

Standard Operating Procedure

Calcium Hydride

This SOP is not complete until it 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 & Biochemistry - Chemical Engineering
Date SOP was written:	December 14, 2012
Date SOP was approved by PI/lab supervisor:	January 18, 2013
Principal Investigator:	Prof. Susannah Scott
Internal Lab Safety Coordinator/Lab Manager:	Stephanie Goubert-Renaudin
Lab Phone:	805-893-8941
Office Phone:	805-893-7403
Emergency Contact:	EH&S 24 hour line: 805-893-3194 (Name and Phone Number)
Location(s) covered by this SOP:	ESB 3324 and 3328. (Building/Room Number)

Type of SOP: Process Hazardous Chemical Hazardous Class

Purpose

Calcium Hydride is a highly water/moisture reactive chemical. In contact with water, it releases flammable gases which may ignite spontaneously. It should therefore be handled under an inert atmosphere. If not handled properly, this can pose a serious threat to the health and safety of laboratory personnel, emergency responders and chemical waste handlers. This SOP helps to understand how to properly store, handle and dispose of Calcium Hydride.

Calcium hydride is widely used as a desiccant for basic solvents such as amines and pyridine. It is also used to pre-dry solvents prior to the use of a more reactive desiccant. Although CaH_2 is indeed convenient and often the drying agent of choice, it has a few drawbacks:

- It is insoluble in all solvents with which it does not react vigorously, in contrast to LiAlH_4 , thus the speed of its drying action can be slow.
- It is incompatible with some solvents, and can in fact explosively react with chlorocarbons (LiAlH_4 also suffers from this disadvantage).
- Because CaH_2 and Ca(OH)_2 are almost indistinguishable in appearance, the quality of a sample of CaH_2 is not obvious visually.

- Since calcium hydride does not remove dissolved oxygen, it is not useful for deoxygenating solvents.

Physical & Chemical Properties/Definition of Chemical Group

CAS#	7789-78-8
Class:	<u>Water Reactive</u>
Molecular Formula:	H ₂ Ca
Form (Physical State):	Light grey, powder, odorless
Boiling Point:	600°C
Melting point:	190°C

Potential Hazards/Toxicity

OSHA Hazards: **Water Reactive**

Pictogram



Potential Health Effects

Inhalation May be harmful if inhaled. May cause respiratory tract irritation.

Skin May be harmful if absorbed through skin. May cause skin irritation.

Eyes May cause eye irritation.

Ingestion May be harmful if swallowed.

Signs and Symptoms of Exposure

Cough, Shortness of breath, Headache, Nausea & Vomiting

Personal Protective Equipment (PPE)

Respiratory protection

General guidelines: Respirators should be used only under any 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.

Note: Self-Contained Breathing Apparatus - SCBA must be used during spill / emergency response.

Hand protection

Handling the chemical with [nitrile or neoprene gloves](#) is recommended. Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with Calcium Hydride.

Eye protection

Safety goggles.

Skin and body protection

Fire/flame resistant lab coat (100% cotton based), full length pants or equivalent, and closed toe shoes.

Hygiene measures

Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling Calcium Hydride.

Engineering Controls

- Recommended storage and usage under inert atmosphere/conditions.

First Aid Procedures

General advice

Consult a physician. Show this safety data sheet to the doctor in attendance. Move out of dangerous area.

If inhaled

If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact

Wash off with soap and plenty of water for at least 15 minutes. Consult a physician.

In case of eye contact

Flush eyes with water for at least 15 minutes as a precaution.

If swallowed

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 and eyes.
- Avoid formation of Calcium Hydride dust and aerosols
- Provide appropriate exhaust ventilation at places where dust is formed.
- Keep away from sources of ignition – Open flames/Bunsen Burner

Conditions for safe storage

- Keep Calcium hydride container tightly closed in a dry and well-ventilated place.
- Hazard communication label on the container must read *Water Reactive* ~~W~~

- Never allow product to get in contact with moisture/water or water based compounds during storage. **Note:** Do not leave the container on the bench top. Do not leave the container near the lab sink, emergency eyewash and safety shower.
- Do not store in humid air/moisture.
- Handle and store under inert gas (Noble gases such as Nitrogen, Argon etc.)
- Recommended to handle inside a **glove box**.
- Keep in a dry place (such as a desiccator or a dry box or glove box).

Chemical stability

Stable under recommended storage conditions.

Possibility of hazardous reactions

Reacts violently with water.

Conditions to avoid

Exposure to moisture.

Materials to avoid

- Strong oxidizing agents, acids and alcohols.
- Reacts violently with water

Hazardous decomposition products

Hazardous decomposition products formed under fire conditions

Reacts with water to form: - Hydrogen gas

Thermal decomposition

600 °C

Spill and Accident Procedure

Fire-fighting & Extinguishing media

Suitable extinguishing media

Carbon dioxide (CO₂) Dry powder / **Class D fire extinguisher**

DO NOT use Water

Special protective equipment for fire-fighters

Wear Self-Contained Breathing Apparatus for fire-fighting if necessary.

Personal precautions

- Wear respiratory protection.
- Avoid dust formation.
- Avoid breathing vapors, mist or gas.
- Ensure adequate ventilation.
- Evacuate personnel to safe areas.
- Avoid breathing dust.

Methods and materials for containment and cleaning up

- Sweep up and shovel.
- Contain spillage, and then collect with an electrically protected vacuum cleaner.
- Do not flush with water.
- Keep in suitable, closed containers for disposal.
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Chemical Spill Dial 9-911 and EH&S (805-893-3194)

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 **9-911 from campus phones (and 805-893-3446 from a cell phone)** and EH&S (805-893-3194) 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 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 immediately.*

Medical Emergency Dial **9-911** (from campus phones)

Life Threatening Emergency, After Hours, Weekends and Holidays – Dial **9-911** (or 805-893-3446 from a cell phone) or go to the Emergency Room of Goleta Valley Cottage Hospital at 351 South Patterson Avenue, Goleta (Phone number: 805-967-3411) *Note: All Serious injuries must be reported to EH&S within 8 hours.*

Non-Life Threatening Emergency – Go to the Student Health Building, Building 588 (phone number: 893-5361, hours: M, T, R, F 8am-4.30pm, W 9am - 4.30pm, R 5pm to 7pm by appointment). After hours go to the Emergency Room of Goleta Valley Cottage Hospital at 351 South Patterson Avenue, Goleta (Phone number: 805-967-3411) *Note: All serious injuries must be reported to EH&S 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 \ and then enter your extension. After hours go to the nearest emergency room: the Emergency Room of Goleta Valley Cottage Hospital at 351 South Patterson Avenue, Goleta (Phone number: 805-967-3411). *Note: All needle stick/puncture exposures must be reported to EH&S within 8 hours.*

Decontamination/Waste Disposal Procedure

NOTE: *Calcium Hydride hazardous waste must be stored away from oxidizing agents/oxidizing chemical waste, acids/acid waste and alcohols (solvent waste).*

General hazardous waste disposal guidelines:

Label Waste

- Affix an on-line hazardous waste tag on all waste containers using the UCSB Hazardous Waste Program 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 or store in glass jar
- Waste must be under the control of the person generating & disposing of it

Dispose of Waste

- Dispose of regularly generated chemical waste within 6 months
- Call EH&S for questions

- Empty Containers
 - Dispose as hazardous waste if it once held extremely hazardous waste (irrespective of the container size)
 - Consult waste pick-up schedule
- Prepare for transport to pick-up location
- Check on-line waste tag
 - Write date of pick-up on the waste tag
 - Use secondary containment

Material Safety Data Sheet (MSDS) Location

MSDS can be accessed online at:

<http://www.sigmaaldrich.com/catalog/product/sial/208027?lang=en®ion=US>

Protocol/Procedure

Please see attached SOP “Procedures for Safe Use of Pyrophoric Solids” (online link: http://web.chem.ucsb.edu/~moretto/SOP_Pyrophoric.pdf)

Prior to starting the reaction, locate the extinguisher, eyewash and safety shower.

In the laboratory, calcium hydride is used as a drying agent for organic solvents.

Calcium hydride is stored in a sealed container in a ventilated cabinet, away from moisture and water.

A flame retardant labcoat, nitrile or neoprene gloves and safety goggles have to be worn at all times when handling calcium hydride.

General procedure for drying organic solvents: Always work in a fume hood and wear appropriate lab attire described above. Add the appropriate solvent to a round bottom flask or side arm Schlenk flask with 10-20g of CaH₂ per liter of solution. Do this under a steady flow of N₂ or Ar. (Note: About ten times the theoretical amount of CaH₂ should be used for drying. The substances to be dried should contain only low amounts of water). When CaH₂ reacts with water, hydrogen is released and bubbles will form. Allow the solvent to stir over CaH₂ overnight or a sufficient length of time. No gas evolution should be present. Once finished using CaH₂ store in a desiccator or in an inert atmosphere (i.e. glove box).

Quenching: Small amounts of unused or unwanted calcium hydride must be destroyed by careful quenching of the residue in the fume hood. Transfer the materials to an appropriate reaction flask for quenching. Dilute significantly with an unreactive solvent such as anhydrous heptane or toluene. Due to the exothermic nature of the reaction, the flask can be placed in an ice water cooling bath. AVOID low boiling diluents such as ether and pentane that tend to condense water upon evaporation. Slowly add isopropanol to quench calcium hydride. Upon completion, add ethanol or methanol as a more reactive quenching agent to ensure completion. Finally, add water dropwise to make sure there are no pockets of reactive materials.

When the solution is unreactive, it is basic and can be disposed in the appropriate basic waste container, kept closed all the time.

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 calcium hydride, designated personnel, i.e. approved users listed below, 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 Calcium Hydride MSDS provided by the manufacturer.
- The Principal Investigator must ensure that his/her laboratory personnel have attended appropriate laboratory safety training or refresher training as required by EH&S.

I have read and understand the content of this SOP:

Name	Signature	Trainer	Date
Prof. Susannah Scott			
Stephanie Goubert-Renaudin			
Gary Kwanyi Ng			
Alessandro Gallo			
Anthony Crisci			
Haibo Yu			
Taeho Hwang			
Bethany Wigington			
Daniel Coller			
Zachary Jones			
Youhong Wang			
Jinghong Zhou			
Jason Fendi			