國立中正大學

National Chung Cheng University

積極創新 修德澤人

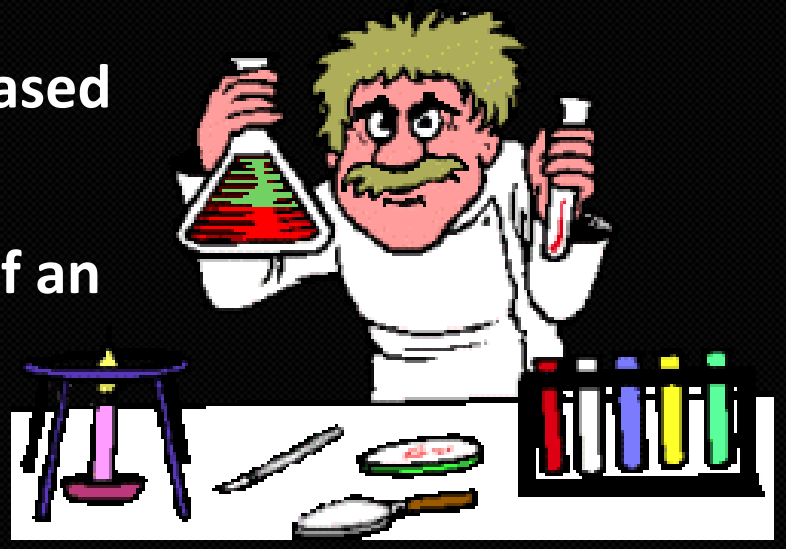
114-2
Exp. 10 – Quantitative Analysis of Trace Cobalt(II)

April 24, 2026 (time required: 1 hours)

1. Objectives

Quantitative analysis of trace cobalt(II) ions using spectrophotometry after complexation with thiocyanate.

- **To form a blue complex between cobalt(II) ions and thiocyanate (SCN^-).**
- **To measure the absorbance of the complex at 620 nm using a spectrophotometer.**
- **To construct a calibration curve based on Beer's law.**
- **To determine the concentration of an unknown cobalt(II) solution.**



2. Definitions

A. Beer's law:

The absorbance (A) of a solution is directly proportional to the concentration (c) and the path length (b).

$T\ (\text{Transmittance}) = P / P_0$ (eq.10-1)

$A\ (\text{absorbance}) = -\log T$ (eq.10-2)

$A = \varepsilon \times b \times c$ (eq.10-3)

ε Mole absorption coefficient ($cm^{-1}M^{-1}$)

b is the path length of light (cm)

c is the concentration (M)

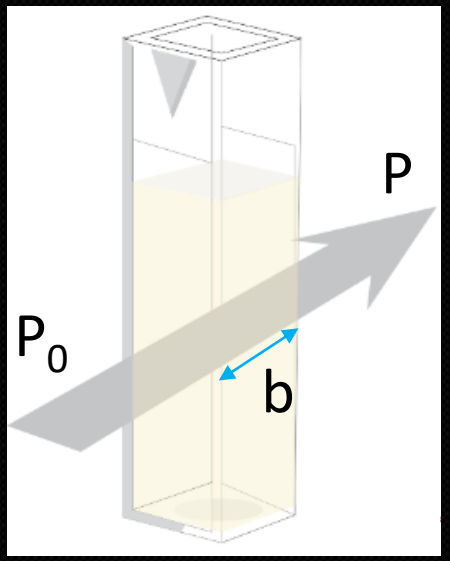


Figure 1. Light transmission diagram

B. Calibration Curve Method:

- Prepare a series of standard solutions with known concentrations.
- Measure their absorbance at the wavelength of maximum absorption (λ_{max} = 620 nm).
- Plot absorbance (y-axis) vs. concentration (x-axis) to obtain a calibration curve.
- Measure the absorbance of the unknown sample and use the curve to determine its concentration.

C. Spectrophotometric Analysis of Cobalt(II)

- **Cobalt Complex Formation:**
  Cobalt(II) ions react with thiocyanate (SCN^-) to form a blue complex.

$$Co^{2+}_{(aq)} + 4\ SCN^-_{(aq)} \rightleftharpoons Co(SCN)_4^{2-} \qquad \text{(eq.10-4)}$$

- **Absorption Maximum:**
  The complex has a maximum absorption peak at **620 nm (visible region)**.

- **Effect of Dilution:**
  Dilution with water reduces SCN^- concentration, causing the complex to dissociate into the pale pink species $Co(SCN)^+$, which make accurate quantification difficult.

- **Use of Acetone:**
  Acetone was added (low dielectric constant) to suppress the dissociation of cobalt complexes.

- **pH Control:**

  Hydrochloric acid (HCl) is added to maintain a constant acidic pH, preventing interference from hydroxide or other ions.

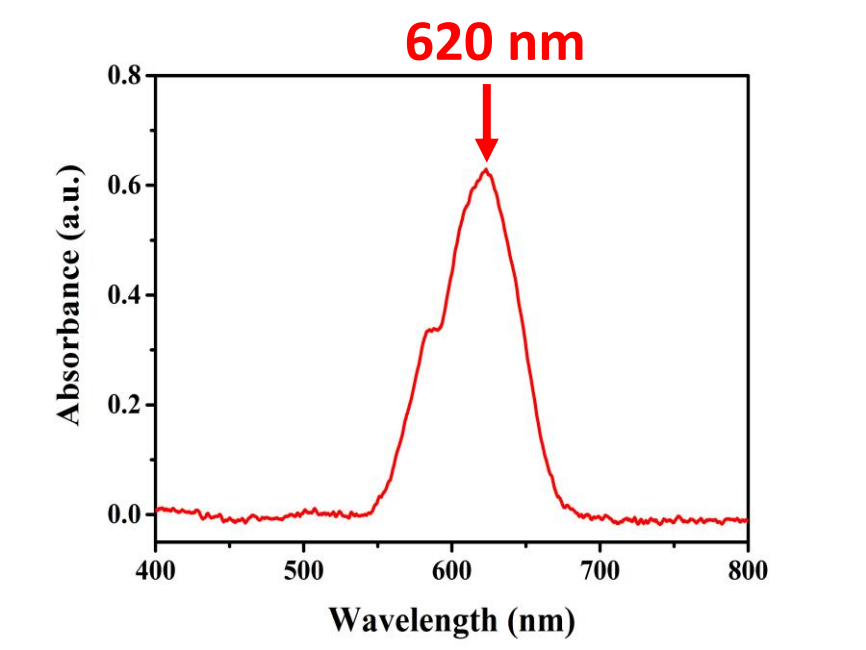


Figure 2. Absorption spectrum of $Co(SCN)_4^{2-}$

3. Equipment and Chemicals (1/2)

Equipment

In cabinet	Provided by TA
Pipet filler × 1	Lens papers
Graduated pipet × 1	Dropper × 6
10 mL Volumetric flask × 5	Cuvette × 1
Robber stopper × 6	Spectrophotometer (shared by two groups)
Erlenmeyer flask × 6	

3. Equipment and Chemicals (2/2)

Chemicals

Cobalt(II) standard solution (0.10 mg/mL)**
Unknown Solution of Cobalt **
50% Potassium thiocyanate (KSCN) solution**
6 M Hydrochloric acid (HCl)***
Acetone (CH_3COCH_3) ** and Deionized water

* : Irritant * : Flammable * : Toxic * : Corrosive

Reference: PubChem (https://pubchem.ncbi.nlm.nih.gov/) - accessed : 2026/04/03

4. Solution Preparation Steps (1/4)

1. **Prepare blank and standard solutions:**
   - Prepare one blank solution (containing all reagents except cobalt) and a series of standard solutions with known cobalt concentrations.
2. **Measure volumes into volumetric flasks:**
   - Pipette the required volume of cobalt standard solution into each 10 mL volumetric flask (see Table 1).
   - Add the same amounts of KSCN, HCl, and acetone to all flasks (including the blank).
3. **Dilute to the mark:**
   - Add deionized water to each flask until the bottom of the meniscus reaches the 10 mL mark, and mix thoroughly.

4. Solution Preparation Steps (2/4)

4. **Transfer to cuvette:**
   - Pour a small amount of the solution into a clean cuvette to rinse it, then discard.
   - Fill the cuvette to approximately 70–80% of its volume (about 2.5–3 mL for a standard cuvette).
5. **Measure absorbance:**
   - Use the blank solution to zero the spectrophotometer at λ_{max} = 620 nm.
   - Measure the absorbance of each standard solution, followed by the unknown solution.

4. Solution Preparation Steps (3/4)

Table 1. Preparation of standard solutions

Standard #	Cobalt Stock (mL)	HCl (mL)	Acetone (mL)	KSCN (mL)	Water to 10 mL
1	0	0.8	4.8	2.0	2.4
2	0.5	0.8	4.8	2.0	1.9
3	1.0	0.8	4.8	2.0	1.4
4	1.5	0.8	4.8	2.0	0.9
5	2.0	0.8	4.8	2.0	0.4

Standard Cobalt(II) Solutions

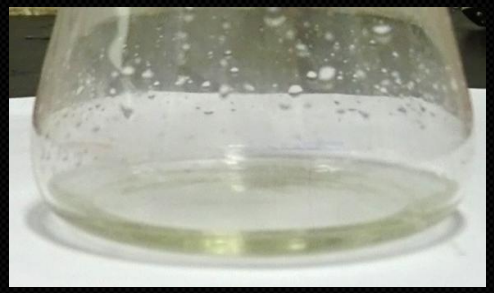


Figure 3. No. 1 0.0 mL cobalt stock solution

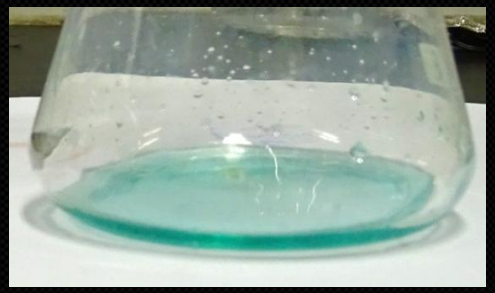


Figure 4. No. 2 0.5 mL cobalt stock solution

Figure 5. No. 3 1.0 mL cobalt stock solution

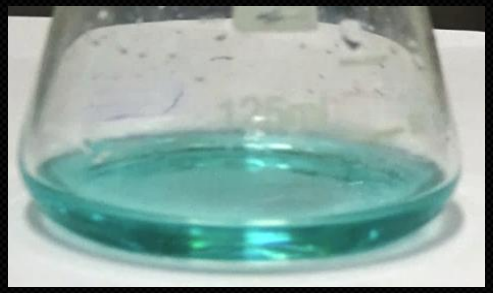


Figure 6. No. 4 1.5 mL cobalt stock solution

Figure 7. No. 5 2.0 mL cobalt stock solution

4. Solution Preparation Steps (4/4)

1. **Prepare the unknown solution:**
   Pipette the required volumes of HCl, acetone, KSCN, and the unknown cobalt solution into a clean 10 mL volumetric flask (see Table 2).
2. **Dilute to the mark:**
   Add deionized water until the bottom of the meniscus reaches the 10 mL mark. Mix thoroughly.
3. **Transfer:**
   Pour the solution into a clean, dry Erlenmeyer flask and seal tightly with a stopper to prevent acetone evaporation.

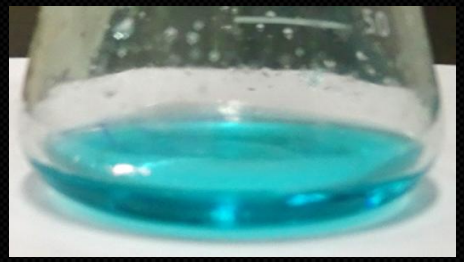


Figure 8. Unknown cobalt solution

Table 2. Preparation of unknown cobalt solution

Unknown cobalt solution (mL)	HCl (mL)	Acetone (mL)	KSCN (mL)	DI water to 10 mL
0.5	0.8	4.8	2.0	1.9

Spectrophotometer

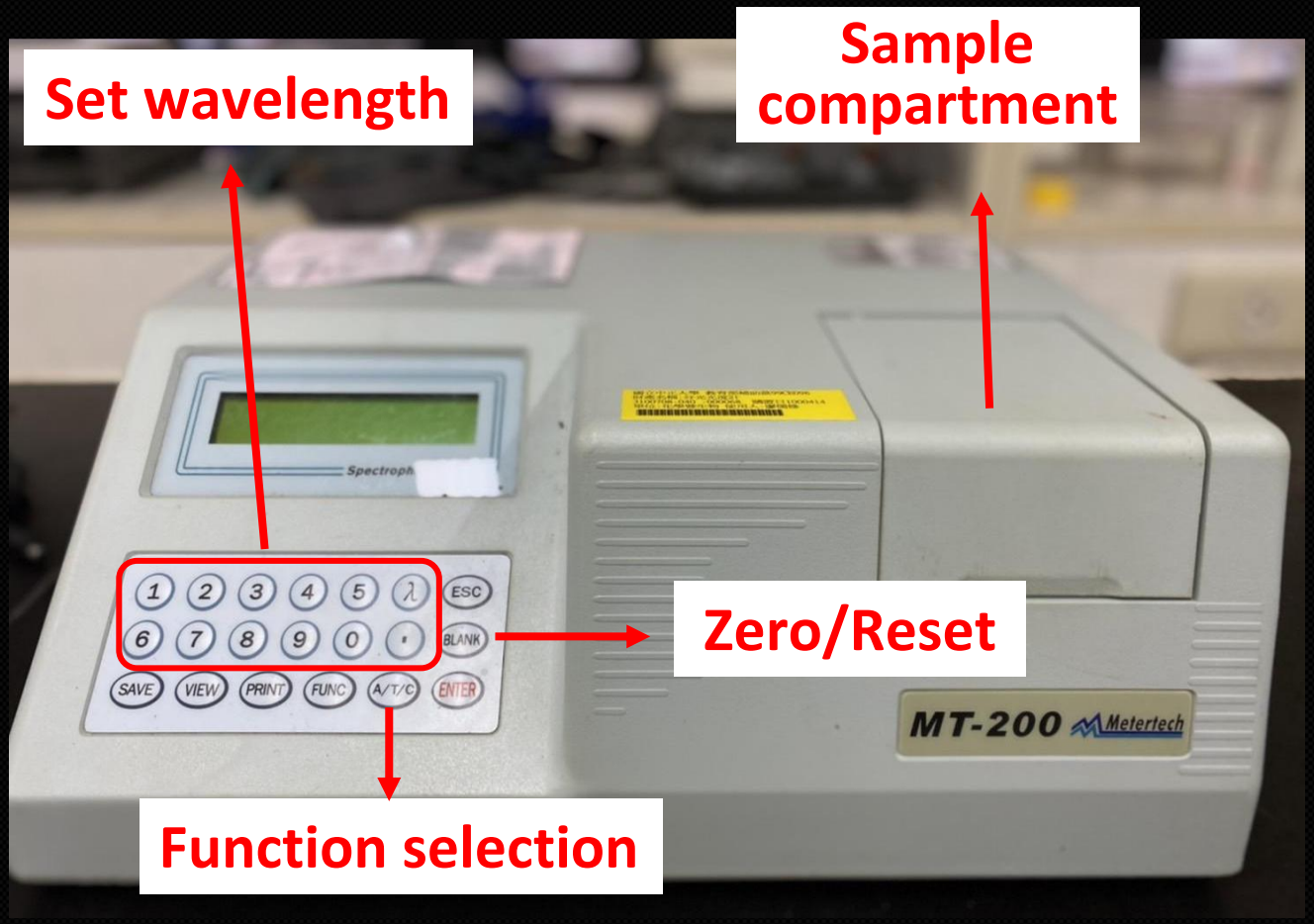


Figure 9. Spectrophotometer (MT-200 Metretech)

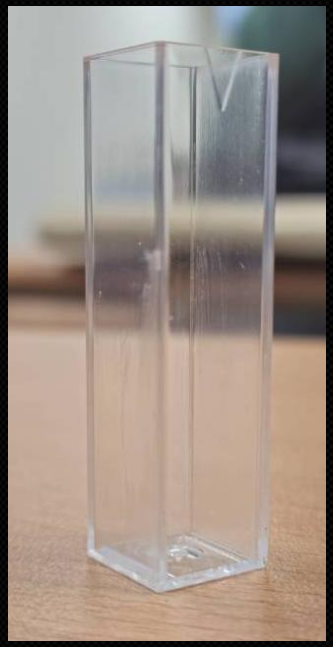


Figure 10. Cuvette

5. How to use spectrophotometer

Figure 11. Power switch

Figure 12. Control panel

1. Plug in the power cord and turn on the instrument.
2. Allow the spectrophotometer to warm up for 20 min before use.
3. Set the wavelength to 620 nm (λ_{max}).
4. Use the reference solution (No. 1, blank) to zero the instrument:
   - Place the blank cuvette into the sample holder.
   - Adjust the absorbance reading to 0.00 (set as blank).
5. Measure the absorbance of each standard and unknown solution.

6. Safety and Precautions (1/2)

1. **Personal Protective Equipment (PPE):** Lab coat, safety goggles, and gloves must be worn at all times.
2. **Chemical Safety:** All chemicals are toxic or hazardous. Follow TA instructions and lab regulations at all times.
3. **No Removal:** Do not remove any chemicals or equipment from the laboratory.
4. **Cuvette Cleaning:** Only the inner walls need to be cleaned. Use deionized water and air dry.
5. **Before Measurement:** Wipe the cuvette exterior with lens paper (not regular tissue) to remove fingerprints.

Figure 13. Proper cuvette handling

6. Safety and Precautions (2/2)

6. **Cuvette Orientation:** Always align the same orientation mark (e.g., arrow) toward the light source for consistent readings.
7. **Waste Disposal:** All waste contains heavy metals (cobalt) and organic solvents (acetone). Dispose in the designated heavy metal waste container.
8. **Sample Sealing:** Seal flasks tightly after preparation to prevent acetone evaporation, which would change concentration.
9. **Cleaning:** Use only deionized water for rinsing all glassware and cuvettes.
10. **Spectrophotometer Warm-up:** Preheat for 20 minutes before use for stable readings.

7. Experimental Results

Analysis wavelength (λ_{max}): 620 nm

Concentration of standard Co(II) solution: 0.10 mg Co^{2+} / mL

(1) Experimental Data for Calibration Curve

Volume of standard cobalt solution (mL)	Concentration after dilution (mg / mL)	**Absorbance** (at 620 nm)
0.5	0.005	
1.0	0.010	
1.5	0.015	
2.0	0.020	

Sample	Volume of cobalt solution (mL)	**Absorbance** (at 620 nm)
unknow	0.5	

(2) Calibration Curve and Unknown Determination

Plot absorbance (y-axis) vs. cobalt(II) concentration (x-axis) to construct the calibration curve.

Determine the concentration of the unknown cobalt solution as follows:

1. **Measure the absorbance of the prepared unknown solution at 620 nm.**
2. **Use the calibration curve to find the diluted concentration (in the 10 mL volumetric flask).**
3. **Calculate the original unknown stock concentration.**

Experimental results for unknown cobalt solution:

Sample ID	Theoretical concentration (mg/mL)*	Experimental concentration (mg/mL)	Relative error (%)
unknow			

*Theoretical concentration is provided by the TA (known value for the unknown sample).
Relative error = |experimental – theoretical| / theoretical × 100%.

8. Questions

1. An acidic solution containing 0.420 mg Fe^{3+} is diluted with KSCN to 100.0 mL. At 480 nm, in a 1.00 cm cuvette, the absorbance is 0.530, Assuming the complex formed is $FeSCN^{2+}$ is wrong. Calculate its molar absorption coefficient (ε).

2. If the standard cobalt solution is contaminated with a small amount of Fe^{3+}, will it affect the quantification of cobalt ions? Why?

3. In this experiment, HCl is added to maintain pH. If a student forgets to add HCl, the pH increases. What issues may arise, and how would that affect absorbance measurements?

4. **Why is it necessary to calibrate the spectrophotometer with a blank solution before measuring samples? What problems occur if this step is omitted?**

National Chung Cheng University
Department of Chemistry and Biochemistry

Chinese version : TA C.-H. LIAO (廖建勳)

English version: TA Y.-C. Tseng (曾 盈臻)

Revised by: Prof. Joyce S. Yu (于淑君)

Revision date: 115.04.16