Standard Operating Procedures

Peroxide Forming Chemicals (PFC) - Class 2

Dicyclopentadiene

Print a copy and insert into your laboratory SOP binder.

<table>
<thead>
<tr>
<th>Department:</th>
<th>Chemistry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Date SOP was written:</td>
<td>June 20, 2013</td>
</tr>
<tr>
<td>Date SOP was approved by PI/lab supervisor:</td>
<td></td>
</tr>
<tr>
<td>Principal Investigator:</td>
<td>Name: Richmond Sarpong</td>
</tr>
<tr>
<td>Signature:</td>
<td>__________________________</td>
</tr>
<tr>
<td>Internal Lab Safety Coordinator or Lab Manager:</td>
<td>Name: Rebecca Murphy</td>
</tr>
<tr>
<td>Lab Phone:</td>
<td>510-643-2485</td>
</tr>
<tr>
<td>Office Phone:</td>
<td>510-642-6312</td>
</tr>
<tr>
<td>Emergency Contact:</td>
<td>Name: Richmond Sarpong</td>
</tr>
<tr>
<td>Phone Number:</td>
<td>626-644-2407</td>
</tr>
<tr>
<td>Location(s) covered by this SOP:</td>
<td>Latimer Hall: 834, 836, 837, 838, 839, 842, 847, 849, 907</td>
</tr>
</tbody>
</table>

1 - Purpose

This SOP covers the precautions and safe handling procedures for the use of Dicyclopentadiene in the Sarpong group, which include the following uses:

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Use</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dicyclopentadiene</td>
<td>1. Prepare the compound cyclopentadiene.</td>
</tr>
<tr>
<td></td>
<td>2. Prepare cyclopentadienyl (Cp) dimer metal compounds.</td>
</tr>
<tr>
<td></td>
<td>3. Used in the lab as a reagent in reactions.</td>
</tr>
</tbody>
</table>

2 - Physical & Chemical Properties/Definition of Chemical Group

CAS#: 77-73-6

Molecular Formula: C_{10}H_{12}

Form: liquid

Color: light yellow

Melting point/freezing point: 33 °C (91 °F) - lit.

Boiling point: 170 °C (338 °F) - lit.

Vapor pressure: 1.86 hPa (1.40 mmHg) at 20 °C (68 °F)

Density: 0.986 g/mL at 25 °C (77 °F)

Flash point: 32 °C (90 °F) - closed cup

Lower explosion limit: 1 % (V)

Upper explosion limit: 10 % (V)
Odor: no data available
Odor Threshold: no data available

3 - Potential Hazards/Toxicity

GHS Classification
Flammable liquids (Category 3)
Acute toxicity, Oral (Category 4)
Acute toxicity, Inhalation (Category 3)
Skin irritation (Category 2)
Eye irritation (Category 2A)
Specific target organ toxicity - single exposure (Category 3)
Acute aquatic toxicity (Category 2)
Chronic aquatic toxicity (Category 2)

GHS Label elements, including precautionary statements

Pictogram

Signal word Danger

Hazard statement(s)
H226 Flammable liquid and vapor.
H302 Harmful if swallowed.
H315 Causes skin irritation.
H319 Causes serious eye irritation.
H331 Toxic if inhaled.
H335 May cause respiratory irritation.
H411 Toxic to aquatic life with long lasting effects.

Precautionary statement(s)
P261 Avoid breathing dust/ fume/ gas/ mist/ vapors/ spray.
P273 Avoid release to the environment.
P305 + P351 + P338 IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
P311 Call a POISON CENTER or doctor/ physician.

Cal/OSHA Permissible Exposure Limits (PEL): 5 ppm (30 mg/m³) - 8-hour TWA

4 - Engineering Controls
Use the engineering controls listed below unless other lab-specific information is included in the Protocol/Procedure section.

• A laboratory type fume hood with the sash position closed as much as possible;
• A glove box for pyrophorics and water reactive chemicals. Glove boxes may also be required for other chemicals, such as regulated carcinogens and particularly hazardous substances;
• Supplemental protective equipment like a blast shield, where appropriate, to protect from explosions when using peroxide formers, pyrophorics, water reactives, and potentially explosive chemicals.

5 - Personal Protective Equipment
For additional information on PPE requirements, go to: http://ceehs.berkeley.edu/section5#Personnel_Protective_Equipment_Required_in_College_Laboratories

Use the PPE listed below unless other lab-specific information is included in the Protocol/Procedure section.

Eye and Face Protection
ANSI-approved safety glasses with side shields or chemical splash goggles must be worn at all times when handling chemicals in the lab.

Skin and Body Protection
1. Gloves are required when handling hazardous chemicals.
   a. Specific glove type recommendations are provided in the Protocol/Procedure section.
   b. Inspect gloves prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Wash and dry hands after handling chemicals, before breaks, and at the end of the workday.
   c. For additional information on glove selection, go to: http://ehs.berkeley.edu/hs/63-laboratory-safety/94-glove-selection-and-usage.html
2. Lab coats are required when handling hazardous chemicals in the lab.
   a. Nomex 3A flame-resistant lab coats are required when working with pyrophorics (H250) and explosives (H200, H201, H202, H203)
   b. Flame resistant lab coats (Nomex or other material) should be worn when working with materials such as Category 1 or 2 flammable liquids (H224 and H225).
3. Cotton-based, non-synthetic clothing (including long pants; no skin exposed below the waist) should be worn.
4. Closed-toe and closed-heel shoes are required in the lab.

Respiratory Protection
Respiratory protection is normally not required for UC Berkeley laboratory activities. Any lab personnel considering the use of a respirator must contact EH&S for a workplace assessment. Respirator users will be provided with specific instructions if a respirator is deemed necessary by EH&S.

6 - First Aid Procedures and Medical Emergencies
Notify supervisor and EH&S immediately. Follow up with a call to 510-642-9090 to report the incident.

Life Threatening Emergency, After Hours, Weekends and Holidays – Call 911 or go to the nearest emergency room. Note: All serious injuries must be reported to EH&S within 8 hours. Follow up with a call to 510-642-9090 to report the incident.

Assess the extent of danger. If you cannot assess the conditions of the environment well enough to be sure of your own safety, do not enter the area. If possible, help contaminated or injured persons. Obtain medical attention for the individual as soon as possible by calling 911. Provide a copy of the appropriate SDS to the emergency responders or physician, as needed.
If inhaled
Move into fresh air. Go to the Occupational Health Facility (Tang Health Center) and after hours, go to the nearest emergency room. If person is not breathing, call 911 and give artificial respiration. If unconscious, call 911.

In case of skin contact
Immediately flush with flowing water for no less than 15 minutes; remove any jewelry or clothing as necessary to facilitate clearing of any residual materials. Wash off with soap and plenty of water for 15 minutes. If skin contact requires medical assistance, go to the Occupational Health Facility (Tang Health Center) and after hours, go to the nearest emergency room. If this is a large or serious injury, call 911.

In case of eye contact
Rinse thoroughly with plenty of water using an eyewash station for at least 15 minutes, occasionally lifting the upper and lower eyelids. Remove contact lenses if possible. Call 911.

If swallowed
Call 911. Do not induce vomiting unless directed otherwise by the SDS. Never give anything by mouth to an unconscious person. Rinse mouth with water. Go to the Occupational Health Facility (Tang Health Center) and after hours, go to the nearest emergency room.

Needle stick/puncture exposure
Wash the affected area with antiseptic soap and warm water for 15 minutes. For mucous membrane exposure such as eyes, mouth and/or nose, flush the affected area for 15 minutes using an eyewash station. Go to the Occupational Health Facility (Tang Health Center) and after hours, go to the nearest emergency room.

All needle stick/puncture exposures must be reported to EH&S within 8 hours. Follow up with a call to 510-642-9090 to report the incident.

7 - Special Handling and Storage Requirements
Lab-specific information on handling and storage may be included in the Protocol/Procedure section.

Working alone - Certain extremely hazardous operations should not be performed if the PI or Lab Safety Contact(s) are not present. Never work alone with extremely hazardous materials/operations. See the Protocol/Procedure section below for specific prohibitions (if any) on working alone.

Precautions for safe handling
- Avoid contact with skin and eyes. Avoid formation of vapors, dusts, mists and aerosols.
- Use appropriate exhaust ventilation.
- Use appropriate personal protective equipment.
- Remove incompatible chemicals from immediate work area.
- Keep flammable, pyrophoric, potentially explosive and water reactive chemicals away from sources of ignition.
- Use care when preparing chemical solutions.

Conditions for safe storage
- Keep quantities to a minimum.
- Keep containers tightly closed and in a cool, dry and well-ventilated location.
- Keep in proper storage cabinets and shelving. Use lowest shelfe possible.
- Assure chemicals are properly labeled.
• Segregate incompatible chemicals.
• Store carcinogens in a designated area.
• Provide secondary containment for chemicals in accordance with the ccEHS "Chemical Hygiene Plan": http://ceehss.berkeley.edu/section5#Chemical_Handling_Storage_and_Transportation

8 - Chemical Spill

Spill – Assess the extent of danger; if necessary request help by calling 911 and 510-642-9090. If you cannot assess the conditions of the environment well enough to be sure of your own safety, do not enter the area. If possible help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors from spill. 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.).

Minor Spill – In the event of a minor spill, if there is no potential for hazardous chemical exposure, report the spill to 510-642-9090 and proceed to clean it, if you are trained. Use appropriate personal protective equipment and clean-up material for chemical spilled. Double bag spill waste in clear plastic bags, label and take to the next chemical waste pick-up location.

Call 510-642-9090 to report the spill to ccEHS and for assistance.

Major Spill – Any hazardous chemical spill that involves chemical exposure, any chemical spill that due to size and/or hazard requires capabilities beyond your training, or any chemical spill that gives the perception (because of odor, for example) that there has been a hazardous release

Call 911 and 510-642-9090 to report the spill to ccEHS and for assistance.

9 - Cleaning and Decontamination

Lab-specific information on decontamination may be included in the Protocol/Procedure section.

• Wearing proper PPE, laboratory work surfaces should be cleaned at the end of each work day.
• Dispose of contaminated materials in accordance with hazardous waste disposal guidelines referenced below.
• Decontaminate all equipment before removing from a designated area.

10 - Hazardous Waste Disposal

Label Waste

Label all containers with the label provided at:

See the EH&S Fact Sheet, “Hazardous Waste Management” for general instructions on procedures for disposing of hazardous waste.

Dispose of Waste

• Dispose of regularly generated chemical waste within 6 months.
• Call EH&S with questions.

11 - Safety Data Sheet (SDS) Location

SDS can be accessed online at http://ucmsds.com
12 - Protocol/Procedure – Dicyclopentadiene

| Preparation                          | Know the location of the nearest fire extinguisher, eyewash, and safety shower before beginning work.  
|                                    | **Never** open a dented or otherwise compromised container of Dicyclopentadiene. Alert lab safety contact immediately.  
|                                    | **Never** open/use/move a Dicyclopentadiene container that is suspected to contain peroxides or is in contact with crystals indicative of peroxide formation. Do not touch the crystals. Alert lab safety contact immediately.  
|                                    | Eliminate incompatible materials (e.g. strong bases, chlorates) from potential spill area.  
|                                    | Eliminate ignition sources such as open flames, hot surfaces, steam baths, and static electricity.  
|                                    | **Never** work alone. Make sure there is another worker present who is also trained in the use of Dicyclopentadiene. |
| Chemical Storage and Disposal       | **Storage**  
|                                    | Store containers in an approved area, such as a ventilated flammable storage cabinet.  
|                                    | Segregate from any incompatible materials, such as strong bases and chlorates.  
|                                    | Keep containers in a cool, dry, well-ventilated area.  
|                                    | Avoid all possible sources of ignition (spark or flame). Protect from flames, static electricity and sources of heat.  
|                                    | Prolonged exposure to air and light may form unstable explosive peroxides unless inhibited against peroxide formation.  
|                                    | Store and transport containers in secondary containment (for example polyethylene bottle carrier).  
|                                    | Keep storage containers closed (air tight) when not in use.  
|                                    | Purchase Dicyclopentadiene with inhibitors added (for peroxide-forming) when possible.  
|                                    | Due to its peroxide-forming hazard, Dicyclopentadiene containers must be dated upon receipt and at the time they are opened (mark the date on the bottle). If tested, note the date it was tested on the bottle. Unopened containers of Dicyclopentadiene have a maximum shelf life of 18 months. Opened containers of Dicyclopentadiene have a maximum shelf life of 12 months. Test open Dicyclopentadiene containers with peroxide test strips every 3 months. Dispose of unused amounts after that period of time has passed (or if peroxides are found to be present by testing).  
|                                    | Degassed Dicyclopentadiene may be stored in resealable Schlenk flasks under an atmosphere of argon or nitrogen, or stored in closed containers in a glove box with a nitrogen or argon atmosphere.  
|                                    | **Disposal**  
|                                    | Dicyclopentadiene waste is considered hazardous. Collect all liquid waste in labeled 1 gallon plastic containers.  
|                                    | Store hazardous waste in closed containers, in secondary containment and in a designated location.  
|                                    | Mix ONLY with compatible waste streams, such as other organic solvents.  
|                                    | Call EH&S with questions. |
| Lab-specific Information           | Cyclopentadiene, formed from dicyclopentadiene, has a strong odor and should be used only in a well-ventilated fume hood.  
<p>|                                    | Cyclopentadiene is highly flammable, so care should be taken to avoid open flames. |</p>
<table>
<thead>
<tr>
<th>Procedure/Use</th>
<th>Scale</th>
<th>Engineering Controls/Equipment</th>
<th>PPE (eye, face, gloves, clothing)</th>
<th>Procedure Steps and Special Precautions for this Procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Dicyclopentadiene is most commonly used to prepare the compound cyclopentadiene through thermal decomposition, or “cracking”</td>
<td>Quantities ranging from 5g to 100g.</td>
<td>All work using dicyclopentadiene must be performed in a ventilated fume hood. The distillation apparatus should be dry and free of all impurities. Joints should be greased. Distillation should be executed under an inert atmosphere (argon or nitrogen). Dicyclopentadiene is cracked by heating the reagent to 180 °C and collecting the distillate in a receiving flask cooled to −78 °C by a dry ice/acetone bath. Eliminate ignition sources such as open flames, hot surfaces, steam baths, static electricity, and operation of mechanical and electrical equipment that is not intrinsically safe. Ensure proper grounding and avoid creating static electricity. Be sure to ground metal containers.</td>
<td><strong>Eye protection:</strong> Wear ANSI approved safety glasses with side shields or tightly fitting safety goggles. <strong>Face Protection:</strong> Wear a face shield when handling containers that are not behind a lab hood sash or blast shield. <strong>Gloves:</strong> Wear nitrile gloves. Use proper glove removal technique (without touching glove’s outer surface) to avoid skin contact with this product. Remove gloves immediately upon contamination. Wash and dry hands after use. <strong>Clothing:</strong> Wear flame resistant lab coat, cotton based clothing; full length pants or equivalent; and close-toed and close-heeled shoes.</td>
<td>All glassware should be inspected. The distilled product should preferably be collected in a Schlenk vial and stored in the absence of oxygen to reduce the danger of peroxide formation. Do not allow to evaporate to near dryness unless absence of peroxides has been shown. If stored in a Teflon sealed storage flask dispense through standard air-free canula and syringe techniques. If Dicyclopentadiene is concentrated on the rotary evaporator, use two dry ice traps to collect the solvent vapors. Distillation/evaporation of Dicyclopentadiene efficiently removes all stabilizers. Collected fractions (rotavap) must be treated as unstabilized Dicyclopentadiene and must be disposed of soon after generation. Pressure can be built up if Dicyclopentadiene is used in reactions. Adequate ventilation (pressure bubbler on Schlenk manifold, or an equilibrating balloon) has to be used to prevent dangerous over pressurization. Pressure is built up if Dicyclopentadiene is used in extractions. Adequate</td>
</tr>
<tr>
<td>Notes</td>
<td>Any deviation from this SOP requires approval from PI.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-------</td>
<td>-----------------------------------------------------</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

when transferring flammable liquids. Use a blast shield if Dicyclopentadiene may be distilled to dryness or evaporated to dryness. Only when absolutely necessary to transfer larger quantities of Dicyclopentadiene, use an appropriately-designed, engineered system that is tested and properly used.

ventilation (open the valve frequently during the extraction) has to be used to prevent dangerous over pressurization. If heated, the reaction apparatus has to be fitted with an adequately sized condenser and an adequate flow of cooling water has to be provided to prevent evaporation. Cooling hoses have to be secured with metal hose clamps to the condenser and the outlet. If distillation or evaporation of Dicyclopentadiene to dryness is possible, a second worker should be present outside the potential explosion zone. Cyclopentadiene is highly flammable, so care should be taken to avoid open flames.
Sarpong Group Notes for Dicyclopentadiene

Dicyclopentadiene is a Peroxide Forming Chemical (PFC). Large quantities are coproduced in the steam cracking of naphtha and gas oils to ethylene. Dicyclopentadiene is primarily used in unsaturated polyester resins and also used in inks, adhesives, and paints. It can also be used in polymerization reactions as a monomer for olefin polymerization or in ring-opening metathesis polymerization.

Precautions for safe handling
Avoid contact with skin and eyes. Avoid inhalation of vapor or mist. Keep away from sources of ignition. Take measures to prevent the build-up of electrostatic charge.

Conditions for safe storage
Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage. Store under inert gas. Segregate the chemical and store in secondary containment. Label the bottle, secondary containment, and storage cabinet as “Peroxide Forming Chemical.” Write the date the chemical was received and date the chemical was opened.

Protocol/Procedure
Dicyclopentadiene is most commonly used to prepare the compound cyclopentadiene through thermal decomposition, or “cracking.”
The distillation apparatus should be dry and free of all impurities. Joints should be greased. Distillation should be executed under an inert atmosphere (argon or nitrogen).
For this process, quantities ranging from 5g to 100g are commonly employed.
The process should only be performed in a certified fume hood, and researchers should wear flame resistant lab coats, gloves, and safety glasses.
Dicyclopentadiene is cracked by heating the reagent to 180 °C and collecting the distillate in a receiving flask cooled to −78 °C by a dry ice/acetone bath.
All glassware should be inspected prior to use to verify that it can stand these high temperatures.
The distilled product should preferably be collected in a Schlenk vial and stored in the absence of oxygen to reduce the danger of peroxide formation.
Cyclopentadiene, formed from dicyclopentadiene, has a strong odor and should be used only in a well-ventilated fume hood.
Cyclopentadiene is highly flammable, so care should be taken to avoid open flames.
13 - Documentation of Training (signature of all users is required)

☑ Prior to conducting any work with dicyclopentadiene, designated personnel must provide training to his/her laboratory personnel specific to the hazards involved in working with this substance, work area decontamination, and emergency procedures.

☑ The Principal Investigator must provide his/her laboratory personnel with a copy of this SOP and a copy of the SDS provided by the manufacturer.

I have read and understand the content of this SOP:

<table>
<thead>
<tr>
<th>Name</th>
<th>Signature</th>
<th>Identifier</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>