Carbon dioxide
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 carbon dioxide (CO_2, CAS No. 124-38-9) used in laboratory. Its purpose is not to have any accident or risk. Carbon dioxide is extremely flammable gas. It may explode if heated.

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

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

OSHA Hazards: Compressed Gas
GHS Classification
Gas under pressure (Liquefied gas)

Signs and Symptoms of Exposure
Nausea, Dizziness, Headache, Low to medium concentrations of carbon dioxide can affect regulation of blood circulation, affect the acidity of body fluids, respiratory difficulties, At high concentrations, Breathing difficulties, Increased pulse rate, and change in body acidity. Very high concentrations can cause unconsciousness and death.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

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

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

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

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

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

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

STORAGE:

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

7. WASTE DISPOSAL

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

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

General hazardous waste disposal guidelines:

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

8. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

9. DESIGNATED AREA

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

10. SAFETY DATA SHEETS

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

11. DETAILED PROTOCOL

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

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

When working in the lab, a laboratory worker must:

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

3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) use carbon dioxide less than 1 bar in any given reaction (higher pressure REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this carbon dioxide 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 carbon dioxide. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

Replace empty gas cylinder

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

Perform IR spectroscopic Experiment

1. A supported metal catalyst disk (e.g. Pt/SiO_2) is placed in an IR vacuum cell.
2. The catalyst is heated at 150 ^oC under vacuum for 30 min in order to eliminate the adsorbed water.
3. The catalysts are than heated form 150 ^oC to 350 ^oC under 5 torr of H_2.
4. The catalyst is kept at 350 ^oC under 200 torr of hydrogen gas for 3-4 hours.
5. Hydrogen gas is removed for 15 min.
6. The catalyst is kept at 350 ^oC under 200 torr of oxygen gas for 3-4 hours.
7. Oxygen gas is removed for 15 min.
8. Repeat 4 to 7 steps three times.
9. The sample is cooled down to room or any desired temperature.
10. Background spectrum is obtained.
11. Carbon dioxide is introduced into the cell up to 10 Torr.
12. Sample spectrum is obtained.
13. Carbon dioxide is pumped out.
14. The IR cell is vented to atmosphere.

CO_2 Calibration 1

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety Goggle.
2. A supported metal catalyst disk (e.g. Pt/SiO_2) is placed in an IR vacuum cell.
3. The catalyst is heated at 150 ^oC under vacuum for 30 min in order to eliminate the adsorbed water.
4. A background spectrum is obtained.
5. Carbon dioxide is introduced into the cell up to 5 Torr.
6. A sample spectrum is obtained.
7. Add carbon dioxide (5 Torr).
8. Another sample spectrum is obtained.
9. Repeat 7 to 8 steps up to 200 Torr
10. The cell is cooled down to room or any desired temperature.
11. Carbon dioxide is pumped out.
12. The IR cell is vented to atmosphere.

CO_2 Calibration 2

1. Wear a nitrile chemical-resistant glove, flame-resistant lab coat, and safety Goggle.
2. Remove any sample pellet in a transmission IR vacuum cell.
3. Evacuate the cell for 30 min.
4. A background spectrum is measured.
5. Fill the cell with carbon dioxide (10 Torr).

6. A sample spectrum is measured.
7. Carbon dioxide is pumped out.
8. The cell is vented.

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 02/01/2013, updated 01/25/2014, 01/21/2022

Carbon monoxide
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 carbon monoxide (CO, CAS No. 630-08-0) used in laboratory. Its purpose is not to have any accident or risk. Carbon monoxide is extremely flammable gas and toxic if inhaled. It contains gas under pressure. Also it causes damage fertility or the unborn child.

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

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

OSHA Hazards: Flammable gas, Compressed Gas, Target Organ Effect (Blood, Nerves), Teratogen

GHS Classification

Flammable gases (Category 1)
Gas under pressure (Compressed gas)
Acute toxicity, Inhalation (Category 3)
Reproductive toxicity (Category 1A)
Specific target organ toxicity – repeated exposure, Inhalation (Category 1)

Signs and Symptoms of Exposure

Blood disorders

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 chemical-resistant glove. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with the chemical and usage.

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

Carbon monoxide monitors and alarms should be connected to an emergency power source. In the event of a power failure, the detection system should continue to operate without interruption, or gas systems should automatically shut down at the source. Power connections, control switches, and adjustments that affect the detection system operation should be protected from direct access by locks on the enclosures.

All gas monitoring systems should have:

- Audible and visible alarms in the following locations: gas supply location, gas use or operator room, and outside the gas use room (e.g., corridor).
- An alarm status and gas concentration readout panel located outside the gas use room.
- Local audible and visual alarms specific and distinct from fire alarm bells and signs to indicate the alarm's meaning and required personnel action.
- The toxic gas alarm level set-point set at the PEL or Threshold Limit Value.

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

All transport of carbon monoxide between on-campus locations must be conducted as follows:

- Gas cylinders must be secured to the transport vehicle (cart, motor vehicle, hand truck, etc.).
- Cylinders must be continuously attended during transport.

- Cylinders must be clearly labeled with content and hazard information.
- Cylinder caps must be in place.

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

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

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

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

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

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

STORAGE:

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

7. WASTE DISPOSAL

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

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

General hazardous waste disposal guidelines:

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

8. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

9. DESIGNATED AREA

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

10. SAFETY DATA SHEETS

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

11. DETAILED PROTOCOL

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

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

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) use carbon monoxide under 1 bar in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this carbon monoxide 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 carbon monoxide. 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) In case of carbon monoxide, ensure carbon monoxide detector is on
2) Close the main cylinder valve.
3) Slowly release pressure from regulator into hood to vent.
4) Close the regulator valves.
5) Disconnect the regulator from an empty cylinder.
6) Screw cylinder cap.
7) Deliver the empty cylinder to the stockroom or store temporally in one of hall cabinets.
8) Bring a new gas cylinder to the rack.
9) Safely secure the cylinder using chain clamp.
10) Unscrew cylinder cap.
11) Ensure the main valve is closed.
12) Unscrew the main valve cap.
13) Connect the regulator to the cylinder.
14) Fully open the regulator valves.
15) Get vacuum in the gas manifold and the regulator.
16) Closed the diaphragm valve.
17) Quickly open and close the main cylinder valve to see if the diaphragm valve is working well.
18) If the good sealing is obtained, go ahead. Otherwise, pump the gas in the line and replace the regulator.
19) Set a delivery pressure as needed.
20) Carefully release pressure from regulator.
21) Fully open the main cylinder valve if needed.

UHV #1, Victor

1. Equip the proper PPEs (flame-resistant lab coat, safety glasses, chemical-resistant nitrile gloves.
2. Ensure that the CO detector is on and in working condition
3. Safely secure the CO cylinder at the base of the chamber and make sure the bottle is in the upright position.
4. Unscrew main valve cap.
5. Attach the appropriate pressure regulator from the gas line to the cylinder.
6. Carefully adjust the outlet pressure to 20 psi.
7. Close the main valve cap and the valve next to the mechanical pump.
8. Purge the gas line by filling it with the carbon monoxide and then opening the valve to the pump.
9. Repeat 3 times to ensure that only CO is present within the line.
10. Fill the line with the carbon monoxide once again for usage
11. Use the leak valve on the chamber to leak CO into the chamber until a dosing pressure (1.0E-06 Torr) is reached.
12. Once dosing is complete, close the leak valve, evacuate the line and close the main valve.

UHV #2, RAIRS

13. Equip the proper PPEs (flame-resistant lab coat, safety glasses, chemical-resistant nitrile gloves.
14. Make sure the CO detector installed on the beams of the ceiling is working.
15. Safely secure the lecture bottle on its holder at the base of the chamber and make sure the bottle is in the upright position.
16. Unscrew main valve cap.
17. Carefully adjust the outlet pressure to 20 psi.
18. Close the main valve cap.
19. Close the valve next to the mechanical pump.
20. Fill the line with the carbon monoxide.
21. Open the valve to pump down, and then close it.
22. Fill the line with the carbon monoxide.
23. After dosing with leak valve ore preparing a gas mixture, evacuate the gas line by opening the valve to the mechanical pump.

UHV #3, Michelle

1. Ensure carbon monoxide detector is on and in working order. The detector is located in the power rack on top of Michelle system.
2. Safely secure carbon monoxide cylinder using a chain clamp or ring clamps.
3. Ensure main valve is completely closed.

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

UHV #4, Praxis

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety glasses.
2. Make sure the CO detector installed on the beams of the ceiling is working.
3. Carbon monoxide comes in a small lecture gas bottle. These are located in CS 135.
4. A gas regulator is necessary to connect the gas lecture bottle to the gas manifold. If the gas bottle is already attached to a regulator, proceed to step 5. To attach a gas regulator to a CO bottle, first make sure that the regulator is compatible with carbon monoxide. Make sure that the valve of the CO bottle is tightly closed at all times.
5. Screw on the regulator to the bottle opening and turn until finger-tight. Use a wrench to turn the connection further. The regulator will have a pressure hand knob that is a metallic rod; it is below the regulator gauges. The current regulator for CO also has a black knob on the opposite side of the metallic knob. The black knob controls gas flow rate. Make sure that the black knob is turned fully left so that the regulator is closed.
6. Slip the CO bottle into the metal ring extension that will hold the CO bottle in place at the gas manifold. Use a screwdriver to close the metal ring; make sure the closure is snug, but that it does not cause strain on the bottle. Connect the end of the regulator to the nut of the Swagelok valve on the gas manifold. Turn the nut until the connection is finger-tight and then use a wrench to finish securing the connection.
7. Make sure that the metallic hand knob that is on the side of the regulator is fully turned away from you (counterclockwise), but not so much that the metal handle comes out of the regulator. This indicates that the regulator is closed. When the hand knob is turned clockwise (or right) it opens the regulator flow. Leave the

regulator metallic knob closed and open the black knob at this point so that the first half of the regulator can be pumped out by the gas manifold pump. Open the Swagelok valve where CO is attached to evacuate the first half of the regulator. Wait until the gas manifold pump pressure gauge that is located at the bottom of the electronics cabinet reaches 20mTorr. The pressure must be at this value to indicate that a leak-proof seal was made when screwing on the regulator. Do not proceed if the pressure is above the normal pressure. You must use a wrench to tighten the nut connection in order to have a better seal and no gas leaks.

8. Now you can empty the second half of the gas regulator; this is the section between the second gauge and the lecture bottle valve. Keep the gas bottle tightly closed. Open the metal hand knob on the side of the gas regulator (clockwise) to pump out the second side of the regulator. Wait for the pump pressure to reach 20mTorr. If the gauge is higher than 20 mTorr, tighten the connection of lecture bottle and regulator. Keep the gas bottle closed tightly when doing so.
9. When the gas manifold pressure is at 20 mTorr with the regulator attached, you can proceed to test the connection by quickly opening and closing the lecture bottle to let in some CO gas to the regulator. Make sure the metal hand knob is fully counterclockwise so that no CO escapes to the second half of the regulator. Also have the Swagelok manifold valve closed to avoid pumping of the line. Wait to see if the pressure on the regulator gauge remains constant and then turn the metal knob clockwise (right) to let CO travel to the next gauge. Turn slowly as this second pressure will be the one flowing out to the chamber. Not much pressure is needed, halfway to the first main black line of the second gauge is good.
10. To introduce CO into the chamber, close the Swagelok that connects the entire manifold to the gas manifold pump. Open the Swagelok valve where the CO bottle is connected and then open the leak valve that is directly attached to the chamber. Control pressure of CO by opening or closing leak valve. CO is used during Temperature-Programmed Desorption (TPD). It is introduced into the chamber at ~130 K and desorbed from the sample crystal at about 85K. For a good TPD, the pressure of gas introduced should not exceed 2E-8 Torr. A TPD pressure between 10E-9 Torr and 12E-9 Torr is the best range in general for gasses used in Praxis. The time that gas is allowed to flow into the chamber depends on the desired experiment time.
11. When CO use is finished, close the chamber leak valve. Close the Swagelok valve that introduces CO. Close the CO lecture bottle valve and open the Swagelok valve that pumps out the manifold.
12. To remove CO from the manifold, make sure that the bottle is closed. Close the metal knob and pump out the first half of the regulator by opening the black knob. Once that side is pumped out, open the metal knob to evacuate the rest of the regulator. Make sure the gas manifold pump gauge has pumped down to 20mTorr before disconnecting the regulator from the manifold line. Store CO on the gas rack that is in CS 135.

UHV #6, NanoReactor

1. Make sure the CO detector installed on the beams of the ceiling is working.
2. The bottle must be firmly tied to the frame.
3. Connect the bottle to the manifold:
4. Connect regulator to bottle.
   b) pump down manifold.
   c) open up manifold section to air.
   d) connect manifold tubing to regulator.
   e) pump down up to the bottle's valve with the valve closed.
   f) close connection to pump when filling the section of manifold.
5. Always pump down the gas in the manifold with a pump that is connected to the exhaust line after use, and especially just before opening up manifold section to air.
6. Disconnect from manifold:
   a) make sure that the bottle's valve is closed.
   b) make sure to pump down the manifold section up to the bottle's valve.

Installation of CO tank into IR system

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Make sure the CO detector installed on the beams of the ceiling is working.
3. Close the valves of old CO tank and evacuate the residual CO remaining in the line.
4. Close the valves around the CO tank and carefully take the old CO tank off from the lines.
5. Connect the new CO tank onto the lines. Check gas-tightness using soap water. Make sure there is no leaking around the valves.
6. Place the old CO tank in designated area or container.

CO titration observed monitored RAIRS spectroscopy

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Make sure the CO detector installed on the beams of the ceiling is working.
3. Load catalyst sample (5 mg) in between the CaF_2 and copper back plate (RAIRS).
4. Assemble the cell and check optical bench clearance.
5. Connect the Teflon tubing to IR cell, and fill the cell with CCl_4.

6. Open H_2 cylinder valve to adjust gas flow by using fine-tuning valve.
7. Run H_2 through IR cell for 30 min.
8. Close H_2 valve and pump the gas line to 0.03 Torr.
9. Open CO valve and close it when the pressure reading is above 900 Torr on pressure gauge 1.
10. Run CO through IR cell for 30 min.
11. Run IR scan in sample channel.

Perform IR spectroscopic Experiment

1. Make sure the CO detector installed on the beams of the ceiling is working.
2. A supported metal catalyst disk (e.g. Pt/SiO_2) is placed in an IR vacuum cell.
3. The catalyst is heated at 150 ^oC under vacuum for 30 min in order to eliminate the adsorbed water.
4. The catalysts are than heated form 150 to 350 ^oC under 5 Torr of H_2.
5. The catalyst is kept at 350 ^oC under 200 Torr of hydrogen gas for 3-4 hours.
6. Hydrogen gas is removed for 15 min.
7. The catalyst is kept at 350 ^oC under 200 Torr of oxygen gas for 3-4 hours.
8. Oxygen gas is removed for 15 min.
9. Repeat 4 to 7 steps three times.
10. The sample is cooled down to room or any desired temperature.
11. Background spectrum is obtained.
12. Carbon monoxide is introduced into the cell up to 10 Torr.
13. Sample spectrum is obtained.
14. Carbon monoxide is pumped out.
15. The IR cell is vented to atmosphere.

CO titration using Transmittance IR spectroscopy 1

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Sample loading and open circulating water.
3. Pump the cell at room temperature.
4. Heat the cell at 150 ^oC for 2 h.
5. Increase the temperature to 350 ^oC
6. Heat the cell at 350 ^oC with oxygen around 200 Torr for 1 h
7. Pump the cell.
8. Heat the cell at 350 ^oC with hydrogen around 200 Torr for 1 h
9. Pump the cell.
10. Repeat the process for 3 times
11. Stop heating and add liquid nitrogen

12. Take IR spectra from 350 °C to -150 °C each 10 °C
13. Fill the cell with carbon monoxide around 20 Torr for 10 min
14. Pump the cell.
15. Start heating.
16. Take IR spectra from -150 °C to 350 °C each 10 °C
17. Stop heating.

CO titration using Transmittance IR spectroscopy 2

1. Equip the proper PPEs (flame-resistant lab coat, safety glasses, chemical-resistant nitrile gloves.
2. Make sure the CO detector installed on the beams of the ceiling is working.
3. Safely secure the lecture bottle on its holder at the base of the chamber and make sure the bottle is in the upright position.
4. Unscrew main valve cap.
5. Carefully adjust the outlet pressure to 20 psi.
6. Close the main valve cap.
7. Close the valve next to the mechanical pump.
8. Fill the line with the carbon monoxide.
9. Open the valve to pump down, and then close it.
10. Fill the line with the carbon monoxide.
11. After dosing with leak valve ore preparing a gas mixture, evacuate the gas line by opening the valve to the mechanical pump.

CO Oxidation

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Sample loading and open circulating water.
3. Pump the cell at room temperature.
4. Heat the cell at 150 °C for 2 h.
5. Increase the temperature to 350 °C.
6. Heat the cell at 350 °C with oxygen around 200 Torr for 2 h.
7. Pump the cell.
8. Heat the cell at 350 °C with hydrogen around 200 Torr for 2 h.
9. Pump the cell.
10. Set the cell to 150 °C.
11. Take an IR background spectrum.
12. Mix O2 (250 Torr) and CO (250 Torr).
13. Take IR spectra at 1, 10, 20, 30, 40, 50, and 60 min.
14. Run the same sample continuously for 5 times.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 10/01/2013, updated 03/01/2014, 03/01/2016, 06/01/2017, 07/01/2018, 01/15/2021

Carbon tetrachloride
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 carbon tetrachloride (CCl_4, CAS No. 56-23-5) is used in laboratory. Its purpose is not to have any accident or risk. Especially carbon tetrachloride is toxic and a CAL/OHSA Select Carcinogen, so may cause cancer and heritable genetic damage. It is toxic by inhalation, in contact with skin and if swallowed. It causes serious damage to health by prolonged exposure through inhalation. Carbon tetrachloride is used as a common solvent for cinchonidine projects in Zaera group, because there are no significant absorption IR bands above 1600 cm^{-1}. So, please be very careful when you handle, or replace it with other solvents if possible.

Synonyms: Tetrachloromethane

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

Please refer its MSDS always before using them.

OSHA Select Carcinogen

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

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

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

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

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

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

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

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

OSHA Hazards: Carcinogen, Target Organ Effect (Nerves, Liver, Eyes, Heart and Kidney), Toxic by Inhalation, Ingestion, and Skin Absorption

GHS Classification

Acute toxicity, Oral (Category 3)

Acute toxicity, Inhalation (Category 3)

Acute toxicity, Dermal (Category 3)

Skin irritation (Category 3)
Eye irritation (Category 2B)
Carcinogenicity (Category 2)
Specific target organ toxicity – repeated exposure (Category 1)
Acute aquatic toxicity (Category 3)
Chronic aquatic toxicity (Category 3)
Hazardous to the ozone layer (Category 1)

Signs and Symptoms of Exposure

Vomiting, Diarrhea, Abdominal pain, Nausea, Dizziness, Headache, Damage to the eyes., Liver injury may occur., Kidney injury may occur., Exposure to and/or consumption of alcohol may increase toxic effects., Contact with skin can cause:, Pain, Erythema, hyperemia

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Respiratory Protection

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

b. Eye Protection

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

c. Skin and Body Protection

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

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

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

d. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with carbon tetrachloride 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 carbon tetrachloride.

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

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

When working in the lab, a laboratory worker must:

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

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

Liquid-Solid IR Cell Experiment

1. Carbon tetrachloride is used as a solvent in the experiment. Before preparing the solution, consult the solute's SOPs to avoid any unexpected contamination.
2. Do not make excess solution. Preparing the solution right upon the beginning of every experiment.
3. Treat used solutions as chemical wastes immediately.

Surface modification of Pt in CCl_4 monitored by ATR-IR

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and *full-face respirator*.
2. *Make a waste bottle labeled as carcinogen hazardous waste. Review the SDS of CCl_4 again; especially remind first-aid measures, handling and storage, PPE, & signs and symptoms of exposure.*
3. Load catalyst sample (5 mg) onto an ATR crystal.
4. Place the ATR crystal in a fume hood and add a few drops of CCl_4 to the sample. *Keep the CCl_4 container tightly closed in a dry and well-ventilated place. The container must be carefully resealed and kept upright to prevent leakage*.
5. Make them evenly dispersed on the crystal.
6. Leave them in the fume hood until the sample is fully dried.
7. Connect the Teflon tubing to IR cell, and fill the cell with CCl_4. *Be careful not to spill CCl_4. Keep watching any leak of CCl_4. 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.*
8. Open H_2 cylinder valve to adjust gas flow by using fine-tuning valve.
9. Run H_2 through IR cell for 30 min. *Keep watching any leak of CCl_4*.

10. Close H_2 valve and flush the cell using CCl_4 solution that has the modifier dissolved in it. *Keep watching any leak of CCl_4*.
11. Run IR scan in sample channel.
12. Pump the gas line and check pressure and the equilibrium reading should be 0.03 torr.
13. *Withdraw all solution left in ATR cell with a 10 mL syringe and transfer it to the carcinogen waste container in a fume hood. Be careful not to spill CCl_4. Keep watching any leak of CCl_4.*
14. Dry ATR crystal in a fume hood and wash it with ethanol thoroughly.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 11/01/2013, updated 03/25/2014, 03/01/2016

Chloroform
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 Chloroform ($CHCl_3$, CAS No. 67-66-3) is used in laboratory. Its purpose is not to have any accident or risk. Chloroform is a CAL/OHSA Select Carcinogen, so may cause cancer and genetic damage. It also causes serious eye and skin irritation. It may be harmful if swallowed.

Synonyms: Trichloromethane, Methylidyne trichloride

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

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

OSHA Select Carcinogen

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

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

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

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

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

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

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

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

OSHA Hazards: Carcinogen, Target Organ (Central nervous system, Blood, Liver, Cardiovascular system, Kidney) Effect, Harmful by Ingestion, Irritant

GHS Classification

Acute toxicity, Oral (Category 4)
Skin irritation (Category 2)
Eye irritation (Category 2A)
Carcinogenicity (Category 2)
Specific target organ toxicity – repeated exposure (Category 2)
Acute aquatic toxicity (Category 3)

Signs and Symptoms of Exposure

Vomiting, Gastrointestinal disturbance, Exposure to and/or consumption of alcohol may increase toxic effects.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with chloroform 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 chloroform.

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

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

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 100 mg of this chloroform in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this chloroform 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 chloroform. 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 nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Introduce chloroform (20 mL) into a two-neck round bottom, followed by adding the reagents and catalyst. All the experiments are conducted in a fume hood.
3. After reaction, the removed solvent needs to be treated as hazardous waste.
4. 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 goggle
2. 9-amino(9-deoxy)*epi*-quinine (1.08 g, 0.3 *m*mol) and AIBN (azoisobutyronitrile) (54 mg, 0.33 *m*mol) 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 *m*mol) is added into the flask.
5. The flask is refluxed at 63 ^{o}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 ^{o}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: 06/01/2013, updated 03/01/2014, 02/07/2020

Chloroform-*d*
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 chloroform-*d* (CCl_3D, CAS No. 865-49-6) is used in laboratory. Its purpose is not to have any accident or risk. Chloroform-*d* is a CAL/OHSA Select Carcinogen, so may cause cancer and genetic damage. It also causes serious eye and skin irritation. It may be harmful if swallowed.

Synonyms: Deuterochloroform

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

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

OSHA Select Carcinogen

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

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

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

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

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

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

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

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

OSHA Hazards: Carcinogen, Target Organ Effect (Kidney, Liver, Cardiovascular system, Central nervous system, Blood), Harmful by Ingestion, Irritant

GHS Classification

Acute toxicity, Oral (Category 4)
Skin irritation (Category 2)
Eye irritation (Category 2A)
Carcinogenicity (Category 2)
Specific target organ toxicity – repeated exposure (Category 2)

Signs and Symptoms of Exposure

Vomiting, Gastrointestinal disturbance, Exposure to and/or consumption of alcohol may increase toxic effects., 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 chloroform-*d* 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 chloroform-*d*.

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

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

When working in the lab, a laboratory worker must:

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

1. Wear nitrile chemical-resistant glove, flame-resistant lab coat, and safety goggles.
2. Bring the sample for NMR measurement and chloroform-*d* into a fume hood.

3. A proper amount of NMR sample is placed in a NMR tube and fill with chloroform-*d* (0.5 mL).
4. Bring the NMR tubes in a second container to the NMR room carefully.
5. After measurement, the sample solution needs to be treated as hazardous waste.
6. Washing and cleaning solvents also need to be treated as hazardous waste.

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

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

Chloroplatinic acid hexahydrate
STANDARD OPERATING PROCEDURE

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

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when chloroplatinic acid hexahydrate ($H_2PtCl_6 \cdot 6H_2O$, CAS No. 18497-13-7) is used in laboratory. Its purpose is not to have any accident or risk. Chloroplatinic acid hexahydrate is corrosive and toxic if swallowed. It causes severe skin burns and eye damage. It is commercially available, and used mostly for synthesis of platinum nanoparticles in Zaera group. A variety of organic solvents are used to clean sample containers. Platinum compounds, especially platinum halogen complexes, are sensitizers. Sensitized persons on a re-exposure to platinum salts will show the clinical features of a Type 1 allergy: asthma and/or rhinitis and/or conjunctivitis and/or urticaria. A contact dermatitis may also occur. Please refer MSDS for the details.

Synonyms: Dihydrogen hexachloroplatinate hexahydrate, Hexachloroplatinate acid hexahydrate, Platinic chloride hexahydrate

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

Please refer their MSDS always before using them.

OSHA Hazards: Corrosive, Toxic by Ingestion, Respiratory sensitizer
GHS Classification
- Acute toxicity, Oral (Category 3)
- Skin corrosion (Category 1B)
- Serious eye damage (Category 1)
- Respiratory sensitization (Category 1)

Signs and Symptoms of Exposure
Cough, 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:

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- Store hazardous waste in closed containers, in secondary containment, and in a designated location. Do not let product enter drains. Discharge into the environment must be avoided.
- Double-bag dry waste using transparent bags.
- Waste must be under the control of the person generating and disposing of it.
- Dispose of routinely generated chemical waste within 90 days.

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

When working in the lab, a laboratory worker must:

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

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

Pt Catalyst Preparation 1

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Bring chloroplatinic acid hexahydrate bottle to a balance
3. Carefully transfer the designated amount of chloroplatinic acid hexahydrate into the designated vessel rapidly. Close and seal the bottle.
4. Dissolve chloroplatinic acid hexahydrate with milli-Q water and mix with support material (e.g. SiO_2, TiO_2, or Al_2O_3).
5. Heat and dry under designated conditions.
6. Any extra unused solution needs to be treated as hazardous waste.

Pt Catalyst Preparation 2

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (50 mL) and a beaker (50 mL) under nitrogen in a fume hood and put a stir bar into the flask.
3. Put sodium hydroxide solution (0.25 g, 12.5 mL, 0.5 M) in the beaker.
4. Put ethylene glycol (12.5 mL) in the beaker.
5. Put chloroplatinic acid hexahydrate (0.25 g, 0.48 *m*mol) in the flask.
6. Put ethylene glycol (12.5 mL) into the flask.
7. Add sodium hydroxide solution to chloroplatinic acid hexahydrate solution.
8. Heat the solution at 160 °C for 3 h, accompanied by N_2 bubbling.

9. Transfer 6 mL aliquot of the resulting solution to a vial.
10. Add hydrogen chloride solution (1 mL of 2 M) and disperse in ethanol containing polyvinylpyrrolidone (12.2 mg).
11. Evaporate the solution.

Pt Catalyst Preparation 3

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dissolve chloroplatinic acid hexahydrate in milli-Q water.
3. Use the solution as the precursor for catalyst preparation.
4. Mix the designated amount of the solution with SBA-15 in a flask.
5. Dry the mixture at room temperature for 24 h.
6. Dry the mixture in oven for 24 h.

PtCu Catalyst Preparation

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Prepare a chloroplatinic acid hexahydrate solution (20 g/L, $H_2PtCl_6 \cdot 6H_2O$ in water).
3. Prepare a copper (II) nitrate hemi(pentahydate) solution (20 g/L, $Cu(NO_3)_2 \cdot 2.5H_2O$ in water).
4. Mix the chloroplatinic acid hexahydrate (0.604 mL) and the copper (II) nitrate (0.091 mL) solutions in the beaker #1 (10 mL).
5. Add a certain amount of D.I. water into the beaker #2 (50 mL).
6. Add the mixture solution (step 4, the beaker #1) to the beaker #2 with SBA-15 (500 mg) dropwise.
7. Mix the SBA-15 with water during each drop with a glass rod.
8. Add D.I. water (0.5 mL) to the beaker #1,
9. Add the water in the beaker #1 to the beaker #2 dropwise.
10. Mix the mixture between each drop.
11. After the mixture becomes colloidal, stop adding water.
12. Keep the mixture under room temperature for 24 h.
13. Cover the beaker of mixture with the aluminum foil.
14. Keep the beaker at 70 ^oC for 24 h.
15. Grind the mixture into powder.
16. Reduce the powder catalyst in furnace under H_2 flow for 3 h at 623 K.

Preparation of Chloroplatinic acid hexahydrate solution (20 g/L)

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, and safety goggles
2. Wash a volumetric flask (50 mL) with DI water
3. Add DI water (2~3 mL) and Chloroplatinic acid hexahydrate(1 g) to the chemical container. Mix solution with a plastic pipette.
4. Transfer the solution completely into a volumetric flask, and rinse the container 3 times with DI water. Transfer residue into volumetric flask also.
5. Add DI water to the 50 mL mark on the volumetric flask.
6. Transfer the solution into a glass bottle.
7. Rinse the volumetric flask with DI water, and put this liquid into a hazardous waste bottle.

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 02/01/2013, updated 06/01/2017, 07/01/2018, 01/23/2019, 10/10/2022

Chlorotrimethylsilane
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 chlorotrimethylsilane (C_3H_9ClSi, CAS No. 74-77-4) used in laboratory. Its purpose is not to have any accident or risk. Chlorotrimethylsilane is highly flammable liquid and vapor, and toxic if swallowed or if inhaled. Harmful in contact with skin. It causes serious eye damage, skin burns, and respiratory irritation.

Synonyms: TMCS, Trimethylchlorosilane, Trimethylsilyl chloride

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

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

OSHA Hazards: Flammable liquid, Toxic by Inhalation, Ingestion
GHS Classification

- Flammable liquids (Category 2)
- Acute toxicity, Oral (Category 3)
- Acute toxicity, Inhalation (Category 3)
- Acute toxicity, Dermal (Category 4)
- Skin corrosion (Category 1A)
- Serious eye damage (Category 1)
- Specific target organ toxicity – single exposure (Category 3), Respiratory system

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

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

Lab workers using chlorotrimethylsilane 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 chlorotrimethylsilane described in this SOP. The researcher must also consult the PI or designated, experienced research coworker for approval to proceed with chemical or biological transformations that have little literature

or local research group precedent. PI approval must also be obtained for significant scale-up (PI defines scale) of new chemistry or biological transformations.

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 10 mL of this chlorotrimethylsilane in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this chlorotrimethylsilane 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 chlorotrimethylsilane. 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.

SBA-15 Silylation 1

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

SBA-15 Silylation 2

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

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 07/08/2020, 07/10/2020

Cinchonidine
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 cinchonidine ($C_{19}H_{12}N_2O$, CAS No. 458-71-2) is used in laboratory. Its purpose is not to have any accident or risk. Cinchonidine has Teratogen hazard. It is |harmful if swallowed, and suspected of damaging the unborn child. Also it may cause an allergic skin reaction and damage to organ through prolonged or repeated exposure if swallowed.

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

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

OSHA Hazards: Harmful by Ingestion, Skin Sensitizer, Teratogen
GHS Classification

Acute toxicity, Oral (Category 4)
Skin sensitization (Category 1)
Reproductive toxicity (Category 2)
Specific target organ toxicity – repeated exposure, Oral (Category 2), Heart, Immune system

Signs and Symptoms of Exposure
Effects due to ingestion may include Rash, Itching, Shortness of breath, photosensitivity of the skin.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with cinchonidine 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 cinchonidine.

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

Lab workers using cinchonidine 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 cinchonidine 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 cinchonidine in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this cinchonidine 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 cinchonidine. 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 nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Weight cinchonidine (1 g).
3. Bring the reagent into a fume hood and add it into a two-neck round bottom flask for reaction.
4. After reaction, the removed solvent needs to be treated as hazardous waste.
5. Washing and cleaning solvents also need to be treated as hazardous waste

Surface modification using Cinchonidine for Et-Py hydrogenation

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Transfer toluene (10 mL) or any other solvent to a beaker (50 mL)
3. Weigh out cinchonidine (5 mg) and transfer it to the beaker.
4. Stir the mixture for 2 minutes and then untrasonicate the slurry for 10 min until all cinchonidine solid is dissolved.
5. Seal the beaker with Para-film.
6. Rinse the stainless cylinder of the high-pressure reactor with ethanol and toluene. Let it dry before adding reactants.
7. Add Pt/Al_2O_3 or other catalyst (1 wt.%, 25 mg) into the cylinder.
8. Inject toluene (4 mL) or any other solvent into the cylinder.
9. Add the ethyl pyruvate/toluene solution (1 mL of a preselected concentration)
10. Transfer the surface modifier solution prepared in step 4 to the cylinder. Place a magnetic stirring bar inside.
11. Mount the cylinder onto the fixed head of the reactor. Tighten all 6 screws and mount the outer band in position.
12. Pressurize the cylinder using UHP H_2 .
13. Start the reaction.

14. When the reaction is done, open the outlet valve to release the pressure.
15. Take a sample for GC analysis.

A modifier for the selective hydrogenation for furfural

1. Wear chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Weigh Cinchonidine (10 mg) on an electronic balance.
3. Transfer and dissolve the sample to a beaker by 1,4-dioxane (~25 mL).
4. Pour the solutions into the volumetric flask (100 mL), using a glass rod.
5. Wash the rod and rinse the beaker several times with 1,4-dioxane.
6. Pour these washings into the volumetric flask.
7. Top up the volumetric flask with 1,4-dioxane, until just below the graduation mark.
8. Top up to the graduation mark with a dropper.
9. Invert and mix to ensure proper mixing of the contents.
10. Transfer the standard solution of Cinchonidine (0.1 mg/mL) into a reagent bottle.
11. Draw Cinchonidine standard solution (2–25 mL) into the reaction mixture. Then reaction under certain conditions. After that, analyze each component by GC.

Addition Reaction with Cinchonidine (Cd)

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 03/01/2014, 04/22/2016, 08/14/2018, 10/01/2022

Cinnamaldehyde
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 cinnamaldehyde (C_9H_8O, CAS No. 104-55-2) used in laboratory. Its purpose is not to have any accident or risk. It causes serious eye and skin irritation. Also it may be harmful if swallowed or in contact with skin.

Synonyms: 3-Phenylprop-2-enal

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)
- Skin sensitization (Category 1)
- Specific target organ toxicity – single exposure (Category 3)

Signs and Symptoms of Exposure
burning sensation, Cough, wheezing, laryngitis, Shortness of breath, Headache, Nausea, Vomiting, 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 cinnamaldehyde 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 cinnamaldehyde.

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

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

UHV #1, Victor Chamber_1

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

UHV #1, Victor Chamber_2

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and full-face respirator.
2. Bring required materials to a fume hood: cinnamaldehyde, a syringe, and a glass tube connected to a Swagelok Valve.
3. Secure the glass tube on the 3-prong clamp.
4. Carefully open the cinnamaldehyde bottle. The bottle must be kept upright at all times to prevent leakage.
5. Use a syringe to transfer cinnamaldehyde (2 mL) from the bottle into the glass tube through the connected Swagelok valve.
6. Close the Swagelok valve and make sure there is no leak.
7. Connect the Swagelok valve to the gas manifold on the chamber.
8. Close the cinnamaldehyde bottle. Check for any leaks of cinnamaldehyde.
9. Put the cinnamaldehyde bottle back to the designated storage cabinet. Keep the cinnamaldehyde container tightly closed in a dry and well-ventilated place.
10. Clean the syringe with acetone and place the waste liquid into the waste container prepared with a carcinogen hazardous label.

UHV #3, NanoReactor

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Bring required materials to a fume hood: cinnamaldehyde, a syringe, a glass tube with fitting to the gas manifold.
3. Secure the glass tube on the 3-prong clamp.
4. Open the cinnamaldehyde bottle.
5. Use a syringe to transfer cinnamaldehyde from the original bottle into the glass tube.
6. Close the cinnamaldehye bottle. Connect the glass tube to the gas manifold of UHV #3, NanoReactor
7. Put the cinnamaldehyde bottle back to the storage place.
8. Clean the syringe with acetone and place the waste liquid into a proper waste container.

Catalytic Hydrogenation of Cinnamaldehyde

1. Wear nitrile chemical resistant gloves, flame-resistant lab coat, and safety goggles.
2. Catalyst (50–200 mg), cinnamaldehyde (0.5–3 *m*mol), and solvent (100 mL) are added into a reactor.
3. Sonicate and stir the mixture.
4. The mixture is purged with pure H_2 (1.0 MPa) five times.

5. The reactor is pressurized to a desired H_2 pressure (2.0 MPa) at room temperature.
6. The reactor is heated to a desired temperature.
7. Begin stirring (900 rpm) and set reaction time to start.
8. Sample (1.0 mL) is taken periodically to determine conversion and selectivity during the reaction process.
9. The catalyst powder is filtered off.
10. The filtrate is analyzed using GC.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 04/01/2016, updated 10/10/2017, 06/23/2021

Copper acetylacetonate
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 copper acetylacetonate ($C_{10}H_{14}CuO_4$, CAS No. 13395-16-9) is used in laboratory. Its purpose is not to have any accident or risk. Copper acetylacetonate causes skin and eye irritation. It may be harmful if inhaled or if swallowed.

Synonyms: Bis(2,4-pentanedionato)copper(II), 2,4-Pentanedionecopper(II) derivative, Cupric acetylacetonate, $Cu(acac)_2$

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
Symptoms of systemic copper poisoning may include: capillary damage, headache, cold sweat, weak pulse, and kidney and liver damage, central nervous system excitation followed by depression, jaundice, convulsions, paralysis, and coma. Death may occur from shock or renal failure. Chronic copper poisoning is typified by hepatic cirrhosis, brain damage and demyelination, kidney defects, and copper deposition in the cornea as exemplified by humans with Wilson's disease. It has also been reported that copper poisoning has lead to hemolytic anemia and accelerates arteriosclerosis. Damage to the eyes., May liberate 2,4-pentanedione upon decomposition. 2,4-Pentanedione has the following toxicological hazards: toxic, irritant, neurological hazard, teratogen, possible mutagen, target organ - thymus. In humans, 2,4-pentanedione is reported to cause contact dermatitis and contact urticaria.

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 copper acetylacetonate 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 copper acetylacetonate.

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

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

Synthesis of Cu@Pt nanoparticle

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (25 mL) under nitrogen in a fume hood.
3. Put copper acetylacetonate (2.04 mg, 0.0078 *m*mol) into the flask.
4. Take ethylene glycol (8 mL) by a syringe in a fume hood and add it slowly to the flask.
5. Rinse syringes several times with ethanol before disposal.
6. The washings should be disposed as hazardous organic waste.
7. Put a syringe needle into sharps-disposal container.
8. Put polyvinylpyrrolidone(5.8 mg) into the flask.
9. Change the pH with NaOH (0.1 M) to pH=10 and close the flask with rubber septum.
10. Reflux the mixture under nitrogen for 20 minutes at 198 ^{o}C. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
11. After cooling to 80 ^{o}C, add platinum acetylacetonate (3.07 mg, 0.0078 *m*mol) and slowly heat the mixture back to reflux for 1 h.
12. Remove the septum from the flask and put SBA-15 (402.93 mg) in the flask and stir the mixture for 2 h.
13. Sonicate the mixture for 1 h.
14. Centrifuge and dispose the liquid

15. Evacuate the flask containing the sample then dry the powder at ca. 60 ^{o}C for 12 h in the fume hood.
16. Grind the product to powder with an agate mortar and pestle
17. Wash the powder with acetone and acetone-ethanol mixture upon sonication for 5 times and centrifuge each time after it's washed.
18. Dry the powder under vacuum in the flask at 60 ^{o}C for 12 h.

ALD Operation

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

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 11/01/2016, updated 11/20/2019

Copper sulfate pentahydrate
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 copper sulfate pentahydrate ($CuO_4S{\cdot}5H_2O$, CAS No. 7758-99-8) used in laboratory. Its purpose is not to have any accident or risk. Copper sulfate pentahydrate is toxic if swallowed. It causes serious eye and skin irritations. Also it may be harmful in contact with skin.

Synonyms: Cupric sulfatepentahydrate

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

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

OSHA Hazards: Toxic by Ingestion, Target organ effect (Liver, Kidney, and Blood)
GHS Classification

Acute toxicity, Oral (Category 3)
Acute toxicity, Dermal (Category 5)
Skin irritation (Category 2)
Eye irritation (Category 2A)
Acute aquatic toxicity (Category 1)

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with copper sulfate pentahydrate 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 copper sulfate pentahydrate.

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

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

Click Chemistry

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Weight 0.050 g of copper sulfate pentahydrate.
3. Bring the reagent into the fume hood and add it into a four-neck round bottom flask for reaction.
4. After reaction, the removed solvent needs to be treated as hazardous waste.
5. Washing and cleaning solvents also need to be treated as hazardous waste

Synthesis of PtCu alloy

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

12. Sonicate the mixture for 1 h.
13. Centrifuge and dispose the liquid.
14. Evacuate the flask containing the sample then dry the powder at ca. 60 °C for 12 h in the fume hood.
15. Grind the product to powder with an agate mortar and pestle
16. Wash the powder with acetone and acetone-ethanol mixture upon sonication for 5 times and centrifuge each time after it's washed.
17. Dry the powder under vacuum in the flask at 60 °C for 12 h.

Synthesis of Pt@Cu nanoparticle

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (25 mL) under nitrogen in a fume hood.
3. Put platinum acetylacetonate (18.582 mg, 0.04725 *m*mol) into the flask.
4. Take ethylene glycol (5.775 mL) by a syringe in a fume hood and add it slowly to the flask.
5. Rinse syringes several times with ethanol before disposal.
6. The washings should be disposed as hazardous organic waste.
7. Put a syringe needle into sharps-disposal container.
8. Put polyvinylpyrrolidone (2.82mg) into the flask.
9. Change the pH with NaOH (0.1 M) to pH=10 and close the flask with rubber septum
10. Reflux the mixture under nitrogen for 2 h at 198 °C. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
11. After cooling to 80 °C, add Copper sulfate pentahydrate (11.814 mg, 0.04725 *m*mol) and slowly heat the mixture back to reflux for 2 h.
12. Remove the septum from the flask and put SBA-15 (598.455 mg) in the flask and stir the mixture for 2 h.
13. Sonicate the mixture for 1 h.
14. Centrifuge and dispose the liquid
15. Evacuate the flask containing the sample then dry the powder at ca. 60 °C for 12 h in the fume hood.
16. Grind the product to powder with an agate mortar and pestle
17. Wash the powder with acetone and acetone-ethanol mixture upon sonication for 5 times and centrifuge each time after it's washed.
18. Dry the powder under vacuum in the flask at 60 °C for 12 h.

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

Approval Date: 06/01/2013, updated 03/01/2014, 11/01/2016

Copper (II) nitrate hemi(pentahydrate)
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 copper (II) nitrate hemi(pentahydrate) (CuN_2O_6, CAS No. 19004-19-4) used in laboratory. Its purpose is not to have any accident or risk. Copper (II) nitrate hemi(pentahydrate) is strong oxidizer, so it may cause fire or explosion. It may be corrosive to metals. It is harmful if swallowed and very toxic to aquatic life. It causes severe skin burns and eye damage.

Synonyms: Cupric nitratehemi(pentahydrate)

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

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

OSHA Hazards: Oxidizer, Harmful by swallowed, Skin burns, Eye Damage.
GHS Classification

- Oxidizing solids (Category 1)
- Corrosive to metals (Category 1)
- Acute toxicity, Oral (Category 4)
- Skin corrosion (Cagegory 1B)
- Serious eye damage (Category 1)
- Acute aquatic toxicity (Category 1)
- Chronic aquatic toxicity (Category 1)

Signs and Symptoms of Exposure
Capillary damage, headache, cold sweat, weak pulse, and kidney and liver damage, central nervous system excitation followed by depression, jaundice, convulsions, paralysis, and coma. Death may occur from shock or renal failure. Chronic copper poisoning is typified by hepatic cirrhosis, brain damage and demyelination, kidney defects, and copper deposition in the cornea as exemplified by humans with Wilson's disease. It has also been reported that copper poisoning has lead to hemolytic anemia and accelerates arteriosclerosis. Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Cough, Shortness of breath, Headache. Stomach - Irregularities - Based on Human Evidence

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 copper(II) nitrate hemi(pentahydrate) 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 copper(II) nitrate hemi(pentahydrate).

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

Lab workers using copper(II) nitrate hemi(pentahydrate) 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 copper(II) nitrate hemi(pentahydrate) described in this

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

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 20 g of this copper(II) nitrate hemi(pentahydrate) in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this copper(II) nitrate hemi(pentahydrate) 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 copper (II) nitrate hemi(pentahydrate). 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.

PtCu Catalyst Preparation

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Prepare a dihydrogen hexachloroplatinate hexahydrate solution (20 g/L, $H_2PtCl_6 \cdot 6H_2O$ in water).
3. Prepare a copper (II) nitrate hemi(pentahydate) solution (20 g/L, $Cu(NO_3)_2 \cdot 2.5H_2O$ in water).
4. Mix the dihydrogen hexachloroplatinate (0.604 mL) and the copper (II) nitrate (0.091 mL) solutions in the beaker #1 (10 mL).
5. Add a certain amount of D.I. water into the beaker #2 (50 mL).
6. Add the mixture solution (step 4, the beaker #1) to the beaker #2 with SBA-15 (500 mg) dropwise.
7. Mix the SBA-15 with water during each drop with a glass rod.
8. Add D.I. water (0.5 mL) to the beaker #1,
9. Add the water in the beaker #1 to the beaker #2 dropwise.
10. Mix the mixture between each drop.
11. After the mixture becomes colloidal, stop adding water.

12. Keep the mixture under room temperature for 24 h.
13. Cover the beaker of mixture with the aluminum foil.
14. Keep the beaker at 70 ^{o}C for 24 h.
15. Grind the mixture into powder.
16. Reduce the powder catalyst in furnace under H_2 flow for 3 h at 623 K.

Synthesis of Cu/SBA-15

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Put the L-ascorbic acid (1 g) into a flask (50 mL).
3. Add water (12.5 mL) to the flask.
4. Prepare a copper nitrate hemi(pentahydrate), $Cu(NO_3)_2{\cdot}2.5H_2O$, solution (20 mg/mL).
5. Add the copper precursor solution (0.64 mL) into the flask.
6. Add SBA-15 (0.1 g) into the flask.
7. Stir (200 rpm) at RT for 48 h.
8. Wash the mixture with H_2O/ethanol 2 times
9. Dry the powder.

Synthesis of Pt@Cu_6/SBA-15, #1

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Put the L-ascorbic acid (1 g) in to a flask (50 mL).
3. Add water (12.5 mL) to the flask.
4. Add Pt nanoparticle solution (15 mL) into the flask.
5. Prepare a copper nitrate hemi(pentahydrate), $Cu(NO_3)_2{\cdot}2.5H_2O$, solution (20 mg/mL).
6. Add the copper precursor solution (0.64 mL) into the flask.
7. Stir (200 rpm) the mixture at RT for 16 h.
8. Add SBA-15 (0.1 g) into the flask.
9. Stir (200 rpm) at RT for 48 h.
10. Wash the mixture with H_2O/ethanol 2 times
11. Dry the powder.

Synthesis of Pt@Cu_6/SBA-15, #2

1. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. Put the L-ascorbic acid (1 g) in to a flask (50 mL).
3. Add water (25 mL) to the flask.

4. Add Pt/SBA-15 (0.1 g) into the flask.
5. Prepare a copper nitrate hemi(pentahydrate), $Cu(NO_3)_2 \cdot 2.5H_2O$, solution (20 mg/mL).
6. Add the copper precursor solution (0.64 mL) into the flask.
7. Stir (200 rpm) the mixture at RT for 16 h.
8. Wash the mixture with H_2O/ethanol 2 times
9. Dry the powder.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 01/26/2019, updated 01/21/2022

Crotonaldehyde
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 crotonaldehyde (C_4H_6O, CAS No. 123-73-9) used in laboratory. Its purpose is not to have any accident or risk. Crotonaldehyde is highly flammable liquid and vapor, and Lachrymator. 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, Toxic by inhalation, Toxic by ingestion, Toxic by skin absorption, Skin sensitizer, Irritant, Mutagen

GHS Classification

Flammable liquids (Category 2)
Acute toxicity, Oral (Category 3)
Acute toxicity, Inhalation (Category 2)
Acute toxicity, Dermal (Category 3)
Skin irritation (Category 2)
Serious eye damage (Category 1)
Skin sensitization (Category 1)
Germ cell mutagenicity (Category 2)
Specific target organ toxicity – single expose (Category 3)
Specific target organ toxicity – repeated expose (Category 2)
Acute aquatic toxicity (Category 1)

Signs and Symptoms of Exposure

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Respiratory Protection

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

b. Eye Protection

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

c. Skin and Body Protection

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

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

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

d. Hand Protection

At a minimum, wear 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 crotonaldehyde 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 crotonaldehyde.

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

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

UHV #1, Victor Chamber_1

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and full-face respirator.
2. Bring the crotonaldehyde bottle to the fume hood in room 135. Open the crotonaldehyde bottle. Use a flint glass pipette to transfer crotonaldehyde (~ 2 mL) into the sealed-off Pyrex glass end tube with 1’33 flange and Swagelok valve.
3. Close the crotonaldehyde bottle. Clean the pipette with acetone and place the waste liquid into a waste container. Put the crotonaldehyde bottle back to the storage place.
4. Connect the glass tube to the leak valve on the UHV system and to the gas line.

5. Submerge the glass tube in liquid nitrogen to freeze crotonaldehyde and slowly open the valve on the gas line to mechanical pump to pump down the frozen liquid sample.
6. Close the valve and thaw crotonaldehyde to release trapped air.
7. Repeat the freeze and pump steps until the pressure does not increase any more when the valve is opened.
8. Fill the delivery line with the vapor of crotonaldehyde by opening and closing the valve on the glass tube.
9. Open the leak valve to dose the crotonaldehyde gas into the chamber for experiments.
10. After experiments, clean the glass tube with acetone and place the waste liquid into the waste container.

UHV #1, Victor Chamber_2

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and full-face respirator.
2. Make a waste bottle labeled as carcinogen hazardous waste. Review the SDS of crotonaldehyde. Make sure to memorize the first-aid measures, handling and storage, PPE, & signs and symptoms of exposure.
3. Bring required materials to a fume hood: crotonaldehyde, a syringe, and a glass tube connected to a Swagelok Valve.
4. Secure the glass tube on the 3-prong clamp.
5. Carefully open the crotonaldehyde bottle. The bottle must be kept upright at all times to prevent leakage.
6. Use a syringe to transfer crotonaldehyde (2 mL) from the bottle into the glass tube through the connected Swagelok valve. Do not overfill the tube. Be careful not to spill crotonaldehyde. In case of exposure, follow procedures outlines in SDS and call 911.
7. Close the Swagelok valve and make sure there is no leak.
8. Connect the Swagelok valve to the gas manifold on the chamber.
9. Close the crotonaldehyde bottle. Check for any leaks of crotonaldehyde.
10. Put the crotonaldehyde bottle back to the designated storage cabinet. Keep the crotonaldehyde container tightly closed in a dry and well-ventilated place.
11. Clean the syringe with acetone and place the waste liquid into the waste container prepared with a carcinogen hazardous label.

UHV #2, RAIRS Chamber: For crotonaldehyde at room temperature

1. Equip the proper Personal Protective Equipment: flame resistant lab coat, safety glasses, nitrile gloves, and insulated gloves for liquid nitrogen manipulation.

2. Under the fume hood, use an injector to extract crotonaldehyde (2 mL) and inject it into a glass tube.
3. Attach the glass tube to the metal adaptor at the end of the leak valve on top of the chamber.
4. Fix the metal adaptor to the gas line.
5. Pour liquid nitrogen in a Dewar cup and submerge the glass tube containing toluene until the liquid freezes.
6. Open the valve next to the glass tube metal adaptor and then open the valve to the mechanical pump.
7. Pump the line down until it reaches the normal base pressure. Close the valve next to the glass tube metal adaptor. Allow the liquid to thaw.
8. Repeat steps 4–6 until the gasses dissolved in the liquid are completely absent.
9. Close the valve to the mechanical pump and open the valve to the to the glass tube metal adaptor. Let the liquid vaporize in the line.
10. For UHV use
    a. Close the valve next to the glass tube
    b. Keep spinning the leak valve to the UHV chamber until desired pressure is reached
    c. Immediately start the timer and close the leak valve quickly when a desired surface coverage (e.g. 10 Langmuir) is achieved
    d. Open the valve to the mechanical and turbo pumps to evacuate the gas line.
11. For high pressure cell reaction:
    a. Slightly open the valve to the gas mixture storage line until desired pressure (e.g. 2 Torr) is reached.
    b. Add other gases needed into the storage line.
    c. After finishing the gas mixture preparation, open the two valves to the high pressure cell and run the reaction.
12. In both experiments (HPC reaction and UHV use), the remaining crotonaldehyde and other gases will be pumped down by mechanical pump, turbo pump, and cryopump, which fulfills the chemical waste disposal.

UHV #3, NanoReactor

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and *full-face respirator*.
2. *Make a waste bottle labeled as lachrymator hazardous waste. Review the SDS of crotonaldehyde again; especially remind first-aid measures, handling and storage, PPE, & signs and symptoms of exposure.*
3. Bring required materials to a fume hood: crotonaldehyde, a syringe, a glass tube with fitting to the gas manifold.
4. Secure the glass tube on the 3-prong clamp.

5. Open the crotonaldehyde bottle. *The container must be carefully resealed and kept upright to prevent leakage*.
6. Use a syringe to transfer crotonaldehyde from the original bottle into the glass tube. *Be careful not to spill crotonaldehyde. Keep watching any leak of crotonaldehyde. Avoid release to the environment. Avoid breathing fume, gas, mist, vapor or spray. If swallowed, immediately call 911. If inhaled, rinse cautiously with water for 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.*
7. Close the crotonaldehyde bottle. *Keep the crotonaldehyde container tightly closed in a dry and well-ventilated place.*
8. Connect the glass tube to the gas manifold of UHV #3, NanoReactor
9. Put the crotonaldehyde bottle back to the storage place. *Keep watching any leak of crotonaldehyde.*
10. Clean the syringe with acetone and place the waste liquid into the waste container prepared with a carcinogen hazardous label. *Keep watching any leak of crotonaldehyde*.

FT-IR #1, Tensor 27

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and full-face respirator.
2. Make a waste bottle labeled as lachrymator hazardous waste. Review the SDS of crotonaldehyde again; especially remind first-aid measures, handling and storage, PPE, & signs and symptoms of exposure.
3. Bring required materials to a fume hood: crotonaldehyde, a syringe, a glass tube with fitting to the gas manifold.
4. Secure the glass tube on the 3-prong clamp.
5. Open the crotonaldehyde bottle. The container must be carefully resealed and kept upright to prevent leakage.
6. Use a syringe to transfer crotonaldehyde from the original bottle into the glass tube. Be careful not to spill crotonaldehyde. Keep watching any leak of crotonaldehyde. Avoid release to the environment. Avoid breathing fume, gas, mist, vapor or spray. If swallowed, immediately call 911. If inhaled, rinse cautiously with water for 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.
7. Close the crotonaldehyde bottle. Keep the crotonaldehyde container tightly closed in a dry and well-ventilated place.
8. Connect the glass tube to the gas manifold of Tensor 27, FT-IR Spectrometer
9. Put the crotonaldehyde bottle back to the storage place. Keep watching any leak of crotonaldehyde.
10. Clean the syringe with acetone and place the waste liquid into the waste container prepared with a carcinogen hazardous label. Keep watching any leak of crotonaldehyde

Reactor #1, Catalysis-Batch Hector

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and full-face respirator.
2. Review the SDS and SOP of crotonaldehyde. Especially remind the signs and symptoms of exposure, first-aid measures, handling, storage, and PPE.
3. Make a waste bottle for crotonaldehyde and acetone.
4. Bring the required materials to a fume hood: crotonaldehyde, a syringe, a domed glass tube with Swagelok fittings to the gas manifold.
5. Secure the glass tube on the clamp.
6. Transfer crotonaldehyde (1 mL) into the domed glass tube carefully not to spill.
7. Put crotonaldehyde bottle back to the designated storage area after required transferring is complete.
8. Connect the glass tube to the gas manifold of the batch reactor.
9. Perform a leak check on the connections. Pressurize the loop with Ar and keep the valve to the glass tube open. Then use some soap solution (snoop) to see if there are any bubble forming at the connections.
10. Run freeze-pump-thaw cycles. Use LN2 to freeze crotonaldehyde. Then, open the valve connecting to the gas manifold. Start pumping to initiate vacuum. After reaching vacuum state, close the valve. Once crotonaldehyde thaws, there will be some gaseous pressure above the liquid phase of crotonaldehyde.
11. Use the gas to feed into the gas manifold and finally the reactor loop.
12. Clean the syringe with acetone.
13. Put the waste liquid into the waste container.
14. After pretreatment of desired catalyst is done, the reaction mixture is introduced for a kinetic data measurement. The valves to the reactor tube are closed before introducing the mixture. Fill the reactor loop with crotonaldehyde (5 Torr), hydrogen (50 Torr) and argon (700 Torr) in sequence.
15. Mix them for 20 minutes by turning on the recirculation pump.
16. Open the valves to the reactor tube.
17. After 2 minutes of reaction, take a gas sample (0.5 mL) for testing with the GC. Use a gas sampling syringe to take a sample through the septum connected in the loop. Then inject the sample in the GC and press the start button in the GC to start collecting data.
18. Take more samples repeatedly as required for testing with the GC.
19. After experimentation is complete, vacuum the reactor loop with the mechanical pump.

IR Measurement

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

2. Make a waste bottle labeled as lachrymator hazardous waste. Review the SDS of crotonaldehyde again; especially remind first-aid measures, handling and storage, PPE, & signs and symptoms of exposure.
3. Bring required materials to a fume hood: crotonaldehyde, a syringe, a glass tube with fitting to the gas manifold.
4. Secure the glass tube on the 3-prong clamp.
5. Open the crotonaldehyde bottle. The container must be carefully resealed and kept upright to prevent leakage.
6. Use a syringe to transfer crotonaldehyde from the original bottle into the glass tube. Be careful not to spill crotonaldehyde. Keep watching any leak of crotonaldehyde. Avoid release to the environment. Avoid breathing fume, gas, mist, vapor or spray. If swallowed, immediately call 911. If inhaled, rinse cautiously with water for 15 min. Remove contact lenses, if present and easy to do. Continue rinsing.
7. Close the crotonaldehyde bottle. Keep the crotonaldehyde container tightly closed in a dry and well-ventilated place.
8. Connect the glass tube to the gas manifold of Tensor 27, FT-IR Spectrometer
9. Put the crotonaldehyde bottle back to the storage place. Keep watching any leak of crotonaldehyde.
10. Clean the syringe with acetone and place the waste liquid into the waste container prepared with a carcinogen hazardous label. Keep watching any leak of crotonaldehyde

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SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 04/01/2016, updated 04/17/2017, 06/23/2021, 09/05/2021, 01/21/2022