Collect:
• Büchner funnel, rubber stopper, suction flask
• Sandpaper, filter paper, iron ring, and dropper
• Styrofoam cup, 30 mL beaker
• Scissors
• Cotton string, aluminum foil, toothpick, crystal seed (obtain when doing part II)

Prepare:
• Prepare your own aluminum can (scrape off paint and inside coating with sandpaper)
• Funnel, stirring rod, graduated cylinder
• 100 mL beaker
Objective and Techniques

Objective:
- Recycle scrap aluminum cans into alum
- To prepare chromium-aluminum alum by displacement

Techniques:
- Suction filtration
- Gravity filtration
- Recrystallization

Scheme of procedure:

$$\text{KOH}_{(aq)} \xrightarrow{\Delta} \text{Al(OH)}_4^- \xrightarrow{\Delta} \text{Al}^{3+}(\text{SO}_4^{2-}, \text{K}^+, \text{H}_2\text{O})$$

$$\xrightarrow{\text{Cool}} \text{KAl(SO}_4)_2 \cdot 12\ \text{H}_2\text{O}_{(s)}$$
Procedure I. Preparation of Alum

Step 1. $\text{Al}_\text{(s)} \rightarrow \text{Al(OH)}_4^-\text{(aq)}$

$\text{Al}_\text{(s)} \overset{\Delta}{\rightarrow} \text{Al(OH)}_4^-\text{(aq)}$
$\text{H}_2\text{SO}_4\text{(aq)} \overset{\Delta}{\rightarrow} \text{Al}^{3+}\text{(SO}_4^{2-}, \text{K}^+, \text{H}_2\text{O}) \overset{\text{cool}}{\rightarrow} \text{KAl(SO}_4^2\cdot 12 \text{H}_2\text{O}_\text{(s)}$

$2\text{Al}_\text{(s)} + 2\text{KOH}_\text{(aq)} + 6\text{H}_2\text{O}_\text{(l)} \rightarrow 2\text{K}^+_\text{(aq)} + 2\text{Al(OH)}_4^-\text{(aq)} + 3\text{H}_2\text{(g)}$

- Scrape clean both sides of aluminum piece and cut into small pieces
- Weigh about 0.5 g, record accurate weight
- Heat in fume hood
- 4 beakers share 1 hot plate
- Set hot plate on 2 or 60 °C
- Reaction is completed when no more $\text{H}_2\text{(g)}$ is evolved
Procedure I. Preparation of Alum

Step 2. Suction filtration & collect filtrate

$$\text{Al}_\text{(s)} + \text{KOH}_{\text{(aq)}} \xrightarrow{\Delta} \text{Al(OH)}_4^- + \text{H}_2\text{SO}_4_{\text{(aq)}} \xrightarrow{\Delta} \text{Al}^{3+} (\text{SO}_4^{2-}, \text{K}^+, \text{H}_2\text{O}) \xrightarrow{\text{cool}} \text{KAl(SO}_4)_2 \cdot 12 \text{H}_2\text{O}_{\text{(s)}}$$

- Fill water in water aspirator (come in from bottom and flow out from top); test the suction
- Water-trap bottle & suction flask should be fixed with extension clamp
- Use rubber stopper to connect Büchner funnel tightly with suction flask
- Select 55-mm filter paper to cover the perforations of Büchner funnel, moisten with small amounts of solvent, and tightly sealed against the bottom by closing the 2-way valve to apply suction
- Pour the solution in to finish filtration
- Pour filtrate into 100 mL beaker
- Rinse suction flask once with 1 mL of water
Procedure I. Preparation of Alum

Step 3. Neutralization

\[ \text{Al}_2(s) + \text{KOH(aq)} \xrightarrow{\Delta} \text{Al(OH)}_4^{-} \xrightarrow{\Delta} \text{Al}^{3+} + \text{SO}_4^{2-}, \text{K}^+, \text{H}_2\text{O} \xrightarrow{\text{cool}} \text{KAl(SO}_4\text{)}_2 \cdot 12\text{H}_2\text{O}_2(s) \]

- Cool the beaker in water bath (for exothermic reaction)
- White, gelatinous precipitate, Al(OH)$_3$, appears
- Heat until all precipitate dissolves
- Al(H$_2$O)$_6^{3+}$ forms
- Volume should be less than 30 mL

Add H$_2$SO$_4$

Heat to boil

Filtrate Al(OH)$_4^-$
**Procedure I. Preparation of Alum**

### Step 4. Crystallization

\[
\text{Al}_{(s)} + \text{KOH}_{(aq)} \xrightarrow{\Delta} \text{Al(OH)}_4^- \xrightarrow{\Delta} \text{Al}^{3+} (\text{SO}_4^{2-}, \text{K}^+, \text{H}_2\text{O}) \xrightarrow{\text{cool}} \text{KAl(SO}_4\text{)}_2 \cdot 12\text{H}_2\text{O}_{(s)}
\]

- Cool the solution to r.t.
- If no crystals are formed, scrape the inside wall of the container with a glass rod to initiate crystallization
- Ice-water bath to allow complete crystallization of alum

Use **Styrofoam cup** to contain ice-water bath
Step 5. Suction filtration to collect products

- Wash the funnel and get a new filter paper
- Stir and mix the crystals with solution, then pour out immediately
- Wash the remaining crystals in beaker with 3~4 mL of ethanol/water solution (1:1)
  * Do not use too much ethanol/water solution for the alum is water soluble
- Allow the crystals to vacuum dry for about 10 min.
- Measure weight and calculate the percent yield
- Recycle the alum (approx. 5~7 g)
  * Do not heat to dry the alum

\[
\begin{align*}
\text{Al}_3(\text{s}) & \xrightarrow{\Delta} \text{Al(OH)}_4^- & \xrightarrow{\Delta} \text{Al}^{3+}(\text{SO}_4^{2-}, \text{K}^+, \text{H}_2\text{O}) & \xrightarrow{\text{cool}} \text{KAl(SO}_4\text{)}_2 \cdot 12 \text{H}_2\text{O}(\text{s})
\end{align*}
\]
Calculation: Percent Yield

I. Preparation of Alum

- Mass of aluminum strips: $W_1$ (g)
- Moles of Al: $n_1 = W_1/26.98$ (mol)
- Mass of alum obtained: $W_2$ (g)
- Theoretical yield of alum: $W_3 = n_1 \times 474.21$ (g)
- Percent yield of alum: $W_2/ W_3 \times 100\% = P \%$

Molar mass:

Al: 26.98 g/mol
KAl(SO$_4$)$_2$·12H$_2$O: 474.21 g/mol
Procedure II.
Synthesis of Al-Cr Alum

- In potassium alum, K⁺ or Al³⁺ can be replaced by other cations
- In this experiment, Cr³⁺ is used to replace parts of Al³⁺
- Cr³⁺ contains d-orbital electrons, hence chromium alum is colored

\[ \text{KAl(SO}_4\text{)}_2 \cdot 12\text{H}_2\text{O(s)} + \text{KCr(SO}_4\text{)}_2 \cdot 12\text{H}_2\text{O(s)} \rightarrow 2\text{K[}(\text{Al,Cr})(\text{SO}_4\text{)}_2] \cdot 12\text{H}_2\text{O(s)} \]
Procedure II.
Synthesis of Al-Cr Alum

Step 1. Preparing saturated solution

- According to table 1, measure exact amounts of alum, Cr-alum, and deionized water in a 100 mL beaker
- Heat and stir the mixture until all solids dissolve, filter by gravity filtration while hot
- Collect filtrate in a clean 30 mL beaker and cool the soln slowly

<table>
<thead>
<tr>
<th>Group*</th>
<th>Alum (g)</th>
<th>Cr-alum (g)</th>
<th>Deionized water (mL)**</th>
</tr>
</thead>
<tbody>
<tr>
<td>A (1)</td>
<td>4</td>
<td>0</td>
<td>25</td>
</tr>
<tr>
<td>B (2)</td>
<td>3</td>
<td>1</td>
<td>25</td>
</tr>
<tr>
<td>C (3)</td>
<td>3</td>
<td>2</td>
<td>25</td>
</tr>
<tr>
<td>D (4)</td>
<td>3</td>
<td>3</td>
<td>25</td>
</tr>
</tbody>
</table>

*Every 4 groups is a cycle; ex, group 1 is A, group 2 is B… etc.

**Measure with graduated cylinder
Gravity Filtration

1. Heat to boil; 2. filter while hot; 3. cool filtrate slowly
Procedure II. Synthesis of Al-Cr Alum

Step 2. Preparing “seed” crystal

- Take a 10 cm-cotton string
- Tie one end to toothpick and one end to the crystal
- Cut off remaining excessive string
- Coat the string with Vaseline to prevent crystal growing on string
  * Do not smudge the seed crystal with Vaseline
- Allow the soln to cool to r.t.
- Suspend seed crystal in the center of the soln
- Fix the position of toothpick with tape
- Cover the beaker with aluminum foil and punch several holes
- Grow the crystal for 2-3 weeks
Setup

- Cover with aluminum foil
- Fix with tape
- Cover with Vaseline
- Seed Crystal

Cotton string on toothpick needs to be fixed also.
Procedure II.
Synthesis of Al-Cr alum

Step 3. After crystallizing for 2-3 weeks
- Take out crystal
- Observe the size and shape of crystal
- Remaining solution that contains heavy metal Cr\(^{3+}\) should be recycled
- Crystals at the bottom of the 30-mL beaker can be saved and used as seed crystals in the future

Pure alum with octahedral shape
Chrome Alum added. Purple crystal
Vaseline are not applied thoroughly to cotton string
Soln is unsaturated; or did not place the crystal in a cooled soln
Notice

- Aluminum pieces can cut through skin, handle with care
- Recycle scrap aluminum cans, do not throw into garbage bin
- Notices on using water aspirator:
  - Cold water enters from the bottom pipe and exits through the top pipe
  - Reduce the flow rate of cooling water after filling with water to avoid flooding
  - The pump needs to be plugged in and turned on power
  - The trap-bottle and suction flask needs to be fixed with universal clamp
- Do not mistake suction filtration and gravity filtration
- Wash and clean suction flask and Büchner funnel after class
- Recycle: solution containing Cr need to be recycled