Synthesis experiment: The preparation of Alum

Materials:

- Aluminum Metal (roughly ½ g)
- 150 mL Beaker
- 10 mL Graduated cylinder
- Glass stirring rod
- Watch glass
- Spatula
- Thermometer
- Bunsen Burner
- Buchner funnel
- Filter paper
- 3 M KOH (10 mL)
- 6 M H_2SO_4 (12 mL)
- 50 : 50 Alcohol/water mixture (10 mL)
- Solid $NaHCO_3$

Part 1: Reacting Aluminum with KOH

1. Obtain mass of beaker
2. Obtain a piece of Aluminum metal approximately ½ g, using a paper towel protect the table and sand down the Aluminum (both sides to get the paint and clear coating off).
3. Cut up 0.4g of the aluminum into small pieces
4. Weight the pieces and record data
5. Transfer the pieces to 150 mL beaker
6. Set up a ring stand with wire gauze and an iron ring

Exercise caution in next steps

7. **Carefully** add 10.0 mL of 3.0 M KOH to the beaker after starting the Bunsen burner
8. Heat on a **Low** flame with caution (do this intermittently as heat is already generated by the reaction, especially at first when the initial reaction is vigorous, being sure to keep the flame away from the top of the beaker, the hydrogen being produced could be ignited by it. If bubbling becomes too vigorous solution could be lost, if that happens remove solution from heat by turning off the Bunsen burner)
9. After initial reaction, continue to **heat and stir** gently (At this point the reaction should become self-sustaining) until the aluminum is completely dissolved (If you see specs of paint floating around, use the spatula to remove them, add distilled water to keep the solution approximately 10 mL, don't worry if black particles are seen that cannot be removed with spatula, the H_2SO_4 will dissolve those)

Part 2: Reacting $KAl(SO_4)_2 \cdot 12H_2O$ with H_2SO_4

10. Cool the solution to approximately room temperature (A cold water bath will speed this process)

Exercise caution in next steps

11. **SLOWLY** (Drop by drop) acidify the solution by adding 6M H_2SO_4 while stirring the mixture, until a total of 10 mL is the acid is to the solution (Watch for any chemical changes)

12. There will most likely be solids present (Cloudy grayish mixture), to further purify the Alum thoroughly stir and heat the solution until the solids are dissolved (Almost clear solution). When you heat the solution, be sure to keep the solutions volume approximately 22-23 mL with distilled water. If this is ignored your solution will result in impure Alum or co-precipitates.

Part 3:

13. Remove the beaker from the heat and place it on the porcelain part of the ring stand (**Not your work bench**)
14. Allow the solution to cool undisturbed for approximately 30 minutes at room temperature
15. Then allow another 15 minutes for the solution to cool in an ice bath (Slow and undisturbed crystal growth will result in purer, larger crystals. Keep beaker in ice bath until its time to filter)

If no crystals form

- 1) Add seed crystal of Alum to initiate the growth via stirring rod
- 2) Scratch the side of the glass with a stirring rod which as NOT been fire polished
- 3) Place beaker in ice bath
- 4) re-heat solution to evaporate a small amount of water, then allow solution to cool again. (This will take 45 or more minutes)

Part 4: Vacuum filtration

16. Obtain Buchner funnel, a side arm filter flask, and filtration paper
17. Place the filter so its covering the holes of the Buchner funnel (be sure it's not running up the sides of the funnel)
18. Wet the filter paper with distilled water, then turn on the water aspirator
19. Transfer most of the crystals to the filter paper using a spatula, leaving some solution in the beaker to aid the transfer of the rest of the solution
20. Rinse the beaker with 9 to 10 mL of the 50:50 mixture of alcohol and water to transfer as much as you can of the crystals to the filtration funnel
21. Transfer the crystals to a labeled watch glass and allow them to dry in a 60° C oven

Part 5: Wrap up (Varies per instructor)

22. Record your final mass OR let the crystals dry in your lab drawer until next lab
23. Calculate theoretical yield of alum based on the mass of aluminum you started with
24. Calculate percent yield

Clean up:

Refer to page 34 in your lab book.