

SOP for Chemicals (F to N)

Ferrous sulfate heptahydrate
Fluorene
Formic acid
Germanium oxide
Glycerol
Glycolic acid
Gold chloride trihydrate
Helium
Hexadecyltrimethylammonium bromide
Hexadecyltrimethylammonium chloride
Hexamethyldisilazane
Hexane
Hydrochloric acid
Hydrogen
Hydrogen fluoride
Hydroxylamine hydrochloride
Hydroxypropyl cellulose
Isopropyl alcohol
Lactic acid solution
Lithium aluminum deuteride
Lithium aluminum hydride
Magnesium sulfate
Mesitylene
Methanol
Methyl bromoacetate
Methylcyclopentadienyl manganese tricarbonyl
Methylene chloride
Molecular sieves
Molybdenum sulfide
Neon
Nitrogen
Nitrogen liquid
Nitromethane
Nitrous oxide

Ferrous sulfate heptahydrate 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 ferrous sulfate heptahydrate ($\text{FeO}_4\text{S} \cdot 7\text{H}_2\text{O}$, CAS No. 7782-63-0) is used in laboratory. Its purpose is not to have any accident or risk. Ferrous sulfate heptahydrate causes skin and eye irritation. It may be harmful if inhaled or if swallowed.

Synonyms: Iron (II) sulfate heptahydrate

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

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

OSHA Hazards: Irritant, Target Organ (Gastro-intestinal system, Liver), Harmful by ingestion

GHS Classification

Acute toxicity, Oral (Category 4)

Skin irritation (Category 2)

Eye irritation (Category 2A)

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 ferrous sulfate heptahydrate 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 ferrous sulfate heptahydrate.

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

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

When working in the lab, a laboratory worker must:

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

- 4) employ < 25 g of this ferrous sulfate heptahydrate in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this ferrous sulfate heptahydrate 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 ferrous sulfate heptahydrate. 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.

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

Approval Date: 06/01/2013

Fluorene

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 fluorene (C₁₃H₁₀, CAS No. 86-73-7) is used in laboratory. Its purpose is not to have any accident or risk. Fluorene causes eye, skin, and respiratory irritations. Also, it may be harmful by inhaled, absorbed through skin, or swallowed.

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

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

OSHA Hazards: not known

GHS Classification

Acute aquatic toxicity (Category 1)

Chronic aquatic toxicity (Category 1)

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with fluorene 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 fluorene.

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

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

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) employ < 50 g of this fluorene in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this fluorene 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 fluorene. 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 of fluorene

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 fluorene 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-TiO₂/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 fluorene 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.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 10/15/2015, updated 05/15/2016

Formic acid

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when formic acid (CH₂O₂, CAS No. 64-18-6) is used in laboratory. Its purpose is not to have any accident or risk. Formic acid is combustible liquid and corrosive. It is toxic if inhaled or swallowed. Also it causes severe skin burns and eye damage.

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

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

OSHA Hazards: Combustible Liquid, Target Organ Effect (Blood, Central nervous system, Kidney), Harmful by Ingestion, Corrosive

GHS Classification

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

Signs and Symptoms of Exposure

Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Spasm, inflammation and edema of the larynx or of the bronchi, Pneumonitis, Pulmonary edema, Burning sensation, Cough, wheezing, laryngitis, Shortness of breath, Headache, Nausea, Vomiting

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

Lab workers using formic acid must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenk 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 formic acid described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale- of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

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

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

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

HPLC Sample Preparation

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Bring formic acid bottle securely to the surface in the fume hood.
3. Take designated amount of formic acid solution into the designated vessel.
4. Close and seal the bottle and put it back to the cabinet.
5. Dilute formic acid using designated solvent.
6. Extra formic acid and used sample solution should be treated as hazardous waste.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 02/01/2014

Germanium(IV) oxide

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 germanium(IV) oxide (GeO₂, CAS No. 1210-53-8) is used in laboratory. Its purpose is not to have any accident or risk. Germanium oxide is toxic by inhalation and harmful by ingestion, so kidney and liver injury may occur.

Synonyms: Germanium dioxide

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, Toxic by Inhalation, Harmful by Ingestion.

GHS Classification

Acute toxicity, Inhalation (Category 4)

Acute toxicity, Oral (Category 4)

Signs and Symptoms of Exposure

Prolonged or repeated exposure can cause Kidney injury, Liver injury, Blood disorders, Electrolyte imbalance, or Neurotoxic effects.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with germanium(IV) oxide 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 germanium(IV) oxide.

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 germanium(IV) oxide must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of germanium(IV) oxide and understand the hazards.

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

- 5) discuss ALL issues or concerns regarding this germanium(IV) oxide 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 germanium(IV) oxide. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Preparation of chiral zeolite

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Take 0.08 g of germanium oxide in a 25 mL Teflon-lined autoclave, in the fume hood.
3. Add other reagents into the autoclave, and the reaction is conducted at 140 °C.
4. After reaction, the filtrate needs to be treated as hazardous waste.
5. Washing and cleaning solvents also need to be treated as hazardous waste.

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

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

Glycerol

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 glycerol (C₃H₈O₃, CAS No. 56-81-5) is used in laboratory. Its purpose is not to have any accident or risk. Glycerol causes skin and eye irritation.

Synonyms: 1,2,3-Propanetriol, Glycerin

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 (Kidney)

GHS Classification

Skin irritation (Category 3)

Eye irritation (Category 2B)

Signs and Symptoms of Exposure

Prolonged or repeated exposure may cause Nausea, Headache, Vomiting

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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 glycerol 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 glycerol.

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

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

Glycerol Oxidation Reaction

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Bring glycerol bottle to the balance. Transfer glycerol into the designated vessel by disposable pipet.
3. Close and seal the bottle and put it back to the cabinet.
4. Add other required reagents into the vessel and assemble the reaction system.

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

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

Glycolic acid

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when glycolic acid (C₂H₄O₃, CAS No. 79-14-1) is used in laboratory. Its purpose is not to have any accident or risk. Glycolic acid is corrosive and harmful if swallowed and if inhaled. It causes severe skin burns and eye damages.

Synonyms: Hydroxyacetic acid

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

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

OSHA Hazards: Corrosive, Harmful by Ingestion

GHS Classification

Acute toxicity, Oral (Category 4)

Acute toxicity, Inhalation (Category 5)

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

Lab workers using glycolic acid must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenk 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 glycolic acid described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

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

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

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

HPLC Sample Preparation

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take glycolic acid out of the fridge and bring the bottle to the balance.
3. Transfer glycolic acid into the designated vessel.
4. Close and seal the bottle and put it back to the fridge.
5. Use designated solvent to dissolve glycolic acid and the sample solution needs to be treated as hazardous waste after measurement.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

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

Gold chloride trihydrate STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when gold chloride trihydrate ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$, CAS No. 16961-25-4) is used in laboratory. Its purpose is not to have any accident or risk. Gold chloride trihydrate is corrosive and causes skin burns and eye damage. It may cause sensitization by skin contact.

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

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

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

OSHA Hazards: Corrosive, Skin sensitizer

GHS Classification

- Skin corrosion (Category 1A)
- Serious eye damage (Category 1)
- Skin sensitization (Category 1)

Signs and Symptoms of Exposure

No data is available.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for gold chloride trihydrate.

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

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

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

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

Gold nanoparticle preparation 1

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

Gold nanoparticle preparation 2

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

Au Nanoparticle (2 nm) Preparation

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

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

Au Nanoparticle (3 nm) Preparation

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

Preparation of gold nanoparticles

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

(hydroxymethyl)phosphonium chloride (96 μ L) into it. Stir and add the mixture to the round bottom flask.

8. Stir the mixture in the closed round-bottom flask for 2 minutes. Then, add the mixture of gold chloride trihydrate and water and stir for another 2 minutes.
9. Save the gold solution in appropriately labeled centrifuge tubes.
10. When using the gold solution, **dispose the waste into the waste bottle labeled toxic and corrosive hazardous waste.**

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

Approval Date: 02/01/2013, updated 10/01/2014, 03/02/2016, 05/15/2016

Helium

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when helium gas (He, CAS No. 7440-59-7) used in laboratory. Its purpose is not to have any accident or risk. Helium contains gas under pressure. It may be harmful if swallowed, if inhaled or if absorbed through skin. Also it may cause skin and eye irritation.

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

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

OSHA Hazards: Compressed Gas

GHS Classification

Gases under pressure (Compressed Gas)

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

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

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

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

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

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

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

STORAGE:

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

7. WASTE DISPOSAL

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

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

8. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

9. DESIGNATED AREA

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

10. SAFETY DATA SHEETS

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

11. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

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

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

Replace empty gas cylinder

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

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

Replacing empty gas cylinder for BET Instrument

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

UHV #3, Michelle

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

12. Open the angle valve to pump the line.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

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

Hexadecyltrimethylammonium bromide

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 hexadecyltrimethylammonium bromide (C₁₉H₄₂BrN, CAS No. 57-09-0) is used in laboratory. Its purpose is not to have any accident or risk. Hexadecyltrimethylammonium bromide causes skin and eye irritation. It may be harmful if inhaled or if swallowed.

Synonyms: CTAB, Cetrimonium bromide, Palmityltrimethylammonium bromide
Cetyltrimethylammonium bromide

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

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

OSHA Hazards: Toxic by ingestion, Irritant

GHS Classification

Acute toxicity, Oral (Category 4)

Skin irritation (Category 2)

Serious eye damage (Category 1)

Specific Target organ toxicity – single exposure (Category 3)

Acute aquatic toxicity (Category 1)

Chronic aquatic toxicity (Category 1)

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with hexadecyltrimethylammonium bromide 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 hexadecyltrimethylammonium bromide.

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

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

When working in the lab, a laboratory worker must:

- 1) not work alone;

- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) employ < 25 g of this hexadecyltrimethylammonium bromide in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this hexadecyltrimethylammonium bromide 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 hexadecyltrimethylammonium bromide. 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.

Au Nanoparticle (3 nm) Preparation

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

SOP Reviewed and Approved by:

 Francisco Zaera
 Print name

 Signature

Approval Date: 06/01/2013, updated 03/18/2016

Hexadecyltrimethylammonium 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 hexadecyltrimethylammonium chloride (C₁₉H₄₂ClN, CAS No. 112-02-7) is used in laboratory. Its purpose is not to have any accident or risk. Hexadecyltrimethylammonium chloride causes skin and eye irritation. It may be harmful if inhaled or if swallowed.

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

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

OSHA Hazards: Irritant

GHS Classification

Acute toxicity, Dermal (Category 5)

Skin irritation (Category 2)

Serious eye damage (Category 1)

Acute aquatic toxicity (Category 1)

Signs and Symptoms of Exposure

N/A

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

Lab workers using hexadecyltrimethylammonium 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 hexadecyltrimethylammonium 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 factor) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) employ < 25 g of this hexadecyltrimethylammonium chloride in any given reaction (larger quantities REQUIRE the approval of PI or designee), and

- 5) discuss ALL issues or concerns regarding this hexadecyltrimethylammonium 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 hexadecyltrimethylammonium 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.

Au Nanoparticle (3 nm) Preparation

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2013, updated 02/15/2015

Hexamethyldisilazane

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when hexamethyldisilazane (C₆H₁₉NSi₂, CAS No. 999-97-3) used in laboratory. Its purpose is not to have any accident or risk. Hexamethyldisilazane is highly flammable liquid and vapor, and toxic in contact with skin. It causes severe skin burns and eye damage. Also it is harmful if swallowed or if inhaled.

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

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

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

OSHA Hazards: Flammable liquid, Target organ effect (Nerves), Corrosive, Toxic by skin absorption, Harmful by Ingestion, Reproductive hazard.

GHS Classification

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

Signs and Symptoms of Exposure

Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin., Spasm, inflammation and edema of the larynx or the bronchi, Pneumonitis, Pulmonary edema, Burning sensation, Cough, wheezing, laryngitis, Shortness of breath, Headache, Nausea, Vomiting

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: <http://ehs.ucr.edu/services/waste.html>

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) employ < 50 mL of this hexamethyldisilazane in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this hexamethyldisilazane with the PI prior to its use.

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

Silylation of Oxide

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

Silylation of Si wafer

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

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

Hexane

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when hexane (C₂H₁₄, CAS No. 110-54-3) used in laboratory. Its purpose is not to have any accident or risk. Hexane is highly flammable liquid. Vapor may cause drowsiness and dizziness. It also causes eye, skin, and respiratory tract irritations. It may be harmful if swallowed, if inhaled, or if adsorbed through skin.

Synonyms: n-Hexane

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

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

OSHA Hazards: Flammable liquid, Target Organ (Peripheral nervous system, Kidney, Testes) Effect, Irritant

GHS Classification

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

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: <http://ehs.ucr.edu/services/waste.html>

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

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

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

For washing solvent

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

4-Methyl-2,6-heptanedione

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Add sodium (1.8 g, 50 mmol) and ethanol (24 mL) into a flask (100 mL).
3. Add 2,4,6-trimethylpyridine (6.5 mL, 50 mmol) into the flask.
4. Reflux the mixture for 1 h at 90 °C in oil bath.
5. Prepare a solution of hydroxylamine hydrochloride (3.6 g, 53 mmol) in 50% ethanol (6.4 mL) and HCl (3.2 mL) in 95% ethanol (6.4 mL).
6. Add the solution slowly into the flask.
7. Reflux the mixture for 2.5 h.
8. Cool down the mixture to room temperature.
9. Remove ethanol from the mixture under evaporator.
10. Add a NaOH solution (3.5 g in 50 mL water) to the residue.
11. Extract the solution with ether (50 mL).
12. Acidify the aqueous solution with 10% H₂SO₄ (35 mL).
13. Add sodium nitrite solution (3.5 g, 50 mmol in 10 mL water) into the mixture.

14. Stir the mixture for 1 h at 0 °C.
15. Extract the mixture with ether (20 mL, 4 times)
16. Wash the organic phase with water and brine.
17. Column the residue with a hexane solution (hexane:ether = 10:2).

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2013, updated 03/01/2014, 03/01/2016

Hydrochloric acid

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when hydrochloric acid (HCl, CAS No. 7647-01-0) is used in laboratory. Its purpose is not to have any accident or risk. Hydrochloric acid is corrosive liquid. It causes severe skin burns and eye damage. Also, It may be harmful if inhaled, if absorbed through skin, or if swallowed.

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

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

OSHA Hazards: Corrosive

GHS Classification

Skin corrosion (Category 1B)

Serious eye damage (Category 1)

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

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

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

Si wafer washing

1. Wear nitrile chemical-resistant gloves, water-resistant lab coat, and safety goggles.
2. Bring the HCl and H₂O₂ solution bottles to a fume hood.
3. Mix HCl, H₂O₂ and H₂O in the volume ratio of 1:1:6, and heat the mixture up to 70 °C. Then load Si wafer into the solution for 10 min.
4. (4)Take the wafer out and wash with DI water for 5mins and blow dry with N₂ gas
5. After treatment, the solution needs to be treated as hazardous

Preparation for pH controlling agent

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

4-Methyl-2,6-heptanedione

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Add sodium (1.8 g, 50 mmol) and ethanol (24 mL) into a flask (100 mL).
3. Add 2,4,6-trimethylpyridine (6.5 mL, 50 mmol) into the flask.
4. Reflux the mixture for 1 h at 90 °C in oil bath.
5. Prepare a solution of hydroxylamine hydrochloride (3.6 g, 53 mmol) in 50% ethanol (6.4 mL) and HCl (3.2 mL) in 95% ethanol (6.4 mL).
6. Add the solution slowly into the flask.
7. Reflux the mixture for 2.5 h.
8. Cool down the mixture to room temperature.
9. Remove ethanol from the mixture under evaporator.
10. Add a NaOH solution (3.5 g in 50 mL water) to the residue.
11. Extract the solution with ether (50 mL).

12. Acidify the aqueous solution with 10% H₂SO₄ (35 mL).
13. Add sodium nitrite solution (3.5 g, 50 mmol in 10 mL water) into the mixture.
14. Stir the mixture for 1 h at 0 °C.
15. Extract the mixture with ether (20 mL, 4 times)
16. Wash the organic phase with water and brine.
17. Column the residue with a hexane solution (hexane:ether = 10:2).

Reaction

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and a safety goggles.
2. Pluronic[®] F-127 (1.0 g) is dissolved in HCl (2 M, 60 mL), KCl (2.5 g), and 1,3,5-trimethylbenzene (1.0 g).
3. This is stirred at room temperature for 24 hours.
4. Tetramethyl orthosilicate (4.1 g) is added drop-wise to the mixture and stirred for 24 hours.
5. The solution undergoes hydrothermal treatment at 100 °C for 48 hours, then filtered and rinsed with H₂O.

Catalytic Reaction

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

Catalytic Reaction

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take 4-hydroxybenzyl alcohol (31 mg) into an Erlenmeyer flask (30 mL) in the fume hood of room 135.
3. Add potassium carbonate (103.7 mg) and water (5 mL).
4. Add aqueous Au-PVP catalyst (0.5 mM, 10 ml, 2 atom.%), stir at 1300 rpm.

5. Quench reaction with HCl (1 M), extract with ethyl acetate, dry organic layer over sodium sulfate.
6. Run on GC.

Oxidation to aldehyde

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

Partial etching of titania shells

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Make a waste bottle labeled as toxic and corrosive hazardous waste. Review the SDS of sodium hydroxide and hydrochloric acid again; especially remind first aid measures, handling and storage, & PPE.*
3. Place an Erlenmeyer flask into a fume hood and put a stir bar into it.
4. Disperse titania shells with a silica core in milli-Q water (20 mL) and add the dispersion to the flask.
5. Take sodium hydroxide solution (2.5 M) from a corrosive base cabinet and place it into the fume hood. With a micropipette add sodium hydroxide (1 mL of 2.5 M) and close with the septum. Stir for 20 minutes.
6. Wash three times with water and *dispose the waste into the waste bottle labeled toxic and corrosive hazardous waste.*
7. Disperse partially etched titania shells with a silica core in milli-Q water (10 mL) and add the dispersion to a clean flask.
8. Take hydrochloric acid solution (0.5 M) from inorganic acid cabinet and place it into the fume hood. With a micropipette add hydrochloric acid (2 mL of 0.5 M) and close with the septum. Stir for 30 minutes.
9. Wash three times with water and two times with ethanol. *Dispose the waste into the waste bottle labeled toxic and corrosive hazardous waste.*
10. Label the centrifuge tube appropriately, cover it with perforated aluminum foil and dry the powder in a vacuum desiccator overnight.

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

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

Hydrogen

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when hydrogen (H₂, CAS No. 1333-74-0) used in laboratory. Its purpose is not to have any accident or risk. Hydrogen is highly flammable gas, and contains gas under pressure. It may explode if heated.

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

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

OSHA Hazards: Flammable gas, Compressed Gas

GHS Classification

Flammable gas (Category 1)

Gas under pressure (Liquefied gas)

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

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

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

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

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

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

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

STORAGE:

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

6. SPILL AND INCIDENT PROCEDURES

Emergency procedure for leaking gas cylinders -

<http://www.airproducts.com/~media/Files/PDF/company/safetygram-11.pdf>

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

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

7. WASTE DISPOSAL

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

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

General hazardous waste disposal guidelines:

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

8. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

9. DESIGNATED AREA

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

10. SAFETY DATA SHEETS

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

11. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

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

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

Replace empty gas cylinder

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

Replacing empty gas cylinder for GC Instrument

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

Calcination of Catalyst in H₂ 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 °C) for 30 min.
7. Set the temperature controller to 150 °C.
8. Open the H₂ cylinder valve, switch from Argon to H₂.
9. Close Argon cylinder valve.
10. Adjust the gas flow rate if needed.
11. Set temperature to 350 °C and calcine the sample in H₂ flow for 2 h.
12. Close H₂ cylinder valve.
13. Close all valves to tube furnace.
14. Open vacuum valve, which is connected to the pump.
15. Check pressure gauge (should be around 0.03 torr).

Perform IR spectroscopic Experiment

1. A supported metal catalyst disk (e.g. Pt/SiO₂) is placed in an IR vacuum cell.
2. The catalyst is heated at 150 °C under vacuum for 30 min in order to eliminate the adsorbed water.
3. The catalysts are then heated from 150 °C to 350 °C under 5 torr of H₂.
4. The catalyst is kept at 350 °C under 200 torr of hydrogen gas for 3-4 hours.
5. Hydrogen gas is removed for 15 min.
6. The catalyst is kept at 350 °C under 200 torr of oxygen gas for 3-4 hours.
7. Oxygen gas is removed for 15 min.
8. Repeat 4 to 7 steps three times.
9. The sample is cooled down to room or any desired temperature.
10. Background spectrum is obtained.

11. Carbon monoxide is introduced into the cell up to 10 Torr.
12. Sample spectrum is obtained.
13. Carbon monoxide is pumped out.
14. The IR cell is vented to atmosphere.

UHV #1, Victor

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

UHV #2, RAIRS

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

UHV #3, Michelle

1. Safely secure Hydrogen cylinder using a chain clamp or ring clamps. The Hydrogen cylinder can only be installed at a place that is at least 20 feet away from the Oxygen cylinder in the same room.

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

UHV #4 Praxis

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

UHV #5, UC Chamber

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

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

UHV #6 Nanoreactor

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

SOP Reviewed and Approved by:

 Francisco Zaera
 Print name

 Signature

Approval Date: 02/01/2013, updated 01/01/2015, 03/01/2016

Hydrogen Fluoride

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when hydrogen fluoride (HF, CAS No. 7664-39-3) is used in laboratory. Its purpose is not to have any accident or risk. Hydrogen fluoride is highly toxic. It is fatal if inhaled, if swallowed or in contact with skin. Also, it causes severe skin burns and eye damage.

Synonyms: Hydrofluoric acid

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

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

OSHA Hazards: Target Organ Effect (Liver, Kidney), Highly Toxic by Inhalation and Ingestion, Skin Absorption, Corrosive

GHS Classification

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

Sign and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

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

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

Si wafer washing

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

SOP Reviewed and Approved by:

 Francisco Zaera
 Print name

 Signature

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

Hydroxylamine hydrochloride 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 hydroxylamine hydrochloride ($H_3NO \cdot HCl$, CAS No. 5470-11-1) is used in laboratory. Its purpose is not to have any accident or risk. Hydroxylamine hydrochloride is corrosive solid, and may be corrosive to metals. It causes skin and eye irritation. Also, It may be harmful if swallowed.

Synonyms: Hydroxylammonium chloride

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

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

OSHA Hazards: **Corrosive**

GHS Classification

- Corrosive to metal (Category 1)
- Acute toxicity, Oral (Category 3)
- Acute toxicity, Dermal (Category 4)
- Skin irritation (Category 2)
- Eye irritation (Category 2A)
- Skin sensitization (Category 1)
- Carcinogenicity (Category 2)
- Acute aquatic toxicity (Category 1)
- Chronic aquatic toxicity (Category 1)
- Specific target organ toxicity – repeated exposure (Category 2)

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

- Dispose of routinely generated chemical waste within 90 days.
- Request a waste pick-up on-line: <http://ehs.ucr.edu/services/waste.html>

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with hydroxylamine hydrochloride 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 hydroxylamine hydrochloride.

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

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

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) employ < 25 g of this hydroxylamine hydrochloride in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this hydroxylamine hydrochloride 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 hydroxylamine hydrochloride. 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.

Growth of Au shells

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Hydroxylamine hydrochloride was prepared by dissolving 130 mg in 1 L water (1.87 mM) in a fume hood.
3. To $\text{HAuCl}_4/\text{K}_2\text{CO}_3$ solution (148 mL), 90~500 mL (Depending on thickness needed) hydroxylamine hydrochloride solution prepared in step 2 was added, was added dropwise using a glass pipette.
4. The pipette is copiously rinsed with DI water and the washings were discarded as hazardous aqueous waste.

4-Methyl-2,6-heptanedione

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

7. Reflux the mixture for 2.5 h.
8. Cool down the mixture to room temperature.
9. Remove ethanol from the mixture under evaporator.
10. Add a NaOH solution (3.5 g in 50 mL water) to the residue.
11. Extract the solution with ether (50 mL).
12. Acidify the aqueous solution with 10% H₂SO₄ (35 mL).
13. Add sodium nitrite solution (3.5 g, 50 mmol in 10 mL water) into the mixture.
14. Stir the mixture for 1 h at 0 °C.
15. Extract the mixture with ether (20 mL, 4 times)
16. Wash the organic phase with water and brine.
17. Column the residue with a hexane solution (hexane:ether = 10:2).

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

Approval Date: 10/01/2014, updated 06/01/2015

Hydroxypropyl cellulose

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 hydroxypropyl cellulose (CAS No. 9004-64-2) is used in laboratory. Its purpose is not to have any accident or risk. Hydroxypropyl cellulose may be harmful if inhaled and swallowed. It may cause skin and eye irritation. Hydroxypropyl cellulose is commercially available, and used mostly for nanostructured catalysts in Zaera group. A variety of organic solvents are used to clean sample containers.

Synonyms: Cellulose, 2-hydroxypropyl ether

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

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

OSHA Hazards: Not known

GHS Classification

N/A

Signs and Symptoms of Exposure

No data is available

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with hydroxypropyl cellulose 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 hydroxypropyl cellulose.

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

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

- 5) discuss ALL issues or concerns regarding this hydroxypropyl cellulose 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 hydroxypropyl cellulose. 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.

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.*

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

Approval Date: 02/01/2013, updated 03/01/2014, 10/01/2014, 05/15/2016

Isopropyl alcohol

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when isopropyl alcohol (C₃H₈O, CAS No. 67-63-0) used in laboratory. Its purpose is not to have any accident or risk. Isopropyl alcohol is highly flammable liquid and vapor. It causes serious eye and mild skin irritation, as well as drowsiness or dizziness

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

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

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

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

GHS Classification

Flammable liquids (Category 2)

Skin irritation (Category 3)

Eye irritation (Category 2A)

Specific target organ toxicity - single exposure (Category 3)

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

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

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

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

For washing solvent

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

Functionalization of Silica nanospheres

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Bring out 20 μ L of 3-aminopropyltriethoxysilane in a sealed vial from the glove box.
3. Add to isopropyl alcohol (20 mL).
4. Add the mixture to the prepared silica spheres (dispersed in ethanol).
5. Heat at 80 $^{\circ}$ C for 2 h, then wash the NH_2 functionalized particles in ethanol.
6. Dispose off the washings as hazardous organic waste.

Oxidation of isopropyl alcohol

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

2. *Make a waste bottle labeled as toxic and carcinogen hazardous waste. Review the SDS of isopropyl alcohol and benzene again; especially remind first aid measures, handling and storage, & PPE.*
3. Place a test tube into a fume hood and put a stir bar into it. Close with a rubber septum and take it to a balance. Weigh P25-TiO₂/Au catalyst (9 mg) and add it into the test tube. Add potassium carbonate (25 mg) and transfer the closed septum back to the fume hood.
4. With a micropipette add toluene (4.5 mL), close with the septum and sonicate for about 1 minute so that the solids disperse well in the solvent.
5. Bring the mixture back to the fume hood and stir it.
6. Before adding the internal standard benzene (12.5 μL) *put on a full-face respirator*. Take a bottle of benzene from the flammable cabinet and place it into the fume hood. *Be careful not to spill benzene. Keep watching any leak of benzene. Avoid release to the environment. Avoid breathing fume, gas, mist, vapor or spray. If swallowed, immediately call 911. If inhaled, rinse cautiously with water for 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.*
7. Remove the septum from the test tube and open the benzene bottle. Add the small amount of benzene into the test tube by using a Hamilton syringe (50 μL). Wash the syringe with benzene three times before adding it into the reaction mixture. After adding it clean the syringe by washing it with ether. *Dispose the waste into the waste bottle labeled carcinogen hazardous waste.* Once adding benzene the handling of the reaction mixture has to be carried out with *the full-face respirator on*.
8. Put the benzene bottle back to the flammable cabinet. *Be careful not to spill benzene. Keep watching any leak of benzene. Avoid release to the environment. Avoid breathing fume, gas, mist, vapor or spray. If swallowed, immediately call 911. If inhaled, rinse cautiously with water for 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.*
9. Take the small vial of isopropanol from the flammable cabinet and put it into the fume hood. Add the reactant (5.5 μL) into the reaction mixture.
10. Close the test tube with the rubber septum, seal with Teflon tape and connect the oxygen supply to the test tube.
11. Open the main valve of oxygen cylinder, which is located in a cupboard in a corridor in front of the room 135. After then, open the oxygen Swagelok needle valve in the fume hood, and fill the balloon with oxygen. Attach the balloon to a needle going through the rubber septum into the reaction mixture.
12. Transfer the test tube into the oil bath and do the catalytic reaction at temperature below 75 °C (boiling point of benzene is 80.1 °C).
13. Collect samples at different reaction times and remember to always *put on the full-face respirator* before working with the mixture. Put a sample (100 μL) into a small centrifuge tube and centrifuge it to remove the solids.

14. After centrifuging bring the closed vial back to the fume hood and transfer the liquid into a new vial.
15. Inject the sample into GC using a Hamilton syringe (10 μ L).

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

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

Lactic acid solution 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 lactic acid solution (C₃H₆N₃, CAS No. 50-21-5) is used in laboratory. Its purpose is not to have any accident or risk. Lactic acid solution causes serious eye damage and skin irritation. It may be harmful if swallowed or in contact with skin.

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

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

OSHA Hazards: Irritant

GHS Classification

- Acute toxicity, Oral (Category 5)
- Acute toxicity, Dermal (Category 5)
- Skin irritation\ (Category 2)
- Serious eye damage (Category 1)

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with lactic acid solution 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 lactic acid solution.

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

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

When working in the lab, a laboratory worker must:

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

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Take lactic acid out of the fridge and bring the bottle to the balance.
3. Transfer lactic acid into the designated vessel.
4. Close and seal the bottle and put it back to the fridge.
5. Discard extra lactic acid to designated chemical waste container.
6. Use designated solvent to dissolve lactic acid and the sample solution needs to be treated as hazardous waste after measurement.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

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

Lithium aluminum deuteride

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 lithium aluminum deuteride (D₄AlLi, CAS No. 14128-54-2) is used in laboratory. Workers should not use lithium aluminum deuteride until they have read and fully understood these safe operating procedures. However, reading these procedures does not substitute for hands-on training. New users of lithium aluminum deuteride must work under the close supervision of an experienced user. Lithium aluminum deuteride is corrosive solid. It causes severe skin burns and eye damage. **In contact with water it releases flammable gases, which may ignite spontaneously.** It is extremely destructive to the tissue of the mucous membranes and upper respiratory tract. Also, It may be harmful if inhaled, if absorbed through skin, or if swallowed.

Synonyms: Deuterated LAH

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, Water Reactive, Toxic by ingestion**

GHS Classification

- Substances, which in contact with water, emit flammable gases (Category 1)
- Acute toxicity, Oral (Category 3)
- Skin corrosion (Category 1B)
- Serious eye damage (Category 1)

Signs and Symptoms of Exposure

Burning sensation, Cough, wheezing, laryngitis, Shortness of breath, spasm, inflammation and edema of the larynx, spasm, inflammation and edema of the bronchi, pneumonitis, pulmonary edema, Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin., Large doses of lithium ion have caused dizziness and prostration, and can cause kidney damage if sodium intake is limited. Dehydration, weight loss, dermatological effects, and thyroid disturbances have been reported. Central nervous system effects that include slurred speech, blurred vision, sensory loss, ataxia, and convulsions may occur. Diarrhea, vomiting, and neuromuscular effects such as tremor, clonus, and hyperactive reflexes may occur as a result of repeated exposure to lithium ion. 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

- Chemical splash goggles or safety glasses with side shields that meet the ANSI Z.87.1 1989 standard must be worn whenever handling pyrophoric chemicals. Ordinary prescription glasses will NOT provide adequate protection unless they also meet this standard. When there is the potential for splashes, goggles must be worn, and when appropriate, a face shield added.
- A face shield is required any time there is a risk of explosion, large splash hazard or a highly exothermic reaction. All manipulations of pyrophoric chemicals which pose this risk should occur in a fume hood with the sash in the lowest feasible position. Portable shields, which provide protection to all laboratory occupants, are advisable.

b. Skin and Body Protection

- A flame resistant lab coat must be worn.
- A chemical-resistant apron worn over the lab coat is required for working with large quantities.
- No open toe shoes are allowed.

c. Hand Protection

- Hand protection requirements for the use of solid pyrophoric chemicals outside an inerted glove box include wearing/donning the appropriate chemical resistant out gloves (Neoprene) and fire resistant (FR) inner gloves/liners.
- Gloves or glove liners composed of the tight weave, inherently flame resistant materials Kevlar[®], Nomex[®], Kerinel[®], or PBI[®], or a blend of those materials, of sufficient thickness to prevent or minimize burn injuries to the extent feasible. Gloves or glove liners meeting MIL-DTL-81188C are also acceptable.
- Acceptable glove liners include the followings:
 - ✓ Ansell Kevlar[®] Goldknit[®] Lightweight 70-200,
 - ✓ Hanz Extremity Wear Nomex[®] Utility 2257C and 2259C,
 - ✓ Other Kevlar[®] gloves with the fabric basis weight of a minimum of 7.7 ounces per square yard and if one layer of the material has a minimum of 35 mils in thickness, and
 - ✓ Other Nomex[®] gloves meeting the specifications for Hans Extremity Wear Nomex[®] Utility 2257G and 2259C.
- Laboratories must have these gloves available and use them for this specific research activity. This requirement may not be superseded by reference to a Safety Data Sheet (SDS).

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/PROTOCOLS

Handling Pyrophoric Solid Reagents

- Pyrophoric solids are ideally used in a sealed glove box flushed with inert gas.
- Many pyrophoric solids are sold as solutions, or dispersions in mineral oil or are covered with hydrocarbon solvents to facilitate use.
- Mildly pyrophoric solids (such as lithium aluminum deuteride, hydride and sodium hydride) may be handled in the air for brief periods of time, but the containers must be flushed with inert gas before storage.

Transferring and Weighing Pyrophoric Solid Reagents

- Gather all necessary experimental equipment first to avoid prolonged exposure of pyrophoric solids to air.
- Weighing alkali metals: Cut desired piece of alkali metal under packing oil using a knife. Using tweezers, transfer to adjacent flask containing toluene or heptane to rinse off oil. Use tweezers again to transfer to a weighed flask of toluene and measure weight to determine mass of metal. Use tweezers again to transfer to desired reaction flask.
- AVOID low boiling rinses such as ether and pentane that tend to condense water upon evaporation.

Specific Recommendations for Working with Pyrophoric Solid Reagents

- Lithium Aluminum Hydride reacts violently with water and has a significant heat of solvation. Therefore DO NOT add solvent to dry LiAlD_4 or LiAlH_4 . Instead, slowly add LiAlD_4 or LiAlH_4 to anhydrous solvent in the reaction flask. The initial small amount of LiAlD_4 or LiAlH_4 will react with any trace amounts of water.
- Potassium metal is considerably more reactive than lithium or sodium.
- Potassium metal oxidizes to potassium oxide (K_2O), potassium peroxide (K_2O_2), and potassium superoxide (KO_2). The yellow peroxides are shock-sensitive and can explode when handled or cut. Therefore dispose of potassium metal as hazardous waste if old or if a significant amount of yellow crust is visible.
- The mineral oil of potassium hydride or sodium hydride dispersions can be rinsed off using a light hydrocarbon solvent such as hexane. This is easily accomplished in a glove box or can be done in a hood UNDER CAREFULLY CONTROLLED CONDITIONS. Weigh out desired amount of dispersion and seal in a flask under nitrogen. Add dry hexane via syringe, swirl, and let metal hydride settle. Slowly syringe off hexane and then carefully discard into a separate flask containing isopropanol. Repeat rinse procedure.
- AVOID low boiling rinses such as ether and pentane that tend to condense water upon evaporation.
- Sodium amalgam, Na(Hg) , (or potassium amalgam) is prepared by dissolving sodium into liquid mercury. This highly exothermic process produces the intermetallic compound NaHg_2 with enough heat to cause local boiling of the

mercury. Thus it must be performed in a hood under dry nitrogen gas. The grey solid produced has the reducing potential of sodium, but is more air stable.

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 pyrophoric chemicals under an inert atmosphere or under kerosene as appropriate.

Avoid storage areas with heat/flames, oxidizers, and water sources.

Containers carrying pyrophoric materials must be clearly labeled with the correct chemical name and hazard warning.

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.).

Spill - Large

- Exert extreme caution due to potential spontaneous combustion.
- Exert extreme caution due to potential ignition of flammable solvents or other materials.
- If anyone is exposed, or on fire, wash with copious amounts of water, ideally in the lab shower.
- Call 911 for emergency assistance.
- Evacuate the spill area.
- Post someone or mark-off the hazardous area with tape and warning signs to keep other people from entering.
- Provide emergency personnel with technical advice on the chemicals involved.

Spill - Small

- Exert extreme caution due to potential spontaneous combustion.

- Exert extreme caution due to potential ignition of flammable solvents or other materials.
- If anyone is exposed, or on fire, wash with copious amounts of water, ideally in the lab shower.
- Call for a coworker to provide backup.
- Place a fire extinguisher nearby.
- Carefully remove nearby flammable materials.
- Powdered lime (calcium oxide, CaO) or dry sand should be used to completely smother and cover any spill that occurs.
- Carefully quench by slow addition of isopropanol.
- After complete quench, double bag spill residues for hazardous waste pickup.
- Call 911 for emergency assistance if necessary.

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>

Disposal of Pyrophoric Solid Reagents by Quenching

- Small amounts of unused or unwanted pyrophoric materials must be destroyed by careful quenching of the residue. Transfer the materials to an appropriate reaction flask for hydrolysis and/or neutralization. Dilute significantly with an unreactive solvent such as heptane or toluene and place the flask in an ice water cooling bath. Slowly add isopropanol to quench pyrophoric materials. Upon completion, add methanol as a more reactive quenching agent to ensure completion. Finally, add water dropwise to make sure there are no pockets of reactive materials. Dispose of as hazardous waste.

- Alternatively, reactive substances can be quenched by slowly adding the dilute solution to dry ice, then adding a mildly reactive quenching agent such as methanol.
- AVOID low boiling diluents such as ether and pentane that tend to condense water upon evaporation.
- Do not leave containers with residues of pyrophoric materials open to the atmosphere due to uncontrolled ignition.

Disposal of Pyrophoric Solid Reagents by Submitting to EHS as Hazardous Waste

- Larger quantities of pyrophoric solid chemicals can be disposed of as hazardous waste.
- Carefully package and label the wastes.
- Specifically Alert EH&S personnel at the collection location to the hazards of any wastes containing pyrophoric solid chemicals.

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 lithium aluminum deuteride 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

Eyewash

Suitable facilities for quick drenching or flushing of the eyes should be within 10 seconds travel time for immediate emergency use. Bottle type eyewash stations are not acceptable.

Safety Shower

A safety or drench shower should be available within 10 seconds travel time from where lithium aluminum deuteride is used.

Fume Hood

Many pyrophoric chemicals release noxious or flammable gases and should be handled in a laboratory hood. In addition, some pyrophoric materials are stored under kerosene (or other flammable solvent), therefore the use of a fume hood (or glove box) is required to prevent the release of flammable vapors into the laboratory.

Fire Extinguisher

- A Class C dry chemical fire extinguisher must be available within 10 seconds travel time from where lithium aluminum deuteride is used.
- Know the location of the nearest Class D fire extinguisher.
- A container of powdered lime (calcium oxide, CaO) should be kept within arm's length when working with lithium aluminum deuteride.

Glove (dry) box

Glove boxes are an excellent device to control pyrophoric chemicals when inert or dry atmospheres are required.

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

Lab workers using lithium aluminum deuteride 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 schlenk 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 lithium aluminum deuteride 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 lithium aluminum deuteride in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this lithium aluminum deuteride 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 lithium aluminum deuteride. 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 Propylene oxide-d₂

1. Wear flame-resistant outer gloves, chemical-resistant inner gloves, flame-resistant lab coat, and safety goggles.
2. Three-necked round flask (250 mL) is dried and attached with reflux condenser, drying tube (CaCl₂) and a dropping funnel (50 mL).
3. LiAlD₄ (2.5 g, 0.06 mol) is put into the round flask and diethyl ether (100 mL) was added to the flask with LiAlD₄.
4. The slurry mixture is cooled in an ice bath.
5. (S)- or (R)-2-chloropropanoic acid (5.4 g, 0.05 mol) and diethyl ether (40 mL) are added to the flask by using dropping funnel.
6. After 15 min, water (5 mL) is added drop by drop.
7. The precipitate is dissolved by addition of sulfuric acid (2 N, 150 mL).
8. The aqueous layer is extracted two times with diethyl ether (50 mL).

9. The combined ether layers are washed with water, sodium carbonate solution and sodium bicarbonate solution.
10. The ether layer is concentrated with a rotary evaporator at atmospheric pressure and dried over sodium sulfate.
11. An oily residue is distilled.
12. The flask (10 mL) with KOH (2.4 g, 0.44 mol) and water (2.5 mL) is put to ice bath.
13. The flask is connected to vacuum trap.
14. The distilled (S)- or (R)-2-chloropropan-1-ol (2.4 g, 0.025 mol) is put into the flask (10 mL).
15. As the cyclization reaction proceeds, a white precipitate of potassium chloride is formed.
16. The oxide is captured by vacuum trap using vacuum.
17. The trapped oxide is dried by calcium hydride and redistilled.

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

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

Lithium aluminum hydride

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 lithium aluminum hydride (H_4AlLi , CAS No. 16853-85-3) is used in laboratory. Workers should not use lithium aluminum hydride until they have read and fully understood these safe operating procedures. However, reading these procedures does not substitute for hands-on training. New users of lithium aluminum hydride must work under the close supervision of an experienced user. Lithium aluminum hydride is corrosive solid. It causes severe skin burns and eye damage. **In contact with water it releases flammable gases, which may ignite spontaneously.** It is extremely destructive to the tissue of the mucous membranes and upper respiratory tract. Also, It may be harmful if inhaled, if absorbed through skin, or if swallowed.

Synonyms: LAH

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, Water Reactive, Toxic by ingestion, Target Organ Effect**

GHS Classification

- Substances, which in contact with water, emit flammable gases (Category 1)
- Acute toxicity, Oral (Category 3)
- Skin corrosion (Category 1B)
- Serious eye damage (Category 1)

Signs and Symptoms of Exposure

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated., Large doses of lithium ion have caused dizziness and prostration, and can cause kidney damage if sodium intake is limited. Dehydration, weight loss, dermatological effects, and thyroid disturbances have been reported. Central nervous system effects that include slurred speech, blurred vision, sensory loss, ataxia, and convulsions may occur. Diarrhea, vomiting, and neuromuscular effects such as tremor, clonus, and hyperactive reflexes may occur as a result of repeated exposure to lithium ion.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

Chemical splash goggles or safety glasses with side shields that meet the ANSI Z.87.1 1989 standard must be worn whenever handling pyrophoric

chemicals. Ordinary prescription glasses will NOT provide adequate protection unless they also meet this standard. When there is the potential for splashes, goggles must be worn, and when appropriate, a face shield added. A face shield is required any time there is a risk of explosion, large splash hazard or a highly exothermic reaction. All manipulations of pyrophoric chemicals which pose this risk should occur in a fume hood with the sash in the lowest feasible position. Portable shields, which provide protection to all laboratory occupants, are advisable.

b. Skin and Body Protection

A flame resistant lab coat must be worn.

A chemical-resistant apron worn over the lab coat is required for working with large quantities.

No open toe shoes are allowed.

c. Hand Protection

- Hand protection requirements for the use of solid pyrophoric chemicals outside an inerted glove box include wearing/donning the appropriate chemical resistant out gloves (Neoprene) and fire resistant (FR) inner gloves/liners.
- Gloves or glove liners composed of the tight weave, inherently flame resistant materials Kevlar[®], Nomex[®], Kerinel[®], or PBI[®], or a blend of those materials, of sufficient thickness to prevent or minimize burn injuries to the extent feasible. Gloves or glove liners meeting MIL-DTL-81188C are also acceptable.
- Acceptable glove liners include the followings:
 - ✓ Ansell Kevlar[®] Goldknit[®] Lightweight 70-200,
 - ✓ Hanz Extremity Wear Nomex[®] Utility 2257C and 2259C,
 - ✓ Other Kevlar[®] gloves with the fabric basis weight of a minimum of 7.7 ounces per square yard and if one layer of the material has a minimum of 35 mils in thickness, and
 - ✓ Other Nomex[®] gloves meeting the specifications for Hans Extremity Wear Nomex[®] Utility 2257G and 2259C.
- Laboratories must have these gloves available and use them for this specific research activity. This requirement may not be superseded by reference to a Safety Data Sheet (SDS).

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/PROTOCOLS

Handling Pyrophoric Solid Reagents

- Pyrophoric solids are ideally used in a sealed glove box flushed with inert gas.
- Many pyrophoric solids are sold as solutions, or dispersions in mineral oil or are covered with hydrocarbon solvents to facilitate use.
- Mildly pyrophoric solids (such as lithium aluminum deuteride, hydride and sodium hydride) may be handled in the air for brief periods of time, but the containers must be flushed with inert gas before storage.

Transferring and Weighing Pyrophoric Solid Reagents

- Gather all necessary experimental equipment first to avoid prolonged exposure of pyrophoric solids to air.
- Weighing alkali metals: Cut desired piece of alkali metal under packing oil using a knife. Using tweezers, transfer to adjacent flask containing toluene or heptane to rinse off oil. Use tweezers again to transfer to a weighed flask of toluene and measure weight to determine mass of metal. Use tweezers again to transfer to desired reaction flask.
- AVOID low boiling rinses such as ether and pentane that tend to condense water upon evaporation.

Specific Recommendations for Working with Pyrophoric Solid Reagents

- Lithium Aluminum Hydride reacts violently with water and has a significant heat of solvation. Therefore DO NOT add solvent to dry LiAlD_4 or LiAlH_4 . Instead, slowly add LiAlD_4 or LiAlH_4 to anhydrous solvent in the reaction flask. The initial small amount of LiAlD_4 or LiAlH_4 will react with any trace amounts of water.
- Potassium metal is considerably more reactive than lithium or sodium.
- Potassium metal oxidizes to potassium oxide (K_2O), potassium peroxide (K_2O_2), and potassium superoxide (KO_2). The yellow peroxides are shock-sensitive and can explode when handled or cut. Therefore dispose of potassium metal as hazardous waste if old or if a significant amount of yellow crust is visible.
- The mineral oil of potassium hydride or sodium hydride dispersions can be rinsed off using a light hydrocarbon solvent such as hexane. This is easily accomplished in a glove box or can be done in a hood UNDER CAREFULLY CONTROLLED CONDITIONS. Weigh out desired amount of dispersion and seal in a flask under nitrogen. Add dry hexane via syringe, swirl, and let metal hydride settle. Slowly syringe off hexane and then carefully discard into a separate flask containing isopropanol. Repeat rinse procedure.
- AVOID low boiling rinses such as ether and pentane that tend to condense water upon evaporation.
- Sodium amalgam, Na(Hg) , (or potassium amalgam) is prepared by dissolving sodium into liquid mercury. This highly exothermic process produces the intermetallic compound NaHg_2 with enough heat to cause local boiling of the mercury. Thus it must be performed in a hood under dry nitrogen gas. The grey solid produced has the reducing potential of sodium, but is more air stable.

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 pyrophoric chemicals under an inert atmosphere or under kerosene as appropriate.

Avoid storage areas with heat/flames, oxidizers, and water sources.

Containers carrying pyrophoric materials must be clearly labeled with the correct chemical name and hazard warning.

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.).

Spill - Large

- Exert extreme caution due to potential spontaneous combustion.
- Exert extreme caution due to potential ignition of flammable solvents or other materials.
- If anyone is exposed, or on fire, wash with copious amounts of water, ideally in the lab shower.
- Call 911 for emergency assistance.
- Evacuate the spill area.
- Post someone or mark-off the hazardous area with tape and warning signs to keep other people from entering.
- Provide emergency personnel with technical advice on the chemicals involved.

Spill - Small

- Exert extreme caution due to potential spontaneous combustion.
- Exert extreme caution due to potential ignition of flammable solvents or other materials.

- If anyone is exposed, or on fire, wash with copious amounts of water, ideally in the lab shower.
- Call for a coworker to provide backup.
- Place a fire extinguisher nearby.
- Carefully remove nearby flammable materials.
- Powdered lime (calcium oxide, CaO) or dry sand should be used to completely smother and cover any spill that occurs.
- Carefully quench by slow addition of isopropanol.
- After complete quench, double bag spill residues for hazardous waste pickup.
- Call 911 for emergency assistance if necessary.

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>

Disposal of Pyrophoric Solid Reagents by Quenching

- Small amounts of unused or unwanted pyrophoric materials must be destroyed by careful quenching of the residue. Transfer the materials to an appropriate reaction flask for hydrolysis and/or neutralization. Dilute significantly with an unreactive solvent such as heptane or toluene and place the flask in an ice water cooling bath. Slowly add isopropanol to quench pyrophoric materials. Upon completion, add methanol as a more reactive quenching agent to ensure completion. Finally, add water dropwise to make sure there are no pockets of reactive materials. Dispose of as hazardous waste.

- Alternatively, reactive substances can be quenched by slowly adding the dilute solution to dry ice, then adding a mildly reactive quenching agent such as methanol.
- AVOID low boiling diluents such as ether and pentane that tend to condense water upon evaporation.
- Do not leave containers with residues of pyrophoric materials open to the atmosphere due to uncontrolled ignition.

Disposal of Pyrophoric Solid Reagents by Submitting to EHS as Hazardous Waste

- Larger quantities of pyrophoric solid chemicals can be disposed of as hazardous waste.
- Carefully package and label the wastes.
- Specifically Alert EH&S personnel at the collection location to the hazards of any wastes containing pyrophoric solid chemicals.

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 lithium aluminum hydride 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

Eyewash

Suitable facilities for quick drenching or flushing of the eyes should be within 10 seconds travel time for immediate emergency use. Bottle type eyewash stations are not acceptable.

Safety Shower

A safety or drench shower should be available within 10 seconds travel time from where lithium aluminum hydride is used.

Fume Hood

Many pyrophoric chemicals release noxious or flammable gases and should be handled in a laboratory hood. In addition, some pyrophoric materials are stored under kerosene (or other flammable solvent), therefore the use of a fume hood (or glove box) is required to prevent the release of flammable vapors into the laboratory.

Fire Extinguisher

- A Class C dry chemical fire extinguisher must be available within 10 seconds travel time from where lithium aluminum hydride is used.
- Know the location of the nearest Class D fire extinguisher.
- A container of powdered lime (calcium oxide, CaO) should be kept within arm's length when working with lithium aluminum hydride.

Glove (dry) box

Glove boxes are an excellent device to control pyrophoric chemicals when inert or dry atmospheres are required.

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

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

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

Magnesium sulfate

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when magnesium sulfate (MgSO₄, CAS No. 7487-88-9) is used in laboratory. Its purpose is not to have any accident or risk. Magnesium sulfate causes skin and eye irritation. It may be harmful if inhaled or if swallowed.

Synonyms: Magnesium sulphate

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 (Central nervous system, Gastrointestinal tract) Effect
 GHS Classification
 Not a dangerous substance according to GHS

Signs and Symptoms of Exposure

Diarrhoea, Vomiting, Central nervous system depression, 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 magnesium sulfate must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

Lab workers using magnesium sulfate must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenk 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 magnesium sulfate described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) employ < 25 g of this magnesium sulfate in any given reaction (larger quantities REQUIRE the approval of PI or designee), and

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

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2013

Mesitylene

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 mesitylene (C₉H₁₂, CAS No. 108-67-8) used in laboratory. Its purpose is not to have any accident or risk. Mesitylene is highly flammable liquid and vapor, and toxic if swallowed, if inhaled, or in contact with skin. It causes serious eye and skin irritation. It may cause respiratory irritation. Keep away from heat, sparks, open flames, or hot surfaces. No smoking, Keep container tightly closed. Use explosion-proof electrical, ventilating, lighting equipment. Avoid breathing dust, fume, gas, mist, vapors, or spray. Use only non-sparking tools. Take precautionary measures against static discharge. Wash skin thoroughly after handling. Use only outdoors or in a well-ventilated area

Synonyms: 1,3,5-Trimethylbenzene

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

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

OSHA Hazards: Flammable liquid, Irritant

GHS Classification

Flammable liquids (Category 3)

Skin irritation (Category 2)

Specific target organ toxicity – single exposure (Category 3)

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 mesitylene 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 mesitylene.

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

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

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) employ < 5 mL of this mesitylene in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this mesitylene 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 mesitylene. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Catalytic Reaction

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Into a round-bottom flask (20 mL), add Au catalyst (1 wt.%), biphenyl (0.1 mmol, internal standard), 2'-hydroxyacetophenone (0.5 mmol), benzaldehyde (0.5 mmol), 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.

Internal standard in oxidation reactions

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

6. Put the mesitylene bottle back to the flammable cabinet.
7. Add the reactant, close the test tube with the rubber septum, seal with Teflon tape and connect the oxygen supply to the test tube.
8. 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).
9. Collect samples at different reaction. Put a sample (100 µL) into a small centrifuge tube and centrifuge it to remove the solids.
10. After centrifuging bring the closed vial back to the fume hood and transfer the liquid into a new vial.
11. Inject the sample into GC using a Hamilton syringe (10 µL).
12. Dispose all the waste into the appropriately labeled waste bottle.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

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

Methanol

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when methanol (CH₄O, CAS No. 67-56-1) used in laboratory. Its purpose is not to have any accident or risk. Methanol is highly flammable liquid and vapor, and toxic if swallowed, if inhaled, or in contact with skin. It causes serious eye and skin irritation.

Synonyms: Methyl alcohol

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

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

OSHA Hazards: Flammable liquid, Target organ effect (Eyes, Kidney, Liver, Heart, Central nervous system), Toxic by Inhalation, Ingestion and Skin absorption, Irritant.

GHS Classification

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

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

- 1) not work alone;

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

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

Photoreaction reagent

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

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

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

SOP Reviewed and Approved by:

Francisco Zaera

 Print name

 Signature

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

Methyl bromoacetate

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when methyl bromoacetate ($C_3H_5BrO_2$, CAS No. 96-32-2) used in laboratory. Its purpose is not to have any accident or risk. Methyl bromoacetate is highly flammable liquid and vapor, and toxic if swallowed, if inhaled, or in contact with skin. It causes serious eye damage and skin burns

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

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

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

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

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

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

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

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

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

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

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

Approval Date: 06/01/2015

(Methylcyclopentadienyl)manganese tricarbonyl 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 (methylcyclopentadienyl)manganese tricarbonyl (C₉H₇MnO₃, CAS No. 12108-13-3) is used in laboratory. Its purpose is not to have any accident or risk. Especially (methylcyclopentadienyl)manganese tricarbonyl is a **OSHA select carcinogen**, so may cause cancer and heritable genetic damage. It is toxic by inhalation, in contact with skin and if swallowed. It causes serious damage to health by prolonged exposure through inhalation. (Methylcyclopentadienyl)manganese tricarbonyl is used as a precursor for atomic layer deposition (ALD) projects in Zaera group. Please be very careful when you handle, or replace it with other precursors if possible.

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

Please refer its MSDS always before using them.

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: Carcinogen, Target Organ Effect (Nerves and Lungs), Toxic by Inhalation, Ingestion, and Skin Absorption

GHS Classification

- Acute toxicity, Oral (Category 2)
- Acute toxicity, Inhalation (Category 1)
- Acute toxicity, Dermal (Category 3)
- Carcinogenicity (Category 2)

Signs and Symptoms of Exposure

Men exposed to manganese dusts showed a decrease in fertility. Chronic manganese poisoning primarily involves the central nervous system. Early symptoms include languor, sleepiness and weakness in the legs. A stolid mask-like appearance of the face, emotional disturbances such as uncontrollable laughter and a spastic gait with tendency to fall in walking are findings in more advanced cases. High incidence of pneumonia has been found in workers exposed to the dust or fume of some manganese compounds.

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-line 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 (methylcyclopentadienyl)manganese tricarbonyl 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 (methylcyclopentadienyl)manganese tricarbonyl.

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 (methylcyclopentadienyl)manganese tricarbonyl must review this SOP and sign the associated training sheet. Lab workers must have specific

training on the proper handling of (methylcyclopentadienyl)manganese tricarbonyl and understand the hazards.

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

(Methylcyclopentadienyl)manganese tricarbonyl Sample Preparation:

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety Goggle.
2. 1'33" flange with sealed-off Pyrex glass end tube should be dried in the oven for 1 hour, cooled down to room temperature.
3. Take the flange and parafilm wrap (2 inch) to Prof. Bocian's lab, follow the procedure of using the glovebox, transfer about 1 ml of this (Methylcyclopentadienyl)manganese tricarbonyl to the flange, and seal the flange with the parafilm wrap.
4. Care should be taken to fast remove the parafilm and mount the flange to the leak vale.
5. Use liquid N₂ to freeze the glass end, fully open the leak valve, close the valve until the pressure goes down, and remove the liquid N₂ to let the liquid inside the glass to thaw.
6. Repeat the freeze-pump-thaw in step 4 two times.
7. Control the leak valve, and do the experiments.
8. After experiment, the silicone sample needs to be treated as solid hazardous waste. The glass tube needs to be cleaned with acetone and water, and the waste solution should be placed into the proper waste container.

SOP Reviewed and Approved by:

 Francisco Zaera
 Print name

 Signature

Approval Date: 06/01/2013, updated 03/01/2014, 03/01/2016

Methylene Chloride

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

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

Synonyms: **Dichloromethane**

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

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

OSHA regulated Carcinogen

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

OSHA Hazards: **Carcinogen**

GHS Classification

Carcinogenicity (Category 2)

Signs and Symptoms of Exposure

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

anesthetic effects, Difficulty in breathing, Headache, Dizziness, Prolonged or repeated contact with skin may cause:, defatting, Dermatitis, Contact with eyes can cause:, Redness, Blurred vision, Provokes tears., Effects due to ingestion may include:, Gastrointestinal discomfort, Central nervous system depression, Paresthesia., Drowsiness, Convulsions, Conjunctivitis., Pulmonary edema. Effects may be delayed., Irregular breathing., Stomach/intestinal disorders, Nausea, Vomiting, Increased liver enzymes., Weakness, Heavy or prolonged skin exposure may result in the absorption of harmful amounts of material., Abdominal pain.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

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

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

Developing Solvent for Column Process

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

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

SOP Reviewed and Approved by:

Francisco Zaera

 Print name

 Signature

Approval Date: 06/01/2015

Molecular sieves

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when molecular sieves (N/A, CAS No. 70955-01-0) is used in laboratory. Its purpose is not to have any accident or risk. Molecular sieves causes serious eye, skin and respiratory irritation.

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

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

OSHA Hazards: Irritant

GHS Classification

Skin irritation (Category 2)

Eye irritation (Category 2A)

Specific target organ toxicity – single exposure (Category 3)

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) employ < 500 g of this molecular sieves in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this molecular sieves with the PI prior to its use.

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

Dehydrate with molecular sieves

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

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

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

Molybdenum(IV) sulfide

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 molybdenum sulfide (MoS₂, CAS No. 1317-33-5) is used in laboratory. Its purpose is not to have any accident or risk. Molybdenum sulfide is toxic if swallowed, if inhaled or in contact with skin.

Synonyms: Molybdenum disulfide

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, Irritant

GHS Classification

Acute toxicity, Inhalation (Category 4)

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 molybdenum sulfide 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 molybdenum sulfide.

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

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

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) employ < 25 g of this molybdenum sulfide in any given reaction (larger quantities REQUIRE the approval of PI or designee), and

- 5) discuss ALL issues or concerns regarding this molybdenum sulfide 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 molybdenum sulfide. 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.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013

Neon

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 neon (Ne, CAS No. 7440-01-9) used in laboratory. Its purpose is not to have any accident or risk. Neon 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 (Liquefied gas)

Signs and Symptoms of Exposure

May be harmful. Nausea, Headache, Vomiting

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

Lab workers using neon 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 schlenk 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 neon 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 neon 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 neon 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 neon 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 temporarily 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.

UHV #3, Michelle

1. Safely secure neon bottle to the base of the chamber and make sure the bottle is in the up straight position.
2. Ensure main valve is completely closed.
3. Unscrew main valve cap.
4. Attach the appropriate pressure regulator and connect to the system using a copper tube.
5. Carefully adjust the outlet pressure to 20 psi.
6. Close the valve next to the mechanical pump.

7. Fill the copper tube with neon gas. Then open the valve to pump down.
8. Repeat steps 6-7 to purge the copper line three times.
9. Carefully pressurize copper line.
10. Slowly open the leak valve to leak the gas into the UHV system at desired pressure.
11. Close the leak valve.
12. Close the valve on the regulator.
13. Close the main valve.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2015

Nitrogen

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when nitrogen (N₂, CAS No. 7727-37-9) used in laboratory. Its purpose is not to have any accident or risk. Nitrogen contains gas under pressure. It may explode if heated.

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

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

OSHA Hazards: Compressed Gas

GHS Classification

Gas under pressure (Liquefied gas)

Signs and Symptoms of Exposure

May be harmful. Nausea, Headache, Vomiting

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

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

Upon receipt of nitrogen gas, cylinders shall be temporarily stored in a well-ventilated area that is attended or locked at all times. All cylinders shall be immediately leak tested with a leak indicating solution and must be clearly labeled with content and hazard

information. Temporary storage locations shall have appropriate signage in place. Cylinders must be seismically secured at all locations with chains at two contact points on the cylinder body, using unistruts or an equivalent. Seismic securing should prevent cylinders from rolling, shifting, or falling.

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

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

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

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

STORAGE:

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

6. SPILL AND INCIDENT PROCEDURES

Emergency procedure for leaking gas cylinders -

<http://www.airproducts.com/~media/Files/PDF/company/safetygram-11.pdf>

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

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

7. WASTE DISPOSAL

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

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

General hazardous waste disposal guidelines:

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

8. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

9. DESIGNATED AREA

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

10. SAFETY DATA SHEETS

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

11. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) use nitrogen gas under 1 bar in any given reaction (higher pressure REQUIRE the approval of PI or designee), and

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

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

Replace empty gas cylinder

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

Replacing empty gas cylinder for GC & BET Instrument

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

6. Fully open the regulator valves and the main cylinder valve and check the pressure.

ALD Reactor

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

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

Nitrogen Liquid

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when **nitrogen liquid** (N₂, CAS No. **7727-37-9**) used in laboratory. Its purpose is not to have any accident or risk. Nitrogen liquid cylinder contains refrigerated gas under pressure. It may cause cryogenic burns or injury. It is considered hazardous by OSHA.

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

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

OSHA Hazards: **Refrigerated Gas**

GHS Classification

Gas under pressure (Refrigerated Liquefied Gas)

Signs and Symptoms of Exposure

Extremely cold material. Liquid can cause burns similar to frostbite, No known significant effects or critical hazards

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye/Face Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

All transport of **nitrogen liquid** between on-campus locations must be conducted as follows:

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

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

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

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

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

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

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

STORAGE:

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

6. SPILL AND INCIDENT PROCEDURES

Emergency procedure for leaking gas cylinders -

<http://www.airproducts.com/~media/Files/PDF/company/safetygram-11.pdf>

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

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

7. WASTE DISPOSAL

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

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

General hazardous waste disposal guidelines:

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

8. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

9. DESIGNATED AREA

A designated area shall be established where limited access, special procedures, knowledge, and work skills are required. A designated area can be the entire laboratory, a specific laboratory workbench, or a laboratory hood. Designated areas must be clearly marked with signs that identify the chemical hazard and include an appropriate warning; for example: WARNING! **NITROGEN LIQUID** WORK AREA!

10. SAFETY DATA SHEETS

Online SDS can be found at <http://www.ehs.ucr.edu/services/msds.html>.

11. DETAILED PROTOCOL

All lab workers who will be using nitrogen liquid must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of nitrogen liquid and understand the hazards.

Lab workers using nitrogen liquid must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenk line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with nitrogen liquid described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) use nitrogen liquid less than 1 L at a time (higher pressure REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this nitrogen liquid with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material(s), the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using nitrogen liquid. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Fill a dewar with LN2 from a tank cylinder

1. Wear flame-resistant lab coat, apron, full-face shield, and waterproof cryo-gloves.
2. Bring a LN2 dewar to LN2 tanks in hallway.
3. Warn pedestrians not to pass the hallway.
4. Open the cap of Dewar
5. Insert the hose of the LN2 tank into the dewar.
6. Open the main valve of the LN2 tank.
7. Fill the dewar slowly.
8. Top the dewar off.
9. Close the main valve of the LN2 tank.
10. Take the hose out.
11. Close the cap of dewar.
12. Bring the dewar into the lab.

SOP Reviewed and Approved by:

Francisco Zaera

 Print name

 Signature

Approval Date: 02/01/2013, updated 05/15/2016

Nitromethane

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 nitromethane (CH₃NO₂, CAS No. 75-52-5) used in laboratory. Its purpose is not to have any accident or risk. Nitromethane is highly flammable liquid and vapor, and harmful if swallowed, if inhaled, or in contact with skin. It causes eye and skin irritation.

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: **Flammable liquid, Carcinogen, Target Organ Effect, Harmful by Ingestion**

GHS Classification

Flammable liquids (Category 3)
Acute toxicity, Oral (Category 3)
Acute aquatic toxicity (Category 3)

Signs and Symptoms of Exposure

Absorption into the body leads to the formation of methemoglobin which in sufficient concentration causes cyanosis. Onset may be delayed 2 to 4 hours or longer.

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 nitromethane 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 nitromethane.

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 nitromethane must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of nitromethane and understand the hazards.

Lab workers using nitromethane 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 schlenk 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 nitromethane described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) employ < 10 mL of this nitromethane in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this nitromethane 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 nitromethane. 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 gloves, flame-resistant lab coat, and safety goggles.
2. Take 2 mL of nitromethane.
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.

Catalytic Reaction

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. 3-Penten-2-ol (0.25 mL) is mixed with ethyl vinyl ether (0.25 mL), nitromethane (1.0 mL) and acid-base silica catalyst (10~20 mg).
3. Reaction occurs over various time frames from 2~24 hours at 40 °C under a nitrogen atmosphere.
4. The solution is treated as hazardous waste after the catalytic reaction has ended.

SOP Reviewed and Approved by:

 Francisco Zaera
 Print name

 Signature

Approval Date: 02/01/2013, updated 03/01/2016

Nitrous oxide

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 nitrous oxide (N₂O, CAS No. 10024-97-2) used in laboratory. Its purpose is not to have any accident or risk. Nitrous oxide is oxidizing gas, and contains gas under pressure. It may cause or intensify fire.

2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Please refer MSDS first always for physical and chemical properties before use.

OSHA Hazards: Oxidizer, Compressed Gas, Target Organ Effect (Blood, Lungs)

GHS Classification

Oxidizing gas (Category 1)

Gas under pressure (Liquefied gas)

Signs and Symptoms of Exposure

Anesthetic effects.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. If chemical has a skin hazard or is a caustic liquid, a face shield should be worn when splashing onto the face is a possibility.

b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

A chemical resistant apron should be used when transferring or using large quantities and splashing is a possibility.

Flame-resistant lab coat will be required, if working with pyrophoric chemicals.

c. Hand Protection

At a minimum, wear a nitrile chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf or <http://www.showabestglove.com/site/default.aspx>

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

4. ENGINEERING/VENTILATION CONTROLS

A ventilation monitor is required on each lab hood or gas manifold in which nitrous oxide 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 nitrous oxide 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 nitrous oxide 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 nitrous oxide 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 nitrous oxide 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 nitrous oxide 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 nitrous oxide 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 nitrous oxide gas cylinders shall be labeled as empty. Depleted nitrous oxide 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 nitrous oxide gas cylinders by EH&S, even when empty, may entail extraordinary costs.

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-line 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 nitrous oxide 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! NITROUS OXIDE 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 nitrous oxide gas must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of nitrous oxide gas and understand the hazards.

Lab workers using nitrous oxide 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 nitrous oxide 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 nitrous oxide 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 nitrous oxide with the PI prior to its use.

If there is an unusual or unexpected occurrence when using this material, the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using nitrous oxide. 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 temporarily 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.

UHV #3, Michelle

1. Safely secure nitrous oxide bottle to the base of the chamber and make sure the bottle is in the up straight position.
2. Ensure main valve is completely closed.
3. Unscrew main valve cap.
4. Attach the appropriate pressure regulator and connect to the system using a copper tube.
5. Carefully adjust the outlet pressure to 20 psi.
6. Close the valve next to the mechanical pump.
7. Fill the copper tube with nitrous oxide gas. Then open the valve to pump down.
8. Repeat steps 6-7 to purge the copper line three times.
9. Carefully pressurize copper line.
10. Slowly open the leak valve to leak the gas into the UHV system at desired pressure.
11. Close the leak valve.
12. Close the valve on the regulator.
13. Close the main valve.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2015