# Acetone
# 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 acetone ($C_3H_6O$, CAS No. 67-64-1) used in laboratory. Its purpose is not to have any accident or risk. Acetone is highly flammable liquid and vapor, and causes serious eye and skin irritation. Also it may cause drowsiness and dizziness.

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

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

OSHA Hazards: Flammable liquid, Target organ effect (Liver and Kidney), Irritant
GHS Classification

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

Signs and Symptoms of Exposure
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 acetone 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 ARE

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

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

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

5) discuss ALL issues or concerns regarding this acetone 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 acetone. 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 a nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggle.
2. Acetone is used to wash and clean the hardware that will be used for UHV system.
3. Always put acetone in a wash bottle.
4. Only use the necessary amount.
5. Collect all the used acetone in a beaker and dispose into a proper waste bottle.

**Coupling catalytic reaction**

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 acetone 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-$TiO_2$-APTES catalyst (50 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 acetophenone from the flammable cabinet and put it into the fume hood. Add the reactant (5.5 µL) into the reaction mixture.
10. Take acetone from the flammable cabinet and put it into the fume hood. Add the reactant (0.5 mL) into the reaction mixture.
11. Close the test tube with the rubber septum, seal with Teflon tape and connect the oxygen supply to the test tube.
12. 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.
13. 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).
14. 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.
15. After centrifuging bring the closed vial back to the fume hood and transfer the liquid into a new vial.
16. Inject the sample into GC using a Hamilton syringe (10 µL).
17. Dispose all the waste into the appropriately labeled waste bottle.

**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 acetone (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.

## 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.

## 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.

**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.

**NiO deposition on SBA-15 via ALD reactor**

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, **AND** a proper face mask at all times (Covid-19) while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
2. Clean the sample holder with acetone
3. Place SBA-15 in the sample holder.
4. Preheat the support (SBA-15) at 200 ℃ for 2 h.

5. Ni precursor (nickel(II) bis(2,2,6,6-tetramethyl-3,5-heptanedionate, Ni(tmhd)) is pretreated at 165 ℃ in an oil bath.
6. The reactor is set to 150 ℃.
7. Dose the Ni precursor for 30 min by using Ar carrier gas (200 mTorr).
8. Purge the reactor with Ar gas (500 mTorr) for 5 min.
9. Dose deionized water (100 mTorr) for 2 min.
10. Purge the reactor with Ar gas (500 mTorr) for 10 min.
11. Repeat 7–10 steps repeatedly until desired growth is obtained.

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

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

**SBA-15 Impregnation**

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, **AND** a proper face mask at all times (Covid-19) while inside the lab. Carry out the following procedures in a fume hood.
2. Clean glassware, such as flask and beaker with acetone.
3. Prepare nickel(II) nitrate hexahydrate solution (2.5 mL, 0.84 M).
4. Pour SBA-15 (100 mg) in a beaker (50 mL)
5. Add ethanol (5 mL) or DI water:ethylene glycol (1:1, 5 mL) to the beaker.
6. Add the desired amount of the Ni solution (20 μL for 1 wt.% Ni loading)
7. The mixture was stirred at 80 ℃ until the ethanol evaporated.

8. The collected powder is transferred to the oven for drying overnight.
9. Calcine the powder in air condition (muffle furnace (Prof. Yin lab) at 500 ℃ for 5 hours (2.5 ℃/m speed).
10. Collect the sample from the furnace.
11. Clean up the flasks with solvents (distilled water, ethanol and acetone).

**Tetrakis(ethylmethylamino)hafnium(IV) for ALD reactor**

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Put SBA-15 (50 mg) into a sample holder in the fume hood of room 135.
3. Spotweld mesh on the sample holder.
4. Attach the sample holder to the ALD reactor.
5. Preheat SBA-15 at 200–300 °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.
14. Use acetone or ethanol to wash the sample holder.

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SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 03/01/2016, 05/15/2016, 10/10/2017, 11/20/2019, 12/11/2019, 07/08/2020, 10/12/2021, 10/18/2021

# Acetonitrile
# 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 acetonitrile ($C_2H_3N$, CAS No. 75-05-8) used in laboratory. Its purpose is not to have any accident or risk. Acetonitrile is highly flammable liquid and vapor, and causes serious eye and mild skin irritation. Also it is harmful if swallowed, if inhaled or in contact with skin.

Synonyms: ACN, Methyl cyanide

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

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

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

GHS Classification

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

Signs and Symptoms of Exposure

Treat as cyanide poisoning. Always have on hand a cyanide first-aid kit, together with proper instructions., The onset of symptoms is generally delayed pending conversion to cyanide., Nausea, Vomiting, Diarrhea, Headache, Dizziness, Rash, Cyanosis, excitement, depression, Drowsiness, impaired judgment, Lack of coordination, stupor, 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:

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- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.

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

## 9. PRIOR APPROVAL/REVIEW REQUIRED

All work with acetonitrile 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 acetonitrile.

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

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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 acetonitrile 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.

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

**HPLC Eluent Preparation**

1. Wear butyl rubber gloves, flame-resistant lab coat, and safety goggles.
2. Take acetonitrile bottle out of the flammable chemical cabinet and place the bottle securely on the surface in a fume hood.
3. Carefully transfer the desired amount of acetonitrile into the designated vessel.
4. Close and seal the bottle and put it back to the flammable chemical cabinet.
5. The used eluent solution needs to be treated as hazardous waste.

**Solvent in Oxidation reaction**

1. Wear nitrile chemical-resistant glove, mask, flame-resistant lab coat, and safety goggles.
2. Add acetonitrile (8.5 mL) to a vial by use of syringe.
3. Add benzyl alcohol (25 mg), potassium carbonate (52 mg), Au-nanoparticles supported on titania (20 mg) to the vial.
4. After reaction is finished, store reaction mixture in an appropriate labeled vial.
5. Keep the vial in refrigerator.

**Catalytic Oxidation Reaction**

1. Wear nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggles.
2. Into a test tube with a stir bar, add L-proline (16.4 mg) and acetonitrile (1 mL).
3. Ultrasonicate and stir the mixture.
4. Add tert-butanol (5 μL).

5. Add benzaldehyde (80 μL) and acetophenone (92 μL).
6. Close with a rubber septum, seal with parafilm, connect the oxygen supply to the test tube, and set the temperature at 50 °C.
7. After reaction, collect the samples.
8. Run GC analysis.

**Making of titania shells**

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

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SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 02/01/2013, updated 03/01/2014, 06/01/2015, 03/03/2016, 05/15/2016

# Acrolein
# 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 acrolein ($C_3H_4O$, CAS No. 107-02-8) used in laboratory. Its purpose is not to have any accident or risk. Acrolein is highly flammable liquid and vapor and a CAL/OHSA Select Carcinogen. Toxic if swallowed and fatal if inhaled. It causes serious eye and skin irritation.

Synonyms: 2-Propenal

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

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

OSHA Select Carcinogen

The OSHA Lab Standard defines a “Select Carcinogen” as any substance, which meets one of the following criteria:

(i) It is regulated by OSHA as a carcinogen; or

(ii) It is listed under the category, "known to be carcinogens," in the Annual Report on Carcinogens published by the National Toxicology Program (NTP); or

(iii) It is listed under Group 1 ("carcinogenic to humans") by the International Agency for Research on Cancer Monographs (IARC); or

(iv) It is listed in either Group 2A or 2B by IARC or under the category, "reasonably anticipated to be carcinogens" by NTP, and causes statistically significant tumor incidence in experimental animals in accordance with any of the following criteria:

(A) After inhalation exposure of 6-7 hours per day, 5 days per week, for a significant portion of a lifetime to dosages of less than 10 mg/m;

(B) After repeated skin application of less than 300 (mg/kg of body weight) per week; or

(C) After oral dosages of less than 50 mg/kg of body weight per day.

OSHA Hazards: Flammable liquid, Target Organ Effect, Highly toxic by inhalation, Highly toxic by ingestion, Highly toxic by skin absorption, Corrosive, Carcinogen

GHS Classification

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

Skin sensitization (Category 1)
Carcinogenicity (Category 2)
Acute aquatic toxicity (Category 1)
Chronic aquatic toxicity (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. Respiratory Protection

Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

### b. Eye Protection

Face shield and ANSI compliant safety glasses with side shields should be worn. Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166 (EU). Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

### c. Skin and Body Protection

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

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

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

### d. Hand Protection

At a minimum, wear nitrile chemical-resistant gloves. 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 acrolein 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 acrolein.

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

Lab workers using acrolein 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 acrolein 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 < 1 mL of this acrolein in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this acrolein 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 acrolein. 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.

## UHV #1, Victor Chamber_1

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and full-face respirator.
2. Bring the acrolein bottle to the fume hood in room 135. Open the acrolein bottle. Use a flint glass pipette to transfer acrolein (~2 mL) into the sealed-off Pyrex glass end tube with 1’33 flange and Swagelok valve.
3. Close the acrolein bottle. Clean the pipette with acetone and place the waste liquid into a waste container prepared with a carcinogen hazardous label. Put the acrolein bottle back to the storage place.
4. Connect the glass tube to the leak valve on the UHV system and to the gas line.
5. Submerge the glass tube in liquid nitrogen to freeze acrolein and slowly open the valve on the gas line to mechanical pump to pump down the frozen liquid sample.
6. Close the valve and thaw acrolein to release trapped air.
7. Repeat the freeze and pump steps until the pressure does not increase any more when the valve is opened.
8. Fill the delivery line with the vapor of acrolein by opening and closing the valve on the glass tube.
9. Open the leak valve to dose the acrolein gas into the chamber for experiments.
10. After experiments, clean the glass tube with acetone and place the waste liquid into the waste container.

## UHV #1, Victor Chamber_2

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 acrolein. Make sure to memorize the first-aid measures, handling and storage, PPE, & signs and symptoms of exposure.
3. Bring required materials to a fume hood: acrolein, a syringe, and a glass tube connected to a Swagelok Valve.
4. Secure the glass tube on the 3-prong clamp.
5. Carefully open the acrolein bottle. The bottle must be kept upright at all times to prevent leakage.
6. Use a syringe to transfer acrolein (2 mL) from the bottle into the glass tube through the connected Swagelok valve. Do not overfill the tube. Be careful not to spill acrolein. In case of exposure, follow procedures outlines in SDS and call 911.
7. Close the Swagelok valve and make sure there is no leak.
8. Connect the Swagelok valve to the gas manifold on the chamber.
9. Close the acrolein bottle. Check for any leaks of acrolein.

10. Put the acrolein bottle back to the designated storage cabinet. Keep the acrolein container tightly closed in a dry and well-ventilated place.
11. Clean the syringe with acetone and place the waste liquid into the waste container prepared with a carcinogen hazardous label.

**UHV #2, RAIRS Chamber: For acrolein at room temperature**

1. Equip the proper Personal Protective Equipment: flame resistant lab coat, safety glasses, nitrile gloves, and insulated gloves for liquid nitrogen manipulation.
2. Under the fume hood, use an injector to extract acrolein (2 mL) and inject it into a glass tube.
3. Attach the glass tube to the metal adaptor at the end of the leak valve on top of the chamber.
4. Fix the metal adaptor to the gas line.
5. Pour liquid nitrogen in a Dewar cup and submerge the glass tube containing toluene until the liquid freezes.
6. Open the valve next to the glass tube metal adaptor and then open the valve to the mechanical pump.
7. Pump the line down until it reaches the normal base pressure. Close the valve next to the glass tube metal adaptor. Allow the liquid to thaw.
8. Repeat steps 4–6 until the gasses dissolved in the liquid are completely absent.
9. Close the valve to the mechanical pump and open the valve to the to the glass tube metal adaptor. Let the liquid vaporize in the line.
10. For UHV use

a. Close the valve next to the glass tube
b. Keep spinning the leak valve to the UHV chamber until desired pressure is reached
c. Immediately start the timer and close the leak valve quickly when a desired surface coverage (e.g. 10 Langmuir) is achieved
d. Open the valve to the mechanical and turbo pumps to evacuate the gas line.

11. For high pressure cell reaction:

a. Slightly open the valve to the gas mixture storage line until desired pressure (e.g. 2 Torr) is reached.
b. Add other gases needed into the storage line.
c. After finishing the gas mixture preparation, open the two valves to the high pressure cell and run the reaction.

12. In both experiments (HPC reaction and UHV use), the remaining acrolein and other gases will be pumped down by mechanical pump, turbo pump, and cryopump, which fulfills the chemical waste disposal.

**UHV #3, NanoReactor**

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 acrolein again; especially remind first-aid measures, handling and storage, PPE, & signs and symptoms of exposure.
3. Bring required materials to a fume hood: acrolein, a syringe, a glass tube with fitting to the gas manifold.
4. Secure the glass tube on the 3-prong clamp.
5. Open the acrolein bottle. The container must be carefully resealed and kept upright to prevent leakage.
6. Use a syringe to transfer acrolein from the original bottle into the glass tube. Be careful not to spill acrolein. Keep watching any leak of acrolein. 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. Close the acrolein bottle. Keep the acrolein container tightly closed in a dry and well-ventilated place.
8. Connect the glass tube to the gas manifold of UHV #3, NanoReactor
9. Put the acrolein bottle back to the storage place. Keep watching any leak of acrolein.
10. Clean the syringe with acetone and place the waste liquid into the waste container prepared with a carcinogen hazardous label. Keep watching any leak of acroline.

**Catalytic Hydrogenation Reaction**

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Place the catalyst in the quartz reactor. About 5 mg of the catalyst is placed in the reactor between some layers of quartz wool above the metal disk placed in the reactor.
3. The reactor is placed inside the heating jacket and connected carefully with the ultratorr connectors to the sampling line. The valves are open and the system is pump. The pressure should be below 60 mTorr. If not, check connections or disconnect the reactor and connect it again. Be careful not to break it.
4. Dry the catalyst, which is PtCu alloy supported by SBA-15 at 150 $^{o}$C for 1 h.
5. Activate the catalyst via reduction cycles at a 200 $^{o}$C for 2 h.
6. After the pretreatment of catalyst, wait until the temperature comes back to 100 $^{o}$C.
7. Turn off the circulation pump and then evacuate the system.
8. Once the vacuum is reached, the gases can be introduced. The acrolein is introduced first (usually Ar is the last one to be introduced due to the high partial pressure use). The dosing line is isolated from the mechanical pump by closing

the proper valve. The valve of the probe molecule is introduced until the desired pressure is reached in the Pirani gauge; the valve that connects the dosing line with the sampling loop is closed (check that the pressure in the sample loop is stable). Evacuate the dosing line (check the pressure with the TC gauge) and, then, be ready to repeat the same procedure followed with the probe molecule for hydrogen and Ar.

9. After all gases are introduced, the circulation pump is turned on.
10. After 2 min, the 6-way valve is used by moving the handle to the other side. Immediately after, the START button in the GC controller is pushed as well as the start button in the DMM software to start the collection of the data. After the peaks have appeared, leave the system run for additional 10 min in order to be sure that nothing else remains in the GC column and then push the STOP button in the GC controller and in the software. Save the collected data and wait until the system is ready for a new run (showed by the LEDs in the GC). If multiple runs are desired, the handle can be moved every 12 min without pressing any button in the software and the GC controller.

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SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 04/01/2016, updated 11/01/2016, 04/17/2017, 06/23/2021

# Compressed Air Gas
# 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 compressed air gas (Air, CAS No. 132259-10-0) used in laboratory. Its purpose is not to have any accident or risk. Compressed air gas cylinder 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 (Compressed gas)

Signs and Symptoms of Exposure
No data available

## 3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

### a. Eye Protection

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

### b. Skin and Body Protection

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

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

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

### c. Hand Protection

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

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

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

## 4. ENGINEERING/VENTILATION CONTROLS

A ventilation monitor is required on each lab hood or gas manifold in which compressed air 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 compressed air 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 compressed air 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 compressed air 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 compressed air 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 compressed air 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 compressed air 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 compressed air gas cylinders shall be labeled as empty. Depleted 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 gas cylinders by EH&S, even when empty, may entail extraordinary costs. Therefore, compressed air 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 compressed air gas must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

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

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

## 9. DESIGNATED AREA

A designated area shall be established where limited access, special procedures, knowledge, and work skills are required. A designated area can be the entire laboratory, a specific laboratory workbench, or a laboratory hood. Designated areas must be clearly marked with signs that identify the chemical hazard and include an appropriate warning; for example: WARNING! COMPRESSED AIR 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 compressed air gas must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of compressed air gas and understand the hazards.

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

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

When working in the lab, a laboratory worker must:

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

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using compressed air 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

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.

## GC Analysis

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Open air, $N_2$ and $H_2$ gas cylinders valves.
3. Load the method on the ChemStation program.
4. Inject a liquid sample.
5. Run the method.

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SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 06/01/2015, 01/26/2019

# Ammonium 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 ammonium chloride ($H_4ClN$, CAS No. 12125-02-9) used in laboratory. Its purpose is not to have any accident or risk. Ammonium chloride is toxic if swallowed. Also it is harmful if inhaled.

Synonyms: Salmiac

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

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

OSHA Hazards: Toxic
GHS Classification

- Acute toxicity, Oral (Category 4)
- Eye irritation (Category 2A)
- Acute aquatic toxicity (Category 2)
- Chronic aquatic toxicity (Category 2)

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

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

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

1. Wear nitrile chemical–resistant gloves, a flame-resistant lab coat, and a safety goggle.
2. **QD-Bn** (8.0 *m*mol), sodium ethanethiolate ($NaSC_2H_5$, 4 eq.) and dimethylform-amide (50 mL) are added to a flask (200 mL).
3. The mixture is stirred at 110 $^oC$ until TLC analysis shows that the starting material is completely consumed within 4–6 h.
4. The mixture is cooled down to room temperature.
5. Ammonium chloride ($NH_4Cl$, 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).

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SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2020

# Ammonium hydroxide
# STANDARD OPERATING PROCEDURE

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

## 1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when ammonium hydroxide ($H_5NO$, CAS No. 1336-21-6) is used in laboratory. Its purpose is not to have any accident or risk. Ammonium hydroxide is combustible toxic liquid and has Lachrymator hazard (a strong and very unpleasant small). It is harmful if inhaled or swallowed. Also, it causes severe skin burns and eye damage.

Synonyms: Ammonia aqueous, Ammonia water

## 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, Toxic by Ingestion
GHS Classification

Acute toxicity, Oral (Category 4)
Skin corrosion (Category 1A)
Serious eye damage (Category 1)
Acute aquatic toxicity (Category 1)

Signs and Symptoms of Exposure
Burning sensation, Cough, wheezing, Laryngitis, Shortness of breath, Spasm, inflammation and edema of the larynx, Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin.

## 3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

### a. Eye Protection

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

### b. Skin and Body Protection

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

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

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

**c. Hand Protection**

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

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

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

## 4. ENGINEERING/VENTILATION CONTROLS

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

## *5.* SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

## 6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

## 7. DECONTAMINATION

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

## 8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

## 9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

## 10. DESIGNATED AREA

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

## 11. SAFETY DATA SHEETS

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

## 12. DETAILED PROTOCOL

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

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

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with ammonium hydroxide described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-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 ammonium hydroxide in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this ammonium hydroxide with the PI prior to its use.

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

**Ammonium Solution Preparation**

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take ammonium hydroxide bottle out of the corrosive chemical cabinet and bring it to a fume hood.
3. Transfer the designated amount of ammonium hydroxide into the designated vessel.
4. Close and seal the bottle and put it back.
5. Dilute ammonium solution with designated amount of water.
6. Extra unused solution needs to be treated as hazardous waste.

**Silica particle preparation 1**

1. Wear nitrile chemical-resistant gloves, mask, flame-resistant lab coat, and safety goggles.
2. Take ammonium hydroxide solution (0.86 mL) by a syringe in a fume hood.
3. Inject ethanol and tetraethyl orthosilicate into the mixture by the syringe.
4. Put the syringe needle into sharps-disposal container.

**Silica particle preparation 2**

1. Wear nitrile chemical-resistant gloves, mask, flame-resistant lab coat, and safety goggles.
2. Take ammonium hydroxide solution (1 mL) by a syringe in a fume hood.
3. Add it to isopropyl alcohol (20 mL).

4. Bring DI water and tetraethyl orthosilicate mixture into the fume hood and add the ammonium hydroxide - isopropanol mixture.
5. After injecting (step 2), put a syringe needle into sharps-disposal container.

**Making of titania shells**

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

**Synthesis of silica spheres**

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Make a waste bottle labeled as toxic hazardous waste. Review the SDS of tetraethyl orthosilicate, ethanol, and ammonium hydroxide again; especially remind first aid measures, handling and storage, & PPE.*
3. Place an Erlenmeyer flask into a fume hood and put a stir bar into it.
4. Add ethanol (72 mL) and milli-Q water (12 mL) to the flask and close with rubber septum. Stir for 5 minutes.

5. Take the bottle of ammonium hydroxide from a corrosive base cabinet and place it into the fume hood. Remove the septum from the flask and add ammonium hydroxide (1.95 mL) by using a micropipette. Stir for 5 minutes.
6. Take tetraethyl orthosilicate (TEOS) from the flammable cabinet and put it into the fume hood. Add TEOS (2.55 mL) to the mixture.
7. Stir for 4 hours at room temperature.
8. Centrifuge and dispose the waste into the waste bottle labeled toxic and corrosive hazardous waste.
9. Label the centrifuge tube appropriately, cover it with perforated aluminum foil and dry the powder in a vacuum desiccator overnight.

**RCA Cleaning Protocol**

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

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

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

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

SOP Reviewed and Approved by:

| Francisco Zaera | |
|---|---|
| Print name | Signature |

Approval Date: 06/01/2013, updated 03/01/2014, 10/01/2014, 03/03/2016, 05/15/2016, 07/16/2019, 02/07/2020

# Argon
# 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 argon (Ar, CAS No. 7440-37-1) used in laboratory. Its purpose is not to have any accident or risk. Argon contains gas under pressure. It may be harmful if swallowed, if inhaled or if absorbed through skin. Also it may cause skin and eye irritation.

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

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

OSHA Hazards: Not known
GHS Classification
N/A

Signs and Symptoms of Exposure
Nausea, Dizziness, 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

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

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

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

All lines or ducts carrying purged or exhausted emissions of argon 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 argon 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 argon 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 argon 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 argon gas cylinders shall be labeled as empty. Depleted argon 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 argon gas cylinders by EH&S, even when empty, may entail extraordinary costs. Therefore, argon 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

## 8. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

## 9. DESIGNATED AREA

A designated area shall be established where limited access, special procedures, knowledge, and work skills are required. A designated area can be the entire laboratory, a specific laboratory workbench, or a laboratory hood. Designated areas must be clearly marked with signs that identify the chemical hazard and include an appropriate warning; for example: WARNING! ARGON 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 argon gas must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of argon gas and understand the hazards.

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

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with argon 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 argon gas under atmospheric condition in any given reaction (higher pressure REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this argon 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 argon. 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.

**Reactor #3, Photocatalysis: Replacing empty gas cylinder for GC**

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

**UHV #1, Victor**

1. Safely secure Argon cylinder using a chain clamp or ring clamps.
2. Ensure the cylinder valve is completely closed.
3. Attach the appropriate pressure regulator to the cylinder and connect it to the gas manifold of the UHV system using copper/stainless steel tubing.
4. Carefully adjust the outlet pressure to about 15 psi using the regulator hand knob.
5. Close the valve between the gas manifold and the mechanical pump.
6. Open the regulator outlet valve and fill the copper tube with Argon gas.
7. Open the valve of the mechanical pump to pump down the gas line.
8. Repeat the steps 5-6 three times to purge the copper line.
9. Carefully pressurize the copper line to deliver the gas.

10. Slowly open the leak valve to leak the gas into the UHV chamber and monitor the pressure in the UHV system.
11. After use, close the leak valve to the UHV system.
12. Close the valve on the regulator.
13. Close the main valve of Argon cylinder.
14. Open the valve of the pump to evacuate the line.

**UHV #2, RAIRS**

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

**UHV #3, Michelle**

1. Safely secure Argon 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 Argon gas. Then open the angle valve to pump down.
7. Repeat the steps 5-6 three times to purge the copper line.
8. Carefully pressurize copper line.
9. Slowly open the leak valve to leak the gas into the UHV system, monitor the pressure in the UHV system
10. Close the leak valve.
11. Close the valve on the regulator.
12. Close the main valve of the Argon cylinder.
13. Open the angle valve to pump the line.

**UHV #4, Praxis**

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

2. Check that the Argon cylinder line is closed.
3. Open the valve, which connects the Argon line and the gas manifold pump, to evacuate the Argon line.
4. Wait until the pressure gauge at the bottom of the electronics cabinet reaches 20mTorr to indicate full gas evacuation.
5. Close the small black valve, which connects the gas manifold pump to the Argon leak valve, to stop pumping of the Argon line.
6. Open the Argon cylinder valve to let gas flow to the chamber leak valve. Adjust the pressure of Argon in the chamber by opening/closing the leak valve.
7. Turn on the ion gun to 'operate' if argon sputtering is to be performed. The ion energy dial can also be adjusted on the ion gun console.
8. When sputtering or Argon use is finished, close the Argon leak valve.
9. Turn down the ion gun energy and switch the ion gun from 'operate' to the 'zero' position.
10. Close the green Argon swagelok valve to stop the flow of gas from the Argon tank into the leak valve.
11. Open the small black valve so that the leak valve can be pumped out.
12. Close the Argon tank valve.

**UHV #5, UC Chamber**

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Turn on the ion gauge controller to ensure the stability of the pressure inside the chamber. If the pressure in the preparation chamber is below 3E-5 Torr, open the leak valve, and wait until the pressure goes down again.
3. Turn on the sputtering system, and ensure the emission dial is turned in completely counter-clockwise orientation, wait for degas process is done.
4. Gently and graduate release the argon leak valve until chamber pressure reaches to 5E-5 Torr, and activate the sputtering process afterwards.
5. Once tasks are done, deactivate the sputtering process, fully close the leak valve, turn off the emission current, and power off the sputtering device.

**UHV #4 Praxis & #6 Nanoreactor**

1. Safely secure Argon cylinder using a chain clamp.
2. Ensure the cylinder valve is completely closed.
3. Attach the appropriate pressure regulator to the cylinder and connect it to the gas manifold of the Nanoreactor system using copper/stainless steel tubing.
4. Carefully adjust the outlet pressure to about 15 psi using the regulator hand knob.
5. Close the valve between the gas manifold and the mechanical pump.
6. Open the regulator outlet valve and fill the copper tube with Argon gas.
7. Open the valve of the mechanical pump to pump down the gas line.

8. Repeat the steps 5-6 three times to purge the copper line.
9. Carefully pressurize the copper line to deliver Argon gas.
10. Slowly open the leak valve to leak Argon gas into the UHV chamber and monitor the pressure in the UHV system.
11. After use, close the leak valve to the UHV system.
12. Close the valve on the regulator.
13. Close the main valve of the Argon cylinder.
14. Open the valve of the pump to evacuate the line.

**Calcination of Catalyst in $H_2$ with Argon pretreatment**

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

**Catalytic Hydrogenation Reaction**

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Place the catalyst in the quartz reactor. About 5 mg of the catalyst is placed in the reactor between some layers of quartz wool above the metal disk placed in the reactor.
3. The reactor is placed inside the heating jacket and connected carefully with the ultratorr connectors to the sampling line. The valves are open and the system is pump. The pressure should be below 60 mTorr. If not, check connections or disconnect the reactor and connect it again. Be careful not to break it.
4. Dry the catalyst, which is PtCu alloy supported by SBA-15 at 150 $^oC$ for 1 h.
5. Activate the catalyst via reduction cycles at a 200 $^oC$ for 2 h.

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

**Oxidation-Reduction Pretreatment of Catalyst**

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

**Reactor #4, ALD Films**

1. Safely secure Argon cylinder using a chain clamp.
2. Ensure the cylinder valve is completely closed.

3. Attach the appropriate pressure regulator to the cylinder and connect it to the system using copper steel tubing.
4. Carefully adjust the outlet pressure to about 15 psi.
5. Close the angle valve next to the mechanical pump.
6. Fill the copper tube with argon gas.
7. Open the angle valve to pump down.
8. Repeat the steps 5-6 three times to purge the copper line.
9. Carefully pressurize copper line.
10. Slowly open the leak valve to leak the gas into the vacuum system, and monitor the pressure in the system
11. Close the leak valve.
12. Close the valve on the regulator.
13. Close the main valve of the argon cylinder.
14. Open the angle valve to pump the line.

**NiO deposition on SBA-15 via ALD reactor**

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

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

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, **AND** a proper face mask at all times (Covid-19) while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
2. Clean the sample holder with acetone
3. Place SBA-15 in the sample holder.
4. Preheat the support (SBA-15) at 200 ℃ for 2 h.

5. Ni precursor (nickel(II) bis(2,2,6,6-tetramethyl-3,5-heptanedionate) is pretreated at 165 ℃ in an oil bath.
6. The reactor is set to 150 ℃.
7. Dose N,N-dimethyltrimethylsilylamine (TMSDMA, 50 mTorr), hexamethyl-disilazane (HMDS, 50 mTorr), or octadecyltrichlorosilane (ODTS, 50 mTorr) for 30 s.
8. Dose the Ni precursor for 30 min by using Ar carrier gas (200 mTorr).
9. Purge the reactor with Ar gas (500 mTorr) for 5 min.
10. Dose deionized water (100 mTorr) for 2 min.
11. Purge the reactor with Ar gas (500 mTorr) for 10 min.
12. Repeat 8–11 steps repeatedly until desired growth is obtained.

**GC #1 Agilent-Batch Reactor: Catalyst Cleaning**

1. Wear nitrile chemical–resistant gloves, a flame-resistant lab coat, and a safety goggle.
2. Clean the catalyst by introducing around 400-500 torr of Argon gas to the reactor loop at room temperature for 1 hour.
   a. Turn on the recirculation pump to circulate the argon gas
   b. After 1 hour turn off the recirculation pump
   c. Initiate vacuum in the system using the mechanical pump to remove the argon gas

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

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

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

8. Slowly open the valve connecting the gas manifold to the rector loop while constantly checking the baratron pressure gauge. Make sure to add 50 torr of $H_2$ to the reactor loop.
9. After adding the desired amount of gas to the loop, close the valve that connects the manifold to the reactor loop and check that the pressure of the reactor loop remains stable
10. Evacuate the gas manifold by opening the valve to the pump for 30 minutes
11. Slowly open the valve for Argon and close the valve after a few seconds
12. Slowly open the valve connecting the gas manifold to the reactor loop while constantly checking the baratron pressure gauge. Make sure to add 545 torr of Argon to the reactor loop.
13. After adding the desired amount of gas to the loop, close the valve that connects the manifold to the reactor loop and check that the pressure of the reactor loop remains stable
14. Evacuate the gas manifold by opening the valve to the pump for 30 minutes, then close the valve.
15. After introducing all the reactant gasses circulate the reactant gas mixture for 20 minutes by turning on the circulation pump.
16. Close the valve at the front of the bypass line, and leave open the valve in the back.
17. Open the valves to the reactor tube, now the reactant mixture will contact the $Pt/Al_2O_3$ catalyst.

**Synthesis of Au NPs on APTES-grafted P25 titania**

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Dry 2 flasks (250 mL) under nitrogen.
3. Put deionized water (100 mL) into the flask.
4. Add 3-aminopropyltriethoxysilane (APTES)-grafted P25 titania nanoparticles (1 g) into the flask
5. After the supports have dispersed evenly, add a solution of tetrachloroauric acid (15 mL, 10 mM).
6. Maintain suspension by stirring the mixture for 2 h at room temperature. If left unattended in a fume hood, put a label with the chemical name and hazard information.
7. Filter the solid from the mixture, and wash twice with deionized water
8. Put deionized water (100 mL) into the other flask.
9. Redisperse the solid into the flask.
10. For the reduction reaction, add sodium borohydride (2.5 g) into the flask.
11. Filter and wash the solid with deionized water.
12. Dispose waste in the properly labeled container.

13. Collect the sample and dry it in an inert atmosphere (i.e. nitrogen, vacuum) at 60°C overnight. If left unattended, put a label with the chemical name and hazard information.
14. Calcinate the sample for 5 h at 450°C in oxygen.
15. Purge with Ar gas for 10 min.
16. Purge with hydrogen at 350 °C for 1 h.
17. After cooling, collect the sample.

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SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 01/04/2014, 03/01/2016, 11/01/2016, 11/28/2019, 10/12/2021, 04/09/2022

# Benzaldehyde
# 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 benzaldehyde ($C_7H_6O$, CAS No. 100-52-7) is used in laboratory. Its purpose is not to have any accident or risk. Benzaldehyde has target organ effect (central nervous system, liver and kidney), and is harmful by ingestion and skin absorption.

Synonyms: Artificial essential oil of almond

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

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

OSHA Hazards: Target Organ Effect, Harmful by ingestion and skin absorption. Skin and respiratory sensitizer, Irritant.

GHS Classification

Flammable liquids (Category 4)
Acute toxicity, Oral (Category 4)
Acute toxicity, Dermal (Category 4)
Skin irritation (Category 2)
Eye irritation (Category 2B)
Respiratory sensitization (Category 1)
Skin sensitization (Category 1)
Acute aquatic toxicity (Category 2)

Signs and Symptoms of Exposure

Central nervous system depression, Prolonged or repeated exposure to skin causes defatting and dermatitis.

## 3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

### a. Eye Protection

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

### b. Skin and Body Protection

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

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

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

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

**c. Hand Protection**

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

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

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

## 4. ENGINEERING/VENTILATION CONTROLS

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

## *5.* SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

## 6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

## 7. DECONTAMINATION

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

## 8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

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

## 9. PRIOR APPROVAL/REVIEW REQUIRED

All work with benzaldehyde 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 benzaldehyde.

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

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

**Henry reaction**

1. Wear nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggles.
2. Weight 0.1 g of benzaldehyde.
3. Bring the reagent into the fume hood and add it into a tube with septum stopper for reaction.
4. After reaction, the removed solvent needs to be treated as hazardous waste.
5. Washing and cleaning solvents also need to be treated as hazardous waste.

**Oxidation reaction**

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

**Oxidation reaction**

1. Wear nitrile chemical–resistant gloves, mask, flame-resistant lab coat, and safety goggles.
2. In the fume hood, add of aluminum isopropoxide (100 μmol) to a round-bottom flask under $N_2$ atmosphere.
3. Add toluene (5 mL).

4. Add benzaldehyde (500 μmol).
5. Stir for 3 h.
6. Take NMR.

**Catalytic Oxidation Reaction**

1. Wear nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggles.
2. Into a test tube with a stir bar, add L-proline (16.4 mg) and acetonitrile (1 mL).
3. Ultrasonicate and stir the mixture.
4. Add tert-butanol (5 μL).
5. Add benzaldehyde (80 μL) and acetophenone (92 μL).
6. Close with a rubber septum, seal with parafilm, connect the oxygen supply to the test tube, and set the temperature at 50 °C.
7. After reaction, collect the samples.
8. Run GC analysis.

**Catalytic Reaction**

1. Wear nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggles.
2. Into a round-bottom flask (20 mL), add Au catalyst (1 wt.%), biphenyl (0.1 *m*mol, internal standard), 2'-hydroxyacetophenone (0.5 *m*mol), benzaldehyde (0.5 *m*mol), and mesitylene (2 mL) by using syringes.
3. Stir the mixture at 130 °C under open air (1 atm.).
4. After injection, clean syringes by thoroughly rinsing with ether. Dispose washing in appropriate wastes containers
5. After reaction is finished, store the reaction mixture in a appropriate labeled vial.

**Coupling catalytic reaction**

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 benzyl 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-$TiO_2$-APTES catalyst (50 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 benzaldehyde from the flammable cabinet and put it into the fume hood. Add the reactant (5 µL) into the reaction mixture.
10. Take acetone from the flammable cabinet and put it into the fume hood. Add the reactant (0.5 mL) into the reaction mixture.
11. Close the test tube with the rubber septum, seal with Teflon tape and connect the oxygen supply to the test tube.
12. 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.
13. 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).
14. 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.
15. After centrifuging bring the closed vial back to the fume hood and transfer the liquid into a new vial.
16. Inject the sample into GC using a Hamilton syringe (10 µL).
17. Dispose all the waste into the appropriately labeled waste bottle.

**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) were added to $CH_2Cl_2$ (4 mL).
3. Bring the reagent 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.

**Catalytic Reaction**

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Methyl acrylate (1.5 mmol), benzaldehyde (1.5 mmol) and catalyst (50 mg) are added to $CH_2Cl_2$, (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.

**Catalytic Reaction**

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

**Addition Reaction with Cinchonidine (Cd)**

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

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SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 03/01/2014, 06/06/2015, 03/03/2016, 05/15/2016, 01/02/2019, 07/01/2019, 10/24/2019, 10/01/2022

# Benzotriazole
# 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 benzotriazole ($C_6H_5N_3$, CAS No. 95-14-7) used in laboratory. Its purpose is not to have any accident or risk. Benzotriazole is toxic if swallowed. Also it is harmful if inhaled and causes serious eye irritation.

Synonyms: 1,2,3-Benzotriazole, 1H-Benzotriazole

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

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

OSHA Hazards: Toxic by Ingestion
GHS Classification
- Acute toxicity, Oral (Category 4)
- Acute toxicity, Inhalation (Category 4)
- Eye irritation (Category 2A)
- Acute aquatic toxicity (Category 3)

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

## 3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

### a. Eye Protection

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

### b. Skin and Body Protection

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

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

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

**c. Hand Protection**

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

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

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

## 4. ENGINEERING/VENTILATION CONTROLS

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

## *5.* SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

## 6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

## 7. DECONTAMINATION

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

## 8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

## 9. PRIOR APPROVAL/REVIEW REQUIRED

All work with benzotriazole 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 benzotriazole.

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

Lab workers using benzotriazole 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 benzotriazole 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 g of this benzotriazole in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this benzotriazole 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 benzotriazole. 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 1,1'-(1,2-Dioxoethane-1,2-diyl)bis-1*H*-benzotriazole**

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Dry a 500 mL flask and a dropping funnel under nitrogen.
3. Put benzotriazole (11.9 g, 100 mmol) into the flask.
4. Add ether (400 mL) into the flask.
5. Put toluene (40 mL and oxalyl chloride (6.35 g, 50 mmol) into the dropping funnel.
6. Drop the oxalyl chloride solution slowly into the flask.
7. Stir the mixture for 20 h at room temperature. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
8. Filter and wash the mixture with ether.
9. Dry the white powder.

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SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2015

# Benzyl 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 benzyl alcohol ($C_7H_8O$, CAS No. 100-51-6) used in laboratory. Its purpose is not to have any accident or risk. Benzyl alcohol is toxic if swallowed. Also it is harmful if inhaled.

Synonyms: Benzenmethanol

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

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

OSHA Hazards: Toxic by Ingestion, Irritant, Carcinogen, Harmful by skin absorption
GHS Classification

Acute toxicity, Oral (Category 4)
Acute toxicity, Inhalation (Category 4)
Acute toxicity, Dermal (Category 4)
Skin irritation (Category 2)
Acute aquatic toxicity (Category 2)

Signs and Symptoms of Exposure
Central nervous system depression, Liver - Irregularities - Based on Human Evidence

## 3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

### a. Respiratory Protection

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

### b. Eye Protection

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

**c. Skin and Body Protection**

Wear chemical resistant lab coat, long pants, and closed-toe shoes. Complete suit protecting against chemicals. The type of protection equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

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

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

**d. Hand Protection**

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

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

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

## 4. ENGINEERING/VENTILATION CONTROLS

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

## *5.* SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

## 6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

## 7. DECONTAMINATION

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

## 8. WASTE DISPOSAL

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

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

Lab workers using benzyl 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 benzyl 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 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;
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4) employ < 5 g of this benzyl alcohol in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this benzyl 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 benzyl 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.

**Oxidation to aldehyde**

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

**Oxidation of benzyl 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 benzyl 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 benzyl alcohol from the flammable cabinet and put it into the fume hood. Add the reactant (5.5 µL) into the reaction mixture.
10. Close the test tube with the rubber septum, seal with Teflon tape and connect the oxygen supply to the test tube.
11. Open the main valve of oxygen cylinder, which is located in a cupboard in a corridor in front of the room 135. After then, open the oxygen Swagelok needle valve in the fume hood, and fill the balloon with oxygen. Attach the balloon to a needle going through the rubber septum into the reaction mixture.
12. Transfer the test tube into the oil bath and do the catalytic reaction at temperature below 75 °C (boiling point of benzene is 80.1 °C).
13. Collect samples at different reaction times and remember to always put on the full-face respirator before working with the mixture. Put a sample (100 µL) into a small centrifuge tube and centrifuge it to remove the solids.

14. After centrifuging bring the closed vial back to the fume hood and transfer the liquid into a new vial.
15. Inject the sample into GC using a Hamilton syringe (10 µL).
16. Dispose all the waste into the appropriately labeled waste bottle.

**Catalytic Reaction**

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

**Hydrogenation of Cinnamaldehyde**

1. Wear a flammable-resistant lab coat, safety goggles and nitrile chemical-resistant gloves.
2. The catalyst is pre-treated under $H_2$ at 350 $^oC$ for 3 h before use.
3. The catalyst (20 mg) is mixed with trans-Cinnamaldehyde (0.8 g), benzyl alcohol (2 mL) and 2-propanol (75 mL).
4. The reaction begins at 100 $^oC$ under $H_2$ (3 MPa) with stirring.
5. The reaction is stopped after an appropriated time.

**Oxidation of Benzyl alcohol**

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. The samples are used without any pretreatment

3. Au/SBA-15 sample (20 mg) is mixed with toluene (20 mL), benzyl alcohol (1 mL) and dodecane (1 mL) in the Parr reactor. Toluene is measured out by a graduated cylinder, while benzyl alcohol and dodecane are measured out by a pipette.
4. The Parr reactor is purged by $O_2$ for 10 min.
5. The reaction begins at 100 $^oC$ and 1 atm $O_2$ pressure with stirring.
6. The reaction is stopped after appropriate time.
7. Liquid samples are filtered and stored in vials.
8. The products are analyzed by GC.

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SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 10/01/2014, updated 03/11/2016, 05/15/2016, 11/15/2016, 10/22/2018, 02/06/2020

# Benzyl 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 benzyl chloride ($C_7H_7Cl$, CAS No. 100-44-7) used in laboratory. Its purpose is not to have any accident or risk. Benzyl chloride 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: α-Chlorotoluene

## 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 (Respiratory system)
GHS Classification

Flammable liquids (Category 4)
Acute toxicity, Oral (Category 4)
Acute toxicity, Inhalation (Category 1)
Skin irritation (Category 2)
Serious eye damage (Category 1)
Skin sensitization (Category 1)
Germ cell mutagenicity (Category 1B)
Carcinogenicity (Category 1B)
Specific target organ toxicity – single exposure (Category 3)

Signs and Symptoms of Exposure
Burning sensation, Cough, wheezing, laryngitis, Shortness of breath, Headache, Nausea, Vomiting,

## 3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

### a. Eye Protection

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

**b. Skin and Body Protection**

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

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

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

**c. Hand Protection**

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

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

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

## 4. ENGINEERING/VENTILATION CONTROLS

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

## *5.* SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

## 6. SPILL AND INCIDENT PROCEDURES

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

Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit

or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

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

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

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

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

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

## 7. DECONTAMINATION

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

## 8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

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

## 9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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).

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2020

# Benzylamine
# 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 benzylamine ($C_7H_9N$, CAS No. 100-46-9) used in laboratory. Its purpose is not to have any accident or risk. Benzylamine 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.

Synonyms: α-Aminotoluene

## 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, Skin burns, Eye damage
GHS Classification

Flammable liquids (Category 4)
Acute toxicity, Oral (Category 4)
Acute toxicity, Dermal (Category 4)
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 benzylamine 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 benzylamine.

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

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

When working in the lab, a laboratory worker must:

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

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

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SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 06/01/2015

# Bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel
# 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 bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel ($C_{22}H_{38}NiO_4$ CAS No. 14481-08-4) is used in laboratory. Its purpose is not to have any accident or risk. Bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel is hygroscopic.

## 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 classified according to GHS

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 bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel 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 bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel.

## 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 bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel and understand the hazards.

Lab workers using bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel 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 bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel 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 < 1 g of this bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel 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 bis(2,2,6,6-tetramethyl-3,5-heptanedionato) nickel. 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.

**UHV #3, Michelle**

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Transfer bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel into the glass-metal container and connect it with Swagelok valve in glove box.
3. Connect it to the leak valve with the Michelle UHV chamber.
4. Open the roughing valve and the Swagelok valve to pump away gases inside the glass-metal sample container and close the valves.
5. Heat the silicon oil bath to 100 °C and leave for 10 min.
6. Rotate and move the sample to face leak valve.
7. Dose the precursor onto the substrate ($SiO_2$/Ta) using a leak valve into the chamber at $1x10^{-6}$ torr to reach a desired coverage.

**Growing $SiO_2$ film on Ta substrate.**

1. Connect the two heating power supply cables to the feedthrough on the $SiO_2$ source. Make sure the connections are good and the wire is not touched each other.
2. Move a sample right in front of the $SiO_2$ source.
3. Leak oxygen ($1x10^{-5}$ torr) inside.
4. Turn the current and voltage knobs counterclockwise on the power supply to minimum.
5. Tune on the power for the HP power supply. Slowly increase the voltage by increasing the coarse settings. Monitor the chamber pressure. Tune both voltage (10 V) and current (10–15 A) knobs. The filament inside the chamber will become red and the temperature of the sample will slowly increase.

6. After the experiment, reduce both current and voltage back to 0.
7. Remove the heating cable from the feedthrough.

**ALD Operation**

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel(II) (1 g) into a glass sample tube (2 mL) in a fume hood.
3. Fix the tube on the homemade ALD reactor in room 135.
4. Heat the tube to 120 °C to increase evaporation.
5. Open the valve to introduce bis(2,2,6,6-tetramethyl-3,5-heptanedionato)nickel(II) into the ALD chamber for 30 s, and then purge with N2 for 120 s.
6. After reaction, close the valve and keep the residual reagent in glass tube for the next reaction.
7. Washing and cleaning solvents also need to be treated as hazardous waste.

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SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 03/01/2019, updated 11/20/2019