

UCSC Laboratory Standard Operating Procedure (SOP) Hydrofluoric Acid

Department:	Chemistry and Biochemistry	Date:	May 20 th , 2015
Principal Investigator/Supervisor:	Yat Li	Office Phone#:	831-502-7363
Procedure Author:	Tianyu Liu, Mingyang Li, and Yi Yang	Lab Phone#:	831-502-7363
Location(s) covered by this SOP/Building/Room#:	PSB 198	Author Email:	tliu23@ucsc.edu

Review any applicable manufacturer/vendor safety information, such as a Safety Data Sheet (SDS), before developing the Standard Operating Procedure (SOP).

Any deviation from this SOP requires approval from the PI.

#1 Brief Experimental Summary: Provide a general description of the process and/or experimental procedure.

The hydrofluoric acid (HF) is used as an etchant to dissolve silica particles.




The mineral acid Hydrofluoric Acid poses unique safety and health hazards that require special precautions, PPE, first aid procedures, and training. While it is classified as a weak acid, HF should be handled with extreme care in comparison to other mineral acids, due to its high reactivity. HF penetrates skin and tissues easily, causes delayed reaction burns at lower concentrations, and exposure can lead to permanent injury and death. Personnel must read the SDS and this SOP carefully before beginning training, and must be fully trained by senior lab personnel before beginning work with HF.

The dangers of working with HF are significant. Personnel **MUST NOT** work alone when using HF at any concentration. Other trained lab personnel **MUST** be present in the same room in which the work is occurring and aware of the procedures taking place.

A tube of calcium gluconate (*e.g.* Calgonate) **MUST** be present and readily accessible for use in the work area when using HF.

Keep calcium nitrate solution (1 M, aqueous) on hand for cleaning up small spills.

List the chemicals that fall under this SOP, include CAS#, and GHS symbols and categories:

Chemical (CAS#)	GHS categories	GHS symbols – choose the appropriate symbols for each chemical
Hydrofluoric Acid (7664-39-3)	Acute toxicity, Oral (Category 2), H300 Acute toxicity, Inhalation (Category 2), H330 Acute toxicity, Dermal (Category 1), H310 Skin corrosion (Category 1A), H314 Serious eye damage (Category 1), H318	
Calcium Hydroxide (1305-62-0)	Skin irritation (Category 2), H315 Serious eye damage (Category 1), H318 Specific target organ toxicity - single exposure (Category 3), Respiratory system, H335 Acute aquatic toxicity (Category 3), H402	
Ethanol (64-17-5)	Flammable liquids (Category 2), H225	



#2 Procedure Description: Include all steps for the procedure from the preparation to waste disposal, along with decontamination/clean-up steps. For each step's description, include any step-specific hazard, personal protective equipment, engineering controls, designated work areas, and specific working alone restrictions in the left hand columns. Note the location and use of any emergency response equipment specific to process (e.g., Calgonate gel, Class D fire extinguisher, inert absorbent material).

Working Alone: Working alone with HF is not permitted. Notify your coworkers prior to conducting this work and ensure that at a minimum of 1 person is present in the same room and aware that the work is occurring. Second person must remain present during the entire time HF is in use. If no one is present in the lab, you can't use HF.

Scale: Work on as small a scale as possible. Only make enough HF solution (**100 mL 5 wt% diluted solution**) for the number of samples to be processed that day. **Do not exceed volume of 20 mL HF 5 wt% solution which is needed per sample and 10 mL of concentrated HF used to prepare the 5 wt% HF diluted solution.**

Procedure Steps	Work Location / Safety Equipment	Precautions
<p>A. Prepare 5 wt% HF diluted solution</p> <ol style="list-style-type: none"> 1) Prepare a 50 mL Teflon beaker with flat bottom and place the beaker in a polyester secondary holder to prevent falling. Label the beaker and holder as "HF 5 wt% solution". 2) Use a polyester pipette to transfer 2 mL HF stock solution (50 wt%) to a Teflon beaker. Polyester pipettes can be found below the bench facing to our CVD system. 3) Use a squeeze bottle to dilute the stock solution to 20 mL using deionized water. 4) Put a plastic stirrer into the solution and stir thoroughly for 5 min (must operate in fume hood). Transfer the beaker with HF diluted solution. 5) Prepare one Teflon vial. Label it as "HF 5 wt% solution". 6) Pipette all the 20 mL 5 wt% HF diluted solution to the Teflon vial. Must use a polyester pipette, not a glass one. <p>B-1. Dissolution of silica spheres/fillers</p> <ol style="list-style-type: none"> 1) De-cap the vial. 2) Slightly tilt the vial and put the sample containing silica on the wall. 3) Gently slide the sample into the HF solution to avoid splashing. 4) Re-cap the vial and place the vial, beaker and secondary container in fume hood for 3 days. 3. Decant the HF solution into saturated calcium hydroxide solution to precipitate any HF residue. 4. Add 20 mL ethanol to sample for solvent exchange. 5. Soak the sample in ethanol for 1 day. 6. Decant the ethanol, replenish ethanol with another 20 mL ethanol and soak the sample for 1 day. Repeat step 6 twice. Decant the solution to the waste bottle labeled "waste HF" 7. Suspend the sample in ethanol until you need the sample. 8. When sample is needed, decant the ethanol solution to the "HF waste" container. Let the sample dry on a paper towel. <p>B-2 Dissolution of silica shell</p> <ol style="list-style-type: none"> 1) De-cap the vial. 2) Gently insert the fluorine-doped indium oxide glass with silica film into the HF solution (5 wt% aqueous 	<p>All procedures must be performed in the fume hood of PSB 198. These procedures including diluting HF solution, dissolving silica and solvent exchange with ethanol.</p> <p>PPE includes a barrier coat with sleeves, disposable Tyvek sleeve guards, goggles, and double nitrile gloves are mandatory For working with concentrated HF or solutions which may pose a splash hazard, wear a full face shield in addition to goggles. For lower concentrations of HF, use two layers of 8 mil nitrile.</p> <p>Check the shelf life of the gel. Place calcium gluconate gel in the fume hood, prior to conducting the procedure.</p>	<ul style="list-style-type: none"> ➤ Hydrofluoric acid readily destroys tissue and bone. HF readily penetrates human skin, allowing it to destroy soft tissues underneath and to decalcify bone. ➤ Do not use HF after hours or when no one is around in the lab in case of emergency. Do not work with HF without notifying others prior to your work. ➤ Solutions containing high concentration of HF greater than milli-molar level must only be used inside of an exhausting fume hood in PSB 198. ➤ Ensure that you have the proper Personal Protective Equipment for the corrosive and hazardous solutions that you will be working with; safety goggles, polyethylene and/or nitrile gloves (two layers), blue vinyl apron with sleeves in case of splashes and Tyvek sleeve guards. Once contaminated, these should be regarded as hazardous waste. ➤ Rinse down work bench with deionized water after using HF. ➤ Before starting any work using HF, obtain a tube of the calcium gluconate which is stored in the fume hood of PSB 198 and be familiar in its use (see HF Safety Guidelines Manual). Check the expiration date on the tube. This tube should be located next to the clean hood area and be readily visible by others as they use the fume hood. ➤ Ensure you have several small (~50 mL) polyethylene bottle in



<p>solution prepared in section A) for 15 s.</p> <ol style="list-style-type: none">3) Taking out the sample with a plastic tweezers and transfer it into a beaker filled with DI water (~50 mL), recap the vial.4) Keep the sample in the DI water for 1 min.5) Take out the glass with a plastic tweezers and wash it with DI water. Blow dry with compressed air.6) Decant the HF solution as well as all used DI water into the waste bottle labeled with “waste HF”. <p>C. Clean-up</p> <ol style="list-style-type: none">1. All used beakers, vials must store in the secondary container labelled as “HF” in the fume hood.2. Pour excessive amount of saturated calcium hydroxide solution into the HF waste to neutralize HF and precipitate fluoride ions. Add calcium hydroxide solution until no white precipitation is formed.3. Spray calcium hydroxide solution to the working area and then cleaned by use of paper towel(s).4. Thoroughly wash hands afterwards and put barrier lab coat in the lab area. No barrier lab coats are allowed in the office.5. All contaminated stuffs should be treated as “hazardous wastes” and dispose appropriately. Refer to “Precautions” column on the right for how to dispose these hazardous wastes.		<p>the hood for any HF contaminated liquid wastes that might be generated. Once used, the bottle must be labelled as “waste HF” with the data immediately. After working, generate a Haz Waste Tag which should be attached to a bag for storage and taped to the outside of the clean-hood until enough volume has been generated to be picked up by EH&S staff.</p> <ul style="list-style-type: none">➤ Post the “Hydrofluoric Acid In Use” warning sign on the glass shield of the hood so that it is easily visible to others working in the lab. Leave this sign posted until HF is no longer in use in the same fume hood.➤ Unless actively being used on a daily basis, store properly labeled HF stock solution in a tightly capped reagent bottle. Store the reagent bottle inside of the corrosive acid cabinet in PSB 198. The HF stock solution should be removed to the cabinet as soon as enough HF stock solution is acquired.➤ Make sure to keep your HF bottle tightly capped and out of the way on the work bench unless actively in use.➤ In case of large spills, evacuate the area, notify others and contact EH&S by calling 9-2553. NEVER try to clean it by yourself.
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Chemical Equation Graphic (optional):

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#3 Personal Protective Equipment (PPE): *List the personal protective equipment used during this process.*

Note: PPE is to be worn by those conducting the work and any adjacent personnel.

Ensure that you are wearing appropriate clothing before donning PPE. Full length pants or skirt and closed-toe shoes must be worn at all times by all individuals that are occupying the laboratory area. The area of skin between the shoe and ankle should not be exposed. PPE for concentrated HF includes a Tyvek, Dacron or Cotton/Dacron blend lab coat, vinyl apron with sleeves, disposable Tyvek sleeve guards, goggles, and 22 mil neoprene or nitrile gloves with a single layer of nitrile or tripolymer gloves underneath. For working with concentrated HF or solutions which may pose a splash hazard, wear a full face shield in addition to goggles. For lower concentrations of HF, use two layers of 8 mil nitrile or 6 mil MAPA Trilites tripolymer gloves.

Eye Protection: ANSI-approved properly fitting safety glasses or goggles. Chemical splash goggles and/or full face shield during activities which pose a splash hazard.



Body Protection: An appropriately-sized lab coat must be worn and buttoned. Laboratory coat sleeves must be of sufficient length to prevent direct skin exposure while wearing gloves. Full length pants (or equivalent) and closed toe/heel shoe attire must be worn at all times by all workers who are occupying or entering a laboratory/technical area. The area of skin between the pants and shoe should not be exposed.

Check box for specialty lab coat: Nomex/Flame Resistant Biological Barrier Other Barrier Lab Coat

Hand Protection: Wear chemical-resistant gloves; remove gloves and wash hands with soap and water after use. Double gloves may provide additional protection for some chemicals. If prolonged contact or immersion is anticipated, consult with EH&S to identify appropriate protective gloves.

Additional Protection: Face Shield Chemical-Proof Apron Respiratory Protection
 Additional Gloves: two layers of nitrile or Trilites triopolymer gloves recommended Other: Calcium gluconate must be available

#4 Incompatible Conditions and Materials: List the incompatible conditions, chemicals, and/or materials that should be avoided, along with the safe storage conditions.

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Material:	Incompatibility:	Storage Conditions:
Hydrofluoric Acid	Metals, Alkali metals, Strong bases	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. Hydrofluoric acid should always be contained in a polyethylene or polypropylene plastic container and kept within a secondary polyethylene or polypropylene containment vessel and stored within an approved acid cabinet.
Calcium Hydroxide	Strong acids	Keep container tightly closed in a dry and well-ventilated place. Air and moisture sensitive. Keep in a dry place
Ethanol	Alkali metals, Ammonia, Oxidizing agents, Peroxides	Hygroscopic.

#5 Training: Training required for all personnel conducting this procedure. Include any specific training requirements.

BEFORE USING HF YOU MUST FIRST ATTEND THE EH&S TRAINING CLASS, READ THROUGH THE HF SAFETY GUIDELINES BINDER LOCATED (“UCSC Illness and Injury Prevention” binder, on the second shelf of the bookshelf in PSB 198B), WHICH CONTAINS THIS SOP AND THE SDS SHEETS FOR ALL HAZARDOUS CHEMICALS IN THIS SOP, AND SIGN THE HF USER AUTHORIZATION FORM AFTER READING CONTENTS OF BINDER. (It is recommended that each lab has a similar binder and authorization form.) First time users should first watch a trained person perform the procedure, then perform the procedure under supervision.

- Complete EH&S online “Laboratory Safety Fundamentals” class available through the UC Learning Center (<http://learningcenter.ucsc.edu/>).
- Review and sign Lab-Specific Training Checklist (<http://ehs.ucsc.edu/lab-safety-manual/training.html#lab-specific%20training>) with PI, Lab Safety Representative, or other designated person.
- Review SOP with knowledgeable person.
- Complete training on specialized equipment prior to use (e.g., ultracentrifuge, hydrogenation apparatus).
- Other EH&S training requirements (e.g., Biosafety, Radiation Safety, Hazardous Waste Management).
- [Click here to enter text.](#)

#6 Clean-Up, Spill, and Emergency Response Procedures (reference the SDS as needed): *Provide any specific information.*

Decontamination/Clean-Up: Wash bench and/or work area with soap and water after using.

Specific Spill Clean-Up Procedures: **Incidental Spill:**

Small spills of dilute HF may be cleaned by lab personnel if they are trained in spill cleanup of Hydrofluoric Acid and have the ability and equipment (such as commercially available HF spill clean-up kit) to safely clean up the spill. Alert all other personnel that may be affected by the spill. Shut off all sources of heat. Keep fume hoods running to dilute and/or remove vapors. Use Calcium Hydroxide or Calcium Carbonate powder to absorb and neutralize the spill. Manage spill debris as hazardous waste.

Note: Only HF specific absorbents should be used on a HF spill. Sand, kitty litter, or other silicon-based absorbent materials (which are common in most solvent spill kits) should not be used on HF spills due to the potential for the generation of silicon tetrafluoride (a toxic and corrosive gas).

Hazardous Spill:

Report the emergency by dialing 911. Contain the spill and prevent from reaching drains if it is possible to do so without exposing yourself to the liquid or vapor. Remove all sources of heat if safe to do so. Warn others in the area. Evacuate to a safe area. Attend to any person that has been exposed to the material, utilizing emergency eyewashes and showers if safe to do so.

Waste Disposal:

Excess Hydrofluoric Acid and all waste material containing Hydrofluoric Acid must be placed in a container which is stored in a Storage Accumulation Area (SAA) with the appropriate label and disposed through EH&S.

Do not attempt to clean up any spill or release for which you are not fully trained and equipped. For assistance with spill cleanup, dial **911** and ask dispatch to page EH&S.

- Isolate the area to prevent the spread of contamination (e.g. close doors to affected area, post warning signs, alert others in immediately vicinity to evacuate).
- Prevent spill from reaching drains or from spilling outside of the fume hood if possible to do so without exposing yourself to liquid or vapor.
- Clean the affected area and all exposed equipment with soap and water to remove any contaminants before resuming work.
- Spill clean-up materials should be disposed of as hazardous waste.

Laboratory Emergency Response Equipment: *All research personnel must know location of nearest fire alarm pull station and emergency shower/eyewash. Do not use fire extinguisher unless you are trained to do so. List locations for nearest fire alarm pull and emergency shower/eyewash.*

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Calcium gluconate gel: stored in a box labeled with “calcium gluconate”, at upper-left corner inside of the fume hood in PSB 198

Fire alarm pull station: Outside the lab, face to the lab entrance

Emergency shower: Near the entrance of the lab area, besides several gas cylinders

Emergency eyewash: The same place as the emergency shower listed above

Emergency Shutdown Procedures: Click here to enter text.

#7 Hazardous Waste(s): *List expected concentrations and amounts of hazardous waste(s) generated during this process. Provide any special/specific waste management. Contact EH&S for specific guidance regarding hazardous waste handling and disposal. General hazardous waste management guidelines: <http://ehs.ucsc.edu/programs/waste-management/index.html>*

Solutions containing HF must be stored in HDPE containers, not glass. Any waste generated must be stored in a proper container, tightly closed, and in secondary containment with appropriate labeling.

NOTE: Segregate acid waste containers from base waste containers.

Waste Labeling

- Affix an on-line hazardous waste tag on all waste containers using the WASTE application <https://ehs.ucop.edu/waste/> as soon as the first drop of waste is added to the container.

SOP: Hydrofluoric Acid

PI: Yat Li



Waste Storage

- Store hazardous waste in closed containers with venting cap, in clean secondary containment, segregated by hazard class, in a marked and designated waste accumulation area.
- Double-bag dry waste using transparent bags.
- Waste accumulation area must be under the control of the person generating the waste.

Waste Disposal

- Hazardous waste must be removed from the lab within 180 days.
- Containers must be clean, sealed, and safe to transport.
- Mark container as ready for pick up in WASTE, move container to accumulation area.

Contact EH&S at x9-3086 or hazwaste@ucsc.edu with any questions.

#8 First Aid / Emergency Procedures: Describe immediate First Aid or medical treatment required in case of personnel exposure.

General advice

In all cases of exposure to HF contact EH&S and medical help immediately by calling 911. Immediate medical attention is required even if burns don't immediately appear, as HF can cause damage up to 24 hours after exposure. Consult a physician. Show the safety data sheet to the doctor in attendance.

The recommendations below are not intended to replace treatment by medical professionals. For more information on HF, including first aid procedures, see http://www.cdc.gov/niosh/ershdb/EmergencyResponseCard_29750030.html

All exposure to or contact with HF should receive immediate first aid and medical evaluation, even if the injury appears minor or no pain is felt. HF can produce delayed effects and serious tissue damage without necessarily producing pain

For immediate medical assistance, dial **911**. Report all serious injuries to EH&S as soon as possible.

- **Eye Contact** Rinse immediately with plenty of water, also under the eyelids, for at least 15 minutes. **Immediate medical attention is required. Do not apply Calgonate Gel to eyes. Use Calgonate emergency eyewash if available, otherwise rinse with water.**
- **Skin Contact** **Immediately** (within seconds) wash affected area of skin at sink if a small area of hand or forearm has been contaminated or at a safety shower if upper arms, torso, or legs are contaminated. Remove any clothing or jewelry in the affected area that could trap HF. Remove goggles last. Face flow of water, close eyes, and pull goggles off over head. Be sure to pay close attention to washing the fingernails in the case of hand exposure. HF burns around the fingernails are extremely painful, difficult to treat, and may require surgical removal of the nail. **Limit rinsing time to 5 minutes so that application of Calgonate Gel (calcium gluconate 2.5%) can be quickly initiated to limit the migration of the fluoride ion.** Continually massage Calgonate Gel into affected area of skin until medical help arrives. Gel must be continually applied to be effective. If victim cannot apply Calgonate gel themselves, ensure that helpers wear full PPE while applying gel. **Immediate medical attention is required.**
- **Inhalation** Move to fresh air. If breathing is difficult, give oxygen. Do not use mouth-to-mouth resuscitation if victim ingested or inhaled the substance; induce artificial respiration with a respiratory medical device. **Immediate medical attention is required.**
- **Ingestion** Do not induce vomiting. Give 4-8 ounces of milk or water as soon as possible to dilute the acid. (Avoid giving too much water, it may induce vomiting.) Give 4-8 ounces of Milk of Magnesia, Mylanta, Maalox or similar product, or Tums, Caltrate or other antacid tablet. The calcium or magnesium in these products may act as an antidote. Never give anything by mouth to an unconscious person. Seek prompt medical attention – have a lab mate call 911 **Call a physician immediately.**
- Seek medical attention immediately, and the SDS should go with the person to the emergency room.
- If Calgonate Gel has been used, dispose of any unused portion of the tube, and ensure that a replacement is obtained before any further work with HF.

General first aid instructions (other than HF exposure):

For immediate medical assistance, dial **911**. Report all serious injuries to EH&S as soon as possible.

- If inhaled, move into fresh air immediately.
- In the case of eye or skin contact, flush with water for a minimum of 15 minutes. Ensure that eyelids are held open while



rinsing eyes.

- If ingested, flush mouth with water (only if the person is conscious).
- In the case of a needlestick/puncture injury, wash the affected area with soap and warm water for 15 minutes. For employees, follow the instructions at the Risk Services website: <http://risk.ucsc.edu/workers-comp/reporting-and-treatment.html>
- Seek medical attention immediately.
- Complete incident report form, <http://risk.ucsc.edu/all-forms/wc-incident-report-form.pdf>, (contact EH&S) and/or follow the instructions at the Risk Services website: <http://risk.ucsc.edu/workers-comp/reporting-and-treatment.html>

As the Principal Investigator, it is your responsibility to ensure that all individuals conducting this protocol are taught the correct procedures for safe handling of the hazardous materials involved. It is also your responsibility to ensure that your personnel complete Laboratory Safety Training and other applicable safety training courses.

- Prior to conducting any work with, the PI or designee must provide training to his/her laboratory personnel regarding the specific 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 year.

I have reviewed and approve this Standard Operating Procedure.

A handwritten signature in black ink, appearing to read "Yat Li".

I understand that checking this box constitutes my approval of this document on [Click here to enter a date.](#)

PI Signature/Approval: Yat Li

DATE

Chemical Information Summary

Provide information for all chemicals included in the SOP. See the SDS for detailed toxicity information. Add more lines as needed.

Physical & Chemical Properties

Chemical	CAS#	Molecular Formula	Structure	Molecular Weight (g/mol)	Density (g/mL)	Form (physical state)	Melting Point (°C)	Boiling point (°C)	Flash point (°C)
Hydrofluoric Acid	7664-39-3	HF	HF	20.01	1.15	Liquid	No data available	No data available	No data available
Calcium Hydroxide	1305-62-0	Ca(OH) ₂	Ca(OH) ₂	74.09	2.24	Powder	>= 450 (>= 842 °F)	No data available	No data available
Ethanol	64-17-5	C ₂ H ₆ O	CH ₃ CH ₂ OH	46.07	0.7974	Liquid	-144.0 (-227.2 °F)	78.0 - 80.0 (172.4 - 176.0 °F)	14.0 (57.2 °F) - closed cup

Exposure Limits/Toxicity Data

Chemical	Color	Odor	Cal/OSHA PEL	Toxicity LD ₅₀
Hydrofluoric Acid	No data available	No data available	0.4 ppm, as F; 1 ppm (STEL)	Oral (rat): > 90 mL/kg
Calcium Hydroxide	Beige	No data available	5 mg/M ³	Oral (rat): 7,340 mg/kg
Ethanol	Colorless	No data available	1,000 ppm	Oral (rat): 7,060 mg/kg; LC ₅₀ Inhalation (rat): 10 h - 20000 ppm

