

(R)-(+)-1-(1-Naphthyl)ethylamine 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 (R)-(+)-1-(1-naphthyl)ethylamine (C₁₂H₁₃N, CAS No. 3886-70-2) is used in laboratory. Its purpose is not to have any accident or risk. (R)-(+)-1-(1-Naphthyl)ethylamine causes skin and eye irritation. It may be harmful if inhaled or if swallowed or if absorbed through skin.

Synonyms: (R)-(+)-α-Methyl-1-naphthalenemethylamine

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 (R)-(+)-1-(1-naphthyl)ethylamine 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 (R)-(+)-1-(1-naphthyl)ethylamine.

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 (R)-(+)-1-(1-naphthyl)ethylamine must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of (R)-(+)-1-(1-naphthyl)ethylamine and understand the hazards.

Lab workers using (R)-(+)-1-(1-naphthyl)ethylamine 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 (R)-(+)-1-(1-naphthyl)ethylamine 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 < 1 g of this (R)-(+)-1-(1-naphthyl)ethylamine in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this (R)-(+)-1-(1-naphthyl)ethylamine 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 (R)-(+)-1-(1-naphthyl)ethylamine. 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 (R)-(+)-1-(1-naphthyl)ethylamine/CCl₄ solution (1 mM).

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 SDSs of (R)-(+)-1-(1-naphthyl)ethylamine and CCl₄ again; especially remind first-aid measures, handling and storage, PPE, & signs and symptoms of exposure.*
3. Bring the bottles of (R)-(+)-1-(1-naphthyl)ethylamine and carbon tetrachloride into a fume hood.
4. Prepare (R)-(+)-1-(1-naphthyl)ethylamine/carbon tetrachloride solution (1 mM). *Be careful not to spill CCl₄. Keep watching any leak of CCl₄. Avoid release to the environment. Avoid breathing fume, gas, mist, vapor or spray. If swallowed, immediately call 911. If inhaled, rinse cautiously with water for 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.*
5. Return the original bottles to the designated store locations.
6. Do the desired experiments in ATR-IR setup. *Be careful not to spill CCl₄. Keep watching any leak of CCl₄. 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. Used and remained solutions need to be treated as hazardous wastes.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 4/22/2016

(R)-(+)-1-(2-Naphthyl)ethanol

STANDARD OPERATING PROCEDURE

Type of SOP: Process Hazardous Chemical Hazard Class

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when (R)-(+)-1-(2-naphthyl)ethanol ($C_{12}H_{12}O$ CAS No. 52193-85-8) is used in laboratory. Its purpose is not to have any accident or risk. (R)-(+)-1-(2-Naphthyl)ethanol may be harmful if inhaled, absorbed through skin, or swallowed. It may cause eye or skin irritation.

Synonyms: (R)-(+)- α -Methyl-2-naphthalenemethanol

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

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

OSHA Hazards: not known

GHS Classification

Not a dangerous substance according to GHS

Signs and Symptoms of Exposure

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 (R)-(+)-1-(2-naphthyl)ethanol must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

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

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

10. DESIGNATED AREA

Work should be completed in a laboratory fume hood designated for (R)-(+)-1-(2-naphthyl)ethanol.

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

All lab workers who will be using (R)-(+)-1-(2-naphthyl)ethanol must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of (R)-(+)-1-(2-naphthyl)ethanol and understand the hazards.

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

The research laboratory requires variation in reaction conditions to develop and optimize new chemical or biological transformations. The researcher must seek literature precedent for reaction conditions that have reasonable similarities to new chemistry that is planned with (R)-(+)-1-(2-naphthyl)ethanol described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

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

- 4) employ < 5 g of this (R)-(+)-1-(2-naphthyl)ethanol in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this (R)-(+)-1-(2-naphthyl)ethanol with the PI prior to its use.

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

Preparation of (R)-(+)-1-(2-naphthyl) ethanol/CCl₄ solution (5 mM)

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and goggles.
2. Measure (R)-(+)-1-(2-naphthyl) ethanol (8.61 mg).
3. Bring the bottle of carbon tetrachloride into a fume hood.
4. Take 10 mL of CCl₄ into a flask.
5. Add the prepared (R)-(+)-1-(2-naphthyl) ethanol into the flask.
6. Return the original bottles to the designated store locations.
7. Do the desired experiments in ATR-IR setup.
8. Used and remained solutions need to be treated as hazardous wastes.

SOP Reviewed and Approved by:

Francisco Zaera

 Print name

 Signature

Approval Date: 02/06/2020

(R)-(+)- α -Methylbenzylamine 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 (R)-(+)- α -methylbenzylamine ($C_8H_{11}N$, CAS No. 3886-69-9) used in laboratory. Its purpose is not to have any accident or risk. (R)-(+)- α -Methylbenzylamine is flammable liquid and vapor, and harmful if swallowed, if inhaled, or in contact with skin. It causes serious eye damage and skin burn.

Synonyms: (R)-(+)-1-Phenylethylamine

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

GHS Classification

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

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with (R)-(+)- α -methylbenzylamine 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 (R)-(+)- α -methylbenzylamine.

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 Flammable Toxic Liquid must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of (R)-(+)- α -methylbenzylamine and understand the hazards.

Lab workers using (R)-(+)- α -methylbenzylamine 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 (R)-(+)- α -methylbenzylamine 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 mL of this (R)-(+)- α -methylbenzylamine in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this (R)-(+)- α -methylbenzylamine 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 (R)-(+)- α -methylbenzylamine. 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 (R)-(+)- α -Methylbenzylamine/ CCl_4 solution (10 mM)

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and goggles.
2. Measure (R)-(+)- α -methylbenzylamine (12.12 mg) by balance.
3. Bring the bottle of carbon tetrachloride (CCl_4) into a fume hood. And take CCl_4 (10 mL) into a flask.
4. Add the prepared (R)-(+)- α -methylbenzylamine into the flask.
5. Return the original bottles to the designated store locations.
6. Do the desired experiments in ATR-IR setup.
7. Used and remained solutions need to be treated as hazardous wastes.

SOP Reviewed and Approved by:

 Francisco Zaera
 Print name

 Signature

Approval Date: 11/06/2020

(S)-(-)-1-(1-Naphthyl)ethylamine

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 (S)-(-)-1-(1-Naphthyl)ethylamine (C₁₂H₁₃N, CAS No. 10420-89-0) is used in laboratory. Its purpose is not to have any accident or risk. (S)-(-)-1-(1-Naphthyl)ethylamine causes skin and eye irritation. It may be harmful if inhaled or if swallowed or if absorbed through skin.

Synonyms: (S)-(-)- α -Methyl-1-naphthalenemethylamine

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 2A)

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 (S)-(-)-1-(1-Naphthyl)ethylamine 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 (S)-(-)-1-(1-Naphthyl)ethylamine.

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 (S)-(-)-1-(1-Naphthyl)ethylamine must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of (S)-(-)-1-(1-Naphthyl)ethylamine and understand the hazards.

Lab workers using (S)-(-)-1-(1-Naphthyl)ethylamine 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 (S)-(-)-1-(1-Naphthyl)ethylamine 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 < 1 g of this (S)-(-)-1-(1-Naphthyl)ethylamine in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this (S)-(-)-1-(1-Naphthyl)ethylamine 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 (S)-(-)-1-(1-Naphthyl)ethylamine. 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 (S)-(-)-1-(1-naphthyl)ethylamine/CCl₄ solution (1 mM).

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 SDSs of (S)-(-)-1-(1-naphthyl)ethylamine and CCl₄ again; especially remind first-aid measures, handling and storage, PPE, & signs and symptoms of exposure.*
3. Bring the bottles of (S)-(-)-1-(1-naphthyl)ethylamine and carbon tetrachloride into a fume hood.
4. Prepare (S)-(-)-1-(1-naphthyl)ethylamine/carbon tetrachloride solution (1 mM). *Be careful not to spill CCl₄. Keep watching any leak of CCl₄. Avoid release to the environment. Avoid breathing fume, gas, mist, vapor or spray. If swallowed, immediately call 911. If inhaled, rinse cautiously with water for 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.*
5. Return the original bottles to the designated store locations.
6. Do the desired experiments in ATR-IR setup. *Be careful not to spill CCl₄. Keep watching any leak of CCl₄. 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. Used and remained solutions need to be treated as hazardous wastes.

SOP Reviewed and Approved by:

 Francisco Zaera
 Print name

 Signature

Approval Date: 11/01/2013, updated 03/01/2016

(S)-(-)-1,1'-Bi-2-Naphthol

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 (S)-(-)-1,1'-bi-2-naphthol (C₂₀H₁₄O₂, CAS No. 18531-99-2) is used in laboratory. Its purpose is not to have any accident or risk. (S)-(-)-1,1'-Bi-2-naphthol causes skin and eye irritation.

Synonyms: (-)-2,2'-Dihydroxy-1,1'-dinaphthyl, (S)-(-)-1,1'-Binaphthalene-2,2'-diol
(S)-BINOL

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)

Signs and Symptoms of Exposure

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated. After absorption: muscle twitching, Convulsions, change in weight, Other dangerous properties cannot be excluded. Handle in accordance with good industrial hygiene and safety practice.

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 (S)-(-)-1,1'-bi-2-naphthol 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 (S)-(-)-1,1'-bi-2-naphthol.

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 (S)-(-)-1,1'-bi-2-naphthol must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of (S)-(-)-1,1'-bi-2-naphthol and understand the hazards.

Lab workers using (S)-(-)-1,1'-bi-2-naphthol 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 (S)-(-)-1,1'-bi-2-naphthol 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 (S)-(-)-1,1'-bi-2-naphthol in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this (S)-(-)-1,1'-bi-2-naphthol 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 (S)-(-)-1,1'-bi-2-naphthol. 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 (S)-(-)-1,1'-Bi(2-naphthol)/CCl₄ solution (5 mM)

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and goggles.
2. Measure (S)-(-)-1,1'-bi-2-naphthol (14.32 mg) by balance.
3. Bring the bottle of carbon tetrachloride (CCl₄) into a fume hood. And take CCl₄ (10 mL) into a flask.
4. Add the prepared (S)-(-)-1,1'-bi-2-naphthol into the flask.
5. Return the original bottles to the designated store locations.
6. Do the desired experiments in ATR-IR setup.
7. Used and remained solutions need to be treated as hazardous wastes.

SOP Reviewed and Approved by:

 Francisco Zaera
 Print name

 Signature

Approval Date: 04/20/2014

1-Butene

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 1-butene (C₄H₁₀, CAS No. 106-98-9) used in laboratory. Its purpose is not to have any accident or risk. 1-Butene is extremely flammable gas under pressure in a cylinder. It may explode if heated, and may be harmful if inhaled or absorbed through skin. It may cause respiratory tract irritation or 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: **Extremely Flammable gas, Compressed Gas**

GHS Classification

Flammable liquids (Category 1)

Gas under pressure (Liquefied gas)

Signs and Symptoms of Exposure

Central nervous system depression, Dizziness, Headache.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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 **1-butene** 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 1-butene.

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 1-butene must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of 1-butene and understand the hazards.

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

GC #1 Agilent-Batch Reactor: Introducing Reaction Gas Mixture

- Note: Introduce the gas with lowest partial pressure first.
 1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety glasses.
 2. Isolate the gas manifold from the mechanical pump by closing the valve connecting the manifold to the mechanical pump

3. Slowly open the valve for the 1-butene and close the valve after a few seconds
4. Slowly open the valve connecting the gas manifold to the reactor loop while constantly checking the baratron pressure gauge. Make sure to add 5 torr of 1-butene to the reactor loop.
5. After adding the desired amount of gas to the loop, close the valve that connects the manifold to the reactor loop and check that the pressure of the reactor loop remains stable
6. Evacuate the gas manifold by opening the valve to the pump for 30 minutes
7. Slowly open the valve for the H₂ and close the valve after a few seconds
8. Slowly open the valve connecting the gas manifold to the reactor loop while constantly checking the baratron pressure gauge. Make sure to add 50 torr of H₂ to the reactor loop.
9. After adding the desired amount of gas to the loop, close the valve that connects the manifold to the reactor loop and check that the pressure of the reactor loop remains stable
10. Evacuate the gas manifold by opening the valve to the pump for 30 minutes
11. Slowly open the valve for Argon and close the valve after a few seconds
12. Slowly open the valve connecting the gas manifold to the reactor loop while constantly checking the baratron pressure gauge. Make sure to add 545 torr of Argon to the reactor loop.
13. After adding the desired amount of gas to the loop, close the valve that connects the manifold to the reactor loop and check that the pressure of the reactor loop remains stable
14. Evacuate the gas manifold by opening the valve to the pump for 30 minutes, then close the valve.
15. After introducing all the reactant gasses circulate the reactant gas mixture for 20 minutes by turning on the circulation pump.
16. Close the valve at the front of the bypass line, and leave open the valve in the back.
17. Open the valves to the reactor tube, now the reactant mixture will contact the Pt/Al₂O₃ catalyst.

SOP Reviewed and Approved by:

 Francisco Zaera
 Print name

 Signature

Approval Date: 04/09/2022

1-Pentene

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 1-pentene (C₅H₁₀, CAS No. 109-67-1) used in laboratory. Its purpose is not to have any accident or risk. 1-Pentene is highly flammable liquid and vapor, and toxic if swallowed, if inhaled, or in contact with skin. It causes serious 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, Irritant

GHS Classification

Flammable liquids (Category 1)

Skin irritation (Category 2)

Eye irritation (Category 2A)

Aspiration hazard (Category 1)

Short-term (acute) aquatic hazard (Category 3)

Long-term (chronic) aquatic hazard (Category 3)

Signs and Symptoms of Exposure

Symptoms of exposure may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and 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 **1-pentene** 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 1-pentene.

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 1-pentene must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of 1-pentene and understand the hazards.

Lab workers using 1-pentene 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 1-pentene 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 < 100 mL of this 1-pentene in any given reaction (larger quantities REQUIRE the approval of PI or designee), and

- 5) discuss ALL issues or concerns regarding this 1-pentene 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 1-pentene. 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.

From QD-a to QD-a_mercapto TES

1. A flask (5 mL) and a reflux condenser are dried under N₂ gas.
2. QD-a (1.44 g, 1 eq., 3.75 mmol) is placed in the flask.
3. 2,2'-Azobisisoputyronitrile (AIBN, 61.6 mg, 0.1 eq. 0.375 mmol) is also put into the flask.
4. Chloroform (5 mL) and 3-mercaptopropyl triethoxysilane (996 mL) are added to the flask.
5. The mixture is refluxed at 62 °C for 1 day.
6. The mixture is cooled down to room temperature.
7. Chloroform is removed by evaporator.
8. Pentene (40 mL) is added to the remained liquid. White powder is precipitated
9. The white powder is filtered and washed with pentene.
10. The powder is dried under vacuum.

SOP Reviewed and Approved by:

Francisco Zaera

 Print name

 Signature

Approval Date: 10/01/2022

1,4-Dioxane

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 1,4-dioxane (C₄H₈O₂, CAS No. 123-91-1) used in laboratory. Its purpose is not to have any accident or risk. 1,4-Dioxane 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: Dioxane, Diethylene oxide

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 2)

Eye irritation (Category 2A)

Carcinogenicity (Category 2)

Specific target organ toxicity – single exposure (Category 3)

Signs and Symptoms of Exposure

Nausea, Vomiting, Weakness, Dizziness, Vertigo, Headache, Sweating, loss of appetite, Kidney injury may occur. Liver injury may occur. 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 **1,4-dioxane** 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 **1,4-dioxane**.

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 **1,4-dioxane** must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of **1,4-dioxane** and understand the hazards.

Lab workers using **1,4-dioxane** 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 **1,4-dioxane** 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 < 100 mL of this 1,4-dioxane in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this 1,4-dioxane 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 1,4-dioxane. 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 Hydrogenation of Furfural

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Catalyst (50–200 mg), furfural (0.5-1 g), and 1,4-dioxane (75 mL) are added into a reactor.
3. Stir the mixture.
4. The mixture is purged with pure H₂ (1.0 MPa) five times.
5. The reactor is pressurized to a desired H₂ pressure (1.0 MPa) at room temperature.
6. The reactor is heated to a desired temperature.
7. Begin stirring (900 rpm) and set reaction time to start.
8. Sample (0.4 mL) is taken periodically to determine conversion and selectivity during the reaction process.
9. The catalyst powder is filtered off.
10. The filtrate is analyzed using GC.

Reference Standard Solutions for Internal GC Calibration

1. Wear chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Weigh 2-methylfuran (0.0709 g) and n-dodecane (internal standard, 0.375 g) on an electronic balance.
3. Dissolve such sample in 1,4-dioxane (35 mL) to get a mixture solution of 2-methylfuran and n-dodecane.
4. Inject the above solution into GC and calculate the peak area ratio for the analyte and internal standard.
5. If the same sample preparation procedure is used to add internal standard to unknown samples, the response ratio can be used to determine the

concentration ratio. Then the concentration of analyte in the sample can be calculated.

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

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 08/07/2018, update 02/07/2020

2-Phenyl-1,2-propanediol 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 2-phenyl-1,2-propanediol ($C_9H_{12}O_2$ CAS No. 4217-66-7) is used in laboratory.

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

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

OSHA Hazards: *not known*

GHS Classification

Not a dangerous substance according to GHS

Signs and Symptoms of Exposure

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-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 **2-phenyl-1,2-propanediol** 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 2-phenyl-1,2-propanediol.

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 2-phenyl-1,2-propanediol must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of 2-phenyl-1,2-propanediol and understand the hazards.

Lab workers using 2-phenyl-1,2-propanediol 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 2-phenyl-1,2-propanediol 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 2-phenyl-1,2-propanediol in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this 2-phenyl-1,2-propanediol 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 **2-phenyl-1,2-propanediol**. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Dialcohol Conversion

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Add Pt/SiO₂ or Au/TiO₂ catalyst (up to 50 mg) and water (3 mL) into a 10 mL of two-neck flask.
3. Add 2-phenyl-1,2-propanediol (76 μL) into the flask.
4. Connect the flask to a reflux condenser.
5. Attach the oxygen gas line to one of necks and adjust oxygen gas flow (10 mL/min) or higher.
6. Put the flask in an oil bath at 60 °C.
7. Stir the mixture for 1 or 2 days in a fume hood. Leave a label with chemical name and hazard information.
8. Extract the mixture with ether (5 mL, 2 times)
9. Wash the extracted organic layer with water (2 mL, 2 times)
10. Remove any volatiles under evaporator.

SOP Reviewed and Approved by:

 Francisco Zaera
 Print name

 Signature

Approval Date: 06/01/2015

2,2-Azobis(2-methylopropionitrile) 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 2,2-azobis(2-methylopropionitrile) (C₈H₁₂N₄, CAS No. 78-67-1) used in laboratory. Its purpose is not to have any accident or risk. 2,2-Azobis(2-methylopropionitrile) is flammable solid, and toxic if swallowed. Also it is harmful if inhaled.

Synonyms: AIBN, Azobisisobutyronitrile, α,α' -Azoisobutyronitrile

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 solid, Target organ effect (Nerves, Liver and Kidney), Toxic by Ingestion

GHS Classification

Self-reactive substances (Type C)
Acute toxicity, Oral (Category 3)
Acute toxicity, Inhalation (Category 4)
Acute aquatic toxicity (Category 3)
Chronic aquatic toxicity (Category 3)

Signs and Symptoms of Exposure

Nausea, Dizziness, Headache, 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 **2,2-azobis(2-methylopropionitrile)** 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 **2,2-azobis(2-methylopropionitrile)**.

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 **2,2-azobis(2-methylopropionitrile)** must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of **2,2-azobis(2-methylopropionitrile)** and understand the hazards.

Lab workers using **2,2-azobis(2-methylopropionitrile)** 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 **2,2-azobis(2-methylopropionitrile)** 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 2,2-azobis(2-methylpropionitrile) in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this 2,2-azobis(2-methylpropionitrile) 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 2,2-azobis(2-methylpropionitrile). Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Preparation of Cd-TEOSPM

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Weigh 0.020 g of 2,2'-azobis(2-methylpropionitrile).
3. Bring the reagent into the fume hood and add it into the two-neck round bottom flask for reaction.
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.

epi-Quinine Tethering

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

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

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

3-(1-Naphthyl)-L-alanine 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 3-(1-naphthyl)-L-alanine ($C_{13}H_{13}NO_2$ CAS No. 55516-54-6) is used in laboratory. Its purpose is not to have any accident or risk. 3-(1-Naphthyl)-L-alanine is harmful to aquatic life.

Synonyms: (S)- α -Amino-1-naphthalenepropanoic 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: not a hazardous substance

GHS Classification: none

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 **3-(1-naphthyl)-L-alanine** 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 3-(1-naphthyl)-L-alanine.

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 3-(1-naphthyl)-L-alanine must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of 3-(1-naphthyl)-L-alanine and understand the hazards.

Lab workers using 3-(1-naphthyl)-L-alanine 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 3-(1-naphthyl)-L-alanine described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

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

- 4) employ < 10 g of this 3-(1-naphthyl)-L-alanine in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this 3-(1-naphthyl)-L-alanine 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 3-(1-naphthyl)-L-alanine. 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 3-(1-Naphthyl)-L-alanine solution (1 mM)

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and goggles.
2. Measure 3-(1-naphthyl)-L-alanine (43.3 mg).
3. Bring the bottle of carbon tetrachloride (CCl₄) into a fume hood. And take carbon tetrachloride (20 mL) into a flask.
4. Add the prepared 3-(1-naphthyl)-L-alanine into the flask.
5. Return the original bottles to the designated store locations.
6. Do the desired experiments in ATR-IR setup.
7. Used and remained solutions need to be treated as hazardous wastes.

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

Approval Date: 09/29/2022

3-(2-Naphthyl)-D-alanine 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 3-(2-naphthyl)-D-alanine ($C_{13}H_{13}NO_2$ CAS No. 76985-09-6) is used in laboratory. Its purpose is not to have any accident or risk. 3-(2-Naphthyl)-D-alanine is harmful to aquatic life.

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

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

OSHA Hazards: not known

GHS Classification

Not a dangerous substance according to GHS

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with **3-(2-naphthyl)-D-alanine** 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 3-(2-naphthyl)-D-alanine.

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 3-(2-naphthyl)-D-alanine must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of 3-(2-naphthyl)-D-alanine and understand the hazards.

Lab workers using 3-(2-naphthyl)-D-alanine 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 3-(2-naphthyl)-D-alanine 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 3-(2-naphthyl)-D-alanine in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this 3-(2-naphthyl)-D-alanine 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 3-(2-naphthyl)-D-alanine. 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 3-(2-Naphthyl)-D-alanine/CCl₄ solution (1 mM)

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and goggles.
2. Measure 3-(2-naphthyl)-D-alanine (4.3 mg).
3. Bring the bottle of carbon tetrachloride (CCl₄) into a fume hood. And take carbon tetrachloride (20 mL) into a flask.
4. Add the prepared 3-(2-naphthyl)-D-alanine into the flask.
5. Return the original bottles to the designated store locations.
6. Do the desired experiments in ATR-IR setup.
7. Used and remained solutions need to be treated as hazardous wastes.

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

Approval Date: 10/05/2022

3-Aminopropyltriethoxysilane 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 3-aminopropyltriethoxysilane (C₉H₂₃NO₃Si, CAS No. 919-30-2) is used in laboratory. Its purpose is not to have any accident or risk. 3-Aminopropyltriethoxysilane is corrosive and harmful if swallowed or in contact with skin. It causes serious eye damage and skin irritation.

Synonyms: APTES, 3-Triethoxysilylpropylamine

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

3-Aminopropyltriethoxysilane is commercially available, and used mostly for silica surface derivation 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: Combustible Liquid, Target Organ Effect (Nerves, Liver, Kidney), Harmful by Ingestion, Corrosive

GHS Classification

- Flammable liquids (Category 4)
- Acute toxicity, Oral (Category 4)
- Acute toxicity, Dermal (Category 5)
- Skin irritation (Category 2)
- Serious eye damage (Category 1)

Signs and Symptoms of Exposure

Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Spasm, inflammation and edema of the 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-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 **3-aminopropyltriethoxysilane** 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 **3-aminopropyltriethoxysilane**.

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 **3-aminopropyltriethoxysilane** must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of **3-aminopropyltriethoxysilane** and understand the hazards.

Lab workers using **3-aminopropyltriethoxysilane** 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 **3-aminopropyltriethoxysilane** 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 < 10 mL of this 3-aminopropyltriethoxysilane in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this 3-aminopropyltriethoxysilane 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 3-aminopropyltriethoxysilane. 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.

Functionalization of silica nanospheres or P25 titania nanoparticles

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Make a waste bottle labeled as toxic hazardous waste. Review the SDS of 3-aminopropyltriethoxysilane, toluene, and P25 titania again; especially remind first aid measures, handling and storage, & PPE.*
3. Place a three-neck round-bottom flask into a fume hood and put a stir bar into it. Close with rubber septum and take it to a balance. Weigh silica or titania powder (1 g) and add it into the round-bottom flask. Closed with septum and take it back to the fume hood.
4. Add toluene (50 mL), close with the septum and sonicate for about 1 minute so that the solid disperse well in the solvent.
5. Take the closed round-bottom flask to the fume hood in the room 162. Attach it to a condenser and close the other two necks with a rubber septum. Put a thermometer probe through one septum into the mixture. Don't forget to open water running through condenser. Change the atmosphere inside the round-bottom flask to nitrogen and set the temperature to 113 °C.
6. Take 3-aminopropyltriethoxysilane from the flammable cabinet and put it into the fume hood. Add the compound (1.5 mL) into the mixture when it starts boiling.
7. Leave the reaction running for 1 day.

8. When the functionalization has finished, remove the condenser and close the middle neck of the round-bottom flask with septum. Bring it into the fume hood in the room 135. Wait for it to cool down and then wash it with ethanol 4 times.
9. Dispose the waste into the waste bottle labeled toxic hazardous waste.

Synthesis of 3-aminopropyltriethoxysilane-grafted SBA-15

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a 3-neck round-bottom flask (50 mL).
3. Pour ethanol (30 mL) into the flask
4. Add 3-aminopropyltriethoxysilane (1.5 mL) into the flask.
5. Put SBA-15 (200 mg) into the flask.
6. Prepare a reflux condenser apparatus in a fume hood.
7. Mix and reflux the mixture at 73 °C for 24 h. If left unattended in a fume hood, put a label with chemical name and hazard information.
8. Transfer the mixture into a centrifuge tube.
9. Centrifuge (3000 rpm) for 10 min.
10. Take out the excess solvent using a pipette.
11. Wash the particles with ethanol (25 mL).
12. Centrifuge (3000 rpm) for 10 min.
13. Take out the excess ethanol using a pipette.
14. Repeat steps 11-13 once more.
15. Dispose waste in the properly labeled container.
16. Dry the white powder in a vacuum chamber.

Synthesis of Au nanoparticles within the SBA-15 support

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry two flasks (250 mL) under nitrogen.
3. Put deionized water (100 mL) into one flask.
4. Add 3-aminopropyltriethoxysilane-grafted SBA-15 (1.0 g) into the flask
5. After the powder is dispersed evenly, add gold chloride solution (15 mL, 10 mM).
6. Maintain the suspension by stirring the mixture for 2 h at room temperature. If left unattended in a fume hood, put a label with chemical name and hazard information.
7. Transfer the mixture into a centrifuge tube.
8. Centrifuge (3000 rpm) for 10 min.
9. Take out the excess solvent using a pipette.
10. Wash the particles with DI water (25 mL).
11. Centrifuge (3000 rpm) for 10 min.

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

Synthesis of aminopropyltriethoxysilane(APTES)-grafted P25 titania

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

11. Dry the white powder.

Synthesis of Au NPs on APTES-grafted P25 titania

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

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

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

3-Mercaptopropyl triethoxysilane 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 3-mercaptopropyl triethoxysilane ($C_9H_{22}O_3SSi$, CAS No. 14814-09-6) is used in laboratory. Its purpose is not to have any accident or risk. 3-Mercaptopropyl triethoxysilane is combustible. It causes serious 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: Combustible Liquid, Irritant

GHS Classification

Flammable liquids (Category 4)

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 3-Mercaptopropyltriethoxysilane 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 (3-mercaptopropyl) triethoxysilane.

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 3-mercaptopropyl triethoxysilane must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of 3-mercaptopropyl triethoxysilane and understand the hazards.

Lab workers using 3-mercaptopropyl triethoxysilane 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 3-mercaptopropyl triethoxysilane 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 defies 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 < 15 g of this 3-mercaptopropyl triethoxysilane in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding this 3-mercaptopropyl triethoxysilane 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 3-mercaptopropyl triethoxysilane. 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.

Condensation reaction

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Weigh 0.3 g of 3-mercaptopropyl triethoxysilane on the balance.
3. Bring the reagent into the fume hood and add it into the two-neck round bottom flask for condensation reaction.
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.

epi-Quinine Tethering

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

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