

Aqua Regia

Standard Operating Procedure

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1. Purpose and application

Aqua regia is a corrosive, fuming yellow solution that is a 1:3 mixture of concentrated nitric and hydrochloric acids. It is commonly used to remove noble metals such as gold, platinum and palladium from substrates.

The etch rate of aqua regia for gold is approximately 10 $\mu\text{m}/\text{min}$ (at room temperature) and can be increased to several 10 $\mu\text{m}/\text{min}$ at elevated temperatures. Palladium, aluminium, copper and molybdenum are also etched by aqua regia. For etching platinum or rhodium, the etching solution has to be heated to attain a reasonable etch rate. Etching of iridium requires strongly heated (boiling) aqua regia [1].

It may also be used to wash glassware in order to remove traces amounts of organic compounds. Its fumes and yellow colour are caused by reaction of nitric acid (HNO_3), with hydrogen chloride (HCl) to form nitrosyl chloride (NOCl), chlorine (Cl_2), and water; both chlorine and nitrosyl chloride are yellow-coloured and volatile [2].

Only fresh solution should be used. If stored, it will quickly loses its effectiveness due to oxidation of its reactive components.

2. Equipment, Chemicals and Supplies

Aqua regia (1:3) is composed out of two chemicals:

1. Concentrated nitric acid (65 %)
2. Hydrochloric acid (37 %)

Concentrated nitric (65 %) is purchased from Sigma Aldrich.

<https://www.sigmaaldrich.com/NL/en/product/sial/30709m>

Hydrochloric acid (37%) is purchased from Sigma Aldrich.

<https://www.sigmaaldrich.com/NL/en/product/sial/30721m>

3. Personal Protective Equipment (PPE)

The following equipment should be used:

- Eye protection: Safety glasses and face shield required.
- Protective gloves: Black neoprene gloves. Check gloves for leaks before use.
- Protective clothing or equipment: Apron.

4. Operational Procedures

1. Get three glass beakers or envelopes which will fit your sample and place them in the bench.
2. Write down your name and type of chemical for every beaker/envelope.
3. Fill two beaker/envelopes with DI water such that it will cover your sample. DI water is used for rinsing the etchant.
4. Carefully add the nitric acid to the hydrochloric acid in the last beaker/envelope such that it will cover your sample. Aqua regia solution is very energetic and potentially explosive. It is very likely to become hot (up to 100 °C). **Handle with care!**
5. If you plan to heat the etch (recommended) set up an au bain-marie with a hotplate in the hood. Put the beaker/envelope filled with etchant in the au bain-marie (use a holder when you use an envelope) to bring it up to temperature. Use an temperature sensor housed within a glass enclosure to measure the temperature of the etchant. **Direct heating of inorganic mixtures at temperatures higher than 80 °C is only allowed in day time and only after personal approval of the set-up by the KN staff.**
6. Calculate the etch time for your sample. You will need to know the thickness of your deposited layer. The etch rate of aqua regia for gold is approximately 10 µm/min at room temperature and can be increased to several 10 µm/min at elevated temperatures. This may not be exact and is highly susceptible to temperature! It is recommended to test this for yourself.
7. Put your sample into the etchant and move your sample for the appropriate amount of time calculated in the previous step. *If etchant at the surface becomes saturated and fresh etchant cannot reach the surface, then etching will slow down. Agitation can be used to bring etchant to the surface and promote etching.* In this case use a magnetic stirrer and carefully swirl your etchant to accelerate the etch and improve uniformity.

DI Water rinse

1. When the etch is complete, transfer the sample carefully to the first DI water rinse beaker/envelope and move the sample for 5 minutes in DI water.
2. If you use tweezers to move the sample, make sure you rinse your tweezers as well.
3. Transfer the sample to the second DI rinse beaker, and rinse for another 5 minutes while moving your sample.

Sample dry

1. After the water rinse is finished, remove your samples and blow them dry with the N₂ gun.

Clean-up

1. Let the etchant cool down to room temperature.
2. When the used etchant is at room temperature, pour it carefully over the other two beakers/envelopes filled with DI water.
3. Fill the beaker/envelope where you had your etchant with DI water.
4. Use the venturi to remove the waste from all the beakers/envelopes.
5. Rinse all the beakers/envelopes three times with DI water.
6. Turn all the beakers/envelopes upside down, wash the outside with DI water and blow them dry with the N₂ gun.

7. Return all labware to its proper location.
8. Clean the area and rinse it with DI water.
9. Wash your black gloves and leave them in the bench.

5. Primary Hazards

Aqua regia solutions are extremely corrosive and may result in explosion or skin burns if not handled with extreme caution. It causes destruction of living tissue at site of contact. Corrosive effects can occur not only on the skin and eyes, but also in the respiratory tract. Aqua regia solutions are extremely corrosive and may result in an explosion, skin burns, or eye/respiratory tract irritation.

6. Engineering Controls to Prevent and Mitigate Hazards

Carry out the procedure in a wet bench. Store bottles of chemicals (sealed tightly) in the inorganic cabinets. Work area should contain an eye wash, safety shower and a bottle of diphoterine. Check where you could find this in your neighbourhood.

The chemical are in the high risk category:

- Working with HIGH risk inorganic chemicals is only allowed during office hours.
- A buddy must be in the same module within eye contact.

7. First Aid and Emergency Procedures

Eye Contact: Immediately flush with diphoterine while lifting upper and lower eyelids occasionally (use the complete 500 ml for one eye and remove contact lenses if possible). After using diphoterine, flush with water for at least 15 minutes. Get immediate medical attention. Press evacuation button.

Skin Contact: Remove contaminated clothing, wash skin with diphoterine. After using diphoterine, flush with water for at least 15 minutes. If there is any irritation. Get medical attention. Press the evacuation button.

Inhalation: Remove to fresh air. Get immediate medical attention. Press the evacuation button.

Ingestion: Get immediate medical attention. Press the evacuation button.

In case of a spill: Press the evacuation button.

In case of a fire: Press the fire button

8. Literature

- [1] „Gold Etching,” [Online]. Available:
https://www.microchemicals.eu/technical_information/gold_etching.pdf. [Geopend 2018].
- [2] „Standard Operating Procedure Aqua Regia,” [Online]. Available:
<https://ehs.yale.edu/sites/default/files/files/aqua-regia-sop.pdf>. [Geopend May 2018].