Lab 01 - Synthesis of a Hydrated Salt

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Chemicals Safety

- Only transfer them in proper containers (lab glassware).
- Do not pour solutions at or above eye level.
- Label any containers used for more than just immediately transferring (e.g. you don't need to label a graduated cylinder, but a beaker for storing it on the bench should be labeled).
- When diluting or adding to a reaction mixture, never pour a solution into a concentrated corrosive as that is more likely to splash the corrosive. Pour the corrosive into the other solution.
- We provide disposable gloves to protect your hands from accidental exposure in the labs. They are a temporary measure in case you get a chemical on them; they typically have a rating for how long before certain compounds pass through to your skin.

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Potassium Hydroxide KOH

Hazards

1. May be corrosive to metals
2. Harmful if swallowed
3. Causes severe skin burns and eye damage
4. May cause respiratory irritation

Precautions

1. Wash face, hands and any exposed skin thoroughly after handling
2. Do not eat, drink or smoke when using this product
3. Do not breathe dust/fume/gas/mist/vapors/spray
4. Wear protective gloves/protective clothing/eye protection/face protection
5. Use only outdoors or in a well-ventilated area
6. Keep only in original container

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Ethanol (C_2H_5OH)

Hazards

1. Highly flammable liquid and vapor
2. Causes serious eye irritation
3. May cause drowsiness or dizziness
4. Can cause central nervous system depression if ingested in large quantities
5. Harmful if inhaled in high concentrations

Precautions

1. Keep away from heat, hot surfaces, sparks, open flames, and other ignition sources. No smoking.
2. Use explosion-proof electrical/ventilating/lighting equipment.
3. Wear protective gloves, protective clothing, and eye protection.
4. Avoid breathing fumes, vapor, or spray. Use only in a well-ventilated area or under a fume hood.
5. In case of fire, use water spray, alcohol-resistant foam, dry chemical, or carbon dioxide.
6. Store in a tightly closed container, in a cool and well-ventilated area.

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Heating Corrosives and Releasing Gasses

Hazards

1. Avoid boiling which could cause spillages.
2. Caution with hot glassware.
3. Heating corrosive substances can cause them to vaporize, leading to exposure to harmful fumes.
4. Gases released during heating can cause respiratory irritation or even poisoning, depending on the substance.
5. If heated in closed containers, the build-up of pressure can cause explosions or container ruptures.
6. Certain corrosives, when heated, may react violently with other substances.

Precautions

1. Always use appropriate glassware rated for heat when working with corrosive substances.
2. Ensure proper ventilation, such as working under a fume hood, to avoid inhaling dangerous fumes.
3. Never heat a closed container, especially if it contains volatile or corrosive substances.
4. When heating, maintain a safe distance and use heat-resistant gloves and protective eyewear.
5. Be aware of the properties of the corrosive you are heating, and avoid overheating to prevent dangerous chemical reactions.

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Guidelines on Disposable Gloves

Hazards

1. Disposable gloves may provide insufficient protection against certain chemicals or corrosive substances.
2. Improper glove fit can lead to tears or reduced dexterity, increasing the risk of spills or exposure.
3. Reusing disposable gloves can lead to contamination, reducing their effectiveness.
4. Certain gloves may cause allergic reactions, particularly those made from latex.
5. Gloves can get punctured or damaged without visible signs, leading to exposure.

Precautions

1. Always choose gloves appropriate for the chemicals and materials being handled (e.g., nitrile, latex, or vinyl gloves).
2. Inspect gloves for visible damage or defects before use, such as tears, holes, or punctures.
3. Discard gloves after each use, especially if they become contaminated or damaged.
4. Remove gloves carefully to avoid skin contact with hazardous substances.
5. Wash hands before and after wearing gloves to minimize contamination risks.
6. Use gloves that fit snugly but allow for free movement to avoid accidental spills.

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Materials

- Aluminum can
- Steel wool or sandpaper
- 250 mL beakers
- 1.5 M potassium hydroxide, KOH
- Hot plate
- Büchner filter flask
- Funnel
- Filter paper
- Vacuum line
- Ice bath
- 9 M sulfuric acid, H_2SO_4
- Ethanol
- Drying oven
- Watch glass

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Procedure

1. Cut open a soda can and remove the top/bottom to get a rectangle of metal. **Sand off the paint and lacquer** from both sides using steel wool or sandpaper. Make sure it's completely clean, or you'll end up with a **sticky yellow product** that won't work later.
2. **Rinse** the metal with water and dry it.
3. Cut the metal into **1 cm squares** and weigh about **1 g** of these (**record the actual mass**).
4. Add the metal squares to a **250 mL beaker** with **40 mL of 1.5 M KOH**. Place the beaker on a **hot plate (in the fume hood)** and gently heat it, but **don't let it boil**. Adjust the heat if bubbles start to form. This will take around **30 minutes**.
5. While waiting, **set up a filtration apparatus** using a **500 mL Buchner flask** and funnel. **Clamp it securely**, place filter paper in the funnel, and **wet it with deionized water**. Have the TF check your setup.
6. Once all the metal dissolves, **use gloves** to remove the beaker from the heat and let it cool slightly.
7. While still hot, **filter the mixture**. The filtrate should be clear—if not, filter again.
8. **Rinse the reaction beaker** with a maximum of **10 mL of deionized water** and pour it over the filter while the vacuum is running.
9. Once filtration is done, **turn off the vacuum** and note the solids on the filter paper and the liquid in the flask.
10. Transfer the filtrate to a **clean 250 mL beaker** and rinse the flask with another **10 mL of deionized water**, adding the rinse to the beaker.
11. Make an **ice bath** with equal parts ice and water, record its temperature, and **cool your filtrate** in it. Don't let the beaker tip!
12. Add **20 mL of 9 M H_2SO_4** to the cold solution while stirring. **Note your observations**, stir for **2 minutes**, and remove from the ice bath.
13. **Heat the mixture** in the fume hood until the **solids dissolve** and the **solution clears**.
14. **Let the solution cool** to room temperature, then place it back in the ice bath to start **crystallization**. If nothing happens after 10 minutes, **scratch the beaker** with a glass rod to help.
15. Once crystallization starts, leave the solution in the ice bath for an additional **10 minutes**.
16. Reassemble and **clean your filtration apparatus**. Filter the crystals using vacuum. **Help transfer the solids** with a spatula and rinse the beaker with a little deionized water to get everything onto the filter.
17. **Wash the crystals** with **10 mL of ethanol** (first, disconnect the vacuum). Then reconnect the vacuum to dry the crystals.
18. Transfer the filtrate to a waste container and **pull air through the flask** for **10 minutes** to fully dry the crystals.
19. **Place the crystals on a watch glass** and put them in the oven at **80°C for 10 minutes** to remove any remaining water and ethanol.
20. Weigh your dried crystals by transferring them to a **pre-weighed watch glass** and **record the mass**.
21. **Label your crystals** with your group name and section, and store them as directed for next week.
22. Dispose of any waste in the **proper containers**, **clean up your workspace**, and return equipment. **Double-check** that the hot plate and vacuum are turned off.
23. Have the instructor **sign off** on your **cleaned workspace**.

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Observations and Records

1. **Mass of metal squares:**

2. **Observations of the reaction in KOH:**

3. **Filtration observations (solids on filter paper and filtrate):**

4. **Temperature of ice bath:**

5. **Observations upon adding 9 M H_2SO_4:**

6. **Observations of solution clearing after heating:**

7. **Observations of crystallization process:**

8. **Final mass of dried crystals:**