Synthesis and *X*-Ray Molecular Structure of *trans*-Dichlorotetramethanolchromium(III) Chloride, $[Cr(MeOH)_4Cl_2]Cl$

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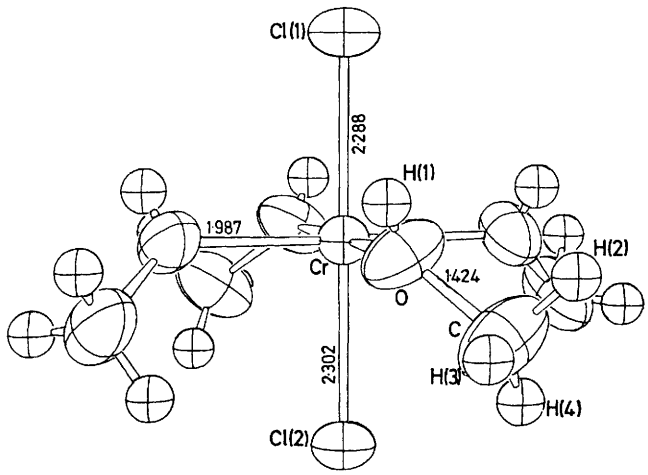
Summary Reaction of methanol with $CrCl_3,6H_2O$ dehydrated *in situ* with 2,2-dimethoxypropane gave *trans*-dichlorotetramethanolchromium(III) chloride; the cation, $[Cr(MeOH)_4Cl_2]^+$, has C_4 symmetry with slightly different Cr–Cl distances but otherwise normal atom-atom distances.

WE report the preparation and structure of an octahedral complex of Cr^{III} containing four methanol groups bonded directly to the central chromium. There have been previous attempts to prepare similar compounds but no definitive characterization has been reported,[1] probably because the alcohol groups are relatively weak Lewis base ligands and are labile to replacement by water.

2,2-Dimethoxypropane (DMP) (0·57 mol) and $CrCl_3,6H_2O$ (0·044 mol) were stirred for 4 h. Flash evaporation at 75 °C then left a brown gum which was dissolved in dry MeOH (100 ml) to give a green solution which was flash evaporated at 45 °C to approximately half-bulk, and kept at 5 °C for 24 h under dry nitrogen. The green crystals formed were filtered off, washed with Me_2CO–DMP (95 : 5) and then ether, and kept under dry nitrogen over conc. H_2SO_4 (50% yield). A 100% yield of a powder rather than the well formed crystals can be obtained if all the MeOH is removed by flash evaporation at 45 °C.

Crystal data: $[Cr(MeOH)_4Cl_2]Cl$: tetragonal, $a = 8·1418(6)$, $c = 9·3123(9)$, space group $P4/n$(No. 85), $Z = 2$, $D_c = 1·495$ g cm^{-3}. Intensities of 564 unique reflections were measured with a Hilger and Watts Y290 four-circle diffractometer and Mo-K_α radiation. The structure was solved by conventional heavy atom techniques. Subsequent refinement of all non-hydrogen atoms anisotropically and hydrogen atoms isotropically resulted in a final value of $R = 0·034$.

The bond distances and molecular geometry of the cation are given in the Figure. The 2 Cr–Cl distances are slightly but significantly different owing to steric repulsion from the methyl groups and also possibly from O—H(1) · · · Cl interactions. All distances are normal and compare favourably with those of the analogous aquo-complex.[2]



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