



# General Chemistry Laboratory

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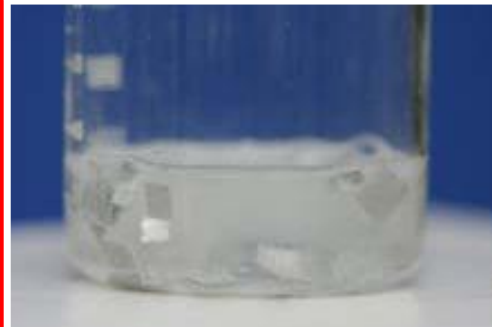
## Preparation of Alum



# Prelab Preparation



- Cut a 5 x 5 cm<sup>2</sup> 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)



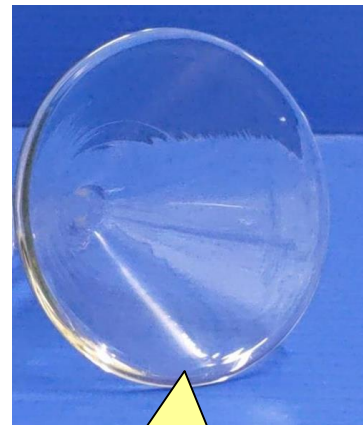
# Prepare Lab Apparatus

## Collect and check the following items

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

## From your personal equipment

- Three 100-mL beakers
- Glass funnel
- Glass stirring rod
- 50-mL graduated cylinder
- Tweezers



**For gravity  
filtration**



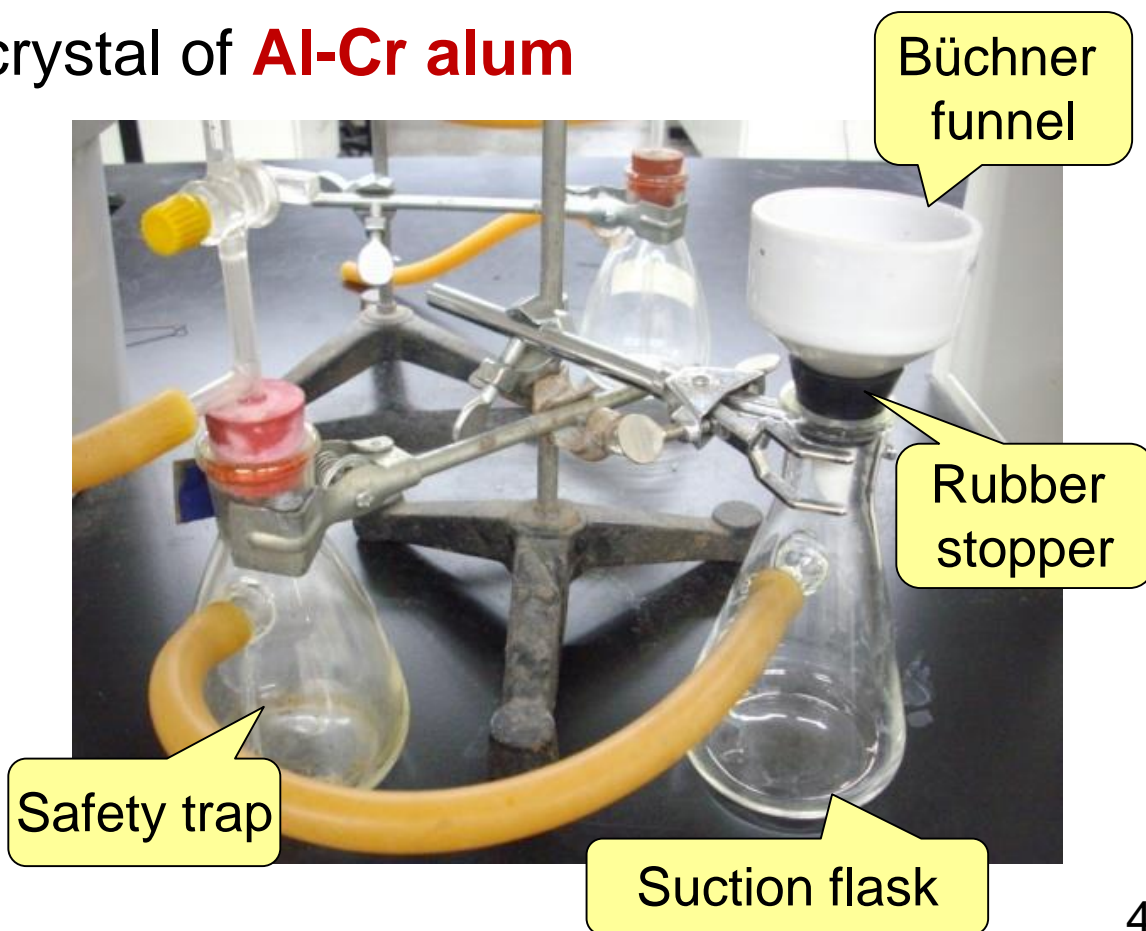
# Objective and Principles

## ■ Objective:

- Synthesize **alum** ( $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ ) from aluminum cans
- Prepare a single crystal of **Al-Cr alum**

## ■ Lab techniques:

- Vacuum filtration
- Gravity filtration
- Recrystallization
- Stirrer/hot plate
- Dispenser

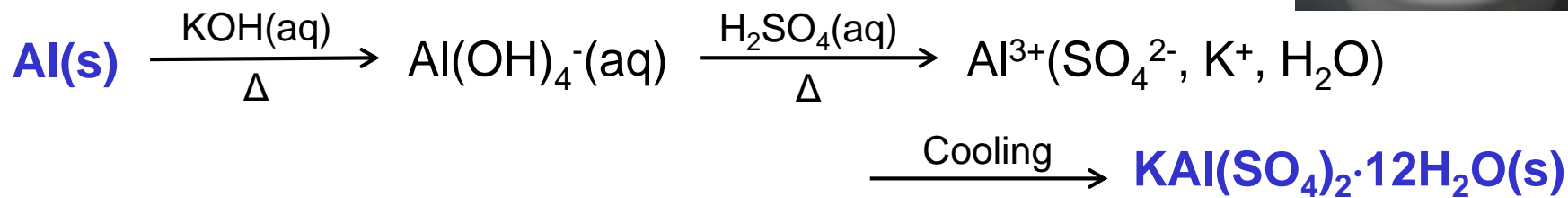




# Experiment Tasks

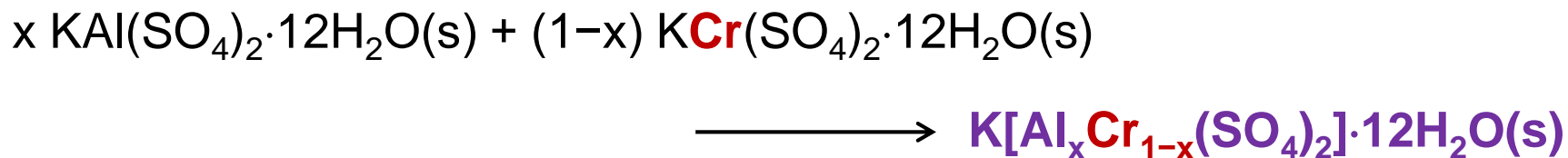
## Task 1

### Amphoteric substance



## Task 2

*These two tasks can be carried out in parallel*

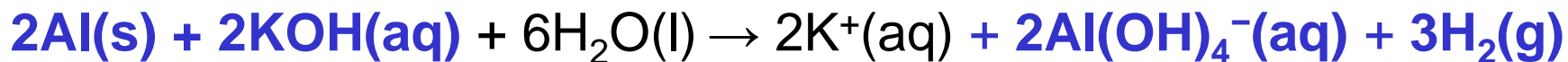


- $\text{Cr}^{3+}$  replaces  $\text{Al}^{3+}$  in the crystal structure of alum
- $d$  electrons of  $\text{Cr}^{3+}$  leads to purple color (crystal field theory)

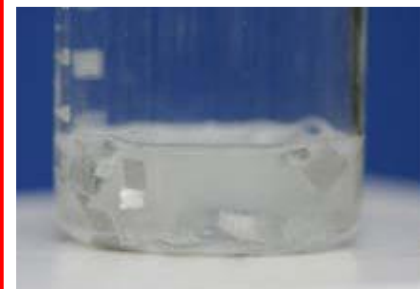




# Step 1.1: Dissolving Al in KOH



- Cut a 5 x 5 cm<sup>2</sup> 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<sub>2</sub>(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 suction 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  $\text{Al}(\text{OH})_4^-$  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  $\sim 1$  mL DI water, and combine with the filtrate



- ✓ Both the safety trap flask and the suction flask should be fixed
- ✓ Place a rubber gasket cone between Büchner funnel and the suction 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 out

Water in

Aspirator pump

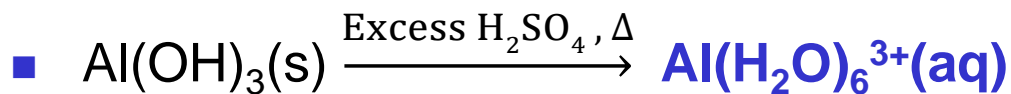
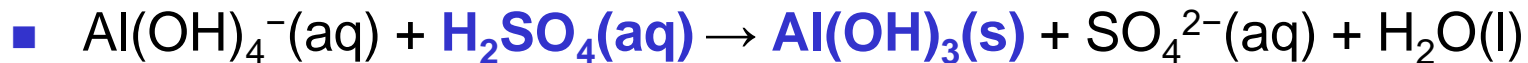
Safety trap

Suction Flask (fixed)

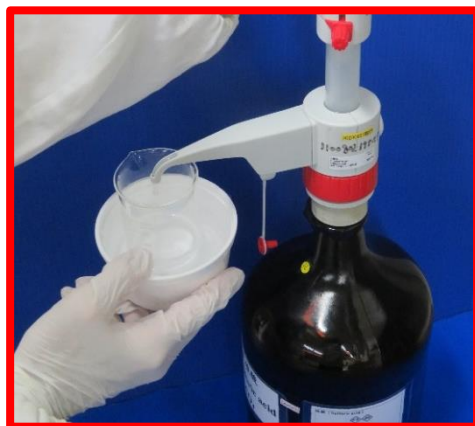




# Step 1.3: Neutralization with $\text{H}_2\text{SO}_4$



Add  $\text{H}_2\text{SO}_4$



Heat to  
boil

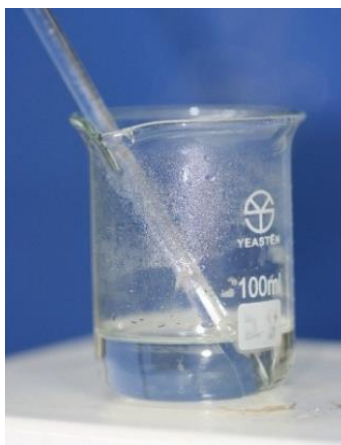
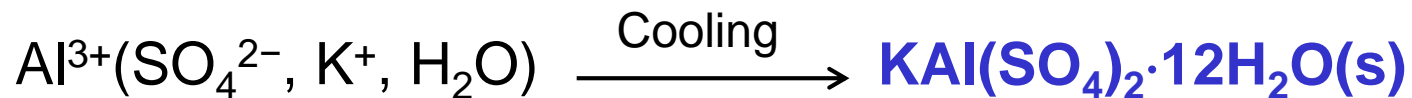


- Collect filtrate in a beaker
- Place the 100 mL beaker in a water bath
- **Dispenser 10 mL of 9 M  $\text{H}_2\text{SO}_4$  to the filtrate in the beaker**
- White gelatinous  $\text{Al}(\text{OH})_3$  appears

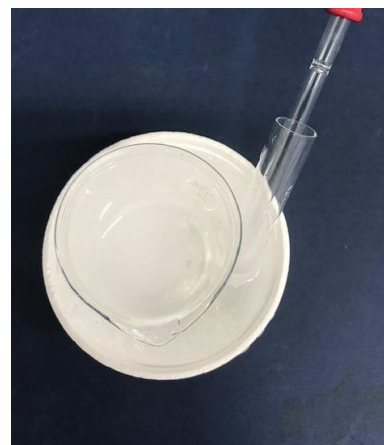
- Use a hot plate in fume hood to boil the solution and dissolve  $\text{Al}(\text{OH})_3$  to  $\text{Al}(\text{H}_2\text{O})_6^{3+}$
- Volume should be **less than 30 mL**



# Step 1.4: Crystallization



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 3-4 mL of ethanol/water solution (1:1 v/v) and cool it in the ice-water bath



# Step 1.5: Vacuum Filtration

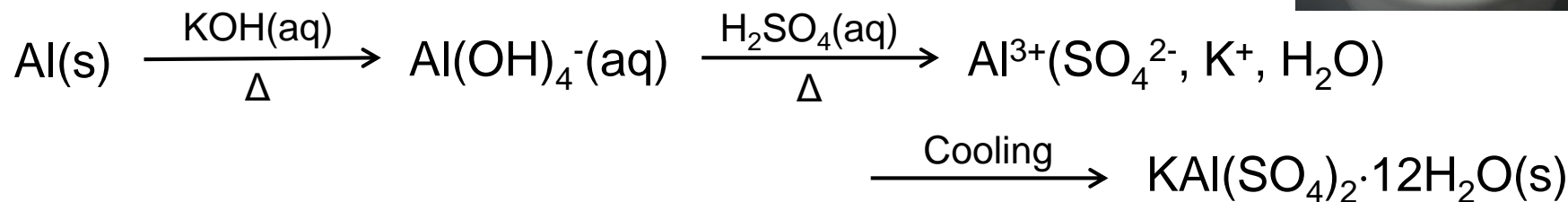


- Use tweezers to remove the used filter paper
- **Wash the Büchner funnel**
- Install a new 55-mm filter paper
- Stir the crystal precipitates in the beaker with a glass rod, then pour the solution **quickly** onto the funnel
- Use a drop pipet to **rinse the beaker and alum crystal** with cooled ethanol/water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



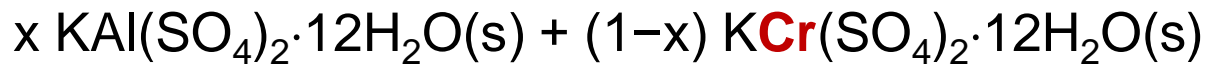
# Experiment Task 2

## Task 1



## Task 2

*These two tasks can be carried out in parallel*



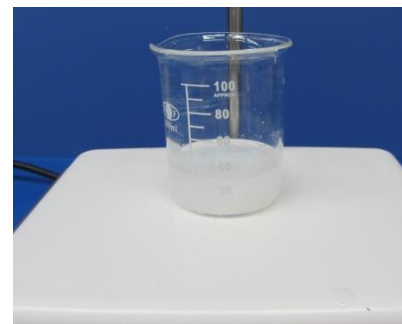
- $\text{Cr}^{3+}$  replaces  $\text{Al}^{3+}$  in the crystal structure of alum
- $d$  electrons of  $\text{Cr}^{3+}$  leads to purple color (crystal field theory)



## 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	1	25
3	3	2	25
4	3	3	25

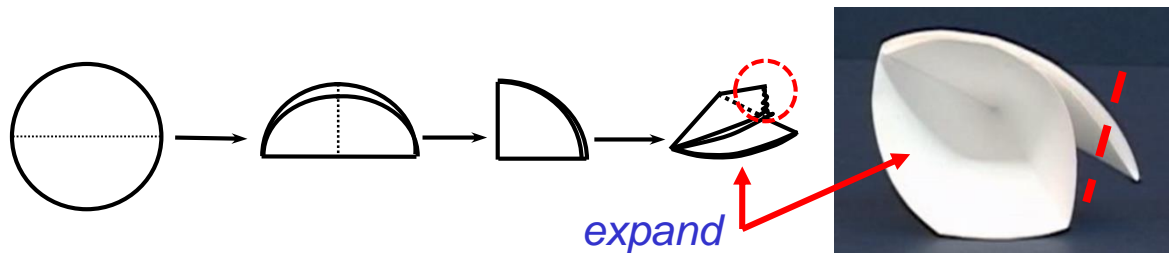


\* TA will instruct on how these groups are assigned



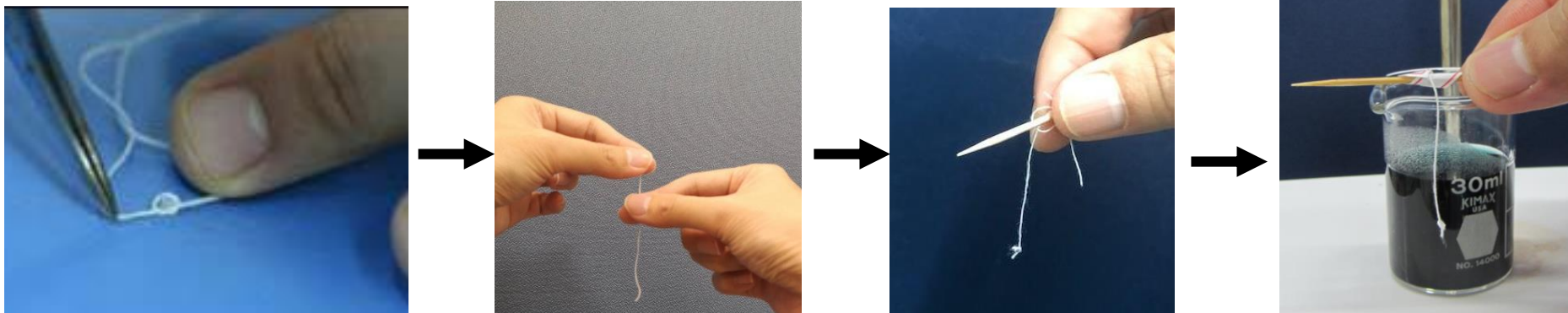
## Step 2.2: Gravity Filtration

- 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

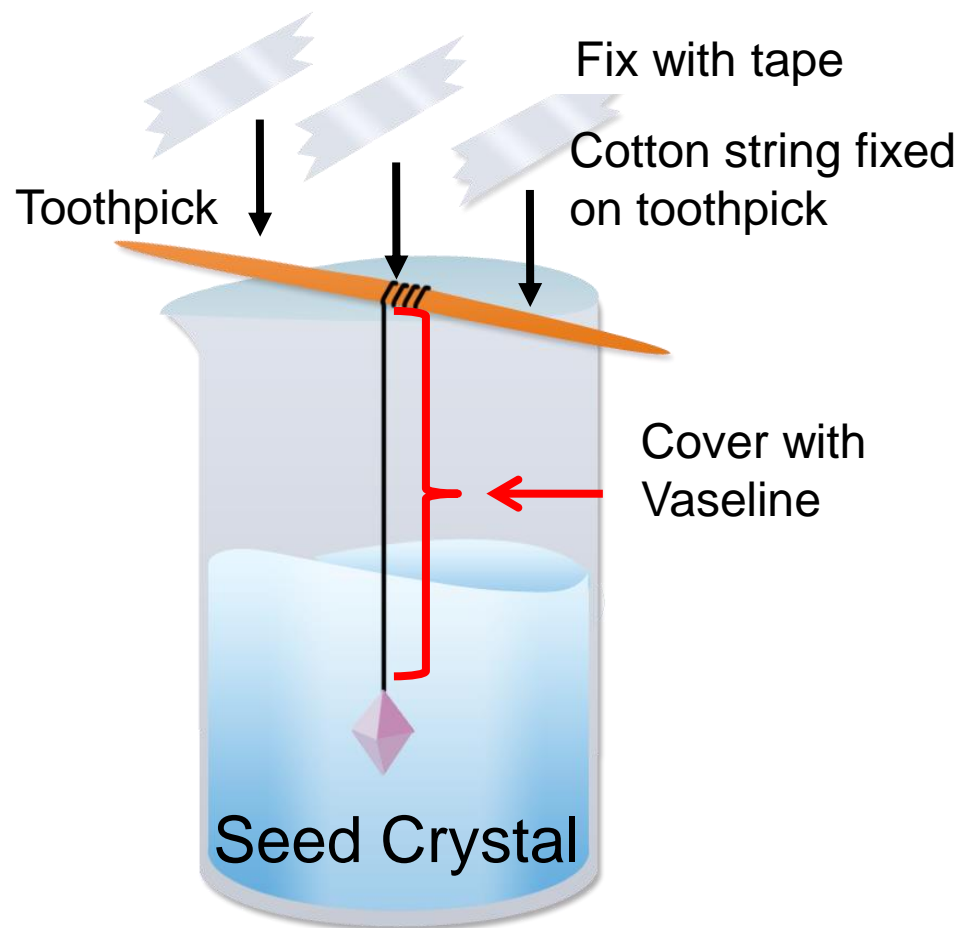
- Cut a cotton string to ~10 cm length
- On one end of the string, use a single knot to tie a seed crystal securely
- Cut the extra string off with a scissor
- Use fingers to coat the cotton string (but not the seed crystal) with Vaseline (crystallization may take place on any uneven surface)
- Tie the 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 toothpick 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
- $\text{Cr}^{3+}$ -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 suction 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:  $P\% = W_2/ W_3 \times 100\%$

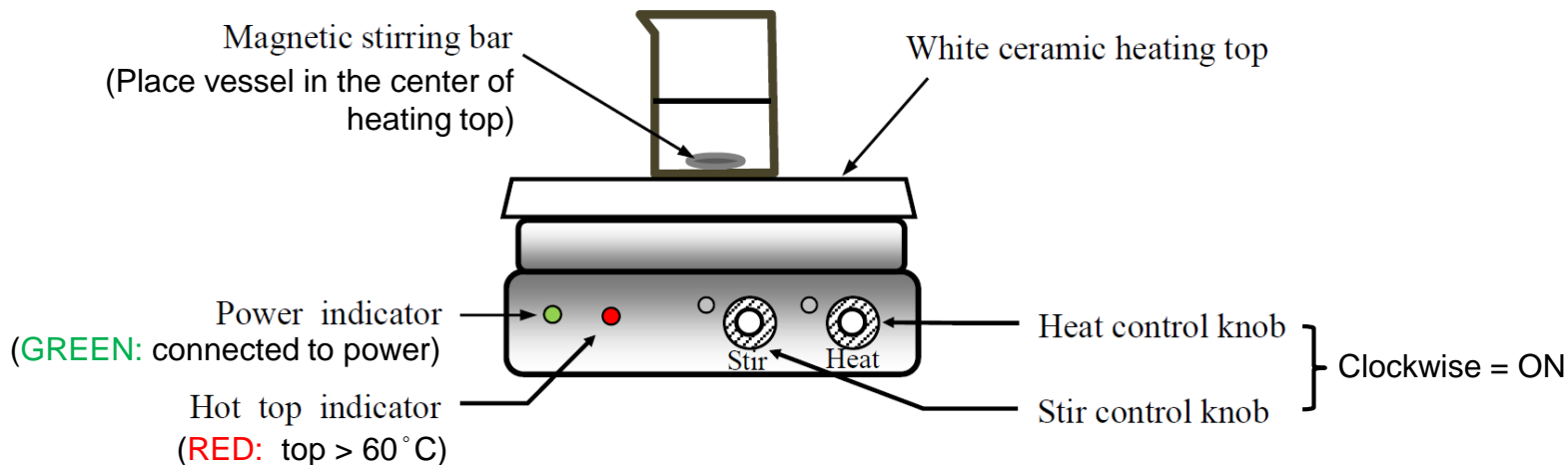
Molar mass:

Al: 26.98 g/mol

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



# Stirrer/Hot Plate



- Connect the stirrer/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”)



# Gravity Filtration

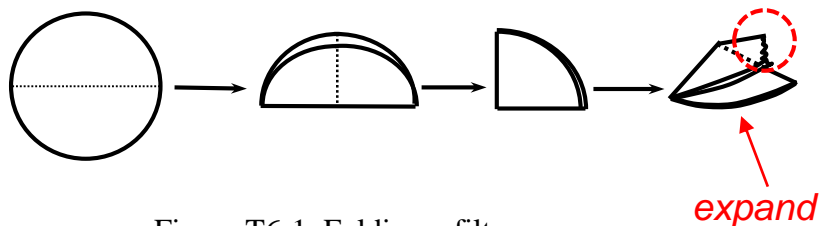


Figure T6-1 Folding a filter cone

- 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)

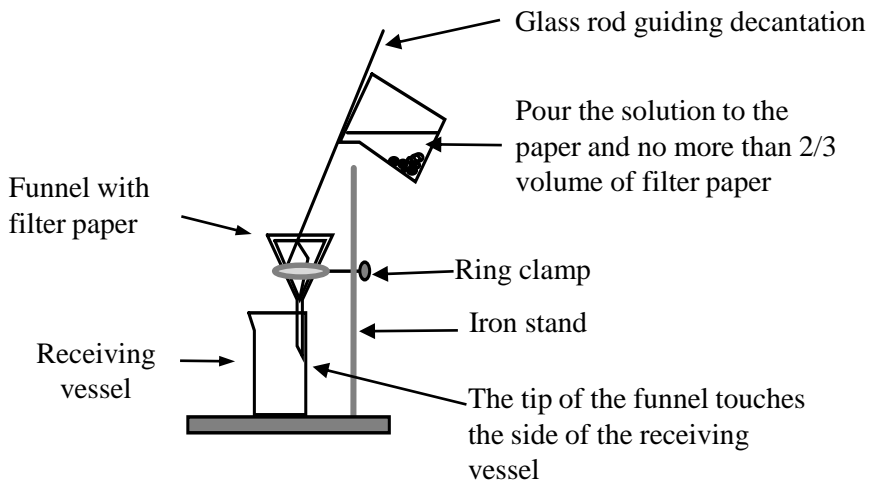


Figure T6-2 Setup of gravity filtration



# 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 suction flask. Use a rubber stopper or a rubber gasket cone to seal the flask

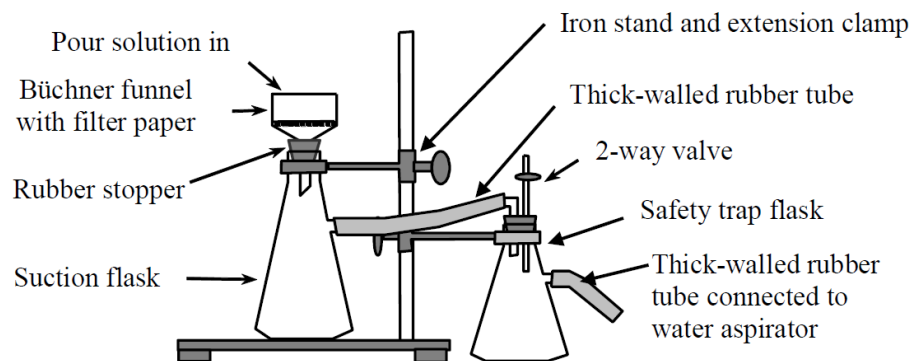


Figure T7-1 Setup of vacuum filtration

- 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



# 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

