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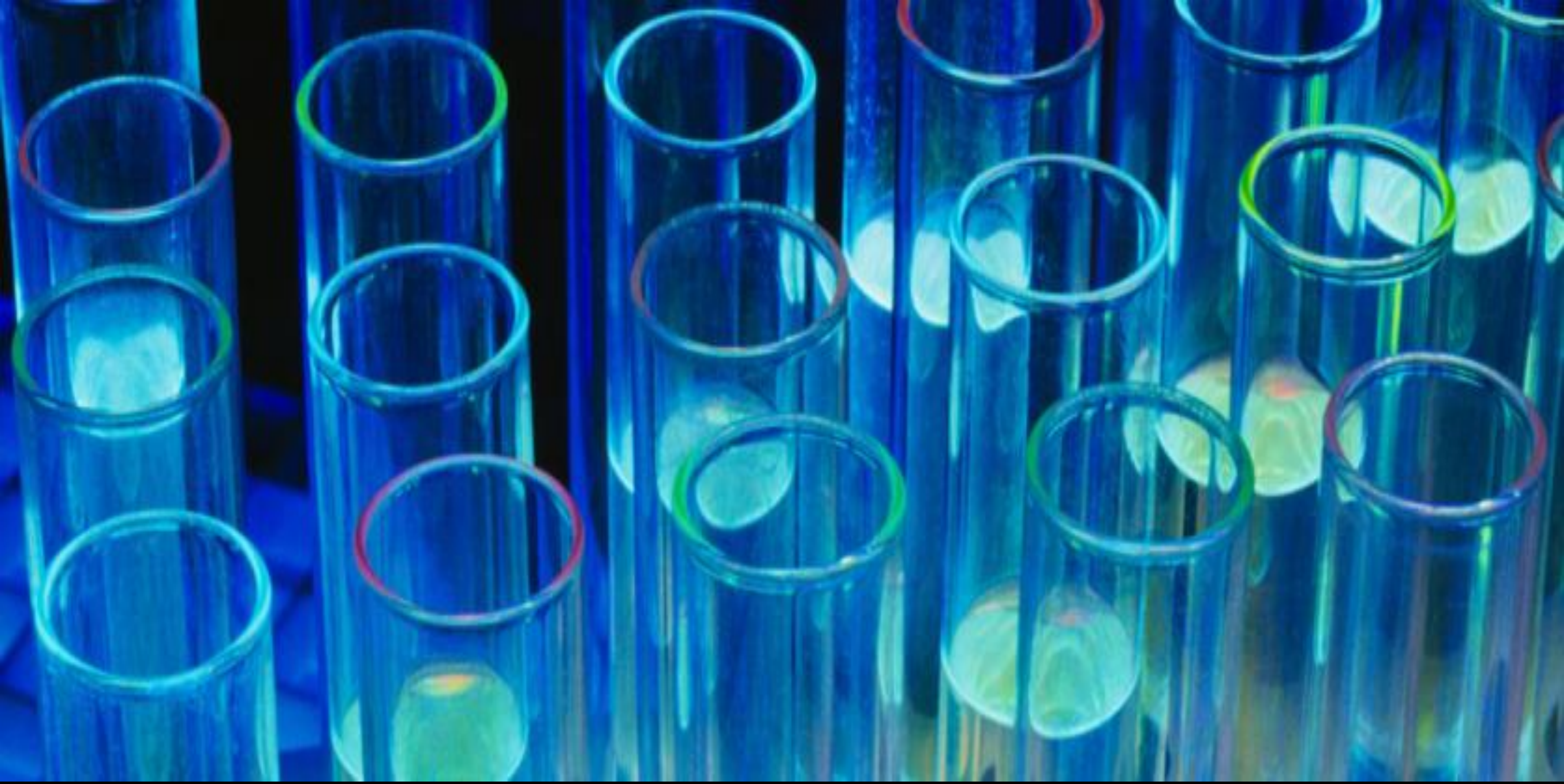


113-2

Expt. 7 - Preparation of Alum

114.05.09 (time required: 2 hours)

PERIODIC TABLE
. OF THE ELEMENTS .

- : NON METALS
- : ALKALINE EARTHS
- : ALKALI METALS
- : TRANSICION METALS
- : OTHER METALS
- : METALLOIDS
- : HALOGENS
- : NOBLE GASES
- : ACTINIDES
- : LANTHANIDES
- : UNKNOWN ELEMENTS

1 H Hydrogen																	2 HE Helium
3 LI Lithium	4 BE Beryllium											5 B Boron	6 C Carbon	7 N Nitrogen	8 O Oxygen	9 F Fluorine	10 NE Neon
11 NA Sodium	12 MG Magnesium											13 AL Aluminium	14 SI Silicon	15 P Phosphorus	16 S Sulfur	17 CL Chlorine	18 AR Argon
19 K Potassium	20 CA Calcium	21 SC Scandium	22 TI Titanium	23 V Vanabium	24 CR Chromium	25 MN Manganese	26 FE Iron	27 CO Cobalt	28 NI Nickel	29 CU Copper	30 ZN Zinc	31 GA Gallium	32 GE Germanium	33 AS Arsenic	34 SE Selenium	35 BR Bromine	36 KR Krypton
37 RB Rubidium	38 SR Stronium	39 Y Yttrium	40 ZR Zirconium	41 NB Niobium	42 MO Molybdenum	43 TC Technatium	44 RU Ruthenium	45 RH Rhodium	46 PD Palladium	47 AG Silver	48 CD Cadmium	49 IN Indium	50 SN Tin	51 SB Antimony	52 TE Tellurium	53 I Iodine	54 XE Xenon
55 CS Caesium	56 BA Barium	* 57-71	72 HF Hafnium	73 TA Tantalum	74 W Tungsten	75 RE Renium	76 OS Osmium	77 IR Iridium	78 PT Platinum	79 AU Gold	80 HG Mercury	81 TL Thallium	82 PB Lead	83 BI Bismuth	84 PO Polonium	85 AT Astatine	86 RN Radon
87 FR Francium	88 RA Radium	** 89-103	104 RF Rutherfordium	105 DB Dubnium	106 SG Seaborgium	107 BH Bohrium	108 HS Hassium	109 MT Meitnerium	110 DS Darmstadtium	111 RG Roentgenium	112 CN Copernicium	113 NH Nihonium	114 FL Flevorium	115 MC Moscovium	116 LV Livermoarium	117 TS Tennessine	118 OG Ognesson

ATOMIC NUMBER — 4
SYMBOL — LI
NAME — Lithium

57 LA Lanthanum	58 CE Cerium	59 PR Proseodymium	60 ND Neodymium	61 PM Promethium	62 SM Samarium	63 EU Europium	64 GD Godolinium	65 TB Terbium	66 DY Dysposium	67 HO Holmium	68 ER Erbium	69 TM Thullium	70 YB Ytterbium	71 LU Lutetium
89 AC Actinium	90 TH Thorium	91 PA Protactiunium	92 U Uranium	93 NP Neptunium	94 PU Plutonium	95 AM Americium	96 CM Curium	97 BK Berkelium	98 CF Californium	99 ES Einstenium	100 FM Fermium	101 MD Mendelevium	102 NO Nobellium	103 LR Lawrencium

1. Objective

- **The purpose of this experiment is to synthesize alum (aluminum potassium sulfate dodecahydrate, $KAl(SO_4)_2 \cdot 12H_2O$) from aluminum cans, including purification through recrystallization.**

2. Principle (1/2)

1. Excess alkali reacts with aluminum foil, as shown in the following reaction equation:

$$2Al(s) + 2OH^{-}(aq) + 6H_2O \rightarrow 2Al(OH)_4^{-}(aq) + 3H_2(g)$$ **(eq. 7-1)**

2. When an acid is added to the solution:

$$Al(OH)_4^{-}(aq) + H^{+}(aq) \rightarrow Al(OH)_3(s)\downarrow + H_2O(l)$$ **(eq. 7-2)**

3. If additional acid is added, $Al(OH)_3$ will convert to Al^{3+} ion and dissolve in the acidic solution.

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

2. Principle (2/2)

4. When alkali is added to $Al(OH)_3$, it reacts to form soluble $Al(OH)_4^-$ ions.

$$Al(OH)_3(s) + OH^-(aq) \rightarrow Al(OH)_4^-(aq) \qquad \text{(eq. 7-4)}$$

5. Substances like $Al(OH)_3$ which can react with both acids and bases, are known as ***amphoteric*** substances. Other examples of amphoteric compounds include $Sb(OH)_3$, $Sn(OH)_2$, $Sn(OH)_4$, $Pb(OH)_2$, $Cr(OH)_3$, $Zn(OH)_2$, $Ga(OH)_3$, and $Ti(OH)_4$.

3. Synthesis of Aluminum Alum (1/4)

1. Aluminum alum has the general formula $M^{I}M^{III}(SO_4)_2 \cdot 12H_2O$. It is an ionic compound that crystallizes from supersaturated solutions containing *sulfate ions*, *trivalent cations* (e.g., Al^{3+}, Cr^{3+}, Fe^{3+}), and *monovalent cations* (e.g., K^+, Na^+, NH_4^+). The crystalline structure incorporates 12 water molecules of hydration.

3. Synthesis of Aluminum Alum (2/4)

2. In this experiment, potassium hydroxide (KOH) is used as the base and sulfuric acid (H_2SO_4) as the acid. Therefore, $M^I = K^+$ and $M^{III} = Al^{3+}$. Under suitable conditions—particularly through the formation of a supersaturated solution—white aluminum alum crystals can form. (aluminum potassium sulfate *dodecahydrate*, $KAl(SO_4)_2 \cdot 12H_2O$)

3. Synthesis of Aluminum Alum (3/4)

3. Aluminum alum is commonly used in water purification, dyeing, papermaking, and as a food additive, among other applications.

4. The experimental procedure is outlined below :

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

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

3. Synthesis of Aluminum Alum (4/4)

5. The overall equilibrium equation for the precipitation of aluminum alum crystals is as follows:

$$K^{+}(aq) + Al^{3+}(aq) + 2SO_4^{2-}(aq) + 12H_2O(l) \rightarrow KAl(SO_4)_2{\cdot}12H_2O(s)$$

4. Recrystallization, Separation, Purification, and Crystal Growing Techniques (1/2)

1. Because different substances exhibit varying solubilities under the same conditions, this property can be utilized to separate and purify substances—a process known as **the crystallization method**.
2. The two most commonly used crystallization techniques are:
   1) **Changing the temperature** to decrease the solubility of the solute, leading to supersaturation and the crystallization of the solute.
   2) **Adding a second solvent** in which the solute is less soluble, thereby reducing its overall solubility and causing it to crystallize out from the mixed solution.

4. Recrystallization, Separation, Purification, and Crystal Growing Techniques (2/2)

3. **In this experiment, we take advantage of aluminum alum's greater solubility in hot water compared to cold water. By using hot water as the solvent and applying recrystallization techniques, we purify the substance and grow single crystals, enabling us to recover pure aluminum alum and observe its crystal structure.**

5. Equipment and Chemicals (1/2)

In cabinet	Provided by TA
Hot plate x 1	Water-jet Pump x 1
Glass Stirring Rod x 1	Vacuum Filtration Flask x 1
Graduated Cylinder 100 mL x 1	Büchner funnel x 1
Beakers 100 mL x 2	Filter paper & sandpaper
Funnel x 1	Sample bottle, labeling paper
Aluminum can x 1	Scissors x 1

5. Equipment and Chemicals (2/2)

Provided by TA
9.0 M H_2SO_4(*aq*)***
1.4 M KOH(*aq*)**
EtOH*/H_2O (2：l, v/v) mixed solution

*：Corrosive *：Toxic *：Dehydrating

Ref. :https://ghs.osha.gov.tw/CHT/masterpage/index_CHT.aspx

6. Experimental Procedures

1. Please prepare an aluminum can (not an iron can) in advance.
2. Cut a piece approximately 5 cm × 5 cm in size and polish both the inner and outer surfaces with sandpaper until smooth. Then, cut the piece into small fragments. This preparation must be completed before the lab session.

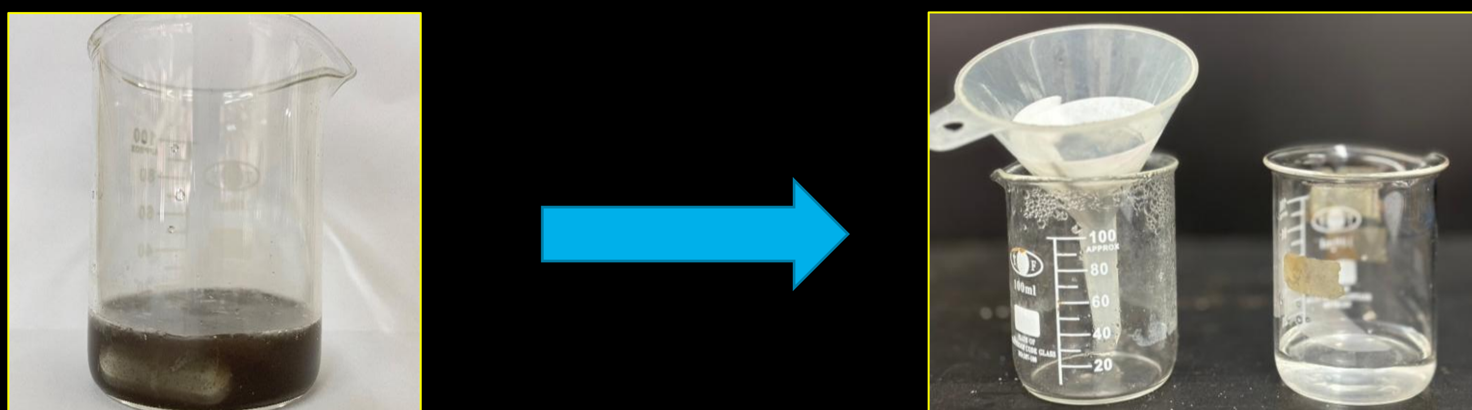
   Note: *Be sure to polish thoroughly to remove paint and coatings, as these can produce unpleasant odors when heated.*

3. Weigh the aluminum piece prepared in **Step 2** to determine its exact mass.

4. Place the aluminum in a 100 mL beaker, add 25 mL of 1.4 M KOH (aq), and heat the mixture to approximately 70 °C using a magnetic stirrer inside a fume hood to facilitate the reaction.

   Note: ***This reaction releases hydrogen gas, which can form an explosive mixture with air. To ensure safety, perform the experiment strictly within a fume hood and keep it away from any ignition sources.***

5. During the reaction, observe the aluminum piece as it becomes buoyant in the solution. The reaction is complete when hydrogen gas bubbling ceases.
6. Filter the hot solution using gravity filtration. Using a dropper, add approximately 5 mL of deionized water to rinse the beaker. Pour the rinse water into the funnel to ensure complete transfer of the solution for filtration. (Figure 1)



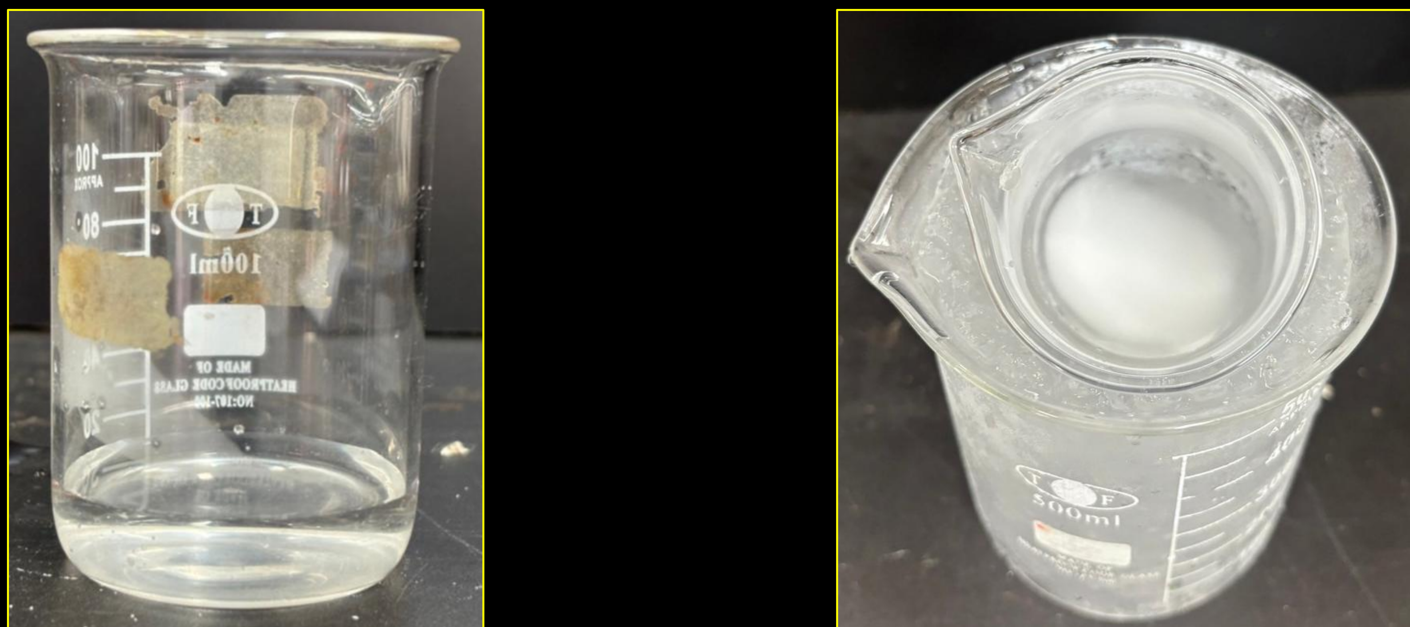
Before filtration

After filtration

Figure 1. Comparison of the solution before and after filtration.

7. **Carefully pour the clear filtrate into a 100 mL beaker. Place the beaker in an ice bath, then slowly add 12 mL of 9 M H_2SO_4 dropwise. (Figure 2)**

Note: ***The addition of acid is exothermic and may generate heat—proceed with caution.***



Before **After**

Figure 2. Comparison of the solution before and after the addition of sulfuric acid.

8. **If white $Al(OH)_3$ precipitate remains after acid addition, gently heat the mixture on a magnetic stirrer to dissolve it. If other insoluble impurities are present, filter them out *using gravity filtration* while the solution is still hot. Do not use vacuum filtration.**

Note: *Ensure the final volume of the solution remains around 15 mL to maintain proper saturation—this is essential for successful crystallization of aluminum alum.*

9. Pour the clarified solution from Step 8 (containing Al^{3+}, K^{+}, and SO_4^{2-} ions) into a clean beaker and allow it to cool to room temperature. If crystals do not form, gently scratch the inner walls of the beaker with a glass stirring rod to induce crystallization. Then, place the beaker in an ice-water bath to complete the crystallization of aluminum alum. (Figure 3)

10. Collect the crystals using vacuum filtration. Use a glass mortar to gently crush and spread the product evenly. Rinse both the beaker and the funnel contents with approximately 12 mL of a 2:1 (v/v) mixture of alcohol and deionized water, added in portions. Continue vacuum filtration for about 10 minutes to dry the product, then weigh it accurately. (Figure 4)

Figure 3. Solution after ice bath cooling

After vacuum filtration

Figure 4. Crystallized aluminum alum Product

11. **Weigh 2.5 g of the crystalline product obtained in Step 10 and transfer it to a 100 mL beaker. Add 20 mL of deionized water and heat the mixture while stirring until all solids dissolve and the solution reaches a gentle boil.**

Note: ***Avoid overheating. Ensure the final volume remains 20~25 mL to prevent excessive formation of small crystals (from too high a concentration) or failure to crystallize (due to over-dilution).***

12. **While the solution is still hot, perform gravity filtration to remove any remaining impurities, collecting the filtrate in a clean sample bottle. Allow the solution to cool and crystallize undisturbed over the course of one to two weeks.**

Note: ***Obtain labeling paper and a 20 mL sample vial from the teaching assistant. Clearly write your department and group number on the label, then submit the labeled sample vial to the teaching assistant.***

13. Remove the recrystallized crystals and examine their shape, color, and size. Submit the sample to the teaching assistant for evaluation, as illustrated in Figure 5.

Figure 5. Recrystallized aluminum alum crystals

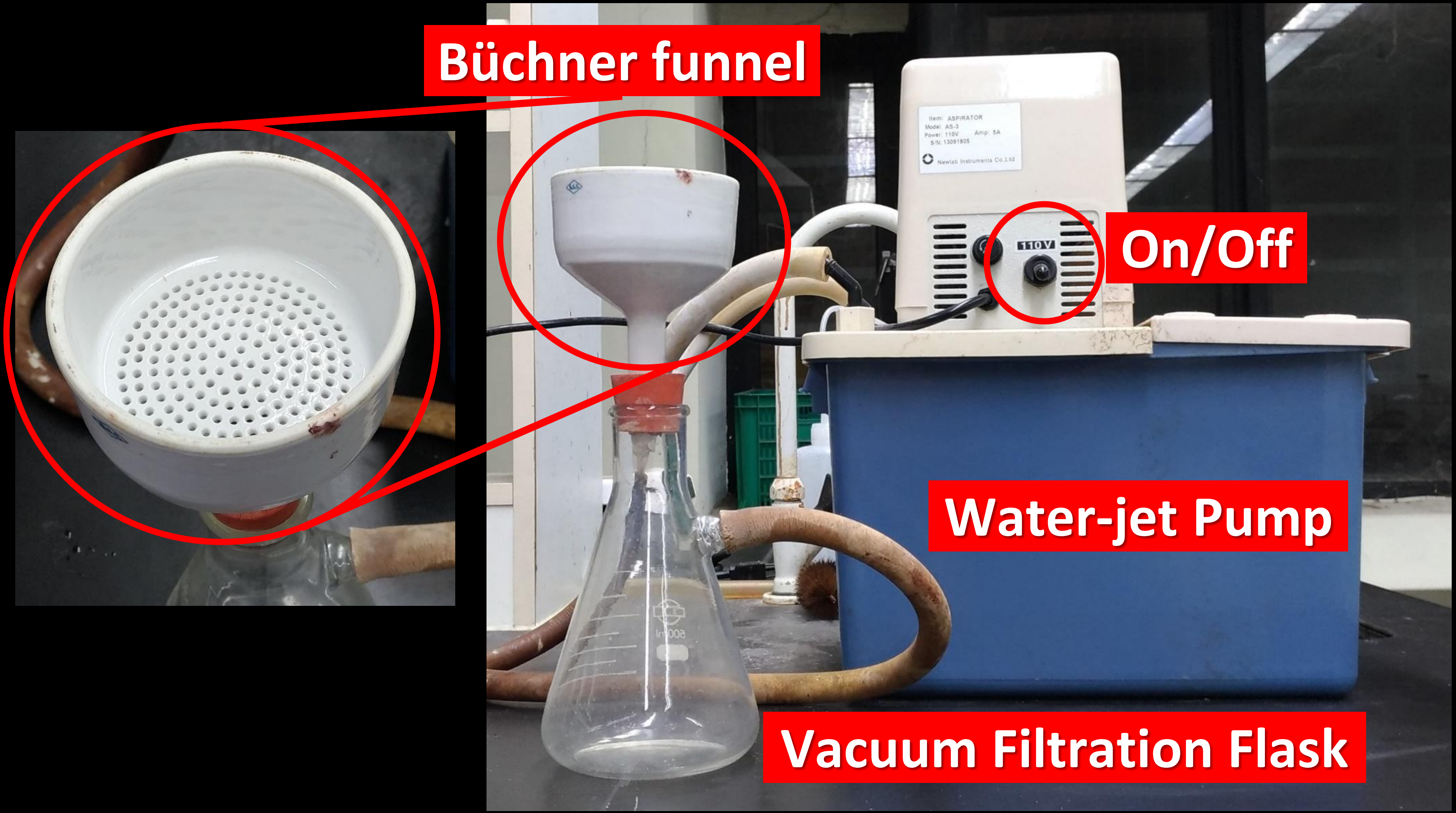


Figure 6. The vacuum filtration apparatus

7. Precautions

1. Hydrogen gas is released when aluminum reacts with aqueous KOH. Since hydrogen mixed with air can form an explosive mixture, it is essential to carry out the reaction in a fume hood.

2. Thoroughly polish the aluminum piece beforehand to remove any paint or coating, which may produce unpleasant odors when heated.

3. **This experiment involves the use of 9 M H_2SO_4 and 1.4 M KOH, both of which are *toxic* and *highly corrosive*. Exercise extreme caution when handling these chemicals. All waste liquids must be disposed of in the designated waste containers—do not pour them down sinks or drains.**

4. **Collect all solid waste generated during the experiment and place it in the designated beaker provided by the teaching assistant. The teaching assistant will manage proper disposal.**

5. **Always wear a lab coat, goggles, gloves, and a mask during the lab session.**
6. **All chemicals used in the laboratory are toxic and potentially hazardous. Please follow all laboratory safety regulations and adhere to the instructions of the teaching assistants to ensure a safe working environment**
7. **Removal of any chemicals or laboratory equipment from the lab is strictly prohibited.**

6. Experimental data

1. Mass of aluminum foil used: ___________________________

2. Mass of aluminum alum obtained: ____________________

3. Mass of recrystallized aluminum alum: _______________

4. Photograph the recrystallized aluminum alum sample.

7. Questions

1. In this experiment, if the clarified solution containing Al^{3+}, K^{+}, and SO_4^{2-} cools to room temperature without forming crystals, gently scratching the inner wall of the beaker with a glass stirring rod can induce crystallization. Why does this occur?

Ref. https://www.ch.ntu.edu.tw/~genchem99/qalab/lab17.htm

2. **In this experiment, aluminum alum is introduced. In addition to aluminum alum, identify two other types of alums. Please provide their chemical formulas and cite a reference source.**

Ref. :https://zh.wikipedia.org/wiki/%E7%9F%BE

3. **In this experiment, which filtration method—gravity filtration or vacuum filtration—is more effective, and why?**

Ref. :https://teaching.ch.ntu.edu.tw/gclab/qalab/lab17.htm

4. **In this experiment, why is crystallization induced by cooling the solution in an ice bath? Would crystallization still occur if the solution were left to cool at room temperature instead?**

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