

## Piranha cleaning

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**Location:** Aisle 2 (Wetdeck 2B)

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## OVERVIEW

This document outlines the process for preparing a piranha bath (3:1 H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O<sub>2</sub>) used for removing organics and metallic contaminants from substrates. Cold piranha (<40 °C) is also used to clean chrome photomasks. Most surfaces are hydroxylated during piranha cleaning, becoming strongly hydrophilic.

## SAFETY PRECAUTIONS

**N.B.** Organics (acetone, isopropyl alcohol, photoresist, polymer films, etc.) should not be placed in a piranha bath or on the piranha wet deck. Contact between piranha and a large volume of organic contaminants will cause a violent reaction and a substantial explosion.

While not carcinogenic, the chemicals used in piranha cleaning are dangerous; care must therefore be taken to avoid physical contact with the cleaning solutions and their fumes. Please refer to the nanoFAB MSDS binder if you have any concerns using these chemicals.

**Sulphuric acid, H<sub>2</sub>SO<sub>4</sub> (96%)** A highly corrosive, strong mineral acid which is clear, colourless to slightly yellow, viscous, and pungent. Sulphuric acid causes severe burns on contact. The harmful vapour and mist can cause burns to eyes, skin, and respiratory tract.

**Hydrogen Peroxide, H<sub>2</sub>O<sub>2</sub> (30%)** A strong oxidizer and corrosive clear liquid. It can cause burns to skin, eyes, and respiratory tract.

Acid gear must be worn when preparing the piranha bath:

- Chemical apron
- Chemical-resistant gloves
- Face shield

### Important safety notes:

- **Mixing the chemicals for a piranha bath causes an exothermic reaction: the solution can self-heat up to 120 °C. Due to the high temperature, the bath should never be agitated.**
- **NEVER use plastic labware—use glass only.**
- **NEVER put piranha solution in a closed container: gas generation and the resulting over pressurization can lead to an explosion.**
- **Piranha solution also explodes if the peroxide concentration exceeds 50%. Consequently, the hydrogen peroxide must always be added to the sulphuric acid, NEVER vice versa.**

NO CHEMICALS ARE TO BE REMOVED FROM THE WET DECK IN OPEN CONTAINERS.

Before bringing any new materials into the nanoFAB for processing, it is necessary to fill out a new chemical import form ([www.nanofab.ualberta.ca/user-information/user-forms/chemical-import-form/](http://www.nanofab.ualberta.ca/user-information/user-forms/chemical-import-form/)).

## OPERATING PROCEDURE

1. Transfer substrates to a Teflon carrier (boat or basket, depending upon substrate size). Blue polypropylene locking tweezers may also be used to hold individual chips.
2. Choose a glass container for the bath which will fit the carrier. Label the container with the bath name (“piranha”), the user name, the date, and the time.
3. Determine the volume of solution required to immerse the substrates completely. This can be done “by eye” or more quantitatively by measuring with a beaker.
4. Calculate the amounts of sulphuric acid and hydrogen peroxide yielding the necessary final volume, using a volumetric  $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$  ratio of 3:1.

The table to the right lists volumes for typical 3:1 piranha baths. When choosing the final volume, it is worth noting that the vigour of the reaction depends on the surface-to-volume ratio of the solution (e.g., 400 mL of piranha in a large beaker may not bubble as violently as 400 mL in a small beaker).

Final volume (mL)	$\text{H}_2\text{SO}_4$ volume (mL)	$\text{H}_2\text{O}_2$ volume (mL)
400	300	100
600	450	150
800	600	200
1000	750	250
1200	900	300
1600	1200	400
2000	1500	500

5. Select two beakers for measuring sulphuric acid and hydrogen peroxide and mark the correct volume on each beaker. Place the measuring beakers and bath container on the wet deck.
6. Check the condition of a pair of chemical-resistant gloves by inspecting for dark spots or other evidence of chemical spills, then filling them with nitrogen using the nitrogen gun to check for leaks. If gloves are not in good condition, discard and obtain a new pair. New gloves may be obtained from the shelf in the sulphuric acid cabinet (Aisle 2); label with the size and date.
7. Don the acid gear: apron, face shield, and gloves.
8. Slowly pour the required amount of sulphuric acid into the marked beaker. Transfer this amount into the bath container labeled piranha and place the empty beaker into dump rinser.
9. Slowly pour the required amount of hydrogen peroxide into the second marked beaker. Transfer this amount into the bath already containing the sulphuric acid. Place the empty beaker into the dump rinser and start the 5× rinsing cycle (press **STOP** → **START**).
10. Carefully place the Teflon carrier of substrates into the piranha bath. There may be a vigorous reaction evident by “bubbling and spitting”. If holding chips with blue tweezers, thread a Teflon rod through the tweezer handles and place the rod across the top of the bath beaker, such that the chips are below the piranha surface.
11. Start a timer for 15 minutes. After 15 minutes the piranha will have cooled such that the reaction is no longer vigorous enough to etch effectively.
12. Wash the chemical bottles on the outside with DI water and pat dry with a cleanroom wipe. Place the bottles back in the appropriate storage cabinets. If you empty a chemical bottle, rinse the inside 3× with DI water, wash the outside, pat dry, cross out the bottle label with a Sharpie marker, write “washed bottle” on the outside, and place in the washed bottle storage bin opposite the gowning room door.
13. Rinse the chemical-resistant gloves with DI water and dry with a cleanroom wipe. Remove acid gear and place back on the appropriate racks.
14. Once the rinse cycle is finished, press **OPEN** to drain the dump rinser, remove the beakers, and place them on the drying rack. Wipe off measuring marks with a cleanroom wipe moistened with acetone or IPA.
15. When the piranha clean has finished, don the acid gear again and slowly remove the carrier from the piranha bath, allowing excess liquid to drip back into the bath.
16. Transfer the carrier carefully into the dump rinser and start the 5× rinsing cycle. If using chips with blue tweezers, hold them over the dump rinser and spray copiously with DI water.
17. Push the piranha bath vessel to the back of the wet deck; nanoFAB staff will aspirate the piranha once cool.
18. Spray the wet deck with DI water to rinse any drips of piranha.
19. Rinse the chemical-resistant gloves with DI water and dry with a cleanroom wipe. Remove acid gear and place back on the appropriate racks.

20. Once the dump rinsing cycle has finished, dry substrates with nitrogen gun or use the spin rinse dryer (if wafers are in a boat).
21. Wipe the front of the wet deck with a cleanroom wipe to ensure there are no water droplets.

## **TROUBLESHOOTING**

If you encounter any unexpected errors or require assistance, please contact the primary or secondary trainer listed above. If they are not available, please contact any nanoFAB staff member for assistance.

## **APPROVAL**

**Qualified Trainer:** Stephanie Bozic

**Group Leader – Fabrication:** Aaron Hryciw