

## UCSC

# Laboratory Standard Operating Procedure (SOP)

### *Precursor Bubbler Change for MOCVD Systems*

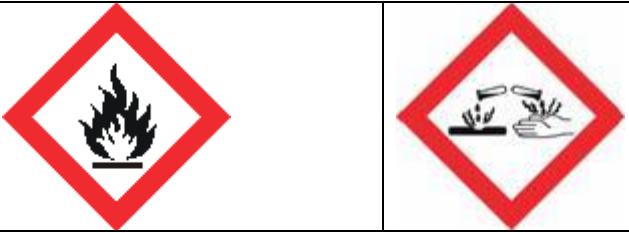
|   |  |                           |  |
|---|--|---------------------------|--|
| <b>Department:</b>                      | Chemistry                                  | <b>Date:</b>              | 02/25/13                                   |
| <b>Principal Investigator:</b>          | Yat Li        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

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.ucscop.com](http://www.ucscop.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>.<sup>1</sup> Attach SDS for each chemical included in this SOP.

*Any deviation from this SOP requires approval from the PI.*

|    |   |
|----|---|
| #1 | <p><b>Scope of Work/Activity:</b> 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 bubblers containing precursors (metal organic compounds) in the Metal Organic Chemical Vapor Deposition (MOCVD) system in the laboratory.</p> <p>Chemicals that fall under this SOP include:</p> <ul style="list-style-type: none"> <li>• Trimethylgallium (1445-79-0; Pyrophoric, water reactive)</li> <li>• Trimethylindium 3385-78-2; Pyrophoric, water reactive)</li> <li>• Trimethylaluminum (75-24-1; Pyrophoric, water reactive)</li> <li>• Bis(η<sup>5</sup>-cyclopentadienyl)magnesium (1284-72-6; Pyrophoric, water reactive)</li> </ul> <p>Note: For operation of the MOCVD system, refer to the document “SOP for Metal Organic Chemical Vapor Deposition (MOCVD) Operation”.</p>  |
| #2 | <p><b>Specific Safety and Environmental Hazards:</b> State specific hazards and potential consequences to person, environment, or property if procedure not followed.</p> <p>Release of metal organic precursors into the hood enclosure and/or lab can result in fire and chemical exposure. Trimethylgallium, Trimethylindium, Trimethylaluminum, Bis(η<sup>5</sup>-cyclopentadienyl)magnesium are pyrophoric and water reactive chemicals. These compounds ignite in air and may react explosively with water. They are always used in a closed, purged system. At normal temperature and pressure, trimethylgallium and trimethylaluminum are liquids, whereas, trimethylindium and bis(η<sup>5</sup>-cyclopentadienyl)magnesium are solids.</p> <p>These materials are extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Exposure may result in burning sensation, cough, wheezing, laryngitis, shortness of breath, spasm, inflammation and edema of the larynx, spasm, inflammation and edema of the bronchi, pneumonitis, pulmonary edema.</p> |

<sup>1</sup> If available, provide the Long Gold SDS version – contact EH&S for assistance.

|  | <p>Include applicable GHS symbols.<sup>2</sup></p>  |  |           |                  |                   |  |                 |                                |                  |  |  |  |
|--|---|--|-----------|------------------|-------------------|--|-----------------|--------------------------------|------------------|--|--|--|
|  | <p>Trimethylaluminum<br/>Trimethylindium<br/>Trimethylgallium<br/>Bis(<math>\eta</math>5-cyclopentadienyl)magnesium</p>   |  |           |                  |                   |  |                 |                                |                  |  |  |  |
| #3                                       | <p><b>Incompatible Conditions and Materials:</b> <i>List the incompatible conditions, chemicals, and/or materials that should be avoided.</i></p> <p><b>Note: the metal organic precursor in the bubbler will ignite spontaneously in air and will react violently with water. Do not open the inlet or outlet valve on the bubbler until it is securely installed in the purged MOCVD system.</b></p> <table border="1" data-bbox="170 892 1461 1155"> <thead> <tr> <th data-bbox="170 892 609 924">Chemical:</th> <th data-bbox="609 892 1461 924">Incompatibility:</th> </tr> </thead> <tbody> <tr> <td data-bbox="170 924 609 976">Trimethylaluminum</td> <td data-bbox="609 924 1461 976">Water, air and other oxidizers, alcohols, halogens</td> </tr> <tr> <td data-bbox="170 976 609 1018">Trimethylindium</td> <td data-bbox="609 976 1461 1018">Water, air and other oxidizers</td> </tr> <tr> <td data-bbox="170 1018 609 1071">Trimethylgallium</td> <td data-bbox="609 1018 1461 1071">Water, air and other oxidizers polyhalogenated compounds</td> </tr> <tr> <td data-bbox="170 1071 609 1155">Bis(<math>\eta</math>5-cyclopentadienyl)magnesium</td> <td data-bbox="609 1071 1461 1155">Water, air and other oxidizers, bases, acids</td> </tr> </tbody> </table>  |  | Chemical: | Incompatibility: | Trimethylaluminum | Water, air and other oxidizers, alcohols, halogens | Trimethylindium | Water, air and other oxidizers | Trimethylgallium | Water, air and other oxidizers polyhalogenated compounds | Bis( $\eta$ 5-cyclopentadienyl)magnesium | Water, air and other oxidizers, bases, acids |
| Chemical:                                | Incompatibility:  |  |           |                  |                   |  |                 |                                |                  |  |  |  |
| Trimethylaluminum                        | Water, air and other oxidizers, alcohols, halogens  |  |           |                  |                   |  |                 |                                |                  |  |  |  |
| Trimethylindium                          | Water, air and other oxidizers  |  |           |                  |                   |  |                 |                                |                  |  |  |  |
| Trimethylgallium                         | Water, air and other oxidizers polyhalogenated compounds  |  |           |                  |                   |  |                 |                                |                  |  |  |  |
| Bis( $\eta$ 5-cyclopentadienyl)magnesium | Water, air and other oxidizers, bases, acids  |  |           |                  |                   |  |                 |                                |                  |  |  |  |
| #4                                       | <p><b>Hazard Controls:</b> <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"> <li>The MOCVD system is installed in a walk-in fume hood. The metal organic precursors are used in a purged closed system within the fume hood.</li> </ul> <p><u>Administrative Controls</u></p> <ul style="list-style-type: none"> <li>Follow procedural steps listed in Section 7 and conduct leak check.</li> <li><b>Working alone restrictions:</b> Do not work alone while performing a bubbler change-out. Have a co-worker standing by ready to call for help, if needed.</li> <li><b>Precautions for safe handling:</b> <b>Note: the contents of the bubbler will ignite spontaneously in air and will react violently with water.</b> Follow the procedural steps in Section 7 to install or remove a bubbler from the MOCVD system. Do not open the inlet or outlet valve on the bubbler until it is securely installed in the purged MOCVD system.</li> <li><b>Conditions for safe storage:</b> Store the bubbler with VCR caps on the inlet and outlet valves in the original DOT packaging the bubbler was delivered in.</li> </ul> <p><u>Training &amp; Competency Requirements</u> <i>Describe necessary training and demonstration of competency for performing the hazardous operation.</i></p> |  |           |                  |                   |  |                 |                                |                  |  |  |  |

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

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|    | <ul style="list-style-type: none"> <li>• Complete EH&amp;S online or instructor-led “Introduction to Laboratory Safety” class</li> <li>• Review and sign Lab-Specific Training Checklist with PI, Lab Safety Representative, or other designated person.</li> <li>• Review SOP with knowledgeable person.</li> <li>• New users must be qualified by Yat Li or Yichuan Ling prior to working with the MOCVD system.</li> </ul> <p><u>Decontamination/Clean-Up</u><br/>Proper installation or removal of a bubbler will not lead to contamination requiring clean-up. Contact EHS before trying to clean-up after a precursor spill incident.</p> <p><u>Emergency shutdown procedure</u></p> <ul style="list-style-type: none"> <li>• If it is safe to do so, close the inlet valve, then the outlet valve on the bubbler.</li> <li>• Close the fume hood doors.</li> <li>• Press the red “Emergency Stop” button near the laboratory door to shut down all gas supply and shut off power to the MOCVD system.</li> </ul>   |
| #5 | <p><u>Personal Protective Equipment (PPE):</u> <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><b>Eye Protection:</b> ANSI-approved properly fitting safety glasses or goggles and full face shield during bubbler change out. Note: worker should not wear contact lenses.</p> <p><b>Skin and Body Protection:</b> 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><b>Hand Protection:</b> <i>State the appropriate chemical-resistant gloves for the material(s) that will be handled. For example:</i></p> <p>Wear Flame resistant gloves during bubbler 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:<br/><a href="http://www.microflex.com/Products/~media/Files/Literature/Domestic%20Reference%20Materials/DOM_Reference_Chemical%20Resistance.ashx">http://www.microflex.com/Products/~media/Files/Literature/Domestic%20Reference%20Materials/DOM_Reference_Chemical%20Resistance.ashx</a></p> <p>Additional Protection: Nomex lab coat.</p> |
| #6 | <p><u>Designated Area:</u> <i>Indicate the area designated for performing this process in the laboratory.</i></p> <p>The MOCVD system is installed in a walk-in fume hood. The precursor bubbler change out is done inside the hood.</p>  |
| #7 | <p><u>Important Steps to Follow:</u> <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><b>Note:</b> There is a possibility of metal organic precursor leakage from the bubbler if the bubbler is not installed properly and leak checked.</p>   |

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|    | <p><b>Note:</b> When installing a new bubbler or removing an old bubbler, the sequence for opening/closing the inlet and outlet valves is very important.</p> <ul style="list-style-type: none"> <li>• <b>When opening the valves</b>, the outlet valve should be opened first, so that the gas pressure inside the bubbler can be released through the outlet. If the inlet valve is opened first by mistake, the gas pressure inside the bubbler may push the liquid MO precursor out of the bubbler through the inlet.</li> <li>• <b>When closing the valves</b>, the inlet valve should be closed first before closing the outlet valve, so that the gas pressure inside the bubbler can be released through the outlet.</li> </ul> <p><b>Removing bubbler:</b></p> <ol style="list-style-type: none"> <li>1. Allow bubbler to come to room temperature.</li> <li>2. Close inlet valve on bubbler, then close outlet valve.</li> <li>3. Purge all system lines with nitrogen for 10 – 15 minutes before removing any connections.</li> <li>4. Remove the flexible lines connected to the bubbler inlet and outlet valves.</li> <li>5. Replace the VCR caps on the bubbler inlet and outlet valves.</li> <li>6. Remove the bubbler from chiller bath and replace into DOT approved packaging for shipment to vendor.</li> </ol> <p><b>Installing bubbler:</b></p> <ol style="list-style-type: none"> <li>1. Remove the bubbler from the protective DOT packaging in the fume hood.</li> <li>2. Secure bubbler in the chiller bath.</li> <li>3. Remove the VCR caps from the bubbler inlet and outlet valves.</li> <li>4. Connect the flexible lines to the bubbler inlet and outlet valves using new gaskets. <b>Do not</b> open the valves at this point.</li> <li>5. Purge all system lines with nitrogen for 10 – 15 minutes.</li> <li>6. Leak check all connections.</li> <li>7. Open the outlet valve on the bubbler first, then open the inlet valve.</li> </ol> |
| #8 | <p><u>Spill Clean-Up Procedures</u> (reference appended SDS as needed):</p> <p>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 <b>911</b> and ask dispatch to page EH&amp;S.</p> <p>In the event of a spill or gas release:</p> <ol style="list-style-type: none"> <li>1. Alert people in the laboratory to evacuate.</li> <li>2. Press the red “Emergency Stop” button by the door to shut down the MOCVD and stop gas flow.</li> <li>3. Close doors to affected area.</li> <li>4. Call for Emergency Response: 911</li> <li>5. Post with danger signs and have person knowledgeable of incident and laboratory assist emergency personnel</li> </ol>  |
| #9 | <p><u>Hazardous Waste(s):</u> <i>List expected concentrations and amounts of hazardous waste(s) generated during this process. Contact EH&amp;S for specific guidance regarding hazardous waste handling and disposal.</i></p> <p>Unreacted precursors in bubblers are to be shipped back to the supplier in their original DOT packaging.</p> <p>General hazardous waste management guidelines: <a href="http://ehs.ucsc.edu/programs/waste-">http://ehs.ucsc.edu/programs/waste-</a></p>  |

|                                | <p><a href="#">management/index.html</a><br/>State if the generated waste cannot be combined with any other waste streams.</p> <p><b>Waste Labeling</b></p> <ul style="list-style-type: none"> <li>Affix an on-line hazardous waste tag on all waste containers using the Online Tag Program (OTP) <a href="http://otp.ucop.edu/">http://otp.ucop.edu/</a> as soon as the first drop of waste is added to the container.</li> </ul> <p><b>Waste Storage</b></p> <ul style="list-style-type: none"> <li>Store hazardous waste in closed containers, in clean secondary containment, segregated by hazard class, in a marked and designated waste accumulation area.</li> <li>Double-bag dry waste using transparent bags.</li> <li>Waste accumulation area must be under the control of the person generating the waste.</li> </ul> <p><b>Waste Disposal</b></p> <ul style="list-style-type: none"> <li>Hazardous waste must be removed from the lab within 180 days.</li> <li>Containers must be clean, sealed, and safe to transport.</li> <li>Mark container as ready for pick up in OTP, move container to accumulation area.</li> <li>Contact EH&amp;S at x9-3086 for questions.</li> </ul>  |      |          |                       |               |                    |     |                   |                  |           |                |               |              |                                |                  |
|--------------------------------|--|------|----------|-----------------------|---------------|--------------------|-----|-------------------|------------------|-----------|----------------|---------------|--------------|--------------------------------|------------------|
| #10                            | <p><b>First Aid / Emergency Procedures:</b> Describe immediate First Aid or medical treatment required in case of personnel exposure.</p> <p>For immediate medical assistance, dial <b>911</b>. Report all serious injuries to EH&amp;S as soon as possible.</p> <ul style="list-style-type: none"> <li>If inhaled, move into fresh air immediately.</li> <li>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.</li> <li>If ingested, flush mouth with water (only if the person is conscious). Do not induce vomiting.</li> <li>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: <a href="http://risk.ucsc.edu/workers-comp/reporting-and-treatment.html">http://risk.ucsc.edu/workers-comp/reporting-and-treatment.html</a></li> <li>Seek medical attention immediately.</li> <li>Complete incident report form, <a href="http://risk.ucsc.edu/all-forms/wc-incident-report-form.pdf">http://risk.ucsc.edu/all-forms/wc-incident-report-form.pdf</a>, (contact EH&amp;S) and/or follow the instructions at the Risk Services website: <a href="http://risk.ucsc.edu/workers-comp/reporting-and-treatment.html">http://risk.ucsc.edu/workers-comp/reporting-and-treatment.html</a></li> </ul> <p><b>Laboratory Emergency Response Equipment:</b> 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="175 1375 1461 1602"> <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 |
| 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   |      |          |                       |               |                    |     |                   |                  |           |                |               |              |                                |                  |

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.*



x I understand that checking this box constitutes my approval of this document on 6/16/2015.

PI Signature/Approval: Yat Li

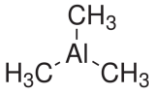
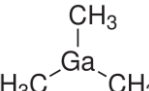
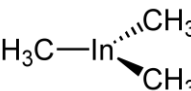

DATE

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) |
|---|-----------|----------------------------|---|---|--------------------------|----------------|-----------------------|---------------------|------------------------|-------------------|
| trimethyl-aluminum                              | 75-24-1   | Pyrophoric, water reactive | C <sub>3</sub> H <sub>9</sub> Al                |    | 72.09                    | 0.725          | L                     | 15                  | 128°C-130°C @ 50 mm Hg | - 17              |
| Trimethyl-gallium                               | 1445-79-0 | Pyrophoric, water reactive | C <sub>3</sub> H <sub>9</sub> Ga                |    | 114.83                   | 1.151          | L                     | - 15.8              | 55.7                   | N/A               |
| Trimethyl-indium                                | 3385-78-2 | Pyrophoric, water reactive | C <sub>3</sub> H <sub>9</sub> In                |   | 159.93                   | 1.57           | S                     | 88.4                | 113.8                  | N/A               |
| Bis(η <sup>5</sup> -cyclopentadienyl)-magnesium |           | Pyrophoric, water reactive | Mg(C <sub>5</sub> H <sub>5</sub> ) <sub>2</sub> |  | 154.49                   | N/A            | S                     | 180 -dec            | 50 @0.1 mm Hg          | N/A               |

## Exposure Limits/Toxicity Data

| Chemical   | Color | Odor | Cal/OSHA PEL                                | Toxicity LD <sub>50</sub> |
|--|-------|------|---|---------------------------|
| trimethylaluminum                                | N/A   | N/A  | 2 mg/m <sup>3</sup> (aluminum alkyls, NOC)  | N/A                       |
| Trimethylgallium                                 | N/A   | N/A  | NE  | N/A                       |
| Trimethylindium                                  | white | N/A  | 0.1 mg/m <sup>3</sup> (indium compounds)    | N/A                       |
| Bis(η <sup>5</sup> -cyclopenta-dienyl)-magnesium | white | N/A  | 10 mg/m <sup>3</sup> (magnesium oxide fume) | N/A                       |







**Revision History:**

| Version | Date       | Revision Author | Summary of Changes                 |
|---------|------------|-----------------|------------------------------------|
| 1       | 01/10/2013 | Nick Filipp     | Initial draft for review by Yat Li |
| 2       | 02/25/2013 | Nick Filipp     | Update template                    |
| 3       | 03/06/2013 | Lisa Wisser     | EH&S Review                        |
|         |            |                 |                                    |
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