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COORDINATION COMPLEXES OF BIS(TRICHLOROVINYL)MERCURY

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Summary

The formation of complexes between bis(trichlorovinyl)mercury and a series of monodentate and bidentate nitrogen, phosphorus and sulphur bases has been studied. Adducts $(Cl_3C_2)_2HgL$ (L = phen dmp, tmp, bipy, diphos) ** have been isolated in the solid state. These are the first reported bis(alkenyl)mercurial complexes and their thermal behaviour and spectral characteristics (infrared, ^{1}H NMR, ^{35}Cl NQR and mass spectra) have been examined. No complexes of the monodentate ligands selected could be isolated, but ^{1}H NMR studies indicated the presence of weak ligand—mercury interaction in solution for triphenylphosphine, thiophene, pyridine as well as for the bidentate *N,N,N′,N′*,-tetramethylethane-1,2-diamine and 2,5-dithiahexane ligands. The acceptor properties of bis(trichlorovinyl)mercury appear to be intermediate between that of diphenylmercury and that of bis(pentafluorophenyl)mercury.

Introduction

The ability of two-coordinate mercury to form complexes with bases is very dependent upon the nature of the groups attached to mercury. Thus while mercury(II) chloride is known to form a wide range of adducts with both monodentate and bidentate ligands [1], no complexes have been isolated for mercury dialkyls. The relative electronegativities of mercury and the adjoining

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** phen, dmp, tmp, bipy, diphos represent abbreviations for 1,10-phenanthroline, 2,9-dimethyl-1,10-phenanthroline, 3,4,7,8-tetramethyl-1,10-phenanthroline, 2,2′-bipyridyl and bis(1,2-diphenylphosphino)ethane, respectively.

groups, together with the resulting influence upon the formal charge on mercury, appear to play an important role in complex formation. Thus the inability of mercury dialkyls to form stable complexes may be a reflection of the similar electronegativities of alkyl groups and mercury (ca. 2.3 and 1.9 respectively) [2], and hence the resulting low formal charge on mercury. Replacement of alkyl by more electronegative groups would be expected to enhance stable complex formation. Certainly the presence of a halogen in the organomercury halides, RHgX, does appear to increase the acceptor character of mercury and complexes, particularly of nitrogenous bases, have been isolated for a range of R and X [3—15]. Substitution in R_2Hg by electron-withdrawing substituents in R should also increase the formal charge on mercury, thereby enhancing the formation of stable addition compounds. Thus, bis(trinitromethyl)mercury [16] and bis(fluoroalkyl)mercurials [17,18] form a wide range of isolable complexes, in contrast to the mercury dialkyls themselves. Similarly, while diphenyl mercury only gives rise to weak complexes of the type Ph_2Hg2L (L = phen, dmp, 2,4,7,9-tetramethyl-1,10-phenanthroline) [19,20], bis(pentafluorophenyl)mercury addition complexes are more numerous and stable [21—24].

As yet, the coordination chemistry of the organomercurials R_2Hg, where R is an alkenyl or halogenated alkenyl group has not been studied. Dialkynyl mercurials $(RC{\equiv}C)_2Hg$ (R = CH_3, $ClCH_2$, $BrCH_2$, C_6H_5) are known [25,26] to form isolable complexes and the dialkenyl mercurials (particularly those containing halogenated alkenyl groups) might be expected to form stable complexes. Herein we report the results of our study into the coordination chemistry of bis(trichlorovinyl)mercury, $(Cl_2C{=}CCl)_2Hg$.

Results and discussion

Bis(trichlorovinyl)mercury forms isolable 1 : 1 complexes with bipy, phen, dmp, tmp and diphos, though no complexes could be isolated with pyridine, 2,4,6-trimethylpyridine, thiophene, *N,N,N',N'*-tetramethylethane-1,2-diamine (tmed), triphenylphosphine or 2,5-dithiahexane. These are the first reported examples of complexes formed by bis(alkenyl)mercurials and, apart from the unusual bis(pentachlorocyclopentadienyl)mercury complex $(C_5Cl_5)_2Hg5/3L$ (L = 1,2,dimethoxyethane) [14], these are also the first examples of chlorinated organomercurial complexes. In terms of the number of complexes formed, bis(trichlorovinyl)mercury would appear to be a stronger acceptor than diphenylmercury which does not form isolable complexes with bipy or diphos [20], but is a weaker acceptor than $(C_6F_5)_2Hg$ [21—24] and $(C(NO_2)_3)_2Hg$ [16]. Mass spectral examination of these complexes shows the relatively weak nature of the metal—ligand bonds. All five complexes dissociated even at low bombarding voltage (20 eV) and the spectra were similar to that of $(Cl_3C_2)_2Hg$ in that the ion of highest molecular mass corresponded to $(Cl_3C_2)Hg^+$.

The thermal behaviour of these complexes and that the parent organomercurial are outlined in Table 1. The thermal stability of the bidentate nitrogen complexes $(Cl_3C_2)_2HgL$, as reflected in the temperature at which onset of mass loss occurs, increases in the order L = bipy, phen, dmp, tmp. The greater

TABLE 1

THERMAL CHARACTERISTICS OF $(Cl_3C_2)_2Hg$ AND $(Cl_3C_2)_2Hg \cdot L$ (L = BIPY, PHEN, DMP, TMP, DIPHOS)

Compound	Temperature (°C)	Thermal Behaviour [a]
$(Cl_3C_2)_2Hg$	100	sublimation begins
	205	mass loss complete
$(Cl_3C_2)_2Hg \cdot L$		
L = bipy	75—190	mass loss in single step, leaving ~2% residue
L = phen	150	onset of mass loss
	150—225	41.1% mass loss attributed to loss of $HgCl_2$ (required 42.9%)
	225—400	45.6% mass loss
	400—800	residue (13.3%) stable
L = dmp	175	onset of mass loss
	175—420	complex thermogram
	[~230]	inflection, 42.1% mass loss, loss of $HgCl_2$ (required 40.6%)
	420—650	residue (28.1%) stable, residue attributed to C_4Cl_4 (required 28.4%)
L = tmp	200	onset of mass loss
	200—230	30.3% mass loss, loss of tmp (required 33.9%)
	230—320	43.0% mass loss, loss of $HgCl_2$ (required 38.9%)
	320—600	residue (26.7%), C_4Cl_4 (required 27.2%) stable
L = diphos	140	onset of mass loss
	550	mass loss complete, leaving 9.5% residue

[a] mass loss and residue are expressed as % of the mass of the original complex.

stability of the 1,10-phenanthroline complex compared to that formed by 2,2'-bipyridyl is in agreement with earlier work on organomercurials [3,17,20,25,27—29]. The relative stabilities has been ascribed [27] to the tendency of organomercurials to preserve approximate linearity upon coordination [8,24,30,31] thus necessitating the approaching ligands to interact with *p*-orbitals on mercury. 1,10-phenanthroline is a planar molecule and could give reasonable overlap with these mercury *p*-orbitals, whereas 2,2'-bipyridyl is twisted to minimise interaction of the 3,3'-hydrogen atoms, leading to poorer overlap and hence weaker mercury—ligand interactions. $(Cl_3C_2)_2Hg(bipy)$ in fact must be an extremely weak complex in the solid state for its melting point and thermal behaviour are very similar to that of the parent mercurial. However, the small yet significant differences between the infrared and NMR spectra of our product compared to the spectra of the free mercurial and bipy itself support the formulation as a complex rather than a loose lattice adduct.

The residue in both the substituted phenanthroline complexes (dmp, tmp) have been ascribed to C_4Cl_4, but this is doubtless polymeric since it does not show any mass spectral features. The decomposition appears to occur in the following manner:

$$n(Cl_3C_2)_2HgL \rightarrow nL + nHgCl_2 + (C_4Cl_4)_n$$

(L = dmp, tmp)

While $(Cl_3C_2)_2Hg(diphos)$ appears to have a comparable thermal stability to the 1,10-phenanthroline complex, onset of mass loss occurring at 140°C, no thermally stable intermediate could be detected in its thermogram.

Comparison of the infrared spectra of the complexes with that of $(Cl_3C_2)_2Hg$ permits frequencies attributable to $(Cl_3C_2)_2Hg$ to be distinguished from those of the neutral ligands. Absorption of the free mercurial at 828 cm^{-1} is shifted significantly to lower frequencies in the spectra of the complexes, as expected for a metal—ligand mode on an increase in coordination number. The ν(CC) mode of 2,2'-bipyridyl at 1582 and 1560 cm^{-1} and the ring breathing mode at 992 cm^{-1} are shifted to higher frequencies (1592, 1570, 1005 cm^{-1} respectively) as expected for the coordinated ligand. Similarly most of the bands associated with phen, dmp, and tmp in the 1350—1620 cm^{-1} region are displaced to higher frequencies on coordination. The most significant features in the spectrum of $(Cl_3C_2)_2Hg$(diphos) are the presence of strong bands at 822 and 1550 cm^{-1}, both absent in the spectrum of the free ligand.

The ^{35}Cl NQR frequencies at 77 K observed for $(C_2Cl_3)_2Hg$ (dmp) and $(C_2Cl_3)_2Hg$(tmp) are reported in Table 2, together with comparable data for $(C_2Cl_3)_2Hg$. Generally an increase in charge on chlorine corresponds to a reduction in NQR frequency, assuming other effects remain constant [32]. The most prominent feature of these spectra is the reduction in frequency of the two lowest frequency resonances on complex formation. These two resonances may be reasonably ascribed to the α-chlorine atoms by comparison with the NQR spectrum of $(CCl_2{=}CCl)HgCl$ [35] in which the α-chlorine resonance (35.42 MHz) is 1.93 MHz lower than the average of the β-chlorine resonances (37.56, 37.13 MHz). The reduction in frequency upon complex formation of the frequencies assigned to the α-chlorine atoms is greater for the dmp complex (0.70 MHz) than for the tmp complex (0.21 MHz) suggesting a greater degree of charge transfer in the dmp complex and this is further supported by our thermal studies. However, there is little difference in the shift of the frequency averaged over all the chlorine atoms, 0.37 MHz for the tmp complex and 0.45 MHz for the dmp complex. This amounts to about a 1% change in average

TABLE 2

^{35}Cl QUADRUPOLE RESONANCE FREQUENCIES (MHz)

	at 77 K [a]	at 293 K [a]
$(Cl_3C_2)_2Hg$ [b]	35.42	
	35.59	
	36.15	
	36.89	
	37.30 (double intensity)	
$(Cl_3C_2)_2Hg$(dmp)	34.74	34.03
	34.87	34.215
	36.28	35.40
	36.68	35.645
	37.03	35.880
	37.14	35.965
$(Cl_3C_2)_2Hg$(tmp)	35.21	
	35.38	35.39 [c]
	36.25 (double intensity)	35.71
	36.97	36.42
	37.15	

[a] ±0.005 MHz. [b] Data from ref. 33. [c] incomplete spectrum.

chlorine charge. An alternative explanation of the shift to low frequencies of the two complexes is the possibility of significant secondary bonding, particularly for the dmp complex, involving the α-chlorine atoms. Recently, the X-ray structure [31] of the tmp complex has shown that intermolecular Hg ... Cl interactions are of little significance, though it is difficult to evaluate the importance of intramolecular Hg ... Cl interactions.

The multiplicity of the NQR spectrum for the dmp complex indicates the presence of six crystallographically inequivalent chlorine atoms whereas for the tmp complex, only five lines were observed, one of which is of double intensity to the remainder, doubtless corresponding to two similarly bonded chlorine atoms. Six lines were also observed at room temperature for the dmp complex but we were unable to find all the resonances in the room temperature spectrum of the tmp complex (Table 2). The temperature coefficients for the two lowest frequency resonances in the dmp complex, though somewhat lower than the temperature coefficients for the other four lines observed, were not sufficiently different to suggest with any degree of certainty the presence of secondary bonding involving the α-chlorine atoms.

In the reaction between bis(trichlorovinyl)mercury and the monodentate ligands selected, no 1 : 1 complexes have been isolated in the solid state. However, ^{1}H NMR studies (Table 3) indicate that weak mercury—ligand interactions do occur in solution. Small downfield shifts of the resonance bands due to the ligands, of the order of 0.05 ppm, are observed. This is in keeping with the withdrawal of electron density from the ligands and indicates the presence of weak interactions, as has been suggested for phosphine-bis(fluoroalkyl)mercury systems from ^{31}P NMR studies [34]. The downfield shifts observed for the isolable complexes $(Cl_3C_2)_2Hg$ L (L = bipy, phen, dmp, tmp, diphos) are significantly larger (Table 4), approximately 0.1 ppm, but still considerably less than that observed in mercury(II) chloride addition complexes (0.25 for example in (tmed)$HgCl_2$). The bidentate ligands 2,5-dithiahexane and tmed, while not forming isolable complexes, appear to form weak interactions in solution with the organomercurial (Table 3).

The trichlorovinyl groups would appear to be sufficiently more electronegative than mercury to enable complex formation to occur and while isolable complexes have only been obtained for bidentate ligands, NMR studies indicate weak mercury—ligand interactions occur for selected monodentate ligands. The

TABLE 3

^{1}H NMR SPECTRAL DATA FOR LIGANDS AND 1 : 1 MIXTURES WITH BIS(TRICHLOROVINYL)-MERCURY (δ (TMS) = 0 ppm, CCl_4 solution)

	L	$(Cl_3C_2)_2Hg$ + L
Pyridine	7.23, 7.29, 7.34, 7.43 7.61, 7.74, 8.71, 8.76	7.30, 7.35, 7.39, 7.49 7.68, 7.81, 8.76, 8.81
Thiophene	7.12, 7.18, 7.34, 7.37	7.17, 7.26, 7.40, 7.44
2,5-Dithiahexane	2.15, 2.70	2.16, 2.72
Tmed	2.21, 2.34	2.27, 2.40
Triphenylphosphine	7.37, 7.42	7.42, 7.47

TABLE 4

^{1}H NMR SPECTRAL DATA FOR LIGANDS AND THEIR COMPLEXES WITH BIS(TRICHLOROVINYL)MERCURY (δ(TMS) = 0 ppm) (CCl_4 solution)

	L	$(Cl_3C_2)_2Hg \cdot L$
phen	7.64, 7.72, 7.81, 7.89, 8.28, 8.40	7.75, 7.82, 7.90, 7.97, 8.38, 8.50
bipy	7.22, 7.35, 7.42, 7.75, 7.88, 8.01, 8.62, 8.76, 8.85	7.33, 7.45, 7.53, 7.81, 7.95, 8.07, 8.48, 8.82, 8.90
dmp	2.96, 7.48, 7.62, 7.76, 8.13, 8.27	3.05, 7.61, 7.75, 7.86, 8.29, 8.43
tmp	2.58, 2.70, 8.08	2.67, 2.76, 8.17
diphos	2.00, 2.07, 2.14, 7.41	2.11, 2.18, 2.25, 7.48

coordination behaviour of bis(trichlorovinyl)mercury appears to be intermediate in character between that of diphenylmercury and that of bis(pentafluorophenyl)mercury.

Experimental

Microanalyses were performed bv the Oxford microanalytical laboratories of Dr. F.B. Strauss. Mass spectra were recorded using an AEI MS30 mass spectrometer at a bombarding voltage of 20 eV and a source temperature of 180°C. ^{1}H NMR spectra were recorded using a Jeol C-60 HL high resolution spectrometer. Thermal measurements were recorded using a DuPont Model 900 thermal analyser. Samples were heated at a rate of 5°C min^{-1} under a dynamic flow of nitrogen of 50 cm^3 min^{-1}. Infrared spectra (4000—650 cm^{-1}) were recorded as KBr discs using a Unicam SP200 spectrometer. Chlorine NQR spectra were observed with a Decca NQR spectrometer using Zeeman modulation and automatic frequency calibration.

^{1}H NMR studies. The spectra of the five complexes isolated were recorded as solutions in carbon tetrachloride. The spectra of equimolar mixtures of the organomercurial and ligand (pyridine, tmed, thiophene, 2,5-dithiahexane or triphenylphosphine) were also recorded as solutions in carbon tetrachloride.

Reagents

Bis(trichlorovinyl)mercury was prepared as previously described and recrystallised from hexane: m.p. 72—73°C lit. 72—73°C [35].

The ligands were commercially available and were used without further purification.

Preparation of complexes

With 2,2'-bipyridyl. Bis(trichlorovinyl)mercury (0.3 g, 0.65 mmol) in hexane was added to 2,2'-bipyridyl (0.11 g, 0.65 mmol) in boiling hexane. On cooling in ice, a white crystalline deposit was formed which was filtered off and recrystallised from hexane as colourless needles, m.p. 75—76°C. [Found: C, 27.46; H, 1.12; N, 4.56. $C_{14}H_8Cl_6HgN_2$ calcd.: C, 27.21; H, 1.30; N, 4.53%.] Infrared absorptions: 1592m, 1570w, 1541m, 1493w, 1478m, 1440s, 1314m, 1242w, 1168w, 1150m, 1098w, 1059w, 1040w, 1005s, 927vs, 838vs, 824s, 758vs, 735m and 682s cm^{-1}.

With 1,2-bis(diphenylphosphino)ethane. Bis(trichlorovinyl)mercury (0.30 g, 0.65 mmol) in chloroform was added to the diphosphine (0.26 g, 0.65 mmol) also dissolved in chloroform solution. The mixture was evaporated to dryness and the oily residue recrystallised from hexane as colourless cubes, m.p. 125°C decomp. [Found: C, 41.75; H, 3.30. $C_{30}H_{24}Cl_6HgP_2$ calcd.: C, 41.88; H, 2.82%.] Infrared absorptions: 1550s, 1488m, 1420s, 1182s, 1165m, 1122m, 1105w, 1098m, 1082m, 1068w, 1030w, 1002w, 938m, 890w, 822s, 792w, 768w, 754m, 743m, 728vs, 704m(sh), 692vs and 672m(sh) cm^{-1}.

With 1,10-phenanthroline. Bis(trichlorovinyl)mercury (0.30 g, 0.65 mmol) in boiling hexane was added to 1,10-phenanthroline monohydrate (0.12 g, 0.65 mmol) dissolved in a 1 : 1 boiling mixture of benzene and hexane. The solvent was then evaporated to dryness and the white residue recrystallised from hexane as fine white needles, m.p. 161—162°C. [Found: C, 29.76; H, 1.26; N, 4.48. $C_{16}H_8Cl_6HgN_2$ calcd.: C, 29.93; H, 1.25; N, 4.36%.] Infrared absorptions: 1622w, 1595w, 1570w, 1545m, 1518m, 1432s, 1350w, 1216w, 1139m, 1099m, 934m(sh), 928m, 864m, 843vs, 837s(sh), 761m, 728s, 710m and 690m cm^{-1}.

With 2,9-dimethyl-1,10-phenanthroline. Bis(trichlorovinyl)mercury, (0.69 g, 1.5 mmol) and 2,9-dimethyl-1,10-phenanthroline hemihydrate (0.33 g, 1.5 mmol) were mixed in hexane solution. The solution was evaporated to crystallisation and the product filtered off and recrystallised from hexane as colourless needles, m.p. 183—184°C. [Found: C, 32.77; H, 1.91; N, 4.26. $C_{18}H_{12}Cl_6HgN_2$ calcd.: C, 32.28; H, 1.81; N, 4.18%.] Infrared absorptions: 1622w, 1598m, 1560w, 1541s, 1512s, 1442m, 1430m(sh), 1414w, 1388m, 1370m, 1311w, 1288w, 1240w, 1221w, 1202w, 1144m, 1110w, 1100m, 1026w, 978w, 924s, 860vs, 832vs, 764m, 730s and 678m cm^{-1}.

With 3,4,7,8-tetramethyl-1,10-phenanthroline. Bis(trichlorovinyl)mercury (0.69 g, 1.5 mmol) dissolved in hexane was added to the ligand (0.35 g, 1.5 mmol) in hexane. The solution was evaporated to crystallisation and the white product filtered off and recrystallised from ethanol/hexane as colourless needles, m.p. 203—205°C decomp. [Found: C, 34.53; H, 2.30; N, 4.13. $C_{20}H_{16}Cl_6HgN_2$ calcd.: C, 34.43; H, 2.31; N, 4.01%.] Infrared absorptions: 1620w, 1582w, 1540s, 1526s, 1438m, 1390m, 1348w, 1272w, 1240m, 1198w, 1018w, 1000w, 952w, 930s, 880m, 862m, 838s, 818m, 726s, 717m and 680m cm^{-1}.

Attempted preparation of other complexes. Mixtures of the mercurial and ligand (pyridine, 2,4,6-trimethylpyridine, thiophene, *N,N,N′,N′*-tetramethylethane-1,2-diamine, triphenylphosphine or 2,5-dithiahexane) in 1 : 1 and 1 : 2 mole ratio were dissolved in hexane and the solution evaporated to crystallisation. In every case, either fractional crystallisation of ligand or the organomercurial occurred and these were identified by their melting points and infrared spectra.

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