Octadecyltrichlorosilane
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 octadecyltrichlorosilane ($C_{18}H_{37}Cl_3Si$, CAS No. 112-04-9) is used in laboratory. Its purpose is not to have any accident or risk. Octadecyltrichlorosilane is combustible liquid and corrosive. It causes severe skin and eye damage.

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

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

OSHA Hazards: Combustible Liquid, Corrosive
GHS Classification
- Flammable liquids (Category 4)
- Skin corrosion (Category 1B)
- Serious eye damage (Category 1)

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

Silyation of Si wafer

1. Wear a nitrile chemical-resistant gloves, a flame-resistant lab coat, and a safety goggle.
2. Bring the toluene solution bottle together with octadecyltrichlorosilane to the fume hood in room 135.
3. Mix octadecyltrichlorosilane and Toluene solution in the volume ratio of 1:250
4. Soak the Si wafer in the octadecyltrichlorosilane /toluene (10 mM) solution for 24 hours and take the wafer out
5. After washing the Si wafer with toluene, all the used solution needs to be treated as hazardous

RCA & Silyation of Si wafer

1. Wear a nitrile chemical-resistant (neoprene) gloves, a flame-resistant lab coat, and a safety goggle.
2. Place wafers in a 3:1 solution of sulfuric acid (12 mL) and hydrogen peroxide (4 mL) for 10 min.
3. Place wafers in a water bath for 5 min.
4. Place wafers in a 1:20 solution of hydrofluoric acid (1 mL) and milli-Q water (20 mL) for 30 s ~ 5 min.
5. Clean wafers with copious amounts of milli-Q water.
6. Place wafers in a solution of milli-Q water (25 mL), hydrogen peroxide (5 mL), and sodium hydroxide (5 mL) for 10 min. at 70 ^{o}C.
7. Place wafers in a water bath for 5 min.
8. Place wafers in a solution of milli-Q water (30 mL), hydrogen peroxide (5 mL), and hydrochloric acid (5 mL) for 10 min. at 70 ^{o}C.
9. Place wafers in a water bath for 15 min.
10. Rinse each wafer under a water stream for 1 min.
11. Dry silicon wafers in N_2 flow.

12. For Liquid-Phase Silylation, immerse SiO_2 wafers into liquid solution of 10 mM octadecyltrichlorosilane dissolved in Toluene.
13. For Gas-Phase Silylation: fill precursor tube about halfway with octadecyltrichlorosilane in glovebox and attach tube to the leak valve. After removal of the setup from the glovebox, install the leak valve onto the UC System chamber.

NiO deposition on ODTS/SBA-15 via ALD reactor:

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

TiO_2/ODTS/SBA-15

1. Wear nitrile chemical resistant gloves, a flame-resistant lab coat, safety goggles, and a proper face mask at all times while inside the lab. Carry out all the procedures in the ALD reactor under rough vacuum conditions.
2. Clean the sample holder with acetone before placing SBA-15 in the sample holder.
3. Preheat the support (SBA-15) at 200 ºC for 2 h.
4. Ti precursor (tetrakis(dimethylamido) titanium, TDMAT) 42 ºC, using heating tape and r
5. The reactor is set to 102 ºC.
6. Dose octadecyltrichlorosilane (ODTS) for 30 s at 50 mTorr.
7. Dose TDMAT for 20 min at 200 mTorr.
8. Purge the reactor with Ar gas for 50 min at 500 mTorr.
9. Dose deionized water vapor for 2 min at 100 mTorr.
10. Purge the reactor with Ar gas for 50 min at 500 mTorr.
11. Repeat 7–10 steps repeatedly until desired growth is obtained.

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 03/01/2014, 02/07/2020, 10/12/2021, 12/21/2021

Oxalyl chloride
STANDARD OPERATING PROCEDURE

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

1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when oxalyl chloride ($C_2Cl_2O_2$, CAS No. 79-37-8) is used in laboratory. Its purpose is not to have any accident or risk. Oxalyl chloride is corrosive toxic liquid. It is toxic if inhaled or swallowed. Also, it causes severe skin burns and eye damage.

Synonyms: Ethanedioyl dichloride

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

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

OSHA Hazards: Corrosive, Toxic by Inhalation, Eye damage, Skin burns
GHS Classification
- Acute toxicity, Inhalation (Category 3)
- Skin corrosion (Category 1A)
- Serious eye damage (Category 1)

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

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

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

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

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

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

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

10. DESIGNATED AREA

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

11. SAFETY DATA SHEETS

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

12. DETAILED PROTOCOL

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

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

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

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

Synthesis of 1,1'-(1,2-Dioxoethane-1,2-diyl)bis-1*H*-benzotriazole

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 06/01/2015

Oxygen
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 oxygen (O_2, CAS No. 7782-44-7) used in laboratory. Its purpose is not to have any accident or risk. Oxygen is oxidizing gas, and contains gas under pressure. It may cause or intensify fire.

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

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

OSHA Hazards: Flammable gas, Compressed Gas
GHS Classification
Flammable gas (Category 1)
Gas under pressure (Liquefied gas)

Signs and Symptoms of Exposure
Nausea, Dizziness, Unconsciousness, May be harmful.

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 oxygen 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 oxygen 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 oxygen 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 oxygen 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 oxygen 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 oxygen 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 oxygen 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 oxygen gas cylinders shall be labeled as empty. Depleted oxygen 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 oxygen gas 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 oxygen 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! OXYGEN 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 oxygen gas must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of oxygen gas and understand the hazards.

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

When working in the lab, a laboratory worker must:

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

3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) use oxygen under 1 bar in any given reaction (higher pressure REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this oxygen 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 oxygen. 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 monoxide is introduced into the cell up to 10 Torr.
12. Sample spectrum is obtained.
13. Carbon monoxide is pumped out.
14. The IR cell is vented to atmosphere.

UHV #1, Victor

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

UHV #2, RAIRS

1. Equip the proper PPEs (flame-resistant lab coat, safety glasses, chemical-resistant nitrile gloves.
2. Unscrew the main valve cap.
3. Carefully adjust the outlet pressure to 20 psi.
4. Close the valve next to the mechanical pump.

5. Fill the gas line with the Oxygen gas.
6. Open the valve to the pump to evacuate the line.
7. Fill the gas lien with the Oxygen gas.
8. After dosing with a leak valve or preparing a gas mixture, evacuate the gas line by opening the valve to the mechanical pump.

UHV #3, Michelle

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

UHV #4, Praxis

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety glasses.
2. Check that the Oxygen tank line is closed.
3. Open the valve, which connects the Oxygen line and the gas manifold pump, to evacuate the Oxygen line.
4. Wait until the pressure gauge at the bottom of the electronics cabinet reaches 20 mTorr to indicate full gas evacuation.
5. Close the green swagelok valve, which connects the gas manifold pump to the Oxygen leak valve, to stop pumping of the Oxygen line.
6. When the single crystal sample has reached the desired temperature for oxygen cleaning (~1000 K for Pd(111)), open the Oxygen tank valve to let gas flow to the chamber leak valve. Adjust the pressure of Oxygen in the chamber by opening/closing the leak valve.
7. When Oxygen use is finished, close the Oxygen leak valve. Shut off the temperature controller.
8. Close the green Oxygen swagelok valve to stop the flow of gas from the Oxygen tank into the leak valve.

9. Open the green swagelok valve that connects to the gas manifold pump so that the leak valve can be pumped out. Close the Oxygen tank valve.

UHV #5, UC Chamber

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

UHV #6, Nanoreactor

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

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 ^{o}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.

Oxidation-Reduction Pretreatment of Catalyst

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

GC #1 Agilent-Batch Reactor: Oxygen Pre-treatment cycle

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Introduce around 400-500 torr of O2 at room temperature by opening the O2 valve
3. Close the O2 valve
4. Turn on the recirculation pump to cycle the O2 gas through the reactor
5. Slowly increase the temperature of the u-shaped tube to the pre-treatment temperature (around 350 °C)
6. After the u-shaped tube reaches the pre-treatment temperature, continue cycling the O2 gas for one hour
7. After 1 hour, slowly reduce the temperature of the catalyst in the u-shaped tube using a cooling fan.

8. After cooling the u-shaped tube and catalyst to room temperature (35 °C is ok to save time), evacuate the reactor loop by opening the valve connecting the reactor loop to the manifold and open the valve connecting the manifold to the pump
9. The reactor loop is evacuated when the pressure is around the same pressure of the loop before introducing the gas. Because the gauge is broken, evacuate the reactor loop by leaving both aforementioned valves open for 30 minutes

Synthesis of Au NPs on APTES-grafted P25 titania

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

SOP Reviewed and Approved by:

Francisco Zaera
Print name

Signature

Approval Date: 02/01/2013, updated 04/05/2014, 03/01/2016, 07/01/2018, 01/25/2019, 04/09/2022, 04/18/2022

Platinum 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 platinum acetylacetonate ($C_{10}H_1PtO_4$, CAS No. 15170-57-7) used in laboratory. Its purpose is not to have any accident or risk. platinum acetylacetonate is toxic if swallowed. Also it is harmful if inhaled.

Synonyms: 2,4-Pentanedioneplatinum(II) derivative, $Pt(acac)_2$

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

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

OSHA Hazards: Toxic
GHS Classification

- Acute toxicity, Oral (Category 4)
- Acute toxicity, Inhalation (Category 4)
- Acute toxicity, Dermal (Category 4)
- Skin irritation (Category 2)
- Eye irritation (Category 2A)
- Reproductive toxicity (Category 2)
- Specific target organ toxicity – single exposure (Category 3)

Signs and Symptoms of Exposure
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. To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.
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 platinum 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 platinum 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 platinum acetylacetonate must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of platinum acetylacetonate and understand the hazards.

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

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

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

Synthesis of Cu@Pt nanoparticle

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

Synthesis of Pt@Cu nanoparticle

1. Wear nitrile chemical–resistant gloves, flame-resistant lab coat, and safety goggles.
2. Dry a flask (25 mL) under nitrogen in a fume hood.

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

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 11/01/2016

Polyvinylpyrrolidone (PVP)
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 polyvinylpyrrolidone ($(C_6H_9NO)_X$, CAS No. 9003-39-8) is used in laboratory. Its purpose is not to have any accident or risk. Polyvinylpyrrolidone may be harmful if swallowed and inhaled. It may cause skin and eye irritation.

Synonyms: PVP, Polyvidone, Povidone

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

Polyvinylpyrrolidone is commercially available polymer, and used mostly as capping agents in nanoparticle synthesis. A variety of organic solvents are used to clean sample containers. Please refer SDS first always for physical and chemical properties before use.

PVP10 (a.m.w. 10,000), PVP40 (a.m.w. 40,000), PVP360 (a.m.w. 360,000)

OSHA Hazards: Not known
GHS Classification
N/A

Signs and Symptoms of Exposure
Unexcreted particles may be phagocytized by cells of the reticuloendothelial system and deposited in storage sites in the liver, spleen, lung, and bone marrow resulting in the storage disease thesaurosis. Severity and symptoms depend on storage site and nature of the particle. Pathological changes are not necessarily attributed to the thesaurosis, but in some cases an inflammation or granulomatoma have occurred.

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 polyvinylpyrrolidone 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 polyvinyl-pyrrolidone.

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

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

When working in the lab, a laboratory worker must:

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

1. Wear dust respirator, nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. Bring polyvinylpyrrolidone bottle to a balance. Use a nickel spoon to transfer polyvinylpyrrolidone into the designated vessel or weighing paper.
3. Close and seal the bottle and put it back.
4. Discard extra polyvinylpyrrolidone to designated chemical waste container. Clean the balance with brush.
5. Add designated amount of water to dissolve polyvinylpyrrolidone. Mix with Pt precursor and reduce the solution under designated conditions.

Silica coating onto the gold nano particle

1. Wear anitrile chemical-resistant gloves, mask, flame-resistant lab coat, and safety goggles.
2. Weighing 0.12 g of polyvinylpyrrolidone.
3. Put the polyvinylpyrrolidone into an Erlenmeyer flask.
4. Pour a proper amount of milli-Q water in the Erlenmeyer flask and stir smoothly.
5. Inject the polyvinylpyrrolidone solution into gold nano particle solution.
6. Stir the mixture smoothly during overnight.

Making Titania Shell

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

2. Disperse SiO_2-Au nanoparticles (100 mg) in ethanol (10 mL) and acetonitrile (3.4 mL).
3. Add polyvinylpyrrolidone (27 mg) and stir for 20 min.
4. Add ammonium hydroxide (77 μL) and stir for 20 min.
5. Add a mixture of ethanol (1.3 mL), acetonitrile (0.42 mL), and titanium butoxide (0.32 mL).
6. Stir the mixture for 2 h and wash 3 times with ethanol.

Treatment of TiO_2 shells

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety goggles.
2. *Make a waste bottle labeled as toxic hazardous waste. Review the SDS of polyvinylpyrrolidone again; especially remind first aid measures, handling and storage, & PPE.*
3. Place an Erlenmeyer flask into a fume hood and put a stir bar into it. Close with a rubber septum and take it to a balance. Weigh polyvinylpyrrolidone (110 mg) and add it into the flask.
4. Take the flask back to the fume hood and add milli-Q water (21 mL) into it.
5. Stir overnight at room temperature.
6. Centrifuge and dispose the waste into the waste bottle labeled toxic 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 °C. If you leave it unattended in a fume hood, put a label with chemical name and hazard information.
11. Remove the septum from the flask and put SBA-15 (558.36 mg) in the flask and stir the mixture for 2 h.
12. Sonicate the mixture for 1 h.

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

Synthesis of Cu@Pt nanoparticle

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

Synthesis of Pt@Cu nanoparticle

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

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

Synthesis of Pt nanoparticles

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 dihydrogen hexachloroplatinate (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 dihydrogen hexachloroplatinate solution.
8. Heat the solution at 160 ^{o}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.

SOP Reviewed and Approved by:

Francisco Zaera	
Print name	Signature

Approval Date: 02/01/2013, updated 03/01/2014, 03/01/2016, 05/15/2016, 11/01/2016, 06/01/2017

Potassium perchlorate
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 potassium perchlorate ($ClKO_4$, CAS No. 7778-74-7) used in laboratory. Its purpose is not to have any accident or risk. Potassium perchlorate is strong oxidizer, so it may cause fire or explosion. It is harmful if swallowed, if inhaled, or absorbed through skin. Also, it causes respiratory tract, skin, and eye irritation.

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

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

OSHA Hazards: Oxidizer, Target Organ (Blood, Thyroid) Effect, Harmful by Ingestion
GHS Classification
Oxidizing solids (Category 1)
Acute toxicity, Oral (Category 4)

Signs and Symptoms of Exposure
Blood disorders, Absorption into the body leads to the formation of methemoglobin which in sufficient concentration causes cyanosis. Onset may be delayed 2 to 4 hours or longer.

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Respiratory Protection

Where risk assessment shows air-purifying respirators are appropriate use a full-face particle respirator type N100 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.

b. Eye Protection

ANSI compliant safety glasses with side shields conforming to EN166 should be worn. Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH. 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 potassium perchlorate 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 potassium perchlorate.

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

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

When working in the lab, a laboratory worker must:

1) not work alone;
2) be cognizant of all of the SDS and safety information presented in this document;
3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
4) employ < 25 g of this potassium perchlorate in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
5) discuss ALL issues or concerns regarding this potassium perchlorate 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 potassium perchlorate. 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. Wear a nitrile chemical–resistant glove, flame-resistant lab coat, and safety goggle.
2. The solution of potassium perchlorate is used for the pretreatment of platinum samples before experiment.
3. To prepare solutions with proper solvents, consult relevant manuals in advance.

SOP Reviewed and Approved by:

Francisco Zaera

Print name

Signature

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

Propane
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 propane (C_3H_6, CAS No. 74-98-6-1) used in laboratory. Its purpose is not to have any accident or risk. Propane is extremely flammable gas, and contains gas under pressure. It may explode if heated, harmful if inhaled or absorbed through skin, or cause respiratory tract 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 gas, Compressed Gas
GHS Classification
Flammable liquids (Category 1)
Gas under pressure (Liquefied gas)

Signs and Symptoms of Exposure
Dizziness, Drowsiness, Unconsciousness

3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

a. Eye Protection

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

b. Skin and Body Protection

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

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

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

c. Hand Protection

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

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

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

4. ENGINEERING/VENTILATION CONTROLS

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

5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS

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

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

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

6. SPILL AND INCIDENT PROCEDURES

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

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

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

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

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

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

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

7. DECONTAMINATION

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

8. WASTE DISPOSAL

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

General hazardous waste disposal guidelines:

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

9. PRIOR APPROVAL/REVIEW REQUIRED

All work with propane 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 propane.

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

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

When working in the lab, a laboratory worker must:

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

Replace empty gas cylinder

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

GC #1 Agilent-Batch Reactor: GC signal calibration

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety glasses.
2. Ensure that the GC is on (if not, turn it on)
3. Vacuum the system for 30 min.

4. Open the gas cylinders (in this case argon, hydrogen and air).
5. Adjust pressure and carrier gas flow rate to the GC setting.
6. Introduce five different pressures of propane gas one at a time to generate a calibration curve (in total, there should be 500 Torr of propane and argon):
    a) Propane:Argon = 1:499 Torr
    b) Propane:Argon = 2:498 Torr
    c) Propane:Argon = 3:497 Torr
    d) Propane:Argon = 4:496 Torr
    e) Propane:Argon = 5:495 Torr
7. This calibration curve can be compared to the data from the reaction.

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 04/18/2022

Propylene
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 propylene (C_3H_6, CAS No. 115-07-1) used in laboratory. Its purpose is not to have any accident or risk. Propylene is highly flammable gas, and contains gas under pressure. It may explode if heated, and may cause drowsiness or dizziness.

Synonyms: Propene

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
GHS Classification
Flammable liquids (Category 1)
Gas under pressure (Liquefied gas)
Specific target organ toxicity – single exposure (Category 3)

Signs and Symptoms of Exposure
Dizziness, Headache, Central nervous system depression

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 propylene 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 propylene.

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

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

When working in the lab, a laboratory worker must:

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

Replace empty gas cylinder

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

UHV #2, RAIRS

Propylene lecture bottle installation:

1. Wear nitrile chemical resistant gloves, a flame-resistant laboratory coat and safety goggles.
2. Make sure the lecture bottle valve is closed all the way. Use a wrench to remove safety screw (and Teflon washer) from the bottle outlet.

3. Attach a High (inlet)/Low (outlet) pressure regulator to the lecture bottle. Check that the regulator valve is closed (loose). Make sure that the Teflon washer is inside the regulator connector. Tighten firmly.
4. Connect the end of the regulator to Propylene gas line. Make sure the threading from each end is aligned before tightening. Tighten firmly.
5. Very slowly pump down the gas line with the mechanical pump that is connected to the gas manifold.
6. Check for leaks. Use squeeze bottle to spray a minimum amount of acetone on the connections you have just tighten. Monitor the pressure. An increase in gas line pressure means that the sprayed connection is loose and needs further tightening. Use paper towels to avoid acetone splashing and to clean up any small spill.

Propylene Dosing (UHV Experiment):

1. Using the mechanical pump connected to the manifold evacuate the gas lines leading to the leak valve on top of the main UHV chamber and the Propylene lecture bottle (make sure that the regulator and lecture bottle valves are closed).
2. Stop pumping when pressure reaches 1E-2 Torr by closing the valve on the main gas line that is closest to the leak valve.
3. Open the lecture bottle valve all the way and then close it.
4. Slowly tighten the regulator valve until you reach the 20 psig mark. Loosen the regulator valve.
5. Slowly open the valve that connects the regulator to the gas line. There will be a pressure drop from 20 psig to ~0 psig on the regulator gauge.
6. Close the valve closest to the leak valve to isolate the gas in the line connected to it.
7. Slowly pump down the gas line leading to the Propylene cylinder.
8. Slowly open the leak valve and monitor the pressure increase inside the UHV chamber until the desired value is reached. Use a timer to attain the desired exposure.
9. After dosing close the leak valve.
10. After all exposures are done pump down the leak valve by slowly opening the valve closest to it.

High Pressure Propylene Hydrogenation:

1. Close the gate-valve to the turbo pump on the gas line located at the High-Pressure end of the system. Make sure that the valve that connects to the gas manifold at the UHV end of the system is also closed.
2. Close the valve that connects the regulator gas line to the High-Pressure end loop. Make sure that the regulator valve is closed (loose).
3. Open the lecture bottle valve all the way and then close it.

4. Slowly tighten the regulator valve until you reach the 20 psig mark. Loosen the regulator valve.
5. Slowly open the valve that connects the regulator to the gas line. There will be a pressure drop from 20 psig to ~0 psig on the regulator gauge.
6. Open the valve that connects the regulator gas line to the High-Pressure end loop.
7. Fill the High-Pressure loop with the desired amount of Propylene. Monitor this measurement with the Baratron Gauge.
8. After the measurement, close the valve to the loop.
9. Open valves to reactor and carry out experiment.
10. Using the mechanical pump connected to the manifold evacuate the gas lines leading to the Propylene cylinder (make sure that the regulator and cylinder valves are closed).
11. Stop pumping when pressure reaches 1E-2 Torr by closing the valve that connects to the manifold on the UHV end.
12. After experiment open valve to High-Pressure loop and pump down with mechanical pump to 1E-2 Torr.
13. Close valve to mechanical pump and open Turbo-Pump gate-valve.

UHV #4 Praxis

1. Wear nitrile chemical-resistant gloves, a flame-resistant lab coat, and safety goggles.
2. Bring the propylene gas lecture bottle into a fume hood.
3. Attach a gas regulator to the Propylene gas lecture bottle by using a wrench.
4. Bring the Propylene lecture bottle to the UHV system carefully and connect to a Swagelok valve on the gas manifold.
5. After measurement, the Propylene gas lecture bottle needs to be stored in the gas cylinder rack of room 135.

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

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

1. Wear nitrile chemical-resistant gloves, flame-resistant lab coat, and safety glasses.
2. Isolate the gas manifold from the mechanical pump by closing the valve connecting the manifold to the mechanical pump
3. Slowly open the valve for the propylene and close the valve after a few seconds
4. Slowly open the valve connecting the gas manifold to the rector loop while constantly checking the baratron pressure gauge. Make sure to add 5 torr of propylene to the reactor loop.

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

SOP Reviewed and Approved by:

Francisco Zaera _____

Print name Signature

Approval Date: 01/01/2013, updated 03/01/2014, 04/09/2022