

AQUA REGIA

STANDARD OPERATING PROCEDURE

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Adapted from UC Berkeley Aqua Regia SOP

Section 1: Purpose

This document describes the standard operating procedures for utilizing an aqua regia etch (3:1 HCl:HNO₃). Specifically, the document aims to establish consistent operational procedures with the principal goal being safety.

Section 2: Application

Aqua regia is a corrosive, fuming yellow liquid prepared by slow mixing of one volume of concentrated nitric acid with three volumes of concentrated hydrochloric acid. It is used to dissolve metals such as gold, platinum, silver, etc. Its fumes and yellow color are caused by reaction of nitric acid, HNO₃, with hydrogen chloride, HCl, to form nitrosyl chloride (NOCl), chlorine (Cl₂), and water; both chlorine and nitrosyl chloride are yellow-colored and volatile. Only fresh solutions should be used.

Section 3: Equipment, Chemicals and Supplies

Aqua regia is composed of two chemicals:

1. Concentrated nitric acid (HNO₃, ACS Grade)
2. Hydrochloric acid (HCl, ACS Grade).

They are typically mixed in a 3:1 HCl:HNO₃ mixture.

Section 4: Personal Protective Equipment

Due to the extreme corrosiveness of aqua regia, all possible precautions must be taken to ensure safety. This includes ensuring that no skin is exposed. The following equipment should be used:

- **Safety glasses and laboratory coat are mandatory.**
- Gloves:
 - For use of <20 ml of etchant **doubled** acid-compatible nitrile gloves. Check gloves for leaks before using. Discard immediately on contact with acid.
 - For use of >20ml of etchant:
 - Thick neoprene gloves. Check for leaks before using.
 - Face mask and chemically resistant acid-compatible lab apron.

- Closed-toe shoes (no sandals) and pants (no shorts).

Section 5: Operational Procedures

Note: Aqua regia is extremely aggressive, and may result in an explosion, skin burns, or eye/respiratory tract irritation if not handled properly. 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.

1. **To use aqua regia solutions you must have specific laboratory safety training and be authorized by the lab safety officer prior to doing any work.**
2. Wear proper safety attire (described in section 4) and prepare working area in the hood.
3. Locate the safety shower, and eye wash.
4. Clear the working area of ALL solvents. Remove methanol and acetone bottles, etc.
5. Prepare proper container for aqua regia and place in secondary containment. Prepare a proper waste disposal container: a glass funnel situated on a glass Erlenmeyer flask to temporarily store spent etchant.
6. Generally, acids are always added to water, since acids are heavy and water on top could cause splattering. Here we add nitric acid to hydrochloric acid, since the main component of aqua regia is HCl.
7. Pour 9 mL of hydrochloric acid slowly into the glass centrifugation tube. Close the lid on the hydrochloric acid bottle and return the bottle to storage.
8. Add 3 mL of the HNO₃ solution. Stir gently with a pipette.
9. Place sample in the solution.
10. Empty centrifuge tube into glass funnel on waste Erlenmeyer after desired etch time, being careful to let all excess aqua regia drip off into the funnel.
11. Rinse the sample collected in the funnel with water. The water from at least the first two rinse cycles should be treated as aqua regia waste and disposed of accordingly. The beaker used to hold the tweezers should also be rinsed in the same manner.
12. Aqua regia should NEVER be sealed in a waste container, since it continues to evolve gases. It should therefore be allowed to sit (overnight, if at all possible), before sealing the lid of the waste container.
13. Examine the workspace carefully to ensure that there are no wet spots. Clean up any wet spots as if they were aqua regia. Also inspect gloves and lab coat.

Section 6. Primary Hazards

Aqua regia is extremely corrosive and can quickly cause extreme burns to skin and severe damage to eyes. As described above, 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. By themselves, its constituents are also reactive:

Concentrated Nitric Acid (65%):

Nitric acid is a strong oxidizer and corrosive and is rated 3 (severe) on the health scale. While it is not combustible, HNO₃ enhances the combustion of other substances and can explode on contact with common organic solvents. It is corrosive to skin. Inhalation can damage the respiratory tract.

Hydrochloric Acid (37%):

Rated 3 (severe) on the health scale. Hydrochloric acid is a corrosive and irritant. Skin contact may produce burns and inhalation can cause severe irritation of the respiratory tract.

Section 7. Engineering Controls to Prevent and Mitigate Hazards

All operations with aqua regia are performed in a fume hood, which is close to an eye wash and shower. All containers which contain aqua regia must have secondary containment at all times.

In addition, researchers should:

- Only prepare the amount you need for immediate use
- 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**

Section 8. First Aid and Emergency Procedures

Eye contact:

Immediately flush eyes with plenty of running water for at least 15 minutes. Speed is extremely important. Obtain medical attention as soon as possible.

Skin contact:

Immediately flush affected areas with water, removing contaminated clothing while under the safety shower. Continue washing and get medical attention.

Ingestion

NEVER MAKE AN UNCONSCIOUS PERSON VOMIT OR DRINK FLUIDS!

Promptly get affected personnel to drink large volumes of water to dilute the swallowed chemical. **DO NOT** induce vomiting. Get medical attention immediately.

In case of a spill:

Minor spills can be diluted and neutralized with soda ash, lime or caustic. Large spills should be contained. EH&S should be notified.

In case of fire:

Do not use water. If water is added to concentrate acid a severe eruption may result. Use a carbon dioxide or dry chemical extinguisher.