

AQUA REGIA SAFETY GUIDELINES

Aqua Regia is widely used as an efficient etchant in microfabrications and microelectronic processing. Its popularity comes from its ability to dissolve noble metals such as Platinum (Pt), Palladium (Pd) and Gold (Au) towards its purification. In chemistry laboratories, this acidic solution is useful for the removal of stubborn organic and inorganic residues from substrates and glassware such as sintered funnels. This method is notably preferred over the more “traditional” chromic acid (CrVI) baths for cleaning NMR tubes as no traces of paramagnetic chromium will remain to later spoil spectra.

The most common Aqua Regia solution consists of a 3:1 *molar* ratio of concentrated Hydrochloric acid (HCl, 35-38%) and concentrated Nitric acid (HNO₃, 65-70%). Therefore, precautions should always be taken when handling this solution. **Anyone who works in laboratories where Aqua Regia solutions are used should familiarize themselves with the hazards related to the mixture and its individual components, and a clear Standard Operating Procedure (SOP) should be established.**

This document discusses the properties, health and safety hazards, how to properly handle and store (*only when necessary*) Aqua Regia solution. Also included are emergency procedures for dealing with accidental exposures to Aqua Regia solution, including first aid treatment information.

☠*WARNING*☠: Aqua Regia solution is VERY DANGEROUS! In addition to being a corrosive liquid and an extremely powerful oxidizer, there are many factors which will cause the reaction to accelerate out of control ranging from foaming out of its bin to an explosion with a huge shock wave with possible acid-gown shredding glass sharps. Aqua Regia can oxidize organic compounds. If you provide sufficient fuel for them (*i.e.* organic solvents, photoresist) they will generate enormous quantities of heat and gas.

1. Properties

Chemical name:	<i>Aqua Regia; Chloronitrous acid; Chloroazotic acid; Nitrohydrochloric acid, diluted; Nitromuriatic acid</i>
Solution:	The usual molar ratio between concentrated Hydrochloric acid and concentrated Nitric Acid is HCl:HNO ₃ is 3:1 . <u><i>This means the volume ratio is usually approx. 4 parts concentrated HCl to 1 part of concentrated HNO₃.</i></u> It is a corrosive and oxidizing liquid with toxic properties.
Appearance:	Freshly prepared, Aqua Regia solution is colorless, but it turns yellow/orange within seconds.
Odor:	Suffocating odor and a continuous release of toxic and corrosive vapours.
Chemical Formulas:	Hydrochloric acid: HCl Nitric acid: HNO ₃
CAS #:	Hydrochloric acid: 7647-01-0 Nitric acid: 7697-37-2

2. Hazard Classification

Hydrochloric acid (concentrated, 35-38%)

WHMIS 2015¹



- Corrosive to metals (Category 1)
- Skin Corrosion/Irritation (Category 1B)
- Serious Eye Damage/Eye Irritation (Category 1)
- Specific target organ toxicity (single exposure) Target Organs – Respiratory system (Category 3)

NFPA²



Flammability: Will not burn under typical fire conditions

Health hazard: Highly toxic; severe acute and chronic health effects

Instability/Reactivity: Normally stable but can become unstable at elevated temperatures and pressures

Nitric acid (concentrated, 65-70%)

WHMIS 2015³



- Oxidizing liquids (Category 3)
- Corrosive to metals (Category 1)
- Acute Inhalation Toxicity – Dusts and Mists (Category 3)
- Skin Corrosion/Irritation (Category 1A)
- Serious Eye Damage/Eye Irritation (Category 1)

NFPA⁴



Flammability: Will not burn under typical fire conditions

Health hazard: Can be lethal under emergency conditions

Instability/Reactivity: Normally stable, even under fire exposure conditions

Special: Possesses oxidizing properties

3. Reactivity and Explosion Hazards

ANY AQUA REGIA SOLUTION IS TO BE HANDLED WITH CARE !

Aqua Regia solution is very energetic, exothermic and potentially explosive. It is very likely to become hot (more than 100°C) when both acids are mixed or when the solution is used.

When preparing the Aqua Regia solution, **ALWAYS add the Nitric acid very slowly to the Hydrochloric acid.** For safe handling, never exceed 38% ratio of concentrated Nitric acid.

Upon mixing of both concentrated acids, chemical reaction results in volatile, toxic and corrosive products Nitrosyl chloride (NOCl) and Chlorine (Cl₂), as evidenced by the fuming nature and characteristic yellow/orange color of the resulting solution. As the volatile products escape from the solution, the Aqua Regia quickly loses its potency and the continuous gas evolution may lead to a build-up of pressure that could cause a sealed container to burst and fail catastrophically.

Aqua Regia solution reacts violently with any organic materials. Avoid mixing with incompatible materials such as acids, bases, organic solvents (acetone, ethyl and isopropyl alcohol), nylon, photoresist or detergents. Always ensure that all substrates are rinsed and dried before using Aqua Regia mixture, as even small amounts of organics could make the solution very unstable. Also, adding any acids or bases to Aqua Regia or spraying it with water will accelerate the exothermic reaction.

Only use clean glass or Pyrex containers. Aqua Regia solutions are not compatible with plastic or other metal containers. The solution will attack metals, often giving off the highly flammable hydrogen gas (H₂). The presence of a source of ignition can thus provoke the gas to burn and explosion can occur. Gown shredding glass sharps can quickly be generated in the event that an Aqua Regia container is maintained tightly closed. Both the user and surrounding people could be hurt should a tightly sealed container become overpressurized and explode.

4. Health Hazards

ANY AQUA REGIA SOLUTION IS TOXIC BY INHALATION AND BOTH VAPOURS AND LIQUID FORMS ARE HIGHLY CORROSIVE !

Aqua Regia solution and vapors emanating from it are extremely corrosive to the skin and respiratory tract. Note that Nitrosyl chloride quickly decomposes further into Nitric oxide (NO) and chlorine, generating the continuous presence of three toxic gases (NOCl, NO, and Cl₂) over the Aqua Regia solution. **These gases are acutely toxic by inhalation.**

Upon contact, both solution and vapors **can cause visible destruction to human tissue, and/or irreversible damage, particularly at high concentrations.** They can erode the skin and the respiratory epithelium through inhalation of vapours, with burning pain in the nose and throat, provoking coughing, wheezing, shortness of breath, and pulmonary oedema. They can be particularly damaging to the eyes, causing burns, irritation and may cause blindness. Note that additional toxicity can be generated by the contaminants present in spent solutions (Chromium species, noble metals ...).

Ingestion of Aqua Regia solution may burn the sensitive lining of the mouth, throat, perforate esophagus and stomach, and cause permanent damage to the digestive tract along with losing the ability to swallow.

5. Safety Precautions for Aqua Regia Solution Use

a) Training

Students and employees who handle Aqua Regia solutions must have received training on the hazards of this type of solution from their respective department (PI or supervisor). More particularly, successful completion of the following EHS-Safety trainings is required. Certifications must be kept up-to-date before any activity related to the production, handling, and waste management of Aqua Regia solution is conducted:

- ✓ WHMIS for Laboratory Personnel,
- ✓ Hazardous Waste Disposal for Laboratory Personnel,
- ✓ Corrosive Substances,
- ✓ Hazardous Materials Minor Spill Response,
- ✓ Safe Storage of Hazardous Materials.

Students and employees must know what to do in the event of a spill or an exposure incident. The SDS of the different components of the solution must always be kept within the immediate vicinity of the working area along with the Standard Operating Procedure (SOP) developed by the student / employee's department.

b) Ventilation / Fume Hood

Because highly corrosive and toxic vapors are generated when preparing Aqua Regia solutions, all work should be conducted **inside a certified chemical fume hood**. The sash should be positioned between the user and the Aqua Regia solution whenever practical to act as a physical barrier and minimize the chance of injuries from splashing.

c) Eye Protection

Safety goggles **and** a face shield **MUST** be worn when handling Aqua Regia solutions.

d) Gloves

Regular nitrile gloves do not provide sufficient protection from Aqua Regia. The double gloving approach must be considered: thicker, more protective reusable gloves worn over top of disposable gloves. Heavy duty neoprene, rubber gloves or any other gloves rated to protect against Hydrochloric AND Nitric acid must be worn. Compatibility charts from suppliers must be consulted. In the event resistant thicker gloves are used, a good dexterity must be maintained.

e) Protective Clothing

The handling of Aqua Regia solutions requires special personal protective equipment in addition to the standard laboratory clothing (lab coat). An acid-resistant Neoprene apron must be worn on top of the lab coat. Legs should also be covered by wearing a full body chemically resistant suit. Closed shoes must also be worn.

f) Safe Work Practices⁵

1. Always consider the use of less hazardous solutions or processes before using Aqua Regia solution.
2. Never use Aqua Regia solution alone or in isolation. Another knowledgeable person should be present in order to assist should an emergency situation occur.
3. Aqua Regia must only be used in a properly functioning fume hood and in a room with a properly functioning eyewash. A safety shower must be available within 10 seconds of travel.
4. Set up a designated clean area in the fume hood for work with Aqua Regia solution, with the appropriate warning signage.
5. Only prepare the amount needed for an immediate use. Work with the smallest practical amount of Aqua Regia solution needed to perform your task.
6. Never take Aqua Regia solution out of the fume hood in which it was prepared. In addition to the inherent vapors emanating from the solution, dissolving metals in this type of solution can also release toxic gases.
7. No other works (especially processes involving organic materials or flammable solvents) should be carried out in the fume hood whenever active Aqua Regia solution is present.
8. Never use plastic or metal containers to prepare or work with Aqua Regia. Glass (preferably Pyrex) containers must be used.
9. **Aqua Regia solutions should never be stored !**
10. Never put Aqua Regia solutions in closed vessels as the solution will continue to off gas and pressure will build-up, potentially resulting in explosion and/or bursting of containers. Gown shredding glass sharps can be generated and hurt surrounding people. **Vented-caps must be used on containers of Aqua Regia solution at all times.**
11. Always add Nitric acid very slowly to Hydrochloric acid. **Never vice versa.**
12. Never pour spent Aqua Regia solutions containing noble metals, chromium or other metals down the drain. Unused or metal-contaminated solutions, neutralized or not, must be considered Hazardous Waste and must be disposed of in accordance with Concordia University's hazardous waste guidelines and policies.
13. Do not use Aqua Regia solution to clean glassware that presents consistent amounts of contaminants. The removal of material in **TRACE amounts only** must be considered under the action of Aqua Regia solution.
14. Ensure containers and substrates are rinsed and properly dried before coming into contact with the Aqua Regia solution.
15. Perform all work in a compatible secondary container (*e.g.* Pyrex, polypropylene tray), free of any organic chemicals or solvents, to contain potential spills.
16. Leave the hot Aqua Regia solution in an open container until cooled down.
17. Never place a container of Aqua Regia near flammables or combustibles.

6. Storage, Spill and Waste Issues

a) Storage and Waste Handling

DO NOT STORE AQUA REGIA SOLUTIONS !

The primary hazard from storage of Aqua Regia solution is the potential for gas generation and over pressurization of the container when the solution is still hot. **If a hot solution of Aqua Regia is stored in an air tight container, it will explode !**

Accidents in laboratories involving the storage of Aqua Regia solutions in tightly capped plastic or Winchester bottles in flammable cabinets have been documented.⁶ Not only do those situations illustrate the explosion hazards this type of solution can represent, but also emphasize further consequences such as surrounding material degradation and failure, or the generation of toxic and corrosive fumes.

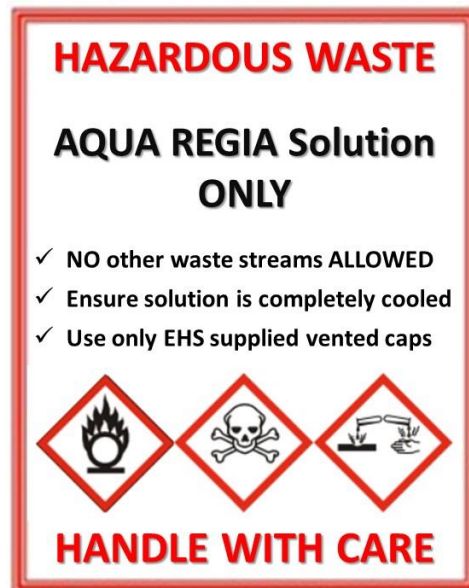
For best results and for safety reasons, make only small, fresh batches of Aqua Regia solution in compatible and open vessels before its use. Avoid having excess unused solution: 100mL represents a reasonable maximum quantity to be handled.

The spent or excess unused Aqua Regia solutions must be left in an open and properly identified container under the fume hood, displaying the appropriate warning signage. These solutions must be left under the fume hood for several hours, ideally overnight or over the week-end, to cool down to ambient temperature and to allow their full decomposition. It is the user's responsibility to make sure that the open container is clearly labeled and left in a safe area. Only once solutions have returned to a yellow color and are no longer evolving any gases, can adapted procedures be followed, depending on the type of contamination and treatment to operate.

1) Waste management of decomposed solution, without any further treatment.

The waste solution has to be poured into a dedicated waste container (ideally a clean and empty hydrochloric or nitric acid 4L-glass bottle) **equipped with a vented cap** or other mechanism to prevent the build-up of pressure. The container must be stored in a properly functioning fume hood, in an area free of any organic solvents or chemicals. The container must display the Concordia EHS Hazardous Waste label, with the name "Aqua Regia Solution", composition (Nitric acid, hydrochloric acid and other contaminants) and related hazards: Corrosive, Oxidizer and Toxic.

It must also include a very visible warning signage mentioning "**Aqua Regia waste only – DO NOT MIX WITH OTHER WASTE**". Only solutions generated from the same experiment should be present in the same waste container. Maintain a log of additions to Aqua Regia waste bottles, displaying dates, amounts, and swirling procedures (if applied).



Initially only add a small amount of the solution to the waste container to ensure there are no residual materials in the container that may cause an adverse reaction, realizing the adverse reaction may take minutes to hours to manifest. If no reaction is observed, continue to pour slowly. At least 25% of the waste container volume should be left empty.

Contact EHS (hazardouswaste@concordia.ca) for the waste pick-up that will be performed at least 3 days after having considered cleared for disposal.

2) Neutralization of Aqua Regia solutions

Aqua Regia solutions can be neutralized safely, provided appropriate PPE are worn and all the material for the neutralization is available. Although different procedures involving the cautious use of basic solutions are described, the most popular procedures share some common safety precautions :

- ✓ Neutralization must always be performed **in a fume hood, in a clean area**.
- ✓ Use **secondary containment**, free of any organic chemical or solvent, underneath the container in which Aqua Regia waste will be treated.
- ✓ The neutralization container should be a **glass beaker** and must never be more than **1/2 or 2/3 full** (even after the dilution is complete).
- ✓ Before diluting the Aqua Regia, **calculate the total volume of water or ice needed**.
- ✓ The beaker in which neutralization will take place must be placed on a stir-plate and a magnetic stir-bar is added before incorporating the Aqua Regia solution to keep **the solution well mixed** during the process.

A) *This procedure is adapted from the one described by University of Illinois Urbana-Champaign – Division of Research Safety.*⁷

- In a fume hood, very carefully pour leftovers or waste Aqua Regia into a beaker, on top a stir-plate and a secondary containment, containing a large quantity of ice (approx. 500 grams of ice per 50 mL of Aqua Regia) and a magnetic stir-bar.
- Neutralize the mixture with the slow addition of an aqueous basic solution, while maintaining a slow stirring to avoid spatters outside the beaker. The basic solution such as **1M or 10% Sodium Hydroxide (NaOH) or saturated Sodium Bicarbonate (NaHCO₃) in water**, is added dropwise with the help of a Pasteur pipette until pH is neutral (measured with a pH strip) or a slightly basic solution is obtained. **Be careful, the addition of NaHCO₃ solution will generate foam !**
- Allow solution to cool to room temperature before moving the container, capping the container, or transferring.

B) *This procedure is adapted from University of Michigan-Flint – Environment, Health & Safety department,*⁸ *and École Polytechnique Fédérale de Lausanne – Faculté des Sciences de la Vie.*⁹

- Inside of a fume hood, add the calculated volume of water (approx.. 7.5-10× dilution) 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 at all times.

- Add the calculated amount of [Magnesium Hydroxide \(Mg\(OH\)₂\)](#) (should be 0.533g per mL of **Aqua Regia**) and a dash of Bromothymol Blue (BB) solution to the beaker. *For BB preparation: add 0.8g BB to 100 mL of water and a small drop of Sodium Hydroxide.*
- Then, SLOWLY add the Aqua Regia to the full volume of water, carefully avoiding overheating and spatters out of the beaker. The BB indicator will turn yellow if you overshoot neutral pH. If your solution turns yellow, but there is still undissolved Mg(OH)₂, let the solution stir longer to equilibrate.
- Test the pH using a pH strip, and add more Mg(OH)₂ if necessary. (pH must be between 6 and 9 for waste pickup).
- Allow solution to cool to room temperature before moving the container, capping the container, or transferring.

The neutralized mixtures generated from both procedures, whether containing heavy metals or not (e.g. gold, platinum, lead, chromium) will be disposed of through the Concordia University Hazardous Waste management system.

b) Spills

Only employees trained in the handling of Aqua Regia solutions should clean up spills.

In the event of a small/minor spill (<100 mL inside the fume hood, with a controlled environment):

1. Notify personnel in the immediate vicinity to stay away from area.
2. Wear appropriate PPE to clean up the spill.
3. If a spill kit is present, use acid neutralizing material to neutralize Aqua Regia solution and yellow HazMat absorbing pads. Test with litmus paper or colour-indicating solution until the spilled material is within the neutral range of pH 6-8.
4. Clean the area with inert absorbent materials (vermiculite, dry sand, oil-sorb, or kitty litter). The area should be soaked with detergent, then rinsed with water. Discard contaminated cleaning materials in hazardous waste containers and clearly label them indicating they contain Aqua Regia-contaminated materials.
5. Advise your supervisor and complete an Injury/Near-miss report (EHS-FORM-042).

In the event of a large spill (>100 mL) located outside a chemical fume hood:

1. Advise and warn co-workers.
2. Evacuate the area immediately.
3. Restrict access to the area.
4. Notify Security at **514 848-3717** or internal extension **3717**, providing them with the following information:
 - a. Location of the spill
 - b. Name of hazardous material
 - c. Quantity involved
 - d. Related health risks and precautions to be taken

5. Provide the Safety Data Sheets (SDS) or appropriate documentation.
6. Advise your supervisor and complete an Injury/Near-miss report.

7. Emergency Procedures

a) Skin Contact

1. Immediately direct the exposed person towards the emergency shower and wash the affected area with large amounts of water until all evidence of the chemical has been removed (15 minutes minimum). Remove contaminated clothing.
2. Call Security at **514 848-3717** or internal extension **3717** for emergency medical assistance.

b) Eye Contact

1. Immediately direct the exposed person towards the emergency eyewash station and wash the affected eye with large amounts of water until all evidence of the chemical has been removed (15 minutes minimum).
2. Do not allow the victim to rub or keep eyes closed.
3. Call Security at **514 848-3717** or internal extension **3717** for emergency medical assistance.

c) Inhalation

1. Immediately move the victim to fresh air.
2. Call Security at **514 848-3717** or internal extension **3717** and ask for medical assistance. Seek medical attention in the event of respiratory irritation, cough, or tightness in the chest. Symptoms may be delayed.

d) Ingestion

Not a likely route of exposure. Call Security at **514 848-3717** or internal extension **3717** and ask for medical assistance. Seek medical attention.

In all cases of exposures, a copy of the Safety Data Sheet (SDS) of the different components must be brought to the emergency room as the treating physician might be unaware of the treatment measures for Aqua Regia solution. All Aqua Regia solution-related incidents must be reported to your Supervisor and to Environmental Health & Safety. An [injury/near-miss report](#) must be completed and submitted to EHS as well, in the event of a spill or a direct exposure.

If you have any concerns or questions about the use of Aqua Regia solution at Concordia University, please contact EHS at ehs@concordia.ca.

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References:

1. Fisher Scientific, Safety Data Sheet of Hydrochloric Acid (concentrated, 35-38%), revision date: 25/04/2019.
2. [Hydrochloric Acid, Solution - CAMEO Chemicals](#), consulted on March 22, 2021.
3. Fisher Scientific, Safety Data Sheet of Nitric Acid (concentrated, 65-70%), revision date: 25/04/2019.
4. [Nitric Acid, Solution \(other than red fuming\) - CAMEO Chemicals](#), consulted on March 22, 2021.
5. a) Princeton University – Office of Environmental Health & Safety – Chemical Safety - [Aqua Regia](#), consulted on March 21, 2021; b) Harvard Environmental Health & Safety – [Aqua Regia Laboratory Safety Guideline](#), consulted on March 21, 2021.
6. Using mixtures of highly corrosive concentrated acids – Ex: Aqua Regia and Piranha solution: https://umanitoba.ca/admin/vp_admin/risk_management/ehso/media/Concentratedacidmixtures.pdf, consulted on March 19, 2021.
7. University of Illinois Urbana-Champaign – Division of Research Safety – [Aqua Regia](#), consulted on March 19, 2021.
8. University of Michigan-Flint – Office of Environment, Health & Safety department – [Laboratory SOP for Aqua Regia](#), consulted on March 19, 2021.
9. École Polytechnique Fédérale de Lausanne – Faculté des Sciences de la Vie – [Guidelines for Aqua Regia Waste Disposal](#), consulted on March 19, 2021.