Diethyl ether
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 diethyl ether ($C_4H_{10}O$, CAS No. 60-29-7) used in laboratory. Its purpose is not to have any accident or risk. Diethyl ether is extremely flammable liquid and vapor, and causes serious eye irritation. It is harmful if swallowed and inhaled. Also it may cause drowsiness and dizziness.

Synonyms: Ether, Ethyl ether

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, Kidney, Skeletal muscle, Gastrointestinal tract), Harmful by Ingestion, Irritant

GHS Classification

Flammable liquids (Category 1)
Acute toxicity, Oral (Category 4)
Acute toxicity, Inhalation (Category 5)
Eye irritation (Category 2A)
Specific target organ toxicity - single exposure (Category 3)

Signs and Symptoms of Exposure

Cough, chest pain, Difficulty in breathing, Dizziness, Drowsiness, Contact with eyes can cause Redness, Provokes tears, and Blurred vision, Prolonged or repeated exposure to skin causes defatting and dermatitis.

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 diethyl ether 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 diethyl ether.

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 diethyl ether must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of diethyl ether and understand the hazards.

Lab workers using diethyl ether 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 diethyl ether 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 < 100 mL of this diethyl ether in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this diethyl ether 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 diethyl ether. 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.

Dialcohol Conversion

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Add Pt/SiO_2 or Au/TiO_2 catalyst (up to 50 mg) and water (3 mL) into a 10 mL of two-neck flask.
3. Add 2-phenyl-1,2-propanediol (76 μL) into the flask.
4. Connect the flask to a reflux condenser.
5. Attach the oxygen gas line to one of necks and adjust oxygen gas flow 10 mL/min or higher.
6. Put the flask in an oil bath at 60 ^oC.
7. Stir the mixture for 1 or 2 days in a fume hood. Leave a label with chemical name and hazard information.
8. Extract the mixture with ether (5 mL, 2 times)
9. Wash the extracted organic layer with water (2 mL, 2 times)
10. Remove any volatiles under evaporator.

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 ^oC 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).

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 mmol) into the flask.
4. Add ether (400 mL) into the flask.
5. Put toluene (40 mL and oxalyl chloride (6.35 g, 50 mmol) 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.

GC Measurement

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Into 0.5 mL of a reaction mixture containing water, benzyl alcohol or other alcohol, add diethyl ether (1.5 mL) via a pipette.
3. Pass through pipette filter containing silica and sodium sulfate.
4. Rinse with diethyl ether (2 mL).
5. Inject 4 μL into GC.
6. Store the reaction mixture in an appropriately labeled vial

Oxidation reaction

1. Wear nitrile chemical-resistant gloves, mask, flame-resistant lab coat, and safety goggles.

2. Take 25 μL of 2-phenylethanol by a syringe in a fume hood, and add it to a reaction mixture containing potassium carbonate (52 mg) in water (8.5 mL).
3. Add Au-nanoparticles supported on titania (20 mg).
4. After injecting, clean syringe by thoroughly rinsing with ether. Dispose washing in appropriate wastes container.
5. After reaction is finished, store reaction mixture in an appropriate labeled vial.

Washing a syringe

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and full-face respirator.
2. *Make a waste bottle labeled as toxic and carcinogen hazardous waste. Review the SDS of benzene, and diethyl ether again; especially remind first aid measures, handling and storage, PPE, & signs and symptoms of exposure.*
3. After adding all the solid parts and a solvent into a test tube, stir it closed with a rubber septum in the fume hood.
4. Take a bottle of benzene from a 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.*
5. Remove the septum from the test tube and open the benzene bottle. Add benzene (12.5 μL) 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 diethyl 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.
6. 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.*

Synthesis of 1,4-Dibenzylpiperazine-2,3,5-trione

1,1'-(1.2-dioxoethane-1,2-diyl)bis-1H-benzotriazole Synthesis

1. Wear nitrile chemical–resistant gloves, a flame-resistant lab coat, and safety goggles.
2. A flask (500 mL) and dropping funnel are dried under nitrogen.
3. Benzotriazole (11.9 g, 100 *m*mol) was put into a flask.

4. Ether (400 mL) is added to the flask.
5. Toluene (40 mL) and oxalyl chloride (6.35 g, 50 *m*mol) are added to the dropping funnel.
6. A solution of oxalyl chloride is slowly dropped to the flask.
7. The mixture is stirred for 20 h at room temperature.
8. The mixture is filtered and washed with ether.
9. White powder is dried.

N-benzyl-2-(benzylamino)acetamide Synthesis

1. Wear nitrile chemical–resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Methanol (40 mL), benzylamine (10.9 mL, 100 *m*mol) and methyl bromoacetate (1.4 mL, 15 *m*mol) are added to a flask (50 mL).
3. The mixture is stirred for a week at room temperature.
4. Methanol is removed by evaporator.
5. The crude is distilled to remove the excess of benzylamine.
6. The residue is purified by column.

1,4-dibenzylpiperazine-2,3,5-trione Synthesis

1. Wear nitrile chemical–resistant gloves, a flame-resistant lab coat, and safety goggles.
2. N-Benzyl-2-(benzylamino)acetamide (150.4 mg) and 1,1'-(1,2-dioxethane-1,2-diyl)bis-1*H*-benzotriazole (180 mg) are put into a vial.
3. 1,4-Dioxane (2 mL) is added to the vial.
4. The mixture is stirred at 90 ^{o}C for 1 day.
5. After reaction, the solvent is removed.
6. The mixture is columned.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 06/01/2015, 03/11/2016, 02/07/2020

Ethanol
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 ethanol (C_3H_6O, CAS No. 67-64-1) used in laboratory. Its purpose is not to have any accident or risk. Ethanol is highly flammable liquid and vapor, and causes serious eye and skin irritation. Also it may cause respiratory 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: Flammable liquid, Target organ effect (Liver and Kidney), Irritant
GHS Classification

Flammable liquids (Category 2)
Skin irritation (Category 3)
Eye irritation (Category 2A)
Specific target organ toxicity - single exposure (Category 3)

Signs and Symptoms of Exposure
Central nervous system depression, narcosis, Damage to the heart.

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 ethanol 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 ethanol.

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 ethanol must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of ethanol and understand the hazards.

Lab workers using ethanol 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 ethanol 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 < 200 mL of this ethanol in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this ethanol 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 ethanol. 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.

For washing solvent

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Ethanol is used to wash and clean the hardware that will be used for UHV system.
3. Always put ethanol in a wash bottle.
4. Only use the necessary amount.
5. Collect all the used ethanol in a beaker and dispose into a proper waste bottle.

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 ^{o}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).

Functionalization of Silica nanospheres

1. Wear nitrile chemical resistant glove, flame-resistant lab coat, and safety goggles.
2. Bring out 20 µL of 3-aminopropyltriethoxysilane in a sealed vial from the glove box.
3. Add to isopropyl alcohol (20 mL).
4. Add the mixture to the prepared silica spheres (dispersed in ethanol).

5. Heat at 80 °C for 2 h, then wash the NH_2 functionalized particles in ethanol.
6. Dispose off the washings as hazardous organic waste.

Functionalization of Silica nanospheres

1. Wear nitrile chemical resistant glove, flame-resistant lab coat, and safety goggles.
2. Bring out 23.6 µL of 3-aminopropyltrimethoxysilane in a sealed vial from the glove box.
3. Syringe it to the prepared silica spheres dispersed in ethanol (200 mL).
4. Stir for 12 h, and then reflux for 1 h,
5. Wash the NH_2 functionalized particles in ethanol.
6. Dispose off the washings as hazardous organic waste.

TiO_2 coating onto the silica particle 1

1. Wear nitrile chemical-resistant gloves, mask, flame-resistant lab coat, and safety goggles.
2. Take titanium butoxide (1 mL) by a syringe in a fume hood.
3. Put ethanol (3 mL) into a glass vial.
4. Inject the titanium butoxide into the glass vial and stir vigorously.
5. Put a syringe needle into sharps-disposal container.

TiO_2 coating onto the silica particle 2

1. Wear nitrile chemical resistant glove, flame-resistant lab coat, and safety goggles.
2. Add titanium butoxide (0.5 mL) via a syringe to ethanol (5 mL).
3. The titanium butoxide solution should be added drop wise into a refluxing suspension of Au coated silica particles (dispersed in absolute ethanol – water 1% mixture).
4. Rinse syringes several times with ethanol before disposal.
5. The washings should be disposed as hazardous organic waste.

Making of titania shells

1. Wear nitrile chemical-resistant glove, mask, flame-resistant lab coat, and safety goggle.
2. *Make a waste bottle labeled as toxic and corrosive hazardous waste. Review the SDS of acetonitrile, ethanol, ammonium hydroxide, hydroxypropyl cellulose and titanium butoxide 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. Close with a rubber septum and take it to a balance. Weigh hydroxypropyl cellulose (50 mg) and add it into the flask. Transfer the closed flask back to the fume hood.
4. Disperse silica spheres with gold nanoparticles in ethanol (21 mL) and add the dispersion to the flask.
5. With a micropipette add acetonitrile (7 mL) and close with the septum. Stir for 20 minutes.
6. Take the bottle of ammonium hydroxide from a corrosive base cabinet and place it into the fume hood. Remove the septum from the flask and add ammonium hydroxide (0.2 mL) by using a micropipette. Stir for 20 minutes.
7. Take titanium butoxide from the flammable cabinet and put it into the fume hood. Add ethanol (3 mL), acetonitrile (1 mL), and titanium butoxide into a vial and be careful not to expose titanium butoxide to air. Mix the vial well and add the mixture slowly into the main mixture. Stir for 2 hours.
8. Wash the mixture with ethanol 4 times.
9. Dispose the waste into the waste bottle labeled toxic and corrosive hazardous waste.

Synthesis of silica spheres

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggle.
2. *Make a waste bottle labeled as toxic hazardous waste. Review the SDS of tetraethyl orthosilicate, ethanol, and ammonium hydroxide 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. Add ethanol (72 mL) and milli-Q water (12 mL) to the flask and close with rubber septum. Stir for 5 minutes.
5. Take the bottle of ammonium hydroxide from a corrosive base cabinet and place it into the fume hood. Remove the septum from the flask and add ammonium hydroxide (1.95 mL) by using a micropipette. Stir for 5 minutes.
6. Take tetraethyl orthosilicate (TEOS) from the flammable cabinet and put it into the fume hood. Add TEOS (2.55 mL) to the mixture.
7. Stir for 4 hours at room temperature.
8. Centrifuge and dispose the waste into the waste bottle labeled toxic and corrosive hazardous waste.
9. Label the centrifuge tube appropriately, cover it with perforated aluminum foil and dry the powder in a vacuum desiccator overnight.

Catalytic Hydrogenation of Cinnamaldehyde

1. Wear nitrile chemical resistant glove, flame-resistant lab coat, and safety goggle.

2. Catalyst (50–200 mg), cinnamaldehyde (0.5–3 *m*mol), and ethanol (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.

For washing solvent

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggle.
2. Ethanol is used to wash and clean the hardware that will be used for high-pressure reactors.
3. Always put ethanol in a wash bottle.
4. Only use the necessary amount.
5. Collect all the used ethanol in a beaker and dispose into a proper waste bottle

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 ethanol to the fume hood in room 135.
3. Dissolve nickel nitrate (0.0495 g) precursor in ethanol (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).

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.
14. Use acetone or ethanol to wash the sample holder.

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.

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 Pt@Cu_6/SBA-15, #2

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 (25 mL) to the flask.
4. Add Pt/SBA-15 (0.1 g) 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. Wash the mixture with H_2O/ethanol 2 times
9. 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 ^oC 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.

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 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: 02/01/2013, updated 06/01/2015, 03/01/2016, 05/15/2016, 10/10/2017, 08/07/2018, 09/27/2021, 10/12/2021, 1018/2021, 10/19/2021, 01/21/2022, 08/24/2022

Ethyl acetate
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 ethyl acetate ($C_4H_8O_2$, CAS No. 141-78-6) used in laboratory. Its purpose is not to have any accident or risk. Ethyl acetate is highly flammable liquid and vapor, and causes serious eye and skin irritation. Also it may be harmful if inhaled and cause drowsiness and dizziness.

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 (Blood, Kidney, Liver and Central nervous system), Irritant

GHS Classification

Flammable liquids (Category 2)
Acute toxicity, Inhalation (Category 5)
Eye irritation (Category 2A)
Specific target organ toxicity - single exposure (Category 3)

Signs and Symptoms of Exposure

Central nervous system depression, Drowsiness, narcosis, anemia

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 ethyl acetate 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 ethyl acetate.

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 ethyl acetate must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of ethyl acetate and understand the hazards.

Lab workers using ethyl acetate 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 ethyl acetate 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 ethyl acetate in any given reaction (larger quantities REQUIRE the approval of PI or designee), and

5) discuss ALL issues or concerns regarding this ethyl acetate 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 ethyl acetate. 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.

TLC monitor

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take ethyl acetate (100 mL) into a flask in a fume hood, and then add another solvent, mix the two solvents together for TLC monitor.
3. The collect solvent needs to be treated as hazardous waste.
4. Washing and cleaning solvents also need to be treated as hazardous waste.

Catalytic Reaction

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take 2-Hydroxybenzyl alcohol (31 mg) into an Erlenmeyer flask (30 mL) 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.

Catalytic Reaction

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take 4-hydroxybenzyl alcohol (31 mg) into an Erlenmeyer flask (30 mL) in the fume hood of room 135.
3. Add potassium carbonate (103.7 mg) and water (5 mL).
4. Add aqueous Au-PVP catalyst (0.5 mM, 10 ml, 2 atom.%), 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.

Oxidation to aldehyde

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take benzyl alcohol (31 mg) into an Erlenmeyer flask (30 mL) in a 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%), 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.

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 B: Purification of the hydrochloride salt

1. A reflux condenser is attached to the top of a flask (250 mL).
2. The yellow salt is put into the flask.
3. Methanol (40 mL) is added to the flask.
4. The mixture is refluxed.
5. Ethyl acetate (20 mL) is slowly added to the flask.
6. The mixture is cooled down to room temperature.
7. The flask is put in refrigerator.
8. The solid is filtered and washed with ethyl acetate (10 mL).
9. The solid is dried under vacuum.

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 02/01/2013, updated 03/01/2014, 03/20/2016, 02/07/2020

Ethyl pyruvate
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 ethyl pyruvate ($C_5H_8O_3$, CAS No. 617-35-6) is used in laboratory. Its purpose is not to have any accident or risk. Ethyl pyruvate is combustible liquid and may cause eye irritation. Also it may be harmful if inhaled, if absorbed through skin, or if swallowed.

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

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

OSHA Hazards: Combustible Liquid
GHS Classification
Flammable liquids (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 ethyl pyruvate 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 ethyl pyruvate.

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 ethyl pyruvate must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of ethyl pyruvate and understand the hazards.

Lab workers using ethyl pyruvate 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 ethyl pyruvate 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:

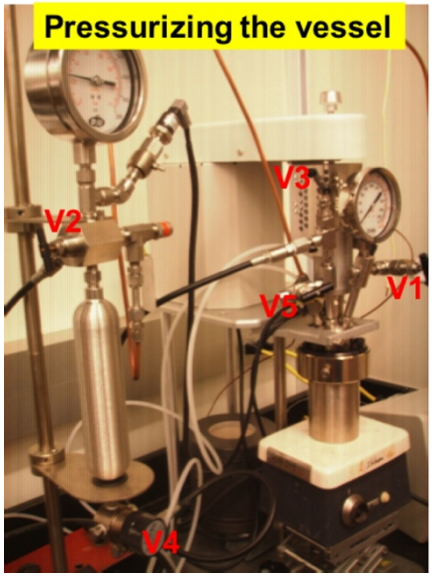
1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);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

5) discuss ALL issues or concerns regarding this ethyl pyruvate 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 ethyl pyruvate. 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.

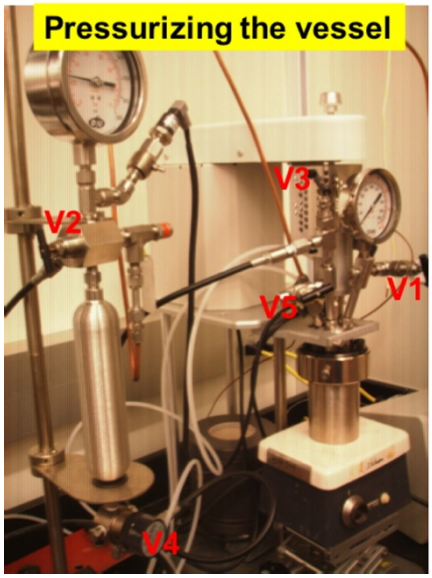
Hydrogenation of Ethyl-Pyruvate (high pressure)

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Transfer toluene (10 mL) or any other solvent to a beaker (50 mL).
3. Add certain amount of ethyl pyruvate into the beaker.
4. Stir the mixture for 2 minutes until ethyl pyruvate is fully dissolved
5. Seal the beaker with Para-film.
6. Rinse the stainless cylinder of the high-pressure reactor with ethanol and toluene. Let it dry before adding reactants.
7. Add Pt/Al_2O_3 or other catalyst (1 wt.%, 25 mg) into the cylinder.
8. Inject toluene (4 mL) or any other solvent into the reactor.
9. Add ethyl pyruvate toluene solution (1 mL) to the cylinder. Place a magnetic stirring bar inside.
10. Mount the cylinder onto the fixed head of the reactor. Tighten all 6 screws and mount the outer band in position.
11. Make sure that the V2, V3, V5 and V1 are closed.
12. Open the valve V2, introduce the H_2 into the high-pressure burette with the pressure is around 40 bar.
13. Close the valve V2.
14. Open the valve V3, introduce the H_2 into the cylinder with the pressure is around 20 bar.
15. Close the valve V3.
16. Open the valve V1 to release the H_2 inside the cylinder.
17. Close the valve V1.
18. Repeat step 11 to 17 four more times.
19. Pressurize the cylinder using UHP H_2
20. Start the reaction.
21. When the reaction is done, open the outlet valve to release the pressure.
22. Take a sample for GC analysis.



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. Make sure that the V2, V3, V5 and V1 are closed.
7. Open the valve V2, introduce the H_2 into the high-pressure burette with the pressure is around 40 bar.
8. Close the valve V2.
9. Open the valve V3, introduce the H_2 into the cylinder with the pressure is around 20 bar.
10. Close the valve V3.
11. Open the valve V1 to release the H_2 inside the cylinder.
12. Close the valve V1.
13. Repeat step 6 to 12 four more times.
14. Pressurize the reactor to 10 bar of H2 with stirring.
15. Release the H2 after 10 min of reaction
16. Take a sample for GC analysis.
17. Repeat this step every 10 minutes until 40min of reaction.



SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2013, updated 04/22/2016, 01/25/2019, 09/10/2019

Ethylene glycol
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 ethylene glycol ($C_2H_6O_2$, CAS No. 107-21-1) used in laboratory. Its purpose is not to have any accident or risk. Ethylene glycol is harmful if swallowed. It may cause damage to organ (Kidney) through prolonged or repeated exposure.

Synonyms: 1,2-Ethanediol

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, Target organ effect (Kidney), Irritant
GHS Classification
Acute toxicity, Oral (Category 4)
Specific target organ toxicity – repeated exposure, Oral (Category 2) Kidney

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 ethylene glycol 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 ethylene glycol.

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 ethylene glycol must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of ethylene glycol and understand the hazards.

Lab workers using ethylene glycol 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 ethylene glycol 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 mL of this ethylene glycol in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this ethylene glycol 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 ethylene glycol. 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 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 12h

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.

Synthesis of Pt nanoparticles

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (50 mL) and a beaker (50 mL) under nitrogen in a fume hood and put a stir bar into the flask.
3. Put sodium hydroxide solution (0.25 g, 12.5 mL, 0.5 M) in the beaker.
4. Put ethylene glycol (12.5 mL) in the beaker.
5. Put dihydrogen hexachloroplatinate (0.25 g, 0.48 *m*mol) in the flask.
6. Put ethylene glycol (12.5 mL) into the flask.
7. Add sodium hydroxide solution to dihydrogen hexachloroplatinate solution.
8. Heat the solution at 160 ^{o}C for 3 h, accompanied by N_2 bubbling.
9. Transfer 6 mL aliquot of the resulting solution to a vial.
10. Add hydrogen chloride solution (1 mL of 2 M) and disperse in ethanol containing polyvinylpyrrolidone (12.2 mg).
11. Evaporate the solution.

SBA-15 Impregnation Protocol

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 ethylene glycol to the fume hood in room 135.
3. Dissolve nickel nitrate (0.0495 g) precursor in ethylene glycol (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 ethylene glycol 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).

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 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).

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 11/01/2016, updated 06/01/2017, 09/27/2021, 10/12/2021

Gold chloride trihydrate
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 gold chloride trihydrate ($HAuCl_4 \cdot 3H_2O$, CAS No. 16961-25-4) is used in laboratory. Its purpose is not to have any accident or risk. Gold chloride trihydrate is corrosive and causes skin burns and eye damage. It may cause sensitization by skin contact.

Synonyms: Tetrachloroauric(III) acid, Hydrogen tetrachloroaurate(III)

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

Gold chloride trihydrate is commercially available, and used mostly for gold nanoparticle synthesis in Zaera group. A variety of organic solvents are used to clean sample containers. Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Corrosive, Skin sensitizer
GHS Classification
- Skin corrosion (Category 1A)
- Serious eye damage (Category 1)
- Skin sensitization (Category 1)

Signs and Symptoms of Exposure
No data is available.

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 gold chloride trihydrate 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 gold chloride trihydrate.

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 gold chloride trihydrate must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of gold chloride trihydrate and understand the hazards.

Lab workers using gold chloride trihydrate 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 gold chloride trihydrate 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 g of this gold chloride trihydrate in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this gold chloride trihydrate 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 gold chloride trihydrate. 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.

Gold nanoparticle preparation 1

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Taking a proper amount of gold chloride trihydrate out of a container by plastic spatula.
3. Putting the gold chloride trihydrate into a glass vial.
4. Pouring a proper amount of Milli-Q water in the glass vial and stirring smoothly.

Gold nanoparticle preparation 2

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Add Milli-Q water (1 mL) to hydrogen tetrachloroaurate trihydrate (1 mg).
3. Prepare stock solution by adding 18 µL of the solution (step 2) into Milli-Q water (10 mL).
4. Add 0.5 mL of this stock solution to Milli-Q water (18.5 mL).
5. Add sodium citrate (10 mM, 0.5 mL) and 0.5 mL of sodium borohydride (0.5 mL).
6. Stir for 5 min.
7. Un-used gold particles should be discarded as hazardous waste.

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.

Au Nanoparticle (3 nm) Preparation

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Prepare Au precursor solution (0.01 M), cethyltrimethylammonium bromide solution (CTAB, 0.1 M), and $NaBH_4$ solution (0.01 M).
3. Add the Au solution (0.25 mL, 0.01 M) and the CTAB solution (7.5 mL, 0.1 M) into a vial.
4. Stir the mixture until bright brown-yellow color.
5. Add ice-cold NaBH4 solution (0.6 mL).
6. Mixing for 2 min
7. After color change to pale brown-yellow, store the vial in refrigerator.

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.

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 ºC for 6 h. If left unattended, leave a label nearby with the chemical names and hazard information.
31. After cooling, collect the sample.

Synthesis of Au NPs on APTES-grafted P25 titania

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Dry 2 flasks (250 mL) under nitrogen.
3. Put deionized water (100 mL) into the flask.
4. Add 3-aminopropyltriethoxysilane (APTES)-grafted P25 titania nanoparticles (1 g) into the flask
5. After the supports have dispersed evenly, add a solution of tetrachloroauric acid (15 mL, 10 mM).
6. Maintain suspension by stirring the mixture for 2 h at room temperature. If left unattended in a fume hood, put a label with the chemical name and hazard information.
7. Filter the solid from the mixture, and wash twice with deionized water
8. Put deionized water (100 mL) into the other flask.
9. Redisperse the solid into the flask.
10. For the reduction reaction, add sodium borohydride (2.5 g) into the flask.
11. Filter and wash the solid with deionized water.
12. Dispose waste in the properly labeled container.
13. Collect the sample and dry it in an inert atmosphere (i.e. nitrogen, vacuum) at 60°C overnight. If left unattended, put a label with the chemical name and hazard information.
14. Calcinate the sample for 5 h at 450°C in oxygen.
15. Purge with Ar gas for 10 min.
16. Purge with hydrogen at 350 °C for 1 h.
17. After cooling, collect the sample.

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 02/01/2013, updated 10/01/2014, 03/02/2016, 05/15/2016, 10/19/2021, 01/22/2022, 04/18/2022

Helium
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 helium gas (He, CAS No. 7440-59-7) used in laboratory. Its purpose is not to have any accident or risk. Helium contains gas under pressure. It may be harmful if swallowed, if inhaled or if absorbed through skin. Also it may cause skin 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: Compressed Gas
GHS Classification
Gases under pressure (Compressed Gas)

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

A ventilation monitor is required on each lab hood or gas manifold in which helium gas is used and stored. Acceptable monitors include audible and visual alarms, magnehelic gauge, inclined manometer, or other devices, which indicate that the enclosure is actively ventilated. Manometers and gauges should be clearly marked to indicate safe pressure limits.

The ventilation device is the elephant trunk, or snorkel, which is connected to the exhaust system. This device is effective for capturing discharges from instruments such as gas chromatographs. The intake of the snorkel must be placed very close to the source to be effective. There are newer designs that are mounted on articulating arms, which make the systems more convenient to use.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Always use a proper dolly to carry gas cylinders in building. Avoid inhalation of vapor or mist. Ensure adequate ventilation. Remove all source of ignition; no smoking or electrostatic charge. Beware of vapor accumulating to form explosive concentration. Vapor can accumulate in low areas. Do use right-sized tools and wear heavy protective gloves when connecting a regulator to gas cylinders. Do not breathe any leaked gas. Work in confined spaces. Prevent further leakage or spillage if safe to do so.

All transport of helium gas between on-campus locations must be conducted as follows:

- Gas cylinders must be secured to the transport vehicle (cart, motor vehicle, hand truck, etc.).
- Cylinders must be continuously attended during transport.
- Cylinders must be clearly labeled with content and hazard information.
- Cylinder caps must be in place.

These requirements apply to all the gas containers, including empty and partially full cylinders.

Upon receipt of helium gas cylinders shall be temporarily stored in a well-ventilated area that is attended or locked at all times. All cylinders shall be immediately leak tested with a leak indicating solution and must be clearly labeled with content and hazard information. Temporary storage locations shall have appropriate signage in place. Cylinders must be seismically secured at all locations with chains at two contact points on the cylinder body, using unistruts or an equivalent. Seismic securing should prevent cylinders from rolling, shifting, or falling.

Laboratory storage of all the gas cylinders shall be in a mechanically ventilated, lockable area. Examples of mechanical ventilation include vented gas manifold and fume hoods. Rooms containing toxic gases shall be locked when not occupied by authorized persons. All cylinders and gas manifold must be clearly labeled with content and hazard information. Cylinders shall be seismically secured at all locations with chains (2 contact points), using unistruts or an equivalent for cylinders larger than lecture bottles. Lecture bottles must be secured to a stable surface. Outdoor storage is only allowed on a short-term basis in a secure area at least 75 feet from an exterior door, window, or air intake location.

All regulators, valves, and lines must be chemically compatible with the gases being used. Compatibility can be determined by contacting the gas vendor or by calling EH&S. Regulator/line systems must be leak tested immediately after assembly and before each use. Regulators shall be compatible with the size and type of gas cylinder being used, and rated for full cylinder pressure.

All lines or ducts carrying purged or exhausted emissions of helium gas must be connected to a mechanical exhaust system that discharges to a safe location (i.e., presents no potential for re-entrainment into any building supply air intake or occupied area). Exhaust duct walls shall be chemically resistant to degradation by the toxic gas in use.

Significant emissions of heilum gas require an emission control device (e.g., scrubber, flare device, adsorbent) before the purged gas can be vented into the exhaust duct system. Significant emissions are defined as duct concentrations that result in duct corrosion or acute health risk to persons exposed near exhaust fan stacks as determined by release modeling. When helium gas is emitted from exhaust systems at concentrations which could pose health risks to rooftop workers, locked gates, doors, or other means shall be used to prevent worker access to stack discharge areas. Warning signs must be conspicuously placed.

STORAGE:

It is essential that helium gas is stored separately from all chemicals with which they may react. Ensure segregation of incompatible chemicals per guidance within the UCR

Chemical Hygiene Plan. Also, follow any substance-specific storage guidance provided in Safety Data Sheet (SDS) documentation.

6. SPILL AND INCIDENT PROCEDURES

Emergency procedure for leaking gas cylinders – (see the process SOP, "Emergency Action for Handling Leaking Compressed Gas Cylinder") or http://www.airproducts.com/~/media/Files/PDF/company/safetygram-11.pdf

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

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

7. WASTE DISPOSAL

All empty helium gas cylinders shall be labeled as empty. Depleted helium gas cylinders should be returnable to the vendor according to their guidelines. The purchase of any gases that will not be completely used in the course of research must be approved by the vendor for return, or by EH&S for disposal as hazardous waste. Disposal of helium gas cylinders by EH&S, even when empty, may entail extraordinary costs. Therefore, helium should be purchased only from vendors who will accept returns.

Staff dealing with hazardous waste disposal should have completed UCR Hazardous Waste Management training - http://ehs.ucr.edu/training/online/hwm/indexlms.html

8. PRIOR APPROVAL/REVIEW REQUIRED

All work with helium gas 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.

9. DESIGNATED AREA

A designated area shall be established where limited access, special procedures, knowledge, and work skills are required. A designated area can be the entire laboratory, a specific laboratory workbench, or a laboratory hood. Designated areas must be clearly marked with signs that identify the chemical hazard and include an appropriate warning; for example: WARNING! HELIUM GAS WORK AREA!

10. SAFETY DATA SHEETS

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

11. DETAILED PROTOCOL

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

Lab workers using helium gas 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 helium gas 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) use helium gas under 1 bar in any given reaction (higher pressure REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this helium with the PI prior to its use.

If there is an unusual or unexpected occurrence when using helium gas, the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using helium gas. 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.

Replace empty gas cylinder

1) Borrow a proper dolly from department stockroom.
2) Close the main cylinder valve.
3) Slowly release pressure from regulator into hood to vent.
4) Close the regulator valves.

5) Disconnect the regulator from an empty cylinder.
6) Screw cylinder cap.
7) Deliver the empty cylinder to the stockroom or store temporally in one of hall cabinets.
8) Bring a new gas cylinder to the rack.
9) Safely secure the cylinder using chain clamp.
10) Unscrew cylinder cap.
11) Ensure the main valve is closed.
12) Unscrew the main valve cap.
13) Connect the regulator to the cylinder.
14) Fully open the regulator valves.
15) Get vacuum in the gas manifold and the regulator.
16) Closed the diaphragm valve.
17) Quickly open and close the main cylinder valve to see if the diaphragm valve is working well.
18) If the good sealing is obtained, go ahead. Otherwise, pump the gas in the line and replace the regulator.
19) Set a delivery pressure as needed.
20) Carefully release pressure from regulator.
21) Fully open the main cylinder valve if needed.

Replacing empty gas cylinder for BET Instrument

1. Close the main valve of empty gas tank.
2. Close the regulator valves.
3. Disconnect the regulator from an empty cylinder.
4. Deliver the empty cylinder to the stockroom and bring a new one to the rack.
5. Connect the regulator to the cylinder.
6. Fully open the regulator valves and the main cylinder valve and check the pressure.

UHV #3, Michelle

1. Safely secure Helium cylinder using a chain clamp or ring clamps.
2. Ensure main valve is completely closed.
3. Attach the appropriate pressure regulator and connect to the system using a copper tube.
4. Carefully adjust the outlet pressure to 15 psi.
5. Close the angle valve next to the mechanical pump.
6. Fill the copper tube with Helium gas. Then open the angle valve to pump down.
7. Repeat steps 5-6 three times to purge the copper line.
8. Carefully pressurize copper line.
9. Slowly open the leak valve to leak the gas into the UHV system, monitor the pressure in the UHV system
10. Close the leak valve.

11. Close the valve on the regulator. Close the main valve.
12. Open the angle valve to pump the line.

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 10/01/2013, updated 03/01/2016

Hexamethyldisilazane
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 hexamethyldisilazane ($C_6H_{19}NSi_2$, CAS No. 999-97-3) used in laboratory. Its purpose is not to have any accident or risk. Hexamethyldisilazane is highly flammable liquid and vapor, and toxic in contact with skin. It causes severe skin burns and eye damage. Also it is harmful if swallowed or if inhaled.

Synonyms: HMDS, 1,1,1,3,3,3-Hexamethyldisilazane

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 (Nerves), Corrosive, Toxic by skin absorption, Harmful by Ingestion, Reproductive hazard.

GHS Classification

- Flammable liquids (Category 2)
- Acute toxicity, Oral (Category 4)
- Acute toxicity, Inhalation (Category 4)
- Acute toxicity, Dermal (Category 3)
- Skin corrosion (Category 1B)
- Serious eye damage (Category 1)
- Acute aquatic toxicity (Category 3)
- Chronic 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 or 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 hexamethyldisilazane 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 hexamethyl-disilazane.

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 hexamethyldisilazane must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of hexamethyldisilazane and understand the hazards.

Lab workers using hexamethyldisilazane 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 hexamethyldisilazane 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 hexamethyldisilazane in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this hexamethyldisilazane 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 hexamethyldisilazane. 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.

Silyation of Oxide

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Take 0.5 mL of hexamethyldisiazane into a vial with screw cap containing 10 mL toluene in the fume hood.
3. Add oxide and seal the vial.
4. The reaction is conducted at room temperature.
5. After reaction, the filtrate needs to be treated as hazardous waste.
6. Washing and cleaning solvents also need to be treated as hazardous waste.

Silyation of Si wafer

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggle.
2. Bring the toluene solution bottle together with HMDS to the fume hood.
3. Mix HMDS and Toluene solution in the volume ratio of 1:25
4. Soak the Si wafer in the mixture solution of HMDS and toluene for 24 hours and take the wafer out

5. After washing the Si wafer with toluene, the sample solution needs to be treated as hazardous

Sample Preparation

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggle.
2. Obtaining samples must be operated inside the hood.
3. As hexamethyldisilazane is contained in a Sure-Seal bottle, use of a syringe and needle is required and should be dried prior to the piercing the septum.
4. Ensure the desired amount of hexamethyldisilazane has been drawn into the syringe, pull the tip of the needle above the level of reagent, remove the needle from the reagent bottle and quickly dispense the contents into the target vial or container.
5. Once the addition of reagent is complete, seal the opening of the target container.
6. Seal the bottle and keep air and/or moisture from entering.
7. The needle can be rinsed with hexanes multiple times, then water, and disposed in broken glassware container.
8. The excess residue needs to be treated as liquid hazardous waste and placed into proper waste container.

NiO deposition on HMDS/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 hexamethyldisilazane (HMDS, 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.

TiO_2/HMDS/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 ^{o}C for 2 h.
4. Ti precursor (tetrakis(dimethylamido) titanium, TDMAT) 42 ^{o}C, using heating tape and r
5. The reactor is set to 102 ^{o}C.
6. Dose hexamethyldisilazane (HMDS) 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.

Silylation Procedure for Pt/SBA-15_HMDS

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. A flask is dried.
3. Take hexamethyldisilazane (0.5 mL) into a flask containing toluene (10 mL) in the fume hood.
4. Pt/SBA-15 (40 mg) is put into the flask.
5. The mixture is stirred at 110 ^{o}C for 24 h.
6. The mixture is cooled down to room temperature.
7. The powder is filtered and washed with Toluene.
8. After reaction, the filtrate needs to be treated as hazardous waste.
9. The powder is dried under vacuum.

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 solvent (toluene or ethanol, 30 mL).
5. Slowly add hexamethyldisilazane (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, 07/01/2018, 10/12/2021, 12/21/2021, 08/24/2022

Hexane
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 hexane (C_2H_{14}, CAS No. 110-54-3) used in laboratory. Its purpose is not to have any accident or risk. Hexane is highly flammable liquid. Vapor may cause drowsiness and dizziness. It also causes eye, skin, and respiratory tract irritations. It may be harmful if swallowed, if inhaled, or if adsorbed through skin.

Synonyms: n-Hexane

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 (Peripheral nervous system, Kidney, Testes) Effect, Irritant

GHS Classification

Flammable liquids (Category 2)
Skin irritation (Category 2)
Eye irritation (Category 2B)
Reproductive toxicity (Category 2)
Aspiration hazard (Category 1)
Acute aquatic toxicity (Category 1)

Signs and Symptoms of Exposure

Prolonged or repeated contact with skin may cause:, defatting, Dermatitis, Contact with eyes can cause:, Redness, Blurred vision, Provokes tears., Effects due to ingestion may include:, Gastrointestinal discomfort, Central nervous system depression, Lung irritation, chest pain, pulmonary edema, giddiness, slowed reaction time, slurred speech, Headache, Dizziness, Drowsiness, Unconsciousness

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 hexane 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 hexane.

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 hexane must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of hexane and understand the hazards.

Lab workers using hexane 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 hexane 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 hexane in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this hexane 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 hexane. 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.

For washing solvent

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Hexane is used to wash and clean the hardware that will be used for UHV system.
3. Always put hexane in a wash bottle.
4. Only use the necessary amount.
5. Collect all the used hexane in a beaker and dispose into a proper waste bottle.

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 ^{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 °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).

Sputter Gun Cleaning

1. Wear nitrile chemical resistance gloves, flame resistance lab coat, and safety goggles.
2. Disassemble the sputter gun. Make sure how it is assembled (take photos).
3. Place six beakers (600 mL) in the fume hood and label them from 1 to 6.
4. Add water (300 mL) into the beaker 1.
5. Add Liquinox detergent (~50 mL) to prepare the soap solution.
6. Place the disassembled sputter gun parts into the beaker 1 with the soap solution.
7. Sonicate it for 5 minutes.
8. Add warm water (300 mL, ~50 °C) into the beaker 2.
9. Transfer the sputter gun parts from the beaker 1 to the beaker 2 using tweezers.
10. Sonicate the beaker 2 for 5 minutes.
11. Add deionized water (300 mL) into the beaker 3.
12. Transfer the sputter gun parts from the beaker 2 to the beaker 3 using tweezers.
13. Sonicate the beaker 3 for 5 minutes.
14. Add methanol (300 mL) into the beaker 4 in the fume hood.
15. Transfer the sputter gun parts from the beaker 3 to the beaker 4 using tweezers.
16. Sonicate the beaker 4 for 5 minutes.
17. Repeat the above step (steps 14-16) with acetone (300 mL) and hexane (300 mL) sequentially.
18. Remove the sputter gun components from the hexane solution.
19. Air-dry it for an hour and assemble it carefully.
20. The sputter gun is mounted in the UHV chamber.
21. Dispose the washing and cleaning solvents properly.

For Injecting into GC

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Turn on GC and set the method to heat up the injection port to above 69 °C.
3. Pour a small amount of hexane into a beaker.
4. Use a syringe to take a sample of hexane from the beaker.
5. Inject hexane into GC.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2013, updated 03/01/2014, 03/01/2016, 11/20/2019, 6/16/2022

Hydrochloric 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 hydrochloric acid (HCl, CAS No. 7647-01-0) is used in laboratory. Its purpose is not to have any accident or risk. Hydrochloric acid is corrosive liquid. It causes severe skin burns and eye damage. Also, It may be harmful if inhaled, if absorbed through skin, or if swallowed.

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
Burning sensation, Cough, wheezing, laryngitis, Shortness of breath, spasm, inflammation and edema of the larynx, spasm, inflammation and edema of the bronchi, pneumonitis, pulmonary edema, 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 hydrochloric 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 hydrochloric 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 hydrochloric acid must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of hydrochloric acid and understand the hazards.

Lab workers using hydrochloric 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 hydrochloric 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-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 hydrochloric acid in any given reaction (larger quantities REQUIRE the approval of PI or designee), and

5) discuss ALL issues or concerns regarding this hydrochloric 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 hydrochloric 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.

Si wafer washing

1. Wear nitrile chemical-resistant gloves, water-resistant lab coat, and safety goggles.
2. Bring the HCl and H_2O_2 solution bottles to a fume hood.
3. Mix HCl, H_2O_2 and H_2O in the volume ratio of 1:1:6, and heat the mixture up to 70 ^oC. Then load Si wafer into the solution for 10 min.
4. (4)Take the wafer out and wash with DI water for 5mins and blow dry with N_2 gas
5. After treatment, the solution needs to be treated as hazardous

Preparation for pH controlling agent

1. Wear nitrile chemical-resistant glove, mask, flame-resistant lab coat, and safety goggles.
2. Take a proper amount of hydrochloric acid in a pipette at the fume hood in the room 135.
3. Pour a calculated amount of Milli-Q water in to the glass bottle.
4. Inject the hydrochloric acid into the glass bottle and stir the mixture smoothly.

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 ^oC 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).

Reaction

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and a safety goggles.
2. Pluronic® F-127 (1.0 g) is dissolved in HCl (2 M, 60 mL), KCl (2.5 g), and 1,3,5-trimethylbenzene (1.0 g).
3. This is stirred at room temperature for 24 hours.
4. Tetramethyl orthosilicate (4.1 g) is added drop-wise to the mixture and stirred for 24 hours.
5. The solution undergoes hydrothermal treatment at 100 °C for 48 hours, then filtered and rinsed with H_2O.

Catalytic Reaction

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and a safety goggles.
2. Take 2-Hydroxybenzyl alcohol (31 mg) into an Erlenmeyer flask (30 mL) 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.

Catalytic Reaction

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take 4-hydroxybenzyl alcohol (31 mg) into an Erlenmeyer flask (30 mL) in the fume hood of room 135.
3. Add potassium carbonate (103.7 mg) and water (5 mL).

4. Add aqueous Au-PVP catalyst (0.5 mM, 10 ml, 2 atom.%), 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.

Oxidation to aldehyde

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take benzyl alcohol (31 mg) into an Erlenmeyer flask (30 mL) in a 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%), 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.

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 (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 (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.

Synthesis of Pt nanoparticles

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (50 mL) and a beaker (50 mL) under nitrogen in a fume hood and put a stir bar into the flask.
3. Put sodium hydroxide solution (0.25 g, 12.5 mL, 0.5 M) in the beaker.
4. Put ethylene glycol (12.5 mL) in the beaker.
5. Put dihydrogen hexachloroplatinate (0.25 g, 0.48 *m*mol) in the flask.
6. Put ethylene glycol (12.5 mL) into the flask.
7. Add sodium hydroxide solution to dihydrogen hexachloroplatinate solution.
8. Heat the solution at 160 ^{o}C for 3 h, accompanied by N_2 bubbling.
9. Transfer 6 mL aliquot of the resulting solution to a vial.
10. Add hydrogen chloride solution (1 mL of 2 M) and disperse in ethanol containing polyvinylpyrrolidone (12.2 mg).
11. Evaporate the solution.

ICP Sample Preparation

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Weigh a sample (~3 mg).
3. Prepare 20 mL of aqua regia solution (V_{HCl}: V_{HNO3} = 3:1)
4. Dissolve samples in the aqua regia solution.
5. Heat the solution at 95 ^{o}C until evaporation to approximately 5 mL.
6. Filter the solution.
7. Wash the beaker and the filter paper.
8. Quantitatively transfer the solution to a volumetric flask (100 mL)
9. Dilute to volume with water.

Flask Washing

1. Wear nitrile chemical-resistant glove, mask, flame-resistant lab coat, and safety goggles.
2. Bring the HCl and HNO_3 solution bottles to a fume hood.
3. Mix HCl, HNO_3 in the volume ratio of 3:1, then pour the mixture into the flask, let it stay for 5 mins.
4. Pour the mixture out and treat it as hazardous.
5. Wash the flask with D.I. water for 3 times and dry in the vacuum oven.

ICP Pretreatment

1. Wear nitrile chemical-resistant glove, mask, flame-resistant lab coat, and safety goggles.
2. Bring the HCl and HNO_3 solution bottles to a fume hood.
3. Mix HCl, HNO_3 in the volume ratio of 3:1, then pour the mixture into the flask with sample inside.
4. Heat the mixture up to 100 ^oC for 1 hour. Then heat up to 120 ^oC for 1 hour.
5. Cool the mixture down to room temperature, then add the mixture to a volumetric flask.
6. Pour desired amount of water to the volumetric flask.

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.

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. THF (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 °C.
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 °C.
11. Water (3.5 mL) is added into the flask.
12. The mixture is stirred overnight at 45 °C.
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: 06/01/2013, updated 03/01/2014, 03/01/2016, 05/15/2016, 06/01/2017, 07/01/2018, 07/16/2019, 02/07/2020

Hydrogen
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 hydrogen (H_2, CAS No. 1333-74-0) used in laboratory. Its purpose is not to have any accident or risk. Hydrogen is highly flammable gas, and contains gas under pressure. It may explode if heated.

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 gas, Compressed Gas
GHS Classification
Flammable gas (Category 1)
Gas under pressure (Liquefied gas)

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

A ventilation monitor is required on each lab hood or gas manifold in which hydrogen gas is used and stored. Acceptable monitors include audible and visual alarms, magnehelic gauge, inclined manometer, or other devices, which indicate that the enclosure is actively ventilated. Manometers and gauges should be clearly marked to indicate safe pressure limits.

The ventilation device is the elephant trunk, or snorkel, which is connected to the exhaust system. This device is effective for capturing discharges from instruments such as gas chromatographs. The intake of the snorkel must be placed very close to the source to be effective. There are newer designs that are mounted on articulating arms, which make the systems more convenient to use.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

Always use a proper dolly to carry gas cylinders in building. Avoid inhalation of vapor or mist. Ensure adequate ventilation. Remove all source of ignition; no smoking or electrostatic charge. Beware of vapor accumulating to form explosive concentration. Vapor can accumulate in low areas. Do use right-sized tools and wear heavy protective gloves when connecting a regulator to gas cylinders. Do not breathe any leaked gas. Work in confined spaces. Prevent further leakage or spillage if safe to do so.

All transport of hydrogen gas between on-campus locations must be conducted as follows:

- Gas cylinders must be secured to the transport vehicle (cart, motor vehicle, hand truck, etc.).
- Cylinders must be continuously attended during transport.
- Cylinders must be clearly labeled with content and hazard information.
- Cylinder caps must be in place.

These requirements apply to all the gas containers, including empty and partially full cylinders.

Upon receipt of hydrogen gas, cylinders shall be temporarily stored in a well-ventilated area that is attended or locked at all times. All cylinders shall be immediately leak tested with a leak indicating solution and must be clearly labeled with content and hazard information. Temporary storage locations shall have appropriate signage in place. Cylinders must be seismically secured at all locations with chains at two contact points on the cylinder body, using unistruts or an equivalent. Seismic securing should prevent cylinders from rolling, shifting, or falling.

Laboratory storage of all the gas cylinders shall be in a mechanically ventilated, lockable area. Examples of mechanical ventilation include vented gas cabinets and fume hoods. Rooms containing toxic gases shall be locked when not occupied by authorized persons. All cylinders and gas manifold must be clearly labeled with content and hazard information. Cylinders shall be seismically secured at all locations with chains (2 contact points), using unistruts or an equivalent for cylinders larger than lecture bottles. Lecture bottles must be secured to a stable surface. Outdoor storage is only allowed on a short-term basis in a secure area at least 75 feet from an exterior door, window, or air intake location.

All regulators, valves, and lines must be chemically compatible with the gases being used. Compatibility can be determined by contacting the gas vendor or by calling EH&S. Regulator/line systems must be leak tested immediately after assembly and before each use. Regulators shall be compatible with the size and type of gas cylinder being used, and rated for full cylinder pressure.

All lines or ducts carrying purged or exhausted emissions of hydrogen gas must be connected to a mechanical exhaust system that discharges to a safe location (i.e., presents no potential for re-entrainment into any building supply air intake or occupied area). Exhaust duct walls shall be chemically resistant to degradation by the toxic gas in use.

Significant emissions of hydrogen gas require an emission control device (e.g., scrubber, flare device, adsorbent) before the purged gas can be vented into the exhaust duct system. Significant emissions are defined as duct concentrations that result in duct corrosion or acute health risk to persons exposed near exhaust fan stacks as determined by release modeling. When hydrogen gas is emitted from exhaust systems at concentrations which could pose health risks to rooftop workers, locked gates, doors, or other means shall be used to prevent worker access to stack discharge areas. Warning signs must be conspicuously placed.

STORAGE:

It is essential that hydrogen gas is stored separately from all chemicals with which they may react. Ensure segregation of incompatible chemicals per guidance within the UCR Chemical Hygiene Plan. Also, follow any substance-specific storage guidance provided in Safety Data Sheet (SDS) documentation.

6. SPILL AND INCIDENT PROCEDURES

Emergency procedure for leaking gas cylinders - http://www.airproducts.com/~/media/Files/PDF/company/safetygram-11.pdf

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

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

7. WASTE DISPOSAL

All empty hydrogen gas cylinders shall be labeled as empty. Depleted hydrogen gas cylinders should be returnable to the vendor according to their guidelines. The purchase of any gases that will not be completely used in the course of research must be approved by the vendor for return, or by EH&S for disposal as hazardous waste. Disposal of hydrogen gas cylinders by EH&S, even when empty, may entail extraordinary costs. Therefore, hydrogen gas should be purchased only from vendors who will accept returns.

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

8. PRIOR APPROVAL/REVIEW REQUIRED

All work with hydrogen gas 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.

9. DESIGNATED AREA

A designated area shall be established where limited access, special procedures, knowledge, and work skills are required. A designated area can be the entire laboratory, a specific laboratory workbench, or a laboratory hood. Designated areas must be clearly marked with signs that identify the chemical hazard and include an appropriate warning; for example: WARNING! HYDROGEN GAS WORK AREA!

10. SAFETY DATA SHEETS

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

11. DETAILED PROTOCOL

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

Lab workers using hydrogen gas 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 hydrogen gas 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) use hydrogen gas under 1 bar in any given reaction (higher pressure REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this hydrogen gas 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 hydrogen gas. 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.

Replace empty gas cylinder

1) Borrow a proper dolly from department stockroom.
2) Close the main cylinder valve.
3) Slowly release pressure from regulator into hood to vent.
4) Close the regulator valves.
5) Disconnect the regulator from an empty cylinder.
6) Screw cylinder cap.
7) Deliver the empty cylinder to the stockroom or store temporally in one of hall cabinets.
8) Bring a new gas cylinder to the rack.
9) Safely secure the cylinder using chain clamp.
10) Unscrew cylinder cap.
11) Ensure the main valve is closed.
12) Unscrew the main valve cap.
13) Connect the regulator to the cylinder.
14) Fully open the regulator valves.
15) Get vacuum in the gas manifold and the regulator.
16) Closed the diaphragm valve.
17) Quickly open and close the main cylinder valve to see if the diaphragm valve is working well.

18) If the good sealing is obtained, go ahead. Otherwise, pump the gas in the line and replace the regulator.
19) Set a delivery pressure as needed.
20) Carefully release pressure from regulator.
21) Fully open the main cylinder valve if needed.

Replacing empty gas cylinder for GC Instrument

1. Close the main valve of empty gas tank.
2. Close the regulator valves.
3. Disconnect the regulator from an empty cylinder.
4. Deliver the empty cylinder to the stockroom and bring a new one to the rack.
5. Connect the regulator to the cylinder.
6. Fully open the regulator valves and the main cylinder valve and check the pressure.

Calcination of Catalyst in H_2 with Argon pretreatment

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Load catalyst sample (200 mg) in a clean dried reaction quartz tube.
3. Mount the tube in place.
4. Check all connections for possible leakage.
5. Open the Argon cylinder valve and regulator to adjust the flow rate to desired value.
6. Run Argon through catalyst at room temperature (25 ^oC) for 30 min.
7. Set the temperature controller to 150 ^oC.
8. Open the H_2 cylinder valve, switch from Argon to H_2.
9. Close Argon cylinder valve.
10. Adjust the gas flow rate if needed.
11. Set temperature to 350 ^oC and calcine the sample in H_2 flow for 2 h.
12. Close H_2 cylinder valve.
13. Close all valves to tube furnace.
14. Open vacuum valve, which is connected to the pump.
15. Check pressure gauge (should be around 0.03 torr).

Perform IR spectroscopic Experiment

1. A supported metal catalyst disk (e.g. Pt/SiO_2) is placed in an IR vacuum cell.
2. The catalyst is heated at 150 ^oC under vacuum for 30 min in order to eliminate the adsorbed water.
3. The catalysts are than heated form 150 ^oC to 350 ^oC under 5 torr of H_2.
4. The catalyst is kept at 350 ^oC under 200 torr of hydrogen gas for 3-4 hours.
5. Hydrogen gas is removed for 15 min.

6. The catalyst is kept at 350 ^{o}C under 200 torr of oxygen gas for 3-4 hours.
7. Oxygen gas is removed for 15 min.
8. Repeat 4 to 7 steps three times.
9. The sample is cooled down to room or any desired temperature.
10. Background spectrum is obtained.
11. Carbon monoxide is introduced into the cell up to 10 Torr.
12. Sample spectrum is obtained.
13. Carbon monoxide is pumped out.
14. The IR cell is vented to atmosphere.

UHV #1, Victor

1. Safely secure Hydrogen cylinder using a chain clamp or ring clamps.
2. Ensure the cylinder valve is completely closed.
3. Attach the appropriate pressure regulator to the cylinder and connect it to the gas manifold of the UHV system using copper tubing.
4. Carefully adjust the outlet pressure to about 15 psi using the regulator hand knob.
5. Close the valve between the gas manifold and the mechanical pump.
6. Open the regulator outlet valve and fill the copper tube with Hydrogen gas.
7. Open the valve of the mechanical pump to pump down the gas line.
8. Repeat the steps 5-6 three times to purge the copper line.
9. Carefully pressurize the copper line to deliver the gas.
10. Slowly open the leak valve to leak the gas into the UHV chamber and monitor the pressure in the UHV system.
11. After use, close the leak valve to the UHV system.
12. Close the valve on the regulator.
13. Close the main valve of Hydrogen cylinder.
14. Open the valve of the pump to evacuate the line.

UHV #2, RAIRS

1. Equip the proper PPEs (flame-resistant lab coat, safety glasses, chemical-resistant nitrile gloves.
2. Unscrew the main valve cap.
3. Carefully adjust the outlet pressure to 20 psi.
4. Close the valve next to the mechanical pump.
5. Fill the gas line with the Hydrogen gas.
6. Open the valve to the pump to evacuate the line.
7. Fill the gas lien with the Hydrogen gas.
8. After dosing with a leak valve or preparing a gas mixture, evacuate the gas line by opening the valve to the mechanical pump.

UHV #3, Michelle

1. Safely secure Hydrogen cylinder using a chain clamp or ring clamps. The Hydrogen cylinder can only be installed at a place that is at least 20 feet away from the Oxygen cylinder in the same room.
2. Ensure main valve is completely closed.
3. Attach the appropriate pressure regulator and connect to the system using a copper tube.
4. Carefully adjust the outlet pressure to 15 psi.
5. Close the angle valve next to the mechanical pump.
6. Fill the copper tube with Hydrogen gas. Then open the angle valve to pump down.
7. Repeat steps 5-6 three times to purge the copper line.
8. Carefully pressurize copper line.
9. Slowly open the leak valve to leak the gas into the UHV system, monitor the pressure in the UHV system
10. Close the leak valve.
11. Close the valve on the regulator. Close the main valve.
12. Open the angle valve to pump the line.

UHV #4 Praxis

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety glasses.
2. Check that the Hydrogen tank line is closed.
3. Open the large black swagelok valve, which connects the Hydrogen line and the gas manifold pump, to evacuate the Hydrogen line. Wait until the pressure gauge at the bottom of the electronics cabinet reaches 20 mTorr to indicate full gas evacuation.
4. Close the black swagelok valve, which connects the entire gas manifold to the gas manifold pump, in order to stop pumping the Hydrogen line.
5. Hydrogen is used in Temperature-Programmed Desorption experiments. During TPD, the sample should reach about 150 K. Open the Hydrogen tank valve to let gas flow to the chamber leak valve. Adjust the pressure of Hydrogen in the chamber by opening/closing the leak valve. For a good TPD, the pressure of gas introduced should not exceed 2E-8 Torr. A TPD pressure between 10E-9 Torr and 12E-9 Torr is the best range in general for gasses used in Praxis. The time that gas is allowed to flow into the chamber depends on the desired experiment time.
6. When Hydrogen use is finished, close the Hydrogen leak valve. Shut off the temperature controller.
7. Close the black swagelok valve on the gas manifold to stop the flow of gas from the Hydrogen tank into the leak valve. Open the black swagelok valve that connects to the gas manifold pump so that the leak valve can be pumped out. Close the Hydrogen tank valve.

UHV #5, UC Chamber

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Gently twist the safety switch allowing the gas molecules moving freely through the pipelines.
3. Turn on the ion gauge controller to ensure the stability of the pressure inside the chamber.
4. When the pressure in the preparation chamber is below 3E-7 torr, open the leak valve, and wait until the pressure goes down again.
5. Gently and gradually release the leak valve while keep monitoring the current pressure until the proper pressure is reached.
6. Once tasks are done, fully close the leak valve.
7. Reset the safety switch back to original lock position.

UHV #6 Nanoreactor

1. Safely secure Hydrogen cylinder using a chain clamp or ring clamps.
2. Ensure the cylinder valve is completely closed.
3. Attach the appropriate pressure regulator to the cylinder and connect it to the gas manifold of the UHV system using copper tubing.
4. Carefully adjust the outlet pressure to about 15 psi using the regulator hand knob.
5. Close the valve between the gas manifold and the mechanical pump.
6. Open the regulator outlet valve and fill the copper tube with Hydrogen gas.
7. Open the valve of the mechanical pump to pump down the gas line.
8. Repeat the steps 5-6 three times to purge the stainless steel line.
9. Carefully pressurize the copper line to deliver the gas.
10. Slowly open the leak valve to leak the gas into the UHV chamber and monitor the pressure in the UHV system.
11. After use, close the leak valve to the UHV system.
12. Close the valve on the regulator.
13. Close the main valve of Hydrogen cylinder.
14. Open the valve of the pump to evacuate the line.

Catalytic Hydrogenation Reaction

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Place the catalyst in the quartz reactor. About 5 mg of the catalyst is placed in the reactor between some layers of quartz wool above the metal disk placed in the reactor.
3. The reactor is placed inside the heating jacket and connected carefully with the

ultratorr connectors to the sampling line. The valves are open and the system is pump. The pressure should be below 60 mTorr. If not, check connections or disconnect the reactor and connect it again. Be careful not to break it.

4. Dry the catalyst, which is PtCu alloy supported by SBA-15 at 150 ^{o}C for 1 h.
5. Activate the catalyst via reduction cycles at a 200 ^{o}C for 2 h.
6. After the pretreatment of catalyst, wait until the temperature comes back to 100 Celsius.
7. Turn off the circulation pump and then evacuate the system.
8. Once the vacuum is reached, the gases can be introduced. The acrolein is introduced first (usually Ar is the last one to be introduced due to the high partial pressure use). The dosing line is isolated from the mechanical pump by closing the proper valve. The valve of the probe molecule is introduced until the desired pressure is reached in the Pirani gauge; the valve that connects the dosing line with the sampling loop is closed (check that the pressure in the sample loop is stable). Evacuate the dosing line (check the pressure with the TC gauge) and, then, be ready to repeat the same procedure followed with the probe molecule for hydrogen and Ar.
9. After all gases are introduced, the circulation pump is turned on.
10. After 2 min, the 6-way valve is used by moving the handle to the other side. Immediately after, the START button in the GC controller is pushed as well as the start button in the DMM software to start the collection of the data. After the peaks have appeared, leave the system run for additional 10 min in order to be sure that nothing else remains in the GC column and then push the STOP button in the GC controller and in the software. Save the collected data and wait until the system is ready for a new run (showed by the LEDs in the GC). If multiple runs are desired, the handle can be moved every 12 min without pressing any button in the software and the GC controller.

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.

Catalytic Hydrogenation of Furfural

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Catalyst (50–200 mg), furfural (0.5-1 g), and 1,4-dioxane (75 mL) are added into a reactor.
3. 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 H2 pressure (1.0 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 (0.4 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.

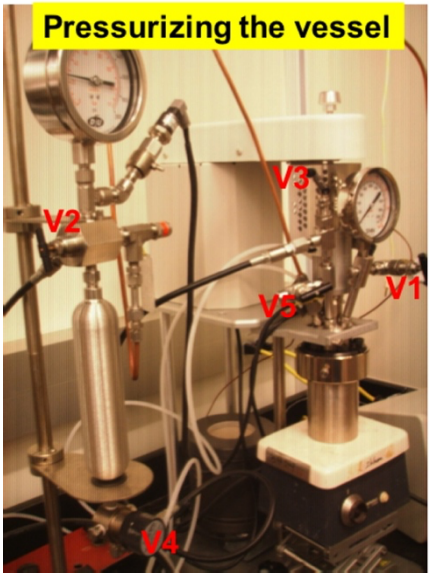
Oxidation-Reduction Pretreatment of Catalyst

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Take the catalyst Pt/Al_2O_3 (200 mg) into a glass sample tube.
3. Fix the tube on the Furnace reactor in room 135.
4. Open the valve to introduce Ar (25 mL/min) for 90 minutes at room temperature
5. Colse the Ar valve.
6. Open the O2 valve (30 mL/min) and treat the catalysts for 120 minutes at 350 °C.
7. Close the O2 valve.
8. Open the Ar valve again to remove the O2 left inside for 5 minutes.
9. Open the H2 valve (30 mL/min) to treat the catalysts for 120 min at 350 °C.
10. Close the H2 valve.
11. The sample is then cooled down under Ar atomosphere (25 mL/min) and transferred to a glass bottle for use.

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. Make sure that the V2, V3, V5 and V1 are closed.

7. Open the valve V2, introduce the H_2 into the high-pressure burette with the pressure is around 40 bar.
8. Close the valve V2.
9. Open the valve V3, introduce the H_2 into the cylinder with the pressure is around 20 bar.
10. Close the valve V3.
11. Open the valve V1 to release the H_2 inside the cylinder.
12. Close the valve V1.
13. Repeat step 6 to 12 four more times.
14. Pressurize the reactor to 10 bar of H2 with stirring.
15. Release the H2 after 10 min of reaction
16. Take a sample for GC analysis.



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 mg) 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. Make sure that the V2, V3, V5 and V1 are closed.
6. Open the valve V2, introduce the H_2 into the high-pressure burette with the pressure is around 40 bar.
7. Close the valve V2.
8. Open the valve V3, introduce the H_2 into the cylinder with the pressure is around 20 bar.
9. Close the valve V3.
10. Open the valve V1 to release the H_2 inside the cylinder.
11. Close the valve V1.
12. Repeat step 5 to 11 four more times.
13. Pressurize the reactor to 10 bar and start stirring.
14. Release the H2 after 10 min of reaction and take a sample for GC analysis.
15. Repeat this step every 10 minutes until 40 min of reaction

GC #1 Agilent-Batch Reactor: Hydrogen Pre-treatment cycle

1. Wear nitrile chemical–resistant gloves, a flame-resistant lab coat, and a safety goggle.
2. Introduce H2 (400-500 Torr) at room temperature by opening the hydrogen valve.

3. Close the hydrogen valve.
4. Turn on the recirculation pump to cycle the hydrogen gas through the reactor.
5. Increase the temperature of U-shaped cell slowly to 350 ^{o}C.
6. Once the cell temperature reaches to 350 oC, continue the hydrogen cycling for 1 h.
7. Cool down the cell to room temperature with a cooling fan.
8. Evacuate the reactor loop by opening the valves to the rotary pump.
9. Wait until the pressure comes back to the base pressure for 30 min.
10. Repeat this cycle as desired.

GC #1 Agilent-Batch Reactor: Loading a Catalyst into the U-shaped Cell

- Note: Introduce the gas with lowest partial pressure first.

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety glasses.
2. Isolate the gas manifold from the mechanical pump by closing the valve connecting the manifold to the mechanical pump
3. Slowly open the valve for the 1-butene (or propylene) and close the valve after a few seconds
4. Slowly open the valve connecting the gas manifold to the rector loop while constantly checking the baratron pressure gauge. Make sure to add 5 torr of 1-butene (or propylene) to the reactor loop.
5. After adding the desired amount of gas to the loop, close the valve that connects the manifold to the reactor loop and check that the pressure of the reactor loop remains stable
6. Evacuate the gas manifold by opening the valve to the pump for 30 minutes
7. Slowly open the valve for the H_2 and close the valve after a few seconds
8. Slowly open the valve connecting the gas manifold to the rector loop while constantly checking the baratron pressure gauge. Make sure to add 50 torr of H_2 to the reactor loop.
9. After adding the desired amount of gas to the loop, close the valve that connects the manifold to the reactor loop and check that the pressure of the reactor loop remains stable
10. Evacuate the gas manifold by opening the valve to the pump for 30 minutes
11. Slowly open the valve for Argon and close the valve after a few seconds
12. Slowly open the valve connecting the gas manifold to the reactor loop while constantly checking the baratron pressure gauge. Make sure to add 545 torr of Argon to the reactor loop.
13. After adding the desired amount of gas to the loop, close the valve that connects the manifold to the reactor loop and check that the pressure of the reactor loop remains stable

14. Evacuate the gas manifold by opening the valve to the pump for 30 minutes, then close the valve.
15. After introducing all the reactant gasses circulate the reactant gas mixture for 20 minutes by turning on the circulation pump.
16. Close the valve at the front of the bypass line, and leave open the valve in the back.
17. Open the valves to the reactor tube, now the reactant mixture will contact the Pt/Al_2O_3 catalyst.

Synthesis of Au NPs on APTES-grafted P25 titania

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Dry 2 flasks (250 mL) under nitrogen.
3. Put deionized water (100 mL) into the flask.
4. Add 3-aminopropyltriethoxysilane (APTES)-grafted P25 titania nanoparticles (1 g) into the flask
5. After the supports have dispersed evenly, add a solution of tetrachloroauric acid (15 mL, 10 mM).
6. Maintain suspension by stirring the mixture for 2 h at room temperature. If left unattended in a fume hood, put a label with the chemical name and hazard information.
7. Filter the solid from the mixture, and wash twice with deionized water
8. Put deionized water (100 mL) into the other flask.
9. Redisperse the solid into the flask.
10. For the reduction reaction, add sodium borohydride (2.5 g) into the flask.
11. Filter and wash the solid with deionized water.
12. Dispose waste in the properly labeled container.
13. Collect the sample and dry it in an inert atmosphere (i.e. nitrogen, vacuum) at 60°C overnight. If left unattended, put a label with the chemical name and hazard information.
14. Calcinate the sample for 5 h at 450°C in oxygen.
15. Purge with Ar gas for 10 min.
16. Purge with hydrogen at 350 °C for 1 h.
17. After cooling, collect the sample.

SOP Reviewed and Approved by:

Francisco Zaera	
Print name	Signature

Approval Date: 02/01/2013, updated 01/01/2015, 03/01/2016, 11/01/2016, 10/10/2017, 08/07/2018, 08/14/2018, 01/25/2019, 09/10/2019, 04/09/2022, 04/18/2022

Hydrogen Fluoride
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 hydrogen fluoride (HF, CAS No. 7664-39-3) is used in laboratory. Its purpose is not to have any accident or risk. Hydrogen fluoride is highly toxic. It is fatal if inhaled, if swallowed or in contact with skin. Also, it causes severe skin burns and eye damage.

Synonyms: Hydrofluoric acid

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 (Liver, Kidney), Highly Toxic by Inhalation and Ingestion, Skin Absorption, Corrosive

GHS Classification

Acute toxicity, Oral (Category 2)
Acute toxicity, Inhalation (Category 2)
Acute toxicity, Dermal (Category 1)
Skin corrosion (Category 1B)
Serious eye damage (Category 1)

Sign and Symptoms of Exposure

Fluoride ion can reduce serum calcium levels possibly causing fatal hypocalcaemia. Material can cause severe burns and blistering which may not be immediately painful or visible. The full extent of tissue damage may not exhibit itself for 12-24 hours after exposure., Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin., necrosis of the 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 hydrogen fluoride 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 hydrogen fluoride.

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 hydrogen fluoride must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of hydrogen fluoride and understand the hazards.

Lab workers using hydrogen fluoride 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 hydrogen fluoride 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 hydrogen fluoride in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this hydrogen fluoride 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 hydrogen fluoride. 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.

Si wafer washing

1. Wear a latex glove first, then Neoprene glove outside, water- resistant lab coat and safety goggle.
2. Take the bottle of 48% HF solution bottle and water bottle to the fume hood in room 135.
3. Mix 48%HF and DI water in the volume ratio of 1:20.
4. Use this diluted solution to treat Si wafer for 1 min.
5. Take the wafer out and wash the wafer with DI water.
6. All the liquid waste needs to be treated as hazardous.

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 ^{o}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 ^{o}C.
10. Dry silicon wafers in a N_2 flow.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 03/01/2014, 07/16/2019

Hydrogen peroxide
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 hydrogen peroxide (H_2O_2, CAS No. 7722-84-1) used in laboratory. Its purpose is not to have any accident or risk. Hydrogen peroxide is oxidizer, so it may intensify fire. It is harmful if swallowed and very 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: Oxidizer, Target Organ Effect (Eyes, Skin, Respiratory), Toxic by Ingestion, Corrosive, Carcinogen

GHS Classification

Oxidizing liquids (Category 1)
Acute toxicity, Oral (Category 4)
Acute toxicity, Inhalation (Category 5)
Skin corrosion (Category 1A)
Serious eye damage (Category 1)
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.

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- 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 hydrogen peroxide 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 hydrogen peroxide.

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 hydrogen peroxide must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of hydrogen peroxide and understand the hazards.

Lab workers using hydrogen peroxide 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 hydrogen peroxide 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 hydrogen peroxide in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this hydrogen peroxide 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 hydrogen peroxide. 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 safety goggles.
2. Into the beaker with stir bar, SH-(NH-Boc-SBA-15), 0.3 g, was added in hydrogen peroxide (30 wt.%, 10 mL).
3. Stir the mxiture at room temperature in air for 24 h.
4. Filter the mixture and wash it with water for several times.

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 ^{o}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 ^{o}C.
10. Dry silicon wafers in a N_2 flow.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 07/01/2018, updated 07/16/2019