#1 Brief Experimental Summary: Provide a general description of the process and/or experimental procedure.

MoO₃ is used as a catalyst to synthesize HₓTiO₂ and tetrabutylammonium hydroxide is used to react with HₓTiO₂ to yield TiO₂ nanosheets.

List the chemicals that fall under this SOP, include CAS#, and GHS symbols and categories:

<table>
<thead>
<tr>
<th>Chemical (CAS#)</th>
<th>GHS categories</th>
<th>GHS symbols — choose the appropriate symbols for each chemical</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium(IV) oxide (TiO₂) (13463-67-7)</td>
<td>Carcinogenicity (Category 2), H351</td>
<td>!</td>
</tr>
<tr>
<td>Potassium carbonate (K₂CO₃) (584-08-7)</td>
<td>Acute toxicity, Oral (Category 4), H302 Skin irritation (Category 2), H315 Eye irritation (Category 2A), H319 Specific target organ toxicity - single exposure (Category 3), Respiratory system, H335</td>
<td>!</td>
</tr>
<tr>
<td>Lithium carbonate (Li₂CO₃) (554-13-2)</td>
<td>Acute toxicity, Oral (Category 4), H302 Eye irritation (Category 2A), H319</td>
<td>!</td>
</tr>
<tr>
<td>Molybdenum(VI) oxide (MoO₃) (1313-27-5)</td>
<td>Eye irritation (Category 2A), H319 Carcinogenicity (Category 2), H351 Specific target organ toxicity - single exposure (Category 3), Respiratory system, H335</td>
<td>!</td>
</tr>
<tr>
<td>Hydrochloric Acid (HCl) (7647-01-0)</td>
<td>Corrosive to metals (Category 1), H290 Skin corrosion (Category 1B), H314 Serious eye damage (Category 1), H318 Specific target organ toxicity - single exposure (Category 3), Respiratory system, H335</td>
<td>![Corrosive]![Eye Irritation]![Specific Target Organ Toxicity]![Respiratory System]</td>
</tr>
<tr>
<td>Tetrabutylammonium hydroxide (2052-49-5)</td>
<td>Skin corrosion (Category 1B), H314 Serious eye damage (Category 1), H318</td>
<td>![Skin Corrosion]![Eye Damage]![Specific Target Organ Toxicity]![Respiratory System]</td>
</tr>
</tbody>
</table>

#2 Procedure Description: Include all steps for the procedure from the preparation to waste disposal, along with decontamination/clean-up steps. For each step’s description, include any step-specific hazard, personal protective equipment, engineering controls, designated work areas, and specific working alone restrictions in the left-hand columns. Note the location and use of any emergency response equipment specific to process (e.g., Calgonate gel, Class D fire extinguisher, inert absorbent material).

Working Alone: Working alone is not recommended. Notify your coworkers prior to conducting this work and ensure that at a
minimum of 1 person is nearby and aware that the work is occurring.

**Scale:** Work on as small a scale as possible. Do not exceed volumes/masses of 5 g, without prior consultation with and approval by the PI.

<table>
<thead>
<tr>
<th>Procedure Steps</th>
<th>Work Location / Safety Equipment</th>
<th>Precautions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Measure TiO(_2), K(_2)CO(_3), MoO(_3), and Li(_2)CO(_3) in a molar ratio of 10:2:1:1 for a total quantity of 5 g. put them into a corundum crucible.</td>
<td>198 PSB/lab coat, safety glasses CVD</td>
<td>MoO(_3) may be harmful if inhaled, Watch out the CVD temperature.</td>
</tr>
<tr>
<td>2. Heat the mixture in air to 900 (^\circ)C in CVD furnace and keep the temperature for 10 h.</td>
<td></td>
<td>Concentrated Acids are corrosive. If you need to dilute the concentration of an acid, <em>slowly</em> add the acid <em>to</em> the water. Heat will be evolved.</td>
</tr>
<tr>
<td>3. Mix the power with 0.008 mol/L HCl to get H(_x)TiO(_2)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4. Add 40% tetrabutylammonium hydroxide in the above solution</td>
<td></td>
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<tr>
<td>5. Filter the solution in the step four using centrifuge to get TiO(_2) nanosheets.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**#3 Personal Protective Equipment (PPE):** List the personal protective equipment used during this process.

*Note:* PPE is to be worn by those conducting the work and any adjacent personnel.

**Eye Protection:** ANSI-approved properly fitting safety glasses or goggles. Chemical splash goggles and/or full face shield during activities which pose a splash hazard.

**Body Protection:** An appropriately-sized lab coat must be worn and buttoned. Laboratory coat sleeves must be of sufficient length to prevent direct skin exposure while wearing gloves. Full length pants (or equivalent) and closed toe/heel shoe attire must be worn at all times by all workers who are occupying or entering a laboratory/technical area. The area of skin between the pants and shoe should not be exposed.

Check box for specialty lab coat: ☐ Nomex/Flame Resistant ☐ Biological Barrier ☐ Other Click here to enter text.

**Hand Protection:** Wear chemical-resistant gloves; remove gloves and wash hands with soap and water after use. Double gloves may provide additional protection for some chemicals. If prolonged contact or immersion is anticipated, consult with EH&S to identify appropriate protective gloves.

**Additional Protection:** ☐ Face Shield ☐ Chemical-Proof Apron ☐ Respiratory Protection ☐ Additional Gloves Click here to enter text.

**#4 Incompatible Conditions and Materials:** List the incompatible conditions, chemicals, and/or materials that should be avoided, along with the safe storage conditions.

Click here to enter text.

<table>
<thead>
<tr>
<th>Material:</th>
<th>Incompatibility:</th>
<th>Storage Conditions:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium(IV) oxide</td>
<td>Strong acids</td>
<td>Keep container tightly closed in a dry and well-ventilated place. Keep in a dry place</td>
</tr>
<tr>
<td>Potassium carbonate</td>
<td>Acids, Strong oxidizing agents</td>
<td>Keep container tightly closed in a dry and well-ventilated place.</td>
</tr>
<tr>
<td>Lithium carbonate</td>
<td>Incompatible with strong acids and oxidizing agents.</td>
<td>Keep container tightly closed in a dry and well-ventilated place.</td>
</tr>
<tr>
<td>Molybdenum(VI) oxide</td>
<td>Strong oxidizing agents, Strong acids</td>
<td>Keep container tightly closed in a dry and well-ventilated place.</td>
</tr>
<tr>
<td>Hydrochloric acid</td>
<td>Bases, Amines, Alkali metals, Metals, permanganates, e.g. potassium permanganate, Fluorine, metal acetylides, hexalithium disilicide</td>
<td>Store in a Corrosives-Acids cabinet, in secondary container (nalgene/polypropylene tray) with other mineral acids. Do not store</td>
</tr>
</tbody>
</table>
with organic acids (acetic acid, formic acid, propionic acid).

| Tetrabutylammonium hydroxide | Strong acids, Corrodes metal | Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage |

#5 Training: Training required for all personnel conducting this procedure. Include any specific training requirements.

- Complete EH&S online “Laboratory Safety Fundamentals” class available through the UC Learning Center (http://learningcenter.ucsc.edu/).
- Review and sign Lab-Specific Training Checklist (http://ehs.ucsc.edu/lab-safety-manual/training.html#lab-specific%20training) with PI, Lab Safety Representative, or other designated person.
- Review SOP with knowledgeable person.
- Complete training on specialized equipment prior to use (e.g., ultracentrifuge, hydrogenation apparatus).
- Other EH&S training requirements (e.g., Biosafety, Radiation Safety, Hazardous Waste Management).
- Click here to enter text.

#6 Clean-Up, Spill, and Emergency Response Procedures (reference the SDS as needed): Provide any specific information.

Decontamination/Clean-Up: Wash bench and/or work area with soap and water after using.

Specific Spill Clean-Up Procedures: Choose an item. Click here to enter text.

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.

- Isolate the area to prevent the spread of contamination (e.g. close doors to affected area, post warning signs, alert others in immediately vicinity to evacuate).
- Prevent spill from reaching drains or from spilling outside of the fume hood if possible to do so without exposing yourself to liquid or vapor.
- Clean the affected area and all exposed equipment with soap and water to remove any contaminants before resuming work.
- Spill clean-up materials should be disposed of as hazardous waste.

Laboratory Emergency Response Equipment: 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. List locations for nearest fire alarm pull and emergency shower/eyewash.

Fire alarm pull station: Outside the lab, face to the lab entrance
Emergency shower: Near the entrance of the lab area, besides several gas cylinders
Emergency eyewash: The same place as the emergency shower listed above.
Emergency Shutdown Procedures: NA

#7 Hazardous Waste(s): List expected concentrations and amounts of hazardous waste(s) generated during this process. Provide any special/specific waste management. Contact EH&S for specific guidance regarding hazardous waste handling and disposal. General hazardous waste management guidelines: http://ehs.ucsc.edu/programs/waste-management/index.html

Click here to enter text.

Waste Labeling

- Affix an on-line hazardous waste tag on all waste containers using the WASTe application https://ehs.ucop.edu/waste/ as soon as the first drop of waste is added to the container.

Waste Storage

- Store hazardous waste in closed containers with cap, 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.
Waste Disposal

- 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 WASTe, move container to accumulation area.
- Contact EH&S at x9-3086 or hazwaste@ucsc.edu with any questions.

#8 First Aid / Emergency Procedures: Describe immediate First Aid or medical treatment required in case of personnel exposure.

Click here to enter text.

For immediate medical assistance, dial 911. Report all serious injuries to EH&S as soon as possible.

- If inhaled, move into fresh air immediately.
- If ingested, flush mouth with water (only if the person is conscious).
- In the case of 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.

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.

- Prior to conducting any work with, the PI or designee must provide training to his/her laboratory personnel regarding the specific hazards involved in working with this substance, work area decontamination, and emergency procedures.
- The Principal Investigator must provide his/her laboratory personnel with a copy of this SOP and a copy of the SDS provided by the manufacturer.
- The Principal Investigator must ensure that his/her laboratory personnel have attended appropriate laboratory safety training or refresher training.

I have reviewed and approve this Standard Operating Procedure.

☒ I understand that checking this box constitutes my approval of this document on 4/13/2016

PI Signature/Approval: Yat Li

DATE 4/13/2016
## Chemical Information Summary

*Provide information for all chemicals included in the SOP. See the SDS for detailed toxicity information. Add more lines as needed.*

### Physical & Chemical Properties

<table>
<thead>
<tr>
<th>Chemical</th>
<th>CAS#</th>
<th>Molecular Formula</th>
<th>Structure</th>
<th>Molecular Weight (g/mol)</th>
<th>Density (g/mL)</th>
<th>Form (physical state)</th>
<th>Melting Point (°C)</th>
<th>Boiling point (°C)</th>
<th>Flash point (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium(IV) oxide</td>
<td>13463-67-7</td>
<td>TiO2</td>
<td>TiO2</td>
<td>79.87</td>
<td>4.26</td>
<td>nano particles</td>
<td>&gt; 350 (&gt; 662 °F)</td>
<td>No data available</td>
<td>No data available</td>
</tr>
<tr>
<td>Potassium carbonate</td>
<td>584-08-7</td>
<td>K2CO3</td>
<td>K₂CO₃</td>
<td>138.21</td>
<td>2.428 g/cm³</td>
<td>Crystalline</td>
<td>891 (1,636 °F) - lit.</td>
<td>No data available</td>
<td>No data available</td>
</tr>
<tr>
<td>Lithium carbonate</td>
<td>554-13-2</td>
<td>Li2CO3</td>
<td>Li₂CO₃</td>
<td>73.89</td>
<td>2.11 g/cm³</td>
<td>Crystalline</td>
<td>618 (1,144 °F) - lit.</td>
<td>No data available</td>
<td>No data available</td>
</tr>
<tr>
<td>Molybdenum(VI) oxide</td>
<td>1313-27-5</td>
<td>MoO3</td>
<td>MoO₃</td>
<td>143.94</td>
<td>No data available</td>
<td>Powder</td>
<td>795 (1,463 °F) - lit.</td>
<td>No data available</td>
<td>Not applicable</td>
</tr>
<tr>
<td>Hydrochloric Acid</td>
<td>7647-01-0</td>
<td>HCl</td>
<td>H-Cl</td>
<td>36.46</td>
<td>1.2</td>
<td>Liquid</td>
<td>-30 (~ -22 °F)</td>
<td>&gt; 100 (&gt; 212 °F) -</td>
<td>NA</td>
</tr>
<tr>
<td>Tetrabutylammonium hydroxide</td>
<td>2052-49-5</td>
<td>C16H37NO·OH</td>
<td></td>
<td>259.47</td>
<td>0.995</td>
<td>Liquid</td>
<td>&gt; 100 (&gt; 212 °F) at 1,013 hPa (760 mmHg)</td>
<td>No data available</td>
<td>No data available</td>
</tr>
</tbody>
</table>
## Exposure Limits/Toxicity Data

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Color</th>
<th>Odor</th>
<th>Cal/OSHA PEL</th>
<th>Toxicity LD50</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium(IV) oxide</td>
<td>White</td>
<td>No data</td>
<td>Particulates not otherwise regulated:</td>
<td>Oral - Rat - &gt; 10,000 mg/kg; Dermal - Rabbit - &gt; 10,000 mg/kg</td>
</tr>
<tr>
<td></td>
<td></td>
<td>available</td>
<td>10 mg/M$^3$, Total Dust</td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td>5 mg/M$^3$, Respirable fraction</td>
<td></td>
</tr>
<tr>
<td>Potassium carbonate</td>
<td>White</td>
<td>No data</td>
<td>No data available</td>
<td>Oral - Rat - 1,870 mg/kg</td>
</tr>
<tr>
<td>Lithium carbonate</td>
<td>White</td>
<td>No data</td>
<td>No data available</td>
<td>Oral - rat - 525 mg/kg; LC$_{50}$ Inhalation - rat - 4 h - &gt; 2.17 mg/l; Dermal - rat - male and female - &gt; 2,000 mg/kg</td>
</tr>
<tr>
<td>Molybdenum(VI) oxide</td>
<td>light grey</td>
<td>No data</td>
<td>0.5 mg/M$^3$, Molybdenum, soluble compounds, as Mo</td>
<td>Oral - Rat - male - 2,689 mg/kg; Oral - Rat - female - 3,830 mg/kg; LC$_{50}$ Inhalation - Rat - male and female - 4 h - &gt; 5.05 mg/l; Dermal - Rat - male and female - &gt; 2,000 mg/kg</td>
</tr>
<tr>
<td>Hydrochloric Acid</td>
<td>Light Yellow</td>
<td>Pungent</td>
<td>0.3 ppm; 2 ppm (Ceiling)</td>
<td>NA</td>
</tr>
<tr>
<td>Tetrabutylammonium hydroxide</td>
<td>Colorless</td>
<td>No data</td>
<td>No data available</td>
<td>No data available</td>
</tr>
</tbody>
</table>
**Documentation of Training** (signature of all users is required)

I have read and understand the content of this SOP:

<table>
<thead>
<tr>
<th>Name</th>
<th>Signature</th>
<th>Date</th>
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