UNIVERSITY OF WATERLOO | Quantum NanoFab

Standard Operating Procedure (SOP): HF Acid Wetbench (HFACID)

Author:	Vito Logiudice
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Revised by:	Nathan Nelson-Fitzpatrick
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	Rev2: clarification regarding proper footwear (section 4)
	Rev3: Revised small spill protocol to include a rinse
	Rev4: Update for QNC

Summary:

This document summarizes the safe operating practices to be followed when working with hydrofluoric acid (HF) based solutions in a Wetbench (Fumehood) which has been specifically designated for such use.

Important:

Wetbenches are dedicated to a specific group of chemicals. **Never** use a chemical in a given fumehood unless the fumehood itself is labeled for use with the chemical in question. Failure to comply may result in **a fire or an explosion.**

If in doubt about compatibility, ask a QNC NanoFab staff member.

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1. EQUIPMENT DESCRIPTION

This SOP is to be used for wetbenches (fumehoods) which are dedicated to Hydrofluoric (HF) acid solutions. These include solutions such as H_2O:HF mixtures and Buffered Oxide Etch (BOE) or Buffered HF (BHF) mixtures which are composed of Ammonium Fluoride (NH_4F) and HF.

For any other mixtures which use HF combined with other chemicals not noted above, consult with facility staff before using these in the lab.

Wetbenches are dedicated to specific groups of compatible chemicals. This procedure is for wetbenches which are strictly dedicated to the use of HF acid solutions.

HF solutions are extremely toxic! You must become familiar with the MSDS which identifies the hazards and the proper emergency response procedures in the event of contact.

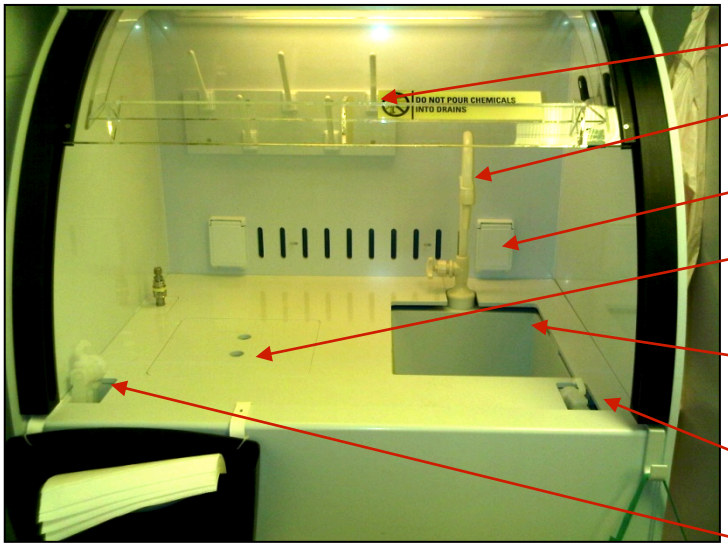
Bare skin contact with HF can lead to severe injury or death if not treated immediately. The best way to prevent all physical contact with such solutions is by strictly following the procedures listed in this document and by working slowly and diligently at all times.

Remember as well that inadvertent mixing of incompatible chemicals such as acids and solvents may result in violent reactions or explosions.

So, NEVER use chemicals in a wetbench which is not specifically identified as being compatible with a chemical you plan to use. Ask a member of staff when in doubt.

Wetbenches are located at multiple locations throughout the facility. Any work which requires the use of HF acid MUST be performed in a bench rated specifically for this chemical.

1.1. GENERAL LAYOUT & FEATURES



Clear, sliding sash (normally kept down)

Foot-pedal actuated DI water gooseneck

Covered power outlets

Recessed, temperature-controlled bath (Ask staff for assistance)

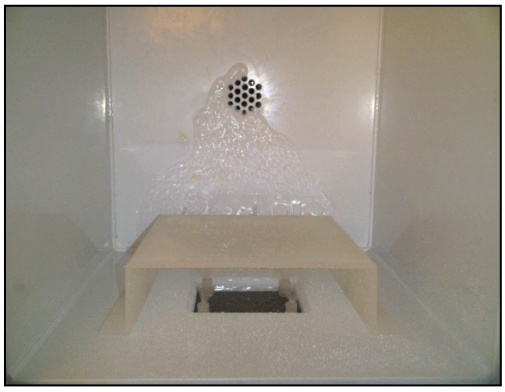
Sink with switch-actuated drain (switch located at top panel, above sink)

Nitrogen blow gun

DI water spray gun

Sink equipped with a DI water spray basket for rinsing wafers or small pieces held in perforated baskets made of an appropriate material.

Spray function is activated by switch located on top panel, above the sink.



NOT AVAILABLE FOR USE: Recessed, temperature controlled bath. Note: Ask staff if you wish to use temperature controlled bath.

Emergency First Aid Kits (Calgonate and Hexafluorine) for hydrofluoric acid burns located next to wet bench.

It is each user's responsibility to learn how to use these kits and to respond to emergency situations by becoming intimately familiar with the "FIRST AID MEASURES" section of the relevant MSDS.

2. REFERENCE DOCUMENTS

- MSDS sheet for chemicals to be used. MSDS sheets are located in the labeled binder by the cleanroom entrance. A sample MSDS for a typical HF solution can be found online: https://fab.qnc.uwaterloo.ca/equipment/bench-acid-HF/manuals/internal/sample-hf-msds/view
- Good reference document from the Government of Western Australia (Working with hydrofluoric acid): https://fab.qnc.uwaterloo.ca/equipment/bench-acid-HF/manuals/internal/HD-ref-doc-govt-australia/view

- Cleanroom Safety Protocols listed online: https://fab.qnc.uwaterloo.ca/policies/safety
- Emergency contact numbers listed online: https://fab.qnc.uwaterloo.ca/data/access/safety/emergency-phone-list
- Process recipes for the chemical solution needed. These PROC documents are available online in the "*Process Info*" folder under each piece of equipment in the *Equipment Info* section:

3. MINIMUM REQUIREMENTS BEFORE USE

Before using this equipment independently, you must have completed the "*Becoming a Lab Member*" requirements listed on the facility website, the major elements of which include:

- Completing and passing all required Safety & Cleanroom training
- Submitting a *Process Review Request* (one for each of your unique process flows)
- Submitting an *Equipment Authorization Request* (one for each equipment needed)
- Receiving one-on-one equipment training by an authorized staff member

Additional requirements specific to this equipment:

- Reserve time on the wetbench through the facility's Badger scheduler well in advance of your session. Remember to "Enable" the tool before you begin using it and "disable" the tool at the end of your session.
- Review and become familiar with the risks and the emergency response procedures listed in the *Material Safety Datasheets* (MSDS) for the chemicals you plan to use.
- Respect the ***Buddy System Policy*** at all times: Make sure there is another lab member with you in the cleanroom at all times while using any wetbench or fumehood. This person will be there to provide assistance or seek help in the event of an emergency.

4. HEALTH, SAFETY & ENVIRONMENT

Extremely toxic and dangerous chemicals and mixtures are routinely used on this equipment. It is important that the safety and environmental requirements and guidelines listed below be followed without exception to ensure the safety of yourself, others and the environment.

Closed shoes MUST be worn to minimize the risk of potential contact in the event of spills. Sandals or similar open footwear (including "Crocs" type shoes which have perforations in them) must not be used in the cleanroom!

Contact lenses should NOT be worn in the cleanroom as these will trap chemicals in the eyes in the event of splashing, thus hindering first aid procedures.

4.1. MANDATORY PERSONAL PROTECTIVE EQUIPMENT (PPE)

The following equipment **MUST** be worn when using this equipment and must be put on **in the order shown**:

1. Cleanroom *Nitrile* gloves

2. Safety glasses

3. *Tychem* yellow apron

4. Faceshield

5. Green *Nitrile* gloves

Important safeguards:

1. Visually inspect all safety gear for any damage prior to donning.
2. Never touch with your white cleanroom gloves with those parts of the green nitrile gloves which may have come into contact with chemicals (ie., fingertips, palm & back of the hand).
3. Before putting on the green gloves, roll the ends so as to make a 1 inch cuff which will help keep chemicals from rolling onto your sleeves (circled in red above).
4. **NEVER** touch any part of your body (apron, faceshield, head, etc.) with your green gloves.
5. During your session at the wetbench, avoid direct contact with chemicals at all times. Never plunge your gloved hands into containers filled with chemicals!
6. Work slowly and deliberately so that you do not splash chemicals onto your protective gear, on the floor or on the wetbench work surface.
7. **NEVER** walk around the cleanroom while wearing any of the protective gear shown above (items 3, 4 & 5) and certainly **NEVER** sit at another workstation while wearing

this gear. Doing so may contaminate other areas of the cleanroom with dangerous chemicals thus making the facility unsafe for yourself and other lab members.

Removing your protective gear at the end of your session:

1. At the end of your session and while still wearing your protective gear, visually inspect the front of your apron to make sure that there are no chemical residues on it. If there are, wipe them off with a cleanroom wipe. Dispose of the wipe in the yellow waste container labeled for acid chemical waste.
2. While still wearing your green gloves, rinse these thoroughly under running water in the sink in the fumehood. Dry them with absorbent wipes.
3. Carefully remove your green gloves by gently pulling them off at the finger tips. Lay the green gloves on the fumehood deck with the fingertips over the edge of the sink.
4. Carefully handle the green gloves by the dry, cuffed sleeves and clip them to the side of the fumehood, over the sink.
5. Remove your faceshield. Inspect it for damage or residues before hanging it up for the next user. Clean it if needed, all while being weary of chemical residues.
6. Remove your apron.
7. If any gear appears damaged or excessively worn, ask a staff member to replace it.

4.2. CHEMICAL WASTE

Never pour chemicals down the drain!

All solutions made up of some combination of:

- H_2O
- HF
- NH_4F

must be disposed of in the waste container labeled "WASTE HF SOLUTIONS ONLY". This container is located in the compartment underneath the sink. If it is full, contact staff.

Place the waste container onto the fumehood work surface and lower the sash to the indicated safe setting (see section 7) before beginning the waste disposal sequence.

When pouring the waste solution into the waste container, do so slowly and deliberately. Avoid splashing or spilling any liquid onto the outside of the container or onto the wetbench work surface.

Any other solutions: Consult with staff for proper disposal procedure.

4.3. IN THE EVENT OF AN EVACUATION ORDER (FIRE ALARM, ETC.)

If time permits:

1. Turn off any flowing water in the wetbench
2. Remove your PPE in the proper order: green gloves then faceshield then yellow apron
3. Pull down the sash to the fully closed position
4. Leave the cleanroom immediately

4.4. IN THE EVENT OF A CHEMICAL SPILL

If contained on the workbench surface and not more than a few milliliters (10 to 20):

1. Remain calm, inform staff if you are uncomfortable with the cleanup or wish to be supervised during cleanup.
2. Gently and slowly rinse the spill and surrounding area towards the sink, use copious amounts of water. Run faucet in the sink to dilute the HF as you rinse.
3. Continue running the sink faucet for two minutes
4. Absorb remaining wetness on deck with cleanroom wipes
5. Use another, water dampened wipe to wipe the surface clean
6. Wipe the surface dry with clean wipes
7. Dispose of the contaminated wipes in the yellow acid chemical waste bin
8. Advise staff so that the chemical waste bin contents can be safely disposed of

If contained on the workbench surface and greater than a few milliliters:

1. Advise a staff member
2. Remain on hand to help with the cleanup as needed

If spilled onto the floor:

1. Remain calm
2. Advise your buddy or someone else in the lab that you had a spill and that you need help
3. Keep at least 3 metres away from the spill and make sure no one else approaches the area
4. Ask your buddy to evacuate the cleanroom and ask him/her to return to help you.
5. Check your personal protective equipment (PPE) to see whether it has come into contact with the spilled solution (ie., apron, green gloves, etc.). Ask your buddy to double check if needed.
6. Dry off any obvious chemical on your PPE with cleanroom wipes.
7. Pile the wipes on the floor off to one side.
8. Carefully remove your PPE in the usual sequence and drop these onto the floor in a pile.
9. Double check to make sure you have not come into contact with any solution (ex: check your shoes).
10. If contact is suspected, immediately rinse with water and commence first aid procedures. Your buddy will be there to help.

11. Evacuate the space and remain on site, just outside the cleanroom until told to leave by staff.

5. MATERIALS & SUPPLIES NEEDED

Plan your work before you begin to make certain that all necessary materials and supplies are on hand when needed. This will keep you from having to remove your protective gear to go fetch needed materials mid-course during your session.

You will likely need some combination of the following items when using this equipment:

- Chemicals. Acids are stored in the labeled, blue chemical cabinet located in the cleanroom. It is your responsibility to safely take the necessary chemicals to your workspace and to return them to the proper storage cabinet when done. See the last bullet in section 7 for proper handling of chemical bottles to/from the fumehood.
- Beakers made of materials compatible with the chemicals you'll be using. In this case, **only** Teflon or polypropylene beakers should be used.

IMPORTANT: HF solutions attack glass and many metals. Thus, glassware and metal tweezers are **not allowed** in this wetbench

NOTE: Teflon beakers as well as polyethylene graduated cylinders are made available for general use in the hood itself. These **MUST** remain in the hood at all times. It is the responsibility of each user to thoroughly rinse, dry and replace these on the drying rack at the back of the hood at the end of each session.

- Graduated cylinders made of materials compatible with the chemicals you'll be using
- Tweezers (non-metal!) of the appropriate size and material for your application
- Substrate carrier(s) for introducing your sample into the chemical baths & for rinsing
- Cleanroom compatible, absorbent wipes
- Etc.

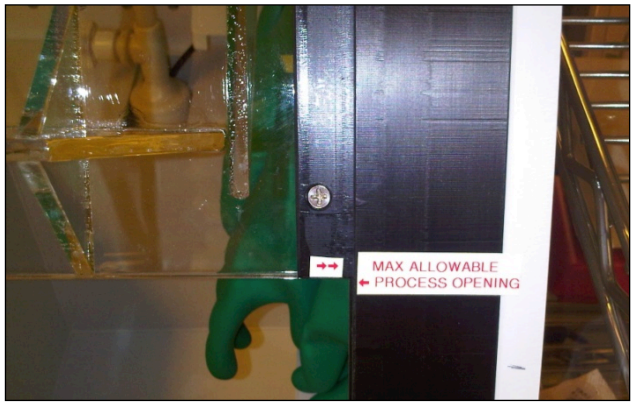
6. VERIFICATIONS BEFORE STARTING

Check the following parameters via the control panel at the top of wetbench:

- Power ON
- Exhaust Monitor ≥ 0.3 inches W.C.
- Light switch ON (switch not shown)

7. GENERAL OPERATING GUIDELINES

- **NEVER** pour any chemical back into its original container as this will contaminate the remaining fresh solution.
- **NEVER** pour chemicals down the drain.
- **NEVER** mix acids with solvents. **These may react violently and cause a fire or an explosion.**
- **NEVER** mix acids with bases. **Such mixtures will react violently.**
- **NEVER** introduce new chemicals into the facility without prior approval by staff.
- **ALWAYS** lower the fumehood sash to the maximum recommended height before opening chemical bottles and commencing procedures.



- **ALWAYS** wear the Protective Personal safety Equipment (PPE) listed in section 4.1.

- **ALWAYS** finish open chemical bottles before opening new ones.
- **ALWAYS** **Add Acid (AAA) to water**, **NEVER** water to acid.
- **ALWAYS** label secondary containers with chemical composition, your name & the date.
- **ALWAYS** return chemicals to their proper storage cabinets at the end of your session via the use of bottle carriers (use green, nitrile gloves to place bottle in carrier):

8. STANDARD OPERATING PROCEDURE

Typical wet chemical processing can routinely be divided into the following three steps.

8.1. PREPARATION

1. Obtain all chemicals needed for your work.

   IMPORTANT: The bottles of chemicals which have been stored in the chemical cabinet may be dirty. If you plan to lay the bottles down on the fumehood work surface during your session, perform the following steps in sequence:

   i. Take the bottle from the bottle carrier and wipe it clean with a new cleanroom wipe. Wipe the bottom of the bottle last. Dispose of the wipe and keep the bottle in hand and off the wetbench work surface until step iii.

   ii. Place a new cleanroom wipe on the wetbench work surface, preferably towards the back or off to one side.

   iii. Place the bottle on the wipe and keep it on this surface until the end of your session or until you are done using it at which point you can check to make sure that it is dry and the cap is on tight before returning it to the bottle carrier on the floor.

2. Obtain all necessary beakers, graduated cylinders, tweezers, wipes, etc.
3. Inspect the hardware for cleanliness. If needed, rinse with DI water as needed (after putting on your personal protective equipment – next step).
4. Put on all safety gear as detailed in section 4.1.
5. Mix your solution. Important:
   - Use graduated cylinders to accurately measure the quantities needed
   - Remember the "**Always Add Acid (AAA) to water**" rule

NOTE: Refer to the relevant process (PROC) document for the solution you plan to use for solution temperature and mixing details.

8.2. PROCESSING

Specific process details on the solution you are using will be referenced in the relevant PROC document. Generally, processing consists of:

1. Soaking your substrate in the solution for a given amount of time
2. Sometimes, gentle back and forth agitation of the sample in the solution is desirable as this tends to improve oxide etch uniformity, particularly in cases where slight oxide etchback is required via the use of very dilute HF solutions.
3. Never leave solutions unattended!
4. At the end of the chemical soaking step, rinse your sample by introducing it in succession into each of three (Teflon or polypropylene) beakers filled with DI water. Gently move your sample around in each beaker for about 30-60sec each.
5. Alternatively, the wetbench's spray feature can be used for rinsing. The spray basket is located in the sink, towards the front of the wetbench. The DI water spray is actuated via the switch located on the top, exterior panel of the fumehood, on the right hand side. Do NOT touch the top panel switches on the hood with the green gloves. To actuate these switches, carefully remove a single green glove and use this hand to work the switches. Remember to put on your green glove before proceeding with work in the hood.
   Spray rinsing should be done for at least 60 seconds.
6. Dry your sample by holding it vertically with your tweezers on a clean wipe and blowing the water off with the nitrogen blow gun located on the right hand side of the wetbench.
7. Repeat as necessary for any samples being processed.

8.3. CLEANUP

At the end of your session:

1. Handle the chemical waste as detailed in section 4.2.
2. Place the original cap on all bottles of fresh, new chemicals which were not used.
3. Inspect bottles to make sure they are clean and dry.
4. Place the bottles in bottle carriers and lay these on the floor, against the wetbench.
5. Rinse the wetbench's work surface if needed and dry with an absorbent wipe.
6. Thoroughly rinse all beakers, graduated cylinders and tweezers.
7. Thoroughly rinse and dry your gloves while still wearing them.
8. With clean, dry, green gloves still on, dry all containers and tweezers with a combination of the nitrogen blow gun and clean absorbent wipes.
9. Wipe dry the wetbench work surface for the next user.

IMPORTANT: As a courtesy to others, inspect the work surface to make sure that it is thoroughly spotless and dry. Solutions like HF or HCl acid look like water so it will be very worrisome for the next user to arrive at a workstation where even a single drop of clear liquid remains on its surface. Please be considerate.

10. If applicable, place your beakers and other containers in your personal container.
11. If the lab supplied beakers were used, replace these on the drying rack inside the wetbench.
12. Carefully remove your personal protective equipment as detailed in section 4.1.
13. Return the chemicals to their proper storage cabinets.
14. Return your personal container (with your beakers, tweezers, etc.) to the centralized storage location for such containers in the cleanroom.

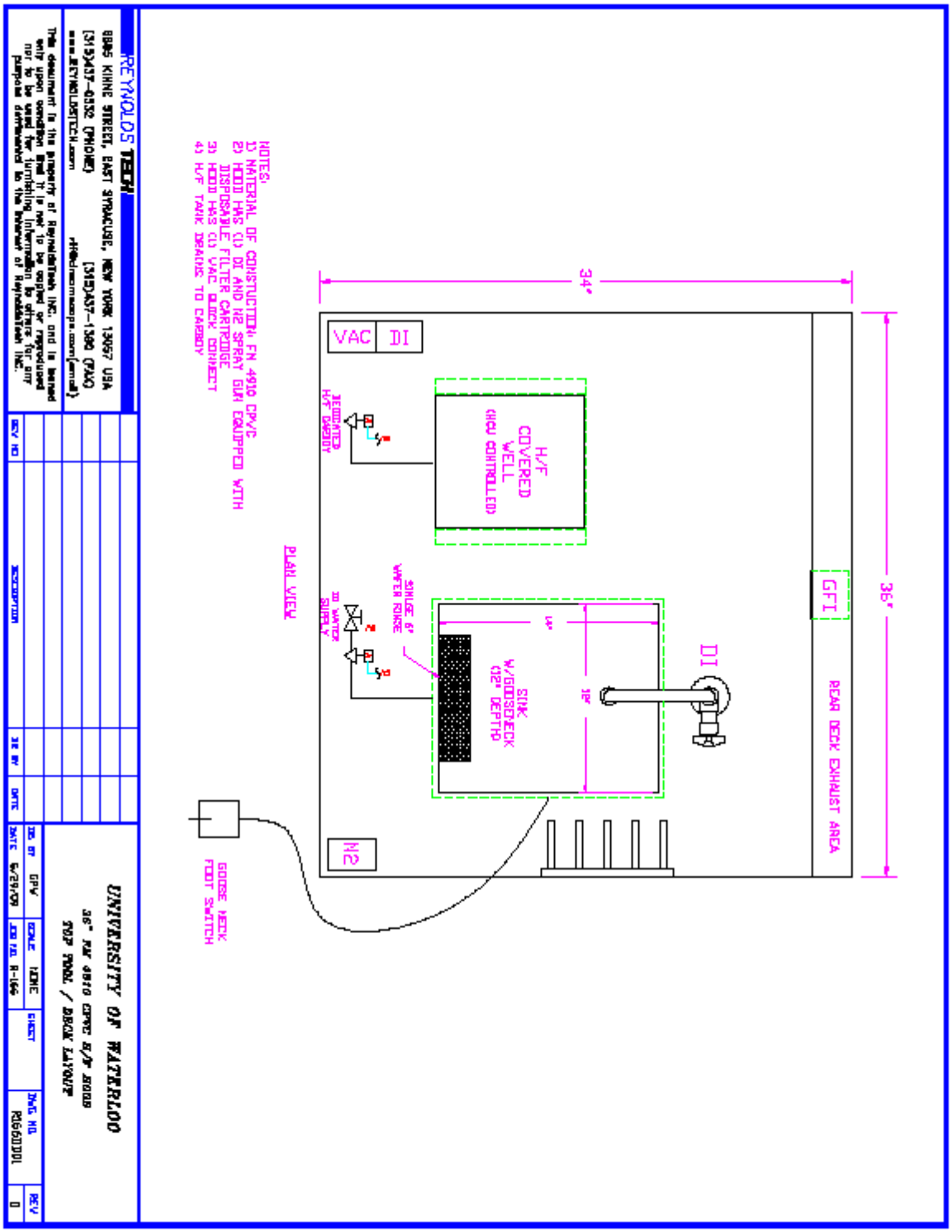
9. TYPICAL PROBLEMS & SOLUTIONS

1. Blotching or contaminants on substrate surface after drying:
   - Often, the source of the problem can be found to be dirty tweezers or beakers.
   - Solution used is incompatible with substrate or films on substrate. Ex., don't use a Piranha solution to clean GaAs substrates!

10. TECHNICAL DATA

None in this version.

APPENDIX


REYNOLDS TECH
UNIVERSITY OF WATERLOO
36" FAB 4010 CPVC H/F HOOD
STOP VALVE / DRAIN LAYOUT
NOTES:
1) MATERIAL OF CONSTRUCTION: FM 4910 CPVC
2) HOOD HAS (1) DI AND N2 SPRAY GUN EQUIPPED WITH DISPOSABLE FILTER CARTRIDGE
3) HOOD HAS (1) VAC QUICK CONNECT
4) H/F TANK DRAINS TO CARBOY
PLAN VIEW
34"
36"
VAC
DI
H/F COVERED WELL (HAS CONTROLLED)
H/F DISPENSER
WATER RINSE
SINK W/GOOSENECK 12" DEPTH
1"
12"
DI
GFI
REAR DECK EXHAUST AREA
N2
GOOSE NECK FOOT SWITCH