



General Chemistry Laboratory

Solubility Product Constant of Silver Acetate



Preparation

Collect the following items

- ☐ One magnetic stir bar (TA will distribute)
- ☐ One 25 mL buret and four 125 mL Erlenmeyer flasks
- ☐ One 10 mL graduated pipet and one pipet filler
- ☐ Two large & one small glass funnels, one 100 mL beaker
- ☐ Ring clamp, Styrofoam cup, NBR gloves



From your personal equipment

- ☐ Four 100 mL beakers



Preparation

- ☐ Take two clean and dry 100 mL beakers to prepare saturated AgOAc solution
- ☐ Wash clean and oven dry two glass funnels (ϕ 70) and three 100 mL beakers (label group no. on beaker)



Objective and Principles

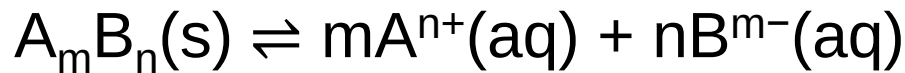
- **Objective:** Determine the solubility product constant (K_{sp}) of silver acetate (AgOAc) at specific temperatures
- **Lab techniques:**
 - Gravity filtration
 - Using a graduated pipet
 - Performing titration using a buret
 - Hot plate/magnetic stirrer
 - Lab dispenser
- **Note:**
 - Take ca. **50 mL 0.020 M** KSCN
 - **0.10 M** AgNO_3





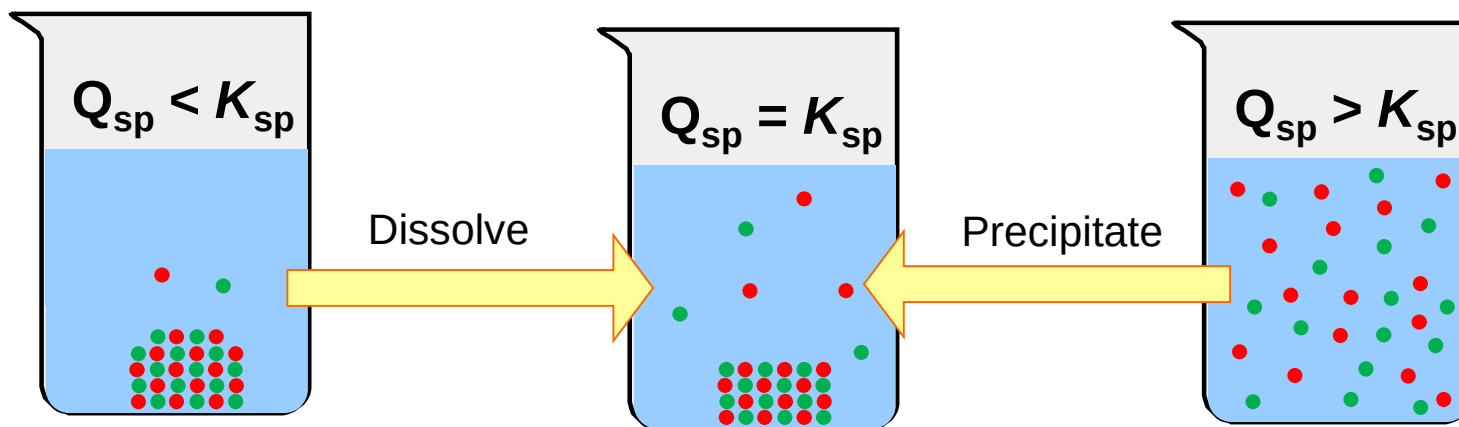
Solubility Product Constant

- K_{sp} is a specific type of equilibrium constant that describes the dissolution process of salts in water:



$$K_{sp} = [A^{n+}]^m[B^{m-}]^n$$

- When the ion product (Q) exceeds K_{sp} , precipitates would form until the Q value in the solution equals to K_{sp}





Solubility and K_{sp}

- For any specific salt, its K_{sp} changes with temperature but stays independent of pH and the concentrations of ions in the solution
- Nevertheless, the *solubility* of salts may be affected by the presence of other ions (**common ion effect**)

Solubility of AgCl in pure water



Initial		0	0
Change	- x	+ x	+ x
Equi.		x	x

$$K_{sp} = [\text{Ag}^+][\text{Cl}^-] = 1.6 \times 10^{-10}$$

$$\begin{aligned}x^2 &= 1.6 \times 10^{-10} \\ \rightarrow x &= 1.3 \times 10^{-5} \text{ (M)}\end{aligned}$$

Solubility of AgCl in 0.01 M NaCl



Initial		0	0.01
Change	- x	+ x	+ x
Equi.		x	0.01 + x

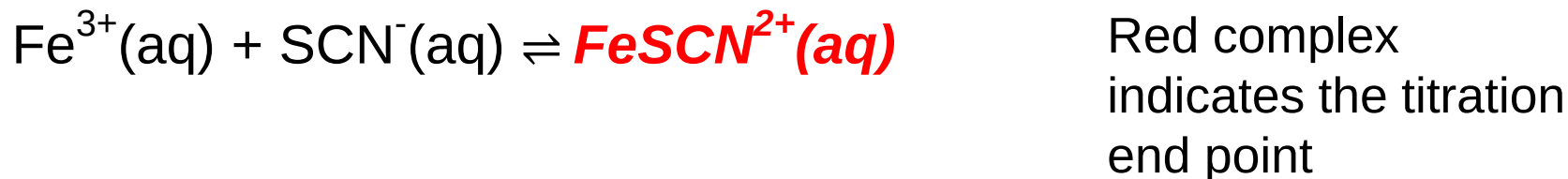
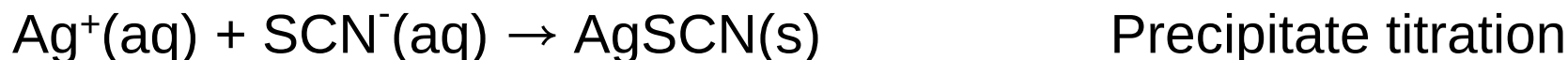
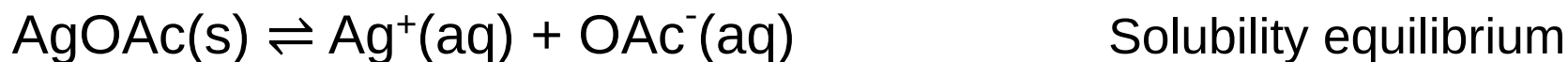
$$K_{sp} = [\text{Ag}^+][\text{Cl}^-] = 1.6 \times 10^{-10}$$

$$\begin{aligned}&x(0.01 + x) \\ &\simeq (0.01)x = 1.6 \times 10^{-10} \\ \rightarrow x &= 1.6 \times 10^{-8} \text{ (M)}\end{aligned}$$



Experiment Tasks

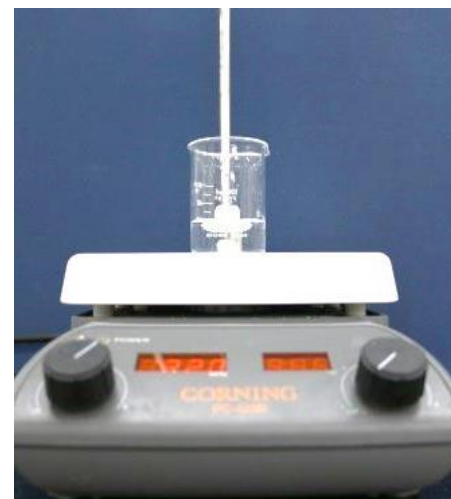
1. Prepare saturated silver acetate solution
2. Remove excess AgOAc precipitate
3. Use a standard SCN^- solution to titrate and determine $[\text{Ag}^+]$
4. Based on the known $[\text{OAc}^-]$, calculate $K_{\text{sp}}(\text{AgOAc}) = [\text{Ag}^+][\text{OAc}^-]$
5. Perform the same procedures in both room temperature and a lower temperature





Step 1: Prepare Saturated AgOAc Solution

- Use a clean and dry 100 mL beaker to take 10.0 mL 0.10 M of $\text{AgNO}_3(\text{aq})$ and 15.0 mL of 0.30 M $\text{NaOAc}(\text{aq})$
- Add the magnetic stirrer into the beaker, stir for 15 minutes
- Record the solution temperature





Step 2: Filtration to Remove AgOAc Ppt

- 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
- Perform gravity filtration to remove AgOAc precipitate and collect the filtrate in a 100 mL beaker
- Rinse a graduate pipet with few AgOAc(aq) filtrate then transfer assigned portion into 125 mL Erlenmeyer flasks
 - **5.00 mL** (coarse titration)
 - **10.00 mL** (fine titration)
- Add **1 mL of Fe^{3+} indicator**

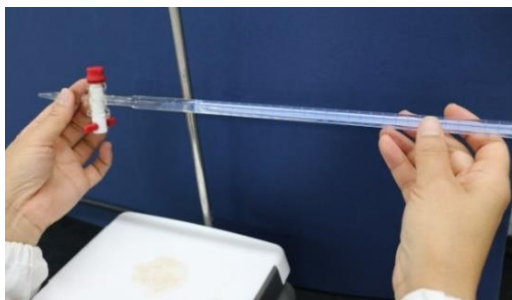




Step 3: Titration of $[\text{Ag}^+]$



- Use a clean and dry 100 mL beaker to take ca. **50 mL 0.020 M** of KSCN(aq)
- Rinse the buret twice with KSCN solution, then fill the buret
- Titrate the AgOAc solution with **0.020 M KSCN** until the solution appears a light orange color (read and record V_i and V_f to 0.01 mL)

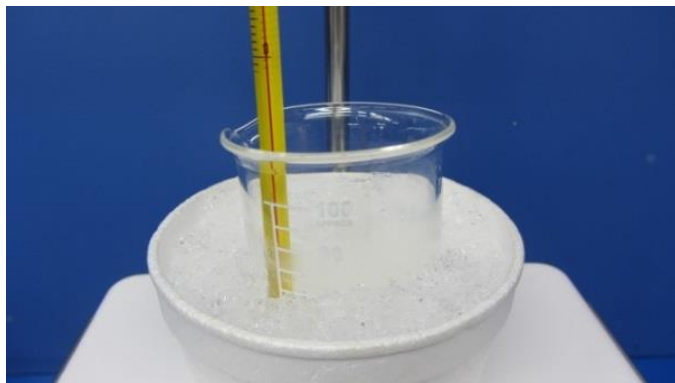


Correct
end point

Over
titration



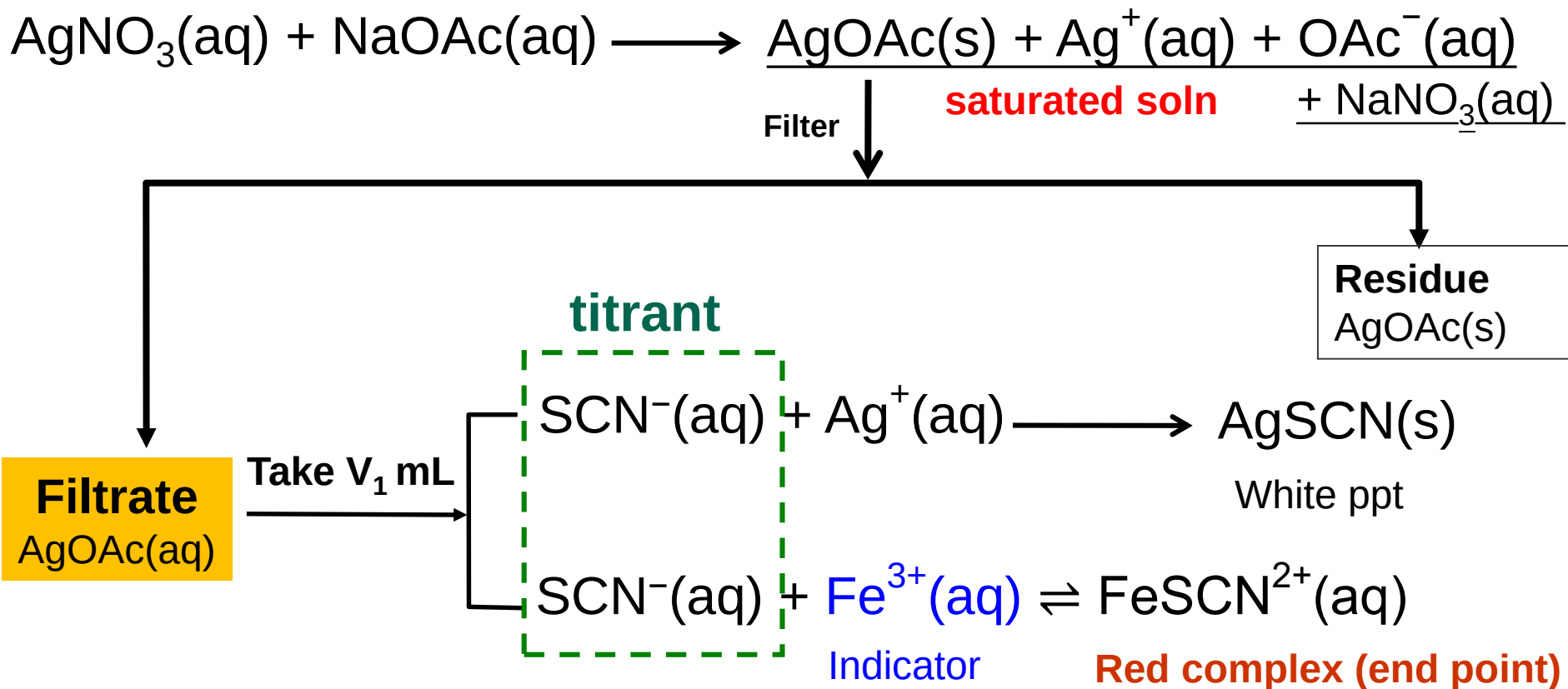
Step 4: K_{sp} at Low Temperature



- Use another clean and dry 100 mL beaker and take 10.0 mL 0.10 M of $\text{AgNO}_3(\text{aq})$ and 15.0 mL of 0.30 M $\text{NaOAc}(\text{aq})$
- Stir and mix the solution in an ice-water bath for 15 min, record the temperature of solution
- Repeat Steps 2 & 3 to determine $[\text{Ag}^+]$ in saturated $\text{AgOAc}(\text{aq})$



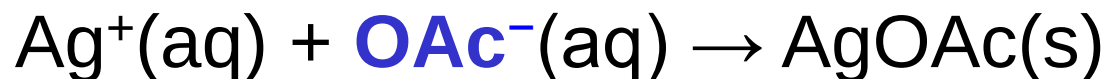
Calculate $[Ag^+]$ (Volhard Method)



- SCN^- reacts with Ag^+ first
- At equivalence point: # moles Ag^+ = # moles SCN^-
- $[Ag^+] \times V_1 = [SCN^-] \times (V_f - V_i)$



Calculate $[OAc^-]$



Before mixing $0.10\text{ M} \times 10.0\text{ mL}$
 $= 1.0\text{ mmol}$

$0.30\text{ M} \times 15.0\text{ mL}$
 $= 4.5\text{ mmol (excess)}$

After mixing $1.0 - 1.0 = 0\text{ mmol}$ $4.5 - 1.0 = 3.5\text{ mmol}$ 1.0 mmol
Ppt. occurs

- Because the K_{sp} of AgOAc is quite small, the precipitation reaction is assumed to be completed with NaOAc being the excess reagent
- Total volume after mixing = 25.0 mL
- Excess reagent:

$$[OAc^-]_{\text{left}} = \frac{3.5\text{ mmol}}{25.0\text{ mL}} = 0.14\text{ M}$$



Calculate K_{sp} of AgOAc



Concentration before
Equilibrium

0 M

0.14 M

Concentration after
Dissolution equilibrium

x M

(0.14 + **x**) M

$$K_{sp} = [\text{Ag}^+][\text{OAc}^-] = \mathbf{x} (0.14 + \mathbf{x})$$



Example of Experimental Data

Test item		Room temp. (°C) (E.g. <u>24.0</u> °C)		Ice bath (°C) (E.g. <u>3.5</u> °C)	
		Coarse titration	Fine titration	Coarse titration	Fine titration
Volume of saturated AgOAc (mL)		5.00	10.00	5.00	10.00
0.020 M KSCN	V_i (mL)				
	V_f (mL)				
Titration volume	ΔV (mL)				



Calculate $[\text{Ag}^+]$, $[\text{OAc}^-]$, and K_{sp}

Temp. ($^{\circ}\text{C}$)	(1) 24.0	(2) 3.5
$[\text{Ag}^+]$ (M)	$[\text{Ag}^+] \times 10.00 = 0.020 \times 11.11$ $[\text{Ag}^+] = 0.023$	
$[\text{OAc}^-]$ (M)	$0.14 + 0.023 = 0.16\text{_}3$	
K_{sp}	$0.023 \times 0.16\text{_}3$ $= 3.7 \times 10^{-3}$	

* Compare the K_{sp} of silver acetate at different temperature



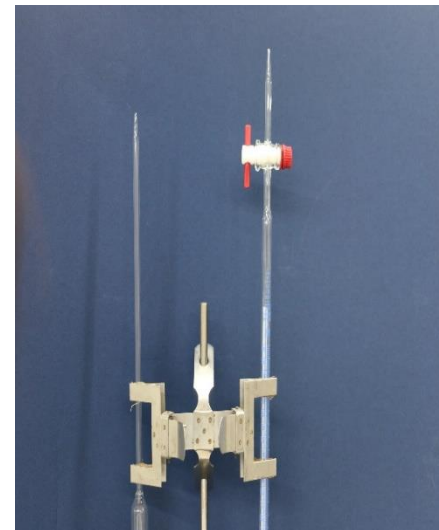
Additional Notes

- Use only clean and dry beakers for mixing AgNO_3 and NaOAc
- Rinse the graduated pipet with sat. AgOAc(aq) , and rinse the buret with KSCN(aq) to ensure that $[\text{Ag}^+]$ can be accurately determined
- Take ca. **50 mL 0.020 M KSCN(aq)** by lab dispenser
- Add **1 mL Fe^{3+}** indicator for each titration (not 1 drop)
- Temperature of AgOAc(aq) should be recorded right before the gravity filtration step
- Avoid contacting $\text{AgNO}_3(\text{aq})$ or AgOAc(aq) with skin (wear NBR gloves at all times)
- **Be careful in not losing the magnetic stir bar into the sink or waste bin**



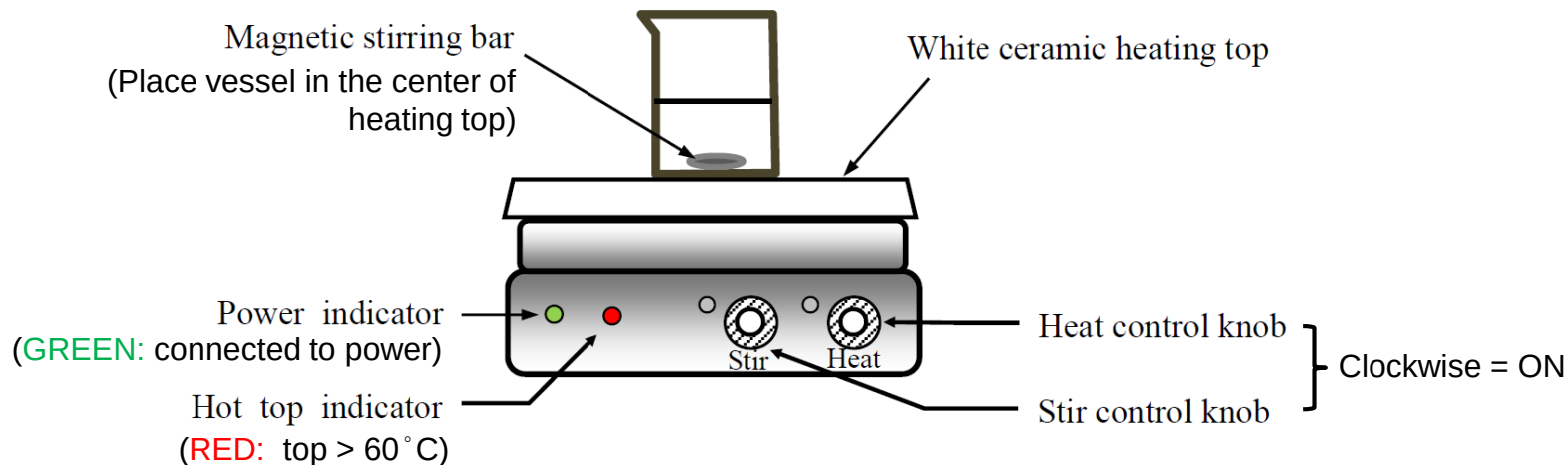
Clean-Up and Check-Out

- Ag-containing solution and precipitates should be disposed into the designated heavy metal waste bin
- Dispose of the unused KSCN(aq) into indicated bottle
- The cleaned buret and transfer pipet should be clamped upside-down on the buret clamp
- Wash Erlenmeyer flasks thoroughly with brush and detergent
- Return the magnetic stir bar to TA
- Clean up the lab bench and check personal equipment inventory (have an associate TA sign the check list)
- This is a **Brief Report** experiment
 - Hand in the report to TA at the end of experiment
- Groups on duty shall stay and help clean up the lab





T2 – 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”)



T6 – Gravity Filtration

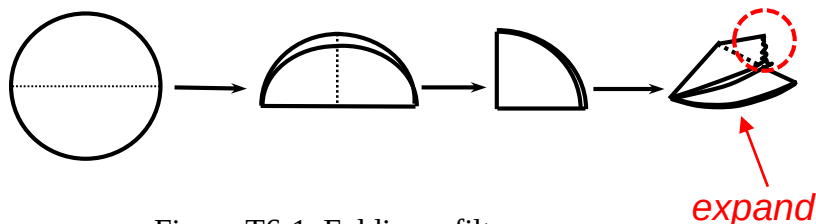


Figure T6-1 Folding a filter cone

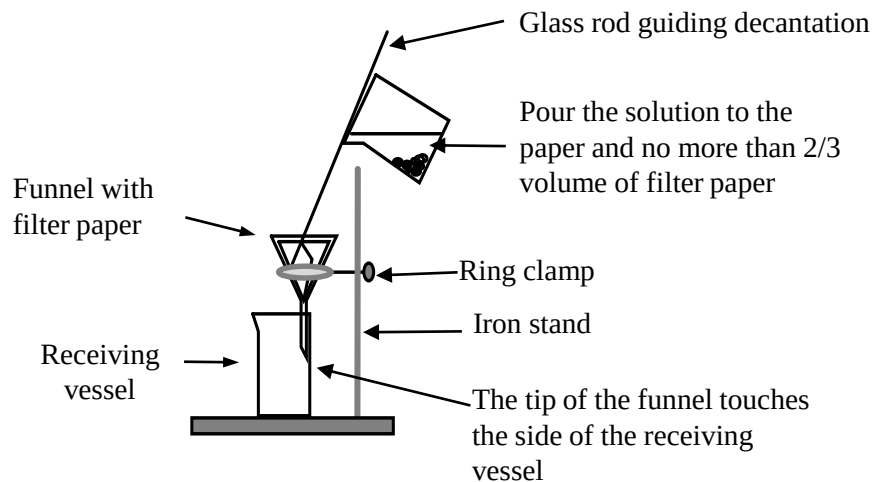


Figure T6-2 Setup of gravity filtration

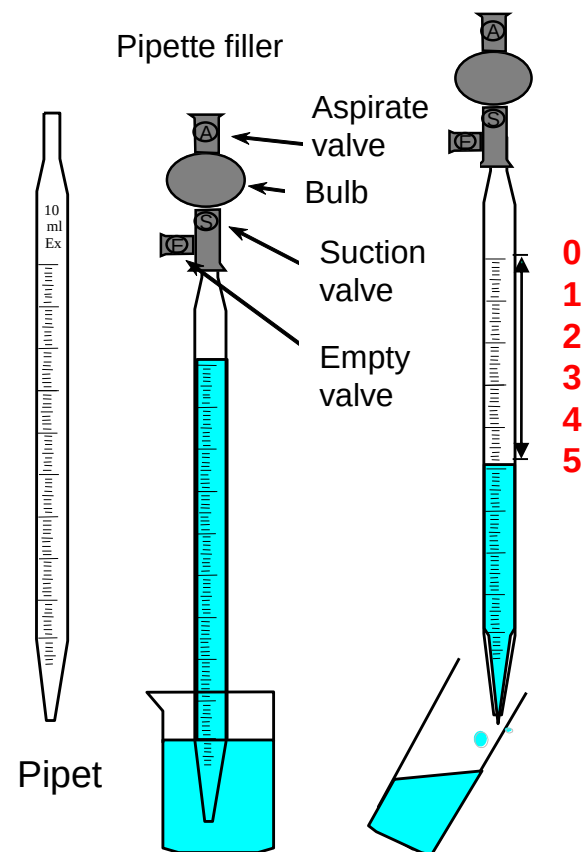
- Fold a round filter paper in half for two times. Tear off a small piece at one of the two thin outside corners
- Expand the filter paper (from the intact fold) into a cone shape. Fit the filter paper into a funnel (the edge of filter paper should not exceed the top of funnel)
- Use a ring clamp to 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 $\frac{2}{3}$ full
- After filtration, use a tweezer to separate the filter paper from funnel (don't use hand)



T12.2 – Measuring (Graduated) Pipet

Deliver 5.00 mL solution – Method 1

- Clean a 10 mL pipet and rinse it twice with small amount of the liquid to be transferred
- Press valve **A** of the pipet filler and simultaneously squeeze the bulb to expel air from it, then insert the top of pipet gently into the pipet filler
- Bring the pipet tip below the liquid surface, press valve **S** to draw liquid to the 0.00 mL marking
- Wipe off any excess liquid near the pipet tip
- Use the other hand to hold the new container. Maintain the pipet in a vertical position and let its tip touch the inner wall of the container. Press valve **E** to drain the liquid to the 5.00 mL marking
- Do not force out any liquid remaining at the tip
- Wash the pipet thoroughly after use

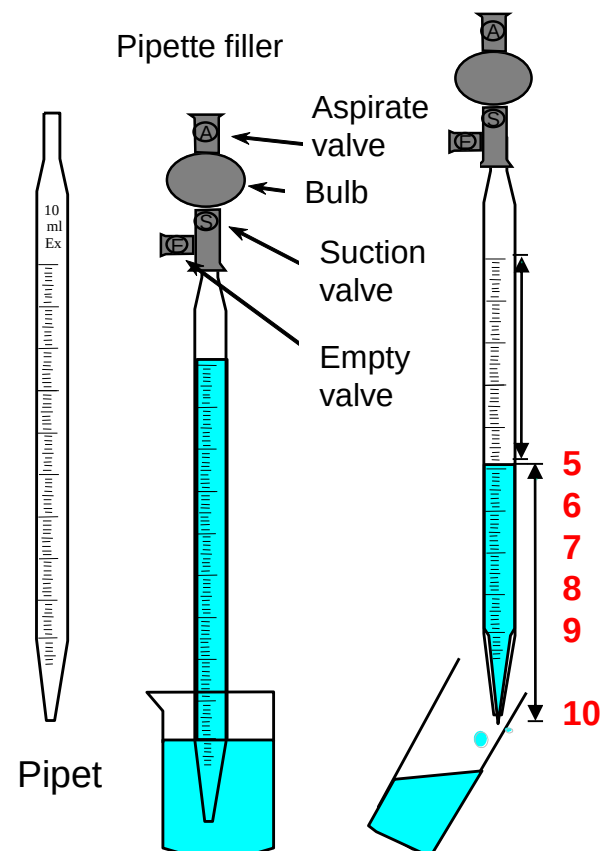




T12.3 – Measuring (Graduated) Pipet

Deliver 5.00 mL solution – Method 2

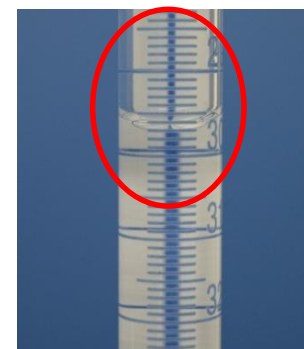
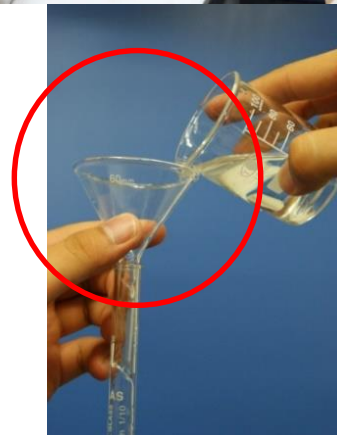
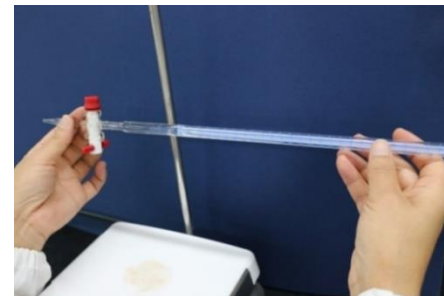
- Clean a 10 mL pipet and rinse it twice with small amount of the liquid to be transferred
- Press valve **A** of the pipet filler and simultaneously squeeze the bulb to expel air from it, then insert the top of pipet gently into the pipet filler
- Bring the pipet tip below the liquid surface, press valve **S** to draw liquid until it rises above the 5.00 mL marking
- Remove the pipet filler and quickly use an index finger to close the top of pipet. Use the finger to adjust the liquid level to the 5.00 mL marking
- Wipe off any excess liquid near the pipet tip
- Use the other hand to hold the new container. Maintain the pipet in a vertical position and let its tip touch the inner wall of the container. Release the index finger so that liquid is transferred
- Do not force out any liquid remaining at the tip
- Wash the pipet thoroughly after use





T14 – Titration

- Clean the buret with DI water, then rinse twice with ~5 mL of titrant (use a funnel to add titrant)
- Open the stopcock to repel the air at the buret tip
- Adjust the height of buret so that its tip is lower than the lid of receiving flask
- Read and record the initial volume (V_i) on the buret to 0.01 mL
- With the stopcock on the right side, use your left hand to control the stopcock while the right hand swirls the receiving flask in a circular motion
- At the titration end point, read and record the final volume (V_f) on the buret to 0.01 mL
- After the experiment, wash the buret and let it dry upside-down on the buret clamp





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) Gently pull the piston pump up until it reaches the end of travel range
- 4) Slowly push the piston down to obtain the solution

