

UCSC


Laboratory Standard Operating Procedure (SOP)

Compressed Gas Cylinder Change for MOCVD and CVD Systems




























Department:	Chemistry	Date:	02/25/13
Principal Investigator:	Yat Li	Office Phone:	Enter text.
Lab Safety Representative:	Enter text. (Name and Phone Number)	Lab Phone:	Enter text.
Location(s) covered by this SOP:	PSB 198	Emergency Contact:	Enter text. (Name and Phone Number)

Review any applicable manufacturer/vendor safety information before developing the Standard Operating Procedure (SOP). Use the Safety Data Sheet (SDS), the UC Center for Laboratory Safety Chemical SOP (www.ucsop.com), and/or other reference materials to complete the chemical information section appended to this SOP. The Online SDSs can be accessed at: <http://www.ucmsds.com/?X>.¹ Attach SDS for each chemical included in this SOP.

Any deviation from this SOP requires approval from the PI.

#1	<p>Scope of Work/Activity: Provide a general description of the process and/or experimental procedure, specifying how the chemicals and other hazardous materials will be used. Include apparatus and special equipment required for the process/procedure.</p> <p>This SOP covers the replacement of compressed gas cylinders (ammonia, hydrogen, and dilute silane/hydrogen mixture) for the MOCVD and CVD systems in the laboratory. Each gas cylinder is installed in a ventilated gas cabinet equipped with a nitrogen purge system.</p> <div style="text-align: center;">  </div> <p>Chemicals that fall under this SOP include:</p> <ul style="list-style-type: none"> • Ammonia (7664-41-7; acutely toxic gas, flammable, corrosive)
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¹ If available, provide the Long Gold SDS version – contact EH&S for assistance.

	<ul style="list-style-type: none"> Hydrogen (1333-74-0; Flammable gas) Silane (7803-62-5; Pyrophoric) <ul style="list-style-type: none"> Note the silane is diluted to 100 ppm in hydrogen, which is below the LFL for silane <p>Note: For operation of the MOCVD and CVD system, refer to the documents “SOP for Metal Organic Chemical Vapor Deposition (MOCVD) Operation” and “Standard Operating Procedure (SOP) for Chemical Vapor Deposition (CVD) Operation”, respectively.</p>															
#2	<p>Specific Safety and Environmental Hazards: <i>State specific hazards and potential consequences to person, environment, or property if procedure not followed.</i></p> <p>Release of process gases into the gas cabinet and/or lab can result in fire and chemical exposure.</p> <ul style="list-style-type: none"> Ammonia is toxic, corrosive and flammable. It is a colorless highly irritating gas with a sharp suffocating odor. Hydrogen is a flammable gas. Silane (100 ppm in hydrogen). Silane is a pyrophoric gas at concentrations > 4.5%. At 100 ppm, the silane concentration is below its lower flammability limit; however, hydrogen is a flammable gas. <p>Include applicable GHS symbols.²</p> <table border="1" data-bbox="172 996 1439 1585"> <tr> <td data-bbox="172 996 632 1182">Ammonia</td> <td data-bbox="632 996 820 1182"></td> <td data-bbox="820 996 1008 1182"></td> <td data-bbox="1008 996 1197 1182"></td> <td data-bbox="1197 996 1439 1182"></td> </tr> <tr> <td data-bbox="172 1182 632 1352">Hydrogen</td> <td data-bbox="632 1182 820 1352"></td> <td data-bbox="820 1182 1008 1352"></td> <td colspan="2" data-bbox="1008 1182 1439 1352"></td> </tr> <tr> <td data-bbox="172 1352 632 1585">Silane</td> <td data-bbox="632 1352 820 1585"></td> <td data-bbox="820 1352 1008 1585"></td> <td colspan="2" data-bbox="1008 1352 1439 1585"></td> </tr> </table>	Ammonia					Hydrogen					Silane				
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#3	<p>Incompatible Conditions and Materials: <i>List the incompatible conditions, chemicals, and/or materials that should be avoided.</i></p> <p>Keep cylinders in ventilated gas cabinets. Keep away from sources of ignition. Take measures to prevent the buildup of electrostatic charge.</p> <p>Dispense these materials only in sealed, purged systems. Handle sealed gas cylinders in accordance with CGA P-1, <i>Safe Handling of Compressed Gases in Containers.</i></p>															

² A Guide to The Globally Harmonized System of Classification and Labelling of Chemicals (GHS), <http://www.osha.gov/dsg/hazcom/ghs.html>

	<p>Chemical:</p> <p>Ammonia</p> <p>Hydrogen</p> <p>Silane/hydrogen</p>	<p>Incompatibility:</p> <p>Oxidizing agents, Iron, Zinc, Copper, Silver/silver oxides, Cadmium/cadmium oxides, Alcohols, Acids, Halogens, Aldehydes</p> <p>Oxidizing agents</p> <p>Bases, Oxidizing agents, Halogens, Halocarbons, Metal oxides, Metal salts, Combustible materials</p>
#4	<p>Hazard Controls: <i>Identify the Engineering Controls (e.g., fume hood, interlocks, shielding) and Administrative Controls (e.g., work practices or procedures, training) that will be employed to reduce hazards to acceptable levels. Address emergency shutdown procedures.</i></p> <p><u>Engineering Controls</u></p> <ul style="list-style-type: none"> The gas cylinders are installed in ventilated gas cabinets equipped with nitrogen purge systems. Each gas cabinet has an exhaust flow monitor and gas leak monitor with alarm. <p><u>Administrative Controls</u></p> <p>There is a possibility of gas leakage into the gas cabinet if the gas lines are not purged prior to loosening connections and if the valve connection to the gas cylinder is not properly made.</p> <ul style="list-style-type: none"> The procedural steps in Section 7 must be followed to ensure a safe cylinder change. The gas cabinet door is kept closed while the cylinder valve is opened or closed and while removing or reinstalling the piping connection to the cylinder. These operations are done by reaching through the access panel in the front of the gas cabinet. Working alone restrictions: Do not work alone while performing a gas cylinder change. Have a co-worker standing by outside the laboratory ready to call for help, if needed. <p>Precautions for safe handling: Follow the procedural steps in Section 7 to install or remove a gas cylinder. Dispense these materials only in sealed, purged systems. Handle sealed gas cylinders in accordance with CGA P-1, <i>Safe Handling of Compressed Gases in Containers</i>.</p> <p>Conditions for safe storage: Keep cylinders in ventilated gas cabinets. Keep away from sources of ignition. Take measures to prevent the buildup of electrostatic charge.</p> <p><u>Training & Competency Requirements</u> <i>Describe necessary training and demonstration of competency for performing the hazardous operation.</i></p> <ul style="list-style-type: none"> Complete EH&S online or instructor-led “Introduction to Laboratory Safety” class Review and sign Lab-Specific Training Checklist with PI, Lab Safety Representative, or other designated person. Review SOP with knowledgeable person. New users must be qualified by Yat Li or Yichuan Ling prior to changing a gas cylinder. <p><u>Decontamination/Clean-Up</u></p> <p>Proper installation or removal of a gas cylinder will not lead to contamination requiring clean-up. Contact EHS before trying to clean-up after a precursor an accidental gas release.</p> <p><u>Emergency shutdown procedure</u></p> <ul style="list-style-type: none"> If it is safe to do so, turn off the gas cylinder valve by reaching into the gas cabinet through the access panel. DO NOT OPEN THE GAS CABINET DOOR. 	

	<ul style="list-style-type: none"> Press the red “Emergency Stop” button near the door to switch off all gas supply.
#5	<p>Personal Protective Equipment (PPE): <i>State the personal protective equipment selected and required. Examples: safety glasses, goggles or face shield; lab coat; specific gloves; chemical-proof apron; respiratory protection.</i></p> <p>Eye Protection: ANSI-approved properly fitting safety glasses or goggles.</p> <p>Skin and Body Protection: Appropriately-sized lab coats must be worn and buttoned to their full length. Laboratory coat sleeves must be of sufficient length to prevent skin exposure while wearing gloves. Full length pants and closed-toe shoes must be worn at all times by all individuals within the laboratory area. The area of skin between the shoe and ankle should not be exposed.</p> <p>Check box for type of lab coat: <input checked="" type="checkbox"/> Flame Resistant <input type="checkbox"/> 100% cotton</p> <p>Hand Protection: <i>State the appropriate chemical-resistant gloves for the material(s) that will be handled. For example:</i></p> <p>Wear leather gloves when performing a gas cylinder change out. Remove gloves and wash hands with soap and water after use.</p> <p>Use the SDS and refer to glove selection chart from the link to determine appropriate glove selection: http://www.microflex.com/Products/~media/Files/Literature/Domestic%20Reference%20Materials/DOM_Reference_Chemical%20Resistance.ashx</p>
#6	<p>Designated Area: <i>Indicate the area designated for performing this process in the laboratory.</i></p> <p>The ammonia and silane/hydrogen cylinders are installed in their respective gas cabinets.</p>
#7	<p>Important Steps to Follow: <i>Provide the steps for the procedure from obtaining the specific reagent bottles to returning those bottles to the appropriate storage location. List the specific sequence of steps required or recommended to mitigate potentially hazardous conditions. In addition, note location and use of any emergency response equipment specific to process (e.g., Calgonate gel, Class D fire extinguisher, inert absorbent material). Include information for special handling and storage requirements.</i></p> <p>Preparation for removing or installing gas cylinders:</p> <ol style="list-style-type: none"> Notify all lab personnel that you will be doing a cylinder change and have them leave the lab. Assign one person to stand outside the lab to keep the area clear and to watch while you perform the cylinder change and be ready to call 911 if help is needed. <p>Removing Gas Cylinder:</p> <ol style="list-style-type: none"> Insert key into the panel on front of gas cabinet (see Image #1 below), and turn it 90 degrees to the left. Close the furnace. Pull the panel out and hold open for 10 seconds. Reach in through the panel (do not open the cabinet door) and turn clockwise the top valve of the gas cylinder (see Image #2) to close the cylinder. Close the panel. Run the nitrogen purge cycle by using the MOCVD control panel. When the purge is complete, reach in through the panel (do not open the cabinet door) and unscrew (anti-clockwise) the side valve to separate the gas cylinder from the gas line in the cabinet (See Image #3). Check the gas monitor on top of the cabinet to make sure there are no leaks. Open the gas cabinet door and secure the gas cylinder cap over the valve on the cylinder. Remove the chains securing the gas cylinder and secure the cylinder to a gas cylinder cart.

10. Remove the cylinder and close the gas cabinet door.

Installing Gas Cylinder:

1. Insert key into the panel on front of gas cabinet (see Image #1 below), and turn it 90 degrees to the left.
2. Pull the panel out and hold open for 10 seconds.
3. Open the gas cabinet door.
4. Place the new gas cylinder inside the cabinet and secure it with chains.
5. Remove the gas cylinder cap from the top of the cylinder.
6. Close the gas cabinet door.
7. Reach in through the panel (do not open the cabinet door) and screw clockwise (see Image #3) the side valve to link the gas cylinder with the gas lines inside the cabinet.
8. Run the nitrogen purge cycle by using the MOCVD control panel.
9. When the purge is complete, reach in through the panel (do not open the cabinet door) and turn anti-clockwise the top valve of the gas cylinder (see image #2) to open the cylinder.
10. Close the panel.
11. Check the gas monitor on top of the cabinet to make sure there are no leaks.

Image #1



Image #2



Image #3



#8 Spill Clean-Up Procedures (reference appended SDS as needed):

Do not attempt to clean up any spill or release for which you are not fully trained and equipped. For assistance with spill cleanup, dial **911** and ask dispatch to page EH&S.

In the event of a gas release:

1. Alert people in the laboratory to evacuate.
2. Press the red "Emergency Stop" button by the door to shut down the MOCVD and stop gas flow.
3. Close doors to affected area.
4. Call for Emergency Response: 911
5. Post with danger signs and have person knowledgeable of incident and laboratory assist emergency personnel

#9 Hazardous Waste(s): List expected concentrations and amounts of hazardous waste(s) generated during this process. Contact EH&S for specific guidance regarding hazardous waste handling and disposal.

Cylinders that contained ammonia or silane/hydrogen must be returned to the supplier.

General hazardous waste management guidelines: <http://ehs.ucsc.edu/programs/waste-management/index.html>

State if the generated waste cannot be combined with any other waste streams.

Waste Labeling

	<ul style="list-style-type: none"> Affix an on-line hazardous waste tag on all waste containers using the Online Tag Program (OTP) http://otp.ucop.edu/ as soon as the first drop of waste is added to the container. <p>Waste Storage</p> <ul style="list-style-type: none"> Store hazardous waste in closed containers, in clean secondary containment, segregated by hazard class, in a marked and designated waste accumulation area. Double-bag dry waste using transparent bags. Waste accumulation area must be under the control of the person generating the waste. <p>Waste Disposal</p> <ul style="list-style-type: none"> Hazardous waste must be removed from the lab within 180 days. Containers must be clean, sealed, and safe to transport. Mark container as ready for pick up in OTP, move container to accumulation area. Contact EH&S at x9-3086 for questions. 														
#10	<p><u>First Aid / Emergency Procedures:</u> Describe immediate First Aid or medical treatment required in case of personnel exposure.</p> <p>For immediate medical assistance, dial 911. Report all serious injuries to EH&S as soon as possible.</p> <ul style="list-style-type: none"> If inhaled, move into fresh air immediately. In the case of eye or skin contact, flush with water for a minimum of 15 minutes. Ensure that eyelids are held open while rinsing eyes. In the case of a needlestick/puncture injury, wash the affected area with soap and warm water for 15 minutes. For employees, follow the instructions at the Risk Services website: http://risk.ucsc.edu/workers-comp/reporting-and-treatment.html Seek medical attention immediately. Complete incident report form, http://risk.ucsc.edu/all-forms/wc-incident-report-form.pdf, (contact EH&S) and/or follow the instructions at the Risk Services website: http://risk.ucsc.edu/workers-comp/reporting-and-treatment.html <p><u>Laboratory Emergency Response Equipment:</u> All research personnel must know location of nearest fire alarm pull station and emergency shower/eyewash. Do not use fire extinguisher unless you are trained to do so.</p> <table border="1" data-bbox="172 1288 1436 1505"> <thead> <tr> <th>Item</th> <th>Location</th> </tr> </thead> <tbody> <tr> <td>Eyewash/Safety Shower</td> <td>Near the door</td> </tr> <tr> <td>Chemical Spill Kit</td> <td>N/A</td> </tr> <tr> <td>Fire Extinguisher</td> <td>Outside the door</td> </tr> <tr> <td>Telephone</td> <td>Student office</td> </tr> <tr> <td>First Aid Kit</td> <td>On the shelf</td> </tr> <tr> <td>Fire Alarm Manual Pull Station</td> <td>Outside the door</td> </tr> </tbody> </table>	Item	Location	Eyewash/Safety Shower	Near the door	Chemical Spill Kit	N/A	Fire Extinguisher	Outside the door	Telephone	Student office	First Aid Kit	On the shelf	Fire Alarm Manual Pull Station	Outside the door
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As the Principal Investigator, it is your responsibility to ensure that all individuals conducting this protocol are taught the correct procedures for safe handling of the hazardous materials involved. It is also your responsibility to ensure that your personnel complete Laboratory Safety Training and other applicable safety training courses.

I have reviewed and approve this Standard Operating Procedure.



3/7/2013

PI Signature:

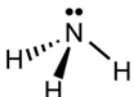
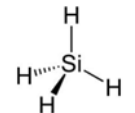
DATE

Note that personnel associated with the protocol must sign the acknowledgement at the end of this document.

Chemical Information Summary

Provide information for all chemicals included in the SOP. See attached SDS for detailed toxicity information.

Physical & Chemical Properties

Chemical	CAS#	Class	Molecular Formula	Structure	Molecular Weight (g/mol)	Density (g/mL)	Form (physical state)	Melting Point (°C)	Boiling point (°C)	Flash point (°C)
Ammonia	7664-41-7	Acutely toxic gas, flammable, corrosive	H ₃ N		17.03	0.0007 (relative vapor density = 0.59)	G	- 78	- 33	132 LFL= 15%; ULF=25%
Hydrogen	1445-79-0	Flammable	H ₂	H-H	2.02	N/A (relative vapor density = 0.08)	G	- 259.2	-252.8	< -150 LFL= 4%; ULF=74%
Silane	7803-62-5	Pyrophoric	SiH ₄		32.12	1.34 kg/m ³ (relative vapor density = 1.11)	G	- 185	- 112.2	N/A LFL= 1.4%; ULF=96%

Exposure Limits/Toxicity Data

Chemical	Color	Odor	Cal/OSHA PEL	Toxicity LD ₅₀
Ammonia	N/A	Sharp/suffocating	25 ppm TWA / 35 ppm STEL	LC ₅₀ 2000 ppm (rat, 4 hr)
Hydrogen	Colorless	N/A	NE	N/A
Silane	Colorless	Choking	5 ppm TWA	LC ₅₀ 19,200 ppm (rat, 1 hr)



Revision History:

Version	Date	Revision Author	Summary of Changes
1	01/09/2013	Yichuan Ling	Initial SOP author
2	01/10/2013	Nick Filipp	Inserted procedure into SOP template.
3	02/25/2013	Nick Filipp	Template updated; added chemical information.
4	03/06/2013	Lisa Wisser	EH&S Review