X-Ray Crystal Structure of the 1:1 Adduct of Trichloromethylmercury Chloride and 1,10-Phenanthroline

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Summary The crystal structure of the 1:1 adduct formed between trichloromethylmercury chloride and 1,10-phenanthroline has been determined from three dimensional *X*-ray diffraction data and reveals different environments for the mercury atoms in the two crystallographically independent molecules.

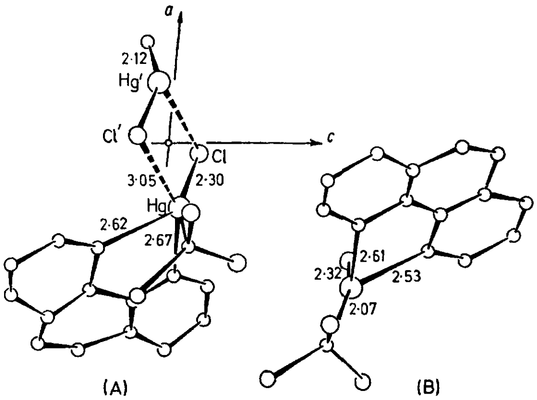
MERCURY(II) chloride forms adducts with a large number of neutral ligands and the crystal structures of many of these complexes have been determined.[1,2] Complexes of organomercury halides are less well documented owing to their tendency to disproportionate. Their preparation is made easier when the organic group has electronegative substituents, and a number of complexes of this type have been characterised.[3] This investigation was prompted by the scarcity of structural data on organomercury halide complexes.

Addition of trichloromethylmercury chloride to 1,10-phenanthroline in chloroform gave a 1:1 adduct. Cl_3CHg-$Cl{\cdot}C_{12}H_8N_2$ crystallises in the triclinic system, spacegroup

$P\bar{1}$, $a = 14{\cdot}05(2)$, $b = 8{\cdot}44(1)$, $c = 15{\cdot}53(2)$ Å, $\alpha = 119{\cdot}8(2)°$, $\beta = 85{\cdot}2(2)°$, $\gamma = 107{\cdot}7(2)°$, $D_m = 2{\cdot}34$ g. cm^{-3}, $Z = 4$, $D_c = 2{\cdot}34$ g. cm^{-3}, μ for Mo-K_α = 110 cm^{-1}. A total of 967 independent non-zero reflections was recorded by visual estimation from precession photographs taken with Mo-K_α radiation ($\lambda = 0{\cdot}7107$ Å). The structure was solved by standard heavy atom procedures and refined using full matrix least squares methods with isotropic thermal parameters for all atoms to a current conventional R factor of 11·9%. Calculated positional parameters for the hydrogen atoms were included in the last cycle of least squares refinement although these parameters were not refined. The data were not corrected for absorption or anomalous dispersion.

The two crystallographically independent molecules are shown in the figure together with some relevant bond lengths. The co-ordination sphere of the mercury atoms is not simple to interpret. It could be considered to be a distorted tetrahedron in both molecules. However in molecule A the distance between Cl′ and Hg is 3·05 Å which is much less than the sum of the van der Waals radii (3·30 Å). If we accept Grdenic's criteria[4] this atom can be included in the effective co-ordination sphere of atom Hg(A) which is then best described as a distorted trigonal bipyramid. Atom Cl′ is related to Cl by an inversion centre thus creating chlorine bridges between pairs of mercury atoms of type A. There are no other intermolecular approaches to either mercury atom of less than 3·30 Å.

The C–Hg–Cl angles are 155°(A) and 145°(B) respectively and this difference appears to be statistically significant (e.s.d. 2°). The latter angle is similar to that found in a variety of mercury(II) chloride adducts where the mercury has distorted tetrahedral co-ordination whilst the former may reflect the interaction of the mercury atom in (A) with the Cl of a neighbouring molecule.



The Hg–N bond lengths (2·61 Å average) are considerably longer than the sums of the van der Waals radii (2·00 Å) and indicate that the phenanthroline is only weakly co-ordinated to the mercury. These values are in good agreement with the Hg–N distances found in $HgCl_2{\cdot}(C_5H_5N)_2$ 2·60 Å[5] and $Fe(CO)_4[HgCl(C_5H_5N)]_2$ 2·51 Å.[6] The Hg–Cl bond distances are 2·30 Å (A) and 2·32 Å (B) respectively.

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