylinder lines for changing the cylinder.

Hazards

1. Arsine is very toxic.
2. Release of high pressure arsine can occur by operator carelessness.
3. Arsine and the hydrogen carrier gas are flammable.
4. Arsine is a colorless gas with a garlic-like odor. The odor threshold is 0.5 ppm. The OSHA TLV-TWA (Threshold Limit Value – Time Weighted Average exposure for an 8-hour shift) is 50 ppb. The DOD ChemLogic CL8 toxic gas monitor is set to give an alarm at this value. If the level of arsine detected in the laboratory space exceeds 100 ppm the building evacuation alarm will be activated. All personnel not equipped with respirators should immediately leave the building.
5. Pure arsine has a specific gravity of 2.695 (air=1).

Precautions

1. Source volume limit: Safety dictated use of a limited volume of AsH_3 as a means of controlling a catastrophic failure. We have limited ourselves to a 26 cu. ft. cylinder of AsH_3.
2. Ventilation: The AsH_3 cylinder is in a separate enclosed and ventilated gas cabinet.
3. Toxic gas monitoring:
    1. The DOD ChemLogic CL8 Toxic Gas Monitor samples the toxic gas cabinet atmosphere. An alarm will activate at the AsH_3 TLV-TWA and shut down the system.
    2. The DOD ChemLogic CL8 Toxic Gas Monitor samples the reactor cabinet atmosphere. An alarm will activate at the AsH_3 TLV-TWA and shut down the system.
    3. The DOD ChemLogic CL8 monitors the outlet of the scrubber. An alarm will activate at the AsH_3 TLV-TWA and shut down the system.
    4. The DOD ChemLogic CL8 Toxic Gas Monitor samples the system cabinet atmosphere. An alarm will activate at the AsH_3 TLV-TWA and shut down the system.
    5. The CL8 monitor also samples the immediate laboratory air in room 240. An alarm will activate at the AsH_3 TLV-TWA and shut down the system. At twice the TLV-TWA the building evacuation alarm will sound. (In case of alarm - leave the area and assist others in leaving the building.)
4. System operation features:
    1. Interlocks prevent the use of AsH_3 except under normal operation of the sealed gas system.
    2. Opening the chamber for loading and unloading samples disables the AsH_3 gas source.
    3. Removal of the AsH_3 cylinder requires purging of the gas regulator inlet line to remove the AsH_3 prior to disconnecting.
    4. Replacement of AsH_3 cylinder requires purging of the gas regulator inlet line to remove air contamination.

5. Gas cylinder change must be done with an observer to double-check procedures and assist in an emergency.

Normal Operation

Normal daily operation once a cylinder has been installed and the delivery lines and regulator are filled. Caution: The regulator and delivery line contain arsine and are not purged during overnight shutdown. Since the cylinder valve is closed at days end, the volume of toxic gas left in the lines represents a small risk in case of line rupture. The alternative of purging the line each day would waste a significant fraction of the small cylinder volume and would add to the load of arsine to be scrubbed from the exhaust gas.

Start of day

1. Open the cylinder valve (Normal operating pressure is 1a psig).
2. Open the regulator low pressure outlet valve, AGMV1.
3. Use AsH_3 from the control panel as appropriate.
4. During a normal growth sequence the switches on the front panel are turned on in a left to right sequence and turned off in a right to left sequence. With all switches closed, AGMFC1 is commanded closed, AGPV4, AGPV1a and AGPV11 are closed and AGPV5 is in the vent position. When the ACTIVE switch is turned on, the signal to command closure of AGMFC1 is removed, AGPV4 opens and the green ACTIVE light comes on. When the GAS SOURCE IN switch is turned on, AGPV1a opens, gas flow as determined by the input voltage to AGMFC1 begins to flow into the vent manifold, and the green GAS SOURCE IN light comes on. During manual operation from the front panel, the flow through AGMFC1 is controlled by the setting of the 1a-turn FLOW SET potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black DVM SELECT button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to AGMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the DVM SELECT button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. When the RUN switch is turned on, the flow at the outlet of AGPV5 is switched from the vent manifold to the run manifold and the yellow RUN light comes on. The system would be shut down in the reverse order.

End of day

1. Secure the AsH_3 flow from the control panel.
2. Close the cylinder valve.
3. Close the valve, AGMV1, at the output of the regulator.

Interconnections

4. ACTIVE controls AGSV1 which drives AGPV4.
5. GAS SOURCE IN controls AGSV2 which drives AGPV10.
6. H_2 FLUSH controls AGSV3 which drives AGPV11.

Interlocks

1. All source valves are deactivated if the following conditions exist:
    1. System is in abort status.
    2. The vacuum exhaust valve, EXPV2, is closed.
    3. The deposition chamber is up to atmosphere.

    4. The manifold diluent gas is in the off position.
2. If the GAS SOURCE IN switch is on (handle up), then the H_2 FLUSH switch is disabled. This prevents AGPV11 from opening if AGPV10 is open.
3. If the H_2 FLUSH switch is on (turned clockwise, the red H_2 FLUSH light will be on), then the GAS SOURCE IN switch is disabled. This prevents AGPV10 from opening if AGPV11 is open.
4. Interlocks2 and 3 are disabled when the key lock PURGE switch is in the on position (In this position the key is in the switch and cannot be removed) The red PURGE light in the panel will be on. This allows the gas circuit to be flushed with either hydrogen or nitrogen during either startup or after repairs have been made. For normal operation the PURGE switch should be in the off position and the key removed.
5. All front panel switches with the exception of the PURGE switch are disabled when the computer commands the circuit to the REMOTE mode.

Programming commands for remote operation

Purging of AsH_3 Lines

Purging must be achieved through the mass flow controller to the vacuum pump. Use alternating evacuation/backfill cycles to remove reactive gases. Due to the limited conductance of the gas lines allow cycle times of a few minutes. The regulator inlet lines can be independently purged for changing cylinders.

Regulator and low-pressure delivery lines

This operation need be performed only to purge the line downstream of the regulator; purge of the high-pressure lines is described in the next section.

1. Close the high-pressure inlet valve, AGMV2 to the regulator.
2. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel and set the N_2 manifold flow controller to maintain a minimum deposition chamber pressure of 1-2 Torr.
3. Evacuation: Open the ACTIVE switch, the GAS SOURCE IN switch, and the RUN switch. Adjust AGMFC1 for maximum flow. Allow several minutes for the line to evacuate.
4. Backfill: Turn the key lock PURGE switch to the on position, and turn the key lock H_2 FLUSH switch on for at least 10 seconds to backfill the line with hydrogen. Close the H_2 FLUSH switch.
5. Repeat the evacuate cycle allowing several minutes for the line to evacuate.
6. Repeat the backfill/evacuate cycle a total of five times. After the last evacuate cycle set AGMFC1 to zero flow, turn the PURGE switch to the off position and remove the key. Turn the H_2 FLUSH switch to the off position and remove the key.
7. Backfill with AsH_3:
    1. Back-off the knob on pressure regulator AGPR1 (fully counterclockwise).
    2. Open the high-pressure regulator inlet valve AGMV2.
    3. Set the knob on pressure regulator AGPR1 to 10 psig at the outlet. Check the excess-flow valve AGEFV1 to see that it has not tripped by pushing in on the red button and observing the gauge on the outlet of AGPR1for any changes in pressure.
    4. Close the RUN SWITCH, the GAS SOURCE IN switch and the ACTIVE switch.

Cylinder Replacement

Cylinder replacement requires purging of only the high-pressure inlet line to the regulator. The high purity, low-pressure delivery lines are not exposed to air during the replacement operation.

1. Cylinder removal:
    1. Tightly close the AsH_3 cylinder valve and prepare to remove gas from the inlet line.

2. Flow AsH_3 through the reactor to reduce the inlet pressure to ""0 psig.
3. Close the regulator inlet valve AGMV2.
4. Lock the excess flow valve AGEFV1 in the open position with tape.
5. Purge the regulator inlet: Turn on the nitrogen supply valve, NMV3, to the vacuum educator and open the AsH_3 regulator vent valve, AGMV4. (DANGER: Be sure the AsH_3 cylinder valve is closed.) After 5 seconds close AGMV4 and NMV3.
6. Backfill the regulator inlet: First insure the tank valve for AGPURGEPR1 is open, that the outlet of regulator AGPURGEPR1 is at least 15 psig, and that AGPURGEMV1 is fully open (clockwise). Backfill the regulator input with nitrogen by opening AGMV3 for at least 2 seconds. Close AGMV3.
7. Repeat the purge-backfill cycle of steps e. and f. twenty times. Finish with a backfill step, close AGMV3 and AGMV4, and disconnect and cap the cylinder. The handheld Matheson toxic gas monitor should be used when unscrewing the cylinder connection. The CGA 350 connection on the AsH_3 cylinder has a left-hand thread. If you are facing the cylinder, the nut on the excess flow valve must be turned clockwise to loosen it. It is very important that the brass cap for the cylinder valve be replaced. The cylinder valves sometimes have small leaks even when tightly closed.

2. Cylinder installation:
    1. With the new AsH_3 cylinder sitting inside the MOCVD cabinet and the exhaust fan on, slightly loosen the brass cap which is plugging the end of the CGA 350 connection on the cylinder valve. The cap has a left-hand thread. Check for the presence of AsH_3 using the portable Matheson toxic gas detector. If a high level release of AsH_3 is detected, the cap on the valve should be retightened and the cylinder returned to the vendor. If a low level release is detected, it may be due to a small amount of arsine trapped between the closed valve on the cylinder and the brass cap. With the brass cap left slightly unscrewed close the door to the MOCVD cabinet and let the tank set for several hours. If no AsH_3 is detected at that point, it is safe to completely unscrew the cap and install the cylinder. Otherwise the cap should be re-tightened and the tank returned to the vendor for replacement.
    2. Connect the excess flow valve, AGEFV1, to the cylinder. Keep the regulator inlet valve, AGMV2, closed as well as the nitrogen backfill valve AGMV3. Hold the excess flow valve, AGEFV1, open by temporarily holding in the red button with a piece of tape.
    3. Purge the regulator input: Turn on the nitrogen flow for the vacuum eductor, by opening NMV3. Open the purge valve, AGMV4, wait 5 seconds and then close AGMV4 and NMV3.
    4. Backfill the regulator input: First insure the tank valve for AGPURGEPR1 is open, that the outlet of regulator AGPURGEPR1 is at least 15 psig, and that AGPURGEMV1 is fully open (clockwise). Backfill the regulator input with nitrogen by opening AGMV3 for at least 2 seconds. Close AGMV3.
    5. Repeat the backfill-purge cycle 20 times finishing with a purge step. Insure that AGMV3, AGMV4, and NMV3 are closed.
    6. Remove the tape from the excess flow valve, AGEFV1.
    7. Open the manual valve on the tank and check for leaks.
    8. Reset the excess flow valve, AGEFV1, by pushing in the red button and check for leaks.
    9. Open the manual valve on the regulator input, AGMV2 and check for leaks.
    10. Close the tank valve on the nitrogen cylinder supplying gas to regulator AGPURGEPR1.

Phosphine (PH3)

Gas Distribution

1. Phosphine at 10 psig output pressure of the gas cylinder regulator is fed to a mass flow controller supplying this gas to the reactor vent/run manifold. The pressure differential between the source and the reactor chamber is accommodated across the flow controller. Alternatively the PH3 can be valved off and hydrogen passed through the flow controller to purge the toxic phosphine.
2. A purge-mode switchable from the front panel allows evacuation and backfill of the delivery lines with H_2 purge gas. Valving at the regulator allows purging of the cylinder lines for changing the cylinder.

Hazards

1. Phosphine is very toxic.
2. Phosphine is pyrophoric.
3. Release of high pressure phosphine can occur by operator carelessness.
4. The hydrogen carrier gas is flammable.
5. Phosphine is a colorless gas with a disagreeable characteristic fishy, garlic-like, or acetylene-like odor. The odor threshold is 21 ppb. The OSHA TLV-TWA (Threshold Limit Value – Time Weighted Average exposure for an 8-hour shift) is 300 ppb. The DOD ChemLogic CL8 toxic gas monitor is set to give an alarm at 50 ppb. If the level of phosphine detected in the laboratory space exceeds 100 ppm the building evacuation alarm will be activated. All personnel not equipped with respirators should immediately leave the building.
6. Pure phosphine has a specific gravity of 1.17 (air=1).

Precautions

1. Source volume limit: Safety dictated use of a limited volume of PH3 as a means of controlling a catastrophic failure. We have limited ourselves to a 26 cu. ft. cylinder of PH3.
2. Ventilation: The PH3 cylinder is in a separate enclosed and ventilated gas cabinet.
3. Toxic gas monitoring:
    1. The DOD ChemLogic CL8 Toxic Gas Monitor samples the toxic gas cabinet atmosphere. An alarm will activate at the AsH_3 TLV-TWA and shut down the system. The more sensitive limit for arsine provides an extra safety margin for phosphine detection.
    2. The DOD ChemLogic CL8 Toxic Gas Monitor samples the reactor cabinet atmosphere. An alarm will activate at the AsH_3 TLV-TWA and shut down the system. The more sensitive limit for arsine provides an extra safety margin for phosphine detection.
    3. The DOD ChemLogic CL8 monitors the outlet of the scrubber. An alarm will activate at the AsH_3 TLV-TWA and shut down the system. The more sensitive limit for arsine provides an extra safety margin for phosphine detection.
    4. The DOD ChemLogic CL8 Toxic Gas Monitor samples the system cabinet atmosphere. An alarm will activate at the AsH_3 TLV-TWA and shut down the system. The more sensitive limit for arsine provides an extra safety margin for phosphine detection.
    5. The CL8 monitor also samples the immediate laboratory air in room 240. An alarm will activate at the AsH_3 TLV-TWA and shut down the system. At twice the TLV-TWA the building evacuation alarm will sound. The more sensitive limit for arsine provides an extra safety margin for phosphine detection. (In case of alarm - leave the area and assist others in leaving the building.)
4. System operation features:

1. Interlocks prevent the use of PH3 except under normal operation of the sealed gas system.
2. Opening the chamber for loading and unloading samples disables the PH3 gas source.
3. Removal of the PH3 cylinder requires purging of the gas regulator inlet line to remove the PH3 prior to disconnecting.
4. Replacement of PH3 cylinder requires purging of the gas regulator inlet line to remove air contamination.
5. Gas cylinder change must be done with an observer to double-check procedures and assist in an emergency.

Normal Operation

Normal daily operation once a cylinder has been installed and the delivery lines and regulator are filled. Caution: The regulator and delivery line contain phosphine and are not purged during overnight shutdown. Since the cylinder valve is closed at days end, the volume of toxic gas left in the lines represents a small risk in case of line rupture. The alternative of purging the line each day would waste a significant fraction of the small cylinder volume and would add to the load of phosphine to be scrubbed from the exhaust gas.

Start of day

1. Open the cylinder valve (Normal operating pressure is 1a psig).
2. Open the regulator low pressure outlet valve, PGMV1.
3. Use PH3 from the control panel as appropriate.
4. During a normal growth sequence the switches on the front panel are turned on in a left to right sequence and turned off in a right to left sequence. With all switches closed, PGMFC1 is commanded closed, PGPV4, PGPV1a and PGPV11 are closed and PGPV5 is in the vent position. When the ACTIVE switch is turned on, the signal to command closure of PGMFC1 is removed, PGPV4 opens and the green ACTIVE light comes on. When the GAS SOURCE IN switch is turned on, PGPV1a opens, gas flow as determined by the input voltage to PGMFC1 begins to flow into the vent manifold, and the green GAS SOURCE IN light comes on. During manual operation from the front panel, the flow through PGMFC1 is controlled by the setting of the 10-turn FLOW SET potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black DVM SELECT button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to PGMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the DVM SELECT button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. When the RUN switch is turned on, the flow at the outlet of PGPV5 is switched from the vent manifold to the run manifold and the yellow RUN light comes on. The system would be shut down in the reverse order.

End of day

1. Secure the PH3 flow from the control panel.
2. Close the cylinder valve.
3. Close the valve, PGMV1, at the output of the regulator.

Interconnections

1. ACTIVE controls PGSV1 which drives PGPV4.
2. GAS SOURCE IN controls PGSV2 which drives PGPV1a.
3. H_2 FLUSH controls PGSV3 which drives PGPV11.

Interlocks

1. All source valves are deactivated if the following conditions exist:
    1. System is in abort status.
    2. The vacuum exhaust valve, EXPV2, is closed.
    3. The deposition chamber is up to atmosphere.
    4. The manifold diluent gas is in the off position.
2. If the GAS SOURCE IN switch is on (handle up), then the H_2 FLUSH switch is disabled. This prevents PGPV11 from opening if PGPV10 is open.
3. If the H_2 FLUSH switch is on (turned clockwise, the red H_2 FLUSH light will be on), then the GAS SOURCE IN switch is disabled. This prevents PGPV10 from opening if PGPV11 is open.
4. Interlocks2 and 3 are disabled when the key lock PURGE switch is in the on position (In this position the key is in the switch and cannot be removed) The red PURGE light in the panel will be on. This allows the gas circuit to be flushed with either hydrogen or nitrogen during either startup or after repairs have been made. For normal operation the PURGE switch should be in the off position and the key removed.
5. All front panel switches with the exception of the PURGE switch are disabled when the computer commands the circuit to the REMOTE mode.

Programming commands for remote operation

Purging of PH3 Lines

Purging must be achieved through the mass flow controller to the vacuum pump. Use alternating evacuation/backfill cycles to remove reactive gases. Due to the limited conductance of the gas lines allow cycle times of a few minutes. The regulator inlet lines can be independently purged for changing cylinders.

Regulator and low-pressure delivery lines

This operation need be performed only to purge the line downstream of the regulator; purge of the high-pressure lines is described in the next section.

1. Close the high-pressure inlet valve, PGMV2 to the regulator.
2. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel and set the N_2 manifold flow controller to maintain a minimum deposition chamber pressure of 1-2 Torr.
3. <u>Evacuation</u>: Open the ACTIVE switch, the GAS SOURCE IN switch, and the RUN switch. Adjust PGMFC1 for maximum flow. Allow several minutes for the line to evacuate.
4. <u>Backfill</u>: Turn the key lock PURGE switch to the on position, and turn the key lock H_2 FLUSH switch on for at least 10 seconds to backfill the line with hydrogen. Close the H_2 FLUSH switch.
5. Repeat the evacuate cycle allowing several minutes for the line to evacuate.
6. Repeat the backfill/evacuate cycle a total of five times. After the last evacuate cycle set PGMFC1 to zero flow, turn the PURGE switch to the off position and remove the key. Turn the H_2 FLUSH switch to the off position and remove the key.
7. <u>Backfill with PH3</u>:
    1. Back-off the knob on pressure regulator PGPR1 (fully counterclockwise).
    2. Open the high-pressure regulator inlet valve PGMV2.
    3. Set the knob on pressure regulator PGPR1 to 10 psig at the outlet. Check the excess-flow valve PGEFV1 to see that it has not tripped by pushing in on the red button and observing the gauge on the outlet of PGPR1for any changes in pressure.
    4. Close the RUN SWITCH, the GAS SOURCE IN switch and the ACTIVE switch.

Cylinder Replacement

Cylinder replacement requires purging of only the high-pressure inlet line to the regulator. The high purity, low-pressure delivery lines are not exposed to air during the replacement operation.

1. Cylinder removal:
    1. Tightly close the PH3 cylinder valve and prepare to remove gas from the inlet line.
    2. Flow PH3 through the reactor to reduce the inlet pressure to ≈0 psig.
    3. Close the regulator inlet valve PGMV2.
    4. Lock the excess flow valve PGEFV1 in the open position with tape.
    5. Purge the regulator inlet: Turn on the nitrogen supply valve, NMV3, to the vacuum educator and open the PH3 regulator vent valve, PGMV4. (DANGER: Be sure the PH3 cylinder valve is closed.) After 5 seconds close PGMV4 and NMV3.
    6. Backfill the regulator inlet: First insure the tank valve for PGPURGEPR1 is open, that the outlet of regulator PGPURGEPR1 is at least 15 psig, and that PGPURGEMV1 is fully open (clockwise). Backfill the regulator input with nitrogen by opening PGMV3 for at least 2 seconds. Close PGMV3.
    7. Repeat the purge-backfill cycle of steps e. and f. twenty times. Finish with a backfill step, close PGMV3 and PGMV4, and disconnect and cap the cylinder. The handheld Matheson toxic gas monitor should be used when unscrewing the cylinder connection. The CGA 350 connection on the PH3 cylinder has a left-hand thread. If you are facing the cylinder, the nut on the excess flow valve must be turned clockwise to loosen it. It is very important that the brass cap for the cylinder valve be replaced. The cylinder valves sometimes have small leaks even when tightly closed.
2. Cylinder installation:
    1. With the new PH3 cylinder sitting inside the MOCVD cabinet and the exhaust fan on, slightly loosen the brass cap which is plugging the end of the CGA 350 connection on the cylinder valve. The cap has a left-hand thread. Check for the presence of PH3 using the portable Matheson toxic gas detector. If a high level release of PH3 is detected, the cap on the valve should be retightened and the cylinder returned to the vendor. If a low level release is detected, it may be due to a small amount of phosphine trapped between the closed valve on the cylinder and the brass cap. With the brass cap left slightly unscrewed close the door to the MOCVD cabinet and let the tank set for several hours. If no PH3 is detected at that point, it is safe to completely unscrew the cap and install the cylinder. Otherwise the cap should be re-tightened and the tank returned to the vendor for replacement.
    2. Connect the excess flow valve, PGEFV1, to the cylinder. Keep the regulator inlet valve, PGMV2, closed as well as the nitrogen backfill valve PGMV3. Hold the excess flow valve, PGEFV1, open by temporarily holding in the red button with a piece of tape.
    3. Purge the regulator input: Turn on the nitrogen flow for the vacuum eductor, by opening NMV3. Open the purge valve, PGMV4, wait 5 seconds and then close PGMV4 and NMV3.
    4. Backfill the regulator input: First insure the tank valve for PGPURGEPR1 is open, that the outlet of regulator PGPURGEPR1 is at least 15 psig, and that PGPURGEMV1 is fully open (clockwise). Backfill the regulator input with nitrogen by opening PGMV3 for at least 2 seconds. Close PGMV3.
    5. Repeat the backfill-purge cycle 20 times finishing with a purge step. Insure that PGMV3, PGMV4, and NMV3 are closed.
    6. Remove the tape from the excess flow valve, PGEFV1.
    7. Open the manual valve on the tank and check for leaks.
    8. Reset the excess flow valve, PGEFV1, by pushing in the red button and check for leaks.
    9. Open the manual valve on the regulator input, PGMV2 and check for leaks.
    10. Close the tank valve on the nitrogen cylinder supplying gas to regulator PGPURGEPR1.

N-Dopant

Gas Distribution

1. This system allows use of either a gaseous or liquid source for layer doping. The gaseous source, a mixture of 5% dimethylzinc (DMZ) in argon at 10 psig output pressure of the gas cylinder regulator, is fed to a mass flow controller supplying this gas to the reactor vent/run manifold. The pressure differential between the source and the reactor chamber is accommodated across the flow controller PDOPMFC1. Alternatively, the DMZ can be valved off and hydrogen passed through the flow controller to purge the dimethylzinc.
2. A purge-mode switchable from the front panel allows evacuation and backfill of the delivery lines with H_2 purge gas. Valving at the regulator allows purging of the cylinder lines for changing the cylinder.
3. The liquid source is tetramethyltin (TMSn) which is a liquid at room temperature with a vapor pressure of 10 Torr at -200C. This means that the pressure of the vapor is 10/760 x 14.7 psia; about 1/100 atmospheric pressure. The vapor is transported from the stainless steel canister which contains the liquid, into the reaction chamber by bubbling hydrogen gas through the liquid.
4. The P-dopant plumbing system is a more complex arrangement which allows the gas streams from either the high pressure or liquid source to be diluted with additional hydrogen carrier gas. In addition, a portion of the total volume of gas may be dumped into the scrubber before it enters the vent/run valve. This arrangement permits a wider range of dopant flow rates to be directed into the deposition chamber. The pressure of the hydrogen gas inside the liquid container is determined by the pressure setting of either PR1 or PR6 depending on the mode of operation. The proper functioning of the various components is discussed in detail in the section on operating procedures.
5. The precise vapor pressure of the TMSn is determined by the temperature of the liquid. This is controlled by immersing the liquid in a coolant bath which is controlled at a precise temperature by a small Brinkmann refrigeration unit.
6. Pressure drop to the reaction chamber occurs across the mass flow controller, PDOPMFC1.
7. The alkyl is exposed to the heated sensor element of the mass flow controller (≈110°C); thus it must be stable at this temperature.
8. Loss of liquid from the bubbler into the gas lines is unlikely because pressure in the gas line is the same as the bubbler. (Closure of the bypass valve, PDOPPV2, is delayed to allow bubbler inlet and outlet pressure to equilibrate.)
9. Upon power failure or other abort conditions, contamination of the upstream H_2lines is prevented by closure of the control valve for mass flow controller PDOPMFC2.
10. Adiabatic cooling could possibly cause alkyl condensation in the mass flow controller valve. It is advisable to keep the alkyl temperature below room temperature.
11. A purge mode overrides some interlocks to allow evacuation and backfill of the bubbler lines.

Hazards

1. TMSn is pyrophoric.
2. The oxides formed from the burning of TMSn are toxic.
3. TMSn reacts violently with water which should never be used to extinguish the burning alkyl. Use a dry chemical fire extinguisher only or else let it burn.
4. The dust (silicon dioxide) formed when dimethylzinc reacts with air is toxic.
5. Release of high pressure dimethylzinc can occur by operator carelessness.

Precautions

1. Source volume limit: Safety dictated use of a limited volume of DMZ as a means of controlling a catastrophic failure. We have limited ourselves to a 26 cu. ft. cylinder of 5% DMZ in argon.
2. Ventilation: The DMZ cylinder is in an enclosed, ventilated MOCVD reactor cabinet.
3. Toxic gas monitoring:
    1. The DOD ChemLogic CL8toxic gas monitor samples the MOCVDcabinet atmosphere. An alarm will activate at the AsH_3 TLV and shut down the system. The more sensitive limit for arsine provides extra margin for dimethylzinc detection.
    2. The DOD ChemLogic CL8 monitor also samples the immediate laboratory air. An alarm will activate at the AsH_3 TLV and shut down the system. At the TLV the building Evacuation alarm will sound. (In case of alarm -leave the area and assist others in leaving the building.)
    3. The DOD ChemLogic CL8 monitors the outlet of the scrubber. If the gas concentration exceeds the arsine TLV threshold, the sources automatically shut down and an alarm on the operator panel sounds.
4. System operation features:
    1. Interlocks prevent the use of DMZ except under normal operation of the sealed gas system.
    2. Opening the chamber for loading and unloading samples disables the DMZ'
    3. Removal of the DMZ cylinder requires purging of the gas regulator inlet line to remove the DMZ prior to disconnecting.
    4. Replacement of DMZ cylinder requires purging of the gas regulator inlet line to remove air contamination.
    5. Gas cylinder change should be done with an observer to double-check procedures and assist in an emergency.
5. The gaseous source is a mixture of 5% dimethylzinc (DMZ) in argon. At this concentration, the gas is labeled flammable (not pyrophoric). Dimethylzinc is especially reactive with air, forming silicon dioxide (Si02) The silicon dioxide forms as a fine white dust which can deposit in the plumbing and valves if air and dimethylzinc are allowed to mix. The dust will prevent valve seats from forming a complete seal and can cause the pressure control valve inside the gas regulator to fail. When this happens the pressure downstream of the gas regulator will creep upward; if left without an excape route the downstream pressure could presumably rise to the full value of the pressure in the source cylinder (=2000 psi). For this reason a relief valve (PDOPPRV1) on the downstream side of the regulator has been installed to vent the gas to the scrubber if the line pressure exceeds 150 psi. This will prevent components from bursting if the regulator should fail. It is therefore extremely important to purge all the air from the components before installing a dimethylzinc cylinder and, likewise, very important to purge all the dimethylzinc from the lines before removing a dimethylzinc cylinder. If this is not done carefully, some of the gas control components could be ruined and others might require a time consuming cleanup process. An excess flow valve (PDOPEFV1) is installed on the dimethylzinc tank. If the gas flow through the valve exceeds 1000 sccm, the valve will trip shutting off the flow. It then stays closed unless it is manually reset by pushing in on the red button. The next valve after the excess flow valve is a pneumatically controlled valve (PDOPPV20) held open by the abort interlock system. This valve automatically closes in the event of power failure, loss of ventilation, operator intervention, fire, low hydrogen pressure, low nitrogen pressure, or detection of toxic gas in the cabinet, room, or at the scrubber outlet.
6. Keep the manual valves on a newly installed bubbler closed until it is connected to the system, leak checked and purged.
7. Avoid gas pressure differentials that can force the alkyl from the bubbler back into the carrier gas piping.

Normal Operation

The P-dopant circuit can provide source material from either the bubbler or the high-pressure cylinder. These flows may be operated with or without additional hydrogen dilution. Each mode of operation will be described in a separate section.

Start-of-day:

Since the pneumatic valves PDOPPV1 and PDOPPV2 seal off the bubbler from the rest of the system, the manual valves on the bubbler inlet and outlet can be left open except when the bubbler is being replaced or removed. At the start-of-day the TMSn is therefore ready to operate from the control panel.

The regulator and delivery line contain dimethylzinc and are not purged during overnight shutdown. Since the cylinder valve is closed at days end, the volume of toxic gas left in the lines represents a small risk in case of line rupture. The alternative of purging the line each day would waste a significant fraction of the small cylinder volume and would add to the load of dimethylzinc to be scrubbed from the exhaust gas.

Operation of bubbler source without additional hydrogen dilution:

With all switches closed, PDOPMFC1, PDOPMFC2, PDOPMFC3,and PDOPMFC4 are commanded closed, PDOPPV1, PDOPPV2, PDOPPV3, PDOPPV4, PDOPPV6, PDOPPV7, PDOPPV8, PDOPPV9, PDOPPV10, and PDOPPV11 are closed and PDOPPV5 is in the vent position. When the ACTIVE switch is turned on, the signals to command closure of PDOPMFC1 and PDOPMFC2 are removed, PDOPPV2, PDOPPV4, and PDOPPV9open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control inputs of PDOPMFC2 and DOPMFC4 which programs their flow settings to 200 sccm. This insures that the flow setting through PDOPMFC2 or PDOPMFC4 will always be larger than the flow through PDOPMFC1and the circuit will not become evacuated; the control valves contained within PDOPMFC2 and PDOPMFC4 will remain wide open. With the switches in this configuration, the hydrogen carrier gas will flow through PDOPMFC2,through the bypass valve PDOPPV2, through PDOPPV9, through PDOPMFC1,through PDOPPV4, through PDOPPV5and into the vent manifold at a rate determined by the current value of the voltage input to PDOPMFC1as determined either by the front panel potentiometer or the D/A converter output for this flow controllers. The circuit pressure will be determined by the setting of PR1 and the pressure drop from the circuit to the chamber will occur across PDOPMFC1. During manual operation from the front panel, the flow through PDOPMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to PDOPMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter.

With the switches in the present configuration, the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve PDOPPV2 is open. When the BUBBLER IN switch is turned on, PDOPPV1 and PDOPPV3open immediately and the green light, BUBBLER IN, turns on. After a two-second delay the red PURGE BUBBLER light goes out and PDOPPV2 closes. This insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when PDOPPV1and PDOPPV3open. When PDOPPV2closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve. When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. Shutdown of the system should proceed by reversing the sequence of the above steps.

The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through PDOPMFC1.

Operation of bubbler source with additional hydrogen dilution:

With all switches closed, PDOPMFC1, PDOPMFC2, and PDOPMFC3 are commanded closed, PDOPPV1, PDOPPV2, PDOPPV3, PDOPPV4, PDOPPV6, PDOPPV7, PDOPPV8, PDOPPV9, PDOPPV10, and PDOPPV11 are closed and PDOPPV5 is in the vent position. When the ACTIVE switch is turned on, the signals to command closure of PDOPMFC1 and PDOPMFC2 are removed, PDOPPV2, PDOPPV4, and PDOPPV9 open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control inputs of PDOPMFC2 and PDOPMFC4 which programs their flow settings to 200 sccm. This insures that the flow setting for PDOPMFC2 will always be larger than the flow setting for PDOPMFC1 and the circuit will not become evacuated; the control valves contained within PDOPMFC2 and PDOPMFC4 will remain wide open.

With the switches in this configuration, the hydrogen carrier gas will flow through PDOPMFC2,through the bypass valve PDOPPV2, through PDOPPV9,through PDOPMFC1, through PDOPPV4, through PDOPPV5and into the vent manifold at a rate determined by the current value of the voltage input to PDOPMFC1as determined either by the front panel potentiometer or the D/A converter output for this flow controller. The circuit pressure will be determined by the setting of PR1 and the pressure drop from the bubbler to the chamber will occur across PDOPMFC1. During manual operation from the front panel, the flow through PDOPMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to PDOPMFC1is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. With the switches in this configuration the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve PDOPPV2 is open.

The H_2 DILUTE switch becomes enabled when the ACTIVE switch is on. It may be turned at any time, however, in a normal growth sequence it would be turned on atter the ACTIVE switch is turned on and before the BUBBLER IN switch is turned on. When the H_2 DILUTE switch is turned on, PDOPPV6, and PDOPPV7 open, the positive closure signal on PDOPMFC3 is removed, and the +10 v control signal which had been keeping PDOPMFC2 fully open is removed. The flow rates through PDOPMFC1, PDOPMFC2, and PDOPMFC3 can now be controlled independently. Hydrogen may be supplied to the circuit through PDOPMFC2 and/or PDOPPV6 and may leave through PDOPMFC1 and/or PDOPMFC3. If the flow meters are set correctly, then the flow through PDOPMFC2 is less than the sum of the flows through PDOPMFC1 and PDOPMFC3. The pressure in the circuit will then drop until it is less than the pressure at the outlet of PR6. At this point the check valve between PDOPPV6and PR6 will open and the circuit pressure will be maintained at the pressure of PR6. The difference between the exit rate of gas through PDOPMFC1 and PDOPMFC3 and the entrance rate through PDOPMFC2 will be maintained by the flow through the check valve. The equivalent flow through valve PDOPPV5 is

$$\frac{Flow_2}{Flow_1 + Flow_3} Flow_1$$

There is nothing to prevent the operator from setting the mass flow controllers such that the flow setting for PDOPMFC2 is greater than the combined flow through PDOPMFC1 and PDOPMFC3. In this case the control valve for PDOPMFC2 would remain fully open and the flow through the circuit would be controlled by the settings on PDOPMFC1and PDOPMFC3. The pressure in the bubbler would remain at the outlet pressure of PR1 and flow through the check valve between PDOPPV6 and PR6 would not occur. Although no damage would occur from operating the system in this manner, there would be no advantage to doing it. In fact, the amount of alkyl flowing through PDOPMFC3would be wasted with no improvement in the accuracy in controlling the flow of a small amount of alkyl through PDOPMFC1.

When the BUBBLER IN switch is turned on, PDOPPV1and PDOPPV3open immediately and the green light, BUBBLER IN, turns on. After a two-second delay the red PURGE BUBBLER light goes out and PDOPPV2 closes. The delay on closure insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when PDOPPV1 and PDOPPV3open. When PDOPPV2closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve.

When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. Shutdown of the system should proceed by turning off, in sequence, the RUN switch, the BUBBLER IN switch, the H_2 DILUTE switch, and the ACTIVE switch.

The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through PDOPMFC1 andfor PDOPMFC3.

Operation of gaseous source without additional hydrogen dilution:

The valve on the top of the dimethylzinc cylinder should be opened and the downstream pressure of regulator PDOPPR1should be checked to see that it is 10 psig. With all switches closed, PDOPMFC1, PDOPMFC2, and PDOPMFC3 are commanded closed, PDOPPV1, PDOPPV2, PDOPPV3, PDOPPV4, PDOPPV6, PDOPPV7, PDOPPV8, PDOPPV9, PDOPPV10,and PDOPPV11 are closed and PDOPPV5 is in the vent position.

When the ACTIVE switch is turned on, the signals to command closure of PDOPMFC1 and PDOPMFC2 are removed, PDOPPV2, PDOPPV4,and PDOPPV9 open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control inputs of PDOPMFC2 and PDOPMFC4 which programs their flow setting to 200 sccm. This insures that the flow setting for PDOPMFC2 and PDOPMFC4 will always be larger than the flow setting for PDOPMFC1 and the circuit will not become evacuated; the control valves contained within PDOPMFC2 and PDOPMFC4 will remain wide open. With the switches in this configuration, the hydrogen carrier gas will flow through PDOPMFC2,through the bypass valve PDOPPV2, through PDOPPV9, through PDOPMFC1, through PDOPPV4, through PDOPPV5 and into the vent manifold at a rate determined by the current value of the voltage input to PDOPMFC1 as determined either by the front panel potentiometer or the D/A converter output for this flow controller. The circuit pressure will be determined by the setting of PR1 and the pressure drop from the bubbler to the chamber will occur across PDOPMFC1. During manual operation from the front panel, the flow through PDOPMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to PDOPMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. With the switches in this configuration the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve PDOPPV2 is open.

The GASEOUS SOURCE IN switch is disabled when the BUBBLER IN switch is on. When the GASEOUS SOURCE IN switch is turned on (with the BUBBLER IN switch off), PDOPPV9 closes, PDOPPV8and PDOPPV10 open and the signal to command closure is removed from PDOPMFC4. Since a +10 v control signal is applied to PDOPMFC4, its control valve will go to a wide open state. Dopant gas from the high-pressure cylinder will begin to flow through the circuit at a rate determined by the setting for PDOPMFC1. The pressure in the circuit will be determined by the pressure setting of PDOPPR1and the pressure drop will occur across PDOPMFC1.When the RUN switch is turned on, the flow at the outlet of PGPV5 is switched from the vent manifold to the run manifold and the yellow RUN light comes on. The system would be shut down by reversing the sequence of the above steps.

Operation of gaseous source with additional hydrogen dilution:

The valve on the top of the dimethylzinc cylinder should be opened and the downstream pressure of regulator PDOPPR1should be checked to see that it is 10 psig. With all switches closed, PDOPMFC1, PDOPMFC2, and
PDOPMFC3 are commanded closed, PDOPPV1, PDOPPV2, PDOPPV3, PDOPPV4, PDOPPV6, PDOPPV7, PDOPPV8, PDOPPV9, PDOPPV10,and PDOPPV11 are closed and PDOPPV5 is in the vent position.

When the ACTIVE switch is turned on, the signals to command closure of PDOPMFC1 and PDOPMFC2 are removed, PDOPPV2, PDOPPV4, and PDOPPV9 open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control inputs of PDOPMFC2 and PDOPMFC4 which programs their flow setting to 200 sccm. This insures that the flow setting for PDOPMFC2 and PDOPMFC4 will always be larger than the flow setting for PDOPMFC1 and the circuit will not become evacuated; the control valves contained within PDOPMFC2 and PDOPMFC4 will remain wide open. With the switches in this configuration, the hydrogen carrier gas will flow through PDOPMFC2,through the bypass valve PDOPPV2, through PDOPPV9, through PDOPMFC1,through PDOPPV4,through PDOPPV5and into the vent manifold at a rate determined by the current value of the voltage input to PDOPMFC1 as determined either by the front panel potentiometer or the D/A converter output for this flow controller. The circuit pressure will be determined by the setting of PR1 and the pressure drop from the bubbler to the chamber will occur across PDOPMFC1. During manual operation from the front panel, the flow through PDOPMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to PDOPMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. With the switches in this configuration the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valvePDOPPV2 is open.

The GASEOUS SOURCE IN switch is disabled when the BUBBLER IN switch is on. When the GASEOUS SOURCE IN switch is turned on (with the BUBBLER IN switch off), PDOPPV9 closes, PDOPPV8 and PDOPPV10 open and the signal to command closure is removed from PDOPMFC4. Since a +10 v control signal is applied to PDOPMFC4, its control valve will go to a wide open state. Dopant gas from the high-pressure cylinder will begin to flow through the circuit at a rate determined by the setting for PDOPOMFC1. The pressure in the circuit will be determined by the pressure setting of PDOPPR1and the pressure drop will occur across PDOPMFC1.

The H_2 DILUTE switch becomes enabled when the ACTIVE switch is on. It may be turned at any time, however, in a normal growth sequence it would be turned on after the ACTIVE and the GASEOUS SOURCE IN switches are turned on. When the H_2 DILUTE switch is turned on, PDOPPV6, and PDOPPV7open, the positive closure signal on PDOPMFC3 is removed, and the +10 v control signal which had been keeping PDOPMFC4fully open is removed. The flow rates through PDOPMFC1, PDOPMFC3,and PDOPMFC4 can now be controlled independently hydrogen may be supplied to the circuit through PDOPMFC4 and/or PDOPPV6 and may leave through PDOPMFC1 and/or PDOPMFC3. If the flow meters are set correctly, then the flow through PDOPMFC4 will be less than the sum of the flows through PDOPMFC1 and PDOPMFC3. The pressure in the circuit will then drop until it is less than the pressure at the outlet of PR6. At this point the check valve between PDOPPV6 and PR6 will open and the circuit pressure will be maintained at the pressure of PR6. The difference between the exit rate of gas through PDOPMFC1 and PDOPMFC3 and the entrance rate through PDOPMFC4 will be maintained by the flow through the check valve. The equivalent flow through valve PDOPPV5 is

$$\frac{Flow_4}{Flow_1 + Flow_3} Flow_1$$

There is nothing to prevent the operator from setting the mass flow controllers such that the flow setting for PDOPMFC4 is greater than the combined flow through PDOPMFC1 and PDOPMFC3. In this case the control valve for PDOPMFC4would remain fully open and the flow through the circuit would be controlled by the settings on PDOPMFC1and PDOPMFC3. The pressure in the bubbler would remain at the outlet pressure of PR1 and flow through the check valve between PDOPPV6 and PR6 would not occur. Although no damage would occur from operating the system in this manner, there would be no advantage to doing it. In fact, the amount of source gas flowing through PDOPMFC3would be wasted with no improvement in the accuracy of controlling the flow through PDOPMFC1.

When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. Shutdown of the system should proceed by turning off, in sequence, the RUN switch, the H_2 DILUTE switch, the GAS SOURCE IN switch, and the ACTIVE switch.

End of day:

The alkyl vapors will accelerate the deterioration of some of the elastomers from which the o-rings are fabricated. Elastomer seals have been eliminated whenever possible in the construction of this system but current technology does not allow them to be completely eliminated. They are present in the mass flow controller and in the vent/run valve. For this reason, if the bubbler source has been used, it is good practice to thoroughly flush the alkyl vapor from PDOPMFC1, PDOPMFC2, PDOPMFC3, and PDOPPV5 before the circuit is shut down. This can be accomplished by allowing hydrogen to flow through PDOPMFC1, PDOPMFC2,and PDOPMFC3 for approximately 10 minutes after the BUBBLER IN switch is closed. During the purging step the ACTIVE and H_2 PURGE switches must be on and there should be a gas flow of at least 10 sccm through PDOPMFC1 and 100 sccm through PDOPMFC2 and PDOPMFC3. After this, the ACTIVE switch can be closed, sealing off the circuit.

Interconnections

1. ACTIVE controls PDOPSV1which drives PDOPPV4.
2. BUBBLER IN controls PDOPSV2which drives PDOPPV1and PDOPPV3.
3. Relays PDOPK1 and PDOPK2control PDOPSV3 which drives PDOPPV2.
4. H_2 DILUTE controls PDOPSV4 which drives PDOPPV6and PDOPPV7
5. GAS SOURCE IN controls PDOPSV5 which drives PDOPPV8 and PDOPPV1O.
6. Relays PDOPK1 and PDOPK6 control PDOPSV7 which drives PDOPPV9.
7. H_2 PUSH controls PDOPSV6 which drives PDOPPV11.

Interlocks

1. All source valves are deactivated if the following conditions exist:
    1. System is in abort status.
    2. The vacuum exhaust valve, EXPV2, is closed.
    3. The deposition chamber is up to atmosphere.
    4. The manifold diluent gas is in the off position.
2. The BUBBLER IN, GAS SOURCE IN, RUN, and H_2 DILUTE switches are disabled if the ACTIVE switch is in the off position.

3. The BUBBLER IN switch is disabled if the GAS SOURCE IN switch is on. This implies that PDOPPV1 and PDOPPV3 can not be opened unless PDOPPV4 is open and PDOPPVa and PDOPPV10 are closed.
4. The GAS SOURCE IN switch is disabled if the BUBBLER IN switch is on. This implies that PDOPPV8 and PDOPPV10 can not be opened unless PDOPPV4 is open and PDOPPV1 and PDOPPV3 are closed.
5. With the PURGE switch on the front panel in the locked position (key removed), PDOPPV2 cannot be opened if PDOPPV1 and PDOPPV3are open.
6. The bypass valve (PDOPPV2) is open if the BUBBLER IN switch is closed (handle down) and the ACTIVE switch is on (handle up).
7. The ACTIVE, BUBBLER IN, GAS SOURCE IN, H_2 DILUTE, and RUN switches on the front panel are disabled when the computer switches the circuit into the REMOTE mode.
8. When the bubbler is connected to the circuit by opening the BUBBLER IN switch (handle up), valves PDOPPV1and PDOPPV3open immediately. Valve PDOPPV2 closes after a 2 second time delay to insure that the pressure on each side of the bubbler is equalized.
9. PDOPMFC1, PDOPMFC2, PDOPMFC3,and PDOPMFC4 are commanded closed if the ACTIVE switch is in the off position. The key lock switch CLOSE MFC2 when turned clockwise will close PDOPMFC2 under all conditions. The key lock switch CLOSE MFC4 when turned clockwise will close PDOPMFC4 under all conditions.
10. When the H_2 DILUTE switch is in the off position, PDOPMFC3 is commanded closed and the set points of PDOPMFC2 and PDOPMFC4 are at 200 sccm. The purpose of setting the PDOPMFC2 and PDOPMFC4 flows to 200 sccm is to insure that their flow settings are always greater that the flow setting for PDOPMFC1 when H_2 DILUTE is in the off position. This prevents the circuit from becoming evacuated. When the H_2 DILUTE switch in on, the closure signal is removed from PDOPMFC3 and the set point voltages for PDOPMFC2 and PDOPMFC4 are determined by either their front panel potentiometers or their remote D/A converters. A closure signal on a mass flow controller will over-ride the set point signal.
11. The manifold diluent gas cannot be turned off if the ACTIVE switch is on.
12. If the GAS SOURCE IN switch is on (handle up), then the H_2 FLUSH switch is disabled. This prevents PDOPPV11 from opening if PDOPV10 is open.
13. All front panel switches with the exception of the PURGE BUBBLER and H_2 FLUSH switches are disabled when the computer commands the circuit to the REMOTE mode.

Programming commands for remote operation

INSERT LABVIEW DOCUMENTATION HERE

Removing and replacing source bubbler

Purging of the bubbler lines must be done through the outlet route. The output can be evacuated with the vacuum pump through valves PDOPPV4and PDOPPV5. The limited conductance of the mass flow controller PDOPMFC1 requires that times of ≈1 minute be used to evacuate the lines to the bubbler. Purging is accomplished by alternately evacuating the lines and backfilling them with hydrogen. During the evacuation cycle it is necessary to open PDOPPV4 and keep PDOPMFC2 closed. For this purpose, a key lock switch, CLOSE MFC2, is installed on the front panel. When the key position is fully clockwise, PDOPMFC2will be closed regardless of the positions of the other switches.

Bubbler removal

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Close the manual valves on the inlet and outlet of the TMSn bubbler.

3. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
4. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the N_2 CARRIER position.
5. On the P-DOPANTcontrol panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for PDOPMFC1 to a flow of 100 sccm.
6. Evacuation: Insert a key in the CLOSE MFC2 switch and turn it clockwise. This will shut off the gas flow through PDOPMFC2. As the gas in the line is evacuated, the flow rate will drop. When the flow rate for PDOPMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated.
7. Backfill: Turn the key in the CLOSE MFC2 switch counter clockwise for 10 seconds. This will allow the bubbler circuit to fill with nitrogen.
8. Repeat steps 6 and 7 twenty times to completely purge the circuit of alkyl vapor. Finish with a backfill step.
9. Turn the BUBBLER IN switch and the ACTIVE switch to the off position.
10. Disconnect and remove the bubbler. CAUTION: This is a potentially hazardous step if the manual bubbler valves have failed to seal. The pyrophoric liquid will ignite when exposed to air. The quantities are usually small enough to easily contain the burning alkyl and the oxidation is limited by the diffusion rate of oxygen into the tubing, which is rather slow. Avoid breathing the oxide fumes. Have a dry chemical or non-flammable dry absorbent medium available to smother the chemical fire should it be necessary. In most cases probably the best remedy is to attach the stainless steel VCR caps to seal the inlet and outlet of the bubbler. DO NOT USE WATER!!
11. Install the stainless steel VCR caps on the bubbler ports. Use new gaskets unless the gaskets are of the self centering type.
12. If a new bubbler is being installed immediately, proceed to the next section. Otherwise, shut down the system as appropriate.

Bubbler installation

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Insure that one of the hydrogen cylinders is actively connected to the hydrogen supply line, that the pressure regulator is properly set and that the hydrogen purifier is turned on and up to temperature.
3. Insure that PR6 is set at 10 psig and PR1 is set at 15 psig.
4. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
5. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the H_2 CARRIER position.

6. Insure that the manual valves on the bubbler are closed. Uncap the inlet and outlet ports on the bubbler and connect the bubbler to the circuit using new gaskets if the previously used gaskets were not of the self-centering type.
7. On the P-DOPANT control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for PDOPMFC1 to a flow of 100 sccm.
8. Evacuation: Insert a key in the CLOSE MFC2 switch and turn it clockwise. This will shut off the gas flow through PDOPMFC2. As the gas in the line is evacuated, the flow rate will drop. When the flow rate for PDOPMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated.
9. Backfill: Turn the key in the CLOSE MFC2 switch counter clockwise for 10 seconds. This will allow the bubbler circuit to fill with hydrogen.
10. Repeat steps 8 and 9 twenty times to completely purge the circuit of air. Finish with a backfill step. Remove the key to the CLOSE MFC2 switch.
11. Open the manual valve on the outlet of the bubbler first. If the pressure in the bubbler should happen to be greater than 10 psig (the setting of PR6) and the inlet valve were opened first, the liquid alkyl would be forced back into the inlet plumbing.
12. Open the manual valve on the inlet of the bubbler.
13. Turn the BUBBLER IN switch and the ACTIVE switch to the off position. Turn the PURGE key lock switch off (counter-clockwise) and remove the key.
14. Shut down the system as appropriate.

Purging of DMZ Lines

Purging must be achieved through the mass flow controller to the vacuum pump. Use alternating Evacuation/backfill cycles to remove reactive gases. Due to the limited conductance of the gas lines allow cycle times of a few minutes. The regulator inlet lines can be independently purged for changing cylinders;

Purge of regulator and low-pressure delivery lines:

This operation need be performed only to purge the line downstream of the regulator; purge of the high-pressure lines is described in the next section.

1. Close the high-pressure inlet valve, PDOPMV2to the regulator.
2. With the vacuum pump running, open the VAC EXHAUSTswitch on the VACUUM CONTROL panel and set the N_2 manifold flow controller to maintain a minimum deposition chamber pressure of 1-2 Torr.
3. Evacuation: On the N DOPANT control panel open the ACTIVE switch, the GAS SOURCE IN switch, and the RUN switch. Adjust PDOPMFC1for maximum flow. Insert a key and turn the key lock PURGE MFC4 switch clockwise. Allow several minutes for the line to evacuate.
4. Backfill: Turn the GAS SOURCE IN switch off. Turn the H_2 DILUTE switch on. Insert a key in the key lock H_2 FLUSH switch and turn it clockwise for at least 10 seconds to backfill the line with hydrogen. Turn the H_2 FLUSH switch counterclockwise and turn the H_2 DILUTE switch off. Turn the GAS SOURCE IN switch on.
5. Repeat the evacuate cycle allowing several minutes for the line to evacuate.
6. Repeat the backfill/evacuate cycle a total of five times. After the last evacuate cycle, set PDOPMFC1 to zero flow, turn the PURGE MFC4 switch to the off position and remove the key. Turn the H_2 FLUSH switch to the off position and remove the key.
7. Backfill with DMZ:
    1. Back-off the knob on pressure regulator PDOPPR1 (fully counterclockwise)

2. Open the high-pressure regulator inlet valve PDOPMV2.
3. Set the knob on pressure regulator PDOPPR1to 10 psig at the outlet. Check the excess-flow valve PDOPEFV1 to see that it has not tripped by pushing in on the red button and observing the gauge on the outlet of PDOPPR1 for any changes in pressure.
4. Close the RUN SWITCH, the GAS SOURCE IN switch and the ACTIVE switch.

Cylinder Replacement

Cylinder replacement requires purging of only the high-pressure inlet line to the regulator. The high-purity, low-pressure delivery lines are not exposed to air during the replacement operation.

8. Cylinder removal:
    1. Tightly close the DMZ cylinder valve and prepare to remove gas from the inlet line.
    2. Flow DMZ through the reactor to reduce the inlet pressure to ""0 psig.
    3. Close the regulator inlet valve PDOPMV2.
    4. Lock the excess flow valve PDOPEFV1 in the open position with tape.
    5. Purge the regulator inlet: Turn on the nitrogen supply valve, NMV3, to the vacuum educator and open the DMZ regulator vent valve, PDOPMV4. (DANGER: Be sure the DMZ cylinder valve is closed.) After 5 seconds close PDOPMV4 and NMV3.
    6. Backfill the regulator inlet: First insure the tank valve for PDOPPURGEPR1 is open, that the outlet of regulator PDOPPURGEPR1 is at least 15 psig, and that PDOPPURGEMV1 is fully open (clockwise). Backfill the regulator input with nitrogen by opening PDOPMV3 for at least 2 seconds. Close PDOPMV3.
    7. Repeat the purge-backfill cycle of steps e and f twenty times. Finish with a backfill step, close PDOPMV3 and PDOPMV4,and disconnect and cap the cylinder. The handheld Matheson toxic gas monitor should be used when unscrewing the cylinder connection. The CGA 350 connection on the DMZ cylinder has a left-hand thread. If you are facing the cylinder, the nut on the excess flow valve must be turned clockwise to loosen it. It is very important that the brass cap for the cylinder valve be replaced. The cylinder valves sometimes have small leaks even when tightly closed.
9. Cylinder installation:
    1. With the new DMZ cylinder sitting inside the MOCVDcabinet and the exhaust fan on, slightly loosen the brass cap which is plugging the end of the CGA 350 connection on the cylinder valve. The cap has a left-hand thread. Check for the presence of DMZ using the portable Matheson toxic gas detector. If a high level release of DMZ is detected, the cap on the valve should be retightened and the cylinder returned to the vendor. If a low level release is detected, it may be due to a small amount of arsine trapped between the closed valve on the cylinder and the brass cap. With the brass cap left slightly unscrewed close the door to the MOCVD cabinet and let the tank set for several hours. If no DMZ is detected at that point, it is safe to completely unscrew the cap and install the cylinder. Otherwise the cap should be re-tightened and the tank returned to the vendor for replacement.
    2. Connect the excess flow valve, PDOPEFV1,to the cylinder. Keep the regulator inlet valve, PDOPMV2, closed as well as the nitrogen backfill valve PDOPMV3. Hold the excess flow valve, PDOPEFV1, open by temporarily holding in the red button with a piece of tape.
    3. Purge the regulator input: Turn on the nitrogen flow for the vacuum eductor, by opening NMV4. Open the purge valve, PDOPMV4, wait 5 seconds and then close PDOPMV4 and NMV4.
    4. Backfill the regulator input: First insure the tank valve for DOPPURGEPR1 is open, that the outlet of regulator DOPPURGEPR1 is at least 15 psig, and that DOPPURGEMV1 is fully open (clockwise). Backfill the regulator input with nitrogen by opening PDOPMV3 for at least 2 seconds. Close PDOPMV3.

5. Repeat the backfill-purge cycle 20 times finishing with a purge step. Insure that PDOPMV3, PDOPMV4, and MV3 are closed.
6. Remove the tape from the excess flow valve, PDOPEFV1.
7. Open the manual valve on the tank and check for leaks.
8. Reset the excess flow valve, PDOPEFV1, by pushing in the red button and check for leaks.
9. Open the manual valve on the regulator input, PDOPMV2and check for leaks.
10. Close the tank valve on the nitrogen cylinder supplying gas to regulator DOPPURGEPR1.

N-Dopant

Gas Distribution

1. This system allows use of either a gaseous or liquid source for layer doping. The gaseous source, a mixture of 5% dimethylzinc (DMZ) in argon at 10 psig output pressure of the gas cylinder regulator, is fed to a mass flow controller supplying this gas to the reactor vent/run manifold. The pressure differential between the source and the reactor chamber is accommodated across the flow controller PDOPMFC1. Alternatively, the DMZ can be valved off and hydrogen passed through the flow controller to purge the dimethylzinc.
2. A purge-mode switchable from the front panel allows evacuation and backfill of the delivery lines with H_2 purge gas. Valving at the regulator allows purging of the cylinder lines for changing the cylinder.
3. The liquid source is tetramethyltin (TMSn) which is a liquid at room temperature with a vapor pressure of 10 Torr at -200C. This means that the pressure of the vapor is 10/760 x 14.7 psia; about 1/100 atmospheric pressure. The vapor is transported from the stainless steel canister which contains the liquid, into the reaction chamber by bubbling hydrogen gas through the liquid.
4. The P-dopant plumbing system is a more complex arrangement which allows the gas streams from either the high pressure or liquid source to be diluted with additional hydrogen carrier gas. In addition, a portion of the total volume of gas may be dumped into the scrubber before it enters the vent/run valve. This arrangement permits a wider range of dopant flow rates to be directed into the deposition chamber. The pressure of the hydrogen gas inside the liquid container is determined by the pressure setting of either PR1 or PR6 depending on the mode of operation. The proper functioning of the various components is discussed in detail in the section on operating procedures.
5. The precise vapor pressure of the TMSn is determined by the temperature of the liquid. This is controlled by immersing the liquid in a coolant bath which is controlled at a precise temperature by a small Brinkmann refrigeration unit.
6. Pressure drop to the reaction chamber occurs across the mass flow controller, PDOPMFC1.
7. The alkyl is exposed to the heated sensor element of the mass flow controller (≈110°C); thus it must be stable at this temperature.
8. Loss of liquid from the bubbler into the gas lines is unlikely because pressure in the gas line is the same as the bubbler. (Closure of the bypass valve, PDOPPV2, is delayed to allow bubbler inlet and outlet pressure to equilibrate.)
9. Upon power failure or other abort conditions, contamination of the upstream H_2lines is prevented by closure of the control valve for mass flow controller PDOPMFC2.
10. Adiabatic cooling could possibly cause alkyl condensation in the mass flow controller valve. It is advisable to keep the alkyl temperature below room temperature.
11. A purge mode overrides some interlocks to allow evacuation and backfill of the bubbler lines.

Hazards

1. TMSn is pyrophoric.
2. The oxides formed from the burning of TMSn are toxic.
3. TMSn reacts violently with water which should never be used to extinguish the burning alkyl. Use a dry chemical fire extinguisher only or else let it burn.
4. The dust (silicon dioxide) formed when dimethylzinc reacts with air is toxic.
5. Release of high pressure dimethylzinc can occur by operator carelessness.

Precautions

1. Source volume limit: Safety dictated use of a limited volume of DMZ as a means of controlling a catastrophic failure. We have limited ourselves to a 26 cu. ft. cylinder of 5% DMZ in argon.
2. Ventilation: The DMZ cylinder is in an enclosed, ventilated MOCVD reactor cabinet.
3. Toxic gas monitoring:
    1. The DOD ChemLogic CL8toxic gas monitor samples the MOCVDcabinet atmosphere. An alarm will activate at the AsH_3 TLV and shut down the system. The more sensitive limit for arsine provides extra margin for dimethylzinc detection.
    2. The DOD ChemLogic CL8 monitor also samples the immediate laboratory air. An alarm will activate at the AsH_3 TLV and shut down the system. At the TLV the building Evacuation alarm will sound. (In case of alarm -leave the area and assist others in leaving the building.)
    3. The DOD ChemLogic CL8 monitors the outlet of the scrubber. If the gas concentration exceeds the arsine TLV threshold, the sources automatically shut down and an alarm on the operator panel sounds.
4. System operation features:
    1. Interlocks prevent the use of DMZ except under normal operation of the sealed gas system.
    2. Opening the chamber for loading and unloading samples disables the DMZ'
    3. Removal of the DMZ cylinder requires purging of the gas regulator inlet line to remove the DMZ prior to disconnecting.
    4. Replacement of DMZ cylinder requires purging of the gas regulator inlet line to remove air contamination.
    5. Gas cylinder change should be done with an observer to double-check procedures and assist in an emergency.
5. The gaseous source is a mixture of 5% dimethylzinc (DMZ) in argon. At this concentration, the gas is labeled flammable (not pyrophoric). Dimethylzinc is especially reactive with air, forming silicon dioxide (Si02) The silicon dioxide forms as a fine white dust which can deposit in the plumbing and valves if air and dimethylzinc are allowed to mix. The dust will prevent valve seats from forming a complete seal and can cause the pressure control valve inside the gas regulator to fail. When this happens the pressure downstream of the gas regulator will creep upward; if left without an excape route the downstream pressure could presumably rise to the full value of the pressure in the source cylinder (=2000 psi). For this reason a relief valve (PDOPPRV1) on the downstream side of the regulator has been installed to vent the gas to the scrubber if the line pressure exceeds 150 psi. This will prevent components from bursting if the regulator should fail. It is therefore extremely important to purge all the air from the components before installing a dimethylzinc cylinder and, likewise, very important to purge all the dimethylzinc from the lines before removing a dimethylzinc cylinder. If this is not done carefully, some of the gas control components could be ruined and others might require a time consuming cleanup process. An excess flow valve (PDOPEFV1) is installed on the dimethylzinc tank. If the gas flow through the valve exceeds 1000 sccm, the valve will trip shutting off the flow. It then stays closed unless it is manually reset by pushing in on the red button. The next valve after the excess flow valve is a pneumatically controlled valve (PDOPPV20) held open by the abort interlock system. This valve automatically closes in the event of power failure, loss of ventilation, operator intervention, fire, low hydrogen pressure, low nitrogen pressure, or detection of toxic gas in the cabinet, room, or at the scrubber outlet.
6. Keep the manual valves on a newly installed bubbler closed until it is connected to the system, leak checked and purged.
7. Avoid gas pressure differentials that can force the alkyl from the bubbler back into the carrier gas piping.

Normal Operation

The P-dopant circuit can provide source material from either the bubbler or the high-pressure cylinder. These flows may be operated with or without additional hydrogen dilution. Each mode of operation will be described in a separate section.

Start-of-day:

Since the pneumatic valves PDOPPV1 and PDOPPV2 seal off the bubbler from the rest of the system, the manual valves on the bubbler inlet and outlet can be left open except when the bubbler is being replaced or removed. At the start-of-day the TMSn is therefore ready to operate from the control panel.

The regulator and delivery line contain dimethylzinc and are not purged during overnight shutdown. Since the cylinder valve is closed at days end, the volume of toxic gas left in the lines represents a small risk in case of line rupture. The alternative of purging the line each day would waste a significant fraction of the small cylinder volume and would add to the load of dimethylzinc to be scrubbed from the exhaust gas.

Operation of bubbler source without additional hydrogen dilution:

With all switches closed, PDOPMFC1, PDOPMFC2, PDOPMFC3,and PDOPMFC4 are commanded closed, PDOPPV1, PDOPPV2, PDOPPV3, PDOPPV4, PDOPPV6, PDOPPV7, PDOPPV8, PDOPPV9, PDOPPV10, and PDOPPV11 are closed and PDOPPV5 is in the vent position. When the ACTIVE switch is turned on, the signals to command closure of PDOPMFC1 and PDOPMFC2 are removed, PDOPPV2, PDOPPV4, and PDOPPV9open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control inputs of PDOPMFC2 and DOPMFC4 which programs their flow settings to 200 sccm. This insures that the flow setting through PDOPMFC2 or PDOPMFC4 will always be larger than the flow through PDOPMFC1and the circuit will not become evacuated; the control valves contained within PDOPMFC2 and PDOPMFC4 will remain wide open. With the switches in this configuration, the hydrogen carrier gas will flow through PDOPMFC2,through the bypass valve PDOPPV2, through PDOPPV9, through PDOPMFC1,through PDOPPV4, through PDOPPV5and into the vent manifold at a rate determined by the current value of the voltage input to PDOPMFC1as determined either by the front panel potentiometer or the D/A converter output for this flow controllers. The circuit pressure will be determined by the setting of PR1 and the pressure drop from the circuit to the chamber will occur across PDOPMFC1. During manual operation from the front panel, the flow through PDOPMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to PDOPMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter.

With the switches in the present configuration, the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve PDOPPV2 is open. When the BUBBLER IN switch is turned on, PDOPPV1 and PDOPPV3open immediately and the green light, BUBBLER IN, turns on. After a two-second delay the red PURGE BUBBLER light goes out and PDOPPV2 closes. This insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when PDOPPV1and PDOPPV3open. When PDOPPV2closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve. When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. Shutdown of the system should proceed by reversing the sequence of the above steps.

The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through PDOPMFC1.

Operation of bubbler source with additional hydrogen dilution:

With all switches closed, PDOPMFC1, PDOPMFC2, and PDOPMFC3 are commanded closed, PDOPPV1, PDOPPV2, PDOPPV3, PDOPPV4, PDOPPV6, PDOPPV7, PDOPPV8, PDOPPV9, PDOPPV10, and PDOPPV11 are closed and PDOPPV5 is in the vent position. When the ACTIVE switch is turned on, the signals to command closure of PDOPMFC1 and PDOPMFC2 are removed, PDOPPV2, PDOPPV4, and PDOPPV9 open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control inputs of PDOPMFC2 and PDOPMFC4 which programs their flow settings to 200 sccm. This insures that the flow setting for PDOPMFC2 will always be larger than the flow setting for PDOPMFC1 and the circuit will not become evacuated; the control valves contained within PDOPMFC2 and PDOPMFC4 will remain wide open.

With the switches in this configuration, the hydrogen carrier gas will flow through PDOPMFC2,through the bypass valve PDOPPV2, through PDOPPV9,through PDOPMFC1, through PDOPPV4, through PDOPPV5and into the vent manifold at a rate determined by the current value of the voltage input to PDOPMFC1as determined either by the front panel potentiometer or the D/A converter output for this flow controller. The circuit pressure will be determined by the setting of PR1 and the pressure drop from the bubbler to the chamber will occur across PDOPMFC1. During manual operation from the front panel, the flow through PDOPMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to PDOPMFC1is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. With the switches in this configuration the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve PDOPPV2 is open.

The H_2 DILUTE switch becomes enabled when the ACTIVE switch is on. It may be turned at any time, however, in a normal growth sequence it would be turned on atter the ACTIVE switch is turned on and before the BUBBLER IN switch is turned on. When the H_2 DILUTE switch is turned on, PDOPPV6, and PDOPPV7 open, the positive closure signal on PDOPMFC3 is removed, and the +10 v control signal which had been keeping PDOPMFC2 fully open is removed. The flow rates through PDOPMFC1, PDOPMFC2, and PDOPMFC3 can now be controlled independently. Hydrogen may be supplied to the circuit through PDOPMFC2 and/or PDOPPV6 and may leave through PDOPMFC1 and/or PDOPMFC3. If the flow meters are set correctly, then the flow through PDOPMFC2 is less than the sum of the flows through PDOPMFC1 and PDOPMFC3. The pressure in the circuit will then drop until it is less than the pressure at the outlet of PR6. At this point the check valve between PDOPPV6and PR6 will open and the circuit pressure will be maintained at the pressure of PR6. The difference between the exit rate of gas through PDOPMFC1 and PDOPMFC3 and the entrance rate through PDOPMFC2 will be maintained by the flow through the check valve. The equivalent flow through valve PDOPPV5 is

$$\frac{Flow_2}{Flow_1 + Flow_3} Flow_1$$

There is nothing to prevent the operator from setting the mass flow controllers such that the flow setting for PDOPMFC2 is greater than the combined flow through PDOPMFC1 and PDOPMFC3. In this case the control valve for PDOPMFC2 would remain fully open and the flow through the circuit would be controlled by the settings on PDOPMFC1and PDOPMFC3. The pressure in the bubbler would remain at the outlet pressure of PR1 and flow through the check valve between PDOPPV6 and PR6 would not occur. Although no damage would occur from operating the system in this manner, there would be no advantage to doing it. In fact, the amount of alkyl flowing through PDOPMFC3would be wasted with no improvement in the accuracy in controlling the flow of a small amount of alkyl through PDOPMFC1.

When the BUBBLER IN switch is turned on, PDOPPV1and PDOPPV3open immediately and the green light, BUBBLER IN, turns on. After a two-second delay the red PURGE BUBBLER light goes out and PDOPPV2 closes. The delay on closure insures that the downstream pressure is not greater than the upstream pressure (a condition which would force the liquid alkyl back into the hydrogen carrier gas piping) when PDOPPV1 and PDOPPV3open. When PDOPPV2closes, the hydrogen carrier gas is no longer allowed to bypass the bubbler, and instead must flow through the bubbler transporting the alkyl vapor to the vent/run valve.

When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. Shutdown of the system should proceed by turning off, in sequence, the RUN switch, the BUBBLER IN switch, the H_2 DILUTE switch, and the ACTIVE switch.

The BUBBLER IN switch should be turned off during standby periods to both conserve the source and to minimize the load of hazardous chemical which enters the scrubber. The alkyl vapor will continue to be transported from the bubbler as long as the BUBBLER IN switch is on and there is gas flow through PDOPMFC1 andfor PDOPMFC3.

Operation of gaseous source without additional hydrogen dilution:

The valve on the top of the dimethylzinc cylinder should be opened and the downstream pressure of regulator PDOPPR1should be checked to see that it is 10 psig. With all switches closed, PDOPMFC1, PDOPMFC2, and PDOPMFC3 are commanded closed, PDOPPV1, PDOPPV2, PDOPPV3, PDOPPV4, PDOPPV6, PDOPPV7, PDOPPV8, PDOPPV9, PDOPPV10,and PDOPPV11 are closed and PDOPPV5 is in the vent position.

When the ACTIVE switch is turned on, the signals to command closure of PDOPMFC1 and PDOPMFC2 are removed, PDOPPV2, PDOPPV4,and PDOPPV9 open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control inputs of PDOPMFC2 and PDOPMFC4 which programs their flow setting to 200 sccm. This insures that the flow setting for PDOPMFC2 and PDOPMFC4 will always be larger than the flow setting for PDOPMFC1 and the circuit will not become evacuated; the control valves contained within PDOPMFC2 and PDOPMFC4 will remain wide open. With the switches in this configuration, the hydrogen carrier gas will flow through PDOPMFC2,through the bypass valve PDOPPV2, through PDOPPV9, through PDOPMFC1, through PDOPPV4, through PDOPPV5 and into the vent manifold at a rate determined by the current value of the voltage input to PDOPMFC1 as determined either by the front panel potentiometer or the D/A converter output for this flow controller. The circuit pressure will be determined by the setting of PR1 and the pressure drop from the bubbler to the chamber will occur across PDOPMFC1. During manual operation from the front panel, the flow through PDOPMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to PDOPMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. With the switches in this configuration the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valve PDOPPV2 is open.

The GASEOUS SOURCE IN switch is disabled when the BUBBLER IN switch is on. When the GASEOUS SOURCE IN switch is turned on (with the BUBBLER IN switch off), PDOPPV9 closes, PDOPPV8and PDOPPV10 open and the signal to command closure is removed from PDOPMFC4. Since a +10 v control signal is applied to PDOPMFC4, its control valve will go to a wide open state. Dopant gas from the high-pressure cylinder will begin to flow through the circuit at a rate determined by the setting for PDOPMFC1. The pressure in the circuit will be determined by the pressure setting of PDOPPR1and the pressure drop will occur across PDOPMFC1.When the RUN switch is turned on, the flow at the outlet of PGPV5 is switched from the vent manifold to the run manifold and the yellow RUN light comes on. The system would be shut down by reversing the sequence of the above steps.

Operation of gaseous source with additional hydrogen dilution:

The valve on the top of the dimethylzinc cylinder should be opened and the downstream pressure of regulator PDOPPR1should be checked to see that it is 10 psig. With all switches closed, PDOPMFC1, PDOPMFC2, and
PDOPMFC3 are commanded closed, PDOPPV1, PDOPPV2, PDOPPV3, PDOPPV4, PDOPPV6, PDOPPV7, PDOPPV8, PDOPPV9, PDOPPV10,and PDOPPV11 are closed and PDOPPV5 is in the vent position.

When the ACTIVE switch is turned on, the signals to command closure of PDOPMFC1 and PDOPMFC2 are removed, PDOPPV2, PDOPPV4, and PDOPPV9 open and the green ACTIVE light comes on. A voltage equal to +10 v is applied to the control inputs of PDOPMFC2 and PDOPMFC4 which programs their flow setting to 200 sccm. This insures that the flow setting for PDOPMFC2 and PDOPMFC4 will always be larger than the flow setting for PDOPMFC1 and the circuit will not become evacuated; the control valves contained within PDOPMFC2 and PDOPMFC4 will remain wide open. With the switches in this configuration, the hydrogen carrier gas will flow through PDOPMFC2,through the bypass valve PDOPPV2, through PDOPPV9, through PDOPMFC1,through PDOPPV4,through PDOPPV5and into the vent manifold at a rate determined by the current value of the voltage input to PDOPMFC1 as determined either by the front panel potentiometer or the D/A converter output for this flow controller. The circuit pressure will be determined by the setting of PR1 and the pressure drop from the bubbler to the chamber will occur across PDOPMFC1. During manual operation from the front panel, the flow through PDOPMFC1 is controlled by the setting of the 10-turn FLOW SET 1 potentiometer on the control panel. The value of the flow setting can be read on the digital panel meter by pushing in on the black FLOW SET 1 button. When the button is released, the meter reads the value of the actual flow. When the gas circuit is operated remotely from the computer, the input voltage to PDOPMFC1 is supplied by a D/A converter in the HP Multiprogrammer and can be remotely programmed. The meter on the panel will continue to indicate the actual flow, and when the FLOW SET 1 button is pushed in, the meter will read the input voltage to the flow controller from the D/A converter. With the switches in this configuration the red PURGE BUBBLER light on the control panel will be on indicating that the bypass valvePDOPPV2 is open.

The GASEOUS SOURCE IN switch is disabled when the BUBBLER IN switch is on. When the GASEOUS SOURCE IN switch is turned on (with the BUBBLER IN switch off), PDOPPV9 closes, PDOPPV8 and PDOPPV10 open and the signal to command closure is removed from PDOPMFC4. Since a +10 v control signal is applied to PDOPMFC4, its control valve will go to a wide open state. Dopant gas from the high-pressure cylinder will begin to flow through the circuit at a rate determined by the setting for PDOPOMFC1. The pressure in the circuit will be determined by the pressure setting of PDOPPR1and the pressure drop will occur across PDOPMFC1.

The H_2 DILUTE switch becomes enabled when the ACTIVE switch is on. It may be turned at any time, however, in a normal growth sequence it would be turned on after the ACTIVE and the GASEOUS SOURCE IN switches are turned on. When the H_2 DILUTE switch is turned on, PDOPPV6, and PDOPPV7open, the positive closure signal on PDOPMFC3 is removed, and the +10 v control signal which had been keeping PDOPMFC4fully open is removed. The flow rates through PDOPMFC1, PDOPMFC3,and PDOPMFC4 can now be controlled independently hydrogen may be supplied to the circuit through PDOPMFC4 and/or PDOPPV6 and may leave through PDOPMFC1 and/or PDOPMFC3. If the flow meters are set correctly, then the flow through PDOPMFC4 will be less than the sum of the flows through PDOPMFC1 and PDOPMFC3. The pressure in the circuit will then drop until it is less than the pressure at the outlet of PR6. At this point the check valve between PDOPPV6 and PR6 will open and the circuit pressure will be maintained at the pressure of PR6. The difference between the exit rate of gas through PDOPMFC1 and PDOPMFC3 and the entrance rate through PDOPMFC4 will be maintained by the flow through the check valve. The equivalent flow through valve PDOPPV5 is

$$\frac{Flow_4}{Flow_1 + Flow_3} Flow_1$$

There is nothing to prevent the operator from setting the mass flow controllers such that the flow setting for PDOPMFC4 is greater than the combined flow through PDOPMFC1 and PDOPMFC3. In this case the control valve for PDOPMFC4would remain fully open and the flow through the circuit would be controlled by the settings on PDOPMFC1and PDOPMFC3. The pressure in the bubbler would remain at the outlet pressure of PR1 and flow through the check valve between PDOPPV6 and PR6 would not occur. Although no damage would occur from operating the system in this manner, there would be no advantage to doing it. In fact, the amount of source gas flowing through PDOPMFC3would be wasted with no improvement in the accuracy of controlling the flow through PDOPMFC1.

When the RUN switch is turned on, the yellow RUN light comes on and the gas flow is switched from the vent manifold to the run manifold and must now pass through the deposition chamber. Shutdown of the system should proceed by turning off, in sequence, the RUN switch, the H_2 DILUTE switch, the GAS SOURCE IN switch, and the ACTIVE switch.

End of day:

The alkyl vapors will accelerate the deterioration of some of the elastomers from which the o-rings are fabricated. Elastomer seals have been eliminated whenever possible in the construction of this system but current technology does not allow them to be completely eliminated. They are present in the mass flow controller and in the vent/run valve. For this reason, if the bubbler source has been used, it is good practice to thoroughly flush the alkyl vapor from PDOPMFC1, PDOPMFC2, PDOPMFC3, and PDOPPV5 before the circuit is shut down. This can be accomplished by allowing hydrogen to flow through PDOPMFC1, PDOPMFC2,and PDOPMFC3 for approximately 10 minutes after the BUBBLER IN switch is closed. During the purging step the ACTIVE and H_2 PURGE switches must be on and there should be a gas flow of at least 10 sccm through PDOPMFC1 and 100 sccm through PDOPMFC2 and PDOPMFC3. After this, the ACTIVE switch can be closed, sealing off the circuit.

Interconnections

1. ACTIVE controls PDOPSV1which drives PDOPPV4.
2. BUBBLER IN controls PDOPSV2which drives PDOPPV1and PDOPPV3.
3. Relays PDOPK1 and PDOPK2control PDOPSV3 which drives PDOPPV2.
4. H_2 DILUTE controls PDOPSV4 which drives PDOPPV6and PDOPPV7
5. GAS SOURCE IN controls PDOPSV5 which drives PDOPPV8 and PDOPPV1O.
6. Relays PDOPK1 and PDOPK6 control PDOPSV7 which drives PDOPPV9.
7. H_2 PUSH controls PDOPSV6 which drives PDOPPV11.

Interlocks

1. All source valves are deactivated if the following conditions exist:
    1. System is in abort status.
    2. The vacuum exhaust valve, EXPV2, is closed.
    3. The deposition chamber is up to atmosphere.
    4. The manifold diluent gas is in the off position.
2. The BUBBLER IN, GAS SOURCE IN, RUN, and H_2 DILUTE switches are disabled if the ACTIVE switch is in the off position.

3. The BUBBLER IN switch is disabled if the GAS SOURCE IN switch is on. This implies that PDOPPV1 and PDOPPV3 can not be opened unless PDOPPV4 is open and PDOPPVa and PDOPPV10 are closed.
4. The GAS SOURCE IN switch is disabled if the BUBBLER IN switch is on. This implies that PDOPPV8 and PDOPPV10 can not be opened unless PDOPPV4 is open and PDOPPV1 and PDOPPV3 are closed.
5. With the PURGE switch on the front panel in the locked position (key removed), PDOPPV2 cannot be opened if PDOPPV1 and PDOPPV3are open.
6. The bypass valve (PDOPPV2) is open if the BUBBLER IN switch is closed (handle down) and the ACTIVE switch is on (handle up).
7. The ACTIVE, BUBBLER IN, GAS SOURCE IN, H_2 DILUTE, and RUN switches on the front panel are disabled when the computer switches the circuit into the REMOTE mode.
8. When the bubbler is connected to the circuit by opening the BUBBLER IN switch (handle up), valves PDOPPV1and PDOPPV3open immediately. Valve PDOPPV2 closes after a 2 second time delay to insure that the pressure on each side of the bubbler is equalized.
9. PDOPMFC1, PDOPMFC2, PDOPMFC3,and PDOPMFC4 are commanded closed if the ACTIVE switch is in the off position. The key lock switch CLOSE MFC2 when turned clockwise will close PDOPMFC2 under all conditions. The key lock switch CLOSE MFC4 when turned clockwise will close PDOPMFC4 under all conditions.
10. When the H_2 DILUTE switch is in the off position, PDOPMFC3 is commanded closed and the set points of PDOPMFC2 and PDOPMFC4 are at 200 sccm. The purpose of setting the PDOPMFC2 and PDOPMFC4 flows to 200 sccm is to insure that their flow settings are always greater that the flow setting for PDOPMFC1 when H_2 DILUTE is in the off position. This prevents the circuit from becoming evacuated. When the H_2 DILUTE switch in on, the closure signal is removed from PDOPMFC3 and the set point voltages for PDOPMFC2 and PDOPMFC4 are determined by either their front panel potentiometers or their remote D/A converters. A closure signal on a mass flow controller will over-ride the set point signal.
11. The manifold diluent gas cannot be turned off if the ACTIVE switch is on.
12. If the GAS SOURCE IN switch is on (handle up), then the H_2 FLUSH switch is disabled. This prevents PDOPPV11 from opening if PDOPV10 is open.
13. All front panel switches with the exception of the PURGE BUBBLER and H_2 FLUSH switches are disabled when the computer commands the circuit to the REMOTE mode.

Programming commands for remote operation

INSERT LABVIEW DOCUMENTATION HERE

Removing and replacing source bubbler

Purging of the bubbler lines must be done through the outlet route. The output can be evacuated with the vacuum pump through valves PDOPPV4and PDOPPV5. The limited conductance of the mass flow controller PDOPMFC1 requires that times of ≈1 minute be used to evacuate the lines to the bubbler. Purging is accomplished by alternately evacuating the lines and backfilling them with hydrogen. During the evacuation cycle it is necessary to open PDOPPV4 and keep PDOPMFC2 closed. For this purpose, a key lock switch, CLOSE MFC2, is installed on the front panel. When the key position is fully clockwise, PDOPMFC2will be closed regardless of the positions of the other switches.

Bubbler removal

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Close the manual valves on the inlet and outlet of the TMSn bubbler.

3. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
4. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the N_2 CARRIER position.
5. On the P-DOPANTcontrol panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for PDOPMFC1 to a flow of 100 sccm.
6. <u>Evacuation</u>: Insert a key in the CLOSE MFC2 switch and turn it clockwise. This will shut off the gas flow through PDOPMFC2. As the gas in the line is evacuated, the flow rate will drop. When the flow rate for PDOPMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated.
7. <u>Backfill</u>: Turn the key in the CLOSE MFC2 switch counter clockwise for 10 seconds. This will allow the bubbler circuit to fill with nitrogen.
8. Repeat steps 6 and 7 twenty times to completely purge the circuit of alkyl vapor. Finish with a backfill step.
9. Turn the BUBBLER IN switch and the ACTIVE switch to the off position.
10. Disconnect and remove the bubbler. CAUTION: This is a potentially hazardous step if the manual bubbler valves have failed to seal. The pyrophoric liquid will ignite when exposed to air. The quantities are usually small enough to easily contain the burning alkyl and the oxidation is limited by the diffusion rate of oxygen into the tubing, which is rather slow. Avoid breathing the oxide fumes. Have a dry chemical or non-flammable dry absorbent medium available to smother the chemical fire should it be necessary. In most cases probably the best remedy is to attach the stainless steel VCR caps to seal the inlet and outlet of the bubbler. DO NOT USE WATER!!
11. Install the stainless steel VCR caps on the bubbler ports. Use new gaskets unless the gaskets are of the self centering type.
12. If a new bubbler is being installed immediately, proceed to the next section. Otherwise, shut down the system as appropriate.

Bubbler installation

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. Insure that one of the hydrogen cylinders is actively connected to the hydrogen supply line, that the pressure regulator is properly set and that the hydrogen purifier is turned on and up to temperature.
3. Insure that PR6 is set at 10 psig and PR1 is set at 15 psig.
4. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 switch on the HYDROGEN/NITROGEN control panel is in the off position. Turn the MANIFOLD N_2 switch on the HYDROGEN/NITROGEN control panel to the on position and adjust the FLOW SET 1 potentiometer to maintain a minimum deposition chamber pressure of 1-2 Torr as indicated on the AdaptTorr pressure controller.
5. On the HYDROGEN/NITROGEN panel set the N_2 CARRIER/H_2 CARRIER switch to the H_2 CARRIER position.

6. Insure that the manual valves on the bubbler are closed. Uncap the inlet and outlet ports on the bubbler and connect the bubbler to the circuit using new gaskets if the previously used gaskets were not of the self-centering type.
7. On the P-DOPANT control panel turn the ACTIVE switch and the BUBBLER IN switch on. Insert a key and turn the PURGE BUBBLER switch clockwise. The red PURGE BUBBLER light should be on. Push in the black DVM SELECT button and adjust the FLOW SET potentiometer for PDOPMFC1 to a flow of 100 sccm.
8. Evacuation: Insert a key in the CLOSE MFC2 switch and turn it clockwise. This will shut off the gas flow through PDOPMFC2. As the gas in the line is evacuated, the flow rate will drop. When the flow rate for PDOPMFC1 reaches zero as indicated on the panel meter, the circuit is completely evacuated.
9. Backfill: Turn the key in the CLOSE MFC2 switch counter clockwise for 10 seconds. This will allow the bubbler circuit to fill with hydrogen.
10. Repeat steps 8 and 9 twenty times to completely purge the circuit of air. Finish with a backfill step. Remove the key to the CLOSE MFC2 switch.
11. Open the manual valve on the outlet of the bubbler first. If the pressure in the bubbler should happen to be greater than 10 psig (the setting of PR6) and the inlet valve were opened first, the liquid alkyl would be forced back into the inlet plumbing.
12. Open the manual valve on the inlet of the bubbler.
13. Turn the BUBBLER IN switch and the ACTIVE switch to the off position. Turn the PURGE key lock switch off (counter-clockwise) and remove the key.
14. Shut down the system as appropriate.

Purging of DMZ Lines

Purging must be achieved through the mass flow controller to the vacuum pump. Use alternating Evacuation/backfill cycles to remove reactive gases. Due to the limited conductance of the gas lines allow cycle times of a few minutes. The regulator inlet lines can be independently purged for changing cylinders;

Purge of regulator and low-pressure delivery lines:

This operation need be performed only to purge the line downstream of the regulator; purge of the high-pressure lines is described in the next section.

1. Close the high-pressure inlet valve, PDOPMV2to the regulator.
2. With the vacuum pump running, open the VAC EXHAUSTswitch on the VACUUM CONTROL panel and set the N_2 manifold flow controller to maintain a minimum deposition chamber pressure of 1-2 Torr.
3. Evacuation: On the N DOPANT control panel open the ACTIVE switch, the GAS SOURCE IN switch, and the RUN switch. Adjust PDOPMFC1for maximum flow. Insert a key and turn the key lock PURGE MFC4 switch clockwise. Allow several minutes for the line to evacuate.
4. Backfill: Turn the GAS SOURCE IN switch off. Turn the H_2 DILUTE switch on. Insert a key in the key lock H_2 FLUSH switch and turn it clockwise for at least 10 seconds to backfill the line with hydrogen. Turn the H_2 FLUSH switch counterclockwise and turn the H_2 DILUTE switch off. Turn the GAS SOURCE IN switch on.
5. Repeat the evacuate cycle allowing several minutes for the line to evacuate.
6. Repeat the backfill/evacuate cycle a total of five times. After the last evacuate cycle, set PDOPMFC1 to zero flow, turn the PURGE MFC4 switch to the off position and remove the key. Turn the H_2 FLUSH switch to the off position and remove the key.
7. Backfill with DMZ:
    1. Back-off the knob on pressure regulator PDOPPR1 (fully counterclockwise)

2. Open the high-pressure regulator inlet valve PDOPMV2.
3. Set the knob on pressure regulator PDOPPR1to 10 psig at the outlet. Check the excess-flow valve PDOPEFV1 to see that it has not tripped by pushing in on the red button and observing the gauge on the outlet of PDOPPR1 for any changes in pressure.
4. Close the RUN SWITCH, the GAS SOURCE IN switch and the ACTIVE switch.

Cylinder Replacement

Cylinder replacement requires purging of only the high-pressure inlet line to the regulator. The high-purity, low-pressure delivery lines are not exposed to air during the replacement operation.

Cylinder removal:

1. Tightly close the DMZ cylinder valve and prepare to remove gas from the inlet line.
2. Flow DMZ through the reactor to reduce the inlet pressure to ""0 psig.
3. Close the regulator inlet valve PDOPMV2.
4. Lock the excess flow valve PDOPEFV1 in the open position with tape.
5. Purge the regulator inlet: Turn on the nitrogen supply valve, NMV3, to the vacuum educator and open the DMZ regulator vent valve, PDOPMV4. (DANGER: Be sure the DMZ cylinder valve is closed.) After 5 seconds close PDOPMV4 and NMV3.
6. Backfill the regulator inlet: First insure the tank valve for PDOPPURGEPR1 is open, that the outlet of regulator PDOPPURGEPR1 is at least 15 psig, and that PDOPPURGEMV1 is fully open (clockwise). Backfill the regulator input with nitrogen by opening PDOPMV3 for at least 2 seconds. Close PDOPMV3.
7. Repeat the purge-backfill cycle of steps e and f twenty times. Finish with a backfill step, close PDOPMV3 and PDOPMV4,and disconnect and cap the cylinder. The handheld Matheson toxic gas monitor should be used when unscrewing the cylinder connection. The CGA 350 connection on the DMZ cylinder has a left-hand thread. If you are facing the cylinder, the nut on the excess flow valve must be turned clockwise to loosen it. It is very important that the brass cap for the cylinder valve be replaced. The cylinder valves sometimes have small leaks even when tightly closed.

Cylinder installation:

1. With the new DMZ cylinder sitting inside the MOCVDcabinet and the exhaust fan on, slightly loosen the brass cap which is plugging the end of the CGA 350 connection on the cylinder valve. The cap has a left-hand thread. Check for the presence of DMZ using the portable Matheson toxic gas detector. If a high level release of DMZ is detected, the cap on the valve should be retightened and the cylinder returned to the vendor. If a low level release is detected, it may be due to a small amount of arsine trapped between the closed valve on the cylinder and the brass cap. With the brass cap left slightly unscrewed close the door to the MOCVD cabinet and let the tank set for several hours. If no DMZ is detected at that point, it is safe to completely unscrew the cap and install the cylinder. Otherwise the cap should be re-tightened and the tank returned to the vendor for replacement.
2. Connect the excess flow valve, PDOPEFV1,to the cylinder. Keep the regulator inlet valve, PDOPMV2, closed as well as the nitrogen backfill valve PDOPMV3. Hold the excess flow valve, PDOPEFV1, open by temporarily holding in the red button with a piece of tape.
3. Purge the regulator input: Turn on the nitrogen flow for the vacuum eductor, by opening NMV4. Open the purge valve, PDOPMV4, wait 5 seconds and then close PDOPMV4 and NMV4.
4. Backfill the regulator input: First insure the tank valve for DOPPURGEPR1 is open, that the outlet of regulator DOPPURGEPR1 is at least 15 psig, and that DOPPURGEMV1 is fully open (clockwise). Backfill the regulator input with nitrogen by opening PDOPMV3 for at least 2 seconds. Close PDOPMV3.

5. Repeat the backfill-purge cycle 20 times finishing with a purge step. Insure that PDOPMV3, PDOPMV4, and MV3 are closed.
6. Remove the tape from the excess flow valve, PDOPEFV1.
7. Open the manual valve on the tank and check for leaks.
8. Reset the excess flow valve, PDOPEFV1, by pushing in the red button and check for leaks.
9. Open the manual valve on the regulator input, PDOPMV2and check for leaks.
10. Close the tank valve on the nitrogen cylinder supplying gas to regulator DOPPURGEPR1.

Vacuum Control

The vacuum control system includes a number of pneumatic valves and interlocks which allow the chambers in the MOCVD system to be evacuated and which at certain times purge portions of the system with nitrogen to prevent backstreaming of the vacuum pump oil. This section begins with a description of the front panel controls and indicators.

Front Panel Functions

Indicator Lamps

REMOTE	The vacuum valve between the deposition chamber and the vacuum pump, EXPV2 can be operated under computer control. In order to enable this, the REMOTE circuit in the VACUUM CONTROL panel must be in the energized state. In remote mode this front panel lamp will be on.
VACUUM EXHAUST	This light comes on if valve EXPV2 between the deposition chamber and the vacuum pump is open. The valve can be opened using either the front panel switch or by computer control.
CONTROL VALVE CLOSED	Indicates that the control valve for the AdapTorr pressure controller is in the fully closed state.
CONTROL VALVE OPEN	Indicates that the control valve for the AdapTorr pressure controller is in the fully open state.
VACUUM IN CHAMBER	Any pressure reading less than the setting for Trip Point 2 on the AdapTorr ACR-26 controller is considered to be vacuum. The output signal from the ACR-26 unit energizes EXK3 when the pressure is less than the trip point setting. EXK3 controls several other interlocks. Trip Point 2 should be set at at 300 Torr. This pressure is about 3 times higher than a normal deposition pressure.
ATM PRESS IN CHAMBER	Any pressure reading greater than the setting for Trip Point 1 on the AdapTorr ACR-26 controller is considered to be atmospheric pressure. The output signal from the ACR-26 unit energizes EXK2 when the pressure is greater than the trip point setting. EXK2 controls several other interlocks. Trip Point 1 should be set at 700 Torr.
VACUUM LATCH	If the pressure in the deposition chamber falls below the setting of Trip Point 2 vacuum in chamber), the vacuum latch relay EXK1 becomes activated and the front panel lamp turns on. If the pressure in the chamber rises above the setting of Trip Point 2, the latch stays engaged until either the vacuum valve EXPV2 closes or the pressure control valve closes completely.
ENTRY CHAMBER UP TO AIR	Indicates that nitrogen gas is flowing into the sample entry chamber. This function is controlled by the switch directly above the lamp.
ENTRY CHAMBER EXHAUST	Indicates that the vacuum valve EXPV4 is open and that the sample entry chamber is being evacuated.
LOAD LOCK OPEN	Indicates that the gate valve between the deposition chamber and the sample entry chamber is open.

LOAD LOCK CLOSED	Indicates that the gate valve between the deposition chamber and the sample entry chamber is closed.
SCRUBBER OXIDATION	Indicates that air is flowing into the inlet of the scrubber.
NITROGEN FLUSH	Nitrogen gas automatically flows into the system at one of three selected ports to insure that the pressure at the vacuum pump inlet does not drop below 100 microns Hg. The purpose of this is to prevent the backstreaming of oil from the pump to the deposition and sample entry chambers. The port that is selected depends on the state of the vacuum exhaust valves and the pressure measured by the AdapTorr pressure controller.
DEP CHAMBER	Indicates that nitrogen is flowing into the port at the base of the spinner shaft. This flow serves two functions. It insures a minimum flow through the deposition chamber to prevent oil backstreaming. It also floods the upper opening of the bellows assembly with nitrogen so as to prevent the flow of source gases into the bellows assembly. If the source gases were to enter the bellows, it is possible that material could be deposited inside the bellows assembly. This lamp will be on and EXPV3 will open if valve EXPV2to the vacuum pump is open and there is adequate pressure in the nitrogen supply line and the vacuum pump is turned on and the pressure is below the setting of Trip Point 2 (vacuum in chamber) and the interlock system is not in abort status. The flow rate is controlled by manual valve EXMV3.
SAMPLE ENTRY CHAMBER	Indicates that nitrogen is flowing into the sample entry chamber. This lamp comes on and EXPV4 will be open if EXPV2 is closed and there is adequate pressure in the nitrogen supply line and the vacuum pump is turned on and valve EXPV6 to the vacuum pump is open and the interlock system is not in abort status. The flow rate is controlled by EXMV4.
PUMP INLET	Indicates that nitrogen is flowing into the particle filter on the inlet of the vacuum pump. The lamp comes on and EXPV5 will be open if there is adequate pressure in the nitrogen supply line and the vacuum pump is on and EXPV2 is closed and EXPV6 is closed. The flow rate is controlled by EXMV5.

Switches

VAC EXHAUST	This switch opens valve EXPV2 between the deposition chamber and the vacuum pump. The switch is disabled when the panel is in the REMOTE mode.
ENTRY CHAMBER UP TO AIR	This switch opens EXPV9 which allows a high flow of nitrogen gas to enter the sample entry to rapidly bring it up to atmospheric pressure. The gas flow rate is controlled by EXMV9. This switch is disabled if either valve EXPV6 between the sample entry chamber and the vacuum pump is open or the gate valve between the deposition chamber and the sample entry chamber is open.
ENTRY CHAMBER EXHAUST	This switch opens EXPV6 between the sample entry chamber and the vacuum pump. EXPV6 will open only if valve EXPV2 between the vacuum pump and the deposition chamber is closed. However, once EXPV6 is opened, EXPV2 can be opened and EXPV6 will not close automatically. The reason for this interlock is to prevent air from being introduced into the deposition chamber. If it is desired to have both EXPV2 and EXPV6open at the same time, such as when a sample is being loaded into the deposition chamber, then it is necessary to first evacuate the sample entry chamber with the valve to the deposition chamber closed. When the sample entry chamber is evacuated then both valves can be opened simultaneously.
LOAD LOCK OPEN/CLOSED	This is a three position momentary contact switch (ON-OFF-ON, normally in the off state) which controls the position of the gate valve between the deposition chamber and the sample entry chamber. When used, the switch should be held in position until the appropriate lamp comes on. The gate valve opens when the switch is pushed upward. This function is disabled unless valve EXPV2 to the deposition chamber is open and valve EXPV6 to the sample entry chamber is open. This prevents the valve from being actuated if there is a pressure differential across it. The gate valve closes when the switch is pushed downward. This function is disabled unless the push rod for sample loading is pulled completely out of the path of the gate valve and hooked to its retainer.
SCRUBBER OXIDATION	This switch opens AUXPV2 which allows air to enter the inlet of the scrubber so than the adsorbed gases and vapors can be oxidized. The switch is disabled if any liquid or vapor source is turned on or if the hydrogen manifold diluent is turned. This prevents hydrogen and air from flowing through the system at the same time. Conversely, if the SCRUBBER OXIDATION switch is on, the interlock system is in C-Level status which prevents any of the valves for the sources or the hydrogen manifold diluent from being turned on. The flow rate of air through AUXPV2 is controlled by the setting of AUXMV2 and indicated by the adjacent rotameter which gives a full scale indication of 300 sccm.

Normal Operation

A normal sequence of operations for loading a substrate, making a deposition, and removing the substrate is as follows. It is assumed that all switches on the VACUUM CONTROL panel are closed.

1. Turn on the ENTRY CHAMBER UP TO AIR switch. When the sample entry chamber is at atmospheric pressure the glass cover can be easily lifted off. Remove the cover and turn off the ENTRY CHAMBER UP TO AIR switch.
2. Place the sample on the loading surface and replace the cover on the chamber.

3. Turn on the ENTRY CHAMBER EXHAUST switch. Wait about 5 minutes for water vapor to be desorbed from the walls of the chamber.
4. Open the VAC EXHAUST switch. Wait one minute.
5. Push the LOAD LOCK OPEN/CLOSED switch upward to open the gate valve.
6. Load the sample onto the susceptor.
7. Remove the sample pushrod from the deposition chamber and make sure its end is attached to the retaining hook.
8. Push the LOAD LOCK OPEN/CLOSED switch downward to close the gate valve.
9. Close the ENTRY CHAMBER EXHAUST switch.
10. Proceed with the deposition.
11. When the deposition is finished, all gases are turned off, the susceptor has cooled, and the susceptor has been lowered; open the ENTRY CHAMBER EXHAUST switch.
12. Push the LOAD LOCK OPEN/CLOSED switch upward to open the gate valve.
13. Use the pushrod to transport the substrate from the deposition chamber to the sample entry chamber. Hook the end of the pushrod to its retainer.
14. Push the LOAD LOCK OPEN/CLOSED switch downward to close the gate valve.
15. Close the ENTRY CHAMBER EXHAUST switch and the VAC EXHAUST switch.
16. Open the ENTRY CHAMBER UP TO AIR switch, remove the cover on the sample entry chamber and turn off the ENTRY CHAMBER UP TO AIR switch.
17. Remove the sample, replace the cover on the chamber and open the ENTRY CHAMBER EXHAUST switch.
18. Wait 5 minutes and close the ENTRY CHAMBER EXHAUST switch.

Interconnections

1. VAC EXHAUST controls EXSV2 which drives EXPV2.
2. ENTRY CHAMBER UP TO AIR controls EXSV9 which drives EXPV9.
3. ENTRY CHAMBER EXHAUST controls EXSV6 which drives EXPV6.
4. LOAD LOCK OPEN controls EXSV7 which drives one side of the pneumatic cylinder for the gate valve.
5. LOAD LOCK CLOSED controls EXSV8 which drives the other side of the pneumatic cylinder.
6. SCRUBBER OXIDATION controls AUXSV2 which drives AUXPV2.

Interlocks

1. If the pressure in the deposition chamber falls below the setting of Trip Point 2 (vacuum in chamber), the vacuum latch relay EXK1 becomes activated and the front panel lamp VACUUM LATCH turns on. If the pressure in the chamber rises above the setting of Trip Point 2, the latch stays engaged until either the vacuum valve EXPV2 closes or the pressure control valve closes completely.
2. The CHAMBER VAC LOSS interlock will activate and place the interlock system in Abort status if the chamber pressure rises above the setting for Trip Point 1 (atmospheric pressure in chamber) with the VACUUM LATCH activated and the valve, EXPV2, between the deposition chamber and the vacuum pump open.
3. Nitrogen will automatically enter the deposition chamber, DEP CHAMBER lamp will be on and EXPV3 will open if valve EXPV2 to the vacuum pump is open and there is adequate pressure in the nitrogen supply line and the vacuum pump is turned on and the pressure is below the setting of Trip Point 2 (vacuum in chamber) and the interlock system is not in abort status.
4. Nitrogen will automatically enter the sample entry chamber, the SAMPLE ENTRY CHAMBER lamp will come on and EXPV4 will be open if EXPV2 is closed and there is adequate pressure in the nitrogen supply line and the

vacuum pump is turned on and valve EXPV6 to the vacuum pump is open and the interlock system is not in abort status.

5. Nitrogen will automatically enter the particle filter on the inlet of the vacuum pump, the PUMP INLET lamp will come on and EXPV5 will be open if there is adequate pressure in the nitrogen supply line and the vacuum pump is on and EXPV2 is closed and EXPV6 is closed.
6. The VAC EXHAUST switch is disabled when the panel is in the REMOTE mode.
7. The ENTRY CHAMBER UP TO AIR switch is disabled if either valve EXPV6 between the sample entry chamber and the vacuum pump is open or the gate valve between the deposition chamber and the sample entry chamber is open.
8. The ENTRY CHAMBER EXHAUST switch which opens valve, EXPV6, will be enabled only if valve EXPV2 between the vacuum pump and the deposition chamber is closed. However, once EXPV6 is opened, EXPV2 can be opened and EXPV6 will not close automatically.
9. The LOAD LOCK OPEN function is disabled unless valve EXPV2 to the deposition chamber is open and valve EXPV6 to the sample entry chamber is open.
10. The LOAD LOCK CLOSED function is disabled unless the push rod for sample loading is pulled completely out of the path of the gate valve and hooked to its retainer.
11. The SCRUBBER OXIDATION switch is disabled if any liquid or vapor source is turned on or if the hydrogen manifold diluent is turned on. Conversely, if the SCRUBBER OXIDATION switch is on, the interlock system is in C-Level status which prevents any of the valves for the sources or the hydrogen manifold diluent from being turned on.

Programming commands for remote operation

A number of the functions of the VACUUM CONTROL panel can only controlled manually until a suitable interface from LabView to the AdapTorr module is in place.

Insert LabView control documentation here

Nitrogen Flush System

If the pressure at the inlet of a mechanical vacuum pump falls below the vapor pressure of the vacuum pump oil, then the oil vapor will begin to migrate upstream through the plumbing system. If this situation is allowed to continue long enough, the entire apparatus being serviced by the pump will become coated with a thin layer of the pump oil. This is the origin of the characteristic oily odor inside the bell jars of many vacuum systems which employ mechanical pumps. If this were to occur inside the deposition chamber of an MOCVD system in which extremely low impurity concentrations must be maintained, then only very highly contaminated layers could be grown. Backstreaming of the pump oil can be eliminated, if a flow of gas into the inlet of the vacuum pump is maintained which keeps the inlet pressure greater than the oil vapor pressure. A pressure of 500 micron Hg is more than sufficient to achieve this. Maintaining a minimum gas flow rate into the pump is sufficiently important that systems have been added to the MOCVD reactor which add nitrogen automatically under all operating conditions. Nitrogen gas automatically flows into the system at one of three selected ports to insure that the pressure at the vacuum pump inlet does not drop below 500 microns Hg. The port that is selected depends on the state of the vacuum exhaust valves and the pressure measured by the AdapTorr pressure controller. Gas enters the port at the base of the spinner shaft in the deposition chamber if valve EXPV2to the vacuum pump is open and there is adequate pressure in the nitrogen supply line and the vacuum pump is turned on and the pressure is below the setting of Trip Point 2 (vacuum in chamber) and the interlock system is not in abort status. The flow rate is controlled by manual valve EXMV3. Gas enters the sample entry chamber if EXPV2 is closed and there is adequate pressure in the nitrogen supply line and the vacuum pump is turned on and valve EXPV6to the vacuum pump is open and the interlock system is not in abort status. The flow rate is controlled by EXMV4. Gas enters the particle filter on the inlet of the vacuum pump if there is adequate pressure in the nitrogen supply line and the vacuum pump is on and EXPV2 is closed and EXPV6 is closed. The flow rate is controlled by EXMV5.

Setting the flow rates for the nitrogen flush system

Deposition chamber:

1. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
2. With the vacuum pump running, open the VAC EXHAUST switch on the VACUUM CONTROL panel. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 and MANIFOLD N_2 switches on the HYDROGEN/NITROGEN control panel are off.
3. Close the needle valve EXMV3 located in the main gas control cabinet.
4. Quickly make a note of the pressure reading on the AdapTorr pressure controller and proceed to Step 5. The total amount of time the pump is left running with no gas flow into the input should not be more than a minute or so.
5. Open EXMV3 until the pressure as indicated on the AdapTorr pressure controller increases by five Torr. Count the number of turns of the valve needed to do this.
6. Use the graph at the end of this section to determine the amount of valve opening (CV value) for the number of turns measured in Step 5. Divide this Cv value by 10 and determine from the graph determine the number of turns open needed to achieve this. Reset EXMV3 to this value. This should give a system pressure of about 0.5 Torr or 500 microns Hg.

Vacuum pump inlet:

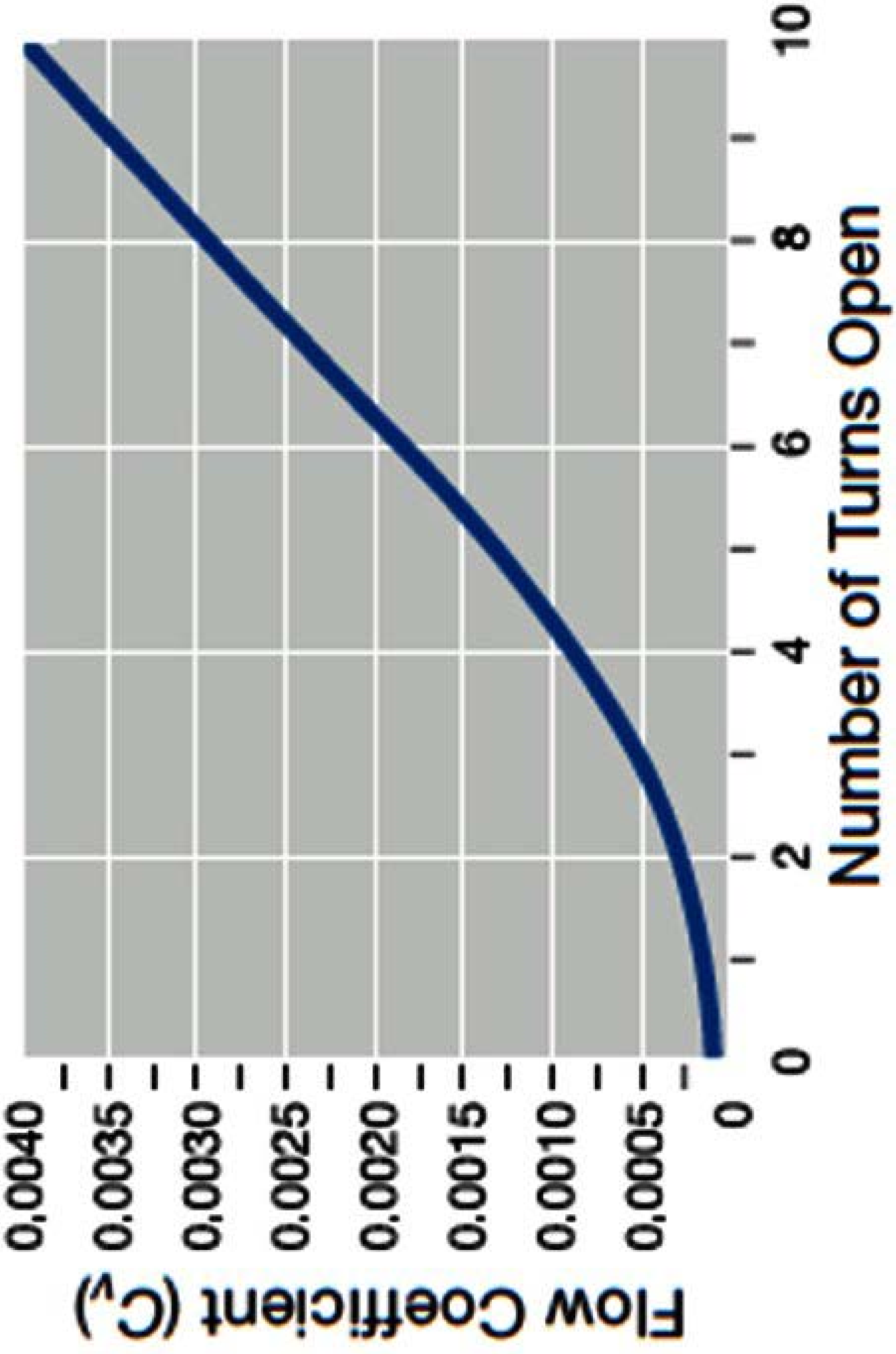
1. Turn off the vacuum pump and the power to the control cabinet. Remove the 1/8" OD pneumatic control tubing from EXPV2. Use a 1/8" union tee and lengths of 1/8" ODtubing to connect the control line for EXPV5 to

both EXPV5 and EXPV2. This will keep EXPV2 open when nitrogen is flowing through EXPV5 so that the pressure can be measured. Turn on the power to the control cabinet and the vacuum pump.

2. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
3. With the vacuum pump running, all the switches on the VACUUM CONTROL panel should be closed. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 and MANIFOLD N_2 switches on the HYDROGEN/NITROGEN control panel are off.
4. Close the needle valve EXMV5 located in the vacuum pump compartment.
5. Quickly make a note of the pressure reading on the AdapTorr pressure controller and proceed to Step 6. The total amount of time the pump is left running with no gas flow into the input should not be more than a minute or so.
6. Open EXMV5 until the pressure as indicated on the AdapTorr pressure controller increases by five Torr. Count the number of turns of the valve needed to do this.
7. Use the graph at the end of this section to determine the amount of valve opening (CV value) for the number of turns measured in Step 6. Divide this Cv value by 10 and determine from the graph determine the number of turns open needed to achieve this. Reset EXMV5 to this value. This should give a system pressure of about 0.5 Torr or 500 microns Hg.
8. Turn off the vacuum pump and the power to the control cabinet. Reconnect the control lines for EXPV2 and EXPV5 in their normal configuration. Turn the power to the control cabinet and the vacuum pump back on.

Sample entry chamber:

1. Turn off the vacuum pump and the power to the control cabinet. Remove the 1/8" OD pneumatic control tubing from EXPV6. Use a 1/8" union tee and lengths of 1/8" OD tubing to connect the control line for EXPV4 to both EXPV4 and EXPV2. This will keep EXPV2 open when nitrogen is flowing through EXPV4 so that the pressure can be measured. Turn on the power to the control cabinet and the vacuum pump.
2. Insure that the tank valve for the nitrogen is on, that the pressure regulator on the tank gives a pressure of 20 psig, and that NMV1 is open.
3. With the vacuum pump running, open the ENTRY CHAMBER EXHAUST switch. Make sure that the CONTROL VALVE OPEN light on the VACUUM CONTROL panel is on. If not press the OPEN button on the AdaptTorr ACR-26 pressure controller to fully open the pressure control valve. Make sure the MANIFOLD H_2 and MANIFOLD N_2 switches on the HYDROGEN/NITROGEN control panel are off.
4. Close the needle valve EXMV410 located in the main gas control cabinet.
5. Quickly make a note of the pressure reading on the AdapTorr pressure controller and proceed to Step 6. The total amount of time the pump is left running with no gas flow into the input should not be more than a minute or so.
6. Open EXMV4 until the pressure as indicated on the AdapTorr pressure controller increases by five Torr. Count the number of turns of the valve needed to do this.
7. Use the graph at the end of this section to determine the amount of valve opening (CV value) for the number of turns measured in Step 6. Divide this Cv value by 10 and determine from the graph determine the number of turns open needed to achieve this. Reset EXMV4 to this value. This should give a system pressure of bout 0.5 Torr or 500 microns Hg.
8. Turn off the vacuum pump and the power to the control cabinet. Reconnect the control lines for EXPV2 and EXPV4 in their normal configuration. Turn the power to the control cabinet and the vacuum pump back on.


Flow Coefficient (C_v)
0.0040
0.0035
0.0030
0.0025
0.0020
0.0015
0.0010
0.0005
0
0
2
4
6
8
10
Number of Turns Open

Substrate Control Panel

The controls for raising and lowering the susceptor, substrate heating and susceptor rotation are contained on a single control panel. The functions of substrate heating and susceptor rotation can be operated by remote control.

Front Panel Switches

BLOWER	Applies power to the blower located inside the Reactor Cabinet. The blower should be used for high temperature growth (>650°C).
HEATER	Applies power to the temperature controller, the induction heater and the blower which cools the quartz deposition chamber. When switch is in the up position, the panel lights for the temperature controller will come on. A time delay circuit which controls the power supplied to the induction heater and the blower keeps the power on for approximately 19 minutes after the switch is turned off. This feature was added so that cooling water will continue to flow through the induction heater and the blower will stay on, as the susceptor cools. Otherwise there would be a rather large temperature rise in the components when the power was turned off. This switch is disabled if the interlock system is in the abort mode.
SPINNER	Applies power to the motor speed controller and tachometer for susceptor rotation. The rotation speed of the susceptor can be adjusted using the SPINNER SPEED CONTROL potentiometer, and the rotation speed is indicated on the tachometer labeled SPINNER RPM. The spinner cannot be turned on unless the RAISE SUSCEPTOR switch is in the up position. The reason for this is that the bellows which allows the susceptor to be raised and lowered touches the rotating shaft which supports the susceptor when the susceptor is in the down position. If the motor were turned on in the down position, the bellows would probably be destroyed. A slow start circuit in the motor speed controller reduces the possibility that the substrate doesn't fly off when the motor is turned on.
RAISE SUSCEPTOR	Applies air pressure to the pneumatic line which raises the susceptor. The front panel RAISE SUSCEPTOR lamp will come on. The rate of travel of the pneumatic cylinder is controlled by pressure regulators and needle valves so that the substrate doesn't go flying when the susceptor is raised or lowered.
LOWER SUSCEPTOR	Applies air pressure to the pneumatic line which lowers the susceptor.

Programming commands for remote operation

Insert LabView control documentation here

Induction Heater

The substrate heating can be turned on and off remotely but the set point temperature must be entered manually into the front panel of the temperature controller. Assuming the tuning procedure for the controller has already been performed, the set point can be adjusted by pressing only the ↑(YES), ↓(NO) arrows on the controller to raise (lower) the setpoint. Consult the operator manual for the controller for further details on its operation and for tuning procedures. The temperature of the substrate can be measured by remote control and recorded in the program output. The same thermocouple used for temperature control is connected to Channel 1 of the low voltage A/D converter in slot 403C of the Multiprogrammer.