

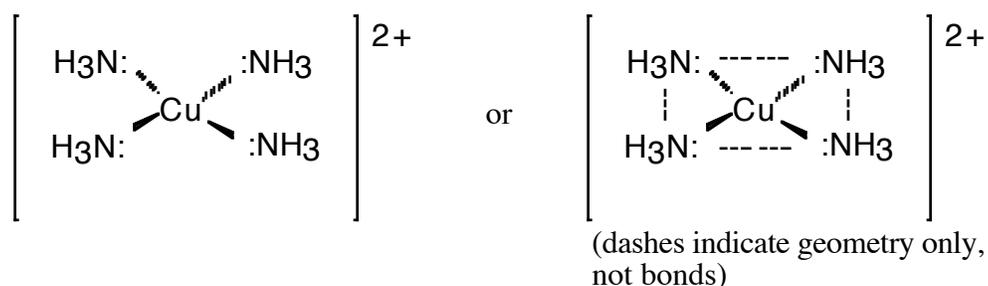
## Experiment 6

### Preparation of an Inorganic Cobalt Complex: $\text{Co}(\text{NH}_3)_n\text{Cl}_3$

#### Discussion

Werner coordination complexes are compounds formed between transition metal ions and a variety of organic and inorganic ions or neutral molecules. The elucidation of the details of the structure of these complexes is of current interest, although Werner correctly predicted the geometry of many of them more than fifty years ago.

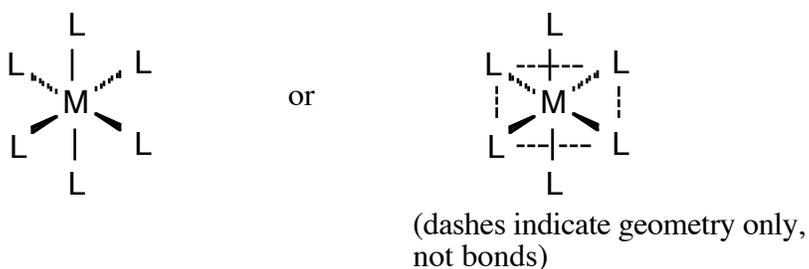
A well-known complex ion is the tetraammine copper(II) ion in which four ammonia molecules are arranged (coordinated) about a central cupric ion at the corners of a square.



The nature of the bonds between the central atom and the coordinating groups (termed ligands) varies with the nature of the species involved. In some complexes the bonding may be described as largely covalent in character, whereas in others electron sharing is relatively unimportant and the interaction between the central atom and the ligands is essentially electrostatic in nature.

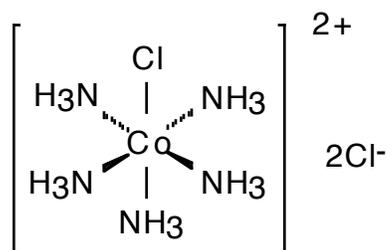
#### Octahedral Complexes

In these, six ligands (L) are arranged about the central atom (M) at the apices of an octahedron. The resultant charge on the complex depends on the nature of M and the ligand L.

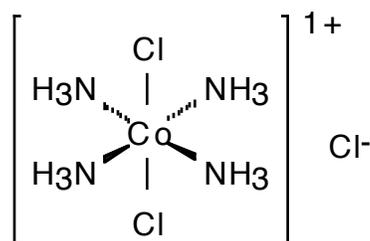


If M is trivalent cobalt ( $\text{Co}^{3+}$ ) and L is ammonia ( $\text{NH}_3$ ), the complex ion will be tripositively charged. An example is, then, hexammine cobalt(III) chloride -  $[\text{Co}(\text{NH}_3)_6]^{3+} 3\text{Cl}^-$  where the three chloride ions are not part of the complex but simply independent ionic species such as present in potassium chloride, calcium chloride, etc.

One or more of the ammonia ligands may be replaced by chloride ions to yield complex compounds such as:



chloropentammine cobalt(III)  
chloride



dichlorotetraammine cobalt(III)  
chloride

etc.

The chloride ions attached to the central metal atom do not ionize readily, and thus the complex compound behaves accordingly. For example, chloropentammine cobalt(III) chloride is a 2:1 electrolyte similar to calcium chloride ( $\text{Ca}^{2+} 2\text{Cl}^-$ ), only two-thirds of the chlorine being outside the complex attached to it by ionic bonds.

In this lab experiment you will synthesize a cobalt coordination complex of the general formula  $\text{CoCl}_3(\text{NH}_3)_x$ , with  $x=3-6$ . In experiment 7 to 9 you will perform four experiments to determine the unknown  $x$ . With any luck, all of your data will point to the same structure.

### i) Preparation of Compound X

**Procedure** (Adapted from *Inorganic Syntheses*, Volume 9, p. 160, 1967)

Fit a 250-mL Erlenmeyer flask with a cork stopper. Measure 20 mL of concentrated ammonia from the dispenser in the hood into your flask. Ammonia is an irritating and pungent gas. Avoid breathing it or dropping it on skin. Always replace stoppers of dispensing bottles immediately. Stopper the Erlenmeyer flask before bringing it out from the hood in order to contain the ammonia.

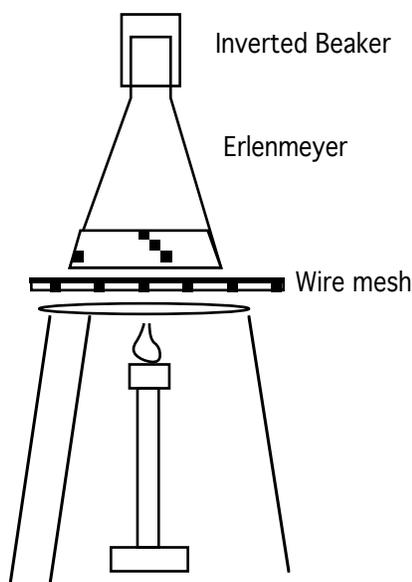
Weigh out 3.0 g of ammonium chloride ( $\text{NH}_4\text{Cl}$ ) to the nearest 0.1 g. Weigh into a square piece of weighing paper whose edges have been folded up sharply. Working in the hood, dissolve the approximately 3.0 g of ammonium chloride in the ammonia solution with a little **gentle** swirling of the loosely stoppered flask.

Weigh accurately by difference about 5 g of granulated cobalt(II) chloride hexahydrate ( $\text{Co}(\text{H}_2\text{O})_6\text{Cl}_2$ ). Proceed as follows:

- 1) Obtain a clean, dry, stoppered weighing bottle from your equipment. **If bottle is not clean**, wash with tap water and cleanser, rinse with distilled water, and dry in oven at  $110^\circ\text{C}$  for about a half-hour. When drying weighing bottles (empty or containing a solid), place cover in a tilted position on top of bottle to permit water vapor to escape. Place bottle and cover within a 250-mL beaker covered with a ribbed watch glass. (This prevents foreign material from dropping into bottle and permits water vapor to escape.) Be sure to identify bottle and beaker by marking the ground-glass area with a **lead** pencil; do **not** use a wax marking pencil. After drying period, remove bottle with tongs and place in covered desiccator to cool.
- 2) Place about 5 g of the cobalt reagent in the weighing bottle. To assist you in judging the appropriate volume, look at the exhibit.
- 3) Weigh the bottle and contents. Record weight of bottle and contents in your laboratory notebook.

- 4) Empty contents of bottle into the ammonia solution **slowly** in small portions with constant swirling of the flask. Wait until each portion appears to have been converted to a yellow-brown precipitate before adding the next portion. The addition should require about ten minutes. It is not necessary to add all of the cobalt salt, but be certain that all of the salt that leaves the bottle enters the reaction flask. A yellow brown slurry is the result at this point.
- 5) Re-weigh bottle and any remaining cobalt reagent in it and record as before.
- 6) Determine the weight of the cobalt salt added to the flask and record.

In the hoods you will find a 30% aqueous solution of hydrogen peroxide ( $\text{H}_2\text{O}_2$ ). (This substance is corrosive; avoid getting it on the skin.) Add 6.0 mL of the  $\text{H}_2\text{O}_2$  solution slowly to the reaction mixture in the flask, with constant swirling. This will produce a vigorous reaction with effervescence of gaseous oxygen. When the effervescence has virtually ceased, add **SLOWLY IN THE HOOD** 20 mL of **concentrated** hydrochloric acid (HCl), measured from a graduated cylinder. During the addition of the acid, the temperature of the reaction mixture rises, and the purple product precipitates leaving a blue supernatant liquid. The mixture is heated for about 15 minutes on a tripod/Bunsen setup. Do not cork the flask. Invert a small beaker over the neck of the Erlenmeyer flask. (See figure 1. below.) Place a clean thermometer in the reaction mixture to determine its temperature. The temperature should be regulated carefully to within  $75\text{-}85^\circ\text{C}$  by adjusting the size and temperature of the flame.



**Figure 1.** Heating liquid in Erlenmeyer Flask. Do not cook your thermometer.

After the 15-minute heating period, the flask is cooled in an ice-water bath. The ice-water bath is prepared by placing two handfuls of ice in a large beaker and filling it half-full of cold water from the tap. (An ice-water bath should always be a mixture of ice and water for rapid cooling, rather than ice alone.)

Prepare a Büchner funnel setup according to figure 2 and the directions given by the laboratory instructor. Important features: filter paper small enough to fit **flat** on the funnel but large enough to cover all of the holes, paper wet with a little water to seal it in place, trap in place, suction on before adding slurry, flask holding the funnel should be clamped to metal stand.