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# THERMODYNAMICS OF METAL—LIGAND BOND FORMATION

## XXVII *. REACTION OF LEWIS BASES WITH ALKYLMERCURY(II) HALIDES

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### Summary

Alkylmercury(II) halides, RHgX, form 1 : 1 adducts in benzene solution with heterocyclic bases; the adduct stability is very low ($K < 1$), though more stable adducts ($10 < K < 100$ approximately) are formed with the bidentate bases, 1,10-phenanthroline and *N,N,N′,N′*-tetramethyl-1,2-diaminoethane. In all cases enthalpies of adduct formation are about $-25$ kJ mol$^{-1}$ . $PBu_3$ forms dimeric adducts, $(RHgX \cdot PBu_3)_2$, of higher stability with enthalpies of formation about $-60$ kJ(g-at.Hg)$^{-1}$ . The perchloro compounds $Cl_3CHgCl$ and $(Cl_3C)_2Hg$ form more stable adducts with *N*-bases and also with tetrahydrothiophene and pyridine-*N*-oxide, but $Cl_3CHgCl$ is symmetrised by phosphines and $(Cl_3C)_2Hg$ reacts slowly with them, depositing mercury. The Lewis acidity of mercury compounds increases in the order: $R_2Hg << RHgCl < (Cl_3C)_2Hg < Cl_3CHgCl < HgCl_2$.

### Introduction

In the past few years there has been increasing interest in the coordination chemistry of organomercury compounds and this field has been recently reviewed [1]. We have previously reported thermodynamic data for the formation of 1/1 adducts of nitrogenous bases with arylmercury(II) halides, RHgX, in benzene solution, though under similar conditions these compounds are symmetrised to $R_2Hg$ and $HgX_2$ by phosphines [2]. In most respects the alkylmercury (II) halides resemble the aryl compounds, but the symmetrisation reaction is much slower: although tertiary phosphines symmetrise MeHgBr in ether solution, it is reported that in benzene addition occurs first, followed by slow symmetrisation [3].

Adducts of phosphines with alkylmercury(II) halides, for example MeHgBr ·

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* For part XXVI see ref. 17.

$PEt_3$, have been isolated and this compound has been shown to be completely dissociated in water; conductimetric titration of EtHgBr with tertiary phosphines in methanol shows that the adducts should be formulated as salts, for example $[EtHg \cdot PR_3]^+ Br^-$, at least in ionising solvents [4]. It has also been shown by NMR studies that the 1/1 adduct is the only product formed when MeHgCl reacts with $PMe_3$ [5]. Adducts have also been reported between a variety of bases with N, P, As, O or S as donor atoms and perfluoroalkylmercury(II) compounds [6,7]; these include both 1/1 and 1/2 adducts of the type $(R_F)_2Hg \cdot B_n$ ($n$ = 1 or 2) and are formed in benzene solution so are presumably not ionised.

The perchloro compounds, $(Cl_3C)_2Hg$ and $Cl_3CHgBr$, have also been reported to form 1/1 adducts with bipy and pyridine respectively [8,9] and $^{35}Cl$ NQR spectra suggest that they form stable solvates when dissolved in ethers [10].

We now report calorimetric studies of the reaction of a variety of alkylmercury(II) halides with pyridine (py), 4-methylpyridine (4-mepy), 1,10-phenanthroline (phen), *N,N,N',N'*-tetramethyl-1,2-diaminoethane (tmed) and tributylphosphine and of $Cl_3CHgCl$ and $(Cl_3C)_2Hg$ with a wider range of bases in benzene solution.

## Adducts of RHgX with nitrogenous bases

The formation of adducts in solution was revealed by liberation of heat when 4-methylpyridine was titrated into solutions of alkylmercury(II) halides in benzene. The most extensive reaction was observed between $PhCH_2HgCl$ and 4-methylpyridine and for this system reproducible enthalpies of adduct formation of about $-24$ kJ mol$^{-1}$ were obtained with an adduct formation constant $K = 0.9$ l mol$^{-1}$. This represents an adduct of such low stability that adduct formation is less than 50% complete even in *M* base solution and the values of $\Delta H^0$ and $K$ must be regarded as very approximate; however, since the total heat liberated is proportional to the total base concentration during the initial part of the titration, the 1/1 stoichiometry is unambiguous. Assumption of a similar enthalpy of adduct formation for the reaction of MeHgBr with 4-mepy leads to a formation constant of about 0.5 l mol$^{-1}$. Since adducts of such low stability fall below the threshold of accurate measurement by the calorimetric technique, no further data for adduct formation by unidentate bases are reported. Liberation of heat, representing formation of 1/1 adducts of very low stability was also observed with pyridine and piperidine, but not with tributylamine, nor with the *S*-bases diethyl sulphide or tetrahydrothiophene.

Although attempts at titration with 2,2'-bipyridine gave almost no evidence of adduct formation, relatively stable adducts of 1/1 stoichiometry were formed with 1,10-phenanthroline. Details are given in Table 1.

The enthalpies of formation of the phen adducts, about $-20$ kJ mol$^{-1}$, are similar to that of the 4-mepy adduct, but they are much more stable with $10 < K < 30$ l mol$^{-1}$; as before, the adduct with the benzyl compound is significantly more stable, $K$ 105 l mol$^{-1}$. The greater stability of phen adducts compared with those of 4-mepy, though without significantly increased enthalpy of formation, was also observed with the rather more reactive arylmercury(II) halides [1]. It was then attributed to the formation of chelate rings in which,

TABLE 1

THERMODYNAMIC DATA FOR FORMATION OF 1/1 ADDUCTS OF BASES WITH RHgX IN BENZENE AT 30°C. [RHgX] in mmol $l^{-1}$, $K$ in l $mol^{-1}$, $\Delta H^0$, $\Delta G^0$ in kJ $mol^{-1}$, $\Delta S^0$ in J $K^{-1}$ $mol^{-1}$.

| R | X | Base | [RHgX] | $K$ | $-\Delta H^0$ | $-\Delta G^0$ | $-\Delta S^0$ |
|---|---|---|---|---|---|---|---|
| Me | Br | 4-mepy | 10 | 0.5 | 24.0 [b] | | |
| Benzyl | Cl | 4-mepy | 8—15 | 0.9 ± 0.1 | 24.0 ± 1.3 | —0.3 ± 0.3 | 80 ± 5 |
| Me | Br | phen [a] | 6—10 | 15.4 ± 1.2 | 26.8 ± 1.7 | 6.9 ± 0.3 | 66 ± 7 |
| Et | Br | phen [a] | 5— 7 | 25.1 ± 2.0 | 17.3 ± 0.6 | 8.1 ± 0.3 | 30 ± 3 |
| Pr | Br | phen [a] | 5— 8 | 12.6 ± 0.9 | 27.5 ± 1.0 | 6.4 ± 0.2 | 70 ± 4 |
| Bu | Br | phen [a] | 6—10 | 18.0 ± 1.0 | 22.2 ± 0.8 | 7.3 ± 0.2 | 49 ± 3 |
| Cyclohexyl | Br | phen [a] | 6— 8 | 27.6 ± 2.2 | 21.0 ± 1.0 | 8.4 ± 0.3 | 42 ± 4 |
| Benzyl | Cl | phen [a] | 2— 4 | 103.1 ± 7.9 | 27.1 ± 2.2 | 11.7 ± 0.2 | 51 ± 8 |
| Me | Br | tmed | 2— 4 | 28.2 ± 3.5 | 34.2 ± 1.3 | 8.4 ± 0.4 | 85 ± 6 |
| Et | Br | tmed | 2— 5 | 10.8 ± 1.0 | 31.5 ± 1.6 | 6.0 ± 0.3 | 84 ± 6 |
| Pr | Br | tmed | 3— 5 | 11.4 ± 0.6 | 28.2 ± 1.4 | 6.1 ± 0.2 | 73 ± 5 |
| Bu | Br | tmed | 2— 5 | 11.6 ± 2.7 | 34.0 ± 2.7 | 6.2 ± 0.7 | 92 ± 11 |
| Cyclohexyl | Br | tmed [a] | 5— 8 | 2.4 ± 0.3 | 38.3 ± 0.5 | 2.2 ± 0.4 | 115 ± 3 |
| Benzyl | Cl | tmed | 2— 3 | 39.1 ± 3.4 | 40.9 ± 1.1 | 9.2 ± 0.3 | 105 ± 5 |

[a] Range of base concentration extended by ampoule experiments.
[b] Arbitrary value.

however, the two Hg—N bonds were weakened due to misfitting of the stereochemistries of phen and the metal atom. A similar effect is observed when bipy adds to $Hg(C_6F_5)_2$, but in that case phen forms an adduct of dramatically higher stability with a much larger enthalpy of formation [11].

The potentially bidentate base tmed also gives a large enthalpy of adduct formation with $Hg(C_6F_5)_2$, indicative of coordination of both N atoms [11]. In the present case enthalpies of adduct formation with alkylmercury(II) halides fall in the range —30 to —40 kJ $mol^{-1}$; this is a little greater than for phen and the adduct stabilities are a little lower. Nevertheless the failure of $NBu_3$ to give any indication of adduct formation shows clearly that tmed is behaving as a chelate. The very low stability of the adduct of tmed with cyclohexylmercury(II) bromide reveals a steric interference from the α-branched alkyl group which would also be unlikely if tmed were only unidentate. Thermodynamic data for the formation of tmed adducts are included in Table 1.

## Adducts of RHgX with phosphines

Titration of tributylphosphine into solutions of RHgX in benzene liberated heat, but the data could not be fitted to computed titration curves for the formation of monomeric 1/1 adducts; formation constants of excellent consistency were obtained assuming that the product was a dimeric 1/1 adduct. $PBu_3$ thus reacts according to the equation:

$$2\,RHgX + 2\,PBu_3 \rightleftharpoons (RHgX \cdot PBu_3)_2$$

$$\text{for which } K = \frac{[(RHgX \cdot PBu_3)_2]}{[RHgX]^2 \times [PBu_3]^2}\ l^3\ mol^{-3}$$

Thermodynamic data for these reactions are given in Table 2, in which $\Delta H^0$,

TABLE 2

THERMODYNAMIC DATA FOR FORMATION OF $(RHgX \cdot PBu_3)_2$ IN BENZENE AT 30°C. [RHgX] in mmol $l^{-1}$, $K'$ in $l^{1.5}$ $mol^{-1.5}$, $\Delta H^0$, $\Delta G^0$ in kJ $(g\text{-at.Hg})^{-1}$, $\Delta S^0$ in J $K^{-1}$ $(g\text{-at.Hg})^{-1}$.

| R | X | [RHgX] | $10^{-3}$ $K'$ | $-\Delta H^0$ | $-\Delta G^0$ | $-\Delta S^0$ |
|---|---|---|---|---|---|---|
| Me | Br | 1—3 | 7.1 ± 0.3 | 65.0 ± 0.7 | 22.3 ± 0.2 | 141 ± 3 |
| Et | Br | 2—4 | 3.2 ± 0.4 | 52.3 ± 0.9 | 20.3 ± 0.4 | 106 ± 4 |
| Pr | Br | 2—4 | 3.3 ± 0.5 | 57.5 ± 1.0 | 20.4 ± 0.5 | 122 ± 5 |
| Bu | Br | 2—2.5 | 3.6 ± 1.2 | 54.7 ± 1.6 | 20.6 ± 0.1 | 112 ± 6 |
| Cyclohexyl | Br | 2—4 | 2.6 ± 0.4 | 48.0 ± 2.0 | 19.8 ± 0.4 | 93 ± 8 |
| Benzyl | Cl | 1—3 | 37.5 ± 1.6 | 64.0 ± 1.3 | 25.5 ± 0.2 | 127 ± 5 |
| *o*-Chlorobenzyl | Cl | 1—2 | 54.0 ± 9.0 | 67.8 ± 2.0 | 27.4 ± 0.4 | 133 ± 8 |

$\Delta G^0$, $\Delta S^0$ are given in kJ $(g\text{-at. Hg})^{-1}$ and $K' = K^{1/2}$.

Although the data are not consistent with the stoichiometric requirements for the alternative symmetrisation reaction:

$$2\,RHgX + 2\,PBu_3 \rightleftharpoons R_2Hg + HgX_2(PBu_3)_2$$

it was confirmed that this was not the reaction taking place by repeating several titrations in the presence of added $R_2Hg$ and showing that this did not affect either $\Delta H^0$ or $K$. Attempts were also made to detect shifts in the Hg—X stretching frequencies in the region 200—600 $cm^{-1}$ and to observe P—H coupling in the NMR spectra, but satisfactory results could not be obtained at the low concentrations involved.

The data in Table 2 show clearly the slightly greater Lewis acidity of MeHgBr compared to the higher homologues; this is an inductive effect in which the higher enthalpy of formation leads to greater adduct stability. There is also a small inductive effect in the opposite sense due to α-branching of the alkyl chain in the cyclohexyl compound. The greater stability of adducts with $C_6H_5CH_2HgCl$ is probably due in part to the replacement of the Br atom by Cl, but adduct stability is still further increased by the inductive effect of *o*-Cl substitution in the benzyl compound.

There was no evidence of reaction when attempts were made to titrate RHgX with $PPh_3$ or diphos. In view of the great stability of the chelate adducts of diphos with mercury(II) halides [12], the failure of RHgX to react with this base is surprising. It is known however that $PPh_3$ [13] and diphos form monomeric adducts with $HgX_2$ in dilute solution and it is possible that the $PBu_3$ adducts of RHgX are stabilised by the halogen bridging.

## Adducts of $Cl_3CHgCl$ and $(Cl_3C)_2Hg$

The very weak acidity of RHgX and the failure of $R_2Hg$ to show Lewis acid properties, together with the known acidity of corresponding fluorinated compounds [6,7] led us to examine the reactions of the more readily accessible perchloro compounds, since in these, too, the inductive effect of the $CCl_3$ group could be expected to lead to increased acidity of the mercury atom. Indeed, adducts of these compounds have been isolated [8,9].

When $Cl_3CHgCl$ was titrated with a tertiary phosphine in benzene solution

TABLE 3

THERMODYNAMIC DATA FOR FORMATION OF 1/1 ADDUCTS OF BASES WITH $(Cl_3C)_2Hg$ IN BENZENE AT 30°C. $[(Cl_3C)_2Hg]$ in mmol $l^{-1}$, $K$ in l $mol^{-1}$, $\Delta H^0$, $\Delta G^0$ in kJ $mol^{-1}$, $\Delta S^0$ in J $K^{-1}$ $mol^{-1}$.

| Base | $[(Cl_3C)_2Hg]$ | $K$ | $-\Delta H^0$ | $-\Delta G^0$ | $-\Delta S^0$ |
|---|---|---|---|---|---|
| py | 2.5—3.5 | 10.9 ± 2.1 | 36.9 ± 0.8 | 6.0 ± 0.5 | 102 ± 5 |
| 4-mepy | 2 —4 | 14.5 ± 2.5 | 38.4 ± 2.8 | 6.8 ± 0.5 | 104 ± 11 |
| 2-mepy | 1.8—2.5 | 20.2 ± 2.1 | 29.4 ± 0.8 | 7.6 ± 0.3 | 72 ± 4 |
| bipy | 1.3—1.8 | 449 ± 26 | 31.4 ± 0.2 | 15.4 ± 0.2 | 53 ± 2 |
| phen | 2 —3 | $>10^4$ | 42.3 ± 0.1 | >23 | <64 |
| $BuNH_2$ | 0.8—1.6 | 199 ± 21 | 41.0 ± 0.2 | 13.3 ± 0.3 | 91 ± 2 |
| $Et_2NH$ | 1 —1.5 | 71.8 ± 5.5 | 34.3 ± 0.2 | 10.8 ± 0.2 | 77 ± 2 |
| pip [a] | 1 —1.6 | 62.9 ± 6.7 | 42.3 ± 1.0 | 10.4 ± 0.3 | 105 ± 5 |
| tmed | 1.3—2.0 | 73.3 ± 7.0 | 49.6 ± 0.9 | 10.8 ± 0.3 | 128 ± 4 |
| py-NO | 2.5—3.5 | 56.3 ± 6.9 | 24.3 ± 1.1 | 10.1 ± 0.4 | 47 ± 5 |
| $C_4H_8S$ | 1.5—2.5 | 7.5 ± 0.9 | 25.3 ± 2.3 | 5.1 ± 0.4 | 67 ± 9 |

[a] pip = piperidine.

heat was liberated but equilibrium was achieved very slowly, suggesting that symmetrisation may occur; titration of $(Cl_3C)_2Hg$ was also unsatisfactory and led to precipitation of metallic mercury. However, satisfactory results were obtained with a variety of other *N*-bases and with pyridine-*N*-oxide and tetrahydrothiophene. Production of heat was also observed with tetrahydrofuran and tributylamine, but the adduct formation constants were too small for evaluation of $K$ or $\Delta H^0$ to be possible. The data for formation of 1/1 adducts with $(Cl_3C)_2Hg$ are summarised in Table 3.

Since $(Cl_3C)_2Hg$ forms reasonably stable adducts with these bases the form of the equilibrium constant observed when $Cl_3CHgCl$ reacts is no longer reliably exclusive of the alternative symmetrisation reaction:

$$2\,Cl_3CHgCl + 2\,B \rightleftharpoons (Cl_3C)_2Hg \cdot B + HgCl_2B$$

That adduct formation rather than symmetrisation occurs is shown by the isolation, from solutions of rather higher concentration than those used in titrations, of the following 1/1 adducts:

| | |
|---|---|
| $Cl_3CHgCl \cdot$ pip * | (Found: C, 15.3; H, 2.5; N, 3.3. Calcd.: C, 16.4; H, 2.5; N, 3.2%.) |
| $Cl_3CHgCl \cdot$ phen | (Found: C, 29.4; H, 1.5; N, 5.2; Calcd.: C, 29.2; H, 1.5; N, 5.2%.) |
| $Cl_3CHgCl \cdot$ py-NO | (Found: C, 16.2; H, 1.1; N, 3.1. Calcd.: C, 16.0; H, 1.1; N, 3.1%.) |

When reaction occurred with n-butylamine, however, the product isolated was $HgCl_2BuNH_2$, showing that symmetrisation had taken place, though possibly only at the stage of crystallisation. With tmed as titrant a reaction stoichiometry of $Cl_3CHgCl$/1.6 tmed was observed with an equilibrium constant too large to measure, but attempts to isolate a crystalline adduct were not successful. In

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* The slightly low percentage of carbon found for this compound is probably due to contamination by a small amount of $Cl_3CHgCl$.

TABLE 4

THERMODYNAMIC DATA FOR FORMATION OF 1/1 ADDUCTS OF BASES WITH $Cl_3CHgCl$ IN BENZENE AT 30° C. [$Cl_3CHgCl$] in mmol $l^{-1}$, $K$ in l $mol^{-1}$, $\Delta H^0$, $\Delta G^0$ in kJ $mol^{-1}$, $\Delta S^0$ in J $K^{-1}$ $mol^{-1}$.

| Base | [$Cl_3CHgCl$] | $K$ | $-\Delta H^0$ | $-\Delta G^0$ | $-\Delta S^0$ |
|---|---|---|---|---|---|
| py | 1.5—3.0 | 16.4 ± 1.8 | 55.0 ± 0.8 | 7.1 ± 0.3 | 158 ± 4 |
| 4-mepy | 2.0—3.0 | 22.5 ± 3.3 | 49.9 ± 1.5 | 7.8 ± 0.5 | 139 ± 7 |
| 2-mepy | 1.8—2.5 | 8.9 ± 1.1 | 45.2 ± 1.5 | 5.5 ± 0.4 | 131 ± 6 |
| bipy | 1.0—1.8 | 326 ± 50 | 31.6 ± 0.8 | 14.6 ± 0.5 | 56 ± 5 |
| phen | 2.0—3.0 | $>10^4$ | 47.7 ± 1.1 | >23 | <82 |
| $Et_2NH$ | 1.3—1.8 | 300 [a] | 41.0 [a] | 14.4 | 88 |
| pip | 0.8—1.5 | 576 ± 49 | 52.9 ± 1.2 | 16.0 ± 0.3 | 122 ± 5 |
| py-NO [b] | 3.0—4.0 | 51.6 ± 3.7 | 26.5 ± 0.7 | 9.9 ± 0.2 | 55 ± 3 |
| $C_4H_8S$ | 1.3—2.0 | 10.7 ± 0.5 | 33.3 ± 0.8 | 6.0 ± 0.2 | 90 ± 3 |

[a] Best fit computed curve to early part of titration data (80% formation of 1/1 adduct). A second reaction appears to occur with excess base. [b] Data also determined for adduct formation between py-NO and $HgCl_2$: $K = 67.1 \pm 6.7$, $\Delta H^0 = -55.0 \pm 1.0$, $\Delta G^0 = -10.6 \pm 0.3$, $\Delta S^0 = -146 \pm 5$.

all other cases calorimetric titrations were consistent with the formation of 1/1 adducts, according to the equation:

$$Cl_3CHgCl + B \rightleftharpoons Cl_3CHgCl \cdot B$$

Data for these reactions are summarised in Table 4.

Comparison of the data in Tables 3 and 4 with data for adducts with $HgX_2$ [12,13] shows steadily increasing Lewis acidity in the order: $R_2Hg << RHgCl < (Cl_3C)_2Hg < Cl_3CHgCl < HgCl_2$, both in terms of enthalpy of reaction and adduct stability. The $CCl_3$ group appears to be almost as effective as Cl in conferring Lewis acidity on the Hg atom; this is shown particularly well by the similarities of both enthalpy and free energy when $Cl_3CHgCl$ or $(Cl_3C)_2Hg$ react with the same base. The stabilities of the adducts of $Cl_3CHgCl$ and $(Cl_3C)_2Hg$ with the *O*-donor, pyridine-*N*-oxide, are unexpectedly high, suggesting that replacement in $HgCl_2$ of Cl, an anion capable of double-bond formation with the mercury atom, by $Cl_3C$, which is not, leads to a slight "hardening" of the mercury atom as a Lewis acid centre.

The potentially bidentate bases, bipy and phen, form much more stable adducts than the unidentate heterocyclic bases, indicating that chelation occurs, with both N atoms coordinated; the enthalpies are however smaller with bipy or phen, showing that only weak Hg—N bonds are formed. This now appears to be a common feature of mercury chemistry [2,7,12,13] and probably arises from misfitting of the small chelate angle with the preferred near linear stereochemistry of the mercury, leading to poor orbital overlap in these cases.

## Experimental

*Materials.* Alkylmercury(II) halides were prepared by the Grignard method and purified by crystallisation from suitable solvents [14]. The chlorinated compounds $Cl_3CHgCl$ and $(Cl_3C)_2Hg$ were prepared by reaction of sodium trichloracetate with mercury(II) chloride in 1,2-dimethoxyethane and purified by crystallisation from chloroform [15]. Liquid bases were purified by distillation and dried over anhydrous potassium carbonate; solid bases were purified by recrystallisation; benzene solutions of 1,10-phenanthroline were dehydrated by azeotropic distillation of solvent before making up to volume. Benzene for use as solvent was purified by freezing, followed by distillation and dried over calcium hydride.

*Calorimetry.* All calorimetric measurements were made on a LKB 8700 titration calorimeter, using previously described techniques [16]. Briefly, a benzene solution of the base was titrated into 100 ml of a benzene solution of the mercury compound and the heat change measured after each incremental addition of titrant. The enthalpy of reaction was obtained from the extrapolated, integrated heat of reaction and the adduct formation constant, $K$, calculated at each point; the enthalpy was then refined iteratively to give constant values of $K$ throughout the titration. Values of the enthalpy in the tables are the average of at least three determinations over the range of mercury concentrations given; uncertainties in $\Delta H^0$ are mean deviations. Values of $K$ are similarly averaged; uncertainties in $K$ are the sum of the mean diviation from different titrations and the average standard deviation in individual titrations. Uncertainties in $\Delta G^0$ and $\Delta S^0$ are derived.

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