SBA-15
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when SBA-15 (SiO_2, CAS No. 7613-86-9) is used in laboratory. Its purpose is not to have any accident or risk. SBA-15 causes skin and eye irritation. It may be harmful if inhaled or if swallowed.

Synonyms: Mesoporous Silica

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Target Organ Effect (Lungs), Irritant
GHS Classification
- Acute toxicity, Oral (Category 5)
- Eye irritation (Category 2A)
- Specific target organ toxicity – single exposure (Category 3)

Signs and Symptoms of Exposure
Prolonged inhalation of crystalline silica may result in silicosis, a disabling pulmonary fibrosis characterized by fibrotic changes and military nodules in the lungs, a dry cough, shortness of breath, emphysema, decreased chest expansion, and increased susceptibility to tuberculosis. In advanced stages, loss of appetite, pleuritic pain, and total incapacity to work. Advanced silicosis may result in death due to cardiac failure or destruction of lung tissue. Crystalline silica is classified as group 1 "known to be carcinogenic to humans" by IARC and "sufficient evidence" of carcinogenicity by the NTP. The chronic health risks are associated with respirable particles of 3-4 um over protracted periods of time. Currently, there is a limited understanding of the mechanisms of quartz toxicity, including its mechanisms for lung carcinogenicity. Additional studies are needed to determine whether the cell transforming activity of quartz is related to its carcinogenic potential.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Respiratory Protection

For nuisance exposures use type P95 (US) particle respirator. For higher level protection use type OV/AGP99 (US) respirator cartridges. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US).

b. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

c. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

d. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Gloves must be inspected prior to use. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.
- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to “Injuries and Medical Treatment” Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with SBA-15 must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for SBA-15.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using SBA-15 must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of SBA-15 and understand the hazards.

Lab workers using SBA-15 must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with SBA-15 described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 10 g of this SBA-15 in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this SBA-15 with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material, the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using SBA-15. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

SBA-15 for ALD reactor

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggle.
2. Add SBA-15 (50 mg) into the sample holder of ALD reactor in the chemical hood.
3. Connect the sample holder with SBA-15 to the ALD reactor.
4. Tighten the 2.75 inch flange.
5. Start slowly pumping the system.
6. Heat the sample holder with SBA-15 to desired temperature.
7. Expose the sample with ALD cycles.

Synthesis of PtCu alloy

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (25 mL) under nitrogen in a fume hood and put a stir bar into it.
3. Put platinum(II) acetylacetonate (8.574 mg, 0.0218 *m*mol) into the flask.

4. Take Ethylene glycol (5 mL) by a syringe in a fume hood and add it slowly to the flask.
5. Rinse syringes several times with ethanol before disposal.
6. The washings should be disposed as hazardous organic waste.
7. Put a syringe needle into sharps-disposal container.
8. Put polyvinylpyrrolidone (0.6 mg) and copper sulfate pentahydrate (5.452 mg, 0.0218 *m*mol) into the flask.
9. Change the pH with NaOH (0.1 M) to pH=10 and close the flask with rubber septum.
10. Reflux the mixture under nitrogen for 2 h at 198 ^{o}C. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
11. Remove the septum from the flask and put SBA-15 (558.36 mg) in the flask and stir the mixture for 2 h.
12. Sonicate the mixture for 1 h.
13. Centrifuge and dispose the liquid.
14. Evacuate the flask containing the sample then dry the powder at ca. 60 ^{o}C for 12 h in the fume hood.
15. Grind the product to powder with an agate mortar and pestle.
16. Wash the powder with acetone and acetone-ethanol mixture upon sonication for 5 times and centrifuge each time after it's washed.
17. Dry the powder under vacuum in the flask at 60 ^{o}C for 12 h.

Synthesis of Cu@Pt nanoparticle

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (25 mL) under nitrogen in a fume hood.
3. Put copper acetylacetonate (2.04 mg, 0.0078 *m*mol) into the flask.
4. Take ethylene glycol (8 mL) by a syringe in a fume hood and add it slowly to the flask.
5. Rinse syringes several times with ethanol before disposal.
6. The washings should be disposed as hazardous organic waste.
7. Put a syringe needle into sharps-disposal container.
8. Put polyvinylpyrrolidone (5.8 mg) into the flask.
9. Change the pH with NaOH (0.1 M) to pH=10 and close the flask with rubber septum
10. Reflux the mixture under nitrogen for 20 minutes at 198 ^{o}C. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
11. After cooling to 80 ^{o}C, add platinum acetylacetonate (3.07 mg, 0.0078 *m*mol) and slowly heat the mixture back to reflux for 1 h.
12. Remove the septum from the flask and put SBA-15 (402.93 mg) in the flask and stir the mixture for 2 h.
13. Sonicate the mixture for 1 h.
14. Centrifuge and dispose the liquid.
15. Evacuate the flask containing the sample then dry the powder at ca. 60 ^{o}C for 12

h in the fume hood.
16. Grind the product to powder with an agate mortar and pestle.
17. Wash the powder with acetone and acetone-ethanol mixture upon sonication for 5 times and centrifuge each time after it's washed.
18. Dry the powder under vacuum in the flask at 60 °C for 12 h.

Synthesis of Pt@Cu nanoparticle

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (25 mL) under nitrogen in a fume hood.
3. Put platinum acetylacetonate (18.582 mg, 0.04725 *m*mol) into the flask.
4. Take ethylene glycol (5.775 mL) by a syringe in a fume hood and add it slowly to the flask.
5. Rinse syringes several times with ethanol before disposal.
6. The washings should be disposed as hazardous organic waste.
7. Put a syringe needle into sharps-disposal container.
8. Put polyvinylpyrrolidone (2.82 mg) into the flask.
9. Change the pH with NaOH (0.1 M) to pH=10 and close the flask with rubber septum
10. Reflux the mixture under nitrogen for 2 h at 198 °C. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
11. After cooling to 80 °C, add copper sulfate pentahydrate (11.814 mg, 0.04725 *m*mol) and slowly heat the mixture back to reflux for 2 h.
12. Remove the septum from the flask and put SBA-15 (598.455 mg) in the flask and stir the mixture for 2 h.
13. Sonicate the mixture for 1 h.
14. Centrifuge and dispose the liquid.
15. Evacuate the flask containing the sample then dry the powder at ca. 60 °C for 12 h in the fume hood.
16. Grind the product to powder with an agate mortar and pestle.
17. Wash the powder with acetone and acetone-ethanol mixture upon sonication for 5 times and centrifuge each time after it's washed.
18. Dry the powder under vacuum in the flask at 60 °C for 12 h.

Catalytic Hydrogenation of Cinnamaldehyde

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Catalyst (50–200 mg), cinnamaldehyde (0.5–3 *m*mol), and solvent (100 mL) are added into a reactor.
3. Sonicate and stir the mixture.
4. The mixture is purged with pure H_2 (1.0 MPa) five times.
5. The reactor is pressurized to a desired H_2 pressure (2.0 MPa) at room temperature.

6. The reactor is heated to a desired temperature.
7. Begin stirring (900 rpm) and set reaction time to start.
8. Sample (1.0 mL) is taken periodically to determine conversion and selectivity during the reaction process.
9. The catalyst powder is filtered off.
10. The filtrate is analyzed using GC.

Surface Modification

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. SBA-15 (1 g) is calcined under vacuum at 200 °C for 2 h.
3. Into the beaker with stir bar, anhydrous toluene (25 mL) and 3-tert-butyloxy carbonylaminopropyltriethoxysilane (0.5 or 1.5 g) are introduced into the flask.
4. Put SBA-15 into the same flask.
5. The reaction is conducted under N2 atmosphere pressure at 110 °C for 48 h.
6. The mixture is filtered, washed with toluene, and dried under vacuum overnight.

Preparation #1 of Ni/SBA-15

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Prepare nickel (II) nitrate hexahydrate (0.01 M, $Ni(NO_3)_2 \cdot 6H_2O$) aqueous solution
3. Add the nickel (II) nitrate hexahydrate aqueous solution (2 mL) into SBA-15 (0.2 g).
4. The slurry is stirred for a while.
5. Leave the slurry at room temperature for 1 day.
6. Dry the slurry in the oven overnight.
7. The powder is calcined at 550 ^oC under air for 6 h.

Preparation #2 of Ni/SBA-15

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Add nickel nitrate hexahydrate (0.01 g, $Ni(NO_3)_2 \cdot 6H_2O$), nitric acid (1.25 mL, HNO_3) and SBA-15 (0.2 g) into water (25 mL).
3. The mixture is sonicated for a while.
4. The mixture is heated to 55 ^oC in a flask.
5. Add urea (1.2012 g) into water (25 mL).
6. Add the urea solution to the first solution (step 2).
7. The mixture is heated to 90 ^oC for 4 h.
8. Cool down to 55 ^oC.

9. The mixture is stirred at 55 °C for 10 min.
10. Cool down the mixture to room temperature.
11. Filter the mixture.
12. Wash the powder for 3 times with water.
13. Dry the powder overnight.
14. The powder is calcined at 550 °C under air for 3 h.

epi-Quinine Tethering

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle
2. 9-amino(9-deoxy)*epi*-quinine (1.08 g, 0.3 *m*mol) and AIBN (azoisobutyronitrile) (54 mg, 0.33 *m*mol) is put into a 50 mL flask.
3. Chloroform (15 mL) is added into the flask.
4. (3-Mercapto)propyltriethoxysilane (0.87 mL, 3.6 *m*mol) is added into the flask.
5. The flask is refluxed at 63 °C for 1 day.
6. The flask is cooled down to room temperature.
7. Chloroform is removed by evaporator.
8. SBA-15 (100 mg) is mixed with the product.
9. Toluene (10 mL) is added to the flask.
10. The mixture is refluxed at 113 °C for 1 day.
11. The solid is filtered and washed by toluene.
12. The powder product is dried under vacuum for 5 h.

SBA-15 Silylation 1

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle
2. Add SBA-15 (200 mg) and two magnetic stir bars to two round-bottom flasks (50 mL).
3. Create inert N_2 or Ar atmosphere in each flask using a combination of a syringe and vent needle. Allow gas to cycle through the flask for 5-10 min.
4. Add toluene (5 mL) to each flask.
5. Add triethylamine (0.3 mL) to each flask.
6. Add Tchlorotrimethylsilane 0.2 mL to one flask and 0.1 mL to the other flask.
7. Allow reaction to stir over magnetic stir plate for 24 hours.
8. Expose both flasks to air and quench each reaction with methanol (5 mL).
9. Vacuum filter both mixtures and wash powder with acetone (10 mL) and DI water for each.
10. Dry both sets of powders by vacuum filtering for 5-10 min.
11. Set each sample (10 mg) aside in a plastic storage container.
12. Set each sample (10 mg) aside in a plastic storage container.

SBA-15 Silylation 2

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle
2. SBA-15 (200 mg) is put into a round-bottom flask (25 mL).
3. The flask is purged with Ar gas for 5 minutes.
4. Toluene (5 mL) is added to the flask
5. Triethylamine (0.3 mL) and chlorotrimethylsilane (0.2 mL) are added to the flask.
6. The resulting mixture is stirred for 24 hours
7. Then the mixture is exposed to air and quenched with methanol (5 mL).
8. The mixture is filtered and washed with water (10 mL) and acetone (10 mL).
9. Finally the mixture is left to dry under vacuum.

SBA-15 Impregnation

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle
2. Prepare a stock solution (0.1 M) and a standard solution (0.01 M) of $Ni(NO_3)_2*6H_2O$.
3. Add a Ni standard solution (0.01 M, 2 mL) to both sets of SBA-15 (200 mg) powder samples into two beakers (100 mL).
4. Stir slurries well for 5-10 min.
5. Leave slurries at room temperature for 24 hours.
6. Dry both sample sets in the oven overnight to drive off the waters of hydration.
7. Calcinate the powder samples, one at a time, in a furnace tube at 550 ^oC under air for ~ 6 hours.
8. Characterize samples by TEM and FTIR.

Ni/SBA-15 catalyst (10 wt.%)

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, face mask, and safety goggle.
2. Bring Nickel (II) nitrate, $Ni(NO_3)_2{\cdot}6H_2O$, bottle and DI water (or ethanol) to the fume hood in room 135.
3. Dissolve nickel nitrate (0.0495 g) precursor in DI water (5 mL) for 10 wt.% Ni/SBA-15 catalyst.
4. Add ethanol (5 mL) to SBA-15 (100 g) in a shallow beaker and mix vigorously while adding the Ni precursor solution.
5. Stir until the resulting slurry is well-mixed and evaporate the ethanol by heating the sample on a hot plate.

6. Collect the dried powder onto a combustion boat and dry over-night in the oven (70 °C).
7. Transfer sample to the muffle furnace (Prof. Yin lab) and calcinate for 5 h at 500 °C (2.5 °C / min).

NiO deposition on SBA-15 via ALD reactor

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, **AND** a proper face mask at all times (Covid-19) while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
2. Clean the sample holder with acetone
3. Place SBA-15 in the sample holder.
4. Preheat the support (SBA-15) at 200 ℃ for 2 h.
5. Ni precursor (nickel(II) bis(2,2,6,6-tetramethyl-3,5-heptanedionate, Ni(tmhd)) is pretreated at 165 ℃ in an oil bath.
6. The reactor is set to 150 ℃.
7. Dose the Ni precursor for 30 min by using Ar carrier gas (200 mTorr).
8. Purge the reactor with Ar gas (500 mTorr) for 5 min.
9. Dose deionized water (100 mTorr) for 2 min.
10. Purge the reactor with Ar gas (500 mTorr) for 10 min.
11. Repeat 7–10 steps repeatedly until desired growth is obtained.

NiO deposition on TMSDMA/SBA-15, HMDS/SBA-15, or ODTS/SBA-15 via ALD reactor

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, **AND** a proper face mask at all times (Covid-19) while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
2. Clean the sample holder with acetone
3. Place SBA-15 in the sample holder.
4. Preheat the support (SBA-15) at 200 ℃ for 2 h.
5. Ni precursor (nickel(II) bis(2,2,6,6-tetramethyl-3,5-heptanedionate) is pretreated at 165 ℃ in an oil bath.
6. The reactor is set to 150 ℃.
7. Dose N,N-dimethyltrimethylsilylamine (TMSDMA, 50 mTorr), hexamethyl-disilazane (HMDS, 50 mTorr), or octadecyltrichlorosilane (ODTS, 50 mTorr) for 30 s.
8. Dose the Ni precursor for 30 min by using Ar carrier gas (200 mTorr).
9. Purge the reactor with Ar gas (500 mTorr) for 5 min.
10. Dose deionized water (100 mTorr) for 2 min.
11. Purge the reactor with Ar gas (500 mTorr) for 10 min.

12. Repeat 8–11 steps repeatedly until desired growth is obtained.

SBA-15 Impregnation

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, **AND** a proper face mask at all times (Covid-19) while inside the lab. Carry out the following procedures in a fume hood.
2. Clean glassware, such as flask and beaker with acetone.
3. Prepare nickel(II) nitrate hexahydrate solution (2.5 mL, 0.84 M).
4. Pour SBA-15 (100 mg) in a beaker (50 mL)
5. Add ethanol (5 mL) or DI water:ethylene glycol (1:1, 5 mL) to the beaker.
6. Add the desired amount of the Ni solution (20 μL for 1 wt.% Ni loading)
7. The mixture was stirred at 80 ℃ until the ethanol evaporated.
8. The collected powder is transferred to the oven for drying overnight.
9. Calcine the powder in air condition (muffle furnace (Prof. Yin lab) at 500 ℃ for 5 hours (2.5 ℃/m speed).
10. Collect the sample from the furnace.
11. Clean up the flasks with solvents (distilled water, ethanol and acetone).

Tetrakis(ethylmethylamino)hafnium(IV) for ALD reactor

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Put SBA-15 (50 mg) into a sample holder in the fume hood of room 135.
3. Spotweld mesh on the sample holder.
4. Attach the sample holder to the ALD reactor.
5. Preheat SBA-15 at 200–300 ºC for 2 h.
6. Take tetrakis(ethylmethylamido)hafnium (TEMAH) into a glass sample tube in the glove box.
7. Fix the tube on the ALD reactor in room 143.
8. Turn on the pump and degas the precursor.
9. Heat the container in a silicon-oil bath up to 60 ºC.
10. Open the valve to introduce TEMAH (1.6 s) and nitrogen (5 s) into the ALD chamber.
11. After reaction, slowly cool down precursor, chamber, and sample to room temperature.
12. Collect and transfer sample into a vial for analysis.
13. Collect the waste of TEMAH and dispose into a proper waste bottle.

Synthesis of 3-aminopropyltriethoxysilane-grafted SBA-15

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (100 mL) under nitrogen.
3. Pour ethanol (50.0 g) into the flask
4. Add 3-aminopropyltriethoxysilane (2.5 g) into the flask.
5. Put SBA-15 (1.0 g) into the flask.
6. Prepare a reflux condenser apparatus in a fume hood.
7. Mix and reflux the mixture for 24 h. If left unattended in a fume hood, put a label with chemical name and hazard information.
8. Filter and wash the mixture with ethanol.
9. Dispose waste in the properly labeled container.
10. Dry the white powder.

TiO_2/SBA-15

12. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, and a proper face mask at all times while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
13. Clean the sample holder with acetone before placing SBA-15 in the sample holder.
14. Preheat the support (SBA-15) up to 200 ^oC for 2 h.
15. Ti precursor (tetrakis(dimethylamido) titanium, TDMAT) is set to 42 ^oC
16. The reactor is set to 102 ^oC
17. Dose TDMAT for 20 min at 200 mTorr.
18. Purge the reactor with Ar gas for 50 min at 500 mTorr.
19. Dose deionized water vapor for 2 min at 100 mTorr.
20. Purge the reactor with Ar gas for 50 min at 500 mTorr.
21. Repeat 6–9 steps repeatedly until desired growth is obtained.

TiO_2/TMSDMA, HMDS or ODTS/SBA-15

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, and a proper face mask at all times while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
2. Clean the sample holder with acetone before placing SBA-15 in the sample holder.
3. Preheat the support (SBA-15) at 200 ^oC for 2 h.
4. Ti precursor (tetrakis(dimethylamido) titanium, TDMAT) 42 ^oC, using heating tape and r
5. The reactor is set to 102 ^oC.
6. Dose an inhibitor reagent (trimethylsilyl dimethylamine, hexamethyldisilazane, or octadecyltrichlorosilane) for 30 s at 50 mTorr.

7. Dose TDMAT for 20 min at 200 mTorr.
8. Purge the reactor with Ar gas for 50 min at 500 mTorr.
9. Dose deionized water vapor for 2 min at 100 mTorr.
10. Purge the reactor with Ar gas for 50 min at 500 mTorr.
11. Repeat 7–10 steps repeatedly until desired growth is obtained.

Synthesis of Cu/SBA-15

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Put the L-ascorbic acid (1 g) into a flask (50 mL).
3. Add water (12.5 mL) to the flask.
4. Prepare a copper nitrate hemi(pentahydrate), $Cu(NO_3)_2{\cdot}2.5H_2O$, solution (20 mg/mL).
5. Add the copper precursor solution (0.64 mL) into the flask.
6. Add SBA-15 (0.1 g) into the flask.
7. Stir (200 rpm) at RT for 48 h.
8. Wash the mixture with H_2O/ethanol 2 times
9. Dry the powder.

Synthesis of $Pt@Cu_6$/SBA-15, #1

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Put the L-ascorbic acid (1 g) in to a flask (50 mL).
3. Add water (12.5 mL) to the flask.
4. Add Pt nanoparticle solution (15 mL) into the flask.
5. Prepare a copper nitrate hemi(pentahydrate), $Cu(NO_3)_2{\cdot}2.5H_2O$, solution (20 mg/mL).
6. Add the copper precursor solution (0.64 mL) into the flask.
7. Stir (200 rpm) the mixture at RT for 16 h.
8. Add SBA-15 (0.1 g) into the flask.
9. Stir (200 rpm) at RT for 48 h.
10. Wash the mixture with H_2O/ethanol 2 times
11. Dry the powder.

Synthesis of 3-aminopropyltriethoxysilane-grafted SBA-15

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a 3-neck round-bottom flask (50 mL).
3. Pour ethanol (30 mL) into the flask

4. Add 3-aminopropyltriethoxysilane (1.5 mL) into the flask.
5. Put SBA-15 (200 mg) into the flask.
6. Prepare a reflux condenser apparatus in a fume hood.
7. Mix and reflux the mixture at 73 ^{o}C for 24 h. If left unattended in a fume hood, put a label with chemical name and hazard information.
8. Transfer the mixture into a centrifuge tube.
9. Centrifuge (3000 rpm) for 10 min.
10. Take out the excess solvent using a pipette.
11. Wash the particles with ethanol (25 mL).
12. Centrifuge (3000 rpm) for 10 min.
13. Take out the excess ethanol using a pipette.
14. Repeat steps 11-13 once more.
15. Dispose waste in the properly labeled container.
16. Dry the white powder in a vacuum chamber.

Synthesis of Au nanoparticles within the SBA-15 support

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry two flasks (250 mL) under nitrogen.
3. Put deionized water (100 mL) into one flask.
4. Add 3-aminopropyltriethoxysilane-grafted SBA-15 (1.0 g) into the flask
5. After the powder is dispersed evenly, add gold chloride solution (15 mL, 10 mM).
6. Maintain the suspension by stirring the mixture for 2 h at room temperature. If left unattended in a fume hood, put a label with chemical name and hazard information.
7. Transfer the mixture into a centrifuge tube.
8. Centrifuge (3000 rpm) for 10 min.
9. Take out the excess solvent using a pipette.
10. Wash the particles with DI water (25 mL).
11. Centrifuge (3000 rpm) for 10 min.
12. Take out the excess DI water using a pipette.
13. Repeat steps 10-12 once more.
14. Dispose waste in the properly labeled waste container.
15. Dry the yellow powder in a vacuum chamber.
16. Dry a clean beaker (150 mL).
17. Put deionized water (100 mL) into the other flask.
18. Redisperse the solid into the flask.
19. For the reduction reaction, add sodium borohydride (2.5 g) into the flask.
20. Maintain suspension by stirring the mixture for 2 h at RT. If left unattended in a fume hood, put a label with the chemical names and hazard information.
21. Transfer the mixture into a centrifuge tube.

22. Centrifuge (3000 rpm) for 10 min.
23. Take out the excess solvent using a pipette.
24. Wash the particles with DI water (25 mL).
25. Centrifuge (3000 rpm) for 10 min.
26. Take out the excess DI water using a pipette.
27. Repeat steps 24-26 once more.
28. Dry the reddish-purple powder in a vacuum chamber.
29. Dispose waste in the properly labeled waste container.
30. After the powder is dry, calcinate the sample at 500 ^{o}C for 6 h. If left unattended, leave a label nearby with the chemical names and hazard information.
31. After cooling, collect the sample.

Silylation Procedure for SBA-15

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. A flask is dried.
3. Put SBA-15 (200 mg) in the flask.
4. Fill the flask with toluene or ethanol (30 mL).
5. Slowly add hexamethyldisilazane or N,N-dimethyltrimethylsilylamine (2 mL) into the flask in the fume hood.
6. The mixture is refluxed at 90 ^{o}C for 24 h.
7. The mixture is cooled down to room temperature.
8. The solution is centrifuged for 10 min to remove the solvent.
9. Collect the powder and wash with isopropyl alcohol and DI water 3 times.
10. Vacuum filter the mixture overnight.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 9/24/2015, updated 11/01/2016, 10/10/2017, 07/01/2018, 01/26/2019, 02/07/2020, 07/08/2020, 07/10/2020, 10/12/2021, 10/15/2021, 10/19/2021, 12/21/2021, 01/21/2022, 08/24/2022

Silicon oil
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when silicon oil (poly(dimethylsiloxane), CAS No. 63148-62-9) is used in laboratory. Its purpose is not to have any accident or risk. Silicon oil is harmful if inhaled, swallowed, or absorbed through skin. It may cause eye, skin, or respiratory tract irritations.

Synonyms: Poly(dimethylsiloxane)

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: not known
GHS Classification
Not a dangerous substance according to GHS

Signs and Symptoms of Exposure
Prolonged or repeated exposure to skin causes defatting and dermatitis., Dizziness, To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.
- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with silicon oil must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.

- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for silicon oil.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using silicon oil must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of silicon oil and understand the hazards.

Lab workers using silicon oil must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with silicon oil described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 100 g of this silicon oil in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this silicon oil with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using silicon oil. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Oil bath

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggle.
2. Silicon Oil is used as the boiling oil in the oil bath, which is used to heat the chemical precursor.
3. Pour the Silicon Oil into a beaker (250 mL). Do not over fill and the volume of the liquid will expand once it is heated.
4. Put the beaker under the precursor container to make sure the entire container is merged into the silicon oil.
5. Heat the beaker using heating stage or heating tape to desired temperature (above 200 ºC).
6. Once finish, stop heating and cover the beaker with aluminum foil.

NiO deposition on SBA-15 via ALD reactor:

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, **AND** a proper face mask at all times (Covid-19) while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
2. Clean the sample holder with acetone
3. Place SBA-15 in the sample holder.
4. Preheat the support (SBA-15) at 200℃ for 2 h.
5. Ni precursor (nickel(II) bis(2,2,6,6-tetramethyl-3,5-heptanedionate, Ni(tmhd)) is pretreated at 165 ℃ in an oil bath.
6. The reactor is set to 150 ℃.
7. Dose the Ni precursor for 30 min by using Ar carrier gas (200 mTorr).
8. Purge the reactor with Ar gas (500 mTorr) for 5 min.
9. Dose deionized water (100 mTorr) for 2 min.
10. Purge the reactor with Ar gas (500 mTorr) for 10 min.
11. Repeat 7–10 steps repeatedly until desired growth is obtained.

NiO deposition on TMSDMA/SBA-15, HMDS/SBA-15, or ODTS/SBA-15 via ALD reactor

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, **AND** a proper face mask at all times (Covid-19) while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
2. Clean the sample holder with acetone
3. Place SBA-15 in the sample holder.
4. Preheat the support (SBA-15) at 200 ℃ for 2 h.
5. Ni precursor (nickel(II) bis(2,2,6,6-tetramethyl-3,5-heptanedionate) is pretreated at 165 ℃ in an oil bath.
6. The reactor is set to 150 ℃.
7. Dose N,N-dimethyltrimethylsilylamine (TMSDMA, 50 mTorr), hexamethyl-disilazane (HMDS, 50 mTorr), or octadecyltrichlorosilane (ODTS, 50 mTorr) for 30 s.
8. Dose the Ni precursor for 30 min by using Ar carrier gas (200 mTorr).
9. Purge the reactor with Ar gas (500 mTorr) for 5 min.
10. Dose deionized water (100 mTorr) for 2 min.
11. Purge the reactor with Ar gas (500 mTorr) for 10 min.
12. Repeat 8–11 steps repeatedly until desired growth is obtained.

Tetrakis(ethylmethylamino)hafnium(IV) for ALD reactor

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Put SBA-15 (50 mg) into a sample holder in the fume hood of room 135.
3. Spotweld mesh on the sample holder.
4. Attach the sample holder to the ALD reactor.
5. Preheat SBA-15 at 200–300 ºC for 2 h.
6. Take tetrakis(ethylmethylamido)hafnium (TEMAH) into a glass sample tube in the glove box.
7. Fix the tube on the ALD reactor in room 143.
8. Turn on the pump and degas the precursor.
9. Heat the container in a silicon-oil bath up to 60 ºC.
10. Open the valve to introduce TEMAH (1.6 s) and nitrogen (5 s) into the ALD chamber.
11. After reaction, slowly cool down precursor, chamber, and sample to room temperature.
12. Collect and transfer sample into a vial for analysis.
13. Collect the waste of TEMAH and dispose into a proper waste bottle.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2013, updated 03/01/2014, 03/01/2016, 10/12/2021, 10/15/2021

Sodium ethanethiolate
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when sodium ethanethiolate (C_2H_5NaS, CAS No. 811-51-8) is used in laboratory. Its purpose is not to have any accident or risk. Sodium ethanethiolate is corrosive solid. It causes severe skin burns and eye damage. Do not breath dust or mist.

Synonyms: Ethanethiolsodium salt, Ethyl mercaptansodium salt

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Corrosive
GHS Classification
Skin corrosion (Category 1B)
Serious eye damage (Category 1)

Signs and Symptoms of Exposure
Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Spasm, inflammation and edema of the larynx, spasm, inflammation and edema of the bronchi, pneumonitis, pulmonary edema, burning sensation, Cough, wheezing, laryngitis, Shortness of breath, Headache, Nausea, Vomiting.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove.Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.

- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with sodium ethanethiolate must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for sodium ethanethiolate.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using sodium ethanethiolate must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of sodium ethanethiolate and understand the hazards.

Lab workers using sodium ethanethiolate must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with sodium ethanethiolate described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;

3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 25 g of this sodium ethanethiolate in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this sodium ethanethiolate with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using sodium ethanethiolate. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Synthesis from QD-Bn to QD-a

1. Wear nitrile chemical–resistant gloves, a flame-resistant lab coat, and a safety goggle.
2. **QD-Bn** (8.0 *m*mol), sodium ethanethiolate ($NaSC_2H_5$, 4 eq.) and dimethylform-amide (50 mL) are added to a flask (200 mL).
3. The mixture is stirred at 110 ^oC until TLC analysis shows that the starting material is completely consumed within 4–6 h.
4. The mixture is cooled down to room temperature.
5. Ammonium chloride (NH4Cl, 40 mL) and water (50 mL) are added to the flask.
6. The pH value of the mixture is determined to be around 7.
7. The mixture is extracted by ethyl acetate (2×200 mL).
8. The organic phase is washed with brine (4×50 mL).
9. The organic phase is dried over sodium sulfate (Na_2SO_4) and concentrated in vacuum.
10. **QD-a** is extracted from the residue by column chromatograph with a solution (methanol:ethyl acetate=1:50 to 1:10).

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 02/01/2020

Sodium hydroxide
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when sodium hydroxide (NaOH, CAS No. 1310-73-2) is used in laboratory. Its purpose is not to have any accident or risk. Sodium hydroxide is corrosive solid. It causes severe skin burns and eye damage. Material is extremely destructive to the tissue of the mucous membranes and upper respiratory tract. Also, It may be harmful if inhaled, if absorbed through skin, or if swallowed.

Synonyms: Caustic soda

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Corrosive
GHS Classification
- Skin corrosion (Category 1A)
- Serious eye damage (Category 1)
- Acute aquatic toxicity (Category 3)

Signs and Symptoms of Exposure
Spasm, inflammation and edema of the larynx, spasm, inflammation and edema of the bronchi, pneumonitis, pulmonary edema, burning sensation, Cough, wheezing, laryngitis, Shortness of breath, Headache, Nausea, Vomiting, Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection
ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection
Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.

- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with sodium hydroxide must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for sodium hydroxide.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using sodium hydroxide must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of sodium hydroxide and understand the hazards.

Lab workers using sodium hydroxide must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with sodium hydroxide described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;

3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 25 g of this sodium hydroxide in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this sodium hydroxide with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using sodium hydroxide. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Sodium Hydroxide Solution Preparation

1. Wear dust respirator, gloves, flame-resistant lab coat, and safety goggles.
2. Bring sodium hydroxide to the balance. Transfer sodium hydroxide into the designated vessel quickly.
3. Close and seal the bottle and put it back.
4. Discard extra sodium hydroxide to designated chemical waste container. Clean the balance with brush.
5. Use designated solvent to dissolve sodium hydroxide and the extra unused solution needs to be treated as hazardous waste.

Preparation of 2 M sodium hydroxide solution for silica etching

1. Wear nitrile chemical-resistant glove, mask, flame-resistant lab coat, and safety goggles.
2. Weigh proper amount of sodium hydroxide.
3. Put the sodium hydroxide into a beaker.
4. Pour a proper amount of milli-Q water in the beaker and stir smoothly.

4-Methyl-2,6-heptanedione

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Add sodium (1.8 g, 50 *m*mol) and ethanol (24 mL) into a flask (100 mL).
3. Add 2,4,6-trimethylpyridine (6.5 mL, 50 *m*mol) into the flask.
4. Reflux the mixture for 1 h at 90 ^{o}C in oil bath.
5. Prepare a solution of hydroxylamine hydrochloride (3.6 g, 53 *m*mol) in 50% ethanol (6.4 mL) and HCl (3.2 mL) in 95% ethanol (6.4 mL).
6. Add the solution slowly into the flask.

7. Reflux the mixture for 2.5 h.
8. Cool down the mixture to room temperature.
9. Remove ethanol from the mixture under evaporator.
10. Add a NaOH solution (3.5 g in 50 mL water) to the residue.
11. Extract the solution with ether (50 mL).
12. Acidify the aqueous solution with 10% H_2SO_4 (35 mL).
13. Add sodium nitrite solution (3.5 g, 50 *m*mol in 10 mL water) into the mixture.
14. Stir the mixture for 1 h at 0 ^oC.
15. Extract the mixture with ether (20 mL, 4 times)
16. Wash the organic phase with water and brine.
17. Column the residue with a hexane solution (hexane:ether = 10:2).

Au Nanoparticle (2 nm) Preparation

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. In a round-bottom flask, Milli-Q water (45.5 mL), NaOH (0.2 M, 1.5 mL), tetrakis(hydroxymethal)phosphonium chloride (1 mL, 120 μL diluted in 10 mL) are added in sequence.
3. The mixture is stirred for 2 min
4. Chloroauric acid solution (25 mM, 2 mL) is added.
5. The colloidal nanoparticle suspension is further stirred for 2 min
6. The suspension is stored in a plastic centrifuge tube in dark at room temperature.
7. Clean the reaction vessel with aqua regia after reaction.

Preparation of sodium hydroxide solution

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Review the SDS of sodium hydroxide and hydrochloric acid again; especially remind first aid measures, handling and storage, & PPE.*
3. Take an Erlenmeyer flask into a fume hood and add milli-Q water (50 mL) into it. Close the flask and take it to a balance.
4. Take the sodium hydroxide from a solid cabinet and take it to a balance. Weigh it (400 mg) and add it into a flask.
5. Take the closed flask into a fume hood and wait for the sodium hydroxide to disperse. Store the solution in a plastic bottle in the corrosive base cabinet.
6. Whenever using sodium hydroxide solution, dispose the waste into the waste bottle labeled corrosive hazardous waste.

Preparation of gold nanoparticles

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Make a waste bottle labeled as toxic hazardous waste. Review the SDS of gold chloride trihydrate, sodium hydroxide, and Tetrakis(hydroxymethyl)phosphonium chloride again; especially remind first aid measures, handling and storage, & PPE.*
3. Place a very clean one neck round bottom flask into a fume hood and put a stir bar into it.
4. Take gold chloride trihydrate from inorganic acid cabinet and take it to a balance. Weigh it (157.6 mg) and add it into a centrifuge tube. Close the centrifuge tube and transfer it to the fume hood. Add milli-Q water (20 mL) to the centrifuge tube.
5. Add milli-Q water (364 mL) to the round-bottom flask.
6. Take sodium hydroxide solution (0.2 M) from a corrosive base cabinet and place it into the fume hood. Add water (12 mL) in the round-bottom flask.
7. Separately in a centrifuge tube prepare a solution of tetrakis(hydroxymethyl) phosphonium chloride in water. First take the bottle of tetrakis(hydroxymethyl) phosphonium chloride from the flammable cabinet and place it into the fume hood. Add milli-Q water (8 mL) into the centrifuge tube. Next, add tetrakis (hydroxymethyl)phosphonium chloride (96 µL) into it. Stir and add the mixture to the round bottom flask.
8. Stir the mixture in the closed round-bottom flask for 2 minutes. Then, add the mixture of gold chloride trihydrate and water and stir for another 2 minutes.
9. Save the gold solution in appropriately labeled centrifuge tubes.
10. When using the gold solution, dispose the waste into the waste bottle labeled toxic and corrosive hazardous waste.

Partial etching of titania shells

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Make a waste bottle labeled as toxic and corrosive hazardous waste. Review the SDS of sodium hydroxide and hydrochloric acid again; especially remind first aid measures, handling and storage, & PPE.*
3. Place an Erlenmeyer flask into a fume hood and put a stir bar into it.
4. Disperse titania shells with a silica core in milli-Q water (20 mL) and add the dispersion to the flask.
5. Take sodium hydroxide solution (2.5 M) from a corrosive base cabinet and place it into the fume hood. With a micropipette add sodium hydroxide solution (1 mL of 2.5 M) and close with the septum. Stir for 20 minutes.

6. Wash three times with water and dispose the waste into the waste bottle labeled toxic and corrosive hazardous waste.
7. Disperse partially etched titania shells with a silica core in milli-Q water (10 mL) and add the dispersion to a clean flask.
8. Take hydrochloric acid solution (0.5 M) from inorganic acid cabinet and place it into the fume hood. With a micropipette add hydrochloric acid solution (2 mL of 0.5 M) and close with the septum. Stir for 30 minutes.
9. Wash three times with water and two times with ethanol. Dispose the waste into the waste bottle labeled toxic and corrosive hazardous waste.
10. Label the centrifuge tube appropriately, cover it with perforated aluminum foil and dry the powder in a vacuum desiccator overnight.

Full etching of titania shells

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Make a waste bottle labeled as toxic and corrosive hazardous waste. Review the SDS of sodium hydroxide and hydrochloric acid again; especially remind first aid measures, handling and storage, & PPE.*
3. Place an Erlenmeyer flask into a fume hood and put a stir bar into it.
4. Disperse titania shells with a silica core in milli-Q water (20 mL) and add the dispersion to the flask.
5. Take sodium hydroxide solution (2.5 M) from a corrosive base cabinet and place it into the fume hood. With a micropipette add sodium hydroxide solution (4 mL of 2.5 M) and close with the septum. Stir for 6 hours.
6. Wash three times with water and two times with ethanol. Dispose the waste into the waste bottle labeled toxic and corrosive hazardous waste.
7. Label the centrifuge tube appropriately, cover it with perforated aluminum foil and dry the powder in a vacuum desiccator overnight.

Synthesis of PtCu alloy

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (25 mL) under nitrogen in a fume hood and put a stir bar into it.
3. Put platinum(II) acetylacetonate (8.574 mg, 0.0218 *m*mol) into the flask.
4. Take ethylene glycol (5 mL) by a syringe in a fume hood and add it slowly to the flask.
5. Rinse syringes several times with ethanol before disposal.
6. The washings should be disposed as hazardous organic waste.
7. Put a syringe needle into sharps-disposal container.
8. Put polyvinylpyrrolidone (0.6 mg) and copper sulfate pentahydrate (5.452 mg,

0.0218 *m*mol) into the flask.
9. Change the pH with NaOH (0.1 M) to pH=10 and close the flask with rubber septum.
10. Reflux the mixture under nitrogen for 2 h at 198 °C. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
11. Remove the septum from the flask and put SBA-15 (558.36 mg) in the flask and stir the mixture for 2 h.
12. Sonicate the mixture for 1 h.
13. Centrifuge and dispose the liquid.
14. Evacuate the flask containing the sample then dry the powder at ca. 60 °C for 12 h in the fume hood.
15. Grind the product to powder with an agate mortar and pestle.
16. Wash the powder with acetone and acetone-ethanol mixture upon sonication for 5 times and centrifuge each time after it's washed.
17. Dry the powder under vacuum in the flask at 60 °C for 12 h.

Synthesis of Cu@Pt nanoparticle

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (25 mL) under nitrogen in a fume hood.
3. Put copper acetylacetonate (2.04 mg, 0.0078 *m*mol) into the flask.
4. Take ethylene glycol (8 mL) by a syringe in a fume hood and add it slowly to the flask.
5. Rinse syringes several times with ethanol before disposal.
6. The washings should be disposed as hazardous organic waste.
7. Put a syringe needle into sharps-disposal container.
8. Put polyvinylpyrrolidone (5.8 mg) into the flask.
9. Change the pH with NaOH (0.1 M) to pH=10 and close the flask with rubber septum.
10. Reflux the mixture under nitrogen for 20 minutes at 198 °C. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
11. After cooling to 80 °C, add platinum acetylacetonate (3.07 mg, 0.0078 *m*mol) and slowly heat the mixture back to reflux for 1 h.
12. Remove the septum from the flask and put SBA-15 (402.93 mg) in the flask and stir the mixture for 2 h.
13. Sonicate the mixture for 1 h.
14. Centrifuge and dispose the liquid.
15. Evacuate the flask containing the sample then dry the powder at ca. 60 °C for 12 h in the fume hood.
16. Grind the product to powder with an agate mortar and pestle.
17. Wash the powder with acetone and acetone-ethanol mixture upon sonication for 5 times and centrifuge each time after it's washed.
18. Dry the powder under vacuum in the flask at 60 °C for 12 h.

Synthesis of Pt@Cu nanoparticle

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (25 mL) under nitrogen in a fume hood.
3. Put platinum acetylacetonate (18.582 mg, 0.04725 *m*mol) into the flask.
4. Take ethylene glycol (5.775 mL) by a syringe in a fume hood and add it slowly to the flask.
5. Rinse syringes several times with ethanol before disposal.
6. The washings should be disposed as hazardous organic waste.
7. Put a syringe needle into sharps-disposal container.
8. Put polyvinylpyrrolidone (2.82 mg) into the flask.
9. Change the pH with NaOH (0.1 M) to pH=10 and close the flask with rubber septum.
10. Reflux the mixture under nitrogen for 2 h at 198 ^{o}C. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
11. After cooling to 80 ^{o}C, add copper sulfate pentahydrate (11.814 mg, 0.04725 *m*mol) and slowly heat the mixture back to reflux for 2 h.
12. Remove the septum from the flask and put SBA-15 (598.455 mg) in the flask and stir the mixture for 2 h.
13. Sonicate the mixture for 1 h.
14. Centrifuge and dispose the liquid.
15. Evacuate the flask containing the sample then dry the powder at ca. 60 ^{o}C for 12 h in the fume hood.
16. Grind the product to powder with an agate mortar and pestle.
17. Wash the powder with acetone and acetone-ethanol mixture upon sonication for 5 times and centrifuge each time after it's washed.
18. Dry the powder under vacuum in the flask at 60 ^{o}C for 12 h.

Acid-Base Titration

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Into the test tube with stir bar, a sample (25 mg) is mixed with HCl solution (0.0224 M, 5 mL).
3. Stir the solution for 20 min at room temperature.
4. Filter the mixture.
5. The filtered solid is washed with water (3 x 10 mL)
6. Add 1-2 drops of phenolphthalein into the filtrate as indicator and then titrate it with a solution of NaOH (0.01 M).

RCA Cleaning Protocol

1. Wear nitrile chemical resistive gloves, a flame-resistant lab coat, and safety goggles.
2. Sonicate silicon wafers in acetone for 20 min.
3. Preclean silicon wafers with acetone and DI water and dry in a N_2 flow.
4. Place wafers in a solution of sulfuric acid (12 mL) and hydrogen peroxide (4 mL) for 10 min.
5. Clean wafers with copious amounts of milli-Q water.
6. Place wafers in a solution of hydrofluoric acid (1 mL) and milli-Q water (20 mL) for 5 min.
7. Clean wafers with copious amounts of milli-Q water.
8. Place wafers in a solution of milli-Q water (20 mL), hydrogen peroxide (5 mL), and ammonium hydroxide (or sodium hydroxide, 5 mL) for 10 min at 80 ^oC.
9. Place wafers in a solution of milli-Q water (20 mL), hydrogen peroxide (5 mL), and hydrochloric acid (5 mL) for 10 min at 80 ^oC.
10. Dry silicon wafers in a N_2 flow.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2013, updated 03/01/2014, 03/01/2016, 05/15/2016, 11/01/2016, 0701/2018, 07/16/2019

Sodium sulfate
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when sodium sulfate (Na_2O_4S CAS No. 7757-82-6) is used in laboratory. Its purpose is not to have any accident or risk. Sodium sulfate is harmful to aquatic life.

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: not known
GHS Classification
Acute aquatic toxicity (Category 3)

Signs and Symptoms of Exposure
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.
- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with sodium sulfate must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for sodium sulfate.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using sodium sulfate must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of sodium sulfate and understand the hazards.

Lab workers using sodium sulfate must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with sodium sulfate described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines factors) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < $^{100\ g}$ of this sodium sulfate in any given reaction (larger quantities REQUIRE the approval of PI or designee), and

5) discuss ALL issues or concerns regarding this sodium sulfate with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using sodium sulfate. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Catalytic Reaction

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and a safety goggles.
2. Take 31 mg of 2-Hydroxybenzyl alcohol into a 30 mL Erlenmeyer flask in the fume hood.
3. Add potassium carbonate (103.7 mg) and water (5 mL).
4. Add aqueous Au-PVP catalyst (0.5 mM, 10 mL, 2 atom.%), and stir at 1300 rpm.
5. Quench reaction with HCl (1 M), extract with ethyl acetate, dry organic layer over sodium sulfate.
6. Run on GC.
7. Dispose off ethyl acetate as hazardous organic waste.

Synthesis from **QD** to **QD-Bn**

1. Wear nitrile chemical–resistant gloves, a flame-resistant lab coat, and a safety goggle.
2. Dimethylformamide, $(CH_3)_2NCOH$, is freshly distilled from a suspension of calcium hydride (CaH_2).
3. Sodium hydride (NaH, 0.68 g, 2.5 eq.) is put into a flask (100 mL).
4. Quinidine (**QD**, 6.2 *m*mol, 2.0 g) is dissolved in dimethylformamide (20 mL).
5. The quinidine solution is added to the flask of sodium hydride.
6. The mixture is stirred at room temperature for 2 h.
7. Benzyl chloride (0.78 mL, 1.1 eq.) is added dropwise via a syringe in 10 min.
8. The mixture is stirred overnight.
9. Brine (20 mL) is added carefully to the flask.
10. The mixture is extracted by ethyl acetate ($CH_3CO_2C_2H_3$, 100 mL).
11. The organic phase is washed with brine (3×50 mL)
12. The organic phase is dried over sodium sulfate (Na_2SO_4) and concentrated in vacuum.
13. **QD-Bn** is extracted from the residue by column chromatograph with a solution (methanol:ethyl acetate=1:40).

Synthesis from QD-Bn to QD-a

1. Wear nitrile chemical–resistant gloves, a flame-resistant lab coat, and a safety goggle.
2. **QD-Bn** (8.0 *m*mol), sodium ethanethiolate ($NaSC_2H_5$, 4 eq.) and dimethylformamide (50 mL) are added to a flask (200 mL).
3. The mixture is stirred at 110 °C until TLC analysis shows that the starting material is completely consumed within 4–6 h.
4. The mixture is cooled down to room temperature.
5. Ammonium chloride (NH_4Cl, 40 mL) and water (50 mL) are added to the flask.
6. The pH value of the mixture is determined to be around 7.
7. The mixture is extracted by ethyl acetate (2×200 mL).
8. The organic phase is washed with brine (4×50 mL).
9. The organic phase is dried over sodium sulfate (Na_2SO_4) and concentrated in vacuum.
10. **QD-a** is extracted from the residue by column chromatograph with a solution (methanol:ethyl acetate=1:50 to 1:10).

Synthesis of 9-amino(9-deoxy)*epi*-quinine

Module D: Neutralization of the salt & Formation of free amine

1. The salt (3 g) is put into a flask (100 mL).
2. Dichloromethane (20 mL) is added to the flask.
3. Ammonium hydroxide (5 M, 15 mL) is slowly added to the flask.
4. The mixture is vigorously stirred for 5min.
5. The mixture is transferred to a separation funnel (60 mL) using dichloromethane to wash the flask (3 mL, 3 times).
6. Aqueous phase is washed with dichloromethane (15 mL, 2 times) and the organic phase is collected.
7. The organic phase is dried with sodium sulfate for 5 min.
8. The solvent is removed under vacuum.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 03/11/2016, 02/07/2020

Sulfuric acid
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when sulfuric acid (H_2O_4S, CAS No. 7664-93-9) is used in laboratory. Its purpose is not to have any accident or risk. Sulfuric acid is corrosive liquid. It causes severe skin burns and eye damage.

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Corrosive, Target Organ Effect (Teeth, Lungs)
GHS Classification
Skin corrosion (Category 1A)
Serious eye damage (Category 1)

Signs and Symptoms of Exposure
Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin., spasm, inflammation and edema of the larynx, spasm, inflammation and edema of the bronchi, pneumonitis, pulmonary edema, burning sensation, Cough, wheezing, laryngitis, Shortness of breath, Headache, Nausea, Vomiting, Pulmonary edema. Effects may be delayed., To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.
- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill

area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with sulfuric acid must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for sulfuric acid.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using sulfuric acid must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of sulfuric acid and understand the hazards.

Lab workers using sulfuric acid must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with sulfuric acid described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;

3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 50 mL of this sulfuric acid in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this sulfuric acid with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using sulfuric acid. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

4-Methyl-2,6-heptanedione

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Add sodium (1.8 g, 50 *m*mol) and ethanol (24 mL) into a flask (100 mL).
3. Add 2,4,6-trimethylpyridine (6.5 mL, 50 *m*mol) into the flask.
4. Reflux the mixture for 1 h at 90 °C in oil bath.
5. Prepare a solution of hydroxylamine hydrochloride (3.6 g, 53 *m*mol) in 50% ethanol (6.4 mL) and HCl (3.2 mL) in 95% ethanol (6.4 mL).
6. Add the solution slowly into the flask.
7. Reflux the mixture for 2.5 h.
8. Cool down the mixture to room temperature.
9. Remove ethanol from the mixture under evaporator.
10. Add a NaOH solution (3.5 g in 50 mL water) to the residue.
11. Extract the solution with ether (50 mL).
12. Acidify the aqueous solution with 10% H_2SO_4 (35 mL).
13. Add sodium nitrite solution (3.5 g, 50 *m*mol in 10 mL water) into the mixture.
14. Stir the mixture for 1 h at 0 °C.
15. Extract the mixture with ether (20 mL, 4 times)
16. Wash the organic phase with water and brine.
17. Column the residue with a hexane solution (hexane:ether = 10:2).

ICP Sample Preparation #1

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Weigh a sample (~ 3 mg).
3. Dissolve the sample in a hot concentrated sulfuric acid solution.

4. Heat the solution at 205 °C for 2 h.
5. Cool down to room temperature.
6. Dilute the solution to around 300 mL.
7. Quantitatively transfer the solution to a volumetric flask (500 mL).
8. Dilute the solution to a volume with water.

RCA Cleaning Protocol

1. Wear nitrile chemical resistive gloves, a flame-resistant lab coat, and safety goggles.
2. Sonicate silicon wafers in acetone for 20 min.
3. Preclean silicon wafers with acetone and DI water and dry in a N_2 flow.
4. Place wafers in a solution of sulfuric acid (12 mL) and hydrogen peroxide (4 mL) for 10 min.
5. Clean wafers with copious amounts of milli-Q water.
6. Place wafers in a solution of hydrofluoric acid (1 mL) and milli-Q water (20 mL) for 5 min.
7. Clean wafers with copious amounts of milli-Q water.
8. Place wafers in a solution of milli-Q water (20 mL), hydrogen peroxide (5 mL), and ammonium hydroxide (or sodium hydroxide, 5 mL) for 10 min at 80 °C.
9. Place wafers in a solution of milli-Q water (20 mL), hydrogen peroxide (5 mL), and hydrochloric acid (5 mL) for 10 min at 80 °C.
10. Dry silicon wafers in a N_2 flow.

ICP Sample Preparation #2

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, safety goggles, AND a proper face mask while inside the lab. Carry out all the procedures in the fume hood.
2. Clean glassware, such as an Erlenmeyer flask (250 mL), a graduated cylinder (50 mL), and a beaker (500 mL) with deionized water and acetone.
3. Measure sample powder (~5 mg) and pour it into the cleaned Erlenmeyer flask. Wrap up the upside part with parafilm to avoid any contamination.
4. Transfer concentrated sulfuric acid (10 mL) from the reagent bottle to a graduated cylinder with a glass pipette.
5. Pour the prepared sulfuric acid (~10 mL) into the Erlenmeyer flask containing the sample powder.
6. Hang the Erlenmeyer flask to the wall in the fume hood and set up the reaction condition for powder dissolution with a silicon oil bath and stirrer.
7. Heat the silicon oil bath to 165 ℃ and leave for 1.5 h. (Check the temperature condition every 15 min)

8. Cool down the reaction conditions to room temperature.
9. Prepare a volumetric flask (500 mL) and fill it up with deionized water (250 mL).
10. Slowly add the dissolved sample-sulfuric acid solution to the volumetric flask in step 9.
11. Fill up the volumetric flask (500 mL) with deionized water.
12. Transfer the plastic bottle to send it to the analysis department.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2015, updated 07/01/2018, 07/16/2019, 07/07/2022

tert-Butylamine
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when tert-butylamine ($C_4H_{11}N$, CAS No. 75-64-9) used in laboratory. Its purpose is not to have any accident or risk. tert-Butylamine is highly flammable liquid and vapor, and harmful if swallowed of toxic if inhaled. It causes severe skin burns and eye damage.

Synonyms: 2-Amino-2-methylpropane

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Flammable liquid, Toxic by Inhalation
GHS Classification

Flammable liquids (Category 2)
Acute toxicity, Oral (Category 4)
Acute toxicity, Inhalation (Category 3)
Skin corrosion (Category 1A)
Serious eye damage (Category 1)
Short-term (acute) aquatic hazard (Category 3)
Long-term (chronic) aquatic hazard (Category 3)

Signs and Symptoms of Exposure
Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Cough, Shortness of breath, Headache, Nausea

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.

- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with tert-butylamine must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for tert-butylamine.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using tert-butylamine must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of tert-butylamine and understand the hazards.

Lab workers using tert-butylamine must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with tert-butylamine described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;

3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 50 mL of this tert-butylamine in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this tert-butylamine with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using tert-butylamine. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Cleaning of the Surfactant on PtCu/SBA-15 Catalyst

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Bring catalyst PtCu/SBA-15 and tert-butylamine into a fume hood.
3. PtCu/SBA-15 (10 mg) is put in a beaker (100 mL) and fill with tert-butylamine (20 mL).
4. Stir the mixture of catalyst with tert-butylamine with a magnetic stirrer for three days at room temperature.
5. After the treatment, the catalyst is separated from the solvent by centrifugation at 6000 rpm for 10 min.
6. After centrifugation, the liquid is treated as hazardous waste.
7. The collected precipitate is washed with ethanol (20 mL) three times to remove excess surface-adsorbed amine.
8. The final product is redispersed in ethanol and then dried over-night.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 11/10/2020

Tetrahydrofuran
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when tetrahydrofuran (C_4H_8O, CAS No. 109-99-9) used in laboratory. Its purpose is not to have any accident or risk. Tetrahydrofuran is flammable liquid and vapor and harmful if swallowed or in contact with skin. It causes serious eye and mild skin irritation. Also it may cause drowsiness and dizziness.

Synonyms: THF

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Flammable liquid, Target organ effect (Central nervous system, Liver and Kidney), Harmful by Ingestion, Irritant, Carcinogen

GHS Classification

Flammable liquids (Category 2)
Acute toxicity, Oral (Category 4)
Acute toxicity, Dermal (Category 5)
Skin irritation (Category 3)
Serious eye damage (Category 1)
Specific target organ toxicity - single exposure (Category 3)

Signs and Symptoms of Exposure

Central nervous system depression, Cough, chest pain, Difficulty in breathing, Exposure to high airborne concentrations can cause anesthetic effects. To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be

buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.
- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to “Injuries and Medical Treatment” Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.

- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with tetrahydrofuran must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for tetrahydrofuran.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using tetrahydrofuran must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of tetrahydrofuran and understand the hazards.

Lab workers using tetrahydrofuran must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with tetrahydrofuran described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 100 mL of this tetrahydrofuran in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this tetrahydrofuran with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using tetrahydrofuran. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Preparation of Cd-TEOSPC

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take tetrahydrofuran (20 mL) as solvent for the reaction into the two-neck round bottom flask in the fume hood.
3. After reaction, the filtrate needs to be treated as hazardous waste.
4. Washing and cleaning solvents also need to be treated as hazardous waste.

Catalytic Reaction

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Dissolve benzaldehyde (10 mmol) and a catalyst (20 mol%) into a tetrahydrofuran solution.
3. Add *trans*-β–nitrostyrene (1 mmol) into the mixture.
4. Leave the mixture at room temperature for 3 days.
5. After reaction, the removed solvent needs to be treated as hazardous waste.
6. Washing and cleaning solvents also need to be treated as hazardous waste.

Synthesis of 9-amino(9-deoxy)*epi*-quinine

Module A: Synthesis of 9-amino(9-deoxy)*epi*-quinine

1. Quinine (5 g, 15.4 *m*mol) and Triphenylphosphine (4.85 g, 18.5 *m*mol) are put into a round flask (250 mL) dried under vacuum.
2. Tetrahydrofuran (60 mL) is added to the flask and stirred for 5 min.

3. Cool down the mixture to 0 ºC and stay for 5 min.
4. Diisopropyl azodicarboxylate (DIAD, 3.64 mL, 18.5 *m*mol) is slowly added to the mixture for 5 min. The color changes to yellowish.
5. At 5 min after addition, diphenyl phosphoryl azide (DPPA, 4. 0 mL, 18.5 *m*mol) is added dropwise for 15 min.
6. After stirring for 15 min, the mixture is warmed up to room temperature.
7. The mixture is stirred for 4 h at room temperature.
8. The mixture is stirred for 2 h at 45 ºC.
9. Triphenylphosphine (4.85 g, 18.5 *m*mol) is added to the mixture in one portion.
10. The mixture is stirred for 2 h at 45 ºC.
11. Water (3.5 mL) is added into the flask.
12. The mixture is stirred overnight at 45 ºC.
13. The mixture is cooled down to room temperature.
14. The mixture is transferred to a flask (500 mL).
15. The solvent is removed by rotary evaporator.
16. Dichloromethane (80 mL) is added to the remained solution.
17. Hydrochloric acid (2 M, 80 mL) is added to the mixture.
18. The organic phase is removed.
19. The aqueous phase is washed with dichloromethane (40 mL, 2 times).
20. The aqueous phase is transferred into a flask (250 mL).
21. The solvent is removed by rotary evaporator and under vacuum.
22. A bright-yellow solid is obtained.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 03/01/2014, 10/24/2019, 02/07/2020

Tetrakis(dimethylamido)titanium
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when tetrakis(dimethylamido)titanium ($C_8H_{24}N_4Ti$, CAS No. 3275-24-9) used in laboratory. Its purpose is not to have any accident or risk. Tetrakis(dimethylamido)titanium is highly flammable corrosive liquid and vapor. It causes severe skin burns and eye damage. In contact with water, it releases flammable gases which may ignite spontaneously.

Synonyms: Tetrakis(dimethylamino)titanium (IV), TDMAT

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Flammable, Corrosive,
GHS Classification

Flammable liquid (Category 2)
Skin corrosion (Category 1B)
Serious eye damage (Category 1)

Signs and Symptoms of Exposure
Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Spasm, inflammation and edema of the larynx, inflammation and edema of the bronchi, pneumonitis, pulmonary edema, burning sensation, Cough, wheezing, laryngitis, Shortness of breath, Headache, Nausea.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.

- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with tetrakis(dimethylamido)titanium must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for tetrakis(dimethylamido)titanium.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using tetrakis(dimethylamido)titanium must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of tetrakis(dimethylamido)titanium and understand the hazards.

Lab workers using tetrakis(dimethylamido)titanium must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with tetrakis(dimethylamido)titanium described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;

3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 1 mL of this tetrakis(dimethylamido)titanium in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this tetrakis(dimethylamido)titanium with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material, the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using tetrakis(dimethylamido)titanium. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Tetrakis(dimethylamido)titanium for ALD reactor

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggle.
2. Add tetrakis(dimethylamido)titanium (0.5 mL) into the metal-glass adaptor container in the glove box in room 135. Connect the container to a Swagelok valve and close the valve inside the glove box.
3. Connect the Swagelok valve with tetrakis(dimethylamido)titanium to the ALD reactor.
4. Tetrakis(dimethylamido)titanium is pumped by mechanical pump. Use a freeze-thaw cycle if necessary.
5. Use tetrakis(dimethylamido)titanium at room temperature.
6. In the ALD reaction, slowly open the Swagelok valve to reach the desired vapor pressure and expose for a certain amount of time (5 s). Close the valve and purge the line with inert nitrogen gas.

TiO_2/SBA-15

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, and a proper face mask at all times while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
2. Clean the sample holder with acetone before placing SBA-15 in the sample holder.
3. Preheat the support (SBA-15) up to 200 ^{o}C for 2 h.
4. Ti precursor (tetrakis(dimethylamido) titanium, TDMAT) is set to 42 ^{o}C
5. The reactor is set to 102 ^{o}C
6. Dose TDMAT for 20 min at 200 mTorr.

7. Purge the reactor with Ar gas for 50 min at 500 mTorr.
8. Dose deionized water vapor for 2 min at 100 mTorr.
9. Purge the reactor with Ar gas for 50 min at 500 mTorr.
10. Repeat 6–9 steps repeatedly until desired growth is obtained.

TiO_2/TMSDMA, HMDS or ODTS/SBA-15

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, and a proper face mask at all times while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
2. Clean the sample holder with acetone before placing SBA-15 in the sample holder.
3. Preheat the support (SBA-15) at 200 ^oC for 2 h.
4. Ti precursor (tetrakis(dimethylamido) titanium, TDMAT) 42 ^oC, using heating tape and r
5. The reactor is set to 102 ^oC.
6. Dose an inhibitor reagent (trimethylsilyl dimethylamine, hexamethyldisilazane, or octadecyltrichlorosilane) for 30 s at 50 mTorr.
7. Dose TDMAT for 20 min at 200 mTorr.
8. Purge the reactor with Ar gas for 50 min at 500 mTorr.
9. Dose deionized water vapor for 2 min at 100 mTorr.
10. Purge the reactor with Ar gas for 50 min at 500 mTorr.
11. Repeat 7–10 steps repeatedly until desired growth is obtained.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 11/01/2015, 12/21/2021

Toluene
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when toluene (C_7H_8, CAS No. 108-88-3) used in laboratory. Its purpose is not to have any accident or risk. Toluene is highly flammable liquid and vapor, and causes serious eye and skin irritation. It is harmful and suspected of damaging fertility or the unborn child. It may be fatal if swallowed and enters airways, and cause drowsiness or dizziness, damage to organs. It is also toxic to aquatic life.

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Flammable liquid, Target organ effect (Bladder, Liver, Kidney and Brain), Irritant, Teratogen, Reproductive hazard.

GHS Classification

Flammable liquids (Category 2)
Acute toxicity, Inhalation (Category 4)
Skin irritation (Category 2)
Eye irritation (Category 2A)
Reproductive toxicity (Category 2)
Specific target organ toxicity - single exposure (Category 2,3)
Aspiration hazard (Category 1)
Acute aquatic toxicity (Category 2)

Signs and Symptoms of Exposure

Lung irritation, chest pain, pulmonary edema, Inhalation studies on toluene have demonstrated the development of inflammatory and ulcerous lesions of the penis, prepuce, and scrotum in animals.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be

buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.
- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.

- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with toluene must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for toluene.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using toluene must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of toluene and understand the hazards.

Lab workers using toluene must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with toluene described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;

2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 100 mL of this toluene in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this toluene with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using toluene. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Hydrogenation of ethyl pyruvate

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take 6 mL of toluene into the high-pressure reactor in the fume hood.
3. Add catalyst and substrate into the reactor, and then the reaction is conducted in the corresponding instrument in room 135.
4. After reaction, the filtrate needs to be treated as hazardous waste.
5. Washing and cleaning solvents also need to be treated as hazardous waste.

Si wafer washing

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Bring the toluene solution bottle to the fume hood.
3. Load this Toluene solution into spray bottle for washing purpose afterwards
4. After washing the Si wafer, the sample solution needs to be treated as hazardous

Synthesis of 1,1'-(1,2-Dioxoethane-1,2-diyl)bis-1*H*-benzotriazole

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (500 mL) and a dropping funnel under nitrogen.
3. Put benzotriazole (11.9 g, 100 *m*mol) into the flask.
4. Add ether (400 mL) into the flask.
5. Put toluene (40 mL) and oxalyl chloride (6.35 g, 50 *m*mol) into the dropping funnel.
6. Drop the oxalyl chloride solution slowly into the flask.

7. Stir the mixture for 20 h at room temperature. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
8. Filter and wash the mixture with ether.
9. Dry the white powder.

Catalytic Oxidation Reaction

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Into the test tube with stir bar, add a heterogeneous catalyst (9 mg) and potassium carbonate (27 mg), and toluene (4.5 mL).
3. Ultrasonicate and stir the mixture.
4. Add a chemical to be oxidized (0.05 *m*mol; e.g. benzyl alcohol, fluorene, p-Cymene, etc.).
5. Close with a rubber septum, seal with parafilm, connect the oxygen supply to the test tube, and do the catalytic reaction at temperatures below 75 ^{o}C (boiling point of benzene is 80.1 ^{o}C).
6. Collect the samples.
7. Run GC.

Oxidation of 1-Methyl-1-cyclohexene

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Into the test tube with stirring bar add P25-TiO_2/Au catalyst (9 mg) and potassium tert-butoxide (13 mg).
3. Add toluene (4.5 mL), ultrasonicate and stir.
4. Add benzene (12.5 µL, internal standard)
5. Add 1-methyl-1-cyclohexene (4.8 mg, 0.05 *m*mol).
6. Close with rubber septum, seal with parafilm, connect the oxygen supply to the test tube and do the catalytic reaction at temperature below 75 °C.
7. Collect the samples and run on GC.

Oxidation of α-Pinene

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Into the test tube with stirring bar add P25-TiO_2/Au catalyst (9 mg) and potassium tert-butoxide (13 mg).
3. Add toluene (4.5 mL), ultrasonicate and stir.
4. Add benzene (12.5 µL, internal standard)
5. Add α-pinene (6.8 mg, 0.05 *m*mol).
6. Close with rubber septum, seal with parafilm, connect the oxygen supply to the test

tube and do the catalytic reaction at temperature below 75 °C.
7. Collect the samples and run on GC.

Oxidation of Cyclooctane

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Into the test tube with stirring bar add P25-TiO_2/Au catalyst (9 mg) and potassium tert-butoxide (13 mg).
3. Add toluene (4.5 mL), ultrasonicate and stir.
4. Add benzene (12.5 µL, internal standard)
5. Add cyclooctane (5.6 mg, 0.05 *m*mol).
6. Close with rubber septum, seal with parafilm, connect the oxygen supply to the test tube and do the catalytic reaction at temperature below 75 °C.
7. Collect the samples and run on GC.

Oxidation reaction

1. Wear nitrile chemical-resistant gloves, mask, flame-resistant lab coat, and safety goggles.
2. In a fume hood, add of aluminum isopropoxide (100 µmol) to a round-bottom flask under N_2 atmosphere.
3. Add toluene (5 mL).
4. Add benzaldehyde (500 µmol).
5. Stir for 3 h.
6. Take NMR.

Oxidation of Fluorene

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Into the test tube with stirring bar, add P25-TiO_2/Au catalyst (9 mg), potassium carbonate (27 mg), and toluene (4.5 mL)
3. Ultrasonicate and stir the mixture.
4. Add benzene (12.5 µL, internal standard).
5. Add fluorene (0.05 *m*mol)
6. Close with rubber septum, seal with parafilm, connect the oxygen supply to the test tube, and do the catalytic reaction at temperature below 75 °C (boiling point of benzene is 80.1 °C.
7. Collect the sample
8. Run GC.

Oxidation of p-Cymene

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Into the test tube with stirring bar, add P25-TiO_2/Au catalyst (9 mg) and potassium carbonate (27 mg), and toluene (4.5 mL)
3. Ultrasonicate and stir the mixture.
4. Add benzene (12.5 μL, internal standard).
5. Add p-Cymene (0.05 *m*mol)
6. Close with rubber septum, seal with parafilm, connect the oxygen supply to the test tube, and do the catalytic reaction at temperature below 75 °C (boiling point of benzene is 80.1 °C.
7. Collect the sample
8. Run GC.

Solvent in oxidation reactions

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Make a waste bottle labeled as toxic and carcinogen hazardous waste. Review the SDS of toluene and benzene again; especially remind first aid measures, handling and storage, & PPE.*
3. Place a test tube into a fume hood and put a stir bar into it. Close with a rubber septum and take it to a balance. Weigh P25-TiO_2/Au catalyst (9 mg) and add it into the test tube. Add potassium carbonate (25 mg) and transfer the closed septum back to the fume hood.
4. With a micropipette add toluene (4.5 mL), close with the septum and sonicate for about 1 minute so that the solids disperse well in the solvent.
5. Bring the mixture back to the fume hood and stir it.
6. Before adding the internal standard benzene (12.5 μL) *put on a full-face respirator*. Take a bottle of benzene from the flammable cabinet and place it into the fume hood. *Be careful not to spill benzene. Keep watching any leak of benzene. Avoid release to the environment. Avoid breathing fume, gas, mist, vapor or spray. If swallowed, immediately call 911. If inhaled, rinse cautiously with water for 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.*
7. Remove the septum from the test tube and open the benzene bottle. Add the small amount of benzene into the test tube by using a Hamilton syringe (50 μL). Wash the syringe with benzene three times before adding it into the reaction mixture. After adding it clean the syringe by washing it with ether. Dispose the waste into the waste bottle labeled carcinogen hazardous waste. Once adding

benzene the handling of the reaction mixture has to be carried out with the full-face respirator on.

8. Put the benzene bottle back to the flammable cabinet. *Be careful not to spill benzene. Keep watching any leak of benzene. Avoid release to the environment. Avoid breathing fume, gas, mist, vapor or spray. If swallowed, immediately call 911. If inhaled, rinse cautiously with water for 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.*
9. Take the reactant (e.g. benzyl alcohol, p-cymene, fluorene, etc.) from the flammable cabinet and put it into the fume hood. Add the reactant (5.5 µL) into the reaction mixture.
10. Close the test tube with the rubber septum, seal with Teflon tape and connect the oxygen supply to the test tube.
11. Open the main valve of oxygen cylinder, which is located in a cupboard in a corridor in front of the room 135. After then, open the oxygen Swagelok needle valve in the fume hood, and fill the balloon with oxygen. Attach the balloon to a needle going through the rubber septum into the reaction mixture.
12. Transfer the test tube into the oil bath and do the catalytic reaction at temperature below 75 °C (boiling point of benzene is 80.1 °C).
13. Collect samples at different reaction times and remember to always put on the full-face respirator before working with the mixture. Put a sample (100 µL) into a small centrifuge tube and centrifuge it to remove the solids.
14. After centrifuging bring the closed vial back to the fume hood and transfer the liquid into a new vial.
15. Inject the sample into GC using a Hamilton syringe (10 µL).
16. Dispose all the waste into the appropriately labeled waste bottle.

Functionalization of silica nanospheres or P25 titania nanoparticles

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Make a waste bottle labeled as toxic hazardous waste. Review the SDS of 3-aminopropyltriethoxysilane, toluene, and P25 titania again; especially remind first aid measures, handling and storage, & PPE.*
3. Place a three-neck round-bottom flask into a fume hood and put a stir bar into it. Close with rubber septum and take it to a balance. Weigh silica or titania powder (1 g) and add it into the round-bottom flask. Closed with septum and take it back to the fume hood.
4. Add toluene (50 mL), close with the septum and sonicate for about 1 minute so that the solid disperse well in the solvent.
5. Take the closed round-bottom flask to the fume hood in the room 162. Attach it to a condenser and close the other two necks with a rubber septum. Put a thermometer probe through one septum into the mixture. Don’t forget to open

water running through condenser. Change the atmosphere inside the round-bottom flask to nitrogen and set the temperature to 113 °C.
6. Take 3-aminopropyltriethoxysilane from the flammable cabinet and put it into the fume hood. Add the compound (1.5 mL) into the mixture when it starts boiling.
7. Leave the reaction running for 1 day.
8. When the functionalization has finished, remove the condenser and close the middle neck of the round-bottom flask with septum. Bring it into the fume hood in the room 135. Wait for it to cool down and then wash it with ethanol 4 times.
9. Dispose the waste into the waste bottle labeled toxic hazardous waste.

GC-MS/LIFDI Sample Preparation

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Bring the compound for GC-MS/LIFDI analysis and toluene into the fume hood or the glove box (for air-sensitive sample).
3. A proper amount of GC-MS/LIFDI sample (~200 μg), is placed in a glass vial (2 mL) and fill with toluene (1 mL).
4. Bring the sample vial in a second container to the MS facility room carefully.
5. After measurement, the sample solution needs to be treated as hazardous waste.
6. Cleaning solvents also need to be treated as hazardous waste.

MS Analysis of Cu ALD Precursor

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Bring the Cu ALD precursor for Mass Spectrometry analysis and the anhydrous Toluene (100 mL) into the glove box.
3. A proper amount of Cu ALD compound (~200 μg) is placed in a glass vial (2 mL). Fill the glass vial with Toluene (1 mL).
4. Close the glass vial with a cap and tightly seal it with Parafilm.
5. Take the MS sample out of the glovebox.
6. Bring the sample vial in a second container to the MS facility room carefully.
7. After measurement, the sample solution needs to be treated as hazardous waste.
8. Cleaning solvents also need to be treated as hazardous waste.

Hydrogenation of Ethyl-Pyruvate in Toluene

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Add Pt/Al_2O_3 (1 wt.%, 25 mg) and toluene (15 mL) into the HP reactor.
3. Prepare an ethyl pyruvate solution (1.4 mL in 7 mL of toluene).
4. Transfer an ethyl pyruvate solution to the mixture in step 2.
5. Connect the cylinder to the head gasket and tighten all 6 screws
6. Flush the reactor with pure H2 for 5 times to remove the air left inside.
7. Pressurize the reactor to 10 bar of H2 with stirring.
8. Release the H2 after 10 min of reaction
9. Take a sample for GC analysis.
10. Repeat this step every 10 minutes until 40min of reaction.

Hydrogenation Reaction

1. Wear nitrile chemical–resistant gloves, a flame-resistant lab coat, and a safety goggle.
2. Add Pt/Al_2O_3 catalyst (1 wt.%, 25 mL) into the stainless steel cylinder and disperse it in toluene (15 mL).
3. Transfer 5-hydroxymethyl-2-furaldehyde solution (1 mL, 3 M in toluene) to the mixture in step 2.
4. Connect the cylinder to the head gasket and tighten all 6 screws.
5. Flush the reactor with pure H2 for 5 times to remove the air left inside.
6. Pressurize the reactor to 10 bar and start stirring.
7. Release the H2 after 10 min of reaction and take a sample for GC analysis.
8. Repeat this step every 10 minutes until 40 min of reaction

Oxidation of Benzyl alcohol

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. The samples are used without any pretreatment
3. Au/SBA-15 sample (20 mg) is mixed with toluene (20 mL), benzyl alcohol (1 mL) and dodecane (1 mL) in the Parr reactor. Toluene is measured out by a graduated cylinder, while benzyl alcohol and dodecane are measured out by a pipette.
4. The Parr reactor is purged by O_2 for 10 min.
5. The reaction begins at 100 ^oC and 1 atm O_2 pressure with stirring.
6. The reaction is stopped after appropriate time.
7. Liquid samples are filtered and stored in vials.
8. The products are analyzed by GC.

epi-Quinine Tethering

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle
2. 9-amino(9-deoxy)*epi*-quinine (1.08 g, 0.3 *m*mol) and AIBN (azoisobutyronitrile) (54 mg, 0.33 *m*mol) is put into a 50 mL flask.
3. Chloroform (15 mL) is added into the flask.
4. (3-Mercapto)propyltriethoxysilane (0.87 mL, 3.6 *m*mol) is added into the flask.
5. The flask is refluxed at 63 ^{o}C for 1 day.
6. The flask is cooled down to room temperature.
7. Chloroform is removed by evaporator.
8. SBA-15 (100 mg) is mixed with the product.
9. Toluene (10 mL) is added to the flask.
10. The mixture is refluxed at 113 ^{o}C for 1 day.
11. The solid is filtered and washed by toluene.
12. The powder product is dried under vacuum for 5 h.

SBA-15 Silylation 1

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle
2. Add SBA-15 (200 mg) and two magnetic stir bars to two round-bottom flasks (50 mL).
3. Create inert N_2 or Ar atmosphere in each flask using a combination of a syringe and vent needle. Allow gas to cycle through the flask for 5-10 min.
4. Add toluene (5 mL) to each flask.
5. Add triethylamine (0.3 mL) to each flask.
6. Add chlorotrimethylsilane 0.2 mL to one flask and 0.1 mL to the other flask.
7. Allow reaction to stir over magnetic stir plate for 24 hours.
8. Expose both flasks to air and quench each reaction with methanol (5 mL).
9. Vacuum filter both mixtures and wash powder with acetone (10 mL) and DI water for each.
10. Dry both sets of powders by vacuum filtering for 5-10 min.
11. Set each sample (10 mg) aside in a plastic storage container.
12. Set each sample (10 mg) aside in a plastic storage container.

SBA-15 Silylation 2

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle
2. SBA-15 (200 mg) is put into a round-bottom flask (25 mL).
3. The flask is purged with Ar gas for 5 minutes.
4. Toluene (5 mL) is added to the flask

5. Triethylamine (0.3 mL) and chlorotrimethylsilane (0.2 mL) are added to the flask.
6. The resulting mixture is stirred for 24 hours
7. Then the mixture is exposed to air and quenched with methanol (5 mL).
8. The mixture is filtered and washed with water (10 mL) and acetone (10 mL).
9. Finally the mixture is left to dry under vacuum.

Synthesis of aminopropyltriethoxysilane(APTES)-grafted P25 titania

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Place a dry three-neck round-bottom flask (100 mL) into a fume hood and put a stir bar into it the flask.
3. Add titania powder (1 g).
4. Pour toluene (30 mL) into the flask and close necks with the septum and sonicate for about 10 min so that the solid disperses well in the solvent.
5. Take the closed round-bottom flask to the fume hood. Attach it to a condenser and close the other two necks with a rubber septum. Flow water through the condenser.
6. Change the atmosphere inside the round bottom flask to nitrogen.
7. Add 3-aminopropyltriethoxysilane (1.5 mL) into the flask.
8. Mix and reflux the mixture for 24 h at 70 °C. If left unattended in a fume hood, put a label with the chemical name and hazard information.
9. Filter and wash the mixture three times with isopropyl alcohol and deionized water to remove excess organosilanes.
10. Dispose waste in the properly labeled container.
11. Dry the white powder.

Silylation Procedure for SBA-15

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. A flask is dried.
3. Put SBA-15 (200 mg) in the flask.
4. Fill the flask with toluene (30 mL).
5. Slowly add hexamethyldisilazane or N,N-dimethyltrimethylsilylamine (2 mL) into the flask in the fume hood.
6. The mixture is refluxed at 90 ^{o}C for 24 h.
7. The mixture is cooled down to room temperature.
8. The solution is centrifuged for 10 min to remove the solvent.
9. Collect the powder and wash with isopropyl alcohol and DI water 3 times.
10. Vacuum filter the mixture overnight.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 03/01/2014, 03/03/2016, 05/15/2016, 07/01/2018, 02/06/2020, 07/08,2020, 0418/2022, 08/24/2022

trans-1-Phenyl-2-buten-1-one
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when trans-1-phenyl-2-buten-1-one (T_2S_3, CAS No. 35845-66-0) used in laboratory. Its purpose is not to have any accident or risk. trans-1-Phenyl-2-buten-1-one is harmful if swallowed.

Synonyms: *trans*-Crotonophenone

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Toxic by Ingestion,
GHS Classification
Acute toxicity, Oral (Category 4)

Signs and Symptoms of Exposure
Effects due to ingestion may include. Liver injury may occur.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Respiratory Protection

Where risk assessment shows air-purifying respirators are appropriate sue a full-face respirator with multi-purpose combination. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US).

b. Eye Protection

Face shield and ANSI compliant safety glasses with side shields should be worn. Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US). Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

c. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. Complete suit protecting against chemicals. The type of protection equipment must be selected according to the concentration and amount of the

dangerous substance at the specific workplace. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

d. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove or butyl-rubber gloves for splash. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.
- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to “Injuries and Medical Treatment” Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.

- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with *trans*-1-phenyl-2-buten-1-one must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for *trans*-1-phenyl-2-buten-1-one.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using *trans*-1-phenyl-2-buten-1-one must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of *trans*-1-phenyl-2-buten-1-one and understand the hazards.

Lab workers using *trans*-1-phenyl-2-buten-1-one must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with *trans*-1-phenyl-2-buten-1-one described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for

approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines factors) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 5 g of this *trans*-1-phenyl-2-buten-1-one in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this *trans*-1-phenyl-2-buten-1-one with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using *trans*-1-phenyl-2-buten-1-one. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Addition Reaction with Cinchonidine (Cd)

1. A round flask (50 mL) is dried.
2. Cinchonidine (14.7 mg, 0.05 mmol) is put into the flask.
3. Dichloromethane (5 mL) is placed in the flask
4. *trans*-1-Phenyl-2-buten-1-one (73.1 mg, 0.5 mmol) and benzaldehyde (50.8 mL, 0.5 mmol) are added to the flask.
5. The mixture is stirred for 1 day at 40 ^{o}C under N2 atmosphere.
6. The mixture is checked with TLC.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 1/01/2022

trans-Chalcone
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when trans-chalcone ($C_{15}H_{12}O$, CAS No. 614-47-1) used in laboratory. Its purpose is not to have any accident or risk. trans-Chalcone is toxic if swallowed. Also it is harmful if inhaled.

Synonyms: Benzylideneacetophenone

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Toxic by Ingestion, Irritant
GHS Classification
- Acute toxicity, Oral (Category 4)
- Eye irritation (Category 2A)
- Specific target organ toxicity – single exposure (Category 3)

Signs and Symptoms of Exposure
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection
ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection
Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.
- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with trans-Chalcone must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for trans-Chalcone.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using trans-Chalcone must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of trans-Chalcone and understand the hazards.

Lab workers using trans-Chalcone must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with trans-Chalcone described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 5 g of this trans-Chalcone in any given reaction (larger quantities REQUIRE the approval of PI or designee), and

5) discuss ALL issues or concerns regarding this trans-Chalcone with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using trans-Chalcone. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Check the position of GC-peak

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Make a waste bottle labeled as toxic hazardous waste. Review the SDS of trans-Chalcone again; especially remind first aid measures, handling and storage, & PPE.*
3. Take trans-chalcone from the solid cabinet to a balance. Weigh 5 mg of trans-chalcone (5 mg) and add it into a vial. Transfer the closed vial to the fume hood.
4. With a micropipette add toluene (2 mL), close the vial and sonicate for about 1 minute so that the solid disperse well in the solvent.
5. Inject the sample into GC using a Hamilton syringe (10 µL).

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name

_____

Signature

Approval Date: 05/01/2016

trans-Cinnamaldehyde
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when *trans*-cinnamaldehyde (C_9H_8O, CAS No. 14371-10-9) used in laboratory. Its purpose is not to have any accident or risk. *trans*-Cinnamaldehyde is combustible liquid, and may cause an allergic skin reaction, serious eye irritation, and respiratory irritation.

Synonyms: *trans*-3-Phenyl-2-propenal

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Flammable liquid, Irritant
GHS Classification
- Flammable liquids (Category 4)
- Skin irritation (Category 2)
- Eye irritation (Category 2A)
- Skin sensitization (Category 1)
- Specific target organ toxicity - single exposure (Category 3)

Signs and Symptoms of Exposure
Cough, Shortness of breath, Headache, Nausea, Vomiting, To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection
ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection
Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.

- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with *trans*-cinnamaldehyde must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for *trans*-cinnamaldehyde.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using *trans*-cinnamaldehyde must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of *trans*-cinnamaldehyde and understand the hazards.

Lab workers using *trans*-cinnamaldehyde must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with *trans*-cinnamaldehyde described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines factor) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;

3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 25 g of this *trans*-cinnamaldehyde in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this *trans*-cinnamaldehyde with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using *trans*-cinnamaldehyde. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Catalytic Reaction

1. Wear a flammable-resistant lab coat, safety goggles and nitrile chemical-resistant gloves.
2. Check the pressure of H_2 gas cylinder is higher than 40 bar.
3. Examine the head gasket carefully to be sure that it is in good condition. Also check the mating surface on the cylinder and head to be sure that they are clean and free from burrs.
4. Open the vessel, and put the cylinder in fume hood.
5. Put isopropanol (7 mL), Benzyl alcohol (41 mg), and *trans*-Cinnamaldehyde (80 mg) into the stainless steel cylinder
6. Add PtCu/SBA-15 (1 wt.%, 10 mg)
7. Add a stirring bar in the cylinder.
8. The cylinder filled with reagents was connected with the head gasket via tightening the six cap screws, followed by the outer band.
9. Open the valves of H_2 gas tank, introduce the H_2 into burette with the pressure is around 40 bar.
10. Flush the reactor for five times with H_2 at the pressure about 10 bar, and then pressurize the reactor with 10 bar for the hydrogenation.
11. Start the reaction with stirring at 300 K.
12. Release the H_2 every 15 min
13. Take a sample for GC analysis.

Hydrogenation of Cinnamaldehyde

1. Wear a flammable-resistant lab coat, safety goggles and nitrile chemical-resistant gloves.
2. The catalyst is pre-treated under H_2 at 350 ^oC for 3 h before use.
3. The catalyst (20 mg) is mixed with trans-Cinnamaldehyde (0.8 g), benzyl alcohol

(2 mL) and 2-propanol (75 mL).
4. The reaction begins at 100 ºC under H_2 (3 MPa) with stirring.
5. The reaction is stopped after an appropriated time.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 11/15/2016, updated 10/22/2018

Triethylamine
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when triethylamine ($C_6H_{15}N$, CAS No. 121-44-8) used in laboratory. Its purpose is not to have any accident or risk. Triethylamine is highly flammable liquid and vapor, and harmful if swallowed, fatal if inhaled, or in contact with skin. It causes serious eye damage and skin burns.

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Flammable liquid, Toxic by Skin absorption, Harmful by Ingestion, Corrosive, Target Organs (Central Nervous system, Liver, Kidney, Heart)

GHS Classification

Flammable liquids (Category 2)
Acute toxicity, Oral (Category 4)
Acute toxicity, Inhalation (Category 1)
Acute toxicity, Dermal (Category 2)
Skin corrosion (Category 1A)
Serious eye damage (Category 1)
Acute aquatic toxicity (Category 3)

Signs and Symptoms of Exposure

Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin, spasm, inflammation and edema of the larynx, spasm, inflammation and edema of the bronchi, pneumonitis, pulmonary edema, burning sensation, Cough, wheezing, laryngitis, Shortness of breath, Headache, Nausea, Vomiting

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be

buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.
- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.

- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with triethylamine must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for triethylamine.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using triethylamine must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of triethylamine and understand the hazards.

Lab workers using triethylamine must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with triethylamine described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 10 mL of this triethylamine in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this triethylamine with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using triethylamine. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

SBA-15 Silylation 1

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle
2. Add SBA-15 (200 mg) and two magnetic stir bars to two round-bottom flasks (50 mL).
3. Create inert N_2 or Ar atmosphere in each flask using a combination of a syringe and vent needle. Allow gas to cycle through the flask for 5-10 min.
4. Add toluene (5 mL) to each flask.
5. Add triethylamine (0.3 mL) to each flask.
6. Add chlorotrimethylsilane 0.2 mL to one flask and 0.1 mL to the other flask.
7. Allow reaction to stir over magnetic stir plate for 24 hours.
8. Expose both flasks to air and quench each reaction with methanol (5 mL).
9. Vacuum filter both mixtures and wash powder with acetone (10 mL) and DI water for each.
10. Dry both sets of powders by vacuum filtering for 5-10 min.
11. Set each sample (10 mg) aside in a plastic storage container.
12. Set each sample (10 mg) aside in a plastic storage container.

SBA-15 Silylation 2

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle
2. SBA-15 (200 mg) is put into a round-bottom flask (25 mL).
3. The flask is purged with Ar gas for 5 minutes.
4. Toluene (5 mL) is added to the flask

5. Triethylamine (0.3 mL) and chlorotrimethylsilane (0.2 mL) are added to the flask.
6. The resulting mixture is stirred for 24 hours
7. Then the mixture is exposed to air and quenched with methanol (5 mL).
8. The mixture is filtered and washed with water (10 mL) and acetone (10 mL).
9. Finally the mixture is left to dry under vacuum.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 07/08/2020, 07/10/2020

Trimethyl(methylcyclopentadienyl)platinum
STANDARD OPERATING PROCEDURE

Type of SOP: ☐ Process ☒ Hazardous Chemical ☐ Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when trimethyl(methylcyclopentadienyl)platinum ($C_5H_4CH_3Pt(CH_3)_3$, CAS No. 94442-22-5) used in laboratory. Its purpose is not to have any accident or risk. Trimethyl(methylcyclopentadienyl)platinum is highly toxic by ingestion. Also it is harmful if inhaled, if swallowed, and if absorbed through skin. It may cause respiratory tract irritation, skin irritation, and eye irritation.

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Toxic by Ingestion, Skin sensitizer
GHS Classification

Acute toxicity, Oral (Category 1)
Acute toxicity, Dermal (Category 2)
Skin sensitization (Category 1)
Acute aquatic toxicity (Category 1)

Signs and Symptoms of Exposure
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or http://www.showabestglove.com/site/default.aspx

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

All chemicals should be transferred and used in an annually certified laboratory chemical fume hood with the sash at the certified position or lower. The hood flow alarm should be checked to be operating correctly prior to using the hood.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Wash thoroughly after handling. Do not ingest or inhale nor get in eyes, skin or clothing. Remove contaminated clothing and wash before reuse.

Store in a tightly closed, labeled container and in a cool, dry well-ventilated area. Segregate from incompatible materials. Secondary containers must be labeled clearly. Follow any substance-specific storage guidance provided in Safety Data Sheet documentation.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

6. SPILL AND INCIDENT PROCEDURES

Chemical Spill - Dial 911 and EH&S 951-827-5528

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

- *Small* – If you have training, use appropriate personal protective equipment and clean-up materials for chemical spilled. Double bag spill waste in clear plastic bags, label, and arrange for chemical waste pick-up.
- *Large*– Dial 911 and EH&S at 951-827-5528 for assistance. Notify others in area of spill. Turn off ignition sources in area. Evacuate area and post doors to spill

area. Remain on the scene, but at a safe distance, to receive and direct safety personnel when they arrive.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. Notify supervisor and EH&S at 951-827-5528 immediately.

Medical Emergency - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

7. DECONTAMINATION

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

8. WASTE DISPOSAL

All waste must be disposed through the EH&S Hazardous Waste Program. Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

General hazardous waste disposal guidelines:

- Affix an on-online hazardous waste tag using the Online Tag Program (OTP - https://otp.ucop.edu/) on all waste containers as soon as the first drop of waste is added to the container.
- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: http://ehs.ucr.edu/services/waste.html

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with trimethyl(methylcyclopentadienyl)platinum must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for trimethyl(methyl-cyclopentadienyl)platinum.

11. SAFETY DATA SHEETS

Online SDS can be found at http://www.ehs.ucr.edu/services/msds.html.

12. DETAILED PROTOCOL

All lab workers who will be using trimethyl(methylcyclopentadienyl)platinum must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of trimethyl(methylcyclopentadienyl)platinum and understand the hazards.

Lab workers using trimethyl(methylcyclopentadienyl)platinum must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with trimethyl(methylcyclopentadienyl)platinum described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale- of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;

2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 0.5 g of this trimethyl(methylcyclopentadienyl)platinum in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this trimethyl(methylcyclopentadienyl)platinum with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using trimethyl(methylcyclopentadienyl)platinum. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Trimethyl(methylcyclopentadienyl)platinum Sample Preparation:

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggle.
2. Leak valve and 1’33” flange with sealed-off Pyrex glass end tube should be dried in the oven for 1 hour, cooled down to room temperature.
3. Take the leak valve, the flange, 1’33” copper gasket, and self-made spatula to Prof. Bocian’s lab, follow the procedure of using the glovebox, transfer about 1 cm^3 of this trimethyl(methylcyclopentadienyl)platinum to the flange, and close the leak valve inside the glovebox.
4. Care should be taken to fast mount the leak valve to chamber.
5. When the pressure in the preparation chamber is below $3x10^{-7}$ torr, open the leak valve, and wait until the pressure is down again.
6. Heat the glass tube to around 303 K, and keep the chamber, especially the parts on the delivery pass, at around 313 K.
7. Control the leak valve, and do the experiments.
8. After each experiment, heating the sample and chamber should be stopped to protect the o-rings around transfer rod.
9. After experiment, the silicon sample needs to be treated as solid hazardous waste. The glass tube needs to be cleaned with acetone and water, and the waste solution should be placed into the proper waste container.

Operating the Glove Box

Trimethyl(methylcyclopentadienyl)platinum is air/moisture sensitive, so it cannot be handled in the fume hood in room 135. It should be handled inside the glove box.

1. A glove box is a sealed container that is designed to allow users to manipulate objects where an inert atmosphere is desired. It has an antechamber, which has two doors connect to the glove box and the outside. Follow the steps below when operate the glove box.
2. Check the pressure gauge on ultrahigh pure Nitrogen cylinder. If the pressure is low (below 200 psi), do not use the glove and change the gas cylinder. Check the pressure gauge on the antechamber chamber. If it is under vacuum, switch the knob below the antechamber chamber from Evacuate to Refill position, and then switch back to CLOSED position. This will isolate the antechamber chamber from the glove box.
3. Open the outside door carefully, and then transfer the new chemicals, container (made by a glass metal adapter) with valves, and tools onto the tray inside. Close the outside door.
4. Switch the knob from Closed to Evacuate, wait for 5 min. Then move to Refill position. The pressure in the antechamber chamber will increase. Evacuate and Refill the antechamber chamber two more times. It is suggested to leave the knob in closed position after the 3rd refill.
5. Put hands in the glove box and open the inside door slowly, monitor the oxygen and water level on the control panel (both should be below 1 ppm). Move the tray inside the glove box and transfer the chemical precursor into the container. Connect the container to a valve and close the valve. Leave the air/sensitive chemical bottle inside the glove box. Label the bottle with chemical name and user name and contact information.

UHV #1, Victor Chamber

1. Wear nitrile gloves, flame-resistant lab coat, and safety goggle.
2. Bring the sealed-off Pyrex glass end tube with 1’33 flange and swagelok valve to the glove box as sample container. Fill the tube with trimethyl(methylcyclopenta dienyl)platinum (~1 cm3) inside the glove box.
3. Close the swagelok valve and seal the tube. Take the container out of the glove box.
4. Connect the sample container to the leak valve on the UHV system. Slowly open the swagelok valve and the leak valve to pump down the container to UHV conditions.
5. To dose trimethyl(methylcyclopentadienyl)platinum, heat up the glass tube to about 303 K using silicone oil and the delivery line to about 313 K using heating tapes. Control the leak valve to obtain proper vapor pressure in the chamber for experiments.

6. After experiments, the Pt metal deposited on substrate can be removed using Ar sputtering and annealing. The glass tube needs to be cleaned with acetone and ethanol and the waste solution should be placed into the proper waste container.

Trimethyl(methylcyclopentadienyl) platinum Sample Preparation

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggle.
2. Clean and then dry the sealed-off Pyrex glass end tube (sample container) in the oven.
3. Cool down to room temperature.
4. Transfer the sample container, Swagelok valve, spatulas, and proper size of wrenches in the glove box. Follow the procedure of using the glove box, transfer Trimethyl(methylcyclopentadienyl)platinum (0.1–0.3 mg) to sample container.
5. Connect the container to the Swagelok valve and close the valve.
6. Take the sample container with valve and connect it to the ALD reactor.
7. Use liquid N_2 to freeze the glass end, slowly open the Swagelok valve to pump down the sample container.
8. In the experiment, the Trimethyl(methylcyclopentadienyl)platinum sample container is held at around 30 °C. Use Swagelok valve to control the exposure pressure.

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

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