## NOTE

# Preparation of Hydroxamic Acid Complexes of $Cr^{3+}$, $Co^{2+}$ and $Ni^{2+}$

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In the present note an attempt to synthesise and characterise complexes of $Cr^{3+}$, $Co^{2+}$ and $Ni^{2+}$ with hydroxamic acid.

The complexing behaviour of hydroxamic acids with meltal ions particularly transition metals has been extensively studied but no work has been done on 1-indenyl acetatohydroxamic acid complexes of Ni(II), Cr(III) and Co(II). Thus we have made an attempt to synthesise and characterise complexes with this ligand.

*Preparation of 1-indenyl acetatohydroxamic acid:* The ligand was prepared as follows: one mole of KOH (56.1 g) was dissolved in 144 mL of methanol and the resulting solution was added to a solution of hydroxylamine hydrochloride (prepared by dissloving 6 mole of hydroxylamine hydrochloride in 240 mL of methyl alcohol). Both the solutions were thoroughly mixed at 30–40°C and the mixture was kept in ice-bath for about 5 min till the complete precipitation of KCl occurred. Now 5 mol of ethyl (1-indenyl) acetate was added in instalments with constant shaking to the above mixture and after the addition was complete, the solution was filtered immediately through suction. The residue in the funnel was washed with a little $CH_3OH$. The filtrate was allowed to stand for 48 h in the Erlenmeyer flask when crystals of the potassium salt formed were filtered. The crystals were washed with a little absolute alcohol and dried in air. About half of the yield was mixed with 80 cc of 1.25 N acetic acid and stirred while heating until a clear solution was obtained. The solution was allowed to cool at room temperature and finally chilled in an ice-bath when 1-indenyl aceto-hydroxamic acid separated out as brownish yellow crystals.

The organic reagents, bases (pyridine, $\alpha$, $\beta$ or $\gamma$ picoline, quinoline) and solvents used in the present investigation were obtained from B.D.H., England or Germany or Bush Ltd. or Fluka, Switzerland.

*Preparation of Ni(II) Complex ($NiL_2 \cdot 2H_2O$):* An ethnolic solution of $NiCl_2 \cdot 6H_2O$ (0.01 mol in 14 mL of $C_2H_5OH$) was treated with aqueous alcoholic solution (0.02 mole) in 1 : 2 ratio and the mixture was refluxed on water bath for 1/2 h. The solution was cooled and made alkaline with a few drops of KOH

solution. The mixture was allowed to stand for 1 h and the pale green precipitate was filtered. It was washed with water and a small amount of alcoholic water and the compound was dried at room temperature.

*Preparation of Co(II) Complex ($CoL_2.2H_2O$):* An aqueous ethanolic cabalt sulphate solution was treated with ligand in 1 : 1 ratio and was just made alkaline. The content was warmed on water bath for 4 h and left at room temperature. Light pink coloured precipitate was obtained which was filtered, washed several times with alcoholic water till free from sulphate ion and dried at 100°C.

*Preparation of Cr(III) Complex ($CrL_3$):* Methanolic solution of chromium chloride and ligand were mixed in the ratio 1 : 3 and the mixture was just warmed on water bath. A greenish precipitate was obtained. It was filtered, washed several times with water and dried at 70°C.

The preparation of complexes of 1-indenyl acetohydroxamic acid with above metal ions was done as above and the estimation of elements in complexes was done by usual procedures and other suitable spectrochemical measurements were tried. Particularly magnetic susceptibility measurements were made by Gouy's method using a number of standard substances like [HgCo[NCS]$_4$], $CuSO_4 \cdot 5H_2O$ and $FeSO_4 \cdot (NH_4)_2SO_4$ The $H_{max}$ of copper sulphate, ferrous ammonium sulphate and HgCoNCS at 20°C are 8.72, 8.78 and 8.75 respectively.

Mean value of field strength = $8.75 \times 10^3$ gauss. The magnetic susceptibilities of the complexes were calculated using the following expressions.

$$\mu_m \text{ susceptibility} = \frac{2 \times l \times m_2}{1.01a \times H_{max} \times w}$$

where $l$ = length, m = change in weight in mg, w = weight of the substance in g, $H_{max}$ = maximum field strength in gauss.

$\mu_{eff}$ was calculated as follows by the following formula:

$$\mu_{eff} = 2.839 \times m \text{ (corrected)} \times T$$

where T = absolute temperature.

## REFERENCES

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2. R.L. Carlin and A.J. Ven Buyneveldt, Magnetic Properties of Transition Metal Complexes, Springer-Verlag, New York (1977).