
i) Preparation of Trans Form:

Six hundred grams of a 10% solution of ethylene diamine is added with stirring, to a solution of 160 gm of cobalt chloride 6-hydrate in 500 ml of water in a 2-litre beaker or bottle. A vigorous stream of air is drawn or passed through the solution for 10 or 12 hours. (Longer aeration causes undesirable secondary reactions to take place) 350 ml of conc. HCl is added and the solution is evaporated on the steam bath until a crust forms over the surface. The solution is allowed to cool and stand overnight before the bright-green square plates of the hydrochloride of the trans form are filtered. These are washed with alcohol and ether and dried at 110°C. At this temperature, the hydrogen chloride is lost, and the crystals crumble to a dull-green powder.

ii) Preparation of Cis Form:

Conversion of trans $\text{Coen}_2\text{Cl}_2\text{Cl}$ to its cis form is brought about by evaporating a neutral solution of the trans form to dryness on the steam bath. The unchanged trans form may be washed out with a little cold water or the transformation
may be completed by repeating the evaporation. It should be repeated not more than two or three times however as some decomposition takes place.

B. Preparation of Cis-aquochloro bis-ethylenediamine Cobalt(III) Sulphate. \( \text{Ref: Werner, A - Annalen 386, 122 (1912)} \)

10 gm of \( \text{trans-Coen}_2\text{Cl}_2\text{-Cl} \) was dissolved in 20 c.c of water and heated on a free flame, until the salt dissolved and the colour became blue-black. It was then allowed to stand for one hour and then 10 gm of finely powdered \( \text{(NH}_4\text{)}_2\text{SO}_4 \) were added. After 12 hrs standing a crust of blue red crystals separated which contained some green crystals. It was then stirred with 10 c.c cold water vigorously, whereby the green salt dissolves. It was filtered and the red sulphate was washed repeatedly with cold water until the wash-water became pure red. It was finally washed with ethanol and dried in air. For recrystallisation the red mass is dissolved in minimum amount of water (by warming if required). It was cooled and finally excess of ethanol was added whereby the recrystallised product is obtained.

C. Preparation of Trans-hydroxochloro bis-ethylenediamine Cobalt(III) Chloride \( \text{Ref. Meisenheimer and Kiderlen - Annalen 438, 241 (1924)} \)

Addition of an aqueous solution of diethylamine to trans-\( \text{Coen}_2\text{Cl}_2\text{-Cl} \) produces a dove-grey paste, which was filtered
off, washed successively with 7:3 ethanol-water, ethanol and ether. The crude product was dissolved in ice-cold water (6.5 gm in 25 ml) and the solution filtered as quickly as possible. Ethanol was added and the mixture cooled in ice. The crystals formed were filtered off, washed with alcohol and ether and dried in a stream of air. At least one more recrystallisation was necessary to give a pure product.
APPENDIX - 2

Methods used during elemental analysis

A) Estimation of Cobalt :

Cobalt was estimated gravimetrically as follows:
Weighed amount of the sample was taken in a vitreous- it was
heated very strongly until it was converted to black-oxide. Then
it was cooled - added little amount of conc. HCl and evaporated
to almost dryness in a very low flame. Finally drops of conc.
H$_2$SO$_4$ were added and it was also evaporated to dryness by a
low flame. Pink coloured CoSO$_4$ thus produced was kept in a
furnace at 400°C for half an hour cooled and weighed.

B) Estimation of Nitrogen :

Nitrogen content of the samples were estimated by Duma's
method.

C) Estimation of Chlorine :

Weighed amount of sample was taken in a beaker, dissolved
in water by the addition of few drops of conc. HNO$_3$ and subse-
quent boiling. Then excess of AgNO$_3$ was added and the solution
was allowed to stand overnight. The AgCl precipitated was filtered
dried and weighed.

D) Estimations of Carbon and Hydrogen :

Estimations of carbon and hydrogen were done by the
Central Drug Research Institute, Lucknow.