

Chemical Training for Nanofab.

EMERGENCY CONTACTS

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1. Scope: The purpose of this training is to familiarize the student with the equipment, procedures and safety protocols associated with wet chemical operations in the NanoFAB cleanroom. Topics include: proper storage of solvents, solvent polarity, hazards of solvents due to toxicity, flammability and reactivity, proper safety procedures, proper disposal of used halogenated and non-halogenated solvents, properties of acids and bases, proper mixing and dilution of acids and bases, proper storage of acids and bases, safety procedures for dealing with acids and bases, and procedures of properly disposing of waste solvents and acids.

2. Required Materials: Students are required to supply their own equipment and materials for this training. “Borrowing” from the teaching fab, Bay 1 hoods or racks, or anybody else will not be permitted. Required materials include, but are not limited to:

Safety Glasses—any person coming for chemical training must have their safety glasses. Regular eyeglasses are not acceptable. It is recommended that personnel obtain a chain or strap, of the type used for hanging reading glasses around the neck, so that they can keep their glasses with them at all times.

Tweezers (both metal and PFA or Teflon). PFA tweezers are cumbersome and have very little gripping strength. For most purposes, metal tweezers and a PFA/Teflon dipper, or holder, are preferable. If whole wafers are to be used, it is advisable to obtain “limited grip” metal tweezers. These instruments have a right-angle bend in the tips, so that only the edge of wafers can be gripped. This prevents accidentally destroying devices on the wafer. For small pieces of wafers, flat “duck-bill” tweezers are superior. The very sharp tweezers used for manipulating individual die are not a good choice for handling wafers. These tweezers will grip at only one point. The wafer will slip and rotate around this point, resulting in a scratched wafer, which will be susceptible to breaking. Teflon coating of tweezers does little to protect the wafers, and does not make them suitable for immersion in acidic solutions. It is important to protect tweezers tips so that they do not develop burrs, which will scratch wafers, ultimately leading to wafer breakage.

Diamond-tipped scribe: All groups whose work requires cleaving wafers should own one or more of these. Protect the tip from damage with a cap, so that the diamond tip will not be knocked off. In order to cleave wafers, make a very small scratch at the edge, then grip the wafer on either side of the scratch with two pair of tweezers. Bend the wafer along a cleavage plane so that the wafer will break cleanly. For (100) material, this means gripping either parallel, or perpendicular, to the flat.

Cleanroom Notebook: This is a plastic-bound, spiral notebook made of lint-free paper. Other types of notebook are not permitted in the Cleanroom. It is available from UTA Book Store.

Wafer holder (PFA or Teflon): Examples of these may be found on <http://www.wafercare.com/Default.asp?G=66>. These are, by far, the most effective means of handling individual wafers in wet-chemistry procedures. It is strongly advised that, at the minimum, each faculty member's group have at least one of these available for Cleanroom work.

Beakers of a size appropriate to the wafers to be processed: Pyrex is suitable for solvent cleaning and for most acid etch procedures. Teflon or PFA is required for procedures using HF. Please consult with the instructor **before** your training session in order to assure that you have the correct size, material and number of beakers. Generally, you will need at least two beakers large enough to hold your material and the wafer holder. One will be used for the etch or clean process, the other will hold DI water, and will be used for rinsing the cleaned or etched wafer. For multi-step solvent cleaning processes, one wafer-holding beaker will be required for each solvent step, as well as one to transport the wafer to the acid hood if the solvent clean is to be followed by an acid clean or etch. Several small beakers (100-250 ml) will be required for pouring reagents into graduated cylinders.

Graduated Cylinders: These should be of a size appropriate to the volumes to be measured. It would be inappropriate to use a 1 liter graduate to measure 10 ml, and it would be equally inappropriate to measure 1000 ml using a 50 ml graduate. Properly used, a graduate cylinder can measure volumes to within 1%. Beakers are rated to +/- 5%, but are usually much worse. Graduate cylinders are available in both pyrex and in heavy-duty plastic suitable for measuring HF.

Pasteur Pipets: These can be either disposable plastic, or disposable borosilicate glass. They are useful for adjusting the level of liquids in graduate cylinders. The borosilicate glass type have a very long tip that is useful for adjusting the volume of liquids in graduate cylinders.

3. Laboratory "Wet" Equipment: The Cleanroom is equipped with a solvent hood, an acid hood, and a "develop" hood. The acid and develop hoods are equipped with city water, which may be used to provide cooling water for heat-generating reactions. All hoods have nitrogen blow guns. The acid hood is equipped with high-purity deionized water.

The hoods are equipped with clear plastic face shields. These serve two purposes: obviously, they provide protection from chemical splashes or other mishaps. They also help protect the operator from toxic vapors. The hood exhaust removes a limited volumetric flow rate of air through the hood. If we assume that this volume is constant, regardless of the cross sectional area (not quite true, but a reasonable operating assumption), then the velocity of the air will be inversely proportional to the area. Thus, maintaining the smallest possible cross sectional area will create the highest possible

velocity, which will more effectively pull fumes away from the operator. Unless there is a genuine need, and there are no sources of toxic vapors present in the hood, the face shields are to be left in “down” position at all times.

Solvent Hood: This hood is to be used for operations requiring non-aqueous solvents. It is made of stainless steel in order to be fire resistant. The hood exhaust ducting is galvanized steel. Consequently, acid solutions should not be used in this hood, as the vapors will corrode both the hood and the exhaust duct. It is permissible to use ultrasonic cleaners or hot plates in this hood, although it is important to watch these processes closely. The cabinet beneath the hood is used for storing solvent waste bottles, and for bottles of general lab use solvents, such as acetone and isopropyl alcohol. No chemicals may be stored in the hood. These must be placed in appropriate, labeled bottles and stored in the Solvent cabinet. Beakers covered with aluminum foil are not acceptable for chemical storage and will be disposed of.

Solvent Cabinet: This cabinet is to the immediate left of the solvent hood. It is made of steel, and is connected to the same exhaust duct as the solvent hood. All chemicals stored in this hood must be in closed bottles or cans, and must be clearly labeled with the contents, the name of the owner, and the date. The label should be updated at least every six months. Containers that have not been updated for six months will be considered to be abandoned, and will be disposed of.

Solvents that are for common use will be labeled “Nanofab.” Solvents with an individual’s name on them are considered private property. If you want to try those, you must contact the owner. If the owner is willing to give you some amount, he must pour that by himself in your empty bottle in your presence. No verbal or email permissions are acceptable.

Acid Hood: The acid hood is a large, rebuilt diffusion tube cleaning hood. Consequently, it has enough space for two to four people to work at the same time. The hood has two sinks, each equipped with city water. This water may be used for cooling water baths when diluting concentrated sulfuric acid, or for rinsing items that do not require ultra-pure water. When ultra-pure water is required, it is obtained from the deionized water (DI) tap. Presently, there is only one tap, on the left side of the hood. An additional tap will be added at a later date. The acid hood is made of polypropylene; thus, it is not suitable for use with solvents, other than water. The hood is connected to a fiberglass exhaust duct, which is flammable. Burning solvent vapors could ignite this material. It is imperative that volatile solvents not be used in this hood.

University policy prohibits putting any chemical down the drain. It is permissible to allow rinse water from empty glassware and from wafers to go down the drain. All waste acids must be collected in proper waste bottles as given in Table 1.

Chemicals may not be stored in the solvent hood. However, the cabinet below the hood is used for storing waste acid bottles (right side) and commonly used fresh acid bottles (left side).

It is permissible to use both hot plates and ultrasonic cleaners in the acid hood.

Acid Cabinet: The acid cabinet is located at the far end of Bay 1, next to the double doors leading to the chase, on the left hand side. The cabinet is polypropylene, and connected to the fiberglass exhaust. It, like the acid cabinet is unsuitable for organic solvent storage. The top two shelves are used for storing both Nanofab (open use) acids, and individually owned bottles of acid. Mixed solutions may also be stored, provided that they are in closed, labeled containers. High density polyethylene bottles are the preferred container. The label must list the contents, the owner and the date prepared. Bottles that are not updated within six months will be considered abandoned and will be disposed of.

Develop Hood: The develop hood is small, and is used primarily for developing photolithographic patterns. All developer solutions are alkali, thus they are used in this hood. The hood is equipped with a nitrogen blow gun and city water. Used developer is to be disposed of in a properly labeled waste bottle, and is stored in the Base cabinet, or in the cabinet under the develop hood. Do not store chemicals in the hood.

Base cabinet: The base storage cabinet is located by the entrance to bay 1. All base solutions and all alkaline materials, such as NaOH and KOH, are stored in this cabinet.

DI water system: The Millipore Ultra Pure water system is located between the acid and the develop hoods. City water is first passed through both particulate and activated carbon filters (located in the chase). The lower unit on the system is the reverse osmosis system. Filtered water first passes through a deionizing column and then a semi-permeable membrane. The membrane removes approximately 99% of the material dissolved in the water. The water then flows to the storage tank above the system. When the point of use tap in the acid hood is opened, water flows from the tank through the polishing system, which contains a high quality deionizing column. The water is illuminated with short wavelength uv light, which oxidizes any bacteria or fungal spores. The oxidized carbon, in the form of carbon dioxide then reacts with the water to form bicarbonate ion. This is removed with yet another deionizing column. When water is being drawn from the tap in the acid hood the display on the unit will indicate the resistivity of the water in M Ω -cm. There is an excellent correlation between the electrical resistivity of water and the amount of dissolved material in the water. The highest resistivity that water can attain, at room temperature, is 18.2 M Ω -cm. Should the indicated resistivity be less than 18.2 M Ω -cm, notify Cleanroom personnel that the system requires service. The unit will also indicate the amount of Total Organic Carbon (TOC) present in the water. When the system is functioning properly, the TOC indication will be <1 ppb. If the TOC indication is >1 ppb, notify Cleanroom personnel that the system requires service. Note that the amber "service required" LEDs on the system may be flashing. These are activated by a clock in the system, rather than by any actual deterioration of the water quality. The system will be serviced based on water quality, rather than on how long the components have been installed in the system.

4. Chemical Operations:

Personal Protective Equipment (PPE): PPE is available for use while using chemicals at NanoFab. It consists of (1) chemical-resistant gloves, (2) protective sleeves, (3)

protective aprons and (4), protective face shields. It should be worn anytime hazardous chemicals are used. Aprons are hung on the right side of the acid hood, by the DI water system. Gloves and sleeves are stored in the cabinet above the Base hood. Face shields are stored on top of the Acid hood. The correct method of donning PPE is as follows:

(1) Put on an apron. The apron will have “FRONT” written on the front side. This side should face away from you. This is done so that, if someone spills a chemical on the front of an apron, the next person does not don the apron with chemical next to his body.

(2) Obtain left and right gloves in an appropriate size from the cabinet on top of the Base hood. If there are no gloves in a size appropriate to your hand, ask a staff member for some.

(3) Inspect the inside of the gloves for moisture. Do not use gloves that are wet inside. Dispose of them and request new gloves.

(4) Test the gloves by first opening them wide, then folding over the open end so that air is trapped inside. Squeeze the glove so that a slight pressure is developed. If you feel air blowing out, there is a leak. Dispose of the gloves and request new gloves.

(5) If the gloves are good, put them on.

(6) Obtain a pair of sleeves from the Base hood cabinet. Assure that they are free from obvious contamination. The sleeves have a large opening, and a small opening. Put your gloved hand through the large opening, and pull them up to your elbow. The small end should be over the wrist portion of the glove. The glove should be inside the sleeve, not on the outside. This way, if chemicals splash on your sleeve they will not run down inside the glove.

(7) Put on a face shield. The face shields can be adjusted to fit your head. Additionally, the tension on the visor can be adjusted so that it can be raised and lowered. If you have difficulty, seek assistance from a staff member.

(8) Remove the equipment, when you are finished with it, in the opposite order. Dispose of any contaminated or defective equipment.

Solvent Cleaning: Organic solvents are commonly used to remove organic contaminants from semiconductor materials. Historically, wafers obtained from manufacturers were contaminated with wax used to hold the wafers in place during polish operations. Heated organic solvents were used to remove this organic contamination from wafers. Modern material suppliers have developed very sophisticated and effective methods of assuring that the wafers they supply are extremely clean. Consequently, it is no longer necessary, or desirable, to conduct organic cleaning operations on new wafers. However, wafers that are being reused may require organic cleaning. The solvents most commonly used for “degreasing” operations are tetrachloroethylene (also known as perchloroethylene) and trichloroethylene. Both are classed as halogenated solvents which means that they

contain halogens, in this case, chlorine. These materials are carcinogenic, and they are also ozone depleting substances (ODS). Their use is strongly discouraged and should be minimized. It is likely that, in the future, their use will be banned. Further, they must be disposed of separately from other solvents. This will be discussed in Section 5, Waste Disposal. The most important distinction between tetrachloroethylene and trichloroethylene is that the boiling point of tetrachloroethylene (121 °C) is higher than that of trichloroethylene (87 °C). Consequently, tetra can be used at higher temperatures without excessive losses due to evaporation. The normal procedure for using these solvents is to place them in a clean beaker, and heat to 80° C (tetra) or 60°C (tri). The wafer to be cleaned is installed in a PFA wafer holder, and immersed in the heated solvent for at least five minutes. In the case of extremely dirty wafers, a second bath of heated solvent is used. At the conclusion of the halogenated solvent cleaning period, the wafer and holder are moved to a clean beaker of acetone. This is necessary because the halogenated solvents are apolar—which is why they solvate organic material—and will not mix with water. Acetone contains a somewhat polar carbonyl moiety, and is therefore soluble in both halogenated solvents and in water (an extremely polar solvent). Acetone is also both extremely volatile (evaporates quickly) and extremely flammable. Consequently, it should not be heated on electric hot plates, and should be kept as far from any ignition source as possible. Graphic testimony to this can be obtained from news accounts of fires in illegal methamphetamine labs. The operators of these labs, who are by no means competent chemists, frequently suffer horrific burns requiring prolonged reconstructive surgery. They are often left permanently disfigured.

The wafer should be allowed to soak in the acetone for five minutes. Even at room temperature, acetone evaporates quickly. If the wafer is left for an extended period, the acetone will evaporate, and any contamination dissolved in the acetone will be deposited on the wafer, and will be extremely difficult to remove.

Methanol (MeOH) is frequently used as an additional step. There is no clear advantage to this. All the cautions given for acetone also apply to methanol, with the additional caution that methanol is rather toxic, and may be absorbed through the skin.

After cleaning with organic solvents the wafer may either be dried, or, if further processing in the acid hood is planned, transferred to a beaker filled with deionized water. If the wafer is to be dried, it is recommended that it first be rinsed over a waste beaker with a stream of isopropyl alcohol (2-propanol, IPA), so that the entire wafer is well wetted. Either acetone, or methanol will evaporate very quickly, leaving a residue that will be nearly impossible to remove; it is necessary to perform this operation quickly. If this cannot be done, it is best to use a final soak of IPA. The wafer is then blown dry with the nitrogen blowgun. High pressure is neither necessary nor desirable. Hold the wafer holder so that the wafer is at an angle of approximately 45° from horizontal, and blow the nitrogen so that the solvent on the surface is swept towards the bottom of the wafer. Hold the wafer over a Cleanroom wipe, so that the solvent will be absorbed. Turn the wafer holder over, and blow the solvent off the backside of the wafer. This will invariably cause some solvent to move to the front of the wafer. Blow this off also.

Using tweezers remove the wafer from the wafer holder and lay it on a clean Cleanroom wipe. Blow any solvent off the surface. The wipe will blot any residual solvent from the backside.

Place the clean wafer in a clean fluoroware container. This should be done with the device side facing down. Fluoroware containers are made with a dished bottom and, with the wafer installed device side down, support the wafer from its edge. This prevents damage to the device side surface. It is good practice to install a plastic “spider” on top of the back side of the wafer before installing the top of the container. Note that the top screws are counterclockwise (left-handed thread). This is so that, when the wafer is removed, the container is held upside down, while the bottom is unscrewed in the normal direction. The wafer is then supported on the spider, device side up, and can be easily removed with tweezers.

Acid Hood Operations: The most common operations performed in the acid hood are etch-cleaning operations. It is common to clean silicon wafers using a procedure known as RCA clean. The original procedure was devised by engineers at RCA . There are presently many variations of the RCA procedure in use. A common formulation is given below:

Organic Clean: Usually, a solution of DI H₂O: NH₄OH: H₂O₂ (5:1:1) at 80° C, for minimum of 10 minutes and a maximum of 20 minutes. Heat water to 90°C; addition of NH₄OH will cause cooling.

Oxide Strip is a solution of DI H₂O: HF (50:1) or (100:1) for from 10 to 15 seconds. Note that HF is never to be used, or disposed of, in glass containers. Use plastic containers and plastic graduated cylinders for measuring.

Important information about HF:

Concentrated HF is considered to be extremely toxic—a “4” on the health hazard scale of 0-4. This is because it is a source of free fluorine ions. Any solution containing a source of free fluorine ions is also hazardous. A concentrated ammonium fluoride solution is “very” toxic (3 on the health hazard scale) and becomes “extremely” toxic when made more acidic, such as in Buffered Oxide Etch (BOE) mixtures. Consequently, BOE presents the same level of toxic hazard to your body as 49% HF even though 20:1 BOE has much less HF in it (about 7% of volume) than “pure” HF (about 49% of volume). It is equally hazardous because it also has about 38% NH₄F, and is acidic.

Fluorine ions (from the HF) easily pass through skin and tissue. Because HF's action can be delayed for many hours, it can distribute throughout your body. The fluorine ions bind very easily and tightly to calcium and magnesium ions to form insoluble salts. In the body, Ca and Mg ions mediate a variety of physiological processes, such as muscle movement. Calcium is also a chief component in bone. The result can be several severe forms of damage to your body, including death.

- Local tissue damage results from free hydrogen ions, which cause corrosive chemical burns and free fluorine ions which cause deep tissue damage including bone loss.
- Systemic conditions include hypocalcemia (loss of calcium) and hyperkalemia (too much potassium). Since calcium and potassium regulate the beating of your heart, an irregular heartbeat and cardiac arrest can result. “Deaths have been reported from concentrated acid burns to as little as 2.5% of body surface exposed to skin contact.” That is the equivalent of a single hand.
- Even dilute HF must be treated with extreme caution. It is highly dangerous to the body because it acts without causing pain. Dilute HF may not cause a visible or painful skin burn (or tissue burn), encouraging victims to ignore exposure. Even so, the fluorine ions can pass through the skin and disturb the body’s Ca and K balance, as well attack the bones. Pain may only occur several hours after the exposure.

Calcium gluconate gel or cream (sometimes called “HF antidote”) is available near the acid hood in a single-use squeeze bottle. Calcium gluconate is discussed more extensively in the “Safety Equipment” section of this document.

Note also that Hydrogen Fluoride gas is also extremely toxic. It very easily dissolves into water to become Hydrofluoric Acid. HF easily moves between the gas phase and liquid phase; consequently, solutions containing HF or NH_4F may generate fumes containing HF. Breathing these fumes makes HF available to the blood and results in the rapid transport of HF throughout the body. Thus, it is imperative to avoid breathing vapors coming from HF containers. These containers may only be opened in a vented acid hood. It is strongly recommended that complete PPE be worn at all times while handling HF.

Ionic Clean solution of DI H_2O : HCl: H_2O_2 (6:1:1) At 80°C , minimum of 10 minutes and a maximum of 20 minutes.

- Note that HF is particularly dangerous
- Hydrofluoric acid (49% 0.2micron filtered SEMI grade) protocol
- Hydrochloric acid (37% 0.2 micron filtered SEMI grade)
- Ammonium Hydroxide (29% 0.2 micron filtered SEMI grade)
- Hydrogen Peroxide (30% 0.2 micron filtered SEMI grade))

General Solution Preparation Procedure:

Determine how much solution will be required to cover your work piece.

Calculate the volumes of each reagent required to make that solution. For example, a single 50 mm wafer in a PFA wafer holder will fit in a 250 ml beaker.

Roughly 200 ml will be required to completely cover the wafer. To prepare the organic clean solution listed above, first add the number of “parts” of the solution. In this case, it would be $5 + 1 + 1 = 7$ (5:1:1). Two hundred ml can be divided into sevenths, yielding 142.9 ml :28.6 ml :28.6 ml. These are somewhat inconvenient numbers. Instead, prepare 210 ml of solution; this requires 150 ml : 30 ml: 30 ml.

First, obtain two clean beakers of size appropriate to the work piece as well as a graduate cylinder appropriate to the volumes to be measured. Rinse them thoroughly with DI water and drain them. A clean “waste” beaker will also be required. Additionally, a clean Pasteur pipet and bulb will be helpful.

Place the required amount of water in the graduated cylinder. Liquids, depending on their surface tension, will climb the wall of the graduate cylinder. This forms a “meniscus.” Measure the volume of the liquid from the bottom of the meniscus. In order to do this accurately, you must squat down so that you are looking straight at the line on the graduate cylinder. Make it a habit not to hold the graduate cylinder when measuring, and especially not to remove it from the hood. If you have spilled hazardous chemicals on the outside of the cylinder your hand will be contaminated. When you have the required amount of water in the graduate cylinder, pour it into the beaker that you will use for your etch.

Water may be measured directly from the DI water tap, and excess disposed of in the drain. Chemicals, however, should be poured into a clean beaker and then decanted into the graduate cylinder. If a small excess is added, it can be removed with a Pasteur pipet and disposed of in the waste chemical beaker. When decanting chemicals into the beaker, pour slightly more than you will need. Dispose of the excess in the waste chemical beaker.

Add chemicals to the solution slowly. Stir frequently with a glass stirring rod. Several chemicals, such as sulfuric acid, liberate significant heat when they are diluted. In some cases it may be advisable to immerse the beaker in a water bath.

As soon as you are through adding a chemical, rinse the glassware used to make the addition and put the chemical bottle back in the appropriate cabinet. Do not allow bottles to accumulate.

After you have prepared your solution(s) and allowed them to reach the correct temperature, perform your etch. You should time the etch using a dedicated timer, rather than your watch or cell phone. At the conclusion of the etch time, the wafer holder should be removed from the beaker. Gently tap the holder on the side of the beaker to dislodge any excess etchant, and then immerse the wafer holder in a beaker of DI water. The beaker holding the wafer holder and DI water should be placed under running DI water. Leave the beaker under running water long enough for the beaker to be filled at least five times. For larger beakers, this may be a significant amount of time. Determine this time before beginning the etch.

5. Waste Disposal: University regulations prohibit pouring any substance down drains. All solvent, acid and alkali waste must be put in properly labeled containers for disposal by Environmental Health and Safety (EHS). Each waste container must have attached a

manila “Hazardous Waste Chemical Inventory Tag.” Before pouring any chemical in the waste bottle, its contents must be entered in the proper waste sheet in the Waste Chemical Folder. This folder is attached to the left hand side of the acid hood in Bay 1.

Solvent disposal: Waste solvents are to be disposed of in the solvent waste bottles, which are stored in the cabinet beneath the solvent hood. These are labeled “Non Halogenated Solvent Waste” and “Halogenated Solvent Waste.” All acetone, methyl alcohol, isopropyl alcohol, ethyl alcohol, hexane, and cyclohexane are to be disposed of in the “Non Halogenated Solvent Waste” bottle. Tetrachloroethylene and trichloroethylene are disposed of in the Halogenated Solvent Waste bottle. If you are unsure which bottle to use for any solvent, please consult a member of the Cleanroom staff before proceeding. Please do not create solvent waste bottles for “acetone,” “methyl alcohol,” etc. There is no need to segregate the various non-halogenated waste solvents, and there is no room to store sufficient waste bottles.

Place the appropriate solvent waste bottle in the hood, remove the cap and insert the funnel that is stored in the hood. Pour the solvent into the bottle, making sure that your face is away from the bottle. Rinse the glassware with acetone from an acetone squeeze bottle several times. This is permissible, even in the “Halogenated Waste Bottle.” Remove the funnel and put the cap on the bottle. Tighten the cap until it is snug, and return it to the bottom cabinet. If the solvent fills the bottle to, or above, the shoulder of the bottle (where the glass narrows from the full diameter of the bottle), then the bottle is full and must be replaced. Take the bottle to the clean storage area, and place it in the Solvent Cabinet. Obtain a new plastic bottle from the bottle rack. Attach one of the manila “Hazardous Waste Chemical Inventory” tags, and write the intended contents on label under “Substance.” The contents must be written in words, not as a chemical formula (e.g., water, not H₂O).

Return to Bay 1 with the new bottle. Obtain an indelible marker from the metal cabinet and use it to cross out the original contents from the bottle label. Write on the bottle, in large print “Halogenated Solvent Waste” or “Non-Halogenated Solvent Waste,” as appropriate. Replace the bottle in the cabinet under the solvent hood.

Do not leave waste solvents in the plastic container that holds the waste funnel. This allows the solvents to evaporate and be discharged with the exhaust air. All of the solvents used in the Cleanroom are considered to be Volatile Organic Compounds (VOCs), and contribute to the formation of photochemical smog. It is a matter of civic responsibility to minimize discharges of environmentally harmful chemicals as much as possible.

If the glassware is well rinsed with acetone, and no obvious contamination is found, allow it to air-dry. If, after drying, inspection shows contamination it will be necessary to clean the glassware with soap and water in the acid hood. Use a very small amount of Alconox and clean using the city water tap. You may need to scrub using a brush or a Cleanroom wipe. When you are satisfied that you have removed the contamination, rinse very thoroughly with city water. When you think you have rinsed it sufficiently, rinse it some more. Soap is very hard to remove completely. Finally, rinse at least twice with deionized water, and allow the glassware to dry.

Acid Waste: Acid waste must be segregated. The necessary categories are: sulfuric acid containing waste, hydrochloric acid containing waste, nitric acid containing waste and hydrofluoric acid containing waste. If sulfuric acid containing waste is mixed with hydrochloric acid containing waste, HCl gas will be generated. This is a toxic and corrosive vapor that will cause eye, nose and throat irritation. Both sulfuric acid and hydrochloric acid are frequently combined with hydrogen peroxide in semiconductor etching processes. Hydrogen peroxide decomposes to water and molecular oxygen (O₂). In a closed container, this results in an increase in pressure that may rupture the container. In order to this problem, when disposing of either sulfuric acid or hydrochloric acid, screw the cap onto the waste bottle completely, then back it off by ¼ turn. This will allow the generated gas to escape, while preventing the cap from coming off if the bottle is inadvertently lifted by the cap.

Sulfuric acid also has a very high “heat of mixing.” This means that when concentrated sulfuric acid is diluted the mixture will become very hot. When it becomes hot it will expand. For this reason, sulfuric acid waste bottles, in particular, must never be filled to the top, and the cap must be left slightly loose, as described above.

When transporting acid waste bottles, use the acid bottle carrier stored between the solvent hood and the solvent cabinet. This will prevent acid from spilling even if the bottle is dropped. When full bottles of acid are brought from chemical storage to the chemical bay they are to be transported in either the acid bottle carrier, or in the acid transport cart, which is stored in the “clean storage” area. Carrying full bottles of acid or acid waste across the Cleanroom has resulted in accidents that required extensive cleanup and caused significant delay to research efforts.

Base Waste: Most of the alkali waste in Nanofab is either used photoresist developer, or ammonium hydroxide-hydrogen peroxide etchants. Other waste solutions will be relatively strong potassium hydroxide or sodium hydroxide solutions used for etching silicon. All are to be stored in the cabinet below the develop hood. Photoresist developers are to be stored in containers marked “developer waste.” Ammonium hydroxide-hydrogen peroxide waste must be stored with the bottle cap slightly loosened, as described for acid-hydrogen peroxide wastes, above. Concentrated hydroxide solutions must be segregated in their own storage bottles (potassium and sodium hydroxide solutions may be mixed, however). Dilution of concentrated hydroxide solutions will result in significant evolution of heat. They must be handled in the same manner as sulfuric acid solutions, as described above.

Table 1. Common Cleanroom Chemicals

Chemical Name	Concentration	Flash Point	Boiling Point	SAFE-T-DATA				Disposal Class	CAS	Incompatibilities
				H	F	R	C			
Acetic acid	99.5% (Glacial)	40	118	3	2	2	4	HOAC Waste Non-Halogenated Solvents	64-19-7	HNO ₃ , HCrO ₄ , HClO ₄ , metals Freezes below 17 C,
Acetone	100%	-20	56.5	1	1	0			67-64-1	Oxidizers, acids Strong oxidizers, acids, halogens, salts of silver & zinc
Ammonium hydroxide	29%	N/A	N/A	3	1	2	3	NH ₄ -OH Waste	7664-41-7	Hydroxides, amines, alkalis, copper, brass, zinc
HCl, Concentrated	39% (12.4 M)	N/A	53	3	0	2	4	HCl/H ₂ O ₂ Waste	7664-93-9	As ₂ O ₃ , P ₂ O ₅ , NH ₃ , CaO, EDTA, H ₂ SO ₄ , <u>Glass</u>
Hydrofluoric Acid	48-52%	N/A	108	4	0	2	4	HF Waste	7664-39-3	Organic material, chlorates, perchlorates, permanganate
Hydrogen Peroxide	30%	N/A	108	3	0	3	4	H ₂ SO ₄ /H ₂ O ₂ Waste Non-Halogenated Solvents	7722-84-1	Strong oxidizers, acetaldehyde, chlorine, ethylene oxide, acids, isocyanates
Isopropyl Alcohol	100%	12	82	1	1	0			67-63-0	
Methyl Alcohol	100%	12	64.5	3	3	1	3	Non-Halogenated Solvents	67-56-1	Strong oxidizers phenol, strong bases, powdered metals organics
Nitric acid	50-70%	N/A	122	4	0	3	4	HNO ₃ Waste Halogenated Solvents	7697-37-2	Strong caustics and bases, reactive metals
Perchloroethylene	100%	N/A	121	3	0	1	2		127-18-4	
Potassium hydroxide	100% (pellets)	N/A	360	3	0	2	4	NaOH Waste	1310-58-3	Acids, water, metals (when wet), halogenated hydrocarbons, maleic anhydride
Sodium hydroxide	100% (pellets)	N/A	1390	4	0	2	4	NaOH Waste	1310-73-2	Water; acids; flammable liquids; organic halogens; metals such as aluminum, tin & zinc; nitromethane
Sulfuric Acid	98% (19 M)	N/A	290	4	0	2	4	H ₂ SO ₄ /H ₂ O ₂ Waste Halogenated Solvents	7664-93-9	Organic material Strong caustics and bases, reactive metals
Trichloroethylene	100%	N/A	87	3	1	1	2		76-01-6	

6. Chemical Spills: **Notify NanoFAB Staff immediately of the spill.** Clean up of chemical spills (outside hoods) shall be performed by NanoFAB Staff, or under NanoFAB staff supervision during normal working hours. Personnel working outside of normal working hours are expected to have sufficient experience to perform spill clean-up safely without supervision. The Spill Kits are located by the base cabinet in Bay 1, and in the Teaching FAB Yellow room. Note that there are plastic jars of neutralizer granules for acids, bases, and absorber granules for solvent spills. The general application method is to pour a ring around the spill to initially contain it and then fill in the ring to complete the absorption and containment. After the granular material completely absorbs the spilled material, it is swept up using the plastic “dust pan” and spatula provided with the spill kit. Place the collected material in the heavy-duty zip-lock bags provided with the spill kit. Write the contents (e.g., Sulfuric acid, hydrogen peroxide, etc.) on the bag label.

7. Accidents: Chemical Showers and Eye Wash Stations

There are five shower / eyewash stations inside the NanoFAB clean room. They are in Bay 1 (by the door), Alcove, TFAB, TFAB yellow room, and MBE.

Chemical Spills on your clothes / body:

Dangerous chemicals on your clothes / body require the immediate use of the shower. Speed is essential. Immediately begin flushing with plenty of water and continue to flush for at least 15 minutes. While showering, remove your affected clothing so as to remove the source of chemical danger from yourself. Concerns about modesty must not interfere with avoiding serious injury. Do not put contaminated clothing back on after rinsing. Summon appropriate medical assistance as soon as possible and medical care.

Chemical Splashes into your eyes:

Use the eyewash stations if chemicals have splashed into your eyes. Call for help so that your buddy can lead you to the eyewash station. Speed is essential. Do not hesitate. Let the water flow over your closed eyes for a few moments before beginning to open them. This will make sure that the water is as clean as possible. Wash out your eyes with water for at least 15 minutes after opening them. If you have splashed chemicals both in your eyes and on your body, then you may want to use the shower first and rinse your eyes in the shower. After showering, rinse your eyes using the eyewash station as well. Remove any affected clothing while rinsing your eyes at the eyewash station, so as to remove the source of chemical danger. Concerns about modesty must not interfere with avoiding serious injury. Summon appropriate medical assistance as soon as possible and obtain medical care.

8. Compressed Gases: Compressed gas cylinders can pose significant safety hazards. A broken valve would allow high-pressure gas to launch the cylinder with devastating force. Improperly handled cylinders containing toxic or pyrophoric gases can injure or kill personnel. Even inert gases can displace sufficient air to cause asphyxiation. **Only personnel authorized by the NanoFAB Management are allowed to change, replace, or otherwise handle compressed gas bottles.** All full sized compressed gas bottles are

to be stored within the service chases. Bottles may only be moved with the protective cap installed over the valve. A cylinder cart is stored in the Gas Room; this must be used whenever cylinders are moved. Cylinders must be properly anchored. Some equipment may require the use of small gas bottles within the machinery – these will be handled on a case-by-case manner. Toxic gas bottles may only be stored and used inside designated toxic gas cabinets appropriate for them. Toxic gas monitors will be employed where necessary for safety. Only authorized personnel may handle these systems. **There are no exceptions.**

Appendix:

List of Commonly Needed Items for Cleanroom Project

<http://www.wafercare.com/>

Item	Product# (look the website for complete product number for your wafer size)	Material
Tweezers*	C20	PFA
Dippers	D11(2" wafers) D14(2" to 5" wafers)	PFA
Dipper Basket	D10	ETFE
Beakers	D50	PFA
Single Wafer Shippers* (For storage and transport of single wafers or pieces. Trays and lids are sold seperately)	H22	Polypropylene or STAT-PRO® 150
Storage Boxes	E04	Black high impact polystyrene
Wafer Carriers (these fit in storage boxes, for storage and transport of up to 25 wafers)	PA72	Blue polypropylene or PFA

<http://www.vwrsp.com/>

Item	Product#	Material
Wafer tweezer for 3" wafer**	63040-518	Stainless steel
Wafer tweezer for 2" wafer**	63040-512	Stainless steel
Tweezers, Flat, Round Point, Uni-Fit**	63040-458	Stainless steel

Tweezers, Long, Narrow, Fine Point, Uni-Fit**	63040-430	Stainless steel
Polyethylene bottles for chemicals, ½ liter* (Polypropylene recommended)	16120-724	HD Polyethylene

For photomasks

<http://www.microtronicsinc.com/>

For buying wafers

<http://www.novawafers.com/>

OR

<http://www.virginiasemi.com/>

OR

<http://www.universitywafer.com/>

Item	Product #	Dimensions	For Equipment	Vendor
Tungston Boat	EVS2B010W	½, 3 ½	NRC Evaporator	Kurt J. Lesker (www.lesker.com)
Alumina-coated W Boat	EVS2BAOW	½, 3 ½	NRC Evaporator	Kurt J. Lesker (www.lesker.com)
Crucible (fits hearth)	EVCFABEB-26	21.9cc	CHA Evaporator	Kurt J. Lesker (www.lesker.com)
Crucible (shallow)	EVCFABEB-1	0.24cc	CHA Evaporator	Kurt J. Lesker (www.lesker.com)
Crucible (base for shallow)	EVCFABEB-20	0.84cc	CHA Evaporator	Kurt J. Lesker (www.lesker.com)
Crucible (sits in 26)	EVCFABEB-2	4.8cc	CHA Evaporator	Kurt J. Lesker (www.lesker.com)
Sputter Targets		3" dia., ¼ " thick	HB Sputter	Kurt J. Lesker (www.lesker.com)
Sputter Targets		4" dia., ¼ " thick	PQ Sputter	Kurt J. Lesker (www.lesker.com)
Boats	992623-01	Tungston	AJA Thermal Evaporator	http://www.mdcvacuum.com/

Boats	992623-02	Molybdenum	AJA Thermal Evaporator	http://www.mdcvacuum.com/
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The above websites are given for guidance only. If you find other good sources of same or similar products, please let us know and we will gladly include those here.

Additional Items:

Clean room notebook (available at UTA Bookstore)*

Glassware*

Plastic beakers polypropylene (Teflon recommended)*

*Required items

**Required at least one of these

Chemicals and Other Materials Provided by NanoFAB

	Chemical name	Grade	Product Number
Solvents	Acetone	CMOS	TXJT 9005-05
	Methanol	CMOS	TXJT 9073-05
	2-propanol	CMOS	TXJT 9079-05
	Trichloroethylene	CMOS	TXJT 9454-03
Acids	Aluminum Etchant 80-15-3-2	CMOS	TXJT 5459-05
	Buffered Oxide Etch 6:1	CMOS	TXJT 1178-03
	HF Dip 10:1	CMOS	TXJT 5397-03
	HF Dip 50:1	CMOS	TXJT 5477-3
	Phosphoric Acid 85%	CMOS	TXJT 0264-05
	Sulfuric Acid	CMOS	TXJT 9684-05
	Nitric Acid	CMOS	TXJT 9606-03
	Hydrofluoric Acid 49%	CMOS	TXJT 9564-06
	Acetic Acid	CMOS	TXJT 9503-05
Hydrochloric Acid	CMOS	TXJT 9539-05	
Bases	Hydrogen Peroxide	CMOS	TXJT 2190-03
	Ammonium Hydroxide	CMOS	TXJT 9731-03

OTHERS

Aluminum foils

Cleanroom wipes

Gowning supplies (Golves, hairnets, coveralls, and shoe covers)

PPE (Chemical gloves, aprons, sleeves, and face shields)

(Safety goggles and respirators are **not provided**)