EXPERIMENT 7 – PREPARATION OF ALUM

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I. Objective

The purpose of this experiment is using recycled aluminum cans to synthesize alum (aluminum potassium sulfate dodecahydrate), including purification through recrystallization.

II. Technique

You will learn the skills of weighing chemicals, measuring liquids, manipulating of gravity filtration, suction filtration, and recrystallization.

III. Introduction

(1) Synthesizing alum

Although aluminum is an active metal, it reacts only slowly with dilute acids because its surface is normally protected by a very thin coating of aluminum oxide. On the other hand, alkaline solutions (containing OH^{-}) dissolve the oxide layer and then react with the metal. Aluminum is converted by excess alkali to the tetrahydroxoaluminate(III) anion (7-1), which is soluble in basic solution. When acid is added slowly to a solution of this complex anion, initial reaction removes one hydroxide ion, causing the precipitation of the white, flocculent $Al(OH)_3$ (7-2) If addition of acid is continued, the aluminum hydroxide is dissolved, eventually forming the hydrated aluminum cation $[Al(H_2O)_6]^{3+}$ (7-3). Addition of alkali to the $Al(OH)_3$ precipitate will also bring about solution, by reforming the complex anion $[Al(OH)_4]^{-}$ (7-4). A substance such as aluminum hydroxide that can be dissolved by either acid or base is said to be amphoteric.

$$2Al(s) + 2KOH(aq) + 6H_2O(l) \rightarrow 2K(aq) + 2Al(OH)_4^{-}(aq) + 3H_2(g) \quad (7\text{-}1)$$

$$Al(OH)_4^{-}(aq) + H^{+}(aq) \rightarrow Al(OH)_3(s) + H_2O(l) \quad (7\text{-}2)$$

$$Al(OH)_3(s) + 3H^{+}(aq) \rightarrow Al^{3+}(aq) + 3H_2O(l) \quad (7\text{-}3)$$

$$Al(OH)_3(s) + OH^{-}(aq) \rightarrow Al(OH)^{4-}(aq) \quad (7\text{-}4)$$

Alums are ionic compounds which crystallize from solutions containing sulfate anion, a trivalent cation (such as Al^{3+}, Cr^{3+} Fe^{3+} and a monovalent cation (K^{+}, Na^{+}. NH_4^{+}). Most alums crystallize readily as octahedral or cubes which, under the appropriate conditions, may grow to considerable size. The crystal lattice contains 12 water molecules, 6 of which are tightly bound to the trivalent cation, with the remaining 6 loosely bound to the sulfate anion and other monovalent cation.

The first purpose of this experiment is to transform scrap aluminum cans into alum by reacting with base. The flow chart of the experiment is shown as follows:

$$2Al(s) \xrightarrow[\triangle]{KOH(aq)} Al(OH)_4^-(aq) \xrightarrow[\triangle]{H_2SO_4(aq)} Al^{3+}\ (SO_4^{2-}, K^+, H_2O)$$

$$\xrightarrow{cool} KAl(SO_4)_2 \cdot 12H_2O(s)$$

The balanced equation for the precipitation of alum is shown as equation 7-5.

$$K^+(aq) + Al^{3+}\ (aq) + 2SO_4^{2-}(aq) + 12H_2O(l) \longrightarrow KAl(SO_4)_2 \cdot 12H_2O(s) \quad (7\text{-}5)$$

IV. Equipment

- water-flow suction pump, suction flask, Büchner funnel, rubber stopper
- hot plate/magnetic stirrer, ring support and stand, funnel, glass rod
- graduated cylinder (50 mL), beaker (100 mL), beaker (500 mL for ice-water bath)
- scissors, sandpaper, filter paper, aluminum soft-drink cans (self-prepared)

V. Chemicals

- 1.4 M Potassium hydroxide, KOH
- 9.0 M Sulfuric acid, H_2SO_4
- Aluminum potassium sulfate dodecahydrate, alum, $KAl(SO_4)_2 \cdot 12H_2O$
- Ethanol/water mixture (1:1, v/v)

VI. Procedure

(1) Preparation of alum

1. Each group is to bring to the laboratory an empty aluminum can.
2. Cut out a piece of aluminum 5 cm by 5 cm, and scrape off any paint as completely as possible with sandpaper. Then cut it into smaller pieces.

   Caution: *It may cut through skin easily while cutting aluminum can for its sharp edges. Handle these pieces with caution.*

   Note: This step can be completed before the laboratory session.
3. Weigh approximately 0.5 g of aluminum pieces. Record this exact mass accurately.

4. Place the aluminum clippings into a 100 mL beaker. Add 25 mL of 1.4 M KOH. Heat the mixture on a hot plate in a fume hood, with stirring to speed up the reaction.

   Caution: *The reaction produces hydrogen gas, which can form a flammable gas mixture with air. A pungent smell is also produced. Hence this step must be performed in the fume hood. Keep any open flames away from the hood.*

5. During the reaction process, the aluminum pieces will rise and fall periodically in the solution. Suggest and write down an explanation in the experimental record. The reaction is complete when the evolution of hydrogen gas ceases.

6. Use gravity filtration to filter this hot solution to remove any solid residue. Rinse the beaker with approximately 1 mL of deionized water and filter again.

   Note: *Refer to the experiment skills videos to learn the technique of suction filtration.*

7. Transfer the clear filtrate into a 100 mL beaker. Rinse the beaker with 1 mL of deionized water, and combine with the filtrate. Cool the beaker in a cool water bath and add 12 mL of 9 M H_2SO_4 slowly for the neutralization reaction is exothermic.

   Note: *As alum is soluble in water, use as little water as possible for rinsing.*

8. After sulfuric acid is added, precipitation of white $Al(OH)_3$ will occur. Wipe dry the outer wall of the beaker and warm the solution on a hot plate with stirring to dissolve this precipitate. If any insoluble solid impurity is present in the solution after boiling, use gravity filtration to remove it while the solution is still hot.

   Note 1: *The final volume of the solution should be less than 15 mL to ensure supersaturation and successful crystallization of the alum.*

   Note 2: *Refer to the experiment skills videos to learn the technique of gravity filtration.*

9. Cool the filtrate obtained in Step 8 (containing Al^{3+}, K^+, SO_4^{2-}) to room temperature. If no crystals are formed, scrape the inside wall of the container with a glass rod to initiate crystallization. Set the reaction beaker into an ice-water bath to ensure complete crystallization of alum.

   Note: *If still no crystals form from the chilled solution, the concentration of alum may be too low to reach saturation. Think of a way to solve this problem.*

10. Collect the alum crystal by suction filtration. Use a glass rod to flatten the product on the filter paper. Rinse the remaining crystals in the beaker with 12 mL of ethanol/water solution (1:1, v/v) and pour the solution into the funnel. Allow the crystals to vacuum dry for about 10 min. After the crystals have dried, record the accurate weight of the product and calculate its yield.

    Note: *Do not use too much ethanol/water solution to rinse the solid in the beaker because the alum will dissolve in the mixture.*

11. Transfer all the alum obtained from **Procedure 10** into a 100 mL beaker. Add 25 mL of deionized water, then heat and stir the mixture on a hot plate until it reaches a boil and all solids are completely dissolved.

    Note: *Do not overheat the solution. The final volume should be maintained at approximately 20–25 mL.*

12. While the solution is still hot, remove any impurities by gravity filtration and collect the filtrate in a sample vial. Then, allow the solution to cool.
13. Loosely place the cap on the sample vial (do not screw it on tightly), and allow the solution to stand undisturbed for two weeks to undergo recrystallization. You may get a nice octahedral crystal and keep it as souvenir.
14. Take out the crystal. Observe and record the crystal shape, color, and size. Crystal growth may be continued for several weeks if desired.

VII. References

1. Irgolic, K.; Peck, L.; O'Connor, R.; Glenn, P. Fundamentals of Chemistry in the Laboratory; 2nd ed., Burgess Publishing Co.: Minnesota, 1981; pp 35-48.
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3. Wyckoff, R. W. G. Crystal Structures; 2nd ed., John Wiley & Sons Inc.: New York, 1965; pp 872-875.
4. The Merck Index; 11th ed., Budavari, S.; O'Neil, M. J.; Smith, A.; Heckelman, P. E., Eds.; Merck & Co., Inc.: New York, 1989;

Experiment 7 : Preparation of ALUM

Name: ________________________ **Student ID:** __________________________

Department: ___________________ **Group:** __________ **Date:** ___________

I. Experimental Data and Results

Preparation of alum

(1) Mass of aluminum strips ____________________________

(2) Mass of alum obtained ____________________________

(3) Theoretical yield of alum ____________________________

(4) Yield of alum ____________________________

Calculation:

II. Question and Discussion

1. Illustrate the types of chemical reactions involved in the synthesis of alum.