Chemistry 3A

Introductory General Chemistry

Experiment 11a

Synthesis of Alum

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Introduction

- This experiment will produce potassium aluminum sulfate dodecahydrate [$KAl(SO_4)_2 \bullet 12\ H_2O$]—called alum from aluminum metal
- Alum uses and purposes are numerous

Industrial and chemical uses

- **Water purification:** Alum acts as a coagulant, causing suspended impurities to clump together and settle out of water, making it a common agent in water treatment plants.
- **Textile dyeing:** It is used as a mordant to help fabric absorb and fix dyes more effectively.
- **Paper manufacturing:** Alum is used for sizing to improve paper strength and surface properties.
- **Fire retardant:** It can be used as a flame retardant in some materials.

Food and culinary uses

- **Pickling:** Alum helps preserve the crispness of fruits and vegetables when pickling.
- **Baking:** Food-grade alum is used as an acidulant in baking powder.
- **Meat tenderizer:** It can be used to tenderize meat by breaking down its protein structure.
- **Crispy foods:** Adding alum to batters can make fried foods crispier.

Health and personal care uses

- **Styptic:** It is used in styptic pencils and blocks to stop bleeding from minor cuts and razor nicks by constricting blood vessels.
- **Astringent:** Alum's astringent properties help tighten skin, reduce pore size, and combat oily skin and acne.
- **Deodorant:** It can be used in natural deodorants by inhibiting odor-causing bacteria.
- **Mouth rinse:** Gargling with alum can help with gum inflammation.
- **Vaccine adjuvant:** Alum is used in some vaccines to enhance the immune response.

Background

There are FOUR reactions involved in alum synthesis:

$$2\ Al\ (s) + 2\ KOH\ (aq) + 6\ H_2O\ (l) \rightarrow 2\ \mathbf{KAl(OH)_4}\ (aq) + 3\ H_2\ (g)$$

SINGLE REPLACEMENT – REDOX – *This reaction evolves a gas*

$$2\ \mathbf{KAl(OH)_4}\ (aq) + H_2SO_4\ (aq) \rightarrow 2\ \mathbf{Al(OH)_3}\ (s) + K_2SO_4\ (aq) + 2\ H_2O\ (l)$$

DOUBLE REPLACEMENT – PRECIPITATION – *This is a precipitation*

$$2\ \mathbf{Al(OH)_3}\ (s) + 3\ H_2SO_4\ (aq) \rightarrow \mathbf{Al_2(SO_4)_3}\ (aq) + 6\ H_2O\ (l)$$

This is an **ACID-BASE NEUTRALIZATION** *of an alkaline precipitate*

$$\mathbf{Al_2(SO_4)_3}\ (aq) + K_2SO_4\ (aq) + 24\ H_2O\ (l) \rightarrow 2\ \mathbf{KAl(SO_4)_2 \bullet 12\ H_2O}\ (s)$$

This is a **HYDRATION & CRYSTALLIZATION** *of a double salt*

$$2\ Al\ (s) + 2\ KOH\ (aq) + 4\ H_2SO_4\ (aq) + 22\ H_2O\ (l) \rightarrow 2\ KAl(SO_4)_2 \bullet 12\ H_2O\ (s) + 3\ H_2\ (g)$$

This is the **NET REACTION** *by combining all steps*

Background

- In these reactions, the product of one step is carried over as a reactant in the succeeding step
- Many reaction types are involved, from a gas-evolving step to a precipitation to an acid-base neutralization and then to a crystallization of a double salt
- A double salt is where a compound involves a combination or mixture of two metal cations, in this case potassium (K) and aluminum (Al) both countered with a sulfate anion (SO_4)
- There are many steps to pay careful attention to their execution

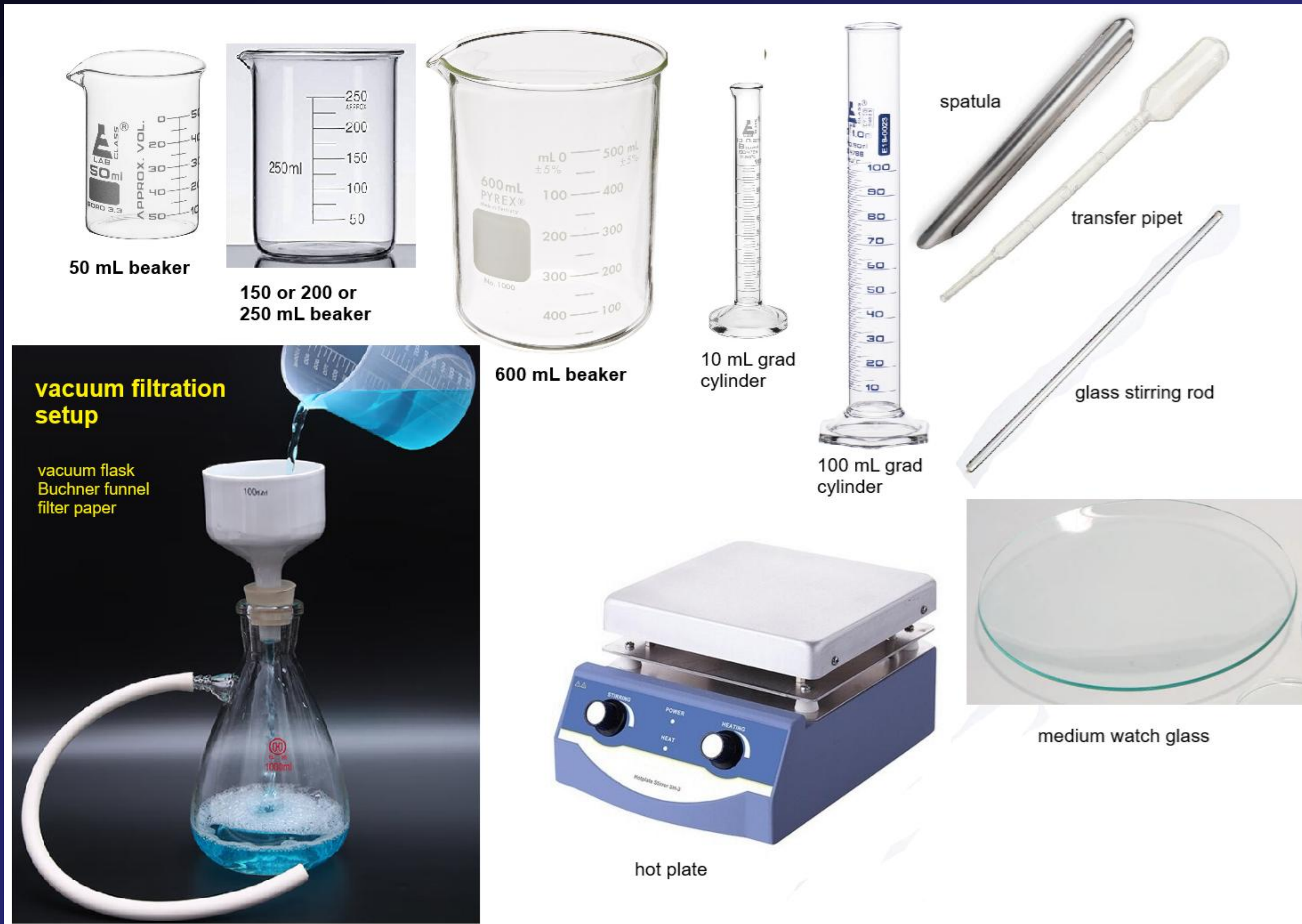
Reproducibility

- Instruments of all kinds do not always give a true reading just one time in use
- Careful scientists will often want to get a mass on a balance not just once, but even THREE times to ensure reproducibility
- Check the web page I wrote in ensuring reproducibility in use of instruments
- http://fresno.chem101.com/Chemistry/BetterLabReports.html

Limiting & Excess Reagents

- You will use three reagents in this experiment
  - aluminum solid (foil or powder)
  - potassium hydroxide
  - sulfuric acid
- Which of these is/are the limiting reagent? Can there be more than one limiting reagent?
- Which of these is/are the excess reagent? Can there be more than one excess reagent?
- About 0.4 g aluminum is used. What is that in moles?
- Why was about 20 mL of 1.5 M KOH used?
- Why was 7.5 mL of 6 M sulfuric acid used?

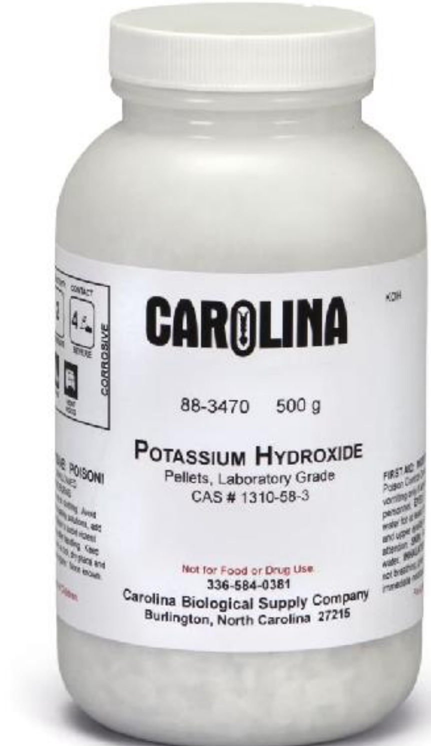
Equipment You Will Use



Consumables You Will Use

Aluminum
foil pieces

6 M H_2SO_4



1.5 M KOH

50% EtOH (ethanol)

Procedure

1. Select a 150, 200, or 250 mL beaker and record its mass
2. Tear about 0.4 g aluminum foil into small pieces and place in beaker
3. Record the mass of beaker with foil pieces
4. Put name on the beaker with Sharpie

Reaction 1 - REDOX (H_2 Gas Evolution) (Step 5)

a. In the fume hood, add 20 mL of 1.5 M KOH to the beaker
b. The foil pieces should dissolve and the solution becomes black. It takes about 20 min for reaction to be complete
c. While reaction occurs, start calculating theoretical yield
d. If reaction slows, put beaker on a hot plate but be prepared to take beaker off the hot plate so it does not boil!
e. Is there a gas produced? What is it?

Procedure

Reaction 2 (Precipitation) [DD]

6. Into a 50 mL beaker, pour 7.5 mL of 6.0 M H_2SO_4 from a 10 mL graduated cylinder
7. While stirring the dissolved aluminum solution with a glass rod in the larger beaker, use a transfer pipet to transfer the acid solution to the aluminum solution DROPWISE. Make sure you stir well after each drop. Look for a greyish white precipitate

Reaction 3 (Acid-Base Neutralization)

8. Cover beaker with watch glass and warm solution on hot plate until the precipitate has dissolved. Be careful NOT to get it to hot and boil the solution!!
9. After full dissolution, let beaker cool for few minutes

Procedure

10. Into a larger beaker set up an ice-water bath
11. Place the reaction beaker in the ice-water bath and let it sit for 15 min

Reaction 4 (Hydration-Crystallization)

12. Swirl beaker when crystal formation is noticed. If after 5 min crystals are not seen, scratch inside of beaker with glass rod to induce nucleation
13. Into a separate small beaker, pour 30 mL of 50% ethanol and place in ice-water bath

Procedure

14. Set up a vacuum filtration apparatus as shown

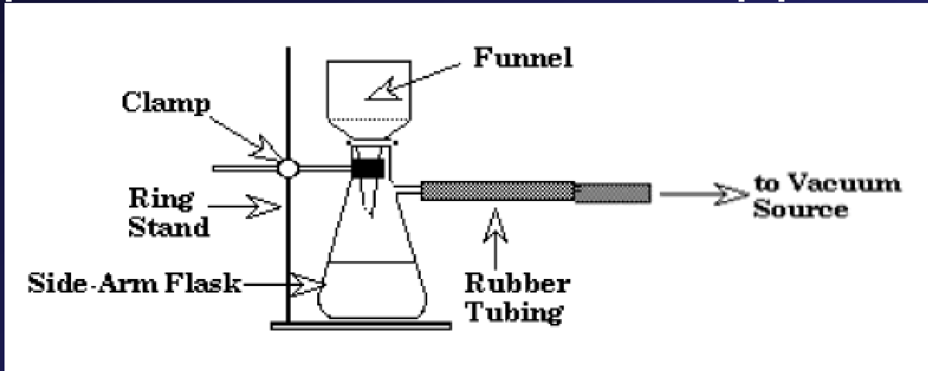


Figure 1: Vacuum Filtration Apparatus

15.
   a. Apply the vacuum to the apparatus and wet the filter paper so that it adheres to funnel bottom with all the holes covered
   b. Swirl the beaker to loosen crystals and pour the crystals with solution on to the paper in the funnel
   c. Pour ~10 mL of cold 50% EtOH into beaker and then swirl to wash out crystals into funnel while also slowly washing the crystals with the ethanol through the filter paper. Repeat ethanol washing of crystals

Procedure

15.
   d. After washing is complete, let the crystals remain on the vacuum until they appear dried by the process. Prodding the crystals with a glass rod should prove how dry they are
16. Record the mass of a clean & dry watch glass
17. Transfer the crystals to the watch glass. Now record the mass of the crystals + watch glass
18. Determine the yield of crystals and use to complete rest of report

Clean Up

- Except only for glass containers that had reaction mixture, just a brief water rinse of glassware and upside-down drip-drying on a sink rack pin is sufficient; this includes grad cylinders & beakers getting KOH and H_2SO_4
- A small amount of soap on brush wash should be sufficient
- There is a solid waste beaker to receive under fume hood near doors
- Any liquid waste there is a large carboy also in that fume hood
- Clean up area as usual. Quick paper towel dry wipe is enough to dry.

Example Data Analysis

REFERENCE VALUES FROM PRE-LAB QUIZ

2. Molar Mass of aluminum 26.98 g/mol
3. Molar Mass of Alum 474.39 g/mol
4. Reaction ratio 1 mol Al : 1 mol $KAl(SO_4)_2 \cdot 12H_2O$

DATA

1. Mass of the reaction beaker 110.586 g
5. Mass of the beaker + aluminum 110.934 g
6. Mass of watch glass 27.3493 g
7. Mass of watch glass + alum 32.0551 g

Example Data Analysis

RESULTS

Show your work for each of the following calculations.

8. Mass of aluminum 0.348 g

$$110.934 - 110.586 = 0.348 \text{ g}$$

9. Theoretical yield of Alum 6.12 g

$$0.348 \text{ g Al} \times \frac{1 \text{ mol Al}}{26.98 \text{ g Al}} \times \frac{1 \text{ mol alum}}{1 \text{ mol Al}} \times \frac{474.39 \text{ g alum}}{1 \text{ mol alum}} = 6.12 \text{ g alum}$$

10. Mass of Alum 4.7058 g (actual yield)

$$32.0551 - 27.3493 = 4.7058 \text{ g alum}$$

Example Data Analysis

11. Percent yield 76.9%

$$\frac{4.7058\ g}{6.12\ g} \times 100\% = 76.9\%$$

12. Discuss your percent yield. (Is this a reasonable value? Why or why not?)

1) Percent yield not greater than 100% which is good for reasons of conservation of mass and a showing of careful technique in determining masses of reactants and products
2) % yield better than 50% generally considered acceptable
3) Sources of a low percent yield could due to
   a) not letting aluminum react fully with KOH
   b) loss in transfer of intermediate products
   c) incomplete crystallization caused by cooling solution too quickly
   d) excessive washing during filtration