Isopropyl alcohol
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 isopropyl alcohol (C_3H_8O, CAS No. 67-63-0) used in laboratory. Its purpose is not to have any accident or risk. Isopropyl alcohol is highly flammable liquid and vapor. It causes serious eye and mild skin irritation, as well as drowsiness or dizziness

Synonyms: 2-Propanol, sec-Propyl alcohol, Isopropanol

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

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

OSHA Hazards: Flammable liquid, Irritant, Target organ effect (Nerves, Kidney, Liver, Cardiovascular system, Gastrointestinal tract)

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. Prolonged or repeated exposure can cause Nausea, Headache, Vomiting, Narcosis, Drowsiness. Overexposure may cause mild, reversible liver effects.

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 isopropyl alcohol 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 isopropyl alcohol.

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

Lab workers using isopropyl alcohol 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 isopropyl alcohol 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 isopropyl alcohol in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this isopropyl alcohol 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 isopropyl alcohol. 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. Isopropyl alcohol is used to wash and clean the hardware that will be used for UHV system.
3. Always put isopropyl alcohol in a wash bottle.
4. Only use the necessary amount.
5. Collect all the used isopropyl alcohol in a beaker and dispose into a proper waste bottle.

Functionalization of Silica nanospheres

1. Wear nitrile chemical resistant gloves, 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 ^{o}C for 2 h, then wash the NH_2 functionalized particles in ethanol.
6. Dispose off the washings as hazardous organic waste.

Oxidation of isopropyl alcohol

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Make a waste bottle labeled as toxic and carcinogen hazardous waste. Review the SDS of isopropyl alcohol and benzene again; especially remind first aid measures, handling and storage, & PPE.*

3. Place a test tube into a fume hood and put a stir bar into it. Close with a rubber septum and take it to a balance. Weigh P25-TiO_2/Au catalyst (9 mg) and add it into the test tube. Add potassium carbonate (25 mg) and transfer the closed septum back to the fume hood.
4. With a micropipette add toluene (4.5 mL), close with the septum and sonicate for about 1 minute so that the solids disperse well in the solvent.
5. Bring the mixture back to the fume hood and stir it.
6. Before adding the internal standard benzene (12.5 µL) *put on a full-face respirator*. Take a bottle of benzene from the flammable cabinet and place it into the fume hood. *Be careful not to spill benzene. Keep watching any leak of benzene. Avoid release to the environment. Avoid breathing fume, gas, mist, vapor or spray. If swallowed, immediately call 911. If inhaled, rinse cautiously with water for 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.*
7. Remove the septum from the test tube and open the benzene bottle. Add the small amount of benzene into the test tube by using a Hamilton syringe (50 µL). Wash the syringe with benzene three times before adding it into the reaction mixture. After adding it clean the syringe by washing it with ether. Dispose the waste into the waste bottle labeled carcinogen hazardous waste. Once adding benzene the handling of the reaction mixture has to be carried out with the full-face respirator on.
8. Put the benzene bottle back to the flammable cabinet. *Be careful not to spill benzene. Keep watching any leak of benzene. Avoid release to the environment. Avoid breathing fume, gas, mist, vapor or spray. If swallowed, immediately call 911. If inhaled, rinse cautiously with water for 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.*
9. Take the small vial of isopropanol from the flammable cabinet and put it into the fume hood. Add the reactant (5.5 µL) into the reaction mixture.
10. Close the test tube with the rubber septum, seal with Teflon tape and connect the oxygen supply to the test tube.
11. Open the main valve of oxygen cylinder, which is located in a cupboard in a corridor in front of the room 135. After then, open the oxygen Swagelok needle valve in the fume hood, and fill the balloon with oxygen. Attach the balloon to a needle going through the rubber septum into the reaction mixture.
12. Transfer the test tube into the oil bath and do the catalytic reaction at temperature below 75 °C (boiling point of benzene is 80.1 °C).
13. Collect samples at different reaction times and remember to always put on the full-face respirator before working with the mixture. Put a sample (100 µL) into a small centrifuge tube and centrifuge it to remove the solids.
14. After centrifuging bring the closed vial back to the fume hood and transfer the liquid into a new vial.
15. Inject the sample into GC using a Hamilton syringe (10 µL).

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 isopropyl alcohol (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 isopropyl alcohol (70 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 (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.
11. Calculate the concentration of analyte by using the calibration curves or response factors.

Hydrogenation of Cinnamaldehyde

1. Wear a flammable-resistant lab coat, safety goggles and nitrile chemical-resistant gloves.
2. The catalyst is pre-treated under H_2 at 350 ^{o}C for 3 h before use.
3. The catalyst (20 mg) is mixed with trans-Cinnamaldehyde (0.8 g), benzyl alcohol (2 mL) and 2-propanol (75 mL).

4. The reaction begins at 100 °C under H_2 (3 MPa) with stirring.
5. The reaction is stopped after an appropriated time.

Copper substrate: Chemical cleaning

1. Wear nitrile chemical resistance gloves, flame resistance lab coat, and safety goggles.
2. Place the polished polycrystalline copper substrate (2 mm thick, 10 mm diameter) in #1 beaker (100 mL) and add acetone (50 mL).
3. Sonicate the beaker for 5 min.
4. Transfer the copper substrate into #2 beaker and add isopropyl alcohol (50 mL).
5. Sonicate #2 beaker 2 for 5 min.
6. Repeat the above step (steps 3-4) with DI water (50 mL) followed by HNO_3 solution (1%, 50 mL).
7. Rinse the copper substrate with isopropyl alcohol for 2 min.
8. Sonicate the copper substrate in a mixture of DI water (60 mL) and glacial acetic acid (2 mL) for 5 min. Then, blow-dry with a flow of N2.
9. Store the samples at low vacuum (10^{-2} torr) or at Ar/ N2 atmosphere immediately to avoid oxidation (until characterize with XPS or mount in the RAIRS chamber).
10. Dispose of the washing and cleaning solvents properly as chemical waste.

Synthesis of aminopropyltriethoxysilane(APTES)-grafted P25 titania

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

Silylation Procedure for SBA-15

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 03/01/2014, 03/01/2016, 05/15,2016, 10/10/2017, 08/14/2018, 10/22/2018, 12/11/2019, 04/18/2022, 08/24/2022

L-Ascorbic 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 L-ascorbic acid ($C_6H_8O_6$, CAS No. 50-81-7) is used in laboratory. Its purpose is not to have any accident or risk. L-Ascorbic acid may cause eye, skin, or respiratory tract irritation. It may be harmful if inhaled, if absorbed through skin, or if swallowed.

Synonyms: Antiscorbutic factor, L-Threoascorbic acid, Vitamin C

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

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

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

Signs and Symptoms of Exposure

Chronic ingestion of large doses may cause gastrointestinal disturbances including nausea and diarrhea, urinary effects involving urine acidification, oxalate and uric crystallization in the bladder and kidney, and decreased reaction times and psychomotor coordination.

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

Lab workers using L-ascorbic 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 L-ascorbic acid described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

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

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

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.

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 06/01/2013, updated 01/21/2022

Methanol
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 methanol (CH_4O, CAS No. 67-56-1) used in laboratory. Its purpose is not to have any accident or risk. Methanol is highly flammable liquid and vapor, and toxic if swallowed, if inhaled, or in contact with skin. It causes serious eye and skin irritation.

Synonyms: Methyl alcohol

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 (Eyes, Kidney, Liver, Heart, Central nervous system), Toxic by Inhalation, Ingestion and Skin absorption, Irritant.

GHS Classification

Flammable liquids (Category 2)
Acute toxicity, Oral (Category 3)
Acute toxicity, Inhalation (Category 3)
Acute toxicity, Dermal (Category 3)
Skin irritation (Category 2)

Signs and Symptoms of Exposure

Methyl alcohol may be fatal or cause blindness if swallowed. It Cannot be made non-poisonous. Effects due to ingestion may include Nausea, Headache, Vomiting, Gastrointestinal disturbance, Dizziness, Weakness, Confusion, Drowsiness, Unconsciousness. May cause convulsions.

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 methanol 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 methanol.

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

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

Photoreaction reagent

1. Wear nitrile chemical-resistant gloves, mask, flame-resistant lab coat, and safety goggles.
2. Take 5 mL of methanol by a syringe in a fume hood.
3. Bring the syringe to the photoreactor and inject the methanol through the outlet of the photoreactor.

Synthesis of N-Benzyl-2-(benzylamino)acetamide

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Put methanol (40 mL) and benzylamine (10.9 mL, 100 *m*mol) in a flask (50 mL).
3. Add methyl bromoacetate (1.4 mL, 15 *m*mol) into the flask.
4. Stir the mixture for a week at room temperature in a fume hood. Leave a label with chemical name and hazard information.
5. Remove methanol under evaporator.
6. Distill the crude to remove the excess of benzylamine.
7. Purify the residue by column.

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 methanol (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 methanol (70 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 (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.
11. Calculate the concentration of analyte by using the calibration curves or response factors.

Washing Solvent

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

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 ^{o}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.

Oxidation of furfural

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. The samples are used without any pretreatment
3. Au/SBA-15 sample (20 mg) is mixed with methanol (50 mL), furfural (1 mL) and decane (1 mL) in the Parr reactor. Methanol is measured out by a graduated cylinder, while furfural and decane are measured out by a pipette.
4. The Parr reactor is purged by O_2 for 10 min.
5. The reaction begins at 100 ^{o}C and 1 atm O_2 pressure with stirring.
6. The reaction is stopped after appropriate time.
7. Liquid samples are filtered and stored in vials.
8. The products are analyzed by GC.

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.

SBA-15 Silylation 1

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

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

SBA-15 Silylation 2

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 03/01/2014, 03/01/2016, 10/10/2017, 08/14/2018, 11/20/2019, 02/06/2020, 07/08/2020, 07/10/2020

Methyl bromoacetate
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 methyl bromoacetate ($C_3H_5BrO_2$, CAS No. 96-32-2) used in laboratory. Its purpose is not to have any accident or risk. Methyl bromoacetate is highly flammable liquid and vapor, and toxic if swallowed, if inhaled, or in contact with skin. It causes serious eye damage and skin burns

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

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

OSHA Hazards: Flammable liquid, Toxic by Ingestion, Eye damage, Skin burns
GHS Classification

Flammable liquids (Category 4)
Acute toxicity, Oral (Category 3)
Skin corrosion (Category 1B)
Serious eye damage (Category 1)

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

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 methyl bromoacetate 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 methyl bromoacetate.

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

Lab workers using methyl bromoacetate 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 methyl bromoacetate described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);

4) employ < 5 g of this methyl bromoacetate in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this methyl bromoacetate 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 methyl bromoacetate. 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 N-Benzyl-2-(benzylamino)acetamide

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Put methanol (40 mL) and benzylamine (10.9 mL, 100 *m*mol) in a 50 mL flask.
3. Add methyl bromoacetate (1.4 mL, 15 *m*mol) into the flask.
4. Stir the mixture for a week at room temperature in a fume hood. Leave a label with chemical name and hazard information.
5. Remove methanol under evaporator.
6. Distill the crude to remove the excess of benzylamine.
7. Purify the residue by column.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2015

Methylene Chloride
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 methylene chloride (CH_2Cl_2, CAS No. 75-09-2) is used in laboratory. Its purpose is not to have any accident or risk. Methylene chloride is a CAL/OHSA Regulated Carcinogen, so may cause cancer and genetic damage. It also causes serious eye and skin irritation. It may be harmful if swallowed. Methylene chloride or dichloromethane (DCM) is commonly used as a reaction solvent, a solvent for extractions in isolating organic compounds, and as an eluent for flash and thin-layer chromatography. Methylene chloride is one of California's Regulated Carcinogens. This SOP documents the safe use of DCM including the minimization of inhalation of dichloromethane. Use of methylene chloride in the laboratory would result in "short term exposure," which the State limits to 125 ppm for 15 minutes.

Synonyms: Dichloromethane

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

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

OSHA regulated Carcinogen

Carcinogens, or agents that cause cancer, are classified by several organizations. The International Agency for Research on Cancer is the most respected voice internationally on the classification of cancer causing substances. The list of substances and definitions can be found online. In the United States, the National Toxicology Program is the organization considered most trusted source in this country on cancer. The most current NTP report on carcinogens can be found online at the National Toxicology Program website. In California, the Department of Industrial Relations, Division of Occupational Safety and Health (Cal/OSHA) regulates occupational use of and exposure to a select set of carcinogens. The substances currently regulated as occupational carcinogens in California can be found at: www.dir.ca.gov/Title8/sb7g16a110.html

OSHA Hazards: Carcinogen
GHS Classification
Carcinogenicity (Category 2)

Signs and Symptoms of Exposure

Methylene chloride is metabolized in the body producing carbon monoxide which increases and sustains carboxyhemoglobin levels in the blood, reducing the oxygen-carrying capacity of the blood., Acts as a simple asphyxiant by displacing air.,

anesthetic effects, Difficulty in breathing, Headache, Dizziness, 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, Paresthesia., Drowsiness, Convulsions, Conjunctivitis., Pulmonary edema. Effects may be delayed., Irregular breathing., Stomach/intestinal disorders, Nausea, Vomiting, Increased liver enzymes., Weakness, Heavy or prolonged skin exposure may result in the absorption of harmful amounts of material., Abdominal pain.

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 methylene chloride 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 methylene chloride.

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

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

Developing Solvent for Column Process

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and *full-face respirator*.

2. *Make a waste bottle labeled as carcinogen hazardous waste. Review the SDS of methylene chloride again; especially remind first-aid measures, handling and storage, PPE, & signs and symptoms of exposure.*
3. Bring the bottles of methylene chloride and acetone into a fume hood.
4. Methylene chloride (95 mL) and acetone (5 mL) are mixed in a screw-capped bottle. *Keep the methylene chloride container tightly closed in a cool, dry and well-ventilated place. The container must be carefully resealed and kept upright to prevent leakage*.
5. The developing solvent (20 mL) is added to silica gel (10 g). *Be careful not to spill methylene chloride. Keep watching any leak. Avoid release to the environment. Avoid inhalation of vapor or mist. If swallowed, never give anything by mouth to an unconscious person and rinse mouth with water. Consult a physician and call 911. If inhaled, move the person into fresh air or give artificial respiration. In case of skin contact, wash off with soap and plenty of water. In case of eye contact, rinse thoroughly with plenty of water for at least 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.*
6. The silica gel is packed into chromatography column in a fume hood.
7. A sample is loaded on the top of the silica gel.
8. The developing solvent (80 mL) is added to the column. *Be careful not to spill methylene chloride. Keep watching any leak.*
9. Dropping solvent is collected in vials. *Be careful not to spill. Keep watching any leak. Avoid release to the environment. Avoid inhalation of vapor or mist.*
10. Check each vial with TLC (thin-layer chromatography).
11. Collect the product in vials. *Be careful not to spill. Keep watching any leak. Avoid release to the environment. Avoid inhalation of vapor or mist.*
12. Remove the solvent under evaporator. *Be careful not to spill. Keep watching any leak. Avoid release to the environment. Avoid inhalation of vapor or mist.*
13. Handle the rest of vials as carcinogen waste. *Transfer them to the carcinogen waste container in a fume hood. Be careful not to spill. Keep watching any leak.*

Catalytic Reaction

1. Wear nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggles.
2. Into a round-bottom flask, add a catalyst (50 mg), methyl vinyl ketone (1.5 *m*mol), benzaldehyde (1.5 *m*mol), and methylene chloride (4 mL).
3. Stir the mixture at room temperature under N2 atmosphere in a fume hood.
4. Every 3 hr, a sample solution (0.1 mL) is filtered and analyzed for GC or TLC.
5. After reaction is finished, wash and clean the flask. The solution needs to be treated as hazardous waste.

Reaction

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Methyl vinyl ketone (1.5 mmol), benzaldehyde (1.5 mmol) and 1,4-diazabicyclo [2.2.2]octane (1.5 mmol) are added to methylene chloride (4 mL).
3. Bring the mixture into the fume hood.
4. Stir it at room temperature under nitrogen atmosphere.
5. For each 3 h, a solution (0.1 mL) is filtered and analyzed with GC or TLC.
6. After the reaction is finished, washing and cleaning the flask and solvents also need to be treated as hazardous waste.

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

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

1. Quinine (5 g, 15.4 *m*mol) and Triphenylphosphine (4.85 g, 18.5 *m*mol) are put into a round flask (250 mL) dried under vacuum.
2. THF (60 mL) is added to the flask and stirred for 5 min.
3. Cool down the mixture to 0 °C 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.

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

1. The salt (3 g) is put into a flask (100 mL).

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2015, updated 01/02/2019, 07/01/2019, 02/07/2020

Molecular sieves
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 molecular sieves (N/A, CAS No. 70955-01-0) is used in laboratory. Its purpose is not to have any accident or risk. Molecular sieves cause serious eye, skin and 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: Irritant
GHS Classification
Skin irritation (Category 2)
Eye irritation (Category 2A)
Specific target organ toxicity – single exposure (Category 3)

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with molecular sieves 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 molecular sieves.

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

Lab workers using molecular sieves 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 molecular sieves 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 < 500 g of this molecular sieves in any given reaction (larger quantities REQUIRE the approval of PI or designee), and

5) discuss ALL issues or concerns regarding molecular sieves 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 molecular sieves. 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.

Dehydrate with molecular sieves

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Weight 100 g of molecular sieves.
3. Bring the solid to the fume hood, and add it to the bottle with solvent need to dehydrate
4. The molecular sieves can be re-activated via calcine in oven.

UHV #3, Michelle: Stop oil vapors backstreaming

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Take a required amount of molecular sieves.
3. Dry molecular sieves in oven.
4. Fill the oil trap by molecular sieves.
5. Dispose the used molecular sieves through the environmental health and safety department

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SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 03/01/2014, 03/06/2019

N,N-Dimethylformamide
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 N,N-dimethylformamide ($C_{10}H_{21}NO_4Si$, CAS No. 68-12-2) is used in laboratory. Its purpose is not to have any accident or risk. N,N-Dimethylformamide is combustible liquid and toxic if inhaled, and has Teratogen hazard. It is harmful in contact with skin. It causes serious eye and mild skin irritation.

Synonyms: DMF

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, Target Organ Effect (Liver, Kidney, Central Nervous system, Cardiovascular system, Blood), Harmful by Skin Absorption, Irritant, Teratogen

GHS Classification

Flammable liquids (Category 3)
Acute toxicity, Oral (Category 5)
Acute toxicity, Inhalation (Category 3)
Acute toxicity, Dermal (Category 4)
Skin irritation (Category 3)
Eye irritation (Category 2A)
Reproductive toxicity (Category 1B)

Signs and Symptoms of Exposure

Warning: Intolerance for alcohol can occur up to 4 days after dimethylformamide exposure. N,N-dimethylformamide is considered to be a potent liver toxin., Vomiting, Diarrhoea, Abdominal pain.

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 N,N-dimethylformamide 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 N,N-dimethyl formamide.

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

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

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Take 0.03 g of N,N-dimethylformamide in a two-neck round bottom flask, in the fume hood.
3. Add other reagents into above flask, remove the air in the flask, fill with N_2, and conduct the reaction with stirring.
4. After reaction, the filtrate needs to be treated as hazardous waste.
5. Washing and cleaning solvents also need to be treated as hazardous waste.

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).

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

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

N,N-Dimethyltrimethylsilylamine
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 N,N-dimethyltrimethylsilylamine ($C_5H_{15}NSi$, CAS No. 2083-91-2) used in laboratory. Its purpose is not to have any accident or risk. N,N-Dimethyltrimethylsilylamine is highly flammable corrosive liquid and vapor. It causes severe skin burns and eye damage.

Synonyms: TMSDMA, (Dimethylamino)trimethylsilane, N-(Trimethylsilyl)dimethylamine

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, Corrosive
GHS Classification
- Flammable Liquids (Category 2)
- Skin corrosion (Category 1B)
- Serious eye damage (Category 1)

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with N,N-dimethyltrimethylsilylamine 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 N,N-dimethyl-trimethylsilylamine.

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

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

N,N-Dimethyltrimethylsilylamine Sample Preparation

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Leak valve and 1’33 flange with sealed-off Pyrex glass end tube should be dried in the oven for 1 hour, cooled down to room temperature.
3. Take the leak valve, the flange, copper gasket (1.33”), and self-made spatula to room 135 and follow the procedures for using the glovebox.
4. Transfer *N,N*- dimethyltrimethylsilylamine (1 mL) to the glass end tube, and close the leak valve inside the glovebox.
5. Care should be taken when mounting the leak valve to the chamber.
6. When the pressure in the preparation chamber is below $3x10^{-7}$ torr, open the leak valve to release residual gas and wait until the pressure drops again.
7. Heat the glass tube to around 303 K and keep the chamber, especially the parts on the delivery pass, at around 313 K.
8. Make sure the leak valve is working properly and continue with experiments.
9. After each experiment, heating the sample and chamber should be stopped to protect the o-rings around the transfer rod.
10. After each experiment, the silicon sample needs to be treated as a solid hazardous waste.
11. The glass tube needs to be cleaned with acetone and DI water, and the waste solution should be placed into the proper waste container.

NiO deposition on TMSDMA/SBA-15 via ALD reactor

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, **AND** a proper face mask at all times (Covid-19) while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
2. Clean the sample holder with acetone
3. Place SBA-15 in the sample holder.
4. Preheat the support (SBA-15) at 200 ℃ for 2 h.
5. Ni precursor (nickel(II) bis(2,2,6,6-tetramethyl-3,5-heptanedionate) is pretreated at 165 ℃ in an oil bath.
6. The reactor is set to 150 ℃.
7. Dose N,N-dimethyltrimethylsilylamine (TMSDMA, 50 mTorr) 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.

Liquid Phase Silylation

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.
2. Have N,N-dimethyltrimethylsilylamine (TMSDMA, 10 mL) in a flask.
3. Immerse SiO2 wafers into the TMSDMA, such that the wafers are fully submerged, under a dry N2 environment.
4. Reflux the samples at 80 oC for 24 h under dry N2 flow.
5. Allow the samples to cool down.
6. Dry each wafer under dry N2 flow.

TiO_2/TMSDMA/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 °C for 2 h.
4. Ti precursor (tetrakis(dimethylamido) titanium, TDMAT) 42 °C, using heating tape and r
5. The reactor is set to 102 °C.
6. Dose trimethylsilyl dimethylamine (TMSDMA) 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.

N,N-(Trimethylsilyl)dimethylamine (TMSDMA) for Michelle UHV Chamber

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggle.
2. Add N,N-(trimethylsilyl)dimethylamine (1 mL) into the metal-glass adaptor container in the glove box of room 135.
3. Connect the container to the leak valve of the Michelle chamber.
4. Wear apron, full-face shield, and waterproof cryo-gloves.
5. Fill liquid nitrogen (LN_2) in a LN_2 dewar from a LN_2 tank in hallway.
6. Slowly pour liquid nitrogen into a LN_2 glass dewar flask.
7. Put the container into the LN_2 glass dewar flask.
8. Purify N-(trimethylsilyl)dimethylamine by repeated freeze-pump-thaw cycles.
9. Heat the container in a silicon-oil bath up to 45 ^oC.
10. 10.Open the container valve.
11. 11.Slowly open the leak valve to leak the N,N-(trimethylsilyl)dimethylamine to reach pressure at 5 x 10^{-7} torr and monitor UHV system.
12. 12.Close leak valve.
13. 13.Close the container valve.
14. 14.Stop heating the silicon-oil bath.
15. 15.Collect waste of N,N-(trimethylsilyl)dimethylamine and dispose into a proper waste bottle.

Silylation Procedure for SBA-15

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. A flask is dried.
3. Put SBA-15 (200 mg) in the flask.
4. Fill the flask with toluene or ethanol (30 mL).
5. Slowly add N,N-dimethyltrimethylsilylamine (2 mL) into the flask in the fume hood.
6. The mixture is refluxed at 90 ^oC 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: 02/15/2021, 10/12/2021, 12/01/2021, 12/21/2021, 05/06/2022, 08/24/2022

Nickel (II) nitrate hexahydrate
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 nickel(II) nitrate hexahydrate ($N_2NiO_6 \cdot 6H_2O$, CAS No. 13478-00-7) used in laboratory. Its purpose is not to have any accident or risk. Nickel(II) nitrate hexahydrate is oxidizer, so it may intensify fire. It is harmful if swallowed and very toxic to aquatic life. It causes serious eye damage and skin 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: Oxidizer, Carcinogen, Target Organ Effect (Lungs), Toxic by Inhalation, Harmful by Ingestion, Skin and Respiratory Sensitizer, Irritant, Teratogen

GHS Classification

Oxidizing solids (Category 3)
Acute toxicity, Inhalation (Category 4)
Acute toxicity, Oral (Category 4)
Skin irritation (Category 2)
Serious eye damage (Category 1)
Respiratory sensitization (Category 1)
Skin sensitization (Category 1)
Reproductive toxicity (Category 1B)
Specific target organ toxicity – repeated exposure, inhalation (Category 1)
Acute aquatic toxicity (Category 1)

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with nickel(II) nitrate hexahydrate 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 nickel(II) nitrate hexahydrate.

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 nickel(II) nitrate hexahydrate must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of nickel(II) nitrate hexahydrate and understand the hazards.

Lab workers using nickel(II) nitrate hexahydrate 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 nickel(II) nitrate hexahydrate 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 < 20 g of this nickel(II) nitrate hexahydrate in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this nickel(II) nitrate hexahydrate 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 nickel(II) nitrate hexahydrate. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Preparation #1 of Ni/SBA-15

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

Preparation #2 of Ni/SBA-15

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Add nickel nitrate hexahydrate (0.01 g, $Ni(NO_3)_2 \cdot 6H_2O$), nitric acid (1.25 mL, HNO_3) and SBA-15 (0.2 g) into water (25 mL).
3. The mixture is sonicated for a while.
4. The mixture is heated to 55 ^oC in a flask.
5. Add urea (1.2012 g) into water (25 mL).
6. Add the urea solution to the first solution (step 2).
7. The mixture is heated to 90 ^oC for 4 h.
8. Cool down to 55 ^oC.
9. The mixture is stirred at 55 ^oC for 10 min.

10. Cool down the mixture to room temperature.
11. Filter the mixture.
12. Wash the powder for 3 times with water.
13. Dry the powder overnight.
14. The powder is calcined at 550 ^{o}C under air for 3 h.

SBA-15 Impregnation 1

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

SBA-15 Impregnation 2

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle
2. A stock solution (25 mL) of $Ni(NO_3)_2{\cdot}6H_2O$ (0.1 M) will be prepared by adding $Ni(NO_3)_2{\cdot}6H_2O$ (0.727 g) into a volumetric flask and filling with water.
3. The stock solution will be used to prepare the standard solution of 0.01 M $Ni(NO_3)_2{\cdot}6H_2O$
4. 0.01 M $Ni(NO_3)_2{\cdot}6H_2O$ solution (0.2 mL) is added into the silylated SBA-15 (200 mg).
5. The resulting slurry is stirred and left at RT for 24 hours
6. Then the slurry is dried in the oven overnight
7. Once dried, the powder is calcinated at 550 °C in a furnace for 6 hours.

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

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, face mask, and safety goggle.
2. Bring Nickel (II) nitrate, $Ni(NO_3)_2{\cdot}6H_2O$, bottle and DI water (or ethanol) to the fume hood in room 135.

3. Dissolve nickel nitrate (0.0495 g) precursor in DI water (5 mL) for 10 wt.% Ni/SBA-15 catalyst.
4. Add ethanol (5 mL) to SBA-15 (100 g) in a shallow beaker and mix vigorously while adding the Ni precursor solution.
5. Stir until the resulting slurry is well-mixed and evaporate the ethanol by heating the sample on a hot plate.
6. Collect the dried powder onto a combustion boat and dry over-night in the oven (70 °C).
7. Transfer sample to the muffle furnace (Prof. Yin lab) and calcinate for 5 h at 500 °C (2.5 °C / min).

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).

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 01/26/2019, 07/08/2020, 07/10/2020, 09/27/2021, 10/12/2021

Nitric 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 nitric acid (HNO_3, CAS No. 7697-37-2) used in laboratory. Its purpose is not to have any accident or risk. Nitric acid is oxidizer, so it may intensify fire. It causes severe skin burns and eye damage.

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

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

OSHA Hazards: Corrosive, Oxidizer, Target Organ Effect (Lungs, Teeth, Cardiovascular system)

GHS Classification

Oxidizing liquids (Category 3)
Skin corrosive (Category 1A)
Serious eye damage (Category 1)

Signs and Symptoms of Exposure

Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin., Inhalation may provoke the following symptoms:, spasm, inflammation and edema of the bronchi, spasm, inflammation and edema of the larynx, pneumonitis, Symptoms and signs of poisoning are:, burning sensation, Cough, wheezing, laryngitis, Shortness of breath, Headache, Nausea, Vomiting, Pulmonary edema. Effects may be delayed., Large doses may cause: conversion of hemoglobin to methemoglobin, producing cyanosis; marked fall in blood pressure, leading to collapse, coma, and possibly death.

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

Lab workers using nitric 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 nitric acid described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

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

3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 10 mL of this nitric acid in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this nitric 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 nitric 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.

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.

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 ^{o}C for 1 hour. Then heat up to 120 ^{o}C 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.

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.

Preparation of Ni/SBA-15

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Add nickel nitrate hexahydrate (0.01 g, $Ni(NO_3)_2{\cdot}6H_2O$), nitric acid (1.25 mL, HNO_3) and SBA-15 (0.2 g) into water (25 mL).
3. The mixture is sonicated for a while.
4. The mixture is heated to 55 ^oC in a flask.
5. Add urea (1.2012 g) into water (25 mL).
6. Add the urea solution to the first solution (step 2).
7. The mixture is heated to 90 ^oC for 4 h.
8. Cool down to 55 ^oC.
9. The mixture is stirred at 55 ^oC for 10 min.
10. Cool down the mixture to room temperature.
11. Filter the mixture.
12. Wash the powder for 3 times with water.
13. Dry the powder overnight.
14. The powder is calcined at 550 ^oC under air for 3 h.

Copper substrate: Chemical cleaning

1. Wear nitrile chemical resistance gloves, flame resistance lab coat, and safety goggles.
2. Place the polished polycrystalline copper substrate (2 mm thick, 10 mm diameter) in #1 beaker (100 mL) and add acetone (50 mL).
3. Sonicate the beaker for 5 min.
4. Transfer the copper substrate into #2 beaker and add isopropyl alcohol (50 mL).
5. Sonicate #2 beaker 2 for 5 min.
6. Repeat the above step (steps 3-4) with DI water (50 mL) followed by HNO_3 solution (1%, 50 mL).
7. Rinse the copper substrate with isopropyl alcohol for 2 min.
8. Sonicate the copper substrate in a mixture of DI water (60 mL) and glacial acetic acid (2 mL) for 5 min. Then, blow-dry with a flow of N2.

9. Store the samples at low vacuum (10^{-2} torr) or at Ar/ N2 atmosphere immediately to avoid oxidation (until characterize with XPS or mount in the RAIRS chamber).
10. Dispose of the washing and cleaning solvents properly as chemical waste.

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 07/01/2018, updated 01/26/2019, 12/11/2019

Nitrogen
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 nitrogen (N_2, CAS No. 7727-37-9) used in laboratory. Its purpose is not to have any accident or risk. Nitrogen 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: Compressed Gas
GHS Classification
Gas under pressure (Liquefied gas)

Signs and Symptoms of Exposure
May be harmful. Nausea, Headache, 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

A ventilation monitor is required on each lab hood or gas manifold in which nitrogen 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 nitrogen 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 nitrogen 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 nitrogen 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 nitrogen 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 nitrogen 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 nitrogen 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 nitrogen gas cylinders shall be labeled as empty. Depleted nitrogen 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 nitrogen gas cylinders by EH&S, even when empty, may entail extraordinary costs. Therefore, nitrogen 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 nitrogen 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! NITROGEN 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 nitrogen must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of nitrogen and understand the hazards.

Lab workers using nitrogen 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 nitrogen 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 nitrogen 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 nitrogen 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 nitrogen 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 & 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.

ALD Reactor

1. Safely secure Nitrogen 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 ALD system using a copper tube.
4. Carefully adjust the outlet pressure to 15 psi.
5. Close the Swagelok valve and angle valve on the ALD reactor that is next to the mechanical pump.
6. Fill the copper tube with Nitrogen gas. Then open the 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 Swagelok valve to leak the gas into the ALD reactor, monitor the pressure in the system
10. Close the Swagelok valve.
11. Close the valve on the regulator. Close the main valve.
12. Open the Swagelok valve and angle valve to pump the line.

Synthesis of PtCu alloy

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (25 mL) under nitrogen in a fume hood and put a stir bar into it.
3. Put platinum(II) acetylacetonate (8.574 mg, 0.0218 *m*mol) into the flask.
4. Take ethylene glycol (5 mL) by a syringe in a fume hood and add it slowly to the flask.
5. Rinse syringes several times with ethanol before disposal.
6. The washings should be disposed as hazardous organic waste.
7. Put a syringe needle into sharps-disposal container.
8. Put polyvinylpyrrolidone (0.6 mg) and copper sulfate pentahydrate (5.452 mg, 0.0218 *m*mol) into the flask.
9. Change the pH with NaOH (0.1 M) to pH=10 and close the flask with rubber septum.
10. Reflux the mixture under nitrogen for 2 h at 198 ^{o}C. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
11. Remove the septum from the flask and put SBA-15 (558.36 mg) in the flask and stir the mixture for 2 h.
12. Sonicate the mixture for 1 h.
13. Centrifuge and dispose the liquid.
14. Evacuate the flask containing the sample then dry the powder at ca. 60 ^{o}C for 12

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

Synthesis of Cu@Pt nanoparticle

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

Synthesis of Pt@Cu nanoparticle

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (25 mL) under nitrogen in a fume hood.
3. Put platinum acetylacetonate (18.582 mg, 0.04725 *m*mol) into the flask.
4. Take ethylene glycol (5.775 mL) by a syringe in a fume hood and add it slowly to the flask.
5. Rinse syringes several times with ethanol before disposal.

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

UHV #3, Michelle

1. Safely secure nitrogen 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 nitrogen gas. Then open the angle valve to pump down.
7. Repeat steps for two times to purge the copper line.
8. Carefully pressurize copper line.
9. Introduce the nitrogen gas into the chamber through the leak valve.
10. Close the leak valve when the pressure inside the chamber equal to atmospheric pressure.
11. Close the valve on the regulator.
12. Close the main valve of the nitrogen cylinder.
13. Open the angle valve to pump the line.

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 °C 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 °C.
10. Open the valve to introduce TEMAH (1.6 s) and nitrogen (5 s) into the ALD chamber.
11. After reaction, slowly cool down precursor, chamber, and sample to room temperature.
12. Collect and transfer sample into a vial for analysis.
13. Collect the waste of TEMAH and dispose into a proper waste bottle.

Synthesis of 3-aminopropyltriethoxysilane-grafted SBA-15

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a 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 °C for 24 h. If left unattended in a fume hood, put a label with chemical name and hazard information.
8. Transfer the mixture into a centrifuge tube.
9. Centrifuge (3000 rpm) for 10 min.
10. Take out the excess solvent using a pipette.
11. Wash the particles with ethanol (25 mL).
12. Centrifuge (3000 rpm) for 10 min.
13. Take out the excess ethanol using a pipette.
14. Repeat steps 11-13 once more.
15. Dispose waste in the properly labeled container.
16. Dry the white powder in a vacuum chamber.

Synthesis of Au nanoparticles within the SBA-15 support

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry two flasks (250 mL) under nitrogen.
3. Put deionized water (100 mL) into one flask.

4. Add 3-aminopropyltriethoxysilane-grafted SBA-15 (1.0 g) into the flask
5. After the powder is dispersed evenly, add gold chloride solution (15 mL, 10 mM).
6. Maintain the suspension by stirring the mixture for 2 h at room temperature. If left unattended in a fume hood, put a label with chemical name and hazard information.
7. Transfer the mixture into a centrifuge tube.
8. Centrifuge (3000 rpm) for 10 min.
9. Take out the excess solvent using a pipette.
10. Wash the particles with DI water (25 mL).
11. Centrifuge (3000 rpm) for 10 min.
12. Take out the excess DI water using a pipette.
13. Repeat steps 10-12 once more.
14. Dispose waste in the properly labeled waste container.
15. Dry the yellow powder in a vacuum chamber.
16. Dry a clean beaker (150 mL).
17. Put deionized water (100 mL) into the other flask.
18. Redisperse the solid into the flask.
19. For the reduction reaction, add sodium borohydride (2.5 g) into the flask.
20. Maintain suspension by stirring the mixture for 2 h at RT. If left unattended in a fume hood, put a label with the chemical names and hazard information.
21. Transfer the mixture into a centrifuge tube.
22. Centrifuge (3000 rpm) for 10 min.
23. Take out the excess solvent using a pipette.
24. Wash the particles with DI water (25 mL).
25. Centrifuge (3000 rpm) for 10 min.
26. Take out the excess DI water using a pipette.
27. Repeat steps 24-26 once more.
28. Dry the reddish-purple powder in a vacuum chamber.
29. Dispose waste in the properly labeled waste container.
30. After the powder is dry, calcinate the sample at 500 ^{o}C for 6 h. If left unattended, leave a label nearby with the chemical names and hazard information.
31. After cooling, collect the sample.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 06/01/2015, 11/01/2016, 03/06/2019, 10/15/2021, 10/19/2021

Nitrogen Liquid
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 nitrogen liquid (N_2, CAS No. 7727-37-9) used in laboratory. Its purpose is not to have any accident or risk. Nitrogen liquid cylinder contains refrigerated gas under pressure. It may cause cryogenic burns or injury. It is considered hazardous by OSHA.

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

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

OSHA Hazards: Refrigerated Gas
GHS Classification
Gas under pressure (Refrigerated Liquefied Gas)

Signs and Symptoms of Exposure
Extremely cold material. Liquid can cause burns similar to frostbite, No known significant effects or critical hazards

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye/Face 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 nitrogen liquid 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 nitrogen liquid 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 nitrogen liquid, 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 nitrogen liquid 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 nitrogen liquid 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 nitrogen liquid 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 nitrogen liquid 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 nitrogen liquid cylinders shall be labeled as empty. Depleted nitrogen liquid 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 nitrogen liquid cylinders by EH&S, even when empty, may entail extraordinary costs. Therefore, nitrogen liquid 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 nitrogen liquid 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! NITROGEN LIQUID 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 nitrogen liquid must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of nitrogen liquid and understand the hazards.

Lab workers using nitrogen liquid 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 nitrogen liquid 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 nitrogen liquid less than 1 L at a time (higher pressure REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this nitrogen liquid 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 nitrogen liquid. 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.

Fill a dewar with LN2 from a tank cylinder

1. Wear flame-resistant lab coat, apron, full-face shield, and waterproof cryo-gloves.
2. Bring a LN2 dewar to LN2 tanks in hallway.
3. Warn pedestrians not to pass the hallway.
4. Open the cap of Dewar
5. Insert the hose of the LN2 tank into the dewar.
6. Open the main valve of the LN2 tank.
7. Fill the dewar slowly.
8. Top the dewar off.
9. Close the main valve of the LN2 tank.
10. Take the hose out.
11. Close the cap of dewar.
12. Bring the dewar into the lab.

UHV #3, Michelle: sample cooling

1. Install a fan in the top side of the manipulator next to the rotary stage.
2. Run the fan all the time during experiment.
3. Insert a funnel into the small inlet at the top of the manipulator.
4. Slowly add about one quarter cup of liquid nitrogen into the manipulator.
5. Keep Adding liquid nitrogen a little at a time (about every 5 minutes) in order to maintain a boiling liquid phase in the manipulator.

UHV #3, Michelle: stop cooling

1. Remove the funnel.
2. Blow dry air through a tube into the manipulator while the liquid nitrogen boils away, and flow until the manipulator is at room temperature and dry.
3. Make certain that there is no resistance to the air stream.
4. Leave the air flow overnight after the experiment.

SOP Reviewed and Approved by:

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Signature

Approval Date: 02/01/2013, updated 05/15/2016, 03/06/2019