



UCSC Laboratory Standard Operating Procedure (SOP) Hydrobromic Acid

Department:	Chemistry	Date:	06/12//2015
Principal Investigator/Supervisor:	Yat Li	Office Phone#:	831-459-1952
Procedure Author:	Yi Yang and Tianyu Liu	Lab Phone#:	831-5027363
Location(s) covered by this SOP/Building/Room#:	PSB198	Author Email:	yyang32@ucsc.edu 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.

Hydrobromic acid is used to prepare methylammonium bromide ($\text{CH}_3\text{NH}_3\text{Br}$), a crucial component for fabricating perovskite-based solar cells.

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
Hydrobromic Acid (HBr) (10035-10-6)	Skin corrosion (Category 1B), H314 Serious eye damage (Category 1), H318 Specific target organ toxicity - single exposure (Category 3), Respiratory system, H335	
Methylamine Solution (74-89-5)	Flammable liquids (Category 2), H225 Acute toxicity, Oral (Category 4), H302 Acute toxicity, Inhalation (Category 3), H331 Skin corrosion (Category 1A), H314 Serious eye damage (Category 1), H318	
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 is not recommended. Notify your coworkers prior to conducting this work and ensure that at a minimum of 1 person is nearby and aware that the work is occurring.

Scale: Work on as small a scale as possible. Do not exceed volumes/masses of 20 mL, without prior consultation with and approval by the PI.

Procedure Steps	Work Location / Safety Equipment	Precautions
All the following procedures must be conducted in a fume hood except heating processes involving electric oven. 1. Place a 150 mL beaker in ice bath and add in 10 mL methylamine using a pipette. 2. Add in 15 mL hydrobromic acid using another	Working in fume hood, wear all applicable PPE.	HBr vapors are hazardous. All work with HBr should be carried out in a fume hood.



<p>pipette and stir thoroughly until a clear solution is formed.</p> <p>3. Warm the prepared solution to room temperature and then put it in an electric oven to heat at 70 °C for 12 h, white precipitation will form under the bottom of the beaker after reaction. The beaker should be covered by a glass petri dish, but not an aluminum foil.</p> <p>4. Filter the precipitation and wash it using absolute ethanol for two times to remove any impurities.</p> <p>5. Dry the collected precipitation in an electric oven at 50 °C for 3 h until white powder is obtained.</p> <p>6. Store the white powder in a labelled vial or other containers.</p>		
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Chemical Equation Graphic (optional):

[Click here to enter text.](#)

#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.

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 [Click here to enter text.](#)

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 [Click here to enter text.](#) Other [Click here to enter text.](#)

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

- 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 flammable liquids in a flammable storage cabinet.

Material:	Incompatibility:	Storage Conditions:
HBr	Strong bases, Strong oxidizing agents, Ammonia, Ozone, Fluorine	Store in secondary containment at all times. Store segregated from bases.
Methylamine	Nitromethane, acids, oxidizing agents, chlorine, hypochlorite, halogenated agents, mercury, copper, copper alloys, zinc, zinc alloys, aluminum, perchlorates.	Keep away from heat, sparks, and flame. Keep away from sources of ignition. Store in a cool, dry, well-ventilated area away from incompatible substances. Flammables-area. Keep away from acids.
Ethanol	Alkali metals, Ammonia, Oxidizing agents, Peroxides	Hygroscopic.

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

- 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: Small Corrosive: Wear appropriate PPE, clean up using available materials. Click here to enter text.

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.*

In case of emergency, fire alarm pull stations are located in the hallway outside of PSB 190. Fire extinguishers are also located in the hallway and have red signs above them. Emergency eyewashes are located at every sink and the emergency shower is located directly to the right as one enters the lab.

Emergency Shutdown Procedures: For emergency shutdown, close fume hood sash and initiate emergency fume hood exhaust. A red button to the top right of the hood will start the emergency exhaust. Grey button next to red button will silence emergency exhaust alarm.

#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>*

Dispose of spent HBr and disposables contaminated with HBr as hazardous waste.

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.

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.

Click here to enter text.

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 be "Yat Li".

X I understand that checking this box constitutes my approval of this document on 6/16/2015

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)
Hydrobromic acid	10035-10-6	HBr	H-Br	80.91	1.49	Liquid	No data available	100 (212 °F) at 1,013.25 hPa (760.00 mmHg)	Not flammable
Methylamine Solution	74-89-5	CH ₅ N	CH ₃ NH ₂	31.06	0.897	liquid, clear	-40 (-40 °F)	48 (118 °F)	-10 (14 °F) - closed cup
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
Hydrobromic acid	light yellow, light brown	Odorless	3 ppm (Ceiling)	LC ₅₀ Inhalation (rat): 1 h - 2858 ppm
Methylamine Solution	Colorless	Unpleasant	5 ppm; 15 ppm (STEL)	Oral (rat): 698 mg/kg; LC ₅₀ Inhalation (rat): 4 h - > 2.1 - < 2.9 mg/l



Chemical	Color	Odor	Cal/OSHA PEL	Toxicity LD ₅₀
Ethanol	Colorless	No data available	1,000 ppm	Oral (rat): 7,060 mg/kg; LC ₅₀ Inhalation (rat): 10 h - 20000 ppm



Documentation of Training (signature of all users is required)

I have read and understand the content of this SOP:

Name	Signature	Date