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## Structures of (I) Bis[benzylidimethyl(phenyl)ammonium] Tetrachloromanganate(II) and (II) Bis[benzylidimethyl(phenyl)ammonium] Tetrachlorocobaltate(II)

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**Abstract.** (I):  $[\text{C}_{15}\text{H}_{18}\text{N}]_2[\text{MnCl}_4]$ ,  $M_r = 621.4$ ,  $a = 13.150$  (6),  $b = 14.105$  (8),  $c = 16.694$  (8) Å,  $V = 3095$  (5) Å<sup>3</sup>,  $D_m = 1.35$ ,  $D_x = 1.33$  Mg m<sup>-3</sup>,  $\mu = 0.812$  mm<sup>-1</sup>,  $F(000) = 1292$ , final  $R = 0.041$  for 967 'observed' reflections. (II):  $[\text{C}_{15}\text{H}_{18}\text{N}]_2[\text{CoCl}_4]$ ,  $M_r = 625.4$ ,  $a = 13.040$  (4),  $b = 14.085$  (9),  $c = 16.450$  (5) Å,  $V = 3021$  (4) Å<sup>3</sup>,  $D_m = 1.38$ ,  $D_x = 1.37$  Mg m<sup>-3</sup>,  $\mu = 0.964$  mm<sup>-1</sup>,  $F(000) = 1300$ , final  $R = 0.044$  for 1091 'observed' reflections. Both (I) and (II): orthorhombic,  $C22_1$ ,  $Z = 4$ ,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $T = 298$  K. The two complexes are isomorphous. The conformation of the N<sup>+</sup>—C—C—C part of the cation is *gauche* and is unaffected by the presence of the bulky anions. The metal ions have almost perfect tetrahedral geometry. The crystal structures are stabilized by Coulombic and van der Waals forces.

**Introduction.** Quaternary amines are active at the cholinergic junctions, their pharmacological activities

being correlated with their conformations. It has been found that benzylidimethyl(phenyl)ammonium chloride has close conformational similarity to the quaternary amine acetylcholine and its structural analogues (Chaudhuri, Banerjee, Deb Purkayastha, Sen Sarma & Bocelli, 1987). The present study was undertaken to study the effect of the presence of bulky anions, like  $\text{MnCl}_4^{2-}$  and  $\text{CoCl}_4^{2-}$ , on the conformation of benzylidimethyl(phenyl)ammonium.

**Experimental.** Pale green crystals of Mn complex (I) and deep blue crystals of Co complex (II) from hot methanol, density by flotation (benzene–bromoform mixture), crystal size (I) 0.15 × 0.15 × 0.19 mm and (II) 0.13 × 0.17 × 0.21 mm, Siemens AED diffractometer, Zr-filtered Mo  $K\alpha$  radiation, accurate cell parameters from least-squares fit of setting angles of (I) 27 reflections ( $8 \leq \theta \leq 20^\circ$ ) and (II) 25 reflections ( $10 \leq \theta \leq 22^\circ$ ) using the *CTDIF* routine (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979),  $C22_1$  (systematic absences:  $hkl$ ,  $h+k$  odd;  $00l$ ,  $l$  odd), 2109 unique reflections measured for (I) and 2017 for (II) ( $2 \leq \theta \leq 30^\circ$ ,  $0 \leq h \leq 18$ ,  $0 \leq k \leq 19$ ,  $0 \leq l \leq 23$  for

\* The early part of the work was done while the author was at Saha Institute of Nuclear Physics, Calcutta 700 064, India.

both), 967 'observed' reflections [ $I \leq 2.5\sigma(I)$ ] for (I) and 1091 for (II), two standard reflections ( $< 2\%$  intensity variation), correction for Lp and statistical fluctuation, absorption ignored. Structure of Mn complex (I) solved by direct and heavy-atom methods; *SHELX76* (Sheldrick, 1976) used with 209  $E$ 's  $\geq 1.4$ , origin- and enantiomorph-defining reflections together with six multiresolution reflections chosen manually from a convergence map; 512 phase sets generated, Mn and two Cl atoms located unambiguously from best  $E$  map, Mn located on a twofold axis; subsequent heavy-atom phased electron density map based on these atoms revealed the remaining 16 non-H atoms of the asymmetric unit; full-matrix least-squares refinement using *SHELX76* with anisotropic thermal parameters for non-H atoms; H atoms, except those of the methyl groups, placed in idealized geometrical positions and assigned isotropic thermal parameters of  $U = 0.05 \text{ \AA}^2$ ; subsequent refinement with fixed parameters for H atoms resulted in  $R = 0.041$ ,  $wR = 0.056$ ,  $S = 1.31$ ,  $w = 1/[\sigma^2(|F_o|) + 0.0071|F_o|^2]$ ;  $(\Delta/\sigma)_{\max} < 0.04$ ;  $\Delta\rho$  in final  $\Delta F$  synthesis in the range  $-0.31$  to  $0.28 \text{ e \AA}^{-3}$ . Co complex (II) isomorphous with Mn complex (I); refinement started with final positional coordinates of corresponding non-H atoms of (I); H-atom positions generated as in (I) and positional and thermal parameters kept fixed; final  $R = 0.044$ ,  $wR = 0.061$ ,  $S = 1.37$ ,  $w = 1/[\sigma^2(|F_o|) + 0.0092|F_o|^2]$ ;  $(\Delta/\sigma)_{\max} < 0.05$ ;  $\Delta\rho$  in final  $\Delta F$  synthesis in the range  $-0.36$  to  $0.31 \text{ e \AA}^{-3}$ . In both (I) and (II), scattering factors of *SHELX76* used for H, C, N, Cl atoms; for Mn and Co coefficients for empirical scattering factor expression from *International Tables for X-ray Crystallography* (1974).

**Discussion.** The positional and equivalent isotropic thermal parameters for the non-H atoms of the two complexes are listed in Table 1.\* Fig. 1 shows a perspective view of the Mn complex together with the labeling scheme used. An identical labeling scheme has been used for the Co complex. Fig. 2 gives a *STEREO* (Chaudhuri, 1984) view of the crystal packing arrangement for the Mn complex. Bond distances, bond angles and some selected torsion angles are listed in Table 2.

The geometry about the  $N^+$  ion in the two complexes is similar to that observed in the structure of benzyldimethyl(phenyl)ammonium chloride (Chaudhuri *et al.*, 1987). The non-bonded contacts between C(15) and C(6) of  $2.80(2) \text{ \AA}$  in the Mn complex and

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters,  $U_{\text{eq}} (\text{\AA}^2)$ , for the non-H atoms, with *e.s.d.*'s in parentheses

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

Mn complex	x	y	z	$U_{\text{eq}}$
Mn	0.0000	0.4103 (1)	0.2500	0.0367 (8)
Cl(1)	0.1136 (2)	0.3150 (2)	0.3232 (2)	0.060 (1)
Cl(2)	0.0896 (2)	0.5073 (2)	0.1605 (2)	0.076 (1)
N	0.1469 (5)	0.2709 (5)	-0.0227 (4)	0.044 (3)
C(1)	0.1788 (6)	0.1974 (6)	0.0386 (5)	0.040 (4)
C(2)	0.2648 (7)	0.1433 (9)	0.0234 (7)	0.055 (6)
C(3)	0.2955 (8)	0.0814 (8)	0.0817 (7)	0.060 (5)
C(4)	0.2471 (9)	0.0738 (8)	0.1533 (7)	0.067 (5)
C(5)	0.1619 (10)	0.1273 (8)	0.1679 (6)	0.070 (5)
C(6)	0.1277 (8)	0.1915 (8)	0.1101 (6)	0.059 (5)
C(7)	0.0367 (7)	0.1732 (8)	-0.1174 (5)	0.052 (4)
C(8)	0.0319 (8)	0.0754 (9)	-0.0962 (7)	0.075 (7)
C(9)	-0.0525 (10)	0.0224 (12)	-0.1129 (10)	0.102 (8)
C(10)	-0.1325 (10)	0.0616 (14)	-0.1504 (10)	0.100 (9)
C(11)	-0.1331 (9)	0.1559 (12)	-0.1718 (7)	0.081 (9)
C(12)	-0.0465 (7)	0.2135 (10)	-0.1549 (6)	0.068 (6)
C(13)	0.1308 (6)	0.2291 (8)	-0.1067 (5)	0.047 (4)
C(14)	0.2371 (9)	0.3406 (9)	-0.0293 (8)	0.064 (5)
C(15)	0.0517 (7)	0.3252 (8)	0.0015 (6)	0.063 (5)
Co complex				
Co	0.0000	0.4125 (1)	0.2500	0.0310 (8)
Cl(1)	0.1115 (2)	0.3202 (2)	0.3197 (2)	0.050 (1)
Cl(2)	0.0858 (2)	0.5061 (3)	0.1619 (2)	0.064 (1)
N	0.1473 (6)	0.2721 (6)	-0.0243 (5)	0.038 (3)
C(1)	0.1789 (7)	0.1984 (7)	0.0384 (6)	0.035 (3)
C(2)	0.2634 (8)	0.1443 (9)	0.0225 (8)	0.047 (4)
C(3)	0.2967 (9)	0.0817 (9)	0.0815 (7)	0.053 (4)
C(4)	0.2492 (10)	0.0739 (9)	0.1552 (8)	0.059 (4)
C(5)	0.1648 (12)	0.1305 (10)	0.1701 (7)	0.066 (5)
C(6)	0.1279 (9)	0.1941 (8)	0.1109 (7)	0.048 (4)
C(7)	0.0354 (7)	0.1734 (9)	-0.1186 (6)	0.044 (4)
C(8)	0.0307 (9)	0.0765 (9)	-0.0971 (8)	0.063 (5)
C(9)	-0.0534 (13)	0.0230 (12)	-0.1139 (12)	0.089 (7)
C(10)	-0.1358 (11)	0.0578 (14)	-0.1499 (10)	0.086 (7)
C(11)	-0.1359 (9)	0.1562 (14)	-0.1715 (8)	0.073 (7)
C(12)	-0.0500 (8)	0.2130 (11)	-0.1562 (7)	0.062 (5)
C(13)	0.1297 (8)	0.2308 (9)	-0.1093 (6)	0.044 (3)
C(14)	0.2365 (10)	0.3437 (10)	-0.0316 (8)	0.056 (4)
C(15)	0.0525 (8)	0.3261 (8)	0.0004 (7)	0.055 (4)

$2.78(2) \text{ \AA}$  in the Co complex account for the deviation in the tetrahedral angles C(1)—N—C(15) [ $113.1(7)^\circ$  in the Mn complex and  $112.6(8)^\circ$  in the Co complex], from the ideal value of  $109.5^\circ$ . This distortion has also been observed in the structure of benzyldimethyl(phenyl)ammonium chloride. Increase in the N—C(13)—C(7) angle, which is a common feature in acetylcholine-like molecules, is observed in the complexes. As in benzyldimethyl(phenyl)ammonium chloride, the endocyclic angles at C(1) and C(7) deviate from the  $sp^2$  value of  $120^\circ$ . This deviation may be attributed to the substituents, tetrahedral N at C(1) and tetrahedral C at C(7) (Domenicano, Mazzeo & Vaciago, 1976). Similar endocyclic angles in phenyl and benzyl rings have been observed in other quaternary amines (Cocouvanis, Hollander & Caffery, 1976; Raegele & Boeyens, 1977).

The torsion angles N—C(13)—C(7)—C(8) [ $-84.5(11)^\circ$  in the Mn complex and  $-83.6(13)^\circ$  in the Co complex] indicate that the N<sup>+</sup>—C(13)—C(7)—C(8) system is in the *gauche* conformation as is the case in benzyldimethyl(phenyl)-

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52187 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

ammonium chloride (Chaudhuri *et al.*, 1987). This conformation is similar to the conformation of the active part of acetylcholine and its analogues (Herdklotz & Sass, 1970; Canepa, Pauling & Sorum, 1966). As in benzyldimethyl(phenyl)ammonium chloride, the C(7)—C(13)—N—C(14) torsion angles [ $-169.7(8)^\circ$  in the Mn complex and  $-169.5(9)^\circ$  in the Co complex] compare well with those in L-(+)-muscarine (Radna, Beveridge & Bender, 1973), acetylcholine chloride (Herdklotz & Sass, 1970) and acetylcholine bromide (Canepa *et al.*, 1966). The  $N^+ \cdots C(8)$  intramolecular contacts are  $3.376(1) \text{ \AA}$  in the Mn complex and  $3.367(2) \text{ \AA}$  in the Co complex compared to  $3.341(4) \text{ \AA}$  in benzyldimethyl(phenyl)ammonium chloride (Chaudhuri *et al.*, 1987), and are in good agreement with those in the halides of acetylcholine (Herdklotz & Sass, 1970; Canepa *et al.*, 1966). These observations indicate that the presence of bulky anions like  $MnCl_4^{2-}$  and  $CoCl_4^{2-}$  in place of  $Cl^-$  have little effect on the conformation of the benzyldimethyl(phenyl)ammonium cation. A very similar observation has been made with respect to the conformation of acetylcholine where the introduction of the bulky tetrabromodioxouranate ion in place of the bromide ion produces insignificant changes in the conformation (Marzotto, Graziani, Bombieri & Forsellini, 1974).

The dihedral angle between the rings is  $73.1(4)^\circ$  in the Mn complex and  $73.9(4)^\circ$  in the Co complex.

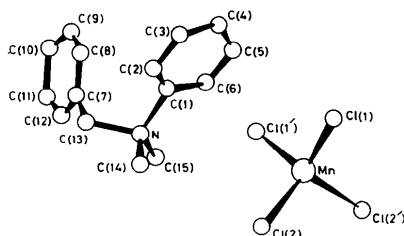


Fig. 1. A perspective view of the Mn complex showing the atom-labeling scheme.

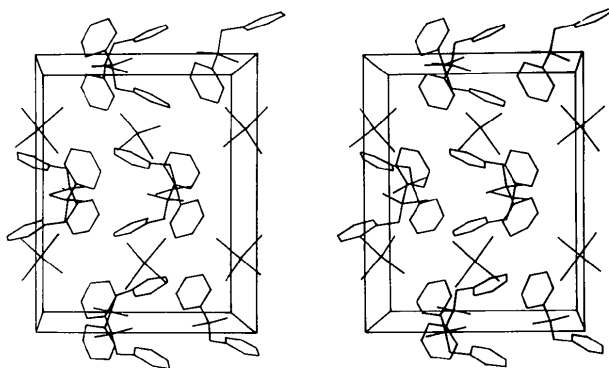


Fig. 2. Stereoview of the crystal packing arrangement for the Mn complex.

Table 2. Bond distances ( $\text{\AA}$ ), bond angles ( $^\circ$ ) and selected torsion angles ( $^\circ$ ), with *e.s.d.*'s in parentheses

	Mn complex	Co complex
$M-Cl(1)$	2.352 (3)	2.262 (3)
$M-Cl(2)$	2.344 (3)	2.256 (4)
$N-C(1)$	1.52 (1)	1.52 (1)
$N-C(13)$	1.54 (1)	1.53 (1)
$N-C(14)$	1.54 (1)	1.54 (2)
$N-C(15)$	1.52 (1)	1.51 (1)
$C(1)-C(2)$	1.39 (1)	1.36 (1)
$C(2)-C(3)$	1.37 (2)	1.38 (2)
$C(3)-C(4)$	1.36 (2)	1.37 (2)
$C(4)-C(5)$	1.37 (2)	1.38 (2)
$C(5)-C(6)$	1.40 (2)	1.41 (2)
$C(6)-C(1)$	1.37 (1)	1.37 (2)
$C(13)-C(7)$	1.48 (1)	1.48 (2)
$C(7)-C(8)$	1.43 (2)	1.41 (2)
$C(8)-C(9)$	1.37 (2)	1.36 (2)
$C(9)-C(10)$	1.34 (2)	1.32 (2)
$C(10)-C(11)$	1.38 (3)	1.43 (3)
$C(11)-C(12)$	1.43 (2)	1.40 (2)
$C(12)-C(7)$	1.38 (1)	1.39 (2)
$Cl(1)-M-Cl(2)$	110.2 (1)	110.0 (1)
$Cl(1)-M-Cl(1')$	110.3 (1)	109.9 (1)
$Cl(1)-M-Cl(2')$	108.8 (1)	109.2 (1)
$Cl(2)-M-Cl(2')$	110.3 (1)	108.5 (1)
$C(1)-N-C(13)$	113.1 (7)	113.6 (8)
$C(1)-N-C(14)$	105.7 (7)	107.1 (8)
$C(1)-N-C(15)$	113.1 (7)	112.6 (8)
$C(13)-N-C(14)$	106.6 (7)	106.8 (9)
$C(13)-N-C(15)$	108.8 (6)	108.4 (8)
$C(14)-N-C(15)$	109.3 (8)	108.0 (8)
$N-C(1)-C(2)$	118.6 (8)	118.0 (10)
$N-C(1)-C(6)$	119.6 (8)	119.4 (9)
$C(2)-C(1)-C(6)$	121.7 (9)	122.4 (10)
$C(1)-C(2)-C(3)$	117.5 (9)	118.4 (11)
$C(2)-C(3)-C(4)$	122.5 (10)	122.2 (12)
$C(3)-C(4)-C(5)$	119.7 (11)	118.2 (12)
$C(4)-C(5)-C(6)$	119.8 (11)	121.1 (13)
$C(5)-C(6)-C(1)$	118.8 (10)	117.8 (11)
$C(13)-C(7)-C(8)$	121.6 (9)	122.6 (10)
$C(13)-C(7)-C(12)$	119.9 (10)	119.5 (11)
$C(12)-C(7)-C(8)$	118.4 (9)	117.7 (11)
$C(7)-C(8)-C(9)$	121.0 (11)	121.4 (12)
$C(8)-C(9)-C(10)$	120.4 (15)	122.8 (16)
$C(9)-C(10)-C(11)$	121.5 (13)	118.1 (14)
$C(10)-C(11)-C(12)$	119.6 (12)	120.6 (13)
$C(11)-C(12)-C(7)$	119.1 (12)	119.4 (13)
$N-C(13)-C(7)$	115.5 (7)	115.2 (9)
$C(1)-N-C(13)-C(7)$	74.6 (9)	72.5 (11)
$C(7)-C(13)-N-C(15)$	-52.0 (10)	-53.4 (11)
$N-C(13)-C(7)-C(12)$	101.1 (11)	102.0 (12)
$C(7)-C(13)-N-C(14)$	-169.7 (8)	-169.5 (9)
$N-C(13)-C(7)-C(8)$	-84.5 (11)	-83.6 (13)

The corresponding dihedral angle in benzyldimethyl(phenyl)ammonium chloride is  $66.67(9)^\circ$ .

The metal ion in the Mn complex has an almost perfect tetrahedral configuration with the  $Cl-Mn-Cl$  angles varying between  $108.8(1)$  and  $110.3(1)^\circ$ . This observation is similar to that in pyridinium tetrachloromanganate(II) (Brassy, Robert, Bachet & Chevalier, 1976). Almost perfect tetrahedral geometry around the metal ion is also observed in the case of the Co complex [the angles are between  $108.5(1)$  and  $110.0(1)^\circ$ ].

The crystal structures of the two complexes are stabilized by van der Waals and Coulombic forces.

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## (1,1,4,7,10,10-Hexaphenyl-1,4,7,10-tetraphosphadecane-*P,P',P'',P'''*)platinum(II) Bis(tetraphenylborate)

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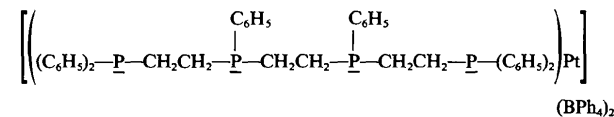
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**Abstract.** [Pt(C<sub>42</sub>H<sub>42</sub>P<sub>4</sub>)]<sub>2</sub>[B(C<sub>24</sub>H<sub>20</sub>)<sub>2</sub>], *M<sub>r</sub>* = 1504.24, monoclinic, *P*<sub>2</sub><sub>1</sub>/*c*, *a* = 23.187 (4), *b* = 13.543 (2), *c* = 28.211 (3) Å, β = 113.29 (1)°, *V* = 8137.0 Å<sup>3</sup>, *Z* = 4, *D<sub>m</sub>* (by flotation) = 1.23, *D<sub>x</sub>* = 1.22 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 1.76 mm<sup>-1</sup>, *F*(000) = 3080, *T* = 294 K, final *R* = 0.079 for 6004 observed reflections. The X-ray structure analysis shows a distorted square-planar structure and the chiral form of the two possible diastereomers of the title compound. The two pairs of P atoms, *trans* to each other, lie above (PPh 0.254, PPh<sub>2</sub> 0.199 Å) and below (PPh 0.273, PPh<sub>2</sub> 0.220 Å) the best plane through the Pt and the P atoms. The Pt atom is shifted out of this plane by 0.039 Å. The Pt—PPh bonds [2.258 (4) Å] are significantly shorter than the Pt—PPh<sub>2</sub> bonds [2.327 (4) Å]. The PhP—Pt—PPh<sub>2</sub> angles and the PhP—Pt—PPh angle are of similar size (mean value 84.3°, Δ<sub>max</sub> = 1.8°), whereas the Ph<sub>2</sub>P—Pt—PPh<sub>2</sub> angle is wide open [109.5 (2)°].

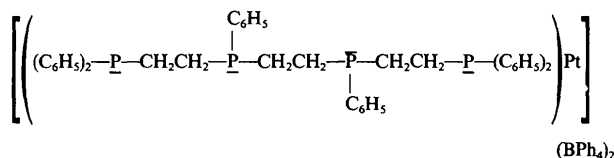
**Introduction.** The tetradentate ligand 1,1,4,7,10,10-hexaphenyl-1,4,7,10-tetraphosphadecane (*P*<sub>4</sub>) has two diastereomeric forms: the chiral *R,R* and *S,S* diastereomer and the *meso*-(*R,S* and *S,R*) form (King, Heckley & Cloyd, 1974). Early attempts to separate the two diastereomers of *P*<sub>4</sub> via reaction of

the ligand mixture with [Mn(CO)<sub>5</sub>X] (*X* = Br, I) failed (Butler & Coville, 1974). Brown & Canning (1984) obtained both diastereomers of *P*<sub>4</sub> in pure form by fractional crystallization of the ligand mixture. Only recently could the two forms of *P*<sub>4</sub> be separated by reaction of commercial *P*<sub>4</sub> with [PtCl<sub>4</sub>]<sup>2-</sup> (Brüggeller, 1989). This reaction led to both diastereomers of [Pt(*P*<sub>4</sub>)](BPh<sub>4</sub>)<sub>2</sub>. In order to distinguish the two complexes, an X-ray structure analysis was performed with one diastereomeric form (1).

*meso*-(1):



chiral (1):



**Experimental.** (1) (Brüggeller, 1989) was recrystallized from CH<sub>3</sub>NO<sub>2</sub> and a crystal with dimensions 0.35 × 0.25 × 0.16 mm was sealed into a capillary. Data collection was with an Enraf–Nonius CAD-4

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