ENVIRONMENT, HEALTH, & SAFETY

Laboratory Standard Operating Procedure for:

Aqua Regia

Principal Investigator (PI) Approval is Required Prior to Performing this Procedure

Description

This standard operating procedure outlines the handling and use of aqua regia. Review this document and supply the information required in order to make it specific to your laboratory. In accordance with this document, laboratories should use appropriate controls, personal protective equipment, and disposal techniques when handling aqua regia.

Aqua regia is a highly corrosive mixture of acids. It can be used to clean glassware since it dissolves any metal and trace organic compounds. Aqua regia is also used to remove noble metals such as gold, platinum and palladium from substrates.

Aqua regia must be prepared and used in a fume hood. To prepare, combine three (3) parts of hydrochloric acid to one (1) part nitric acid in a glass container. The hydrochloric acid must be measured into the glassware first, and then the nitric acid is *slowly* added.

- Never exceed 38% nitric acid.
- Never use plastic or metal containers to prepare or use aqua regia.

Potential Hazards

- Aqua regia has many potential physical and health hazards. A less hazardous solution/process should be used if possible.
- Aqua regia is an oxidizer. Oxidizers are agents that initiate or promote combustion in other materials, generally through the release of oxygen.
- Aqua regia will oxidize over time to form toxic gases (nitrosyl chloride, nitrogen dioxide, and chlorine).
- Aqua regia solution is very energetic, very likely to become hot (> 100C) and potentially explosive.
- Aqua regia solutions are extremely corrosive. Corrosive materials can cause destruction of living tissue by chemical action at the site of contact and can be solids, liquids, or gases. Corrosive effects can occur not only on the skin and eyes, but also in the respiratory tract and, in the case of ingestion, in the gastrointestinal tract as well.
- If aqua regia is not handled properly, an explosion, skin burns, or eye/respiratory tract irritation may result.

Concentrated nitric acid is considered to be a Particularly Hazardous Substance (PHS) due to its acute toxicity and reactivity. The MIOSHA 8-hour Permissible Exposure Limit (PEL-TWA) for nitric acid is 2 ppm and 4 ppm for a 15-minute short-term exposure limit (STEL).

The toxic gases formed as aqua regia oxidizes – nitrosyl chloride, nitrogen dioxide, and chlorine – are also PHSs. Nitrosyl Chloride does not have a PEL. Nitrogen dioxide has a PEL of 1 ppm (STEL). The PEL for Chlorine gas is 0.5 ppm (PEL-TWA) and 1.0 ppm (STEL).

Engineering Controls

An eyewash and safety shower must be available in the immediate work area for any work with aqua regia.

When working with aqua regia, always work in a clean fume hood that contains NO organic material and keep the sash down while reactions are in progress.

Work Practice Controls

- Set up a designated area for storage and work with aqua regia.
- Only prepare the amount you need for immediate use and follow the instructions for dilution and neutralization of unused solution.
- Work with the smallest practicable amount of aqua regia needed to perform your task.
- Never put aqua regia in a closed vessel; evolved gases will cause a pressure build-up and possible explosion.
- Never take aqua regia out of the fume hood.
- Once work with aqua regia is complete, wipe down the area with a soap and water solution.

When working with aqua regia you must have specific HazMat absorbent pads or pillows that can be used on nitric acid, and these must be available in the immediate work area. These absorbent pads will be needed for clean-up in case of a small spill inside of the fume hood.

Protective Equipment (PPE)

Goggles, lab coat, chemical-resistant gloves (18 mil neoprene, Silver Shield, or any other glove rated to protect against hydrochloric AND nitric acid).

Face shield and acid-resistant apron are recommended if working with a larger volume (>200ml).

Transportation and Storage

- Never store aqua regia for later use; only make enough for immediate use.
- Never put aqua regia in an organic solvent cabinet; it is a strong oxidizer and incompatible with organic chemicals/solvents and any reducing agents.
- Never put a container of aqua regia near flammables or combustibles.
- Ensure primary and secondary containment is free from organic chemicals/solvents.

Waste Disposal

Dilution and Neutralization: before disposing of waste, dilute and neutralize following the steps below.

1. Use secondary containment for the container in which aqua regia waste will be neutralized. (The secondary containment must be free from all organic chemicals/solvents).
2. The neutralization container should be a glass beaker and must never be more than 2/3 full (even after the dilution is complete). See below to determine the required size of beaker.
3. Before diluting the aqua regia, calculate the total volume of water needed. This should be approximately a 7.5x dilution (e.g. 3 L of water would be needed to dilute 400ml of aqua regia).
4. Calculate the amount of magnesium hydroxide needed. This should be 0.533 g per ml of aqua regia (e.g. 213 g of magnesium hydroxide would be needed to neutralize 400 ml of aqua regia).

5. Inside of a fume hood, add the total volume of water to a glass beaker (use secondary containment), keeping in mind that the beaker should be no more than 1/2-2/3 full of water. Place the beaker on a stir-plate and add a stir-bar to keep the solution well mixed.
6. Add the magnesium hydroxide and a dash of bromothymol blue solution to the beaker. For bromothymol blue (BB) preparation: add 0.8g BB to 100 ml of water and a small drop of sodium hydroxide.
7. SLOWLY add the aqua regia to the full volume of water, carefully avoiding overheating. The bromothymol blue indicator will turn yellow if you overshoot neutral pH. If your solution turns yellow, but there is still undissolved magnesium hydroxide, let the solution stir longer to equilibrate. Test the pH using a pH strip, and add more magnesium hydroxide if necessary. (pH must be between 6 and 9 for waste pickup.)
8. Allow solution to cool to room temperature before moving the container, capping the container, or transferring the solution.

Disposal: Obtain piranha waste bottles and piranha venting caps from Environment, Health and Safety (EHS) at (810) 766-6763. Be sure the waste bottles and caps are present in the lab before you prepare any aqua regia. Prior to adding any waste to the bottle, place a completed hazardous waste label on it. Be sure to put the accumulation start date on the label when you begin adding waste.

Exposures/Unintended Contact

 ***If the employee is in need of emergency medical attention, call 911 immediately.*** 

For an actual chemical exposure/injury:

- Flush exposed eyes or skin with water for at least 15 minutes, then seek medical attention (see below).
- Note: In case of inhalation, symptoms may be delayed up to 24 hours
- If swallowed, DO NOT INDUCE VOMITING. Never give anything by mouth to an unconscious person. Wash mouth with water, give plenty of water slowly to drink and ***obtain urgent medical attention***.

Contact EHS for advice on symptoms of chemical exposure, or assistance in performing an exposure assessment.

Report all work related accidents, injuries, illnesses or exposures to UM-Flint DPS. Additionally, employees and supervisors must be sure to report the injury to EHS and complete and submit the Illness and Injury Report Form to WorkConnections within 24 hours. Follow the directions on the WorkConnections website Forms Instructions to obtain proper medical treatment and follow-up.

If you were involved in or observed an incident or near miss, please complete the EHS Laboratory Incident and Near-Miss Report Form. This will be valuable in improving laboratory safety on UM-Flint campus.

TREATMENT FACILITIES:

MAJOR INJURIES	MINOR INJURIES –During Business Hours	MINOR INJURIES –After Business Hours
Genesys Hospital One Genesys Parkway Grand Blanc, MI 48439 (810) 606-5710 **Hurley Medical Center** One Hurley Plaza Flint, MI 48503 (810) 262-9000 **McLaren Hospital Flint** 401 South Ballenger Hwy Flint, MI 48532 (810) 342-2000	**Genesys Occupational Health Network** 1460 Center Rd. Burton, MI 48509 (810) 715-4620 Mon. to Fri. 7:30 am to 10 pm Sat. & Sun. Noon to 8 pm **McLaren Flint-Burton OCC Center** 1459 S. Center Rd. Burton, MI 48509 (810) 496-0900 Mon. - Fri. 8 am to 8 pm Sat & Sun 10 am to 2 pm	**Downtown Flint** 420 S. Saginaw St. Flint, MI 48502 (810) 762-1550 **Genesys East** 1096 S. Belsay Rd, Suite F Burton, MI 48509 (810) 743-3351 **Genesys North** 4154 W. Vienna Rd Clio, MI 48420 (810) 686-7397 **Genesys South** 8447 N. Holly Rd Grand Blanc, MI 48439 (810) 603-0856 Mon. - Fri. 6 to 10pm / Sat. & Sun. 1-10pm

Click here for more information on the UM – Flint Emergency Preparedness and Response Plan.

Spill Procedure

- When a spill occurs, ***personal safety should always come first***.
- Alert and clear everyone in the immediate area where the spill occurred.
- Use proper personal protective equipment as indicated above.
- Collect residue, place in container and contact EHS at (810) 766-6763 for proper disposal.

If less than 200 ml of spent solution (no longer evolving gases) is spilled in the fume hood, lab personnel can soak up spill with absorbent/neutralizer for acid spills. Dispose of absorbent material as solid hazardous waste. Wipe contaminated area again with soap and water solution.

If reactive solution (still evolving gases) is spilled in the fume hood, close the sash, leave the lab and call the UM-Flint Department of Public Safety (DPS) at 911 to request assistance from EHS. Do not take any action to cover the spill. Post a warning on the lab and do not allow others to enter. Have a person available that has knowledge of the incident and laboratory to assist emergency personnel.

If any aqua regia solution is spilled OUTSIDE of the fume hood, open the hood sash fully, leave the lab and call UM-Flint DPS at 911 to request assistance from EHS. Do not take any action to cover the spill. Post a warning on the lab and do not allow others to enter. Have a person available that has knowledge of the incident and laboratory to assist emergency personnel.

Additional Spill Response Steps:

MAJOR CHEMICAL SPILL

- Attend to injured or contaminated persons and remove them from exposure.
- Alert people in the laboratory to evacuate.
- **Call UM-Flint DPS at *911* immediately from any university telephone for assistance or (810) 762-333 from any cell phone or non-university telephone.**
- Close doors to affected area.
- Post warnings to keep people from entering the area.
- Have person available that has knowledge of incident and laboratory to assist emergency personnel.

Additional Spill Links:

- www.oseh.umich.edu/pdf/chemspil.pdf
- http://www.oseh.umich.edu/emer-chemical.shtml.

Report all emergencies, suspicious activity, injuries, spills, and fires to the UM-Flint Department of Public Safety (DPS) at 911 from any university telephone or (810) 762-3333 from cell phone or non-university telephone. Register with the University of Michigan-Flint Emergency Alert System via Wolverine Access. Also, preprogram the UM-Flint DPS telephone number (810) 762-3333 into your cell phone for quick, easy use.

Training of Personnel

All personnel are required to complete Laboratory Safety Training. Documentation of the training is required. This training can be accomplished by completing the ***Comprehensive Laboratory Safety*** session (**BLS009** *or equivalent*) via MyLINC, or UM-Flint EHS on-line training or other equivalent approved by EHS. Furthermore, all personnel shall read and fully adhere to this SOP when handling aqua regia.

Certification

I have read and understand the above SOP. I agree to contact my Supervisor or Lab Manager if I plan to modify this procedure.

Name	Signature	UM ID #	Date

Principal Investigator _____ **Revision Date** _____