Quantitative Analysis of Cobalt (II) Ions

Collect

- 50 mL Erlenmeyer flask (6) (wash clean ➔ dry ➔ cool)
- Test tube (1) (wash clean ➔ dry ➔ cool)
- 10 mL Volumetric flask (1)
- 2 mL graduated pipet (1)
- Pipet filler (1)
- Cork stoppers (6)
- Cuvettes (1) (do not clean with brush; return after class)
- Dropper (1)
- Lens cloth (2)
- Unknown cobalt(II) ion solution: 6 mL per group (given out by TA)
- About 100 mL of distilled water
Objective & Skills

Objective

- To determine trace amounts of cobalt(II) ions by performing a quantitative spectroscopic analysis of the complex of cobalt(II) ion with thiocyanate.

\[
\text{Co}^{2+}_{(aq)} + 4\text{SCN}^-_{(aq)} \rightarrow \text{Co(SCN)}_4^{2-}
\]

\[\lambda_{\text{max}}: 620 \text{ nm}\]

Skills

- Pipet
- Volumetric flask
- Spectrophotometer
Beer’s Law $A = \varepsilon bc$

$\lambda_{620 \text{ nm}}$ $\xrightarrow{b}$ $\frac{I_t}{I_o}$

$T$ (transmittance) $= \frac{I_t}{I_o}$

Abs (absorbance) $= -\log T$

$= \varepsilon bc$

Co(SCN)$_4^{2-}$ (aq)

Complementary color
Procedure 1: Prepare Blank Solution (without Co²⁺)

(1) 0.80 mL 6 M HCl
(2) 2.0 mL 50% KSCN
(3) 4.8 mL acetone
Add to volumetric flask from dispenser

- Firmly stopper the flask
- Invert repeatedly
- Assure mixing well and solids disappear

Transfer soln in clean Erlenmeyer flask
Stopper to prevent evaporation
Procedure 2: Prepare Cobalt Sample Solution with Standard Concentration

Accurately measure standard cobalt(II) solution (0.10 mg/mL) with 2 mL pipet

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<table>
<thead>
<tr>
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</thead>
<tbody>
<tr>
<td>0.50</td>
<td>1.00</td>
<td>1.50</td>
<td>2.00 mL</td>
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</tbody>
</table>

Accurately measure \( x \) mL unknown cobalt (II) solution

\( 0.2 < x \leq 2.0 \)

10 mL volumetric flask

Add 0.80 mL 6 M HCl

2.0 mL 50% KSCN

4.8 mL acetone

Add water till 10 mL mark

Mix thoroughly

Transfer to

Clean Erlenmeyer flask, stopper
Formula of Standard Cobalt Sample Solution

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Cobalt(II) soln (mL)</th>
<th>6 M HCl</th>
<th>50% KSCN</th>
<th>Acetone</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
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</tr>
<tr>
<td>1</td>
<td>0.50</td>
<td>0.8 mL</td>
<td>2.0 mL</td>
<td>4.8 mL</td>
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<tr>
<td>2</td>
<td>1.00</td>
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<td></td>
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</tr>
<tr>
<td>3</td>
<td>1.50</td>
<td>0.8 mL</td>
<td>2.0 mL</td>
<td>4.8 mL</td>
</tr>
<tr>
<td>4</td>
<td>2.00</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unknown</td>
<td>0.2 &lt; x ≤ 2.0</td>
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</tbody>
</table>

Standard solutions:
- Color increases in intensity
- Without white precipitate
- Checked by TA

Transparent Blue Solution
Procedure 3: Calibration of Spectrophotometer

(1) Turn on power and warm up for 20 min.
(2) Press Mode button and set to A (Absorbance)
(3) Set the analytical wavelength (i.e. 620 nm)
(4) Press “BLANK” button to zero set

(1) Power switch
(2) A/T/C Mode
(3) Wavelength setting
(4) Blank

Cuvette holder
Empty cuvette holder
**Calibration: Blank Adjust**

**Blank soln**
- Rinse cuvette twice with blank sample
- Add soln until 1/2 height
- Wipe with lens cloth

**Cuvette**
- Hold top
- Do not clean with brush
- Do not use a test tube

- Use same cuvette
- Align cuvette in same direction to control the path of the light
- Close lid
- Press “Blank” button to blank adjust
Procedure 4: Absorbance of Standard and Unknown Cobalt Sample Solution

- Start testing from lower concentration
- Rinse cuvette with test solution twice
- Fill cuvette with 1/2 of solution
- Wipe cuvette with lens cloth
- Place cuvette in cuvette holder
- Align cuvette in same direction
- Close lid and read the Absorbance
Notice

- Familiar with the manipulation of pipet, volumetric flask, and spectrophotometer first.
- Use 2 mL graduated pipet to measure standard Co(II) solution accurately. Do not use graduated cylinder instead.
- Waste solution contains heavy metals and organic solvent; must be recycled.
- After class, hand out the cuvette to TA.
- Do not leave the cuvette in the spectrophotometer.
Calibration Curve and Concentration of Unknown Cobalt Solution

- 5-points plot (blank soln included)
- X-Y scattering plot (label x,y axes)
- Show linear regression \( y = mx + b \) and \( R^2 \)
- The absorbance of the unknown solution should be within the range of the linear line
- Concentration of unknown solution should take into account its dilution factor \( (x \text{ mL}) \)

\[
y = 32.5x + 0.0052
\]

\[
R^2 = 0.9997
\]