

Preparation of an Alum

Pages 75 – 84

Pre-lab = pages 81 to 82, all questions

No lab questions, a lab report is required by the start of the next lab

What is an alum?

- They are white crystalline double sulfates of univalent and trivalent atoms.
- $M^+M'^{3+} (SO_4)_2 \cdot 12H_2O$
- M^+ = univalent cation: Na^+ , K^+ , Tl^+ , NH_4^+
- M^{3+} = Al^{3+} Fe^{3+} Cr^{3+} Ti^{3+} Co^{3+}

Uses



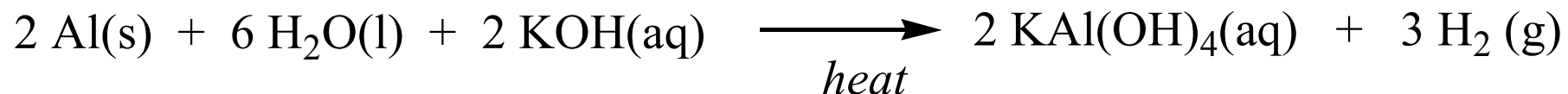
- These salts are employed in dyeing and various other industrial processes. They are soluble in water; have an astringent, acid, and sweetish taste; react acid to litmus; and crystallize in regular octahedra.
- When heated they liquefy; and if the heating is continued, the water of crystallization is driven off, the salt froths and swells, and an amorphous powder remains.

- **Shaving alum** is a powdered form of alum used as an astringent to prevent bleeding from small shaving cuts. The styptic pencils sold for this purpose contain aluminum sulfate or potassium aluminum sulfate. Similar products are also used on animals to prevent bleeding after nail-clipping. Alum in block form (usually potassium alum) is used as an aftershave, rubbed over the wet freshly shaved face.
- **Crystal deodorant:** Alum was used in the past as a natural underarm deodorant in Mexico, Thailand, and the Far East. Typically potassium alum is used.

- **Wax:** Alum is used in the Middle East as a component in wax, compounded with other ingredients to create a hair-removal substance.
- **Foamite:** Alum is used to make foamite which is used in many fire extinguishers for chemical and oil fires.
- **Adjuvant:** Alum is used regularly as an adjuvant (enhances immune response to a given immunogen when given with it) in human immunizations.

- **Alum powder**, found amongst spices at most grocery stores, is used in pickling recipes as a preservative, to maintain crispness, and as an ingredient in some play dough recipes. It is also commonly cited as a home remedy or pain relief for canker sores.
- **Water treatment:** Alum (aluminum sulfate) is used in water treatment. The addition of alum to raw water causes small particles and colloids to stick together and form heavier particles (floc) which will settle in water. This process is called coagulation or flocculation.
- **Fire retardant:** By soaking and then drying cloth and paper materials they can be made fireproof.

Reaction A



- Al foil will be used as the Al source.
- Weigh approx. 0.5 grams
- Tear into small pieces (to increase the surface area, and reactivity)
- Add KOH and swirl *carefully*
- Heat with a cool flame

Important Points

- Make sure your solution does not evaporate to dryness, by adding DI water.
- Once all your Al is dissolved – gravity filter the warm reaction mixture.
- This removes any impurities
- Keep the filtrate (the solution you collect)
- Your solution contains $\text{Al}(\text{OH})_3$

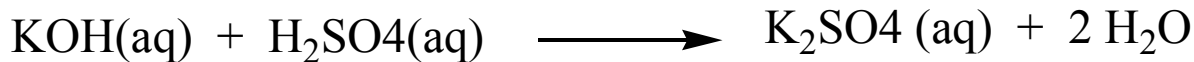
Amphoterism

- The ability of some chemicals to act either as an acid or a base is called amphoterism.
- Whether an amphoteric chemical acts as an acid or a base depends on what other chemicals happen to be around.
- Aluminum Hydroxide is amphoteric



- Aluminum hydroxide, Al(OH)_3 , reacts as a base with common acids to form salts, e.g., with sulfuric acid, H_2SO_4 , to form aluminum sulfate, $\text{Al}_2(\text{SO}_4)_3$.
- It reacts as an acid with strong bases to form aluminates, e.g., with sodium hydroxide, NaOH , to form sodium aluminate, $\text{Na[Al(OH)}_4(\text{OH}_2)_2]$.

Adding H₂SO₄



- Add the acid SLOWLY (1ml at a time)
- The reaction is exothermic
- Add approximately 15 mls of acid

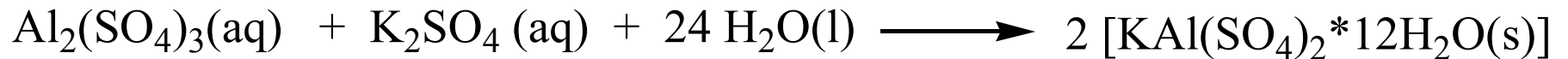
- The sulfuric acid reacts with potassium aluminum hydroxide to form aluminum hydroxide, $\text{Al}(\text{OH})_3$.
- The aluminum hydroxide precipitates from solution as a solid and excess potassium hydroxide, KOH , is neutralized to potassium sulfate

- After adding the acid the solution may need to be heated
- Heating dissolves the $\text{Al}(\text{OH})_3(\text{s})$ and promotes the formation of aluminum sulfate, $\text{Al}_2(\text{SO}_4)_3$.



Crystallization

- On cooling, the aluminum sulfate, potassium sulfate, and water combine to form alum, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

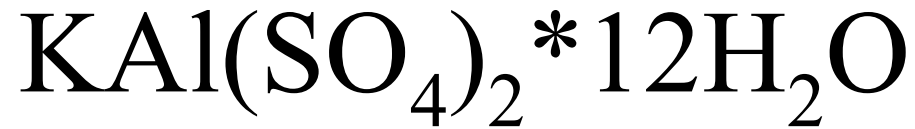


Growing alum crystals

- Crystallization is the process in which a dissolved solute comes out of solution and forms a solid. In synthesis reactions, crystallization is often used to purify a product.
- At low concentrations, solutes (molecules or ions) dissolved in a solvent are well separated from each other.
- As the solute concentration increases, some solutes unite, forming a nucleate. Nucleates can be as small as four or five molecules.
- As the solute concentration increases, so do the number of nucleates formed. When a solution can no longer hold any more solute for an extended period of time, it is said to be saturated.

Theoretical Yield

- In theory, the amount of product that could be produced if the reaction were 100% efficient.
- Always start by calculating moles of known species (Al) and then doing a mole to mole ratio to determine the moles of the desired species (potassium alum).
- •The last step converts the moles of the desired species to the needed units. If a solid, generally find grams. If aqueous solution, generally find Molarity or volume.



- When calculating your theoretical yield, the water molecules need to be included in the calculation.
- Without the water molecules being present the alum would not crystallize out.
- Heating the alum could cause the hydrated water molecules to be lost

Actual or Experimental Yield

- The actual amount of product recovered in the experiment.
- Never weigh chemicals directly on a balance, weigh your product by difference weighing.
- $\text{Weight of watch glass + product} - \text{Weight of watch glass alone} = \text{Weight of product}$

- **Percent Yield (% yield) = (Actual Yield / Theoretical Yield) x 100**

Lab Techniques



Vacuum Filtration

- Vacuum filtration is a technique for separating a solid product from a solvent or liquid reaction mixture.
- The mixture of solid and liquid is poured through a filter paper in a Buchner funnel.
- The solid is trapped by the filter and the liquid is drawn through the funnel into the flask below, by a vacuum.

Setting up and performing a vacuum filtration



- To prepare for a vacuum filtration, gather together a filter flask, Buchner funnel, tubing, filter paper, clean solvent, disposable dropper, and your sample.
- Vacuum outlets are located in the hoods, which are connected to a central vacuum pump for the entire building.
- The tubing should be connected from the vacuum nozzle to the side arm of your filter flask.

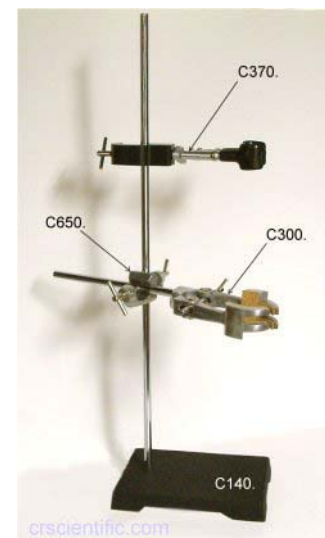


- Turn on the vacuum using the knob on the outside of the hood. Check the vacuum by feeling for suction at the end of your tubing. The vacuum should be strong enough to hold the tubing to your finger without falling off.



- Place the black rubber ring adapter in the top of the flask and then the Buchner funnel.

- Check for good suction by placing your gloved hand across the top of the funnel.
- If you do not feel strong suction, there is a poor connection and a possible leak somewhere in your system.
- Ensure your flask is clamped in a secure position.

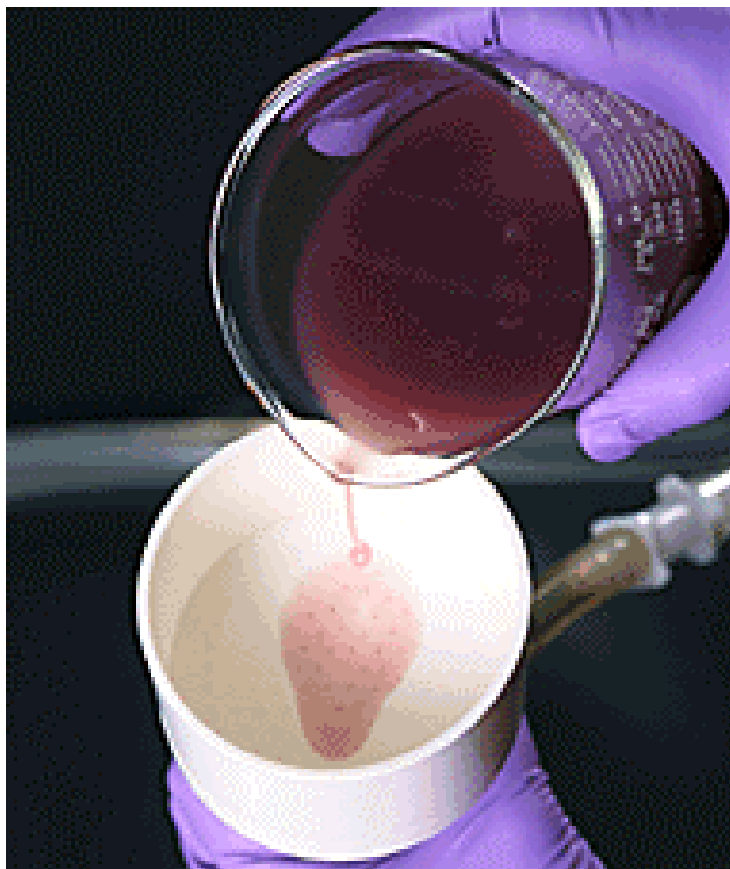




- Prepare to filter your sample by placing a filter paper in the Buchner funnel and wetting it with clean solvent.



- You should see the paper being sucked down against the holes in the funnel and the solvent should quickly pass through into the filter flask.



- To filter your sample, slowly pour into the center of the filter paper.
- Use more clean solvent to rinse your beaker, so that all the solid is collected.
- Continue to draw air through the solid, to evaporate any remaining solvent in your sample.
- When you are finished, break the vacuum at the connection between the flask and the trap. Then turn off the vacuum with the tap on the outside of the hood.

- You may notice that more crystals formed in the filter flask during the washings. This second crop of crystals may also be collected,
- If you choose to collect these crystals, they should be kept separate from the main crop.
- It is always good laboratory practice to keep different crops of crystals separate until the identity and purity of each crop is determined (second crops almost always contain more impurities than the first crop and the time needed to purify them sometimes far outweighs the additional yield).

Recording the Melting Point

- There are instruments in the Organic Lab
- Some melting point apparatus will also be available in the lab.
- Record the melting point of a couple of samples simultaneously, as the oil / apparatus takes a while to cool down.

- The purer the material. The sharper the melting point.
- Impure materials have a broad melting point.
- Heat your sample slowly. This will make your result more accurate.
- Record the temperature range over which it melts, e.g. 92-93°C or 101 – 111°C

Lab Report

- Introduction / Objectives of the Lab
- Results and Discussion
- Conclusion

- Three pages maximum

- DO NOT WRITE YOUR REPORT IN THE FIRST PERSON!!!
- It must be written in the third person.
- For example..
- I made some alum in the lab, I added potassium hydroxide, I stirred it, filtered it and got 100 g out.

How to write:

- Alum was prepared by the reaction of potassium hydroxide with aluminum metal affording the desired compounds $\text{KAl}(\text{SO}_4)_4$ in high yield.

Objectives

- 8 points
- What are you making and why?
- Give the equation
- Uses of alum

Results and Discussion

- 12 points
- Describe what you did, include the techniques you employed.
- Include your observations
- Give your yield, and how it was calculated

Conclusion

- 10 points
- The conclusion summarizes what you did in the experiment
- How did the reaction go?
- Could it have been improved, if so how.

Reference Sites

- www.pubs.acs.org (Journal of American Chemical Society)
- www.rsc.org
(Chemical Communication)
- These journals can be looked at for examples of how to write up experimental details.