Procedure Title: Mixing and Using Etchants or Solutions that Contain Picric Acid

Minimum Review Requirements: Annually

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1. Version History

Version #:  1
Supersedes:  n/a

Handwritten amendments to the official procedures can be made by a single line through the text, along with the date, and initialed by the authorized individual making the correction. Changes are to be noted below. Formal changes to this SOP are made on the date of revision or sooner, where required.

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2. Introduction

This SOP provides the framework for using Picric Acid containing etchants in room 0C14 of the Engineering building. Usage of Picric Acid is not allowed outside of 0C14 or by personnel or students who have not been specifically authorized to handle Picric Acid. Picric Acid will be kept in a locked cabinet, with supervisors given keys.

All faculty supervisors of students working with Picric Acid must have read, understood and signed this SOP.

It is the responsibility of the Faculty Supervisors to ensure that all users of picric acid under their supervision/direction are competent in the use of the provided PPE and understand the hazards involved with using picric acid. Faculty Supervisors also agree to cover the cost of any services related to picric acid disposal not covered by the University Waste Management Facility.

All people wanting to use picric acid must first submit a proposal outlining why it is necessary and what alternatives have been considered and the reason for their rejection. Unauthorized use of picric acid will result in immediate loss of laboratory privileges.

3. Definition

SOP: Standard Operating Procedure
MSDS: Material Safety Data Sheet
WHMIS: Workplace Hazardous Materials Information System
PPE: Personal Protective Equipment
WSEP: Workplace Safety and Environmental Protection
Acetic-Picral Etchant: 5 ml Acetic acid, 6 g Picric Acid, 100ml ethanol, 10ml distilled water

4. Personnel

Persons authorized to perform this SOP:

By signing this form I acknowledge that I have read and understand this SOP, as well as the applicable MSDS’s and that I will conduct myself in accordance with this SOP and the general laboratory rules.

NOTE: ALL SIGNATURES MUST BE PRESENT ON THE SOP LOCATED IN THE YELLOW BINDER IN ROOM 2C26 – Mechanical Engineering Materials Lab, digital copies of SOP’s are made available for reference and convenience only. Printed SOP’s are valid for 24 hours only, after that time their accuracy must be verified with the OFFICIAL HARDCOPY VERSION.
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<tr>
<td>Prof. Akindele Odeshi</td>
<td>Faculty</td>
<td>ME</td>
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Reference Only
5. Safety

5.1 Chemical Safety and Hazards

- Preparation/usage of picric acid and picric acid containing etchant(s) must be conducted inside the laboratory fume hood. The fume hood’s sash must be at an appropriate height, and the exhaust fan operational providing a face velocity of at least 100fpm.
- Proper PPE including disposable nitrile gloves (for splash protection, not immersion) must be worn. Hands must be washed with water and soap upon removal of gloves.
- Picric Acid is toxic by all routes of entry.
- Picric Acid is very toxic if inhaled. It is also corrosive to eyes and skin. It may cause permanent injury to eyes. It can hurt target organs such as blood and kidney.

5.2 Explosion Hazards

- Picric Acid is explosive when dry (<10% moisture content).
- Explosions can occur due impact or friction (such as bottle screw threads) on the surface of dry picric acid crystals.
- Picric Acid must be stored under distilled water, and the bottle threads and cap must be kept free of any picric acid (either in solution or crystalized).
- Picric acid is highly reactive with a wide variety of materials (e.g., copper, zinc, lead, salts, concrete, ammonia, etc.) and extremely susceptible to the formation of picrate salts. Many of these salts are even more reactive and shock sensitive than the acid itself.
- Before closing a container of picric acid, the lid of the bottle and the threaded portion of bottle must be cleaned with a wet cloth(s) before resealing. The cap of the container must not be metallic.
- *Picric Acid and Picric containing compounds cannot be used with any metals: i.e. lids, spatulas, work benches etc.*
6. Procedure

6.1 Weighing Picric Acid:

- Must be conducted in a fume hood – with a NON-metallic bench top.
- If the balance has a metal weighing surface it must be covered with a non-metallic and non-porous material.
- Weigh boats must be plastic.
- Spatulas must be plastic or glass.

1. Place a small piece of filter paper on top of some paper towels inside the fume hood.
2. Place a small quantity of wet picric acid onto the filter paper.
3. Allow some of the moisture to drain (for a few minutes at most).
4. While the Picric Acid is still damp:
   a. Weigh the picric acid in a plastic weigh boat.
   b. Pour or spoon the picric acid into the solvent being used.
   c. NOTE: The picric acid should always be damp and NEVER allowed to DRY.
5. Triple rinse the spatula and weigh boat with water, collecting the rinse with a properly labelled and sized chemical waste container. The triple rinsed spatula and weigh boat can then be thrown in the regular garbage (ensure they are fully clean).
6. Collect the filter paper and paper towels underneath it and place in a waste disposal container (A sealable plastic bag is fine), that has enough water in it to keep the materials from drying out (it doesn’t have to be filled with water, just a small amount to keep the moisture content higher than 10%). Seal the bag fully to keep the moisture content in the bag high.
7. Wipe the cap and threads of the original Picric Acid Bottle with a damp wipe to ensure no deposition has occurred, dispose of the wipe in the same bag as the filter paper and repeat.
8. Ensure that the water level in the picric acid bottle is sufficient, if not add some distilled water.
9. Seal the Picric Acid Bottle with Parafilm and then replace the cap.
10. Enter the usage and inspection on the log sheet provided.
11. Wet a small paper towel or absorbent cloth with water and wipe the surface underneath where the filter paper was, as well as the balance and the path between the filter paper, balance and solvent container. Place the paper towel in the labelled solid waste container (sealable plastic bag) used in step 6.
12. Repeat step 11 two more times, for a total of 3 wipe downs.
13. Wipe the etchant bottle’s threads to ensure no picric acid has been deposited and place the wipe in the solid waste disposal container (sealable plastic bag) used in step 6.
14. Seal the etchant with Parafilm and replace the cap.
15. Survey the area to ensure no contamination has occurred.
16. Put the Picric Acid away in its secondary containment.
17. Picric Acid waste must be disposed of weekly to prevent drying out, if any waste (solid or liquid is generated) fill out the appropriate waste disposal forms and arrange for pick up.
6.1.1 Cleaning glassware, labware and other utensils

- Please see Mat0001 and Mat0002 for detailed information on general lab cleaning.
- NOTE: All chemical waste must be collected (and not allowed to go down the drain or in a garbage). For Picric Acid special care must be taken to ensure that all articles (spatulas, wipes etc. remain wetted at all times).
- NOTE: Parafilm cannot be cleaned, so it must be disposed of as contaminated waste.

6.2 Etching with Picric Acid

Etching with Picric acid containing etchants should be conducted in accordance with Mat0005 for submersion etching, with the additional information in this SOP used to ensure no dry picric acid occurs, and that waste disposal is completed in an appropriate manner.

- Any solution containing Picric Acid must be used in a fume hood with a NON-Metallic bench top.
- After etching is completed, the area (fume hood bench top) must be wiped down, and the etchant bottle threads cleaned as outlined in section 6.0, collecting the waste as described in section 6.0.
- After etching with picric acid the etchant and rinse containers must be triple rinsed with water (and the waste collected), and then triple wiped with a damp cloth (inside and out), with the wipes being collected as hazardous waste as described in section 6.0.

6.3 Labeling and Storing of Picric Acid

6.3.1 Labeling of Picric Acid

- Please see Mat0001 and Mat0002 for detailed information on labelling, Picric Acid and all etchants must be labelled in accordance with University and WHMIS regulations.
- In addition to the regular labeling requirements all Picric Acid and Picric Acid containing solutions must in addition have a label with the date of last opening (along with the person who used the bottle). Figure 1 shows a sample of these labels, these labels must be stuck to each bottle containing Picric Acid.

<table>
<thead>
<tr>
<th>Date</th>
<th>User</th>
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<tr>
<td>Sept. 10, 2012</td>
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<td>Yes</td>
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<td>Jane Doe</td>
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6.3.2 Storage of Picric Acid

- Picric acid must be stored in a flammable liquids storage cabinet, inside its locked cabinet. With the log on the bottle filled out each time it is used.
- All Picric acid containing bottles should be sealed with Parafilm and then the threaded cap put on the container. Any used Parafilm must be disposed of as hazardous waste, contaminated with trace Picric acid.
- Picric acid and its derivatives containers should be inspected monthly for evidence of crystallization or leaks, and the water level replenished (with log entry).
- All picric acid containing solutions must be stored in secondary containment (plastic).
- Dry picric acid or picrate salts should not be touched or moved under any circumstances. If there are crystals either in the bottle or on the thread of the bottle immediately contact waste management.
- When working with picric acid ETCHANTS, the etchants cannot be stored for more than 7 days.

6.4 Picric Acid Spills

- If spilled outside a chemical fume hood, the spill must be cleaned by Chemical Safety personnel.
  1. Evacuate the area and close the doors - restrict access to area and post a sign to prevent others from entering;
  2. Call WSEP immediately.

- If spilled inside a chemical fume hood, the spill can be cleaned by laboratory staff if they have the correct equipment, understand the hazards, know how to clean up the spill safely and dispose of the waste properly:
  1. Do Not allow the spill to get to a drain, use a sorbent pad to block drain access.
  2. Dampen some paper towel or sorbent pad and wipe up the spill, collecting the paper towel as hazardous waste.
  3. Continue to wipe the area with new paper towels (wet) until no picric acid is present.
  4. After you fill the area is clean of picric acid, triple wipe with wet paper towel and collect the waste.
  5. Remove the drain barrier and triple wipe that area with damp paper towel, collecting it as hazardous waste.
  6. Fill out in incident report.

- NOTE: if you suspect any picric acid may have made it to a drain, contact WSEP as soon as possible so that they can handle the situation.

- After two years of initial receipt, it is necessary to dispose of the original picric acid container as a hazardous waste.
- For more information on general lab safety, please see Mat0001 and Mat0002.
6.5 Hazardous Waste Disposal Procedures

1. Follow all labeling and disposal procedures found in Mat0001 and Mat0002.
2. Excess picric acid and all waste material containing picric acid (such as etchants) must be placed in a container with a screw cap which is stored in secondary containment and disposed through Department of Health, Safety & Environment. The container’s contents must be kept wet.
3. All of the contaminant substances must be disposed on Friday of each week before 2:30 PM through the Department of Health, Safety & Environment of University of Saskatchewan. For more details, please see Mat0001.

7. Equipment or Materials Required

1. WHMIS workplace labels
2. Lab coat, disposable nitrile gloves, goggles, safety glasses, face shield, fume hood, safety shower.
3. Chemical Waste Disposal Labels
4. Substance Classification Sticker (colored Dot)
5. Proper containers must have a screw cap and they must be less than 10 Litres in volume for liquid storage.
6. Plastic bags
7. Paper towel
8. Sorbent pads
9. Marker and/or pen

8. Regulatory / Standards

- Standard Operating Procedures # Mat0001, Mat0002 and Mat0005.

9. References

University of Saskatchewan, Department of Mechanical Engineering, Standard Operating Procedure # Mat0001, Mat0002 and MAT0005

Information on Picric Acid, Environmental Health & Safety Stanford University

Guidelines for the Safe Use of Picric Acid, Environmental of health and Safety, Mcgill University.