

The Structures of $[\text{Co}(\text{en})_3][\text{HgCl}_4\text{I}]$ and *cis*- $[\text{Co}(\text{en})_2(\text{NH}_3)(\text{py})][\text{Hg}_2\text{Cl}_7]$ – Complexes Containing Novel Halo-mercury(II) Anions

DONALD A. HOUSE, VICKIE MCKEE and WARD T. ROBINSON

Department of Chemistry, University of Canterbury, Christchurch (New Zealand)

(Received October 3, 1988)

Abstract

The single crystal X-ray structures of the title complexes have been determined. Racemic $[\text{Co}(\text{en})_3][\text{HgCl}_4\text{I}]$ (monoclinic, $P2_1/a$, $a = 13.488$, $b = 11.716$, $c = 11.910$ Å, $\beta = 109.82^\circ$, $V = 1770.6$ Å³, $Z = 4$, $D_{\text{calc}} = 2.67$ g/cc) consists of 1el ob₂ cations associated with $[\text{HgCl}_3\text{I}]^{2-}$ and Cl^- anions. Halo-mercury(II) distances within the irregular tetrahedral $[\text{HgCl}_3\text{I}]^{2-}$ anion are $\text{Hg}-\text{I} = 2.65$ Å and $\text{Hg}-\text{Cl}$ varies from 2.46 to 2.63 Å. Racemic-*cis*- $[\text{Co}(\text{en})_2(\text{NH}_3)(\text{py})][\text{Hg}_2\text{Cl}_7]$ (orthorhombic, $Pbcn$, $a = 22.663$, $b = 14.137$, $c = 14.459$ Å, $V = 4632$ Å³, $Z = 8$, $D_{\text{calc}} = 2.66$ g/cc) consists of isolated $\delta\lambda$ cations and weakly interacting dibridged $[\text{Hg}_2\text{Cl}_7]^{3-}$ anions. Within each $[\text{Hg}_2\text{Cl}_7]^{3-}$ unit, $\text{Hg}-\text{Cl}$ distances vary from 2.34–2.97 Å, and the closest $\text{Hg}-\text{Cl}$ approach between two adjacent $[\text{Hg}_2\text{Cl}_7]^{3-}$ units is 3.36 Å.

Introduction

We have recently been interested in structures adopted by chloro-mercury(II) anions with trivalent cations, [1, 2] as the stoichiometry of the salt rarely reflects the nature of the anions in the crystal lattice [3].

This paper reports the nature of the anions formed when *rac*- $[\text{Co}(\text{en})_3]\text{I}_3$ or *rac-cis*- $[\text{Co}(\text{en})_2(\text{NH}_3)(\text{py})](\text{ClO}_4)_3$ [4] are crystallized from 3 M HCl containing excess HgCl_2 . In the case of $[\text{Co}(\text{en})_3]\text{I}_3$, the first product to form was $[\text{Co}(\text{en})_3][\text{HgCl}_5]^*$ (as needles) which were unsuitable for single crystal X-ray analysis. The mother liquor subsequently deposited $[\text{Co}(\text{en})_3][\text{HgCl}_4\text{I}]^*$ (1) as large irregular blocks. With *cis*- $[\text{Co}(\text{en})_2(\text{NH}_3)(\text{py})](\text{ClO}_4)_3$, the only product isolated was *cis*- $[\text{Co}(\text{en})_2(\text{NH}_3)(\text{py})][\text{Hg}_2\text{Cl}_7]^*$ (2).

We report here the results of single crystal X-ray structural analyses of 1 and 2.

*This represents the stoichiometric formula only and it would be unwise to assume the existence of any particular anionic structural unit in the lattice.

Experimental

Racemic $[\text{Co}(\text{en})_3]\text{Cl}_3 \cdot x\text{H}_2\text{O}$ was prepared according to literature procedures [5] and converted to the triiodide by metathesis (with NaI).

$[\text{Co}(\text{en})_3]\text{I}_3$ (2 g) was dissolved in 3 M HCl (100 ml) at 50 °C and a solution of HgCl_2 (5 g) in 3 M HCl (50 ml), also at 50 °C, was added. On spontaneous cooling to room temperature a considerable quantity (~2 g) of $[\text{Co}(\text{en})_3][\text{HgCl}_5]^*$ deposited as needles and these were removed by filtration after five hours. Anal. Calc. for $\text{C}_6\text{H}_{24}\text{N}_6\text{Cl}_5\text{CoHg}$: C, 11.68; H, 3.92; N, 13.62. Found: C, 11.80; H, 4.28; N, 13.61%. The still yellow mother liquor was allowed to evaporate spontaneously at room temperature in an open beaker for 7–10 days, during which time crystals of $[\text{Co}(\text{en})_3][\text{HgCl}_4\text{I}]^*$ (1) suitable for single crystal X-ray structural analysis, slowly deposited.

This same product can be obtained more easily by dissolving $[\text{Co}(\text{en})_3][\text{HgCl}_5]^*$ (2 g) in 3 M HCl (150 ml, 80 °C) and adding NaI (~1 g). If too much iodide is added, a flocculant yellow precipitate deposits (at 80 °C). This can be redissolved by addition of sufficient HgCl_2 (~1 g) to give a clear yellow solution. $[\text{Co}(\text{en})_3][\text{HgI}_4\text{Cl}]^*$ deposits (irregular blocks) on slow cooling to room temperature.

$[\text{Co}(\text{en})_2(\text{NH}_3)(\text{py})][\text{Hg}_2\text{Cl}_7]$ (2) deposits slowly from a room temperature solution prepared from 1.5 g of the perchlorate salt [4] in 50 ml of 3 M HCl followed by the addition of 5 g of HgCl_2 in 50 ml of 3 M HCl.

X-ray Structural Analyses

Crystal parameters and data collection details (Nicolet R3m diffractometer) for 1 and 2 are presented in Table 1. The non-unique data were averaged and the data corrected for Lorentz, polarization and absorption effects and check reflection fluctuations. The structures were solved using Patterson calculations and conventional difference Fourier synthesis methods (SHELXTL) [6]. In the final cycles, hydrogen atoms were included in idealized calculated positions. For 1, all Hg, halogen and Co atoms were refined using anisotropic thermal

TABLE 1. Crystal data for $(\pm)$ -[Co(en)₃][HgI₄] (1) and $(\pm)$ -[Co(en)₂(NH₃)(py)][Hg₂Cl₇] (2)

Molecular formula	C ₆ H ₂₄ N ₆ ICl ₄ Hg (1)	C ₉ H ₂₄ N ₆ Cl ₇ CoHg ₂ (2)
Formula weight	708.2	924.4
Space group	<i>P</i> 2 ₁ / <i>a</i>	<i>Pbcn</i>
<i>a</i> (Å)	13.488(6)	22.663(8)
<i>b</i> (Å)	11.716(4)	14.137(4)
<i>c</i> (Å)	11.910(4)	14.459(4)
α (°)	90	90
β (°)	109.82(3)	90
γ (°)	90	90
<i>V</i> (Å ³)	1771(1)	4632(2)
<i>Z</i>	4	8
Temperature (°C)	−130	−135
<i>D</i> _{calc} (g cm ^{−3})	2.67	2.66
<i>D</i> _{meas} (g cm ^{−3})	2.60	2.62
<i>F</i> (000)	1320	3407
Absorption coefficient, μ (cm ^{−1})	119	148
Absorption correction	empirical	empirical
Transmission factors	max. 0.964 min. 0.443	max. 0.432 min. 0.192
Mo K α (λ) (Å)	0.71069	0.71069
Scan mode	ω scans 6° min ^{−1}	ω scans var. 2.93–29.3° min ^{−1}
Octants	<i>h</i> 0–17, <i>k</i> 0–14, <i>l</i> 0–±15	<i>h</i> 0–25, <i>k</i> 0–16, <i>l</i> 0–16
2 θ _{max} (°)	50	45
Reflections measured	3418	3366
Reflections used	2509	2255
Parameters refined	109	111
Weighting, <i>g</i>	8.8 × 10 ^{−4}	10 × 10 ^{−4}
<i>R</i>	0.0411	0.0492
<i>R</i> ₂	0.0536	0.0665

parameters and for **2** all non-hydrogen atoms were refined similarly. Non-hydrogen atom coordinates are listed in Tables 2 and 3. See also 'Supplementary Material'.

Results and Discussion

[Co(en)₃][HgCl₄I] consists of Δ - plus Λ -[Co(en)₃]³⁺ cations and isolated Cl[−] and [HgCl₃I]^{2−} anions (Fig. 1). The three chelate rings in the [Co(en)₃]³⁺ cations adopt the 1el ob₂ configuration (Fig. 2) as does the racemic triiodide salt [7–9]. While salts containing more or less tetrahedral [HgCl₄]^{2−} or [HgI₄]^{2−} anions are known [1, 10–12], the above is the first characterized [13] example of the [HgCl₃I][−] anion (Fig. 2, Table 4). The mean Hg–Cl distance of 2.53 Å is quite comparable to that observed (~2.49 Å) [10, 11] in isolated tetrahedral [HgCl₄]^{2−} anions. However, the Hg–I distance of 2.651(1) Å is considerably shorter than those observed in tetrahedral [HgI₄]^{2−} anions (2.75–2.80 Å) [12], and is similar to the distance observed in various forms of HgI₂ (~2.62 Å) [14, 15].

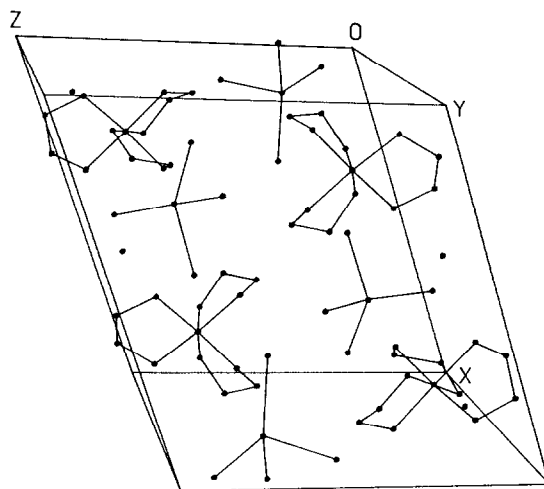
TABLE 2. Non-hydrogen atom coordinates (×10⁴) for $(\pm)$ -[Co(en)₃][HgI₄] (1)

Atom	<i>x</i>	<i>y</i>	<i>z</i>
Hg(1)	3634(1)	5245(1)	7369(1)
I	1777(1)	6262(1)	6453(1)
Cl(1)	5252(2)	6385(2)	7536(3)
Cl(2)	4251(2)	3917(2)	9069(2)
Cl(3)	3727(2)	3702(2)	5799(2)
Cl(4) ^a	9073(2)	6167(2)	943(2)
Co	7499(1)	4024(1)	7787(1)
N(1)	6362(7)	4293(7)	6250(8)
C(1)	6192(9)	3226(9)	5521(10)
C(2)	6251(9)	2255(9)	6354(10)
N(2)	7229(7)	2402(7)	7394(8)
C(3)	7069(9)	3943(9)	9965(10)
N(3)	6497(7)	3999(7)	8655(8)
C(4)	8059(8)	3260(9)	10181(10)
N(4)	8579(7)	3704(7)	9345(8)
N(5)	7767(7)	5662(8)	8071(9)
C(5)	8705(9)	6014(10)	7754(11)
C(6)	8717(9)	5318(9)	6701(11)
N(6)	8574(7)	4090(7)	7016(9)

^aIsolated chloride ion.

TABLE 3. Non-hydrogen atom coordinates ($\times 10^4$) for ($\pm$)-*cis*-[Co(en)₂(NH₃)(py)][Hg₂Cl₇] (2)

Atom	x	y	z
Hg(1)	1407(1)	2213(1)	4423(1)
Hg(2)	1853(1)	4656(1)	3068(1)
Co	1188(1)	1818(1)	338(1)
Cl(11)	1138(1)	3019(2)	2914(2)
Cl(12)	852(1)	2579(2)	5776(2)
Cl(13)	2268(1)	1263(2)	4107(3)
Cl(21)	2160(1)	3896(2)	4638(2)
Cl(22)	1091(1)	5774(2)	3220(2)
Cl(23)	2566(2)	4111(4)	2013(3)
Cl(0)	535(1)	737(2)	3909(2)
N(1)	823(4)	1335(6)	1471(6)
N(2)	1487(4)	519(7)	197(7)
N(3)	503(4)	1483(6)	-426(6)
N(4)	1555(4)	2181(7)	-848(6)
N(5)	879(4)	3106(7)	556(5)
N(6)	1902(4)	2144(8)	1051(7)
C(1)	1104(6)	467(9)	1762(9)
C(2)	1206(7)	-116(9)	906(8)
C(3)	570(5)	1871(9)	-1355(8)
C(4)	1210(5)	1773(9)	-1625(8)
C(5)	1218(5)	3893(9)	504(8)
C(6)	1027(5)	4771(8)	752(7)
C(7)	463(5)	4874(9)	1058(8)
C(8)	100(5)	4112(8)	1095(8)
C(9)	316(4)	3231(8)	863(7)

Fig. 1. A packing diagram for (1el ob₂)-($\pm$)-[Co(en)₃][HgCl₄I] (1) showing the Δ -($\lambda\delta\delta$) plus Λ -($\delta\lambda\lambda$) cations and the Cl⁻ and [HgCl₃I]²⁻ anions.

The other cobalt(III) complex, of formal stoichiometry, [Co(en)₂(NH₃)(py)][Hg₂Cl₇], consists of Δ - plus Λ -*cis*-[Co(en)₂(NH₃)(py)]³⁺ cations and [Hg₂Cl₇]³⁻ anions. In the cations, the two chelate rings adopt the $\delta\lambda$ conformation (Fig. 3, Tables 5 and 6) and the Co-N(py) distance [1.971(9) Å] is not significantly different from the other

TABLE 4. Selected bond lengths (Å) and bond angles (°) for ($\pm$)-[Co(en)₃][HgI₄] (1)

Cation			
Co-N(1)	1.974(8)	Co-N(2)	1.962(9)
Co-N(3)	1.960(11)	Co-N(4)	1.968(8)
Co-N(5)	1.960(9)	Co-N(6)	1.964(12)
Co-N (mean) = 1.965(5)			
N(1)-C(1)	1.494(14)	C(1)-C(2)	1.493(16)
N(2)-C(2)	1.482(12)	C(3)-C(4)	1.502(16)
N(3)-C(3)	1.489(13)	C(5)-C(6)	1.500(18)
N(4)-C(4)	1.492(17)		
N(5)-C(5)	1.496(17)		
N(6)-C(6)	1.515(14)		
N(1)-Co-N(4)	176.9(4)		
N(2)-Co-N(5)	176.2(4)		
N(3)-Co-N(6)	176.1(3)		
N(1)-C(1)-C(2)-N(2)	-50.1(1.2)		
N(3)-C(3)-C(4)-N(4)	47.5(1.0)		
N(5)-C(5)-C(6)-N(6)	-49.2(1.0)		
Anion, HgCl ₃ I ²⁻			
Hg-Cl(1)	2.508(3)	Hg-Cl(2)	2.464(3)
Hg-Cl(3)	2.634(3)	Hg-I	2.651(1)
I-Hg-Cl(1)	118.0(1)		
I-Hg-Cl(2)	129.1(1)		
I-Hg-Cl(3)	106.4(1)		
Cl(1)-Hg-Cl(2)	103.2(1)		
Cl(1)-Hg-Cl(3)	99.9(1)		
Cl(2)-Hg-Cl(3)	93.8(1)		

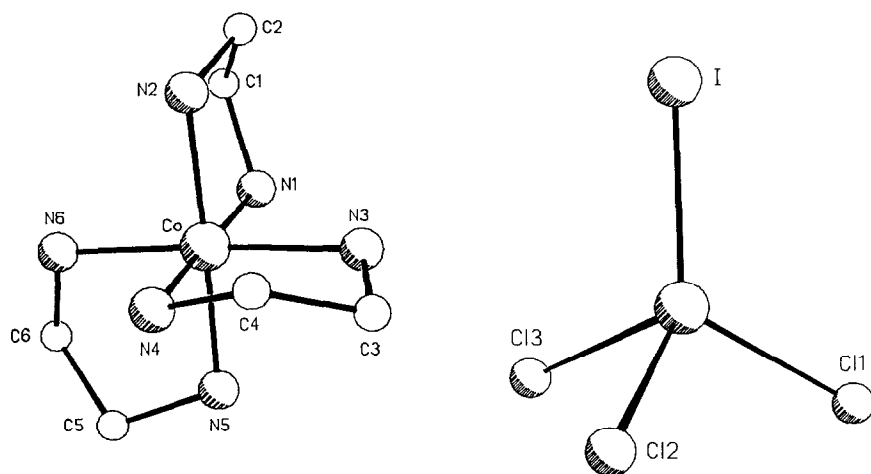


Fig. 2. Perspective views of the Λ -($\delta\lambda\lambda$)-[Co(en) $_3$] $^{3+}$ cation and the [HgCl $_3$ I] $^{2-}$ anion. In the lattice there are equal amounts of the Δ -($\lambda\delta\delta$) cation.

TABLE 5. Selected bond lengths (Å) and bond angles (°) for ($\pm$)-*cis*-[Co(en) $_2$ (NH $_3$)(py)]Hg $_2$ Cl $_7$ (2)

Cation			
Co—N(1)(en)	1.954(8)	N(1)—C(1)	1.443(16)
Co—N(2)(en)	1.964(10)	N(2)—C(2)	1.501(16)
Co—N(3)(en)	1.958(8)	N(3)—C(3)	1.456(14)
Co—N(4)(en)	1.970(9)	N(4)—C(4)	1.482(15)
Co—N(5)(py)	1.971(9)	C(1)—C(2)	1.502(18)
Co—N(6)(NH $_3$)	1.970(9)	C(3)—C(4)	1.506(15)
N(5)—C(5)	1.351(15)	C(6)—C(7)	1.359(17)
N(5)—C(9)	1.360(13)	C(7)—C(8)	1.354(17)
C(5)—C(6)	1.359(17)	C(8)—C(9)	1.377(16)
N(1)(en)—Co—N(4)(en)	174.6(4)		
N(2)(en)—Co—N(5)(py)	176.7(4)		
N(6)(NH $_3$)—Co—N(3)(en)	177.1(4)		
N(3)—C(3)—C(4)—N(4)	-50.8(1.2)		
N(1)—C(1)—C(2)—N(2)	48.3(1.3)		
Anion, Hg $_2$ Cl $_7$ $^{3-}$			
Hg(1)—Cl(0)	2.961(3)	Hg(2)—Cl(11)	2.828(3)
Hg(1)—Cl(12)	2.376(3)	Hg(2)—Cl(21)	2.601(3)
Hg(1)—Cl(13)	2.408(3)	Hg(2)—Cl(22)	2.346(3)
Hg(2) ... Cl(13)'	3.36		
Hg(1) ... Hg(2)	4.09		
Cl(11)—Hg(1)—Cl(0)	86.6(1)		
Cl(11)—Hg(1)—Cl(21)	82.4(1)		
Cl(11)—Hg(1)—Cl(13)	106.3(1)		
Cl(11)—Hg(1)—Cl(12)	118.8(1)		
Cl(12)—Hg(1)—Cl(0)	90.4(1)		
Cl(12)—Hg(1)—Cl(21)	92.5(1)		
Cl(12)—Hg(1)—Cl(13)	134.7(1)		
Cl(13)—Hg(1)—Cl(0)	95.7(1)		
Cl(13)—Hg(1)—Cl(21)	90.1(1)		
Cl(21)—Hg(1)—Cl(0)	168.6(1)		
Cl(11)—Hg(2)—Cl(23)	94.3(1)		
Cl(11)—Hg(2)—Cl(22)	97.8(1)		
Cl(11)—Hg(2)—Cl(21)	83.4(1)		
Cl(21)—Hg(2)—Cl(23)	104.3(1)		
Cl(21)—Hg(2)—Cl(22)	113.1(1)		
Cl(22)—Hg(2)—Cl(23)	141.8(1)		
Hg(1)—Cl(11)—Hg(2)	99.3(1)		
Hg(1)—Cl(21)—Hg(2)	94.9(1)		

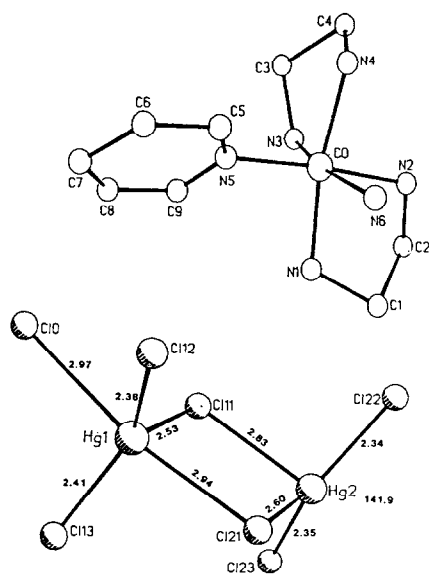


Fig. 3. Perspective views of the Δ -($\delta\lambda$)-[Co(en)₂(NH₃)(py)]³⁺ cation and the Hg₂Cl₇³⁻ anion. In the lattice, there are equal amounts of the Λ -($\lambda\delta$) cation.

Co–N bond lengths. This Co–N(py) distance is similar to the distance found in ($\pm$)-*cis*-[CoCl(en)₂(py)]²⁺ [16], namely, 1.992(2) Å. Mean Co–N(en) distances in these Co(III) cations [1.965(5) for 1 and 1.961(7) Å for 2] are remarkably consistent. As with [M(en)₃]³⁺ systems [9], the ring conformation adopted in the solid state for *cis*-[M(en)₂XY]Z systems appears to be anion dependent (Table 6).

Although various ionic or polymeric forms corresponding to [Hg₂Cl₇]³⁻ have been reported previously [1, 17] (Table 7) we would regard the anion found in structure 2 to be an isolated dibridged [Hg₂Cl₇]³⁻ unit (Fig. 3).

The next closest Cl atom to Hg(2) [Cl(13)] (Fig. 3) is at 3.36 Å, compared with a distance of 3.14 Å where linear singly bridged [Hg₂Cl₇]³⁻_n chains are proposed [1] and the Cl(23)–Hg–Cl(22) angle of 141.8° observed in the monomeric unit (Fig. 3), is opened to 165.3° in the chain structure [1].

Thus, this isolated [Hg₂Cl₇]³⁻ anion can be regarded as a chloro adduct of the known dimeric, chloro-bridged, Hg₂Cl₆²⁻ anion [36–38].

TABLE 6. Ordered ring conformations in some *cis*-[Co(XY)(en)₂]Z complexes

Complex	Enantiomer	Conformation ^a	Dihedral angles ^b	Reference
[CoCl(en) ₂ (NH ₃)]Cl ₂	(±)	($\delta\lambda$)	50.2, -49.5	16
[CoCl(en) ₂ (py)]Cl ₂ ·H ₂ O	(±)	($\lambda\lambda$)	-49.9, -50.0	16
Na ₂ [Co(SO ₃) ₂ (en) ₂]ClO ₄ ·3H ₂ O	(±)	($\delta\delta$)	46.6, 49.2	18
[Co(CN) ₂ (en) ₂]Cl·H ₂ O	(+)	Λ ($\lambda\lambda$)	-47.2, -46.0	19
[CoCl ₂ (en) ₂]Cl·H ₂ O	(+)	Λ ($\delta\delta$)	48.1, 53.3	20
[CoCl ₂ (en) ₂]Cl·H ₂ O	(±)	($\delta\lambda$)		21
[Co(NO ₂) ₂ (en) ₂]NO ₃	(±)	($\delta\lambda$)	44.9, -44.0	22
[Co(NO ₂) ₂ (en) ₂][Co(mal) ₂ (en)]·2H ₂ O	(-)	Δ ($\delta\delta$)	47.9, 45.9	23
[Co(NO ₂) ₂ (en) ₂][Co(NO ₂) ₂ (ox)(NH ₃) ₂]·H ₂ O	(-)	Δ ($\delta\lambda$)		24
[Co(NO ₂) ₂ (en) ₂][Co(NO ₂) ₄ (NH ₃) ₂]	(+) ^c	Λ ($\delta\lambda$)	46.6, -46.6	25
[Co(NO ₂) ₂ (en) ₂]Cl	(+) ^c	Λ ($\delta\lambda$)	50.2, -48.1	26
[Co(NCS) ₂ (en) ₂]Cl·H ₂ O	(±)	($\delta\lambda$)	48.3, -47.6	27
[Co(N ₃) ₂ (en) ₂] ₂ [Sb ₂ (+)-(tart) ₂]	(+)	Λ		28
[CoCl(en) ₂ (imid)]Cl ₂	(±)	($\lambda\lambda$)	-56.6, -45.9	29
[CoCl(en) ₂ [(S)-prol]]ZnCl ₄	(-)	Δ ($\delta\lambda$)	45.2, -45.3	30
[CoCl{adenine(-1)}(en) ₂]Br·H ₂ O	(±)	$\lambda\lambda$		31
[CoCl{theophylline(-1)}(en) ₂]ClO ₄	(±)	$\lambda\lambda$		32
[Co(en) ₂ (NH ₃)(py)](Hg ₂ Cl ₇)	(±)	$\delta\lambda$	48.3, -50.8	d
[CoBr(en) ₂ (py)](NO ₃) ₂	(±)	$\delta\lambda$	42.9, -44.2	33
[Co(NO ₂)(SO ₃)(en) ₂]	(±)	($\delta\lambda$)		34
[Co(N ₃)(SO ₃)(en) ₂]	(±)	($\delta\delta$)		34
[Co(NO ₂)(SSO ₃)(en) ₂]	(±)	($\delta\lambda$)	48.8, -50.7	35

^aFor a racemic crystal, the lattice will contain equal amounts of the $\Delta\lambda\lambda$ and $\Lambda\delta\delta$ or $\Delta\delta\lambda$ and $\Lambda\delta\lambda$ cations. The data reported in this Table are those assigned to the particular absolute configuration used in the structure analysis. ^bIn many instances these have been calculated from the cited atom coordinates. Where numbers are not given, either the atom coordinates are not available to us, or unreasonable dihedral angles were calculated. ^cThe sign of rotation reported in this work [25, 26] has been incorrectly assigned (I. Bernal, personal communication) and should be (+). ^dThis research.

TABLE 7. Structures adopted by the 'Hg₂Cl₇³⁻' anion

Cation	Structure	Reference
<i>u-fac</i> -[Cr(dien) ₂] ³⁺	(HgCl ₃ ⁻) _n chains and weakly associated (Hg Cl = 3.3–3.4 Å) HgCl ₄ ²⁻ anions	1
<i>s-fac</i> -[Cr(dien) ₂] ³⁺	2HgCl ₄ ²⁻ , Hg ₂ Cl ₆ ²⁻ and Hg ₄ Cl ₁₄ ⁶⁻ anions	1
<i>s-fac</i> -[CrCl(dien)(Hdien) ₂ N,N)] ³⁺	[Hg ₂ Cl ₇ ³⁻] _n chains (Hg Cl = 3.21 Å between chains)	1
<i>cis</i> -[Co(en) ₂ (NH ₃)(py)] ³⁺	weakly associated (Hg Cl = 3.36 Å) dibridged Hg ₂ Cl ₇ ³⁻ anions	this research
(±)-[Co(en) ₃] ³⁺	unknown	17

We will discuss in detail, the whole question of bonding in chloro-mercury(II) anions in a subsequent review paper.

Supplementary Material

Tables of structure factors, hydrogen atom coordinates and anisotropic thermal parameters are available from the authors.

Acknowledgements

We thank the New Zealand Universities Grants Committee for funds to purchase instruments used in this research, and Miss S. E. Miller for the preparation of the crystals.

References

- 1 D. A. House, V. McKee and W. T. Robinson, *Inorg. Chim. Acta*, **157** (1989) 15.
- 2 G. H. Searle and D. A. House, *Aust. J. Chem.*, **40** (1987) 361.
- 3 D. Grdenic, *Q. Rev.*, **19** (1965) 303.
- 4 D. A. House and R. S. Visser, *J. Inorg. Nucl. Chem.*, **38** (1976) 1157.
- 5 G. G. Schlessinger, *Inorganic Laboratory Preparations*, Chem. Publishing Co., New York, 1962, p. 186.
- 6 G. M. Sheldrick, *SHELXTL User Manual*, Revision 5, Nicolet XRD Corp., Madison, WI, 1986.
- 7 A. Whulcr, P. Spinat and C. Brouty, *Acta Crystallogr., Sect. B*, **36** (1980) 1086.
- 8 D. A. House, in G. Wilkinson (ed.), *Comprehensive Coordination Chemistry*, Vol. 2, Pergamon, New York, Ch. 13.1, Table 5.
- 9 S. E. Miller and D. A. House, *Inorg. Chim. Acta*, **157** (1989) 29.
- 10 A. Ben Salah, J. W. Bats, H. Fuess and A. Daoud, *Inorg. Chim. Acta*, **63** (1982) 169.
- 11 B. Kamenar and A. Nagl, *Acta Crystallogr., Sect. B*, **32** (1976) 1414.
- 12 M. Korfer, H. Fuess and J. W. Bats, *Z. Anorg. Allgem. Chem.*, **543** (1986) 104.
- 13 J. G. Contreras and G. V. Seguel, *Inorg. Chim. Acta*, **61** (1982) 99.
- 14 D. Schwarzenbach, *Z. Kristallogr.*, **128** (1969) 97.
- 15 G. A. Jeffrey, *Inorg. Chim. Acta*, **6** (1967) 396.
- 16 J. MacB. Harrowfield, B. W. Skelton, A. H. White and F. R. Wilner, *Aust. J. Chem.*, **39** (1986) 339.
- 17 A. W. Herlinger, J. N. Brown, M. A. Dwyer and S. Pavkovic, *Inorg. Chem.*, **20** (1981) 2366.
- 18 C. I. Raston, A. H. White and J. K. Yandell, *Aust. J. Chem.*, **32** (1979) 291.
- 19 K. Matsumoto, S. Ooi and H. Kuroya, *Bull. Chem. Soc. Jpn.*, **40** (1967) 2988.
- 20 K. Matsumoto, S. Ooi and H. Kuroya, *Bull. Chem. Soc. Jpn.*, **43** (1970) 3801.
- 21 K. Matsumoto, S. Ooi and H. Kuroya, *Nippon Kagaku Zasshi*, **89** (1968) 167.
- 22 O. Bortin, *Acta Chem. Scand., Ser. A*, **30** (1976) 657; **23** (1969) 3273.
- 23 K. Matsumoto and K. Huroya, *Bull. Chem. Soc. Jpn.*, **45** (1972) 1775.
- 24 H. Shintani, S. Sato and Y. Saito, *Acta Crystallogr., Sect. B*, **32** (1976) 1184.
- 25 I. Bernal, *Inorg. Chim. Acta*, **101** (1985) 175.
- 26 I. Bernal, *Inorg. Chim. Acta*, **96** (1985) 99.
- 27 U. Schubert, B. Zimmer-Gasser, K. C. Dash and G. Ray Chaudhury, *Cryst. Struct. Commun.*, **10** (1981) 251.
- 28 K. Miyoshi, Y. Matsumoto and Y. Yoneda, *Inorg. Chem.*, **20** (1981) 1057, cited in ref. 49 of this paper. No further information appears to have been published.
- 29 B. Zimmer-Gasser and K. C. Dash, *Inorg. Chim. Acta*, **55** (1980) 43.
- 30 D. A. House, G. Hall, A. J. Matheson, W. T. Robinson, F. C. Ha and C. B. Knobler, *Inorg. Chim. Acta*, **39** (1980) 257.
- 31 T. J. Kistenmacher, L. G. Marzilli and C. H. Chang, *J. Am. Chem. Soc.*, **95** (1973) 5817.
- 32 T. J. Kistenmacher and D. J. Szalda, *Acta Crystallogr., Sect. B*, **31** (1975) 90.
- 33 O. Bortin, *Acta Chem. Scand.*, **23** (1969) 3274.
- 34 I. Bernal, *Inorg. Chim. Acta*, **142** (1988) 21.
- 35 A. R. Murdock, T. Tyree, W. O. Herbain, L. Kinney, M. Carreras, J. N. Cooper and R. C. Elder, *Inorg. Chem.*, **24** (1985) 3674.
- 36 K. Brodersen, G. Pezzeti and G. Thiele, *Z. Anorg. Allg. Chem.*, **499** (1983) 169.
- 37 P. L. Goggin, P. King, D. M. McEwan, G. E. Taylor, P. Woodward and M. Sandstrom, *J. Chem. Soc., Dalton Trans.*, (1982) 875.
- 38 R. P. Shibaera and L. P. Rozenberg, *Sov. Phys. Crystallogr.*, **26** (1981) 695; *Chem. Abstr.*, **96** (1982) 27120.