Quantitative Analysis of Cobalt(II) Ions

Last revised: 2022/11/12

Preparation

Collect the following items

- Six 50 mL Erlenmeyer flasks + Six cork stoppers
- One 10 mL volumetric flask
- One 2 mL graduated pipet and one pipet filler
- One cuvette + Two lens tissues
- One drop pipet

From your personal equipment

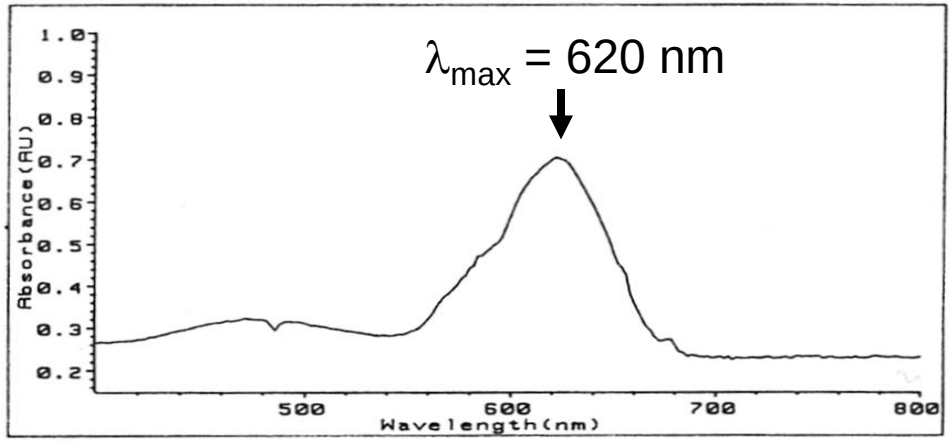
- Use one clean and dry test tube to take 8 mL standard Co^{2+} solution
- 10 mL and 50 mL graduated cylinders
- The TA will distribute a Co^{2+} solution with unknown concentration to each group

Objective and Principles

- **Objective:** Determine trace amounts of Co^{2+} ions by quantitative analysis of $[Co(SCN)_4^{2-}]$ with a spectrophotometer

$$Co^{2+}(aq) + 4SCN^-(aq) \rightleftharpoons [Co(SCN)_4]^{2-}(aq)$$

Light pink — Blue

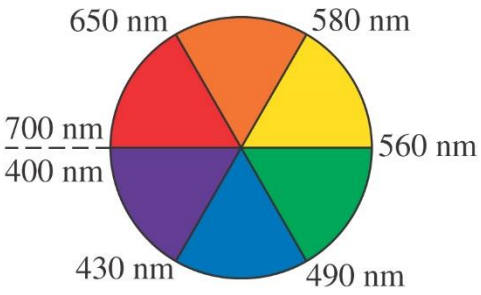


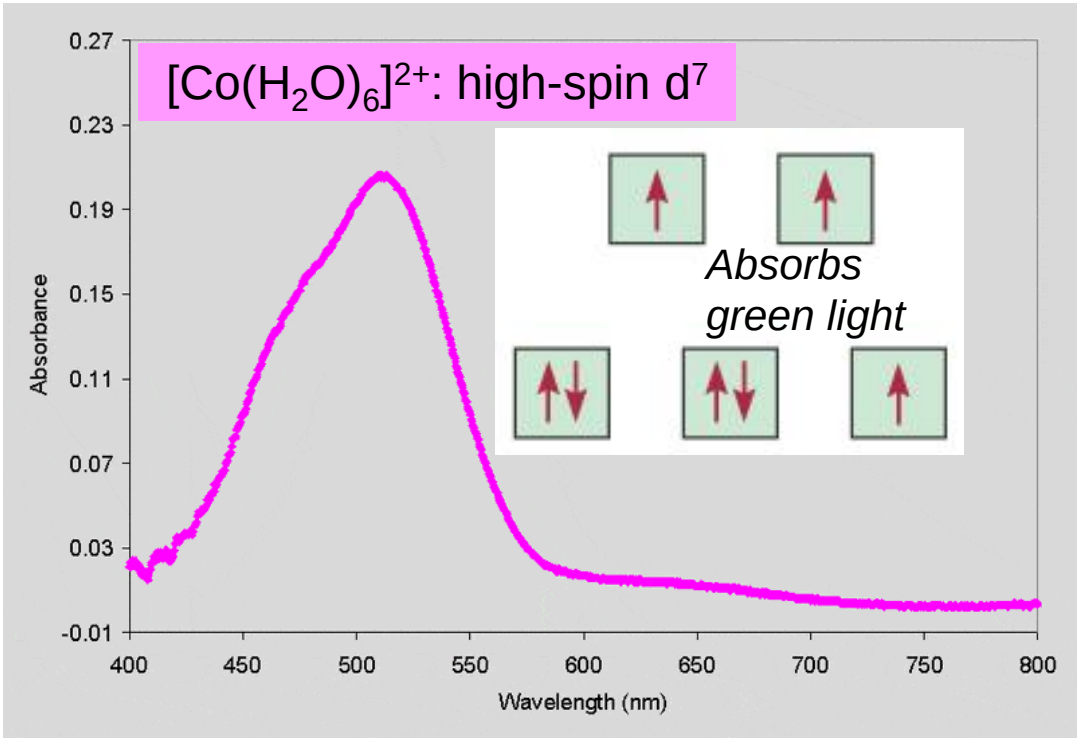
- **Lab techniques:**
  - Operate a spectrophotometer
  - Volumetric flask
  - Graduated pipet

Color and Absorption of $[Co(H_2O)_6]^{2+}$

- Co^{2+} has 7 electrons in the 3d orbitals
- In aqueous environment, each Co^{2+} is coordinated by six H_2O ligands (oxygen atom donates lone pair electrons) and forms a $[Co(H_2O)_6]^{2+}$ complex ion
- The energy of 3d orbitals are splitted in the octahedral crystal field
- Low absorption probability

A compound's visible color is complementary to the color it absorbs:

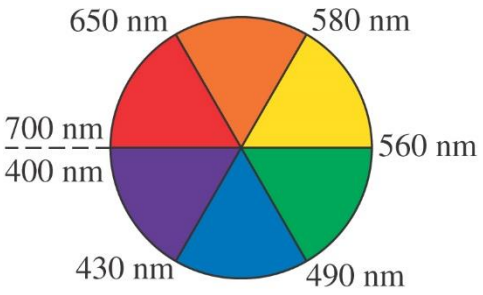


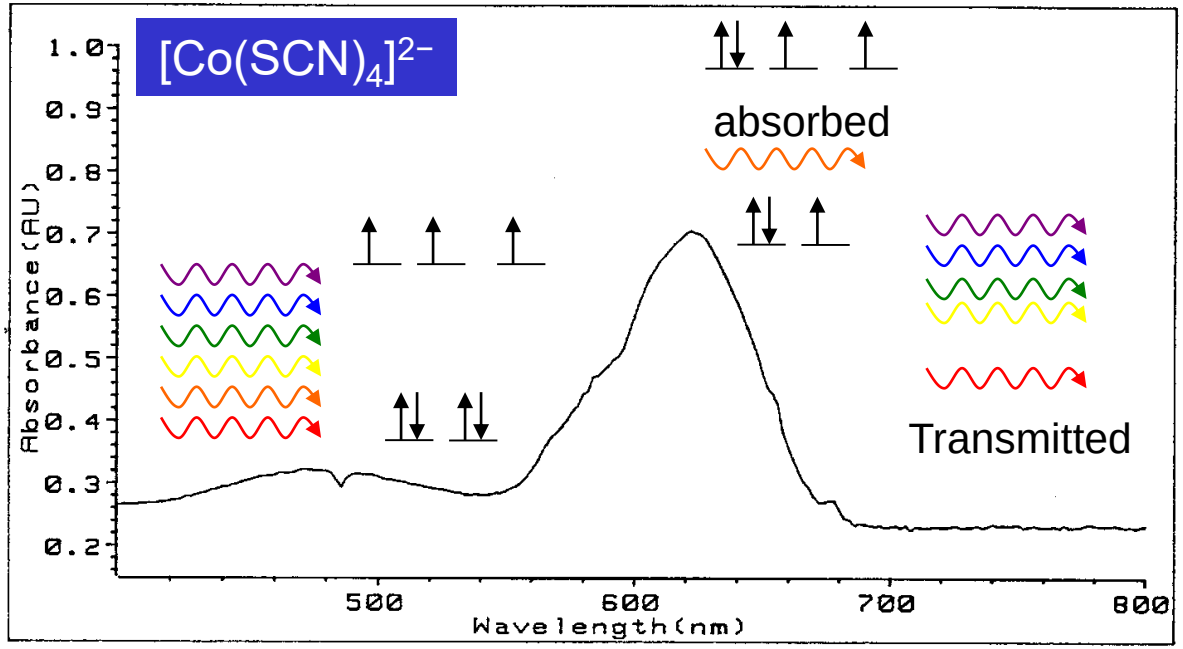


Color and Absorption of $[Co(SCN)_4]^{2-}$

- Each Co^{2+} is coordinated by four SCN^- ligands (nitrogen atom donates lone pair electrons) and forms a tetrahedral $[Co(SCN)_4]^{2-}$ complex ion
- The energy of 3d orbitals are splitted differently in this tetrahedral crystal field (2 orbitals have lower energy than the other three orbitals)
- Absorbs red/orange light with higher probability (stronger absorption)

A compound's visible color is complementary to the color it absorbs:

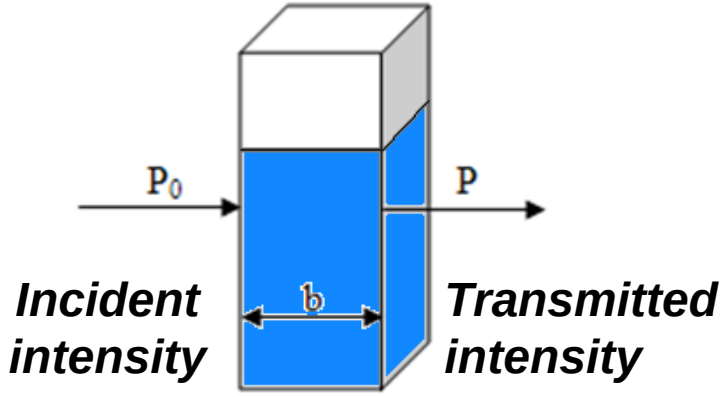




Beer's Law

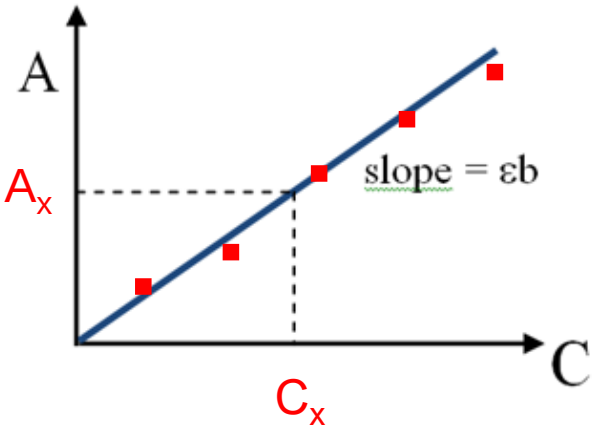
$$A(\lambda) = \varepsilon(\lambda) \times b \times c$$

A: Measured absorbance
ε : Extinction coefficient
b: Path length
c: Concentration of the absorbing substance



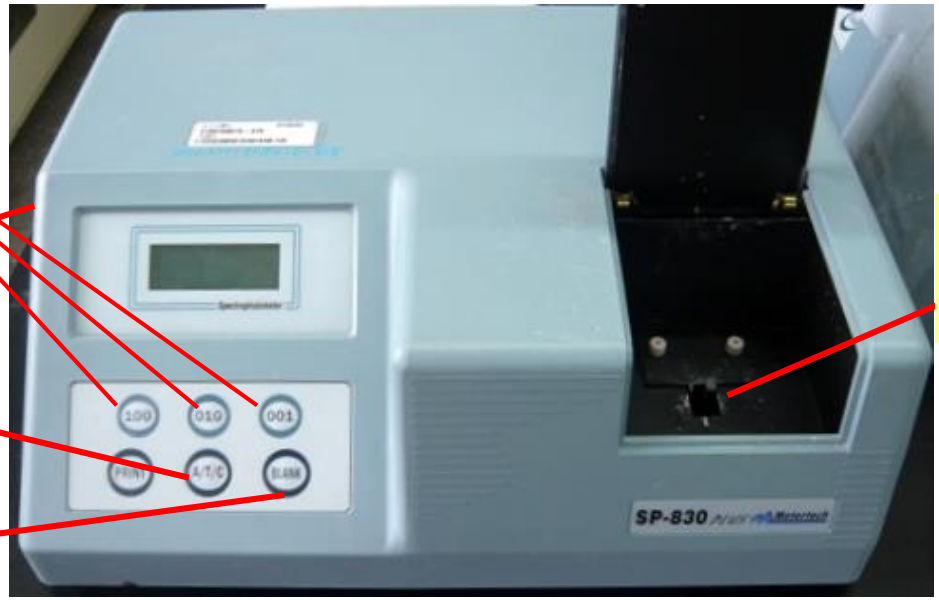
- **Transmittance: $T = P/P_0$**
- **Absorbance: $A = -\log T$**
  $A = -\log(P/P_0)$

- For a dilute solution, the absorbance is proportional to the concentration
- The extinction coefficient ε can be calculated from the Abs vs. concentration calibration curve
- With ε known, the unspecified conc. of sample solution can be determined from its absorbance



Step 1: Calibration of Spectrophotometer

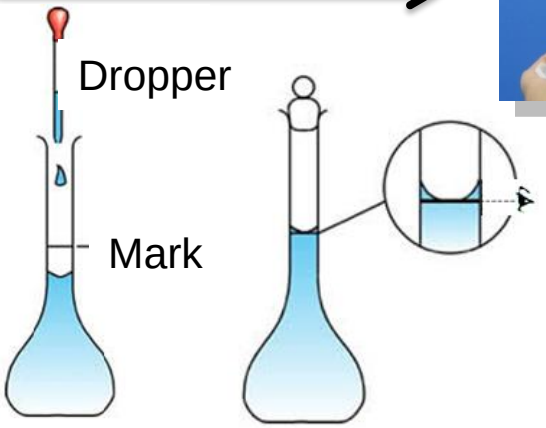
1) Switch on the power and let warm up for at least 20 minutes
2) Check whether the cuvette holder is empty, then close the lid of sample compartment
3) Press the "A/T/C" button to set the measurement mode to A (absorbance)
4) Set the analytical wavelength to 620 nm
5) Press the "BLANK" button to zero the absorbance reading

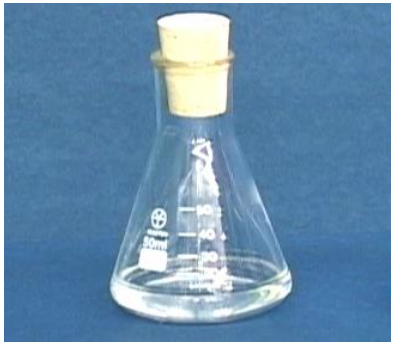


Step 2: Prepare Blank Solution

- Measure 0.8 mL 6 M HCl, 2.0 mL 50% KSCN, and 4.8 mL acetone into a clean 10 mL volumetric flask
- Add DI water until the liquid level matches the inscribed mark; Install the stopper cap (hold with a finger), invert the flask several times to ensure thorough mixing
- Transfer the solution to a clean and dry 50 mL Erlenmeyer flask, and seal it with a cork stopper (acetone is a volatile solvent)

Add DI water
to 10 mL mark





✓ White precipitates may appear depending on the sequence of mixing chemicals (why?)

Step 3: Prepare Co^{2+} Solutions

- According to the values listed in the table below, use a 2 mL graduated pipet to accurately measure 0.50 – 2.00 mL standard Co^{2+} solution (0.10 mg/mL) and transfer it to the clean 10 mL volumetric flask
- Add 0.8 mL 6 M HCl, 2.0 mL 50% KSCN, and 4.8 mL acetone into the same volumetric flask
- Dilute each solution to 10 mL with DI water, mix thoroughly, and transfer to clean and dry Erlenmeyer flasks (sealed with cork stoppers)

	Sample No.	0.10 mg/mL Co^{2+} (mL)	6 M HCl	50% KSCN	Acetone
Blank solution (step 2) →	**0**	**0**	0.8 mL	2.0 mL	4.8 mL
For calibration curve	**1**	**0.50**			
	2	**1.00**			
	3	**1.50**			
	4	**2.00**			

✓ Use the same volumetric flask to prepare all solutions in the sequence 0 → 4

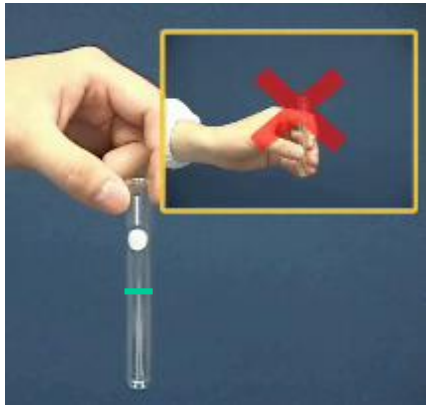
Step 4: Prepare Unknown Co^{2+} Sample

- Use a 2 mL graduated pipet to transfer x mL of the unknown conc. Co^{2+} solution (assigned to your group) into a clean 10 mL volumetric flask
- Add 0.8 mL 6 M HCl, 2.0 mL 50% KSCN, and 4.8 mL acetone into the same volumetric flask
- Dilute the solution to 10 mL with DI water, mix thoroughly, and transfer to clean and dry Erlenmeyer flask (sealed with a cork stopper)

	Sample No.	0.10 mg/mL Co^{2+} (mL)	6 M HCl	50% KSCN	Acetone
Blank solution (step 2) →	**0**	**0**	0.8 mL	2.0 mL	4.8 mL
For calibration curve (step 3)	**1**	**0.50**			
	2	**1.00**			
	3	**1.50**			
	4	**2.00**			
Unknown sample (step 4) →	**Unknown**	**$0.5 \leqq x \leqq 2.0$**			

Step 5: Blank Adjust

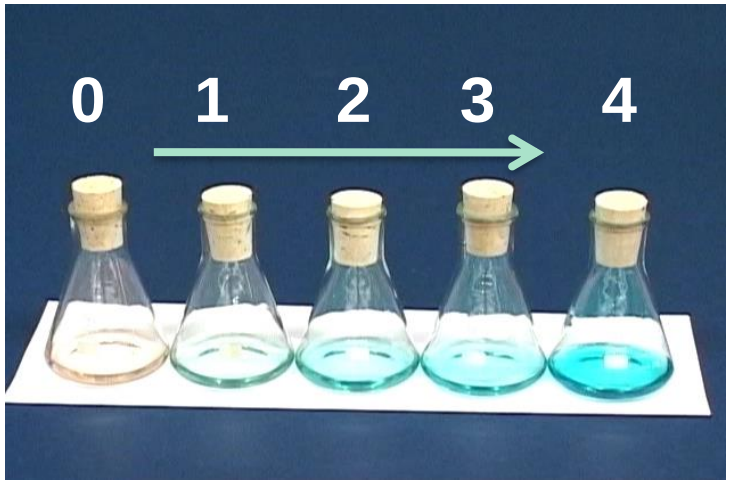
Blank solution

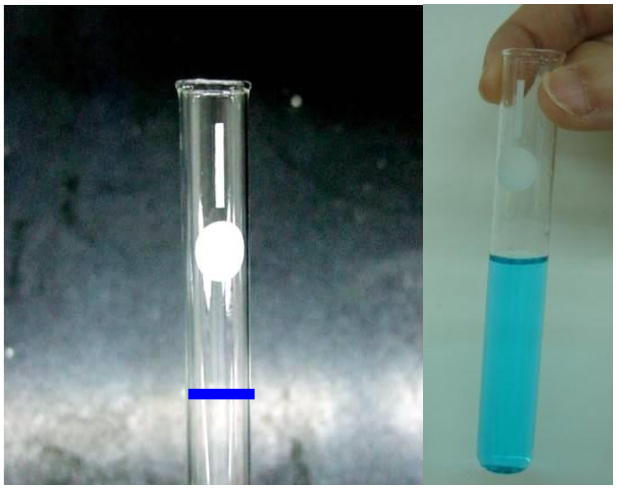


- Use a dropping pipet to rinse the cuvette twice with blank solution, then add blank solution into the cuvette to ~ ½ height
- Hold only the top part of the cuvette, and clean off any residual liquid outside the cuvette with a lens tissue
- Place the cuvette gently into the cuvette holder and align the vertical white lines
- Close the lid, then press the "BLANK" button to blank adjust

✓ Do NOT use regular test tubes in place of cuvette

Step 6: Absorbance Mesurement





- Perform the measurement in the sequence of 1→4 (lower to higher concentration)
- Pour the solution back to the original flask, then rinse the cuvette with the next tested solution twice; filled to ½ height
- Wipe off any residual liquid outside the cuvette, then place the cuvette into the cuvette holder (align the vertical white lines)
- Close the lid, and read the absorbace value at 620 nm

Clean-Up and Check-Out

- Wash all 50 mL Erlenmeyer flasks and place them in the oven
- Waste solution that contains Co^{2+} ions and organic solvent (acetone) should be disposed into the designated container in the fume hood
- Turn off the spectrophotometer and put the dust cover on
- Clean cuvette only with water (**to avoid scratching the cuvette, do not use brush to clean**), return the cuvette to TA
- Clean up the lab bench and check personal equipment inventory (have an associate TA sign the check list)

- This is a **Full Report** experiment:
  - Member A: **Hand in prelab to the TA**
  - Member B: **Have the lab notes and results checked by the TA, and hand in the report next week**

- Groups on duty shall stay and help clean up the lab

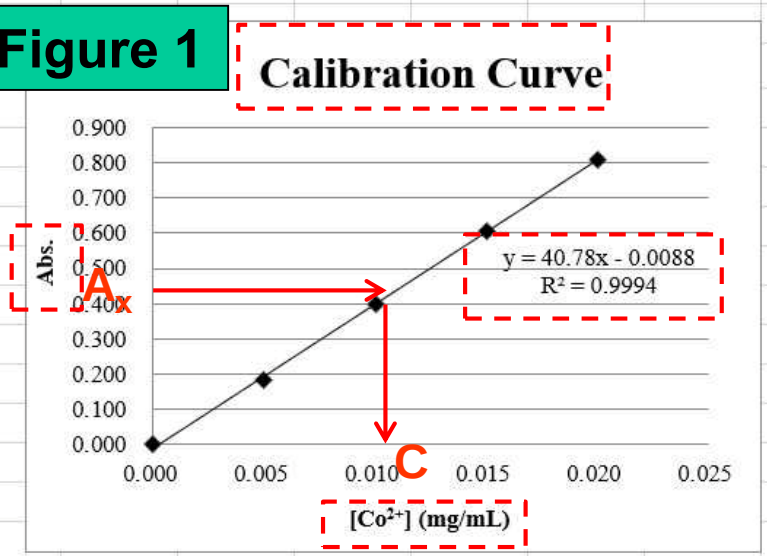
Plot the Calibration Curve

- 5-point scattered plot (including the blank solution) and label both the x and y axes properly; Include both the data table (Table 1) and the calibration curve (Figure 1) in your report
- Plot the linear regression line (y = mx + b) – show the result and the R^2 value together with the plot
- Calculate the original concentration of unknown Co^{2+} solution prior to dilution; the absorbance of the unknown sample should fall within the range of the calibration curve

Table 1

$[Co^{2+}]$ (mg/mL)	Abs.
0	0.000
0.0050	0.183
0.010	0.399
0.015	0.604
0.020	0.809

Figure 1



Final Report (Full Version)

- Four experiments (E3, E8, E10, E12)
- Complete the data analysis and calculation part in the lab manual
- Plot data correctly and discuss potential sources of errors
- Hand in the report in the following week together with the prelab and lab records
- 50 points per report (5 pts deduction for late submission < 1 week)

I. Prelab exercise

- ✓ Objectives
- ✓ Principles
- ✓ Chemicals
- ✓ Procedures

15 pts

+

II. Lab Notes

- ✓ Observation
- ✓ Operation
- ✓ Reaction condition
- ✓ Data and results

10 pts

+

III. Final report

- ✓ Data analysis
- ✓ Elaborate results
- ✓ Error analysis
- ✓ ~~Questions and discussion~~

25 pts

Lab Report Grading Rubrics

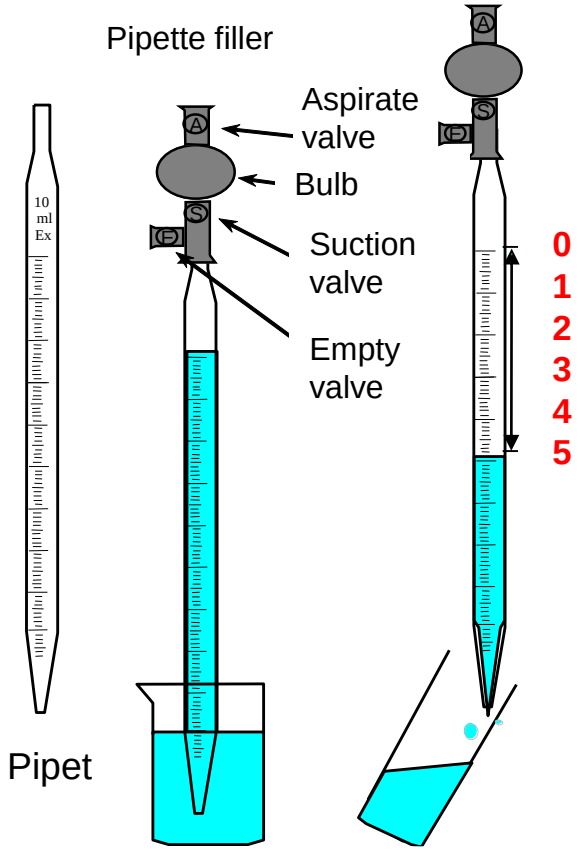
Category	Guidelines	Pts
I. Prelab exercise	1. Briefly summarize main principles and relevant equations	5
	2. List the chemicals' toxicity and physical and chemical properties	5
	3. Use flow chart to explain the experimental procedures	5
II. Lab notes	4. Record data with correct significant figures and units	5
	5. Record observations, operations, and reaction conditions in details	5
III. Final report	6. Process data correctly (calculation included)	5
	7. Present final results with correct significant figures and units	5
	*8. Analyze the results with appropriate error discussions**	*5*
	*9. Plot the results with correct XY axes and labeling**	*5*
	*10. Elaborate findings and provide constructive suggestions**	*5*

**Only for full reports*

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

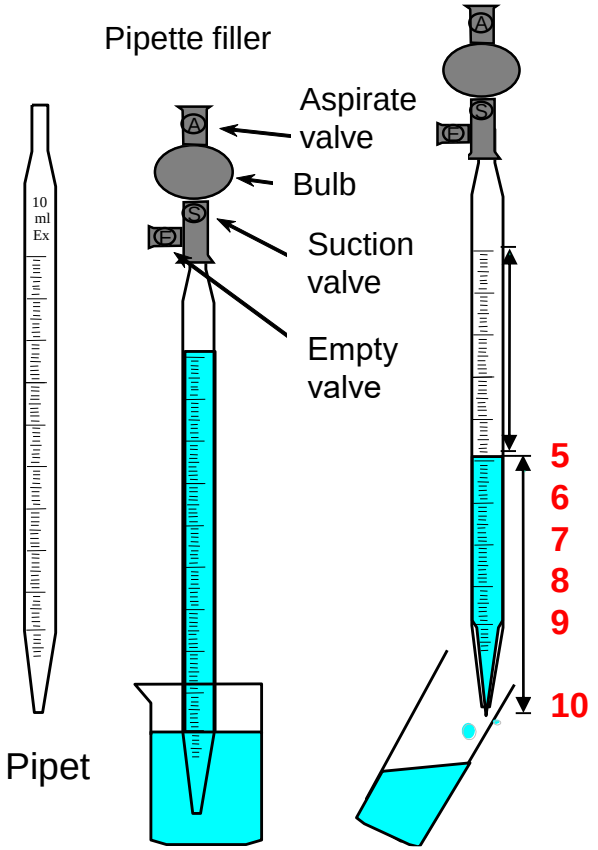


T12 Video (YouTube link)

T12.3 – Measuring (Graduated) Pipet

Deliver 5.00 mL solution – Method 2

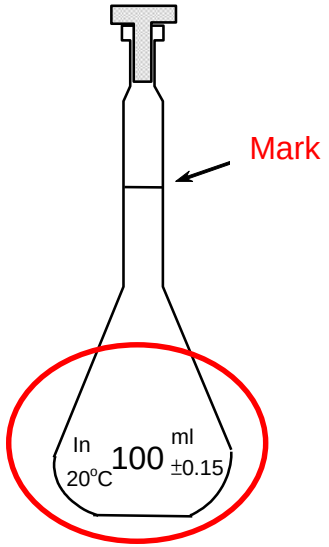
- Clean a 10 mL pipet and rinse it twice with small amount of the liquid to be transferred
- Press valve **A** of the pipet filler and simultaneously squeeze the bulb to expel air from it, then insert the top of pipet gently into the pipet filler
- Bring the pipet tip below the liquid surface, press valve **S** to draw liquid until it rises above the 5.00 mL marking
- Remove the pipet filler and quicky 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



T12 Video (YouTube link)

T13 – Volumetric Flask

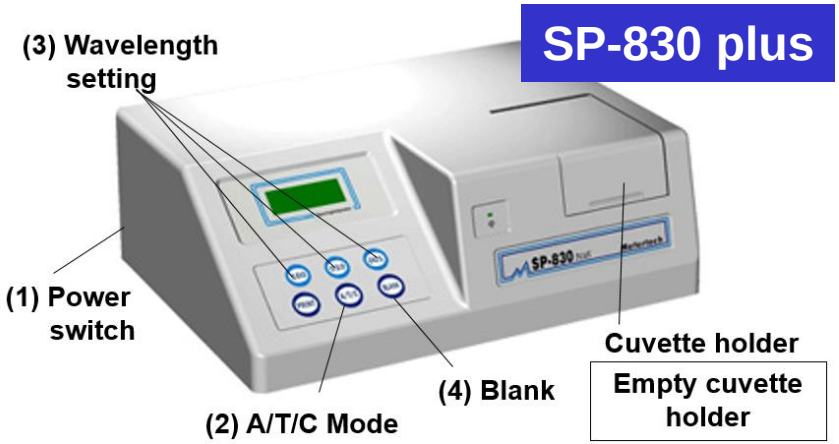
- Clean the volumetric flask thoroughly, then rinse it with a small amount of solvent
- Using a funnel, transfer the solution to be diluted into the volumetric flask
- Fill solvent into the flask until about half full, swirl the flask to let the solution mix
- Add more solvent so that the liquid level approaches (but does not exceed) the inscribed mark
- Use a dropper pipet to add solvent slowly, so that the liquid level matches the inscribed mark
- Install the stopper cap (hold with a finger), invert the flask several times to ensure thorough mixing
- Pour the solution into a beaker for later use (do not store solution in the flask)
- Wash the volumetric flask immediately after use and let it air dry (do not put flask on a hot plate or in an oven)



T13 Video (YouTube link)

T17 – Spectrophotometer

- Turn on the power switch and let the instrument warm up for at least 20 minutes
- Ensure the *cuvette holder* is empty
- Press the "Mode" button several times until "A" (absorbance) appears on the screen
- Set the wavelength to the desired value (e.g. 620 nm)
- Press the "Blank" button to zero the reading
- Place a *cuvette* with blank solution into the cuvette holder. Align the white line on the cuvette toward you (do NOT use regular test tubes in the spectrophotometer)
- Press the "Blank" button to calibrate
- Place a sample solution into cuvette holder
- Close the lid of the sampel compartment, record the absorbance reading



Note – the T17 video shows the older Spectronic 20 model instead of the currently used SP-830

T17 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