UCSC
Laboratory Standard Operating Procedure (SOP)
Molybdenum (VI) oxide and Tetrabutylammonium hydroxide

Department:	Chemistry and Biochemistry	**Date:**	April 11th, 2016
Principal Investigator/Supervisor:	Yat Li	**Office Phone#:**	831-502-7363
Procedure Author:	Bin Yao	**Lab Phone#:**	831-502-7363
Location(s) covered by this SOP/Building/Room#:	PSB 198	**Author Email:**	byao4@ucsc.edu

Review any applicable manufacturer/vendor safety information, such as a Safety Data Sheet (SDS), before developing the Standard Operating Procedure (SOP).

Any deviation from this SOP requires approval from the PI.

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

MoO_3 is used as a catalyst to synthesize H_xTiO_2 and tetrabutylammonium hydroxide is used to react with H_xTiO_2 to yield TiO_2 nanosheets.

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

Chemical (CAS#)	GHS categories	GHS symbols – *choose the appropriate symbols for each chemical*
Titanium(IV) oxide (TiO_2) (13463-67-7)	Carcinogenicity (Category 2), H351	
Potassium carbonate (K_2CO_3) (584-08-7)	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	
Lithium carbonate (Li_2CO_3) (554-13-2)	Acute toxicity, Oral (Category 4), H302 Eye irritation (Category 2A), H319	
Molybdenum(VI) oxide (MoO_3) (1313-27-5)	Eye irritation (Category 2A), H319 Carcinogenicity (Category 2), H351 Specific target organ toxicity - single exposure (Category 3), Respiratory system, H335	
Hydrochloric Acid (HCl) (7647-01-0)	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	
Tetrabutylammonium hydroxide (2052-49-5)	Skin corrosion (Category 1B), H314 Serious eye damage (Category 1), H318	

#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.

Procedure Steps	Work Location / Safety Equipment	Precautions
1. Measure TiO_2, $K2CO_3$, MoO_3 and Li_2CO_3 in a molar ratio of 10:2:1:1 for a total quantity of 5 g, put them into a corundum crucible. 2. Heat the mixture in air to 900 ^oC in CVD furnace and keep the temperature for 10 h. 3. Mix the power with 0.008 mol/L HCl to get H_xTiO_2 4. Add 40% tetrabutylammonium hydroxide in the above solution 5. Filter the solution in the step four using centrifuge to get TiO_2 nanosheets.	**198 PSB/lab coat, safety glasses** **CVD**	**MoO_3 may be harmful if inhaled, Watch out the CVD temperature.** Concentrated Acids are corrosive. If you need to dilute the concentration of an acid***, slowly*** add the acid ***to*** the water. Heat will be evolved.

Chemical Equation Graphic (optional):
Click here to enter text.

#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. ☐ Other 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.

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

		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.
- 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.
- If ingested, flush mouth with water (only if the person is conscious).
- 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

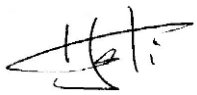
As the Principal Investigator, it is your responsibility to ensure that all individuals conducting this protocol are taught the correct procedures for safe handling of the hazardous materials involved. It is also your responsibility to ensure that your personnel complete Laboratory Safety Training and 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

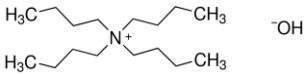
PI Signature/Approval: Yat Li DATE 4/13/2016



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

Chemical	CAS#	Molecular Formula	Structure	Molecular Weight (g/mol)	Density (g/mL)	Form (physical state)	Melting Point (℃)	Boiling point (℃)	Flash point (℃)
Titanium(IV) oxide	13463-67-7	TiO2	TiO_2	79.87	4.26	nano particles	> 350 (> 662 ℉)	No data available	No data available
Potassium carbonate	584-08-7	K2CO3	K_2CO_3	138.21	2.428 g/cm^3	Crystalline	891 (1,636 ℉) - lit.	No data available	No data available
Lithium carbonate	554-13-2	Li2CO3	Li_2CO_3	73.89	2.11 g/cm^3	Crystalline	618 (1,144 ℉) - lit.	No data available	No data available
Molybdenum(VI) oxide	1313-27-5	MoO3	MoO_3	143.94	No data available	Powder	795 (1,463 ℉) - lit.	No data available	Not applicable
Hydrochloric Acid	7647-01-0	HCl	H-Cl	36.46	1.2	Liquid	-30 (-22 ℉)	> 100 (> 212 ℉) - lit.	NA
Tetrabutylammonium hydroxide	2052-49-5	C16H37NO		259.47	0.995	Liquid	> 100 (> 212 ℉) at 1,013 hPa (760 mmHg)	No data available	No data available

Exposure Limits/Toxicity Data

Chemical	Color	Odor	Cal/OSHA PEL	Toxicity LD_{50}
Titanium(IV) oxide	White	No data available	Particulates not otherwise regulated: 10 mg/M^3, Total Dust 5 mg/M^3, Respirable fraction	Oral - Rat - > 10,000 mg/kg; Dermal - Rabbit - > 10,000 mg/kg
Potassium carbonate	White	No data available	No data available	Oral - Rat - 1,870 mg/kg
Lithium carbonate	White	No data available	No data available	Oral - rat - 525 mg/kg; LC_{50} Inhalation - rat - 4 h - > 2.17 mg/l; Dermal - rat - male and female - > 2,000 mg/kg
Molybdenum(VI) oxide	light grey	No data available	0.5 mg/M^3, Molybdenum, soluble compounds, as Mo	Oral - Rat - male - 2,689 mg/kg; Oral - Rat - female - 3,830 mg/kg; LC_{50} Inhalation - Rat - male and female - 4 h - > 5.05 mg/l; Dermal - Rat - male and female - > 2,000 mg/kg
Hydrochloric Acid	Light Yellow	Pungent	0.3 ppm; 2 ppm (Ceiling)	NA
Tetrabutylammonium hydroxide	Colorless	No data available	No data available	No data available

Documentation of Training (signature of all users is required)

I have read and understand the content of this SOP:

Name	Signature	Date