1.0 CONTACT INFORMATION

1.1 Tool Owners:
1.1.1 Olivia McPherson
1.1.2 E-mail: mcperson@cns.fas.harvard.edu
1.1.3 Telephone: (617-49)6-7102
1.1.4 Hao-Yu Greg Lin
1.1.5 E-mail: hlin@cns.fas.harvard.edu
1.1.6 Telephone: (617-38)4-5028
1.1.7 T. Fettah Kosar
1.1.8 E-mail: fkosar@cns.fas.harvard.edu
1.1.9 Telephone: (617-49)5-1738

1.2 Center for Nanoscale Systems – Material Synthesis and Characterization Facility
1.2.1 E-mail: Materials@cns.fas.harvard.edu
1.2.2 Telephone: (617-38)4-7411

1.3 Emergency:
1.3.1 Telephone: 911

1.4 Harvard University Operations Center
1.4.1 Telephone: (617-49)5-5560

1.5 Harvard Police
1.5.1 Telephone: (617-49)5-1212

2.0 PURPOSE & SCOPE

2.1 This document describes safety features of the two ReynoldsTech wet benches and is required reading for anyone using the wet benches in the Chemical Nanotechnology Facility (Soft Lithography Foundry and Nanoparticle Facility) located in LISE G06.

3.0 APPLICABLE DOCUMENTS

3.1 CNS Safety Manual (required reading)
3.2 CNS Form FM106 “Chemical Nanotechnology G06 Room Wetbench Training Checklist”
3.3 MSDS Sheets: http://cns.fas.harvard.edu/safety/msds.php
3.4 CNS FM006 “Material Request Form”

4.0 DEFINITIONS

4.1 Personal Protective Equipment = PPE
4.2 Nitrogen = N2
4.3 Material Safety Data Sheet = MSDS
4.4 Deionized Water = DI = DI Water
4.5 Minute = min
4.6 Second = s = sec
4.7 Rinse = a process of either immersing, spraying and cascading wafers in DI water for no less than one minute.
4.8 Nitrogen Dry = a process of spraying wafers with nitrogen until water and/or IPA is no longer present on either wafer surface.
4.9 Chemical Handling: Means pouring, placing or retrieving components from beakers or tanks, rinsing acid bottles, moving beakers with liquid in them inside wet benches etc.

5.0 WORK INSTRUCTIONS

5.1 EQUIPMENT, MATERIALS AND SUPPLIES:

5.1.1 PPE
   5.1.1.1 Safety Goggles
   5.1.1.2 Face Shield
   5.1.1.3 VR Polyolefin Apron
   5.1.1.4 Vented wet bench
   5.1.1.5 Nitrile Gloves
   5.1.1.6 Yellow MAPA Roll Pruf gloves for all chemical use in wet benches
   5.1.1.7 Safety glasses

5.1.2 “Over-the-glasses” safety glasses placed over prescription glasses

5.1.3 Wafer or substrate

5.1.4 Beakers

5.1.5 Teflon-tipped tweezers for acid processing

5.1.6 Stainless steel tweezers for solvent processing

5.1.7 Petri dish

5.1.8 Cleanroom Wipes

5.1.9 Appropriate chemicals (must be authorized by CNS and MSDS supplied)

5.2 SAFETY INSTRUCTIONS:

5.2.1 Certification
   5.2.1.1 Users must have taken the “LISE G06 Room Training” and submitted the completed CNS Form FM106 “Chemical Nanotechnology G06 Room Wetbench Training Checklist” after taking this wet bench safety training.

5.2.1.2 Users must be trained AND certified in the use of the wet benches before proceeding.

5.2.2 Personal Protective Equipment (PPE)
   5.2.2.1 Minimum PPE when handling any amount of acids and bases in wet benches and when pouring solvents in quantities greater than one liter is as follows:
      5.2.2.1.1 Face shield
      5.2.2.1.2 Blue Apron
      5.2.2.1.3 Yellow Chemical gloves over the nitrile gloves
      5.2.2.1.4 Goggles or over-sized Safety Glasses under face shield
      5.2.2.1.5 Over-the-glasses safety glasses if wearing prescription glasses.
5.2.2.2 How to Don PPE Equipment:
   5.2.2.2.1 Inspect Apron
   5.2.2.2.2 Use 10% IPA in water on a wipe and clean face shield before donning
   5.2.2.2.3 Adjust if necessary
   5.2.2.2.4 Put on yellow chemical gloves over apron sleeves

5.2.2.3 How to Remove Personnel Protective Equipment
   5.2.2.3.1 Wash gloves with DI water
   5.2.2.3.2 Dry gloves using clean room wipes
   5.2.2.3.3 Remove outer layer chemical glove and discard.
   5.2.2.3.4 Remove face shield
   5.2.2.3.5 Place face shield on bin
   5.2.2.3.6 Hang apron

5.2.3 Safety equipment for all benches
   5.2.3.1 Emergency shut down of all benches is accomplished by striking the EMO
   (emergency off) button on the upper right corner of each wet bench
   5.2.3.2 In the event of fire, pull the local bench fire alarm, located at the bottom right of
   the stainless steel benches or on wall.
   5.2.3.3 If able, lower the bench shield to its lowest point and still be able to work in the
   bench comfortably. Lower shield to lowest point when done using…this will
   help conserve energy.

5.2.4 Steel bench safety equipment
   5.2.4.1 UV/IR/thermal fire sensors are located in each bench of stainless steel
   construction and high pressure carbon dioxide will discharge in the event of fire.
5.2.5 Spill Procedure

5.2.5.1 Do not expose yourself or attempt to cleanup a spill that is a serious health hazard, or one that you are uncomfortable handling. Instead immediately seek assistance.

5.2.5.2 Spills inside a wet bench

5.2.5.2.1 If you cause or discover spilled hydrofluoric acid seek assistance. Contact CNS staff during normal business hours (M-F 8am to 5pm) or contact the Operations Center during off-hours.

5.2.5.2.1.1 Warn other users of the presence of a spill using an A-frame “Chemical Spill Keep Away” floor sign or a handmade sign.

5.2.5.2.2 Any other chemical spill (excluding HF) in the wet benches can be picked up by CNS users if they feel comfortable. If not, contact CNS staff for assistance.

5.2.5.2.2.1 Spill Kits can be found on the PPE rack between the wet benches (labeled “spill kit”), and there are absorbent spill pads on the shelves.

5.2.5.2.2.2 If an acid or base solution spill occurs in any of the white plastic wet benches you can use the water hose to wash the spill into the sink.

5.2.5.2.3 Spills outside of any wet bench

5.2.5.2.3.1 For any chemical spill outside the wet bench CNS users must seek assistance before attempting clean up. Seek assistance from CNS staff during normal business hours (M-F 8am to 5pm). During off-hours seek assistance from the Operations Center at 617-(49)5-5560.

5.2.5.2.3.2 Warn other users of the presence of a spill using an A-frame “Chemical Spill Keep Away” floor sign or a handmade sign.

5.2.5.2.3.3 Leave clean room if vapors are irritating.

5.2.6 Injuries

5.2.6.1 Minor injuries such as burns and cuts can be treated with the first aid kit located in the Gowning Area. This kit can be removed from the wall if necessary.

5.2.6.2 Injuries requiring treatment by a health care professional must be documented.

5.2.6.2.1 This includes filling out an Occupational Accident, Injury or Illness Investigation Report.

5.2.6.2.1.1 These forms are available in the CNS main office (LISE 306) and should be completed within 24 hours of medical treatment or on the Monday following a weekend.

5.2.6.2.2 Additionally a CNS Incident Report form must be completed and submitted to the CNS Administrative office.

5.2.6.3 LISE G06 users familiar with first aid may come to the aid of colleagues.
5.3 MAINTENANCE AND CALIBRATION CRITERIA

5.3.1 Ensure that all wet benches have an adequate air draw and that certification stickers are current.
5.3.2 Ensure that no alarms are active, such as alarms for low air flow, full waste container, or waste container overflow.

5.4 CHEMICAL SUPPLIES

5.4.1 LISE G06 users employ chemicals stocked by CNS and approved chemical provided by users themselves.
5.4.2 Chemicals stocked in the LISE G06 are for use in the LISE G06 only and not for use in other departments or research facilities.
5.4.3 Removing CNS-provided chemicals from CNS spaces is considered a significant safety violation with disciplinary consequences including loss of all CNS access privileges.
5.4.4 Familiarize yourself with any chemicals you plan to use by reading the Material Safety Data Sheets.
   5.4.4.1 Several references are available on the CNS web site.
   5.4.4.2 http://cns.fas.harvard.edu/safety/msds.php
   5.4.4.3 Consult Harvard University EH&S or the chemical manufacturer regarding toxicology of your prospective chemicals.
5.4.5 PRIOR TO USE, ALL MATERIALS BROUGHT INTO CNS LABS MUST BE APPROVED with the following form:
   5.4.5.1 CNS FM006 “Material Request Form”
   5.4.5.2 http://cns.fas.harvard.edu/users/Forms/Chemical_Request_form.pdf
   5.4.5.3 This includes, but is not limited to: new solvents, acids, bases, proprietary solutions, adhesives, nanoparticles and polymer mixes.
5.4.6 CNS has restrictions on the use of some chemicals such as elemental mercury and mercury compounds. Not all materials requests will be approved.
5.4.7 Label all chemical containers with chemical name, user name, date and contact information. An unlabeled chemical container is a safety violation and is the basis for disciplinary consequences including loss of laboratory access privileges.
5.4.8 LISE G06 staff will actively seek and dispose of any chemical containers without an approved label or any other unlabeled chemicals in the LISE G06.

5.5 TRANSPORTING CHEMICALS

5.5.1 Chemicals must be transported through the lab with special protection. Never carry chemical bottles without the proper carrier.
5.5.2 Individual glass bottles must be placed in polyethylene safety carriers during all transport such as from the Gowning Area to point-of-use.
5.5.3 If a bottle is not in the bay where you need to work, find the chemical in another bay and transport it from one bay to another in the designated carrier.
5.6 CHEMICAL EXPOSURE

5.6.1 If exposed to chemicals on your body find nearest emergency shower and remove all clothing and rinse body for at least 15 minutes.

5.6.2 Flush the affected areas with water for 15 minutes (see section 5.7 for Hydrofluoric Acid exposure).

5.6.3 Safety showers and eyewashes are located in G05 and G06.

5.6.4 Exposure of the eyes requires flushing with water for at least 15 minutes.
  5.6.4.1 If eyes are exposed to a chemical find the nearest emergency eyewash station.
  5.6.4.2 Hold eyes open and flush continuously for 15 minutes.
  5.6.4.3 After flushing the exposed area with water seek medical attention.

5.6.5 Contact a staff member as soon as possible for assistance or call 911.

5.6.6 If exposure occurs in the evening or on a weekend, contact 911 or the Operations Center 5-5560.

5.6.7 All injuries occurring in the LISE G06 must be reported to the Manager of the Materials Group within 24 hours or on the Monday following a weekend.

5.7 HYDROFLUORIC ACID (HF) HAZARDS

5.7.1 Hydrofluoric acid (HF) burns are extremely serious and may result in death due to tissue damage occurring as fluoride ions diffuse through the body.

5.7.2 Inhalation of hydrofluoric acid vapors may be lethal and shall be avoided by only working with HF inside of designated vented wet benches.

5.7.3 Skin contact with HF may not be painful at first with life threatening deep burns developing hours after exposure.

5.7.4 If skin is contacted with HF, immediately flush the area well for five minutes. This may include under and around fingernails, as under fingernails and cuticles are common places where people receive burns.

5.7.5 Remove all contaminated clothing while rinsing.

5.7.6 After rinsing the area contaminated by HF, gently massage calcium gluconate ointment into skin using a gloved hand. Calcium gluconate is located on the wet bench where HF is used and in the First Aid kit in the Gowning Area.

5.7.7 If HF has contacted the eyes, use an eye wash station for at least 15 minutes with eyes wide open. Do not apply calcium gluconate gel to eyes.

5.7.8 While washing the affected area, have someone call 911 for emergency medical assistance. All HF exposures need to be evaluated by a physician.

5.7.9 Report any HF burns to the Manager of the Materials Group and EH&S. with the Incident Report form.

5.8 CHEMICAL STORAGE

5.8.1 Chemicals must be stored in appropriate chemical storage cabinets.

5.8.2 Chemical bottles cannot be left on the floor areas of the LISE G06.

5.8.3 Screw-top containers are the only acceptable means of liquid storage.
  5.8.3.1 These containers are safe and protect against contamination.
  5.8.3.2 Plastic wrap or aluminum foil covers are not acceptable. These containers will be disposed of immediately as they pose an unsafe condition.

5.8.4 All chemical containers must be labeled with the lab member’s name, contact information, date, and detailed contents of the container.
5.8.5 Each wet bench contains a label indicating the contents of stocked chemicals.
5.8.6 Photoresists and developers are located under spinner wet benches
5.8.7 If a chemical is out of stock, contact LISE G06 staff.

5.9 CHEMICAL USE IN BENCHES

5.9.1 Always work with chemicals in a ventilated wet bench.
5.9.2 Always place fuming containers toward the back of the wet benches where there is maximum exhaust.
5.9.3 Do not leave chemical processes unattended unless absolutely necessary.
5.9.4 All chemicals in use must be clearly labeled with chemical name, user name, date, time, expected time of return, and where able to be reached.

5.10 CHEMICAL POURING

5.10.1 Create a label using a wipe for the chemicals planned for use before you pour any chemical.
5.10.2 Ensure the glass or plastic containers intended for use are clean and dry.
5.10.3 Ensure proper compatibility among containers and chemicals.
   5.10.3.1 Only Teflon and polypropylene should be used with HF.
   5.10.3.2 HF will attack some plastics, especially polyethylene and will certainly etch glass.
   5.10.3.3 Rule of thumb: match the chemical bottle type with the type of container planned for use.
5.10.4 Minimize the number of chemical bottles on the bench surface at one time.
5.10.5 Hold chemical bottle with two hands when pouring.
5.10.6 If measurements are required, pour the chemical into a small beaker and then pour from the small beaker into the graduated cylinder to measure out the quantity required.
   5.10.6.1 If excess chemical remains after measurement, dispose of it properly.
   5.10.6.2 Never pour any chemicals back into the container from which they came.
5.10.7 When mixing acids with water, ALWAYS ADD ACID (AAA) to water, and never add water to acid.
   5.10.7.1 An exception to this rule is the wafer cleaning solution called Piranha
   5.10.7.2 Read the Piranha Fact Sheet before mixing any piranha solution.
   5.10.7.3 This solution is a mixture of sulfuric acid and hydrogen peroxide.
   5.10.7.4 When making Piranha solution adds H₂O₂ to H₂SO₄.
5.10.8 When mixing several acids with water, add the weakest one first.
5.10.9 Once chemicals are poured, use a wipe and wipe off the lip and side of the bottle to ensure that no chemical is left exposed on the bottle.
   5.10.9.1 Rinse and throw the used acid/base wipe into the trash.
   5.10.9.2 Place used solvent/resist wipe in waste collection bin.
5.10.10 Rinse and dry each beaker and graduated cylinder before pouring the next chemical.
5.10.11 Only a MINIMAL amount of chemical needs to be used. Do not pour more chemical than needed, especially developer and solvents.
5.10.12 Rinse and dry PPE gloves between pouring different chemicals.

5.11 As a common courtesy, inform others working at the wet bench which chemicals you will be pouring or using.

5.12 NEVER POUR ANY ACIDS INTO THE CUP SINKS IN THE STAINLESS STEEL WET BENCHES. THIS COULD CREATE AN EXOTHERMIC REACTION.
6.0

6.1 USE OF LABWARE IN BENCHES

6.1.1 Ensure open containers (beakers, chemical supplies, graduated cylinders, etc.) are at least 6-inches from the front of the bench.

6.1.2 Wet bench users shall utilize beakers rather than tanks if the following criteria are met:
   6.1.2.1 A process has potential to react with or contaminate a tank.
   6.1.2.2 A sample is not a whole substrate/wafer and there are no appropriate holders available for use.
   6.1.2.3 If a metal lift off process requires the use of a lab swab.
   6.1.2.4 If a process will make the chemical unusable.
   6.1.2.5 If the chemical does not have a tank assigned.

6.2 USE OF BENCH TANKS

6.2.1 Wet bench users shall utilize process dunk tanks if the following criteria are met:
   6.2.1.1 A commercially available holder exists for handling wafers/substrates.
   6.2.1.2 More than one whole wafer/substrate is to be processed.
   6.2.1.3 The chemical to be used is available in a tank and it does not follow the characteristics for beaker processing.
   6.2.1.4 Never dump a tank when heated. Make sure solution is cooled to room temperature before draining.

6.3 HYDROFLUORIC ACID (HF) WASTE COLLECTION

6.3.1 HF must be disposed of in waste bottles located at dedicated acid wet benches.
   6.3.1.1 Ensure waste bottle has proper labeling.
   6.3.1.2 Use a funnel to pour HF into its waste bottle.
   6.3.1.3 When a waste bottle is 75% full, it is no longer usable and a fresh waste container must be employed.

6.3.2 Rinse the HF waste bottle with a sprayer if any waste drips on the side of the waste container then dry when rinsed. Test with pH strips if necessary.

6.4 USE OF WATER SINK

6.4.1 The primary function of sinks is rinsing/draining wafers, containers, and gloves.

6.4.2 Sink drain leads to LISE waste neutralization tank in basement, and ultimately to the environment.

6.4.3 Chemical disposal in a sink is not permissible for:
   6.4.3.1 Photolithographic waste developers
   6.4.3.2 Organic solvents
   6.4.3.3 Photoresist

6.4.4 NEVER POUR ACID directly down a drain; always uses the aspirator. The LISE G06 acid wet bench has an aspirator.

6.5 USE OF SOLVENT CUP SINKS

6.5.1 Solvent and photoresist waste must be poured into cup sinks (solvent drains) located in dedicated solvent benches.

6.5.2 Cup sinks (pictured below) are to be used for:
6.5.2.1 Disposal of Non-Halogenated solvents at a solvent bench. Example: SU-8 Developer (PGMEA), IPA, Methanol, Ethanol, Acetone, Hexane, etc.
6.5.2.2 Spraying samples with non-halogenated solvents from a hand held spray bottle.

6.6 WET BENCH DESIGNATIONS
6.6.1 The stainless steel bench is referred to as “SB08 – Solvent Bench”. The white polymer bench is referred to as “AB04 – Acid/Base/Aqueous Bench”.
6.6.2 Each bench is labeled in its upper right corner.

6.7 CONTROLS AND INTERLOCKS
6.7.1 ReynoldsTech wet benches have several alarm which may disable the bench completely or partly disabled for use until cleared.
6.7.2 Do not use a bench in alarm. Seek staff assistance.
6.7.3 Below are examples of various causes and required action.

<table>
<thead>
<tr>
<th>Alarm/Error Message</th>
<th>Cause</th>
<th>Required Action</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>Heated Tank Low Level</em></td>
<td>Proper tank operating level was not maintained in the specified tank</td>
<td>Fill process tank to proper operating level</td>
</tr>
<tr>
<td><em>Heated tank over</em></td>
<td>An over temperature</td>
<td>First ensure proper operating level was maintained.</td>
</tr>
</tbody>
</table>
temperature situation has occurred in the specified tank. The temperature controller not configured properly. Fill the tank with colder medium to reduce the temperature of the bath below the over temp setpoint. If this does not work reprogramming of the temperature controller may be required.

Leak detected in drip pan There is a liquid leak in the specified location Using all facility personal protective protocols, clean the spill appropriately.

Bottle wash lid open The lid of the bottle wash opened somehow during operation Close the lid of the bottle wash

Waste carboy not in place The carboy may have moved or may not have been pushed all the way back to resting point. Check to see if the carboy is in place. If not, put it in place or if the carboy drawer is not in its home position, push it back

Waste carboy high level The carboy has reached high level Empty the carboy properly following all facility safety protocols

Check waste bottle level Photo resist waste hasn’t been checked for nine hours Pull the bottle out of its resting spot to check level and replace if necessary

Waste bottle not in place Waste bottle was not replaced Replace waste bottle

6.8 EQUIPMENT SHUT-DOWN

6.8.1 Rinse all equipment and materials with DI water and IPA three times.
6.8.2 Wipe-down all surfaces and any fixtures with DI water and IPA until free of all process residues.
6.8.3 Clean PPE as necessary with IPA.
6.8.4 Beakers must be emptied, cleaned and wiped dry. If leaving a beaker full for overnight processing, label it, date it and sign it.
6.8.5 Static/ultrasonic baths, etching and lift-off processes may take several hours and often users leave their wafers processing overnight with a label.
6.8.6 If wafers are seen inside a tank, bath or on a hot plate, do not turn off this unit.
6.8.7 If leaving beakers or pans with a chemical content and samples in it, leave a note by the beaker with name, chemical, the date, time, and return time.
6.8.8 Dispose of all wipes used.
6.8.9 Unused wipes must be left on a table outside the wet bench.
6.8.10 Sinks should be emptied, drained and free of beakers, tweezers etc.
6.8.11 Chemical resistant gloves must be washed and dried with a tex wipe, then stored appropriately.
6.8.12 Nitrogen guns must be placed back inside their compartments.
6.8.13 Lower wet bench shield.
6.8.14 Turn off light if bench unlikely to be used soon.

7.0 RESPONSIBILITIES

7.1 Trained personnel conducting these operations are responsible for ensuring compliance with the all Harvard University - Center for Nanoscale Systems laboratory use protocols and/or documented procedures.
7.2 Any user who damages or contaminates these systems is responsible for restoring the system to its original state.

7.3 Users must report unsafe conditions or activities to CNS staff or EHS.

8.0 RECORDS

8.1 Any problems or issues must be documented with CNS FM032 “Incident Report Form”.

9.0 STATISTICAL TECHNIQUES

9.1 NA
10.0  REVISION HISTORY

<table>
<thead>
<tr>
<th>Revision #</th>
<th>Date</th>
<th>Author(s)</th>
<th>Changes/Additions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>Aug 4, 2010</td>
<td>E. Martin</td>
<td>Compressed file, minor edits.</td>
</tr>
<tr>
<td>1.2</td>
<td>Jul 28, 2011</td>
<td>H. Lin</td>
<td>Minor edits.</td>
</tr>
<tr>
<td>1.4</td>
<td>Aug 26, 2011</td>
<td>E. Martin</td>
<td>Correct the names of the benches.</td>
</tr>
<tr>
<td>1.5</td>
<td>Aug 26, 2011</td>
<td>H. Lin</td>
<td>Update the required CNS form 106.</td>
</tr>
</tbody>
</table>