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| **AUP Acid Etching at the Chemical Fume Hood Procedure** |
| **Document Number:** | AUPPS-PR-CHEM-CAV-ACID | **Effective Date:** | DD Mmm YYYY |
| **Revision Number:** | 1 | **Periodic Review Date:** | DD Mmm YYYY |
| **Document Owner:** | River Fiedler | **Department Owner:** | SRF Operations |

# Purpose and Scope

This procedure covers parts etching procedure performed at the chemical fume hood.

This procedure applies to acid work related to AUP

# Definitions and Diagrams

The following terms have specific meanings within this procedure.

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| **Term** | **Definition** |
| Component | For the simplicity of this procedure, the terms cavities, components, and/or parts are considered interchangeable and will be generally referred to as “component”. If a component is a mix of the following metals or an unusual size or shape, the PI/TR and/or a supervisor should be consulted on the appropriate method to use |
| Hardware/Fastener | Nuts, bolts, washers. These should not be degreased in the same container or ultrasonic as components or feedthroughs and other parts that potentially see beam or face the inside of a cavity |
| DI/UPW | Deionized (DI) and Ultra-Pure Water (UPW) are used interchangeably in this procedure. |
| Ultrasonic, USC, and sonic | Are used interchangeably in this procedure. The container or tank may also be referred to as a bath |
| N2/Nitrogen | Filtered nitrogen is most commonly used |
| Quick Dump Rinser | A sink that fills and empties water to rinse components several times. |

# Roles and Responsibilities

The following roles have responsibilities described in this document.

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| **Role** | **Responsibility** |
| Technician | A trained, qualified person who will execute this procedure. |
| PI/PM/TR/SL/WCL | Principal Investigator, Project Manager, Technical Representative, Scientific Lead, and Work Center Lead (supervisor). Someone knowledgeable of or in charge of the project or item in question who can provide guidance if questions arise. |

# Safety

Individual must keep safety as the first priority in the process; before beginning any job, the user must assure they have the correct PPE for the individual job. Maintaining the level of safety and secure nature of the work area is paramount. Assure personal safety by using caution in movement and taking necessary steps to avoid unnecessary personnel in the immediate area.

# Procedure

***NOTE: The following steps of this procedure involve interaction with various acids. Users are to keep an acute awareness of surroundings and maintain professional decorum at all times. All activities involving the use of concentrated acids shall be performed at the chemical fume hood with a properly operating fume extraction system while wearing appropriate PPE.***

***Refer to the work center OSP for further details.***

## Acid Preparation:

To prepare acid for component processing several steps must be followed to safely perform the tasks plus a plan for chemical storage and waste disposal must be thought out and communicated to the room safety warden and safety personnel.

**ES&H must have approved the recipe prior to conducting any acid work.**

***NOTE: The operator is responsible for utilizing appropriately sized and constructed containers/fixtures prior to executing this procedure. Ensure chemical compatibility.***

Premixing steps:

1. Components to be etched may require cleaning prior to any acid work. If any cleaning is necessary, refer to the cleaning procedure for the appropriate method based on the type of metal.
2. Prior to performing a task determine if a water “dry run” is needed. If so, perform tasks with water to determine volume, hardware, and steps necessary to perform the task safely.
3. Determine the amount of acid necessary for the process or determine if used acid is available to minimize storage. If new acid is necessary, measure out the process with water in a mock assembly as necessary to determine the methods of transfer and minimal amount.
4. If mixing new acid, choose a container that is adequate for the necessary volume. Use HDPE constructed containers specifically purchased for acid storage. Inspect containers to ensure integrity.
5. Determine if there is space in the acid storage cabinet to store the new acid container(s). If using pre-used acid, determine if the individual acid stock is adequate for the volume to be mixed.
6. Locate the proper NFPA hazard label prior to starting and fill out label with: chemical name, date, and owners name. If using used acid, ensure it is within the acceptable date of use.
7. Determine if the DI water plant is operational and available.
8. If space is not available for the new acid contact the safety warden before proceeding.
9. If mixing new acid, plan ahead and prepare any labels or triple rinse stickers as needed. It may be helpful to label any beakers or containers being used to measure acid with the volumes intended to be mixed.

Safety PPE:

For any acid work, PPE is required to perform any of the tasks. The PPE identified is as follows: Rubber Boots, Rubber apron tied tight around back and neck, approved hood with powered air purifying respirator, inner gloves (nitrile) and outer gloves (neoprene).

Refer to the work center OSP for further details.

1. Inspect PPE prior to donning; look for excessive wear, damage, punctures etc.
2. Test inner and outer gloves by capturing air and pressurizing to the finger tips by reducing the volume. The gloves should hold air with no leaks if not replace
3. Don PPE and check for exposed skin
4. Replace any warn or damaged PPE before beginning work, dispose of used PPE properly.

Wet Bench Usage:

The wet bench is necessary to remove hazardous acid fumes formed during the operation. The fume hood is marked on the side of the sash when the highest position the sash can be opened when acid is present and opened in the hood. The mark indicates 100fpm of air flow through the opening and the hood has an alarm for the exhaust failure.

1. If the exhaust alarm goes off during the processing or acid mixing or cleanup, close any open acid container then close the sash, exit the room and report this to the safety warden.
2. If the alarm is going off prior to starting do not ignore it or start working, report it to the safety warden

General Acid Mixing Guidelines

**Note: If a recipe calls for water, ALWAYS add acid into water, NEVER water into acid.**

1. Check Wet bench for adequate space prep work area.
2. Turn on the DI water and wet bottom of the wet bench and leave flowing at a low flow throughout the mixing process.
3. Place labeled containers in wet bench.
4. If water is an ingredient, always start with water first. Water should never be poured into acid as it could cause a dangerous reaction.
5. Transfer a bottle of virgin acid to the wet bench using bottle carriers for secondary containment.
6. Open the virgin acid bottle and transfer an appropriate volume to a “measuring” plastic beaker (such as a graduated cylinder) and then transfer to an empty pre-labeled storage container or mixing container.
7. Repeat for the appropriate types and volume of other acids needed for recipe.
8. Use one acid at a time and, when finished, rinse the outside of each bottle with water and return to storage cabinet or triple rinse the bottle and label with a “Triple Rinsed” sticker for disposal if empty.
9. Ensure all containers with acid in them are closed and sealed. Thoroughly rinse all containers that contained acid with DI water.
10. Clean up work area by returning bottles to the storage cabinet and triple rinsing bottles appropriately.
11. Agitate the mixed acid to properly blend.
12. Pour mixed acid into the storage container and transfer to the acid storage cabinet for later use or proceed to etching steps if being use immediately.
13. Rinse all wet bench areas and PPE to ensure no residual acid remains, turn off DI water
14. Squeegee floor around work area to remove any excess water resulting from rinse process.

BCP: Hydrofluoric: Nitric: Phosphoric 1:1:1 or 1:1:2

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BCP 1:1:1 is a combination of 1 part Hydrofluoric Acid, 1 part Nitric Acid, and 1 part Phosphoric Acid.

BCP 1:1:2 is a combination of 1 part Hydrofluoric Acid, 1 part Nitric Acid, and 2 parts Phosphoric Acid.

Copper Bright Dip

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Copper Bright is a mixture of 55% Phosphoric Acid, 20% Nitric Acid, and 25% Acetic Acid.

Mixing Stainless Steel Etch

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Stainless Steel Etch is a mixture of 66% Water, 4% Hydrofluoric Acid, and 30% Nitric Acid.

An aggressive etch can be mixed using 15% Water, 35% Hydrofluoric Acid, and 50% Nitric Acid.

Acid Etching with BCP:

**NOTE:** Fresh BCP 1:1:1 yields an average etch rate of ~8µm/min at a temperature of 20°C. However, when using used acid, take an etch rate sample and adjust the time to achieve the desired removal. During the etching process, keep track of the acid temperature. The user can implement an ice bath into the procedure to maintain a lower temperature if necessary.

1. Setup hardware for BCP etching, acid, and rinse containers.
2. Don PPE.
3. Turn on DI water.
4. Setup components to be etched and appropriate containers in or near the chemical hood.
5. Transfer the bottle of premixed BCP acid to the wet bench using bottle carriers for secondary containment.
6. Open mixed acid container and transfer to pre-determined etching container.
7. Submerge each component to be etched into the acid for the prescribed amount of time. The acid should be agitated by either stirring the acid or by moving the component. Monitor acid temperature to ensure controlled etch rate.
8. Thoroughly rinse the component immediately upon removal.
9. Transfer the rinsed component to a bath of UPW for a minimum of five minutes. This will help eliminate the potential of trapped acid.
10. Repeat as needed for additional components.
11. Return acid to the storage container.
12. Ensure all acid containers are closed and sealed. Thoroughly rinse off the mixed acid container with DI water and return it to the acid storage cabinet using bottle carriers for secondary containment. If necessary, allow acid to cool prior to storage.
13. Meticulously rinse off all process hardware and associated containers and tooling.
14. Remove all hardware from the acid fume hood and return it to storage locations
15. Clean up wet bench, rinse all surfaces with DI water and test with pH paper
16. Squeegee floor around work area to remove any excess water resulting from rinse process.

Acid Etching with Copper Bright

**Note: Oxides form on the surface of copper very quickly. Do not allow components to dry between steps.**

1. Setup hardware for etching, acid, and rinse containers.
2. Don PPE.
3. Turn on DI water.
4. Setup components to be etched and appropriate containers in or near the chemical hood.
5. Transfer the bottle of premixed acid to the wet bench using bottle carriers for secondary containment.
6. Open mixed acid container and transfer to pre-determined etching container.
7. Submerge each component to be etched into the acid for the prescribed amount of time. **(Generally about 45 seconds).** The acid should be agitated by either stirring the acid or by moving the component.
8. Thoroughly rinse the component immediately upon removal. Save this first batch of rinse water and dispose of properly.
9. If the surface finish is not satisfactory for the desired outcome, the part may be etched again in the acid bath until the desired finish is achieved.
10. Transfer the rinsed component to a bath of UPW. This will help eliminate the potential of trapped acid.
11. Immediately transfer to an acetone bath.
12. Repeat as needed for additional components.
13. Return acid to the storage container.
14. Ensure all acid containers are closed and sealed. Thoroughly rinse off the mixed acid container with DI water and return it to the acid storage cabinet using bottle carriers for secondary containment. If necessary, allow acid to cool prior to storage.
15. Meticulously rinse off all process hardware and associated containers and tooling.
16. Remove all hardware from the acid fume hood and return it to storage locations
17. Clean up wet bench, rinse all surfaces with DI water and test with pH paper
18. Squeegee floor around work area to remove any excess water resulting from rinse process.
19. Copper parts are dried with filtered N2 immediately upon removal from the acetone bath. The components are then sealed in nitrogen in bagging material.

Acid Etching Stainless Steel

1. Setup hardware for etching, acid, and rinse containers.
2. Don PPE.
3. Turn on DI water.
4. Setup components to be etched and appropriate containers in or near the chemical hood.
5. Transfer the bottle of premixed acid to the wet bench using bottle carriers for secondary containment.
6. Open mixed acid container and transfer to pre-determined etching container.
7. Submerge each component to be etched into the acid for the prescribed amount of time. **(Generally about an hour).** The acid should be agitated by either stirring the acid or by moving the component.
8. Thoroughly rinse the component immediately upon removal.
9. Transfer the rinsed component to a bath of UPW for a minimum of five minutes. This will help eliminate the potential of trapped acid.
10. Repeat as needed for additional components.
11. Return acid to the storage container.
12. Ensure all acid containers are closed and sealed. Thoroughly rinse off the mixed acid container with DI water and return it to the acid storage cabinet using bottle carriers for secondary containment. If necessary, allow acid to cool prior to storage.
13. Meticulously rinse off all process hardware and associated containers and tooling.
14. Remove all hardware from the acid fume hood and return it to storage locations
15. Clean up wet bench, rinse all surfaces with DI water and test with pH paper
16. Squeegee floor around work area to remove any excess water resulting from rinse process.

# References

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| **Document No.** | **Title** |
| SRF-06-PR-001 | Records Management Procedure |
| SRF-07-PR-001  | Document Management Procedure |
| <Doc Id> | <Document Title> |

# Release and Revision History

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| **Rev #** | **Major Changes** | **Revision Date:** |
| 1 | Initial version (Utilizing SRF-07-FM-005 SRF OPS Procedure Template, R1) | DD Mmm YYY |

# Approvals

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| --- | --- |
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For Project Procedures: Refer to the Project Execution Procedure SRF-11-PR-001

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