UCLA ENVIRONMENT, HEALTH & SAFETY

Standard Operating Procedure

3-Chloroperbenzoic acid (MCPBA)

This is an SOP template and is not complete until: 1) lab specific information is entered into the box below 2) lab specific protocol/procedure is added to the protocol/procedure section and 3) SOP has been signed and dated by the PI and relevant lab personnel.

Print a copy and insert into your
Laboratory Safety Manual and *Chemical Hygiene Plan*.
Refer to instructions for assistance.

Department:	Chemistry
Date SOP was written:	5/25/2017
Date SOP was approved by PI/lab supervisor:	5/25/2017
Principal Investigator:	Rongbiao Tong
Internal Lab Safety Coordinator/Lab Manager:	Jingxun Yu
Lab Phone:	23587397
Office Phone:	23587357
Emergency Contact:	Rongbiao Tong 53484541 *(Name and Phone Number)*
Location(s) covered by this SOP:	*CYT/6014* *(Building/Room Number)*

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

Purpose

3-Chloroperbenzoic acid (MCPBA) is a strong oxidizing agent that may cause a fire upon contact with heat and other flammable materials. It may be harmful if ingested, inhaled, or absorbed through the skin. It causes irritation to the gastrointestinal tract, respiratory tract, skin, and eyes. It may cause burns by all exposure routes. MCPBA is mainly used for the oxidation of aldehydes and ketones to esters, olefines to epoxides, sulfides to sulfoxides and sulfones, and amines to nitroalkanes, nitroxides, and *N*-oxides. The compound is not particularly stable above 70% purity so is normally contaminated with 3-chlorobenzoic acid.

Physical & Chemical Properties/Definition of Chemical Group

CAS#: 937-14-4

Class: **Organic peroxide, skin sensitizer, irritant, corrosive**

Molecular Formula: $C_7H_5ClO_3$

Form (physical state): Solid

Color: White

Boiling point: N/A

Potential Hazards/Toxicity

3-Chloroperbenzoic acid is an organic peroxide that may cause a fire when exposed to heat. It may undergo hazardous decomposition. It may be harmful if ingested, inhaled, or absorbed through the skin. It causes irritation to the gastrointestinal tract, respiratory tract, skin, and eyes. It may cause an allergic skin reaction and serious eye irritation. May cause burns by all exposure routes.

Personal Protective Equipment (PPE)

Respirator Protection
Use a full-face particle respirator with type N100 (US) respirator cartridges.

Respirators should be used only under any of the following circumstances:

- As a last line of defense (i.e., after engineering and administrative controls have been exhausted).
- When Permissible Exposure Limit (PEL) has exceeded or when there is a possibility that PEL will be exceeded.
- Regulations require the use of a respirator.
- An employer requires the use of a respirator.
- There is potential for harmful exposure due to an atmospheric contaminant (in the absence of PEL)
- As PPE in the event of a chemical spill clean-up process

Lab personnel intending to use/wear a respirator mask must be trained and fit-tested by EH&S. This is a regulatory requirement. (http://map.ais.ucla.edu/go/1004655)

Hand Protection
Handle with gloves. Nitrile gloves are recommended.

NOTE: Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with 3-chloroperbenzoic acid.

Refer to glove selection chart from the links below:
http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf
OR
http://www.allsafetyproducts.biz/page/74172
OR
http://www.showabestglove.com/site/default.aspx
OR
http://www.mapaglove.com/

Eye Protection
ANSI approved, tight-fitting glasses/goggles. Face shields are recommended.

Skin and Body Protection
Flame-resistant lab coat preferably made of anti-static material, long pants, closed-toe shoes. Refer to UCLA policy 905.

Hygiene Measures
Avoid contact with skin, eyes, and clothing. Wash hands before breaks and after handling.

Engineering Controls

Chemical fume hood. Adequate exhaust and capture filtration. Electrically grounded lines and equipment.

First Aid Procedures

If inhaled
Move person into fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Consult a physician.

In case of skin contact
Flush with plenty of water for at least 15 minutes while removing contaminated clothing. Consult a physician.

In case of eye contact
Flush eyes with plenty of water for at least 15 minutes lifting upper and lower eyelids and removing contact lenses. Consult a physician.

If swallowed
Do not induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

Special Handling and Storage Requirements

Precautions for safe handling: Avoid contact with skin, eyes, and clothing. Avoid inhalation and ingestion. Provide adequate ventilation. Keep away from heat and sources of ignition- No smoking.

Conditions for safe storage: Keep container tightly closed in a cool, dry, and well-ventilated area. Store in original container. Recommended storage temperature is 2-8 °C. Avoid strong oxidizing agents, strong bases, strong reducing agents, organic materials, manganese/manganese oxides, acids, metals, combustible material, and flammable materials.

Spill and Accident Procedure

Chemical Spill Dial 911 and x59797

Spill – 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 (<1 L) – If you have training, you may assist in the clean-up effort. 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.

Large (>1 L) – Dial **911** (or 310-825-1491 from cell phone) and EH&S at x59797 for assistance.

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 x59797 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 x59797 immediately.*

Medical Emergency Dial 911 or x52111

Life Threatening Emergency, After Hours, Weekends And Holidays – Dial **911** (or 310-825-1491 from cell phone) or contact the Ronald Reagan UCLA Medical Center (emergency room) directly at **x52111** (located at 757 Westwood Plaza, enter from Gayley Avenue). *Note: All serious injuries must be reported to EH&S at **x59797** within 8 hours.*

Non-Life Threatening Emergency – Go to the Occupational Health Facility (OHF), **x56771,** CHS room 67-120 (This is on the 6th floor, 7th corridor, room 120. Enter through the School of Dentistry on Tiverton Drive and proceed to the "O" elevator to the 6th floor.)Hours: M - F, 7:30 a.m. to 4:30 p.m. At all other times report to Ronald Regan UCLA Medical Center (emergency room) at **x52111**. *Note: All serious injuries must be reported to EH&S at x59797 within 8 hours.*

Needle stick/puncture exposure (as applicable to chemical handling procedure) – Wash the affected area with antiseptic soap and warm water for 15 minutes. For mucous membrane exposure, flush the affected area for 15 minutes using an eyewash station. Page the needle stick nurse by dialing **231** from a campus phone, enter **93333** when prompted and then enter your extension. Hours: M – F, 8:00 a.m. to 4:00 p.m. At all other times report to Ronald Regan UCLA Medical Center (emergency room) at **x52111**. *Note: All needle stick/puncture exposures must be reported to EH&S at x59797 within 8 hours.*

Decontamination/Waste Disposal Procedure

Wearing proper PPE, decontaminate equipment and bench tops with soap and water. Sweep up or shovel spills avoiding dust formation. Dispose of the used chemical and contaminated disposables as hazardous waste following the guidelines below.

General hazardous waste disposal guidelines:

Label Waste

- Affix an on-line hazardous waste tag on all waste containers using the Online Tag Program http://otp.ucop.edu/ as soon as the first drop of waste is added to the container

Store Waste

- Store hazardous waste in closed containers, in secondary containment and in a designated location
- Double-bag dry waste using transparent bags http://map.ais.ucla.edu/go/1002774
- Waste must be under the control of the person generating & disposing of it

Dispose of Waste

- Dispose of regularly generated chemical waste within 90 days
- Call EH&S at x61887 for questions
- Empty Containers
    - Dispose as hazardous waste if it once held extremely hazardous waste (irrespective of the container size) http://ehs.ucla.edu/Pub/ExtremelyHazardousWaste.pdf
    - Consult waste pick-up schedule http://ehs.ucla.edu/pub/HazWaste%20Pickup%20Schedule.pdf

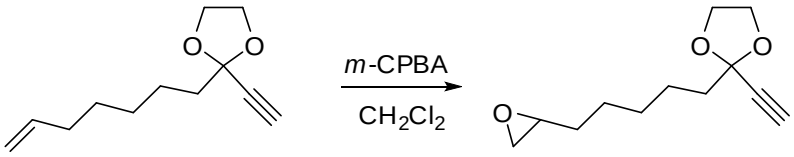
Prepare for transport to pick-up location

- Check on-line waste tag
- Write date of pick-up on the waste tag
- Use secondary containment

Safety Data Sheet (SDS) Location

Online SDS can be accessed at http://msds.ehs.ucla.edu.

Protocol/Procedure (Add lab specific Protocol/Procedure here)



A solution of *m*-CPBA (10.4 g, 70%, 42.3 mmol) in CH_2Cl_2 (80 mL) was added to a solution of olefin (3.73 g, 19.2 mmol) in CH_2Cl_2 (40 mL). The reaction was stirred for 12 h and cooled to 0 °C. 2-Methyl-2-butene (8.1 mL, 76.9 mmol) was added and the resulting mixture was slowly warmed to 25 °C and stirred for 4 h to consume excess *m*-CPBA. The mixture was diluted with saturated $NaHCO_3$ and extracted by CH_2Cl_2. The combined CH_2Cl_2 extracts were washed with saturated Na_2SO_3 (until potassium iodide starch test paper showed no color), 5% NaOH (2 × 30 mL), and water (2 × 30 mL), dried ($MgSO_4$), and concentrated to afford crude epoxide. Flash chromatography on silica gel (10:1 hexanes/EtOAc) yielded 4.03 g (99%) of pure epoxide as colorless oil. (*Org. Lett.*, 8(7), 1283-1286)

GUIDELINES FOR HANDLING ORGANIC PEROXIDES

1. Use only the minimum quantity required.

2. Wear nitrile gloves, eye protection and body protection such as a lab coat or apron.

3. Conduct procedures inside a chemical fume hood or from behind a protective shield.

4. Do not return unused peroxides to the container.

5. Clean up liquid spills up immediately. Keep spills of solid peroxides wet with an appropriate inert solvent (e.g. water or aliphatic hydrocarbon). Cover the spill with a wet (water) mixture (1:1:1, by weight) of sodium carbonate, "kitty litter" or vermiculite, and sand. Dispose promptly as hazardous waste. See Armour (Armour, M. A., Hazardous Laboratory Chemicals Disposal Guide, CRC Press, Boca Raton, FL, 1991.) for recommended procedures for specific

compounds. v Reduce the sensitivity of peroxides to heat and shock by dilution with inert solvents (e.g. aliphatic hydrocarbons).

6. Avoid using solutions of peroxides in volatile solvents. Solvent evaporation should be controlled to avoid dangerous concentration of the peroxide.

7. Do not allow peroxides to contact iron or compounds of iron, cobalt, or copper, metal oxide salts, acids or bases, or acetone.

8. Do not use metal spatulas to handle peroxides.

9. Do not allow open flames, other sources of heat or sparks, friction, grinding or forms of impact near peroxides.

10. Do not use glass containers with screw-cap lids or glass stoppers. Use polyethylene containers, screw caps or stoppers.

11. Protect from heat and light.

12. Store peroxides at the lowest possible temperature consistent with their solubility and freezing point.

CAUTION: Do not store liquid or solutions of peroxides at a temperature below that at which the peroxide freezes or precipitates. Peroxides in this form are extremely shock and heat-sensitive. Refrigerated storage of peroxides or other flammable chemicals must be ONLY in "Lab-Safe" or explosion-proof units.

A reported procedure involving use of mcpba:

Reaction. Geraniol (1.00 g, 6.48 mmol) is weighed into a 100-mL round-bottom flask. CH2Cl2 (25 mL), sodium bicarbonate (NaHCO3, 598 mg, 7.12 mmol), and a magnetic stir bar are introduced into the flask and the mixture is stirred and cooled in a 0 °C ice/water bath. A solution of mCPBA6 (1.76 g, 7.12 mmol, 57–86%—Aldrich, nominal value 70% used for stoichiometry) in 35 mL of CH2Cl2 is added slowly via an addition funnel over a period of 5 min. Almost immediately, a heavy white precipitate forms and the mixture becomes very difficult to stir. The resulting slurry is stirred for 30 min at 0 °C, the ice bath is removed, and the reaction is allowed to stir for 2 hours.

Isolation of Product. The mixture is transferred portionwise to a separatory funnel and is washed twice (or until the aqueous layer is basic to pH paper) with an equal volume of saturated sodium carbonate. The entire organic layer is then washed twice with an equal volume of saturated Na2S2O3 solution. The CH2Cl2 layer is dried over Na2SO4, filtered, and concentrated to give 1.1 g of a mixture of epoxide products.

Purification of Racemic Epoxides by Column Chromatography. A 19- × 300-mm column is packed with a slurry of 25 g of 200–400 mesh silica gel in 30% EA/70% PE. A total of 300 mL of the solvent system is required to both prepare the column and elute the desired epoxide product. The crude epoxide is loaded onto the column and 30 mL of solvent is forced through the column. Fractions are then collected in 12-mL portions; the desired epoxide is eluted in vials 6–9. These fractions are combined and concentrated under vacuum to yield 337 mg (1.98 mmol, 31% yield) of racemic 2, 3-epoxygeraniol as a colorless oil. TLC: silica gel on aluminum backing, 30% EA/70% PE, KMnO4 dip used to visualize; Rf = .50, geraniol; Rf = .31, 2, 3-epoxygeraniol; Rf = .19, 6,7-epoxygeraniol; Rf = .10, 2,3,6,7-diepoxygeraniol (sometimes observed).

Reference: Lynn M. Bradley, Joseph W. Springer, Gregory M. Delate, and Andrew Goodman. Epoxidation of Geraniol: An Advanced Organic Experiment that Illustrates Asymmetric Synthesis. Journal of Chemical Education • Vol. 74 No. 11 November 1997, pp1336-1338.

NOTE

Any deviation from this SOP requires approval from PI.

Documentation of Training (signature of all users is required)

- Prior to conducting any work with 3-chloroperbenzoic acid, 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.
- The Principal Investigator must ensure that his/her laboratory personnel have attended appropriate laboratory safety training or refresher training within the last one year.

I have read and understand the content of this SOP:

Name	Signature	Date
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