Preparation of Alum

Last revised: 2024/9/16

Prelab Preparation

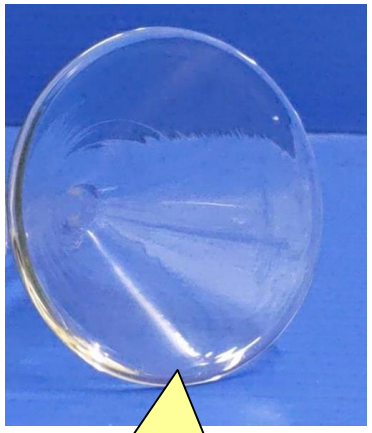
- Cut a 5 x 5 cm^2 Al foil. **Be careful for sharp edges**
- Use a sandpaper to remove the coating on both sides of aluminum foil

- Cut the aluminum foil to small pieces
- Weigh ~ 0.5 g of Al, transfer to a 100 mL beaker, and add **25 mL** 1.4 M KOH
- Heat the mixture in fume hood to 60°C (heating dial = Low or 2)

Prepare Lab Apparatus

Collect and check the following items

- ❑ Rubber stopper, **Büchner funnel**, and **filter flask (wash thoroughly)**
- ❑ **30-mL beaker (wash thoroughly)**
- ❑ Iron ring and three-prong clamp
- ❑ Styrofoam cup
- ❑ Dropping pipet
- ❑ Funnel



For gravity filtration

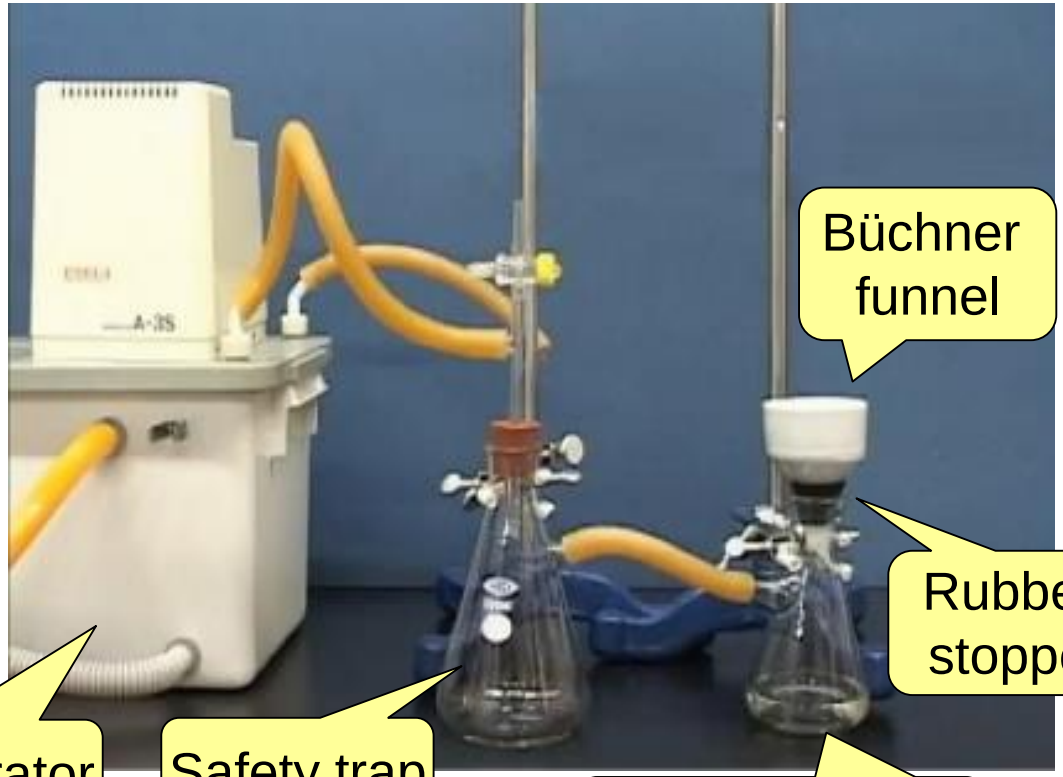
From your personal equipment

- ❑ Three 100-mL beakers
- ❑ Glass rod
- ❑ 50-mL graduated cylinder
- ❑ Tweezers

Objective and Principles

- **Objective:**
  - Synthesize **alum ($KAl(SO_4)_2 \cdot 12H_2O$)** from aluminum cans
  - Prepare a single crystal of **Al-Cr alum**

- **Lab techniques:**
  - **Vacuum filtration**
  - **Gravity filtration**
  - Recrystallization
  - Stirrer/hot plate
  - Dispenser



Experiment Tasks

Task 1

Amphoteric substance

$$\mathbf{Al(s)} \xrightarrow[\Delta]{KOH(aq)} [Al(OH)_4]^-(aq) \xrightarrow[\Delta]{H_2SO_4(aq)} Al^{3+}(SO_4^{2-}, K^+, H_2O) \xrightarrow{Cooling} \mathbf{KAl(SO_4)_2{\cdot}12H_2O(s)}$$

Task 2

These two tasks can be carried out in parallel

$$x\ KAl(SO_4)_2{\cdot}12H_2O(s) + (1-x)\ K\mathbf{Cr}(SO_4)_2{\cdot}12H_2O(s) \longrightarrow \mathbf{K[Al_xCr_{1-x}(SO_4)_2]{\cdot}12H_2O(s)}$$

- Cr^{3+} replaces Al^{3+} in the crystal structure of alum
- *d* electrons of Cr^{3+} leads to purple color (crystal field theory)

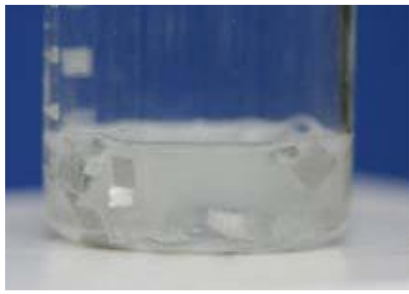
Step 1.1: Dissovling Al in KOH

$2Al(s) + 2KOH(aq)$ + $6H_2O(l) \rightarrow 2K^+(aq)$ + **$2[Al(OH)_4]^-(aq) + 3H_2(g)$**

- Cut a 5 x 5 cm^2 foil from the aluminum can, then use a sandpaper to remove the coating on both sides of aluminum foil
- Using a scissor to cut the aluminum foil to small pieces
- Weigh ~0.5 g of Al foil, then transfer into a 100 mL beaker with 25 mL of 1.4 M KOH. Heat the mixture in fume hood to 60°C (heating dial = 2)
- **Reaction is completed when no more $H_2(g)$ is evolved**







- ✓ The aluminum foil have sharp edges and need to be handled with care (wear cloth gloves)
- ✓ Dispose the remaining aluminum cans into the recycle bin

Step 1.2: Vacuum Filtration

- Fill the tank of water aspirator with tap water
- Maintain a slow overflow rate
- **Wash the filter flask** and set up the apparatus
- Use a **55-mm** diameter filter paper to cover the perforations of Büchner funnel
- **Rinse the filter paper** with some DI water
- **Close the 2-way valve** on the safety trap
- **Test the suction** with your palm
- Pour the $[Al(OH)_4]^-$ solution into the Büchner funnel
- Open the 2-way valve to **release vacuum**
- Pour the filtrate into a **clean 100 mL beaker**
- Rinse the suction flask with ~ 1 mL DI water, and combine with the filtrate

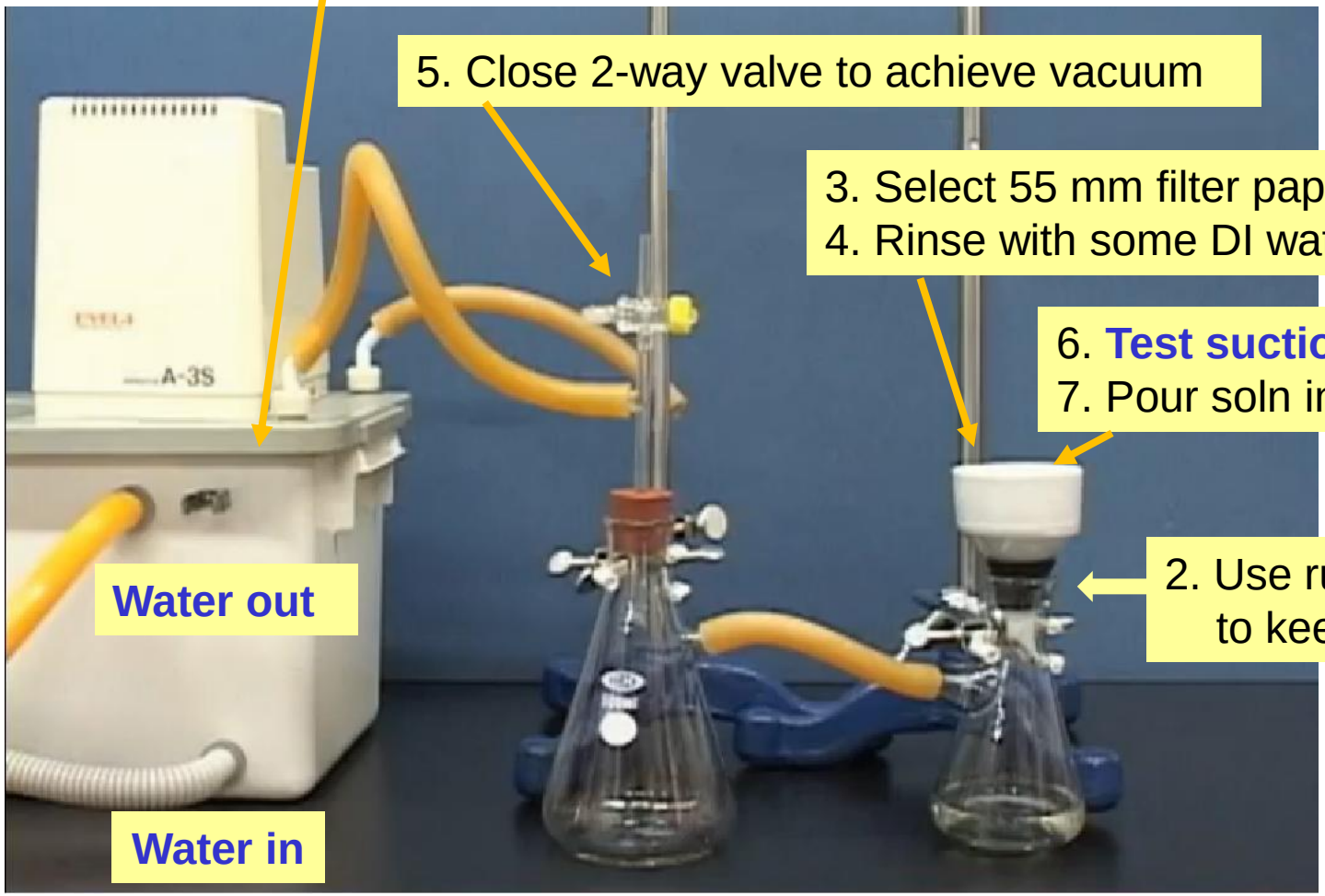
✓ Both the safety trap flask and the filter flask should be fixed

✓ Place a rubber gasket cone between Büchner funnel and the filter flask

✓ The tip of Büchner funnel should point away from the short glass tube

Setup of Vacuum Filtration

1. Fill the tank with water, maintain a slow overflow rate; turn on the power



5. Close 2-way valve to achieve vacuum

3. Select 55 mm filter paper
4. Rinse with some DI water

6. **Test suction**
7. Pour soln into Büchner funnel

2. Use rubber stopper to keep air tightness

Water aspirator Safety trap Filter Flask (fixed)

Step 1.3: Neutralization with H_2SO_4

- $[Al(OH)_4]^-(aq) + \mathbf{H_2SO_4(aq)} \rightarrow \mathbf{Al(OH)_3(s)} + SO_4^{2-}(aq) + H_2O(l)$
- $Al(OH)_3(s) \xrightarrow{\text{Excess } H_2SO_4\,,\,\Delta} \mathbf{[Al(H_2O)_6]^{3+}(aq)}$

Add H_2SO_4

Heat to boil

- Collect filtrate in a beaker
- Place the 100 mL beaker in a water bath
- **Dispenser 10 mL of 9 M H_2SO_4 to the filtrate in the beaker**
- White gelatinous $Al(OH)_3$ appears

- Use a hot plate in fume hood to boil the solution and dissolve $Al(OH)_3$ to **$[Al(H_2O)_6]^{3+}$**
- Volume should be **less than 30 mL**

Step 1.4: Crystallization

$$Al^{3+}(SO_4^{2-}, K^+, H_2O) \xrightarrow{\text{Cooling}} \mathbf{KAl(SO_4)_2 \cdot 12H_2O(s)}$$

Cool to r.t.

Ice-water bath

- Let the solution **cool naturally to room temperature and grow crystal**
- If no crystal can be seen, **scrap** the inner wall of the beaker near the liquid surface with a glass rod to generate nucleation centers
- Place the beaker into a mixed ice/water bath
- Allow the crystallization of alum to complete
- Use a test tube to take 4 mL of ethanol/water solution (1:1 v/v) and cool it in the ice-water bath

Step 1.5: Vacuum Filtration

- Use tweezers to remove the used filter paper
- **Wash the Büchner funnel**
- Install a new 55-mm filter paper
- Stir the crystal precipitates in the beaker with a glass rod, then pour the solution **quickly** onto the funnel
- Use a drop pipet to **rinse the beaker and alum crystal** with cooled ethanol/water solution

- Vacuum dry the alum crystals for ~10 min.
- Transfer the dry alum crystals on a weighing paper and measure the weight
- Calculate the percent yield
- Have your alum product **check by TA**, and recycle into the designated bottle

Experiment Task 2

Task 1

$$Al(s) \xrightarrow[\Delta]{KOH(aq)} [Al(OH)_4]^-(aq) \xrightarrow[\Delta]{H_2SO_4(aq)} Al^{3+}(SO_4^{2-}, K^+, H_2O) \xrightarrow{Cooling} KAl(SO_4)_2{\cdot}12H_2O(s)$$

Task 2

These two tasks can be carried out in parallel

$$x\ KAl(SO_4)_2{\cdot}12H_2O(s) + (1-x)\ K\mathbf{Cr}(SO_4)_2{\cdot}12H_2O(s) \longrightarrow \mathbf{K[Al_xCr_{1-x}(SO_4)_2]{\cdot}12H_2O(s)}$$

- Cr^{3+} replaces Al^{3+} in the crystal structure of alum
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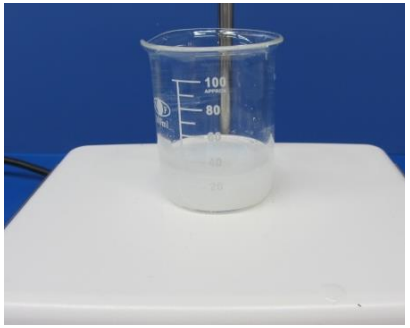
Step 2.1: Prepare Saturated Solution

- Measure **alum** and **chrome alum** according to the table shown below, record the exact weights and transfer to a 100 mL beaker
- Use a graduated cylinder to measure **25 mL DI** water into the beaker
- **Heat** and stir the solution on a hot plate **to boil** (dial = **4-5**, operate in fume hood); and all the solids are dissolved

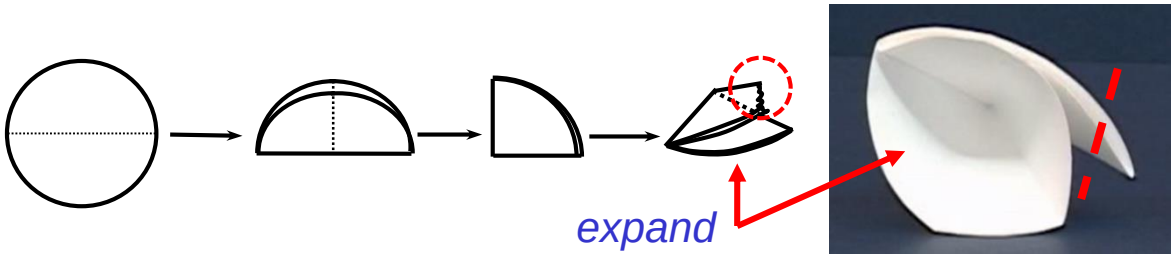
Group*	Alum (g)	Cr-alum (g)	DI water (mL)
1	**3.5**	**0**	**25**
2	**3.0**	**0.5**	**25**
3	**3.0**	**1.0**	**25**
4	**3.0**	**2.0**	**25**

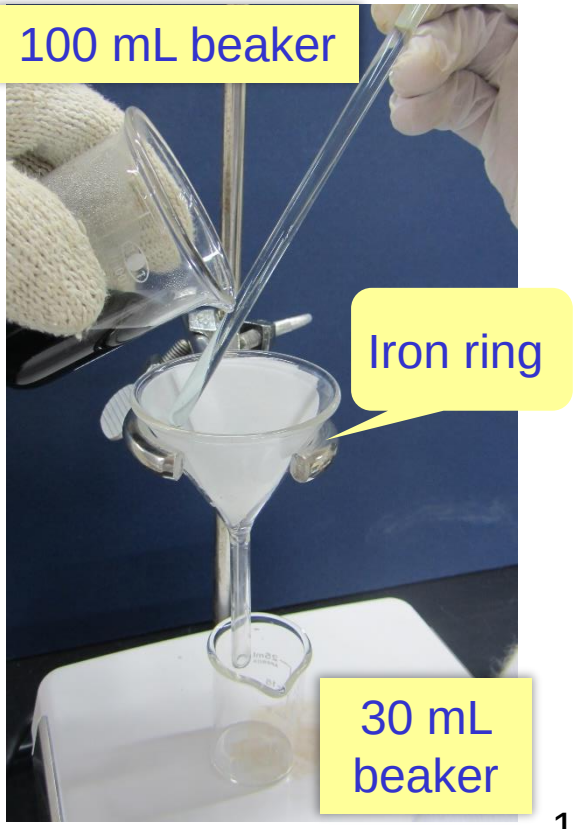


* TA will instruct on how these groups are assigned

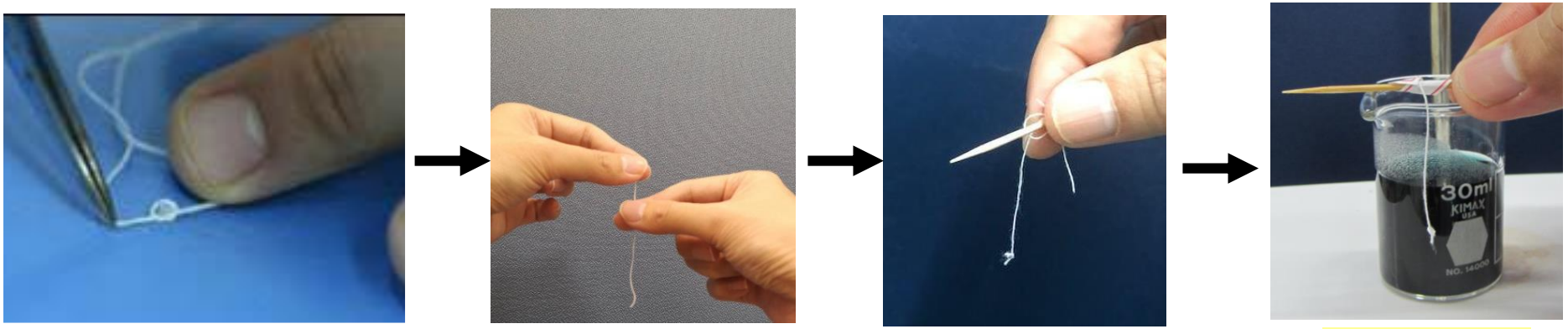
Step 2.2: Gravity Filtration

- Take a **110-mm diameter filter paper**, fold it twice and tear off a small piece at one of the two thin outside corners
- Use a **ring clamp** to set up a glass funnel; expand the filter paper and install it in the funnel
- Wash a **30 mL beaker** thoroughly and place it below the funnel; the tip of funnel should touch the inner wall of the beaker
- While the Al/Cr alum solution is **still hot**, use a glass rod to guide the solution into the glass funnel
- Let the filtrate cool down slowly





Step 2.3: Prepare Seed Crystal



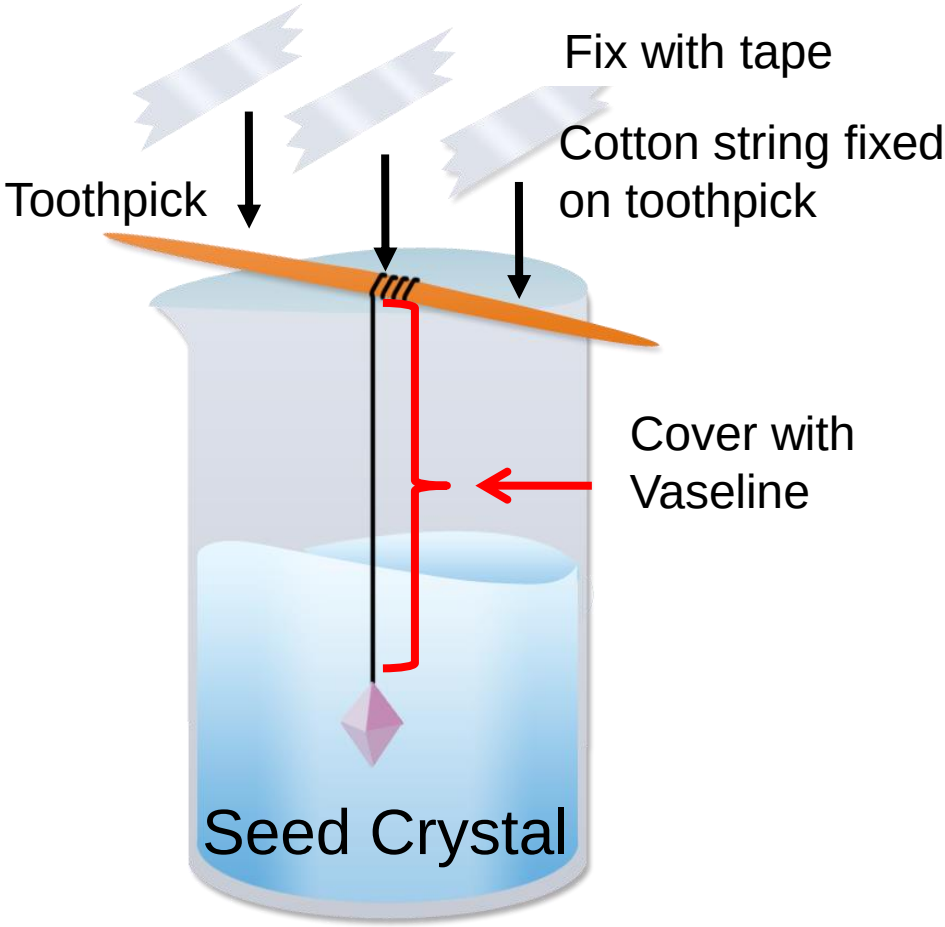
20-25 mL

- Cut a cotton string to ~10 cm length
- On one end of the string, use a single knot to tie a seed crystal securely
- Cut the extra string off with a scissor
- Use fingers to coat the cotton string (but not the seed crystal) with Vaseline (crystallization may take place on any uneven surface)
- Tie the other end of string to a tooth pick

✓ Ideal seed crystal: > 1 mm in size, has only one octahedral crystal domain (not multiple small crystals fusing together)

Step 2.4: Recrystallization Setup

- Allow the solution to **cool down to room** temperature
- Suspend the seed crystal **in the center** of the solution
- **Fix** both ends of the tooth pick with tapes
- Give your beaker to TA to store
- Check the grown crystal after 2-3 weeks



✓ The volume of filtrate in 30 mL beaker should be about 20-25 mL

Step 2.5: Collect the Grown Crystal

- **After 2-3 weeks**, remove the crystal from the solution
- Observe the size and shape of grown crystal
- Cr^{3+}-containing solution should be recycled into the heavy metal waste bin
- Smaller crystals at the bottom of 30 mL beaker can be recycled as seed crystal candidates

Pure alum crystal (octahedral shape)

Purple colored crystal of Cr-alum

Vaseline not applied to cotton string

Solution concentration too low; or solution had not been cooled down to RT

Additional Notes

- Handle cut aluminum cans with care
- **Water aspirator:**
  - Tap water goes in from the bottom and overflows through the top hose
  - Too fast overflow rate may cause flooding
  - Is the power cord properly plugged in?
- **Check the suction** before pouring the solution
- Do not substitute gravity filtration by suction filtration
- For task II, perform the gravity filtration as soon as possible while the solution is **hot**

Clean-Up and Check-Out

- This is a **Brief Report** experiment
  - **Observation**: color, ppt, reaction time, exo- or endothermic.....
  - **Percent yield**: list calculation, s.f……
- After weighing your alum product, hand it to TA and recycle
- Recycle scrap aluminum cans
- Clean the filter flask and the Büchner funnel before and after use
- Empty the water in the safety trap and the aspirator
- Clean up the lab bench and check personal equipment inventory (have an associate TA sign the check list)
- Groups on duty shall stay and help clean up the lab

Calculate the Percent Yield

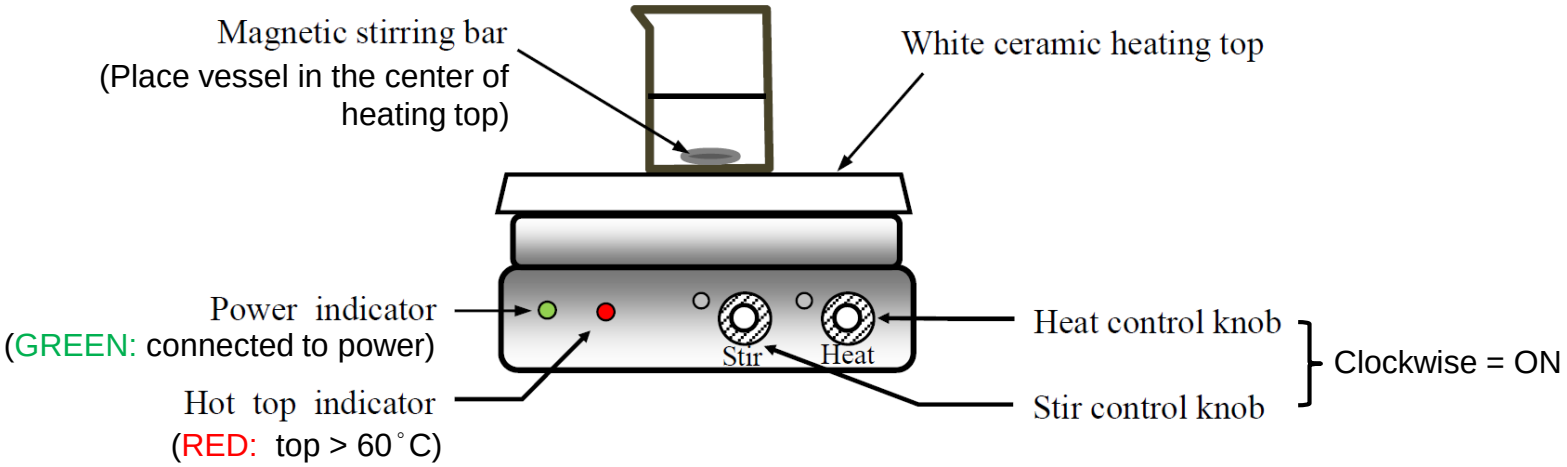
- Mass of aluminum strips: W_1 (g)
- Moles of Al: $n_1 = W_1/26.98$ (mol)
- Mass of alum obtained: W_2 (g)
- Theoretical yield of alum: $W_3 = n_1 \times 474.21$ (g)
- Percent yield of alum: $P\% = W_2/ W_3 \times 100\%$

Molar mass:

Al: 26.98 g/mol

$KAl(SO_4)_2 \cdot 12H_2O$: 474.21 g/mol

Stirrer/Hot Plate



- Connect the stirre/hot plate to a grounded 110 V power outlet (replace damaged power cord and plug immediately)
- Keep power cord away from the ceramic heating top
- Clean the heating top with non-corrosive detergent after use or when liquid spills
- NEVER heat a large amount of volatile and flammable liquid (e.g. ether, acetone) directly on the hot plate
- If the stirring bar jumps erratically, turn the stirring function off and adjust the vessel position, then restart the stirring
- Do not remove the stirring bar from solution with hand – instead use a Teflon-coated magnetic rod (“fishing pole”)

T2 Video (YouTube link)

Gravity Filtration

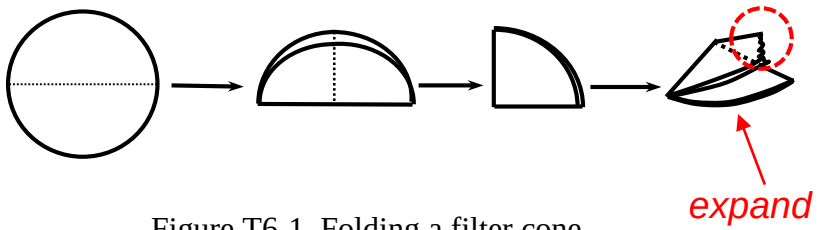


Figure T6-1 Folding a filter cone

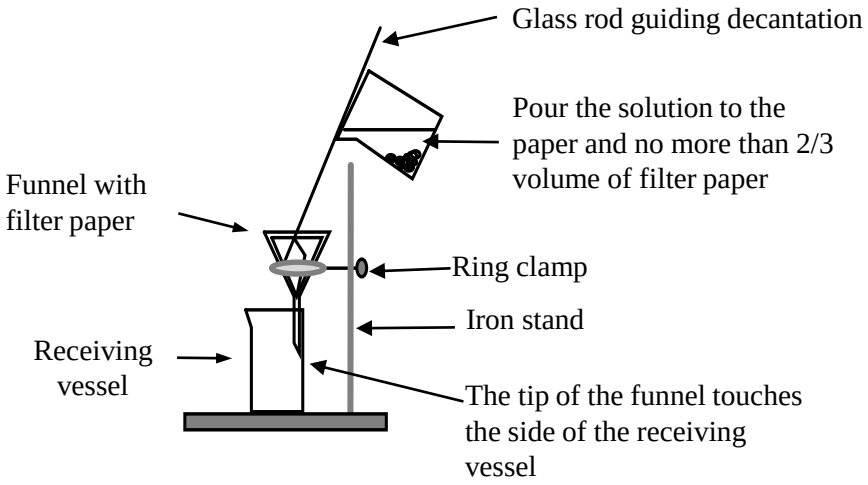


Figure T6-2 Setup of gravity filtration

- Fold a round filter paper in half for two times. Tear off a small piece at one of the two thin outside corners
- Expand the filter paper (from the intact fold) into a cone shape. Fit the filter paper into a funnel (the edge of filter paper should not exceed the top of funnel)
- Use a ring clamp to support the glass funnel. The tip of the funnel should touch the sidewall of the receiving vessel
- Pour the liquid into the paper cone (not on the glass funnel). Use a glass rod to decant the liquid
- Fill the paper cone no more than 2/3 full
- After filtration, use a tweezer to separate the filter paper from funnel (don't use hand)

T6 Video (YouTube link)

Vacuum (Suction) Filtration

- Fill the tank of water aspirator with tap water (bottom in, top out) and maintain a slow overflow rate
- Fix both the safety trap (buffer flask) and the suction flask with extension clamps
- Install a Büchner (or Hirsch) funnel on the filter flask. Use a rubber stopper or a rubber gasket cone to seal the flask

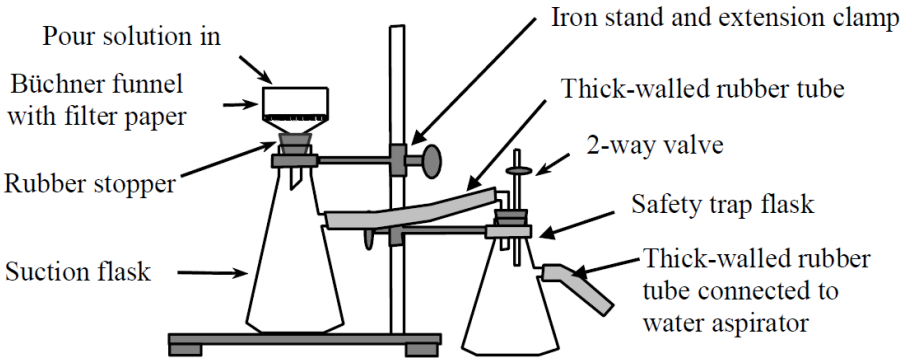


Figure T7-1 Setup of vacuum filtration

- Cover the perforations of funnel with an unfolded circular filter paper of suitable size
- Moisten the filter paper with a small amount of solvent. Switch on the water aspirator and close the 2-way valve on the safety trap (stopcock in horizontal position)
- Pour the solution onto the filter paper. Wash the precipitate with a small amount of solvent or wash liquid. Let the precipitate air-dry for ~5 minutes.
- Open the 2-way valve on the safety trap (stopcock in vertical position). Switch the water aspirator off if no one else is using it.
- Turn the water flow off and empty the water aspirator tank

T7 Video (YouTube link)

Lab Dispenser

(1) Check the pre-set volume on the dispenser. Do not change the setting unless instructed to do so

(2) Place the receiving flask under the tip of dispenser

(3) To remove the air bubbles in the dispenser, lightly pull the piston pump up and down for several times

(4) Gently pull the piston pump up until it reaches the end of travel range, then slowly push the piston down to obtain the solution