I. Group III Basic Sulfide Precipitation

Tommy, Trojan
Chemistry 105B Lab
Monday 5-9 p.m
T.A. Diane

II. To familiarize ourselves with the qualitative analysis of Group III cations & analyze according to procedure.

III. Procedure:

A) Preparation of Group III Cations (Unknown Solution)

\[ 50 \text{ mg} \text{ of unknown} + 1 \text{ mL} 12\text{M HCl} \rightarrow \]

Immersion in boiling HNO_3 & stir 2 glass stirring rods until completely dissolved.

\[ 1 \text{ mL} \text{ H}_2\text{O} \]

B) Precipitation of Group III Cations

\[ \text{Add 15 drops 6 M NaOH} \]

Centrifuge & save precipitate for part C.

Decant supernatant into test tube for part F.

C) Test for Mn^{2+}

\[ 1-2 \text{ mL} 15\text{M HNO}_3 \rightarrow \]

Heat in hot HNO_3 & transfer 0.5 mL into another 75 mm test tube.

Save the rest for part D.
C. (cont)

2. Add slight excess solid Na₂BiO₃ to dissolve.
   → Centrifuge. Deep purple MnO₄⁻ ion confirms presence of manganese.

D. Test for Fe³⁺

1. 10 drops 1 M NH₄Cl
   15 M NH₃ in hood until basic to litmus.
   → Brown ppt. indicates iron may be present.
   Centrifuge & decant.
   Save decant in test tube for part E.
   Remaining soln. from part C.1.

2. 6 M HCl + 2 drops 0.1 M NH₃SCN.
   → Blood-red color identifies iron as Fe(SCN)²⁻.

E. Test for Ni²⁺

1. Add 3 drops H₂DMAQ
   → Pink precipitate indicates Ni²⁺ as Ni(H₂DMAQ)₂⁻.
   Decant from part D.2.

F. Test for Al³⁺

1. 6 M HNO₃
   Acidify until blue litmus turns red.
   → White gelatinous ppt. indicates presence of aluminum.
   Centrifuge & decant supernatant for part G.
   Add drops 6 M NH₃ until basic to litmus.
   + 5 more drops.
   Heat in hot H₂O.
F. (cont)

2. 2-3 drops 6M HNO₃

⇒ 2 drops aluminum reagent - stir.

⇒ 4 drops of 6M NH₃ until basic to litmus.

⇒ If red ppt., indication of Al₃⁺.

G. Test for Zn²⁺

1. Add 6M HCl until acidic to litmus.

⇒ Supernatant from Part E.

⇒ 3 drops 0.2M K₃[Fe(CN)₆]

⇒ Stir.

⇒ Light green ppt. indicates Zn²⁺.

IV. Safety Procedures:

<table>
<thead>
<tr>
<th>MSDS Info.</th>
<th>Health Hazards</th>
<th>Precautions</th>
<th>1st Aid</th>
<th>Flammability</th>
<th>Cleaning Smoke</th>
</tr>
</thead>
<tbody>
<tr>
<td>HCl</td>
<td>Corrosive</td>
<td>Avoid contact &amp; breathing vapor</td>
<td>Flush 15 min.</td>
<td>reacts with metals</td>
<td>H₂O</td>
</tr>
<tr>
<td>H₂SO₄</td>
<td>Corrosive</td>
<td>Don't get in eyes, gasses</td>
<td>Flush 15 min.</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>NaOH</td>
<td>Corrosive</td>
<td>goggles/slasses</td>
<td>Flush 15 min.</td>
<td>N/A</td>
<td>Neutralize &amp; wash down</td>
</tr>
<tr>
<td>NH₄Cl</td>
<td>eye/skin irritant</td>
<td>goggles</td>
<td>Flush 15 min.</td>
<td>N/A</td>
<td>Swab, moist breathing mask.</td>
</tr>
<tr>
<td>Aluminum Reagent</td>
<td>eye/skin irritant</td>
<td>goggles</td>
<td>Flush 15 min.</td>
<td>N/A</td>
<td></td>
</tr>
</tbody>
</table>

NH₄SCN

Mn, Fe, Zn, Ni

Eye/skin irritant gasses

\[\text{Neutralize with dilute acid.}\]
V. Data:

**Flow Chart**

\[
\text{Al}^{3+}, \text{Ni}^{2+}, \text{Zn}^{2+}, \text{Fe}^{2+}, \text{Mn}^{2+}
\]

\[\Downarrow \text{12 M HCl}\]

\[\Downarrow \text{6 M NaOH}\]

Supernatant

\[\text{Al(OH)}_3, \text{Zn(OH)}_4^{2-} (aq)\]

\[\Downarrow \text{6 M HNO}_3\]

\[\Downarrow \text{NH}_3\]

\[\text{Zn(NH}_3)_4^{2+}\]

\[\Downarrow \text{6 M HCl}\]

\[\text{K}_2\text{Zn}[\text{Fe(CN)}_6]_2\]

(light green)

\[\Downarrow \text{6 M NH}_3\]

\[\text{Al(OH)}_3\]

(aluminum, red)

[aluminum, red]

\[\Downarrow \text{aluminum, reagent}\]

\[\Downarrow \text{Ni}^{2+}, \text{Fe}^{2+}\]

\[\text{H}_2\text{DMSO}\]

\[\Downarrow \text{SCN}^-\]

(purple)

\[\text{MnO}_4^-\]

(blood-red)

\[\text{Fe(SCN)}_6^{3-}\]

(brick-red)

\[\text{Ni(HDMSO)}_2\]

(blood-red)

\[\text{Fe(OH)}_3, \text{Ni(OH)}_2, \text{Mn(OH)}_2\]

\[\text{Ppt}\]

\[\text{Na}_3\text{BiO}_3\]
I. Esters: Synthesis & Analysis

Tommy Trojan

Chem 105
T.A.: Diane
16 November 24

II. Purpose: To prepare a sample of oil of wintergreen in order to determine the purity.

III. Procedure:

1. 125 mL round bottom flask
   - 6g salicylic acid
   - 40 mL acetone
   - 10 drops conc. H₂SO₄
   - Heat flask over
   - 5 min

2. 10 mL ice/H₂O (0°C)
   - Chill in ice bath

3. Decant liquid & add 20 mL H₂O
   - Shake & chill again

4. Heat
   - H₂O & salicylic acid
   - Add 1% ferric chloride (1 drop)
   - to each test tube
B. Purification of Aspirin By Recrystallization

1. 20 mL ethanol
   Add 50 mL H2O
   Warm mixture.

2. Aspirin solubility = 0.25 g/100 mL H2O
   Calculate % yield.

C. Purity of Aspirin

1. 0.3 g of prepared aspirin
   25 mL 95% ethanol
   Add 2 drops phenolphthalein indicator.

2. Prepare base 0.1 M NaOH.

3. Titrate aspirin sample until faint pink.

D. Synthesis of Methyl Salicylate

5 mL methyl alcohol
Add 3 drops conc. sulfuric acid.
Place in warm bath for 15 min.
II. Purpose: The purpose of this experiment is to conduct and observe an electrochemical reaction and the relationships between current, time, and the moles of chemicals consumed and produced. Using the data obtained from our observations, we will determine the value of the Faraday.

III. Procedures:

1. Clean flat copper electrode with steel wool.
2. Weigh electrode and record mass.
3. Set up the circuit with power supply and ammeter.
4. Use a rubber tube to connect the vacuum trap.
5. Collect 30 mL gas.
6. Continue electrolysis for one more minute.
7. Rinse electrode and acetone.
8. Rinse black deposit and dissolve the Cu wire.
9. Record voltage.
10. Repeat procedure.
Investigating the Reaction of Potassium Permanganate and Oxalic Acid

**Purpose:** The purpose was to find out the rate of reaction of potassium permanganate and oxalic acid under different conditions. The rate equation for the reaction was determined with the equation: \( \text{RATE} = k \cdot [\text{oxalic acid}]^x \cdot [\text{potassium permanganate}]^y \).

The reaction will be either a 1st or 2nd order reaction. A 1st order reaction was when the rate of reaction doubles when concentration of 1 reactant was doubled. (2nd = both reactants)

The overall order was determined \( x + y = \) and the effect of temperature on rate of reaction was also studied.

**Flow Chart:**

1. Stirred mixture
2. Solution purple to red to yellow
3. Start timer when \( \frac{1}{2} \) KMnO₄ was added
4. Stopped timer when last trace of red disappears

<table>
<thead>
<tr>
<th>Reactants</th>
<th>( \text{exp} #1 )</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>vol of oxalic acid, mL</td>
<td>5.00</td>
<td>10.00</td>
<td>5.00</td>
</tr>
<tr>
<td>vol of KMnO₄, mL</td>
<td>1.00</td>
<td>1.00</td>
<td>2.00</td>
</tr>
<tr>
<td>vol of distilled H₂O, mL</td>
<td>6.00</td>
<td>1.00</td>
<td>6.00</td>
</tr>
</tbody>
</table>

**Exp. #1**

- Oxalic acid is poisonous
- Added 5.00 mL oxalic acid + 6.00 mL H₂O (distilled)
- For initial time
  - Start timer when \( \frac{1}{2} \) KMnO₄ was added

- Recorded
  1. Molarity of each reactant
  2. Vol of distilled H₂O
  3. Time
  4. Room temp

- Repeated until time within 10 sec

**Diagram:**

- 20 x 15 cm test tube
- Mixed w/ glass rod
- Avoided contact w/ KMnO₄
Exp 2: 3
Repeated Ex 1
using quantities of
reagents from table
Exp 4
did at 10 degree
↑ room temp

1000 mL
beaker

 kep warm
$\frac{1}{2}$ (10-12 hr)
$\frac{1}{2}$ $\text{H}_2\text{O}$

Maintained temp
w/ in ±1 degree

Recorded
1. Vol/molarity reactants
2. Vol dist. $\text{H}_2\text{O}$
3. Time
4. Temp of $\text{H}_2\text{O}$

Added
1.00 mL
$\text{KOH}$

Added
5.00 mL
0.1 M acetic acid + 6.00 mL
dist $\text{H}_2\text{O}$

20x150
test tube

Mixed

Stopped timer w/ last trace of
red

Mixed

Started timer
when $\frac{1}{2}$ acid was
added

After 10 min
transferred

Added 0 to
Water bath

Exp 5: 6
Used procedure
from 4, but 4
exp 5 20 degrees
above room temp
and 30 degrees
room temp for
Exp ± 6

Repeate till
collapsed time can
be repro'd w/in 10
seconds

Recorded data
I. Determination of the Formula & Dissociation Constant of a Complex Ion

Tommy Trojan
31 October 94
T.A.: Diane

II. Purpose: To allow us to experimentally determine the formula of the ammonia-silver complex ion (Ag(NH₃)ₓ⁺), as well as determine the dissociation constant (Kf) of the complex by using the data from the above determination along with the value of the solubility product constant (Ksp) of Silver-Iodide (AgI).

III. Procedure:

1. 3-250mL Erlenmeyer flasks

   **1.** Add 20.0mL 0.01M AgNO₃ to 3 flasks labeled #1, #2, and #3.

   **2.** Add 2.0M NH₃ to one 20mL Erlenmeyer flask and titrate with KBr solution until a faint, permanent cloud of silver bromide forms.

   **3.** Repeat procedure #1 but when adding HNO₃, add 0.01M NaBr instead of KBr.