

STANDARD OPERATING PROCEDURE

Procedure Title	Aqua Regia (King acid)
Procedure Author	Angel T. Garcia-Esparza
Date of Creation/Revision	2025-07-10
Name of Responsible Person (The PI, Lab Supervisor, or Autonomous Researcher)	Angel T. Garcia-Esparza
Location to be Performed (building/lab #, beam lines)	SSRL B131, Rms. 209; Bldg 120, Rms. 257
Proposal #s:	
#1	Process or Experiment Description: Briefly summarize the process or experiment, including an estimate of how long the process takes and how frequently it will be conducted. Include total quantities (volume, mass) of the materials you to expect to use. Preparation of cleaning agent: Aqua Regia. Aqua Regia is a 3:1 mixture of concentrated hydrochloric acid (HCl, $\leq 37\%$) and concentrated nitric acid (HNO_3, $\leq 70\%$). The preparation procedure is conducted only once at the beginning of the experiment to clean the substrates and electrodes. The precursor acids are handled with glass pipets. The cleaning agents are contained in 20 ml glass vials placed in secondary container (do not use plastic containers for Aqua Regia). The final volume is always equal or less than 20 ml. For example, Aqua Regia is made of 6 ml hydrochloric acid and 2 ml nitric acid (total 8 ml). Aqua Regia is a corrosive, fuming yellow liquid. It is used to remove a wide variety of inorganic contaminants and dissolve metals. Its fumes and color are caused by reaction of nitric acid, HNO_3, with hydrogen chloride, HCl, to form nitrosyl chloride (NOCl), chlorine (Cl_2), nitric oxide (NO), and nitrogen dioxide (NO_2) and water; where chlorine and nitrosyl chloride are yellow-colored, and volatile. These gases are toxic inhalation hazards. The cap is never to be tighten completely because pressure can build up due to gas evolution within the container leading to explosive failure of the glass vessel. Only use the 20 ml volume for one-time usage.
#2	Risk Assessment: Identify potential safety hazards. For chemical hazards, be specific (e.g. flammability, corrosivity, reactivity/explosion, acute toxicity, or carcinogenicity). List OSHA Hazards, NPFA ratings, and occupational exposure limits.
Hydrochloric Acid Corrosive to Metals (Category 1), H290; Skin corrosion (Category 1B), H314; Serious eye damage (Category 1), H318; Specific target organ toxicity - single	

exposure (Category 3), Respiratory system, H335

- Workplace exposure limit basis USA. ACGIH Threshold Limit Values (TLV) – 2 ppm
- NFPA Ratings: Health – 3; Flammability – 0; Instability – 1; None

Nitric acid

- Oxidizing liquids (Category 3), H272; Corrosive to Metals (Category 1), H290; Acute toxicity, Inhalation (Category 3), H331; Skin corrosion (Category 1A), H314; Serious eye damage (Category 1), H318
- Workplace exposure limit basis USA. ACGIH Threshold Limit Values (TLV) – 2 ppm
- NFPA Ratings: Health – 4; Flammability – 0; Instability – 1; Oxidizer

Aqua Regia

- Fuming liquid – must be stored in fume hood in a vented container
- Corrosive to Metals (Category 1), H290; Skin corrosion (Category 1A), H314; Serious eye damage (Category 1), H318; Acute Toxicity (Category 4), H303; Oxidizing liquids (Category 3), H272
- Workplace exposure limit basis USA. ACGIH Threshold Limit Values (TLV) – 2 ppm
- NFPA Ratings: Health – 3; Flammability – 0; Instability – 0; Oxidizer

#3

Safety Equipment: *Specify all equipment needed to safely perform research or experiment.*

#3a Engineering/Ventilation Controls: *fume hood use, glass secondary container, and absorbent bench paper.*

All work with Aqua Regia must be performed inside a chemical fume hood. The fume hood sash shall be kept as low as possible and should be positioned between the researcher and the Aqua Regia solution to minimize the chance of splashing.

#3b Personal Protective Equipment and Other Safety Equipment: *(e.g safety glasses, nitrile gloves, cryo gloves)*

All chemicals will only be handled with chemically-resistant butyl rubber or natural latex

gloves > 0.6 mm in thickness. Safety glasses or goggles, chemical splash resistant lab coat.

A warning sign, visible by any user working in the area, must be always posted to indicate the fume hood contains an Aqua Regia mixture.

#3c Location of Nearest Emergency Safety Equipment:

Item:	Location:
Eyewash/Safety Shower	Eyewash drench hoses are located, next to the sinks (B120, Rm 257, 258; B131, Rm 113, 209). An eyewash/facewash is located next to the water system, near the lab entrance (B120, Rm 257). Safety showers are located in front of the sink (B131, Rm 113), near the water system and cold room entrance (B131, Rm 209), and near the lab entrance (B120, Rm 257).
First Aid Kit	First aid kits are located at 4 Safety Stations around the experimental floor, near BL2-2, 9-3, 6-2, and 4-2. Contact SLAC Medical (x2281) or Security (x5555) for non-life threatening injuries. Call 911 for life threatening situations.
Chemical Spill Kit	Spill kits are located near the sinks (B120, Rm 257, 258), underneath the bench tops (B131, Rm 113), or next to the entrance to the lab (B131, Rm 209).
Fire Extinguisher	Fire extinguishers are located near the lab entrance (B120, Rm 257, 258; B131, Rm 209) or just outside the laboratory door (B120, Rm 260; B131, Rm 113).
Telephone	Telephones are located near the entrance to the labs.
Fire Alarm Manual Pull Station	A fire alarm pull station is located outside the laboratory, on the outside stairway door frame between rooms 252A& 254 (B120) or near the building exit door, between beam lines 11-2 and 4-3 (B131).
Safety Stations	Safety Stations around the experimental floor, near BL2-1, 9-3, 6-2, and 4-2. Contact SLAC Medical (x2281) or Security (x5555) for non-life threatening injuries. Call 911 for life threatening situations.

#4	Shipping and Receiving Requirements: <i>Describe shipping or receiving requirements, especially for highly toxic, highly reactive/ unstable materials, highly flammables, and corrosives. .</i>
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Follow standard SSRL shipping and receiving protocols.

#5 **Designated Area:** *Where highly toxic materials,, highly flammable/pyrophoric, highly corrosive, reactive/unstable, or nanomaterials are used, identify the designated work area(s), and the necessary personnel decontamination after completion of work.*

Fume hood in B.131 WET LAB 209 or fume hood in B.120 WET LAB 257. Dispose gloves and contaminated equipment following the established hazardous waste guidelines.

#6 **Step-By-Step Operating Procedure:** *Provide a sequential description of work, including details such as chemical concentrations and when special safety equipment is to be utilized. Include temperature, pressure, and other experimental conditions. Schematics, or pictures are suggested for complex set ups.*

- Be familiar with the location and content of any applicable Safety Data Sheets (SDSs) for the chemicals to be used.
- Implement good laboratory practices, including good workspace hygiene.
- Aqua Regia must only be used in a room with a properly functioning eyewash. A safety shower is available within 10 s of travel.
- Do not remove Aqua Regia from the fume hood where it was made.
- Never place Aqua Regia in a sealed container as glass failure can occur due to gas generation and consequential over pressurization.
- Whenever preparing/handling Aqua Regia, glass containers (preferably borosilicate) should be used. Secondary containment (e.g., glass, polypropylene or polyethylene tray) shall be used for all Aqua Regia work.
- Only prepare the amount you need for immediate use (≤ 20 ml). Work with the smallest practicable amount of Aqua Regia needed to perform the task.
- Place all required materials such as glass pipets and glass vials inside the fume hood prior to handling the concentrated acids.
- Place concentrated acids bottles and the Aqua Regia vessel in secondary containers (the secondary containment must be free from organic chemicals/solvents).
- **ALWAYS add nitric acid (HNO₃) to hydrochloric acid (HCl), never vice versa.**

Preparation Aqua Regia

1. Prepare aqua regia in a fume hood by slowly adding 1 volume equivalent of concentrated nitric acid to 3 volume equivalents of concentrated hydrochloric acid in an appropriately sized glass beaker. Before use, let the mixture stand for 1 or 2 minutes as the solution changes from colorless to golden.
2. Perform the addition slowly to prevent uncontrolled boiling and potential splashing.
3. For 16 ml, Aqua Regia is prepared by slowly 4 ml of concentrated HNO_3 to 12 ml of concentrated HCl in 20 ml glass vessel.
4. Upon mixing, the colorless solution quickly becomes hot and turns yellow in color, which successively darkens to an orange or red fuming solution over several minutes. The yellow-brown fumes contain NOCl , Cl_2 , NO , and NO_2 , which must not be inhaled.
5. Let the Aqua Regia 20-ml vessel rest and return the concentrated acid bottle as soon as practically possible to their respective corrosive cabinets to avoid having large volume acids in the fume hood at the same time.
6. The Aqua Regia solution is now ready for use, which must only occur in open or vented glass vessels. Once the solution returns to a yellow color and gas evolution ceases, Aqua Regia has lost its potency.
7. Transfer the Aqua Regia to the contaminated glassware to be cleaned inside secondary containment. Let the Aqua Regia rest on the contaminated glass.
8. For disposal, use secondary containment for the container of Aqua Regia waste (the secondary containment must be free from organic chemicals/solvents).
9. Allow solution to cool to room temperature before moving the container, capping the container with a vented cap.
10. Dispose the waste solution following hazardous waste disposal regulations.

#7

Special Handling Procedures, Transport & Storage Requirements: *Describe special handling and storage requirements for hazardous chemicals in your laboratory, especially for highly reactive/ unstable materials, highly flammables, and corrosives. Describe transport and secondary containment requirements, between the laboratory and beam lines or between facilities.*

Aqua Regia should only be used in the fume hood within secondary containment.

#8 **Beam Line Handling and Storage Requirements:** *Describe sample handling procedures and sampling set up at the beam lines. Are samples sealed or open? Is ventilation required? Are heating, cooling, or gas distribution systems present?*

Not to be handled at the beam line. Aqua Regia should only be used in the fume hood.

#9 **Emergency Procedures:** *Indicate how spills, personnel exposure/ injury, and other accidents should be handled and by whom. List emergency contact numbers.*

Safety Shower/Eye Shower:

Any serious chemical self-contamination.

Evacuate the Lab, and call 911 if:

1. Serious chemical self-contamination
2. Large hazardous material release.
3. Smoke, chemical fire, explosion.

Call x5555 if:

1. Non-life threatening injury
2. Non-life threatening chemical or radiation incident.

Additional Emergency Procedures:

#10 **Waste Disposal:** *Identify amounts of waste anticipated and appropriate disposal procedures.*

Waste will be segregated by hazard class (e.g. flammable, corrosive) and state (e.g. solid, liquid), appropriately labeled, and placed in the laboratory's hazardous waste cabinet.

Additional Waste Guidelines:

Allow solution to completely cool. Use vented caps only. Submit waste pick-up request.

#11 **Training Requirements:** *List the general and lab-specific trainings required*

Course 204, Sample Preparation Laboratory Training

Additional Training Requirements:

#12	Approval Required: <i>Identify any tasks that require prior approval by the PI/ Laboratory Supervisor (e.g., use of Restricted Chemicals and other higher hazard chemicals, and running of higher hazard operations). List Subject Matter Experts (SMEs) consulted for approval.</i>
Procedures must be approved by PI/Spokesperson, Laboratory Manager and Directorate ESH Coordinators.	
<u>Additional Approvers:</u> 	

Approved by: _____ Approval date: _____

Approved by: _____ Approval date: _____

Approved by: _____ Approval date: _____

Approved by: _____ Approval date: _____