Experiment 4: Preparation of Chloropentaamminecobalt (III) chloride and Nitropentaamminecobalt(III) chloride

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We pledge our honor we have abided by the Stevens Honor Code.

Procedure

Preparation of Chloropentaamminecobalt (III) chloride

1. Combine 5.0 g of ammonium chloride and 30 mL of conc. aqueous ammonia in a 250 mL Erlenmeyer flask
2. Add stir bar to flask and start stirring
3. Slowly add 10 g of fine powder cobalt (II) chloride 6-hydrate.
4. While stirring, dropwise add 8mL of 30% H_2O_2
5. Once reaction ceases, slowly add 30 mL of conc. HCl
6. Turn on hot plate to 85C, heat for 20 minutes
7. Cool to room temperature and Filter $[Co(NH_3)_5Cl]Cl_2$
8. Wash crystals with ice water several times(max 20 mL), wash with 20 mL cold HCl (6 M)
9. Dry in 100 C oven for 2 hrs.

Preparation of Pentaamminenitritocobalt(III) chloride

1. Prepare solution of 8 mL ammonia (aq) in 80 mL of water
2. Heat on hot plate, and a stir bar
3. Add 5.0 g of $[Co(NH_3)_5Cl]Cl_2$ (dry)
4. Continue heating / stirring until colored product dissolves
5. Filter off dark brown/black cobalt oxide

Procedure cont.

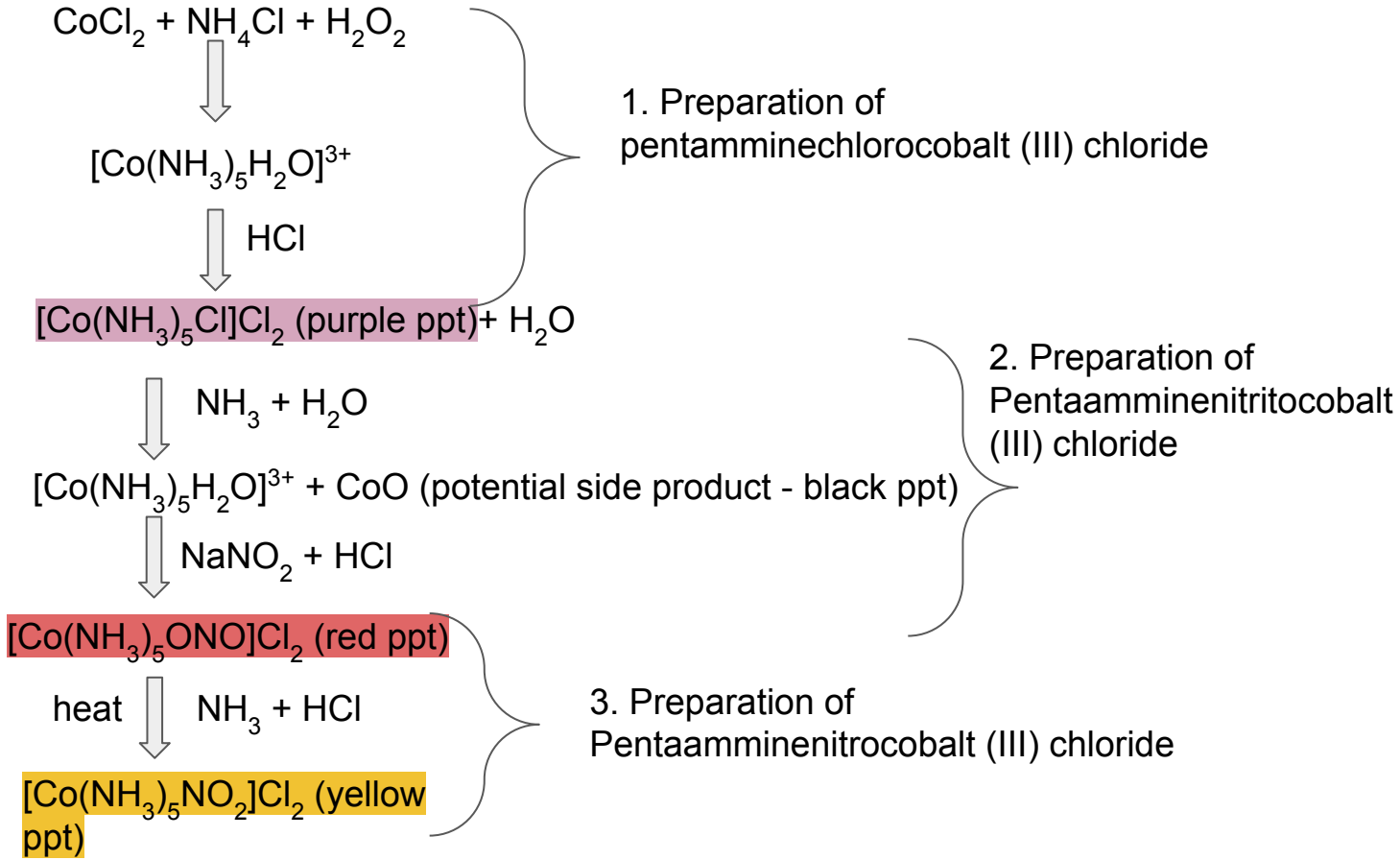
Preparation of Pentaamminenitirtocobalt (III) chloride

6. Cool solution to ~10C
7. Add 2M HCl slowly, until neutral
8. Add 5.0 g of $NaNO_2$ and 5 mL of 6M HCl.
9. Place in ice bath for 1 hr.
10. Filter precipitated $[Co(NH_3)_5ONO]Cl_2$
11. Wash product with 25 mL ice water, 25 mL of alcohol, then dry on lab bench for 1 hr

Preparation of Pentaamminenitrocobalt (III) chloride

1. Boil 20 mL of water
2. Add a few drops of ammonia (aq) and 2.0 g of $[Co(NH_3)_5ONO]Cl_2$
3. As solution cools, add 20 mL of concd/ HCl
4. Filter crystalized $[Co(NH_3)_5NO_2]Cl_2$ with Buchner funnel
5. Wash crystals with 13 mL alcohol
6. Allow to dry in air for 2 hrs

Flowchart

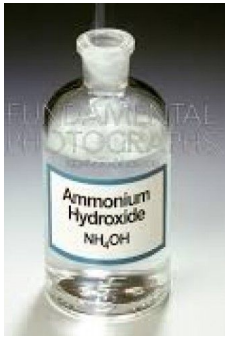


ppt - precipitate

1. Preparation of pentaamminechlorocobalt (III) chloride

1. Make a solution of 5.0 g ammonium chloride in 30 mL concentrated aqueous ammonia in 250 mL Erlenmeyer flask.
2. Place flask on magnetic stirrer hot plate, turn on stirring, and add 10 g powdered cobalt (II) chloride hexahydrate.
3. With continued stirring, add 8 mL 30% hydrogen peroxide dropwise.

5.0 g



30 mL

Turn on stirring

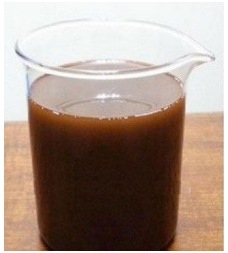
Add 10g

Cobalt (II) chloride hexahydrate

8 mL

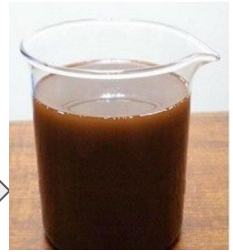
1. Preparation of pentaamminechlorocobalt (III) chloride

4. When evidence of reaction has ceased, slowly add 30 mL concentrated HCl.
5. With continued stirring, turn hot plate and adjust temperature to 85°C. Heat for 20 minutes at this temperature.
6. Cool mixture to room temperature.



When no more color change or gas evolution

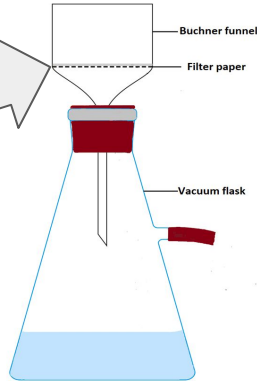
Add 30 mL conc. HCl



85°C, 20 min, with stirring

Cool to room temp

Maroon color



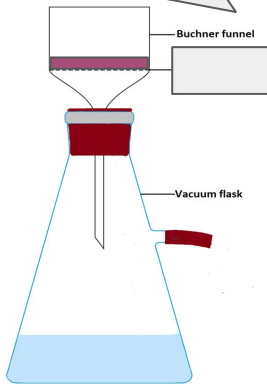
Vacuum filter precipitate

1. Preparation of pentaamminechlorocobalt (III) chloride

7. Wash purple crystals with ice water not exceeding 20 mL, then 20 mL cold 6M HCl, and dry in 100℃ oven for two hours.

Ice water up to 20 mL

20 mL cold 6M HCl

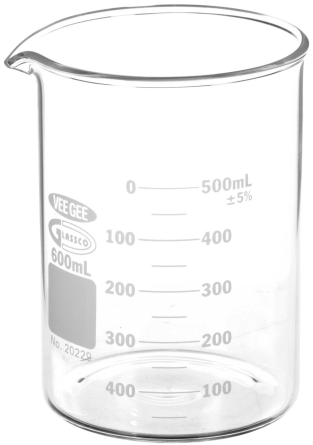


100℃, 2 hours

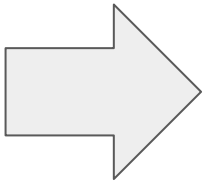
Chloropentaamminecobalt (III) chloride

2. Preparation of Pentaamminenitritocobalt (III) chloride

80 mL water

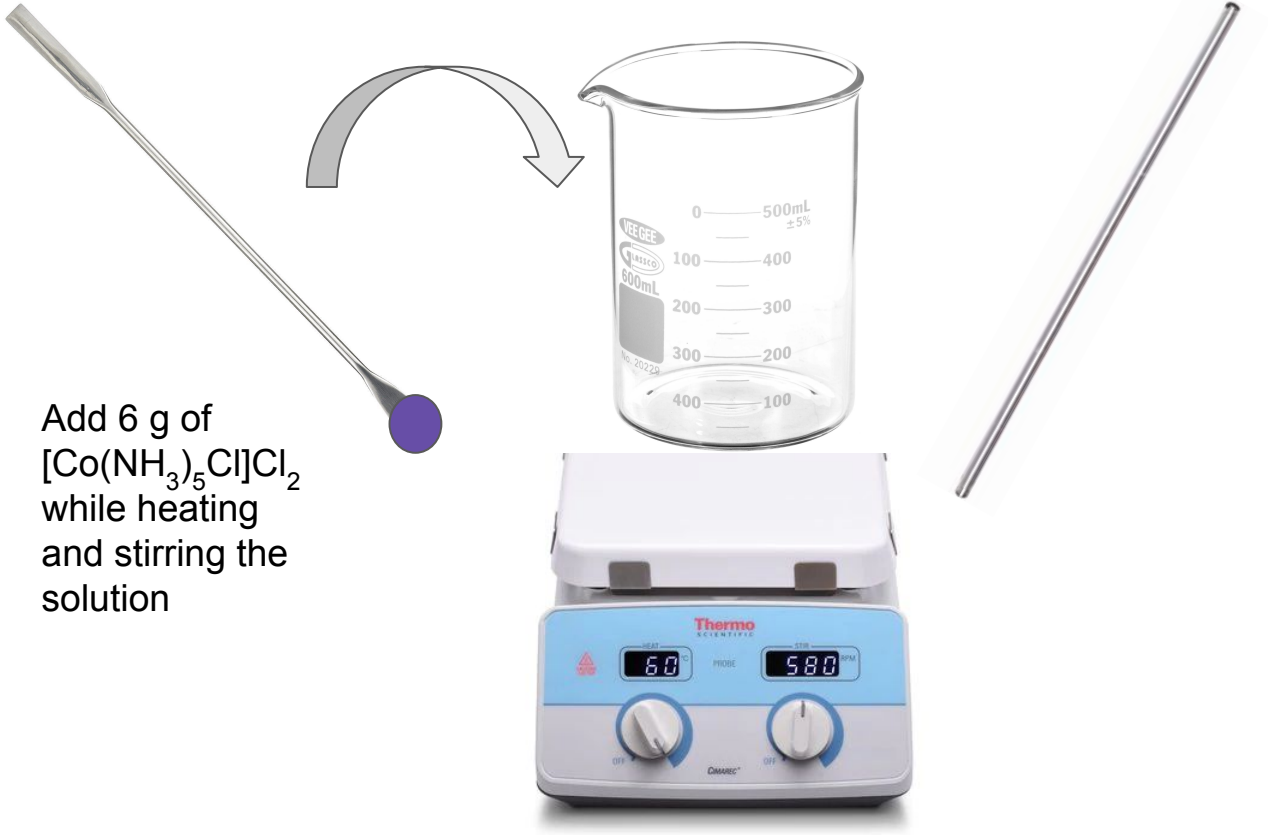


8 mL ammonium (aq)



heat a solution on the stirrer-hot plate

2. Preparation of Pentaamminenitritocobalt (III) chloride

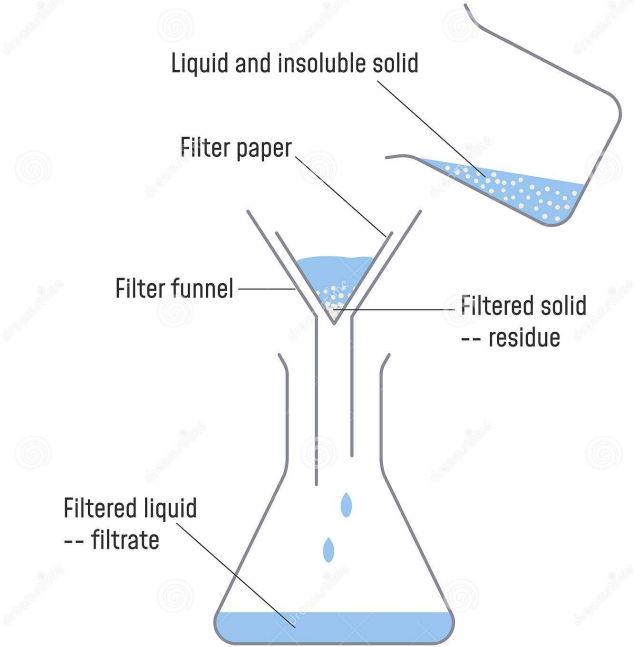


Add 6 g of $[Co(NH_3)_5Cl]Cl_2$ while heating and stirring the solution

Heat until completely dissolved

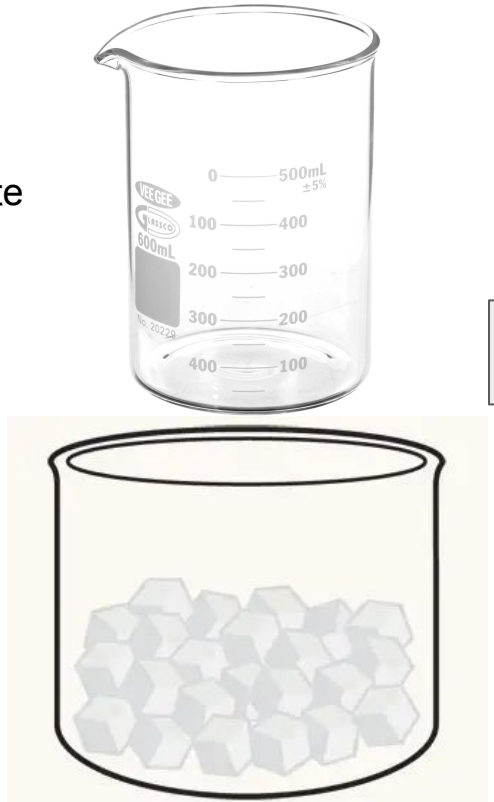
2. Preparation of Pentaamminenitritocobalt (III) chloride

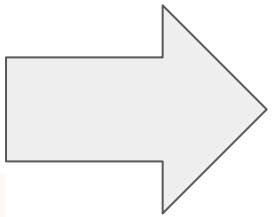
If a dark brown to black precipitate of cobalt oxide forms, filter it off



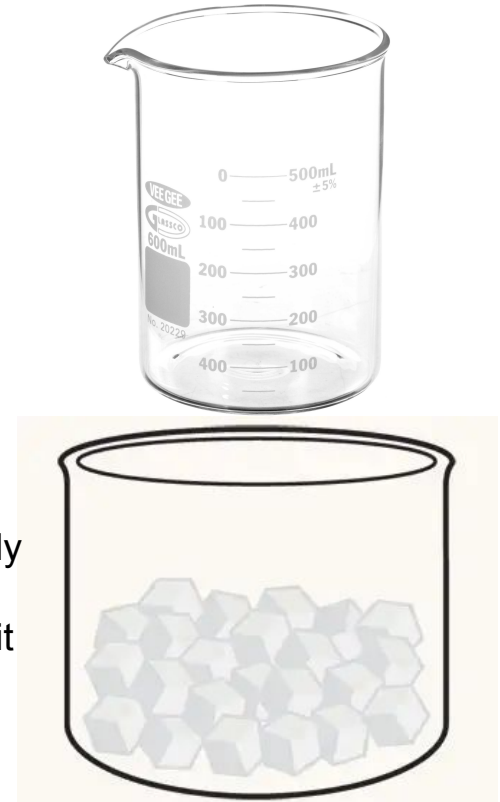
2. Preparation of Pentaamminenitritocobalt (III) chloride

Cool the filtrate which should be a clear solution to about 10°C.



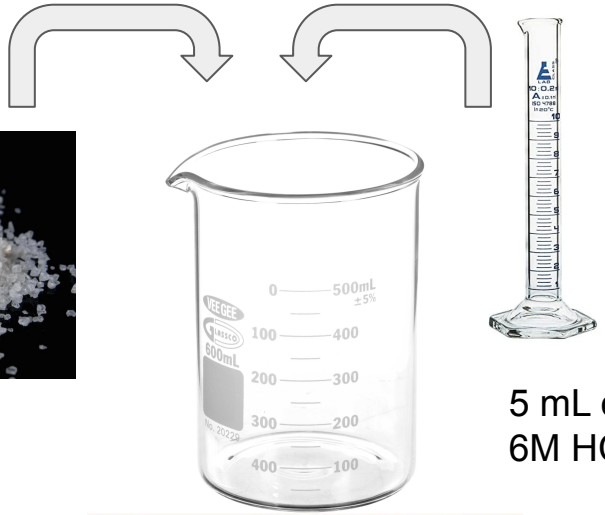


Add 2 M HCl slowly while keeping the solution cold until it is just neutral to litmus.

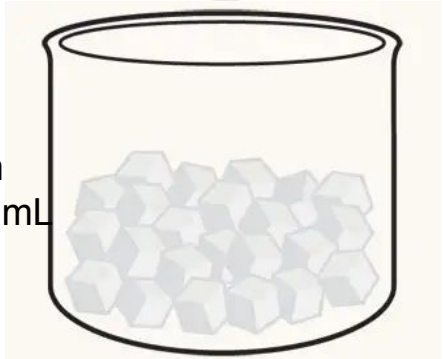


2. Preparation of Pentaamminenitritocobalt (III) chloride

5.0 g of sodium nitrite

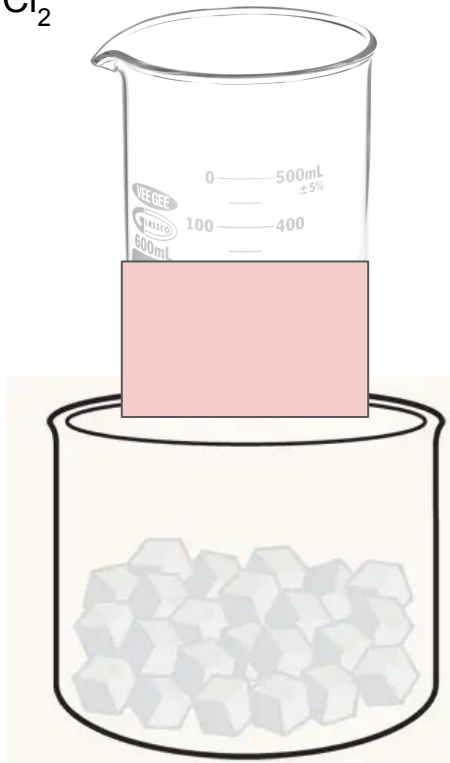


5 mL of 6M HCl

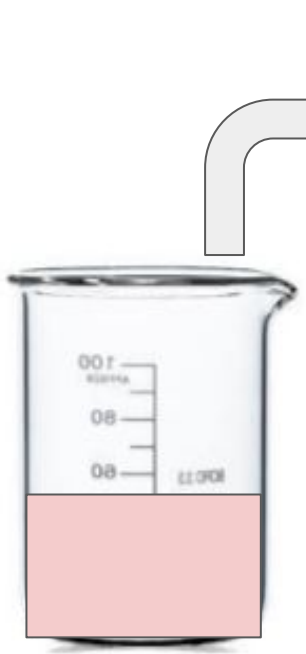


Add 5.0 g of sodium nitrite followed by 5 mL of 6 M HCl.

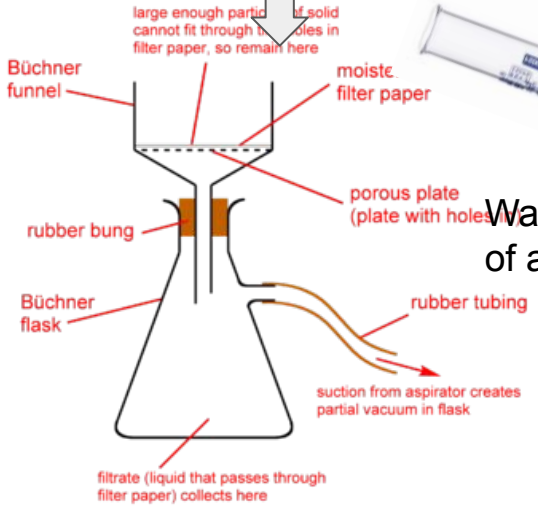
Should see salmon pink $[Co(NH_3)_5ONO]Cl_2$

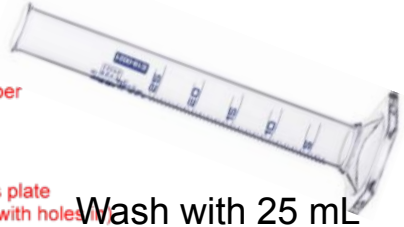


2. Preparation of Pentaamminenitritocobalt (III) chloride



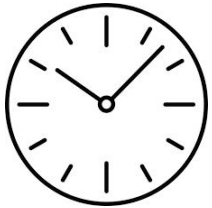
Wash with 25 mL
of ice cold water





Wash with 25 mL
of alcohol

$[Co(NH_3)_5ONO]Cl_2$ precipitate
allowed to air dry for 1 hour



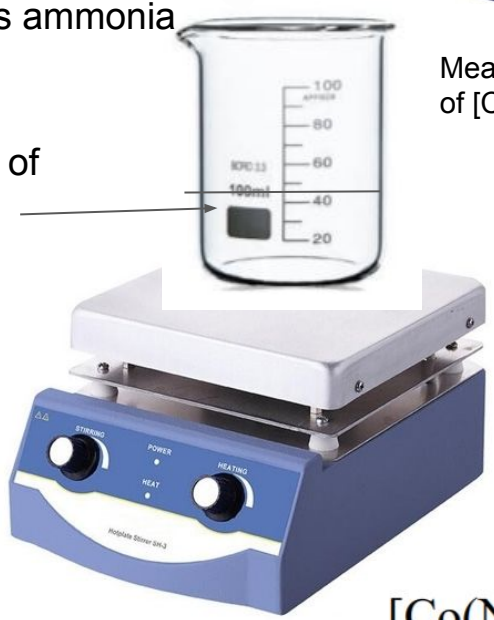
3. Preparation of Pentaamminenitrocobalt (III) chloride

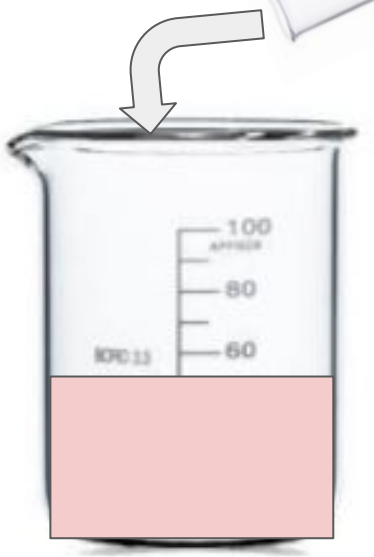
Add few drops of aqueous ammonia

Measure and add 2.0 g of $[Co(NH_3)_5ONO]Cl_2$

Add 20 mL HCl

20 mL of water boiled





Let solution cool

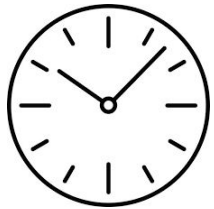
$$[Co(NH_3)_5ONO]^{2+} \xrightarrow{\text{heat}} [Co(NH_3)_5NO_2]^{2+}$$

3. Preparation of Pentaamminenitrocobalt (III) chloride

Filter product

Wash with 13 mL alcohol

Crystallized $[Co(NH_3)_5NO_2]Cl_2$ allowed to air dry for 2 hours



Conclusion

Accomplished: In this experiment, pentaamminechlorocobalt (III) chloride was prepared using H_2O_2 to oxidize Co^{2+} into Co^{3+} and HCl for the ligand exchange. The pentaamminechlorocobalt (III) chloride was then used to prepare pentaammminenitritocobalt (III) chloride using $NaNO_2$ for a ligand exchange. Lastly, the pentaammminenitritocobalt (III) chloride was isomerized into pentaammminenitrocobalt (III) chloride by heating, since these two compounds are linkage isomers.

Learned: Throughout this lab, we learned about complex ions and coordination compounds. The different types of structural isomers (coordination and linkage) were learned through the experiment, as well as how they can be prepared in the laboratory through ligand exchange reactions and isomerization.

Any Issues: The main issues in the experiment were not obtaining enough precipitate of the coordination compounds to proceed to the next step in the procedure due to inefficient reactions or improper heating during the reactions.

Future Recommendations / Applications: To increase the amount of product obtained in each step of the reaction the temperature should be very closely monitored throughout the process and adjusted to be at the optimal level for each step.

Post Lab Questions

1. What is the name of the following type of reaction?
   a. $[Co(NH_3)_5H_2O]^{3+} + 3Cl^- \rightarrow [Co(NH_3)_5Cl]Cl_2 + H_2O$
   b. This reaction is a ligand exchange reaction where the counterion and ligand switch, forming the coordination isomer.
2. Why did ammonia not get displaced in the above reaction?
   a. Ammonia is not displaced because its interaction with cobalt is more stable than that of water, so the water can be easily replaced, whereas the ammonia can not.
3. Name two different techniques which can be used to identify whether it is nitrito complex $[Co(NH_3)_5ONO]^{2+}$ or nitro complex $[Co(NH_3)_5NO_2]^{2+}$?
   a. The first technique is by observation of the different colors. The nitrito complex is a red color and the nitro complex is a yellow color. The second technique is by melting point determination, the H-bonds that are possible in nitro will increase the melting point because the solid nitro complex is more stable than the nitrito complex.