

2,2'-Bipyridyl and 1,10-Phenanthroline Complexes of Oxochromium (v)

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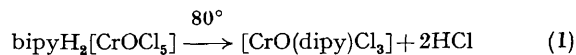
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Summary $[\text{CrO}(\text{bipy})\text{Cl}_3]$ and $[\text{CrO}(\text{phen})\text{Cl}_3]$ have been isolated from dehydrochlorination of the corresponding monodi-imine salts of hypothetical $\text{H}_2[\text{CrOCl}_5]$ in an inert atmosphere.

Few five-valent chromium complexes have been reported and all are moisture sensitive. Among these, the only six-co-ordinate complex is the alkali metal salt of the ion, $[\text{CrOCl}_5]^{2-}$.¹ Here we report the preparation and characterization of two stable complexes of oxochromium(v), $[\text{CrO}(\text{bipy})\text{Cl}_3]$ (I) and $[\text{CrO}(\text{phen})\text{Cl}_3]$ (II).

The complex $\text{bipyH}_2[\text{CrOCl}_5]$ was obtained as brown crystals, stable in dry air, from the reaction of CrO_3 with 2,2'-bipyridyl hydrochloride in concentrated HCl at 0°C. The compound was characterized by elemental analysis, bulk susceptibility measurement (1.69 B.M. at 24°), i.r. $[\nu(\text{Cr}=\text{O}) \text{ at } 934 \text{ cm}^{-1}]$, and reflectance spectrum (λ_{max} 13,800 and 23,000 cm^{-1}). The complex $[\text{CrO}(\text{bipy})\text{Cl}_3]$ was obtained (brown crystals, stable in air, insoluble in water and common solvents but slightly soluble in acetone, readily soluble in aqueous alkalis to give a brown solution which slowly disproportionates) from the dehydrochlorination of

the dipyriddy salt in dry CO_2 atmosphere at 80° according to reaction (1). The compound was characterized by



elemental analysis, inability to form any AgCl on trituration with AgNO_3 solution, nonconducting solution in acetone (18 $\text{ohm}^{-1} \text{ cm}^2$), bulk susceptibility measurement (1.85 B.M. at 24°), i.r. $[\nu(\text{Cr}=\text{O}) \text{ at } 954 \text{ cm}^{-1}]$, and reflectance spectrum (λ_{max} at 14,920, 18,870, and 24,100 cm^{-1}). Compound (II) was obtained by using phenanthroline instead of bipyridyl; it had similar properties to that of compound (I) and was characterized as for compound (I) ($\mu_{\text{eff}} = 1.87$ at 24°, $\nu_{\text{Cr}=\text{O}} = 957 \text{ cm}^{-1}$, λ_{max} 15,000, 18,570, and 24,350 cm^{-1}). Results are consistent with the studies for $[\text{CrOCl}_5]^{2-}$ (ref. 2) and similar to the corresponding molybdenum analogues.³ Further studies are in progress.

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¹ R. F. Weinland and M. Fiederer, *Ber.*, 1907, **40**, 2090, and reference therein.

² H. B. Gray and C. R. Hare, *Inorg. Chem.*, 1962, **1**, 363; E. Wending and R. Rohmer, *Bull. Soc. chim. France*, 1967, 8.

³ P. C. H. Mitchell, *J. Inorg. Nucl. Chem.*, 1963, **25**, 963; 1964, **26**, 1967.