

changes can be attributed to a strong  $\sigma$  Pt—N interaction.

The benzothiazole and acetoxybenzene rings are essentially planar with no atom deviating from the least-squares planes by more than 0.02 (1) Å. The dihedral angles between the PtNBr<sub>3</sub> plane and the benzothiazole and acetoxybenzene planes are 85.7 (1) and 71.3 (3)° respectively. The distances and angles within the tetraethylammonium cation are all normal.

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## Structure of Tris(tri-*p*-methoxyphenylphosphine)mercury(II) Diperchlorate

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**Abstract.** [Hg{(C<sub>7</sub>H<sub>7</sub>O)<sub>3</sub>P<sub>3</sub>}(ClO<sub>4</sub>)<sub>2</sub>],  $M_r = 1456.6$ , triclinic,  $P\bar{1}$ ,  $a = 10.569$  (4),  $b = 16.697$  (4),  $c = 19.631$  (7) Å,  $\alpha = 74.91$  (2),  $\beta = 84.34$  (3),  $\gamma = 71.80$  (2)°,  $V = 3176.9$  Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.51$ ,  $D_x = 1.523$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 2.645$  mm<sup>-1</sup>,  $F(000) = 1472$ ,  $T = 296$  K, final  $R = 0.041$  for 4943 observed reflections. The unit cell contains ClO<sub>4</sub><sup>-</sup> anions and [Hg(PR<sub>3</sub>)<sub>3</sub>]<sup>2+</sup> cations. Hg has a trigonal planar coordination [P—Hg—P = 126.7 (1), 113.1 (1), 115.5 (1)°] with a small pyramidal distortion.

**Introduction.** Crystal structures of HgX<sub>2</sub> adducts have been determined for various substituted phosphines, but

X is generally a soft ligand (halide, SCN, CN) occupying a site in the metal coordination sphere (Podlahová, Kratochvil, Loub & Paulus, 1986; Allen, Bell, Fong, March & Nowell, 1985; Buergi, Fischer, Kunz, Parvez & Pregosin, 1982; Alyea, Dias, Ferguson & Khan, 1979a; Makhija, Rivest & Beauchamp, 1979; Aurivillius & Wendel, 1976). Even harder donors like nitrate (Whitlow, 1974; Alyea, Dias, Ferguson & Restivo, 1977; Alyea, Dias, Ferguson & Parvez, 1979; Buergi *et al.*, 1982; Alyea, Dias, Ferguson & Siew, 1983) and carboxylates (Roberts, Ferguson, Goel, Ogin & Restivo, 1978; Alyea, Dias, Ferguson & Khan, 1979b; Alyea, Dias, Ferguson & Siew, Khan & Roberts, 1979; Alyea, Dias, Ferguson & Siew,

Table 1. Refined coordinates ( $\times 10^4$ , Hg  $\times 10^5$ ) and equivalent isotropic temperature factors ( $\text{\AA}^2 \times 10^3$ )

$$U_{eq} = (\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j) / 3.$$

	x	y	z	$U_{eq}$		x	y	z	$U_{eq}$
Hg	23309 (4)	34710 (3)	26869 (2)	46	C(41)	417 (8)	5054 (6)	1300 (4)	44
Cl(1)	5992 (3)	3846 (2)	2637 (2)	81	C(42)	-712 (10)	5189 (7)	1733 (5)	63
Cl(2)	2776 (3)	8543 (2)	1890 (2)	81	C(43)	-1630 (10)	5990 (8)	1668 (6)	77
P(1)	1574 (2)	4270 (2)	3639 (1)	56	C(44)	-1415 (11)	6687 (7)	1169 (6)	85
P(2)	1623 (3)	4003 (2)	1428 (1)	61	C(45)	-275 (13)	6577 (7)	757 (6)	88
P(3)	3082 (3)	1835 (2)	3155 (1)	69	C(46)	649 (10)	5764 (7)	824 (5)	63
O(1)	4654 (13)	4274 (12)	2632 (7)	104	C(47)	-3385 (19)	7705 (13)	1363 (14)	251
O(2)	6547 (19)	3813 (14)	3211 (10)	133	C(51)	3001 (9)	4021 (6)	811 (5)	47
O(3)	6574 (19)	4258 (13)	2086 (12)	143	C(52)	4197 (10)	4037 (7)	1025 (5)	60
O(4)	6166 (16)	3039 (15)	2587 (9)	136	C(53)	5283 (9)	3971 (7)	576 (6)	63
O(5)	3420 (15)	7777 (8)	2271 (6)	211	C(54)	5197 (10)	3905 (7)	-102 (5)	63
O(6)	1915 (12)	8437 (8)	1497 (7)	169	C(55)	4006 (12)	3920 (8)	-332 (5)	87
O(7)	3517 (13)	8907 (9)	1426 (7)	194	C(56)	2902 (10)	3990 (8)	119 (5)	71
O(8)	2128 (14)	9068 (8)	2283 (6)	187	C(57)	6362 (14)	3638 (11)	-1162 (8)	137
O(17)	5301 (7)	3785 (5)	5862 (4)	74	C(61)	880 (9)	3249 (6)	1239 (5)	48
O(27)	-1840 (6)	2235 (4)	5384 (3)	66	C(62)	-476 (10)	3403 (6)	1190 (6)	62
O(37)	-1670 (7)	7945 (4)	2666 (4)	82	C(63)	-951 (10)	2778 (7)	1086 (7)	78
O(47)	-2198 (10)	7515 (6)	1050 (6)	151	C(64)	-141 (11)	1989 (7)	1038 (6)	73
O(57)	6355 (7)	3789 (5)	-487 (4)	89	C(65)	1215 (11)	1808 (7)	1090 (6)	72
O(67)	-738 (8)	1406 (5)	948 (5)	111	C(66)	1699 (9)	2443 (7)	1183 (5)	59
O(77)	-1449 (10)	384 (7)	3518 (6)	147	C(67)	55 (18)	576 (10)	915 (13)	198
O(87)	5584 (7)	1154 (5)	5904 (4)	79	C(71)	1701 (9)	1405 (6)	3293 (5)	52
O(97)	6411 (10)	541 (6)	829 (5)	125	C(72)	1759 (11)	619 (7)	3733 (7)	82
C(11)	2804 (9)	4237 (6)	4215 (5)	42	C(73)	729 (12)	269 (7)	3819 (7)	92
C(12)	3982 (9)	3548 (6)	4339 (5)	51	C(74)	-395 (12)	697 (8)	3439 (7)	87
C(13)	4800 (10)	3431 (6)	4879 (5)	58	C(75)	-502 (11)	1503 (8)	3003 (6)	85
C(14)	4451 (9)	3999 (6)	5319 (5)	53	C(76)	560 (11)	1833 (7)	2919 (5)	74
C(15)	3353 (9)	4718 (6)	5189 (5)	51	C(77)*	-1916 (32)	293 (25)	3075 (20)	161
C(16)	2544 (9)	4820 (6)	4642 (5)	51	C(77)*	-1534 (24)	-352 (14)	3844 (18)	125
C(17)	4970 (12)	4261 (8)	6388 (6)	83	C(81)	3859 (10)	1526 (6)	4000 (5)	54
C(21)	499 (8)	3705 (5)	4189 (4)	39	C(82)	3068 (10)	1632 (7)	4598 (5)	66
C(22)	595 (9)	3460 (6)	4914 (5)	48	C(83)	3597 (10)	1506 (7)	5232 (5)	67
C(23)	-172 (9)	2970 (6)	5337 (5)	48	C(84)	4952 (10)	1267 (6)	5302 (5)	62
C(24)	-1068 (9)	2726 (6)	5022 (5)	49	C(85)	5750 (10)	1148 (7)	4721 (6)	74
C(25)	-1169 (10)	2973 (7)	4293 (5)	61	C(86)	5218 (10)	1272 (7)	4076 (6)	70
C(26)	-413 (10)	3461 (6)	3892 (5)	56	C(87)	4810 (13)	1424 (8)	6481 (6)	93
C(27)	-1930 (11)	2090 (8)	6124 (6)	83	C(91)	4209 (9)	1357 (6)	2508 (5)	56
C(31)	584 (8)	5391 (5)	3332 (4)	37	C(92)	5083 (11)	1761 (7)	2140 (6)	71
C(32)	-734 (9)	5691 (6)	3553 (5)	48	C(93)	5827 (12)	1485 (8)	1594 (7)	91
C(33)	-1445 (9)	6538 (6)	3318 (5)	53	C(94)	5691 (11)	792 (7)	1395 (6)	75
C(34)	-891 (9)	7117 (6)	2864 (5)	56	C(95)	4811 (12)	379 (7)	1757 (7)	81
C(35)	427 (10)	6831 (6)	2624 (5)	56	C(96)	4051 (10)	668 (6)	2301 (6)	69
C(36)	1160 (9)	5971 (6)	2868 (5)	54	C(97)*	6706 (27)	1058 (19)	261 (12)	104
C(37)	-1202 (13)	8550 (8)	2150 (8)	109	C(97)*	6166 (42)	130 (28)	479 (21)	199

\* Half occupancy.

1984) can satisfy the Lewis acidity of Hg and prevent the formation of complexes with a ligand:metal ratio greater than two, although the bulky phosphines used in many cases probably contribute to keeping the phosphine content low. The only crystal structure known for a complex containing more than two phosphines per Hg appears to be that of an ionic compound obtained by Cotton, Duraj & Roth (1985), containing discrete  $[\text{Cl}_5\text{Ta}-\text{O}-\text{TaCl}_5]^{2-}$  anions and tetrahedral  $[\text{Hg}(\text{PPhMe}_2)_4]^{2+}$  cations.

By using the  $\text{ClO}_4^-$  salt, whose hardness and poor basicity are well known, a 1:3  $\text{Hg}(\text{ClO}_4)_2$  adduct was isolated with tri-*p*-methoxyphenylphosphine (Allman & Goel, 1984). Its structure was investigated in order to characterize the Hg coordination sphere in this phosphine-rich compound.

**Experimental.** Compound prepared as previously described (Allman & Goel, 1984). Crystals grown from ethanol,  $0.178 (010-0\bar{1}0) \times 0.084 (001-00\bar{1}) \times 0.38 (110-\bar{1}\bar{1}0)$  mm, density by flotation in bromoform/bromobenzene. Space group determined from precession and cone-axis photographs. Enraf-Nonius

CAD-4 diffractometer, accurate cell dimensions from 25 centered reflections ( $10 < \theta < 15^\circ$ ), detailed procedure for data collection described elsewhere (Bélanger-Gariépy & Beauchamp, 1980),  $\text{Mo K}\alpha$  graphite-monochromatized radiation,  $2\theta_{\text{max}} = 45^\circ$ ,  $-11 \leq h \leq 11$ ,  $-18 \leq k \leq 18$ ,  $0 \leq l \leq 21$ ; orientation checked every 100 measurements, intensity of three standards checked every hour, max. fluctuation 1.6%, 8265 independent reflections measured, 4943 with  $I > 3.0\sigma(I)$ . Data corrected for Lp and absorption, transmission range 0.60–0.77. Hg located from Patterson map, other atoms from  $\Delta F$  map; structure refined on  $|F|$  isotropically by full-matrix least squares, then anisotropically by block-diagonal least squares. Methyl groups C(77) and C(97) disordered over two equally populated positions. At least one H located on  $\Delta F$  map for each methyl group, used to calculate ideal coordinates for each set of methyl H (C–H = 0.95 Å, isotropic  $B = 7 \text{\AA}^2$ ). Ring H fixed at idealized positions ( $B = 5 \text{\AA}^2$ ). H repositioned after each least-squares cycle. Both  $\text{ClO}_4^-$  ions exhibit high thermal motion, but there is no evidence for disorder over two or more discrete orientations. Final  $R = 0.041$ ,  $wR = 0.045$

(weights based on counting statistics),  $S = 1.41$ , (shift/ $\sigma$ ), mean = 0.04, max. = 0.09. Residual electron density on final  $\Delta F$  map: general background from  $-0.31$  to  $0.30 \text{ e } \text{Å}^{-3}$ , peaks  $\pm 10.6-0.8 \text{ e } \text{Å}^{-3}$  near Hg. Scattering factors from Cromer & Mann (1968), except for H (Stewart, Davidson & Simpson, 1965). Anomalous-dispersion  $f'$  and  $f''$  terms for Hg, Cl and P from Cromer & Liberman (1970). Programs used listed elsewhere (Authier-Martin & Beauchamp, 1977). Refined coordinates in Table 1.\*

\* Lists of observed and calculated structure-factor amplitudes, anisotropic thermal parameters, distances and angles in the  $\text{ClO}_4^-$  ions and aromatic rings, torsion angles for the methoxy groups, and details on least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44992 (49 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

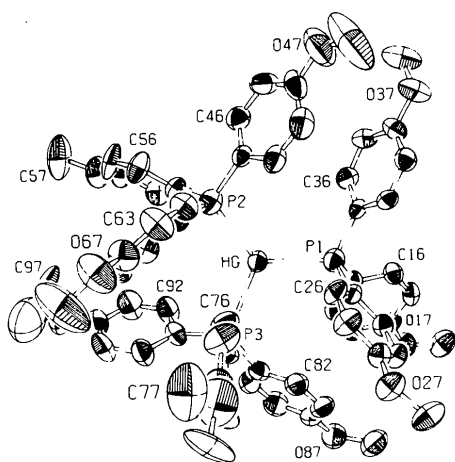


Fig. 1. Structure of the complex cation. The ellipsoids correspond to 50% probability. The 'open' ellipsoids represent the alternative positions of the disordered methyl groups. The first digit in the numeric part of the symbols for aromatic carbons corresponds to the ring number, the second to the position in the ring (1 for P-bonded carbon, sequential numbering, 7 for methyl group).

Table 2. Selected interatomic distances (Å) and bond angles ( $^\circ$ )

Hg—P(1)	2.500 (3)	P(2)—C(41)	1.790 (10)
Hg—P(2)	2.503 (3)	P(2)—C(51)	1.803 (10)
Hg—P(3)	2.534 (3)	P(2)—C(61)	1.805 (10)
Hg—O(1)	3.125 (16)	P(3)—C(71)	1.787 (11)
P(1)—C(11)	1.785 (10)	P(3)—C(81)	1.803 (10)
P(1)—C(21)	1.801 (9)	P(3)—C(91)	1.818 (10)
P(1)—C(31)	1.810 (9)		
P(1)—Hg—P(2)	126.7 (1)	Hg—P(2)—C(41)	109.7 (3)
P(1)—Hg—P(3)	113.1 (1)	Hg—P(2)—C(51)	113.0 (3)
P(2)—Hg—P(3)	115.5 (1)	Hg—P(2)—C(61)	108.1 (3)
P(1)—Hg—O(1)	79.8 (3)	C(41)—P(2)—C(51)	110.9 (4)
P(2)—Hg—O(1)	101.0 (3)	C(41)—P(2)—C(61)	109.3 (4)
P(3)—Hg—O(1)	112.0 (3)	C(51)—P(2)—C(61)	105.6 (5)
Hg—P(1)—C(11)	117.7 (3)	Hg—P(3)—C(71)	111.3 (3)
Hg—P(1)—C(21)	103.5 (3)	Hg—P(3)—C(81)	111.3 (3)
Hg—P(1)—C(31)	114.3 (3)	Hg—P(3)—C(91)	108.6 (3)
C(11)—P(1)—C(21)	105.3 (4)	C(71)—P(3)—C(81)	106.1 (5)
C(11)—P(1)—C(31)	108.1 (4)	C(71)—P(3)—C(91)	108.2 (5)
C(21)—P(1)—C(31)	107.0 (4)	C(81)—P(3)—C(91)	111.4 (5)

**Discussion.** The unit cell contains  $\text{ClO}_4^-$  ions and the  $[\text{Hg}\{\text{P}(\text{C}_6\text{H}_4\text{OCH}_3)_3\}_3]^{2+}$  complex cation shown in Fig. 1. The environment of Hg is best described as roughly trigonal planar, with a significant but relatively small pyramidal distortion, displacing Hg  $0.32 \text{ Å}$  out of the plane containing the three P atoms. Deviations of the P—Hg—P angles ( $126.7$ ,  $113.1$  and  $115.5^\circ$ ,  $\sigma = 0.1^\circ$ ) (Table 2) from  $120.0^\circ$  are significant, but again relatively small. The Hg—P bond lengths ( $2.500$ ,  $2.503$  and  $2.534 \text{ Å}$ ,  $\sigma = 0.003 \text{ Å}$ ) are on the high side of the range ( $2.359-2.589 \text{ Å}$ ) found for complexes with phosphines of similar sizes (Makhija *et al.*, 1979; Makhija, Beauchamp & Rivest, 1973; Gagnon & Beauchamp, 1979; Whitlow, 1974; Bell, Dee, Goldstein, McKenna & Nowell, 1983; Buergi *et al.*, 1982; Bell, Goldstein, Jones & Nowell, 1980; Allen *et al.*, 1985). All these complexes contained only one or two phosphines per Hg, and the long bonds in the present case probably reflect mutual steric hindrance between the three phosphines. In the tetrahedral  $[\text{Hg}(\text{PPhMe}_2)_4]^{2+}$  cation, the Hg—P bonds are longer still [ $2.537(10) \text{ Å}$ ] (Cotton *et al.*, 1985).

The packing pattern (Fig. 2) shows that the perchlorate ions containing Cl(2) are surrounded by aromatic rings and well removed from Hg. The  $\text{ClO}_4^-$  ions containing Cl(1) lie closer to the complex cation, in which one side is not well shielded by aromatic rings. Perchlorate oxygen O(1) at  $3.125(16) \text{ Å}$  from Hg could participate in a slight bonding interaction, but  $\text{Hg}\cdots\text{O}$  contacts above  $3.0 \text{ Å}$  correspond to a vanishingly small binding effect. In  $\text{Hg}(\text{ClO}_4)_2[\text{P}(\text{cyclohexyl})_3]_2$  (Alyea, Dias, Ferguson & Khan, 1979b), the two inequivalent anions gave  $\text{Hg}\cdots\text{O}$  contacts of  $2.928(10)$  and  $3.079(11) \text{ Å}$  as the shortest interactions with an otherwise linear two-coordinated  $\text{Hg}(\text{PR}_3)_2$  unit. A much shorter Hg— $\text{OClO}_3$  bond of  $2.73(2) \text{ Å}$  was observed in  $\text{Hg}[\text{P}(o\text{-tolyl})_3]\text{Cl}(\text{ClO}_4)$  (Alyea, Dias, Ferguson & Khan, 1979a). Although Hg in the present structure is displaced from the plane of the three P atoms in the direction of O(1), considering the weakness of the  $\text{Hg}\cdots\text{O}$  contact, the metal is probably best regarded as forming a three-coordinated  $[\text{Hg}(\text{PR}_3)_3]^{2+}$  cation.

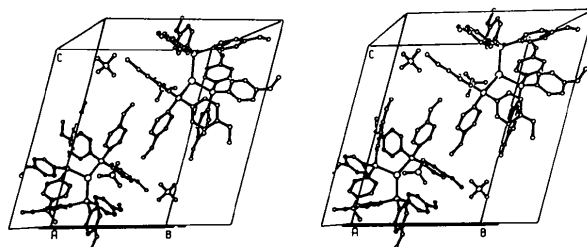


Fig. 2. Stereoview of the unit cell down the  $a^*$  axis. The atoms are represented by spheres of arbitrary size: large (Hg), medium (Cl, P), small (C, O). Hydrogens are omitted. Only one orientation of the disordered C(77) and C(97) methoxy groups is shown.

Details on the geometry of the anions and ligands are provided in the supplementary material. Phosphine coordination increases the C–P–C angles (mean 108.0°) compared with the free ligand (101.5°) (Allman, Goel & Beauchamp, 1986), as already pointed out (Makhija *et al.*, 1973). In the uncoordinated phosphine, the O–CH<sub>3</sub> bond is almost perfectly eclipsed with respect to the adjacent ring (C–C–O–C torsion 5°). In the present case, this orientation is observed for six of the nine independent aromatic rings, although the deviations reach 20° (supplementary material). On the other hand, ring number 8 shows a *gauche* orientation [67 (5)°]. For the two remaining rings (numbers 7 and 9), the methyl groups are involved in a twofold disorder ('open' atoms in Fig. 1). In both cases, one orientation is roughly eclipsed, the other is *gauche*. The mean ring–O and O–CH<sub>3</sub> distances are 1.357 (15) and 1.396 (15) Å, respectively, and the ring–O–CH<sub>3</sub> angle is 119.0 (9)°. Similar values were reported by Nyburg & Faerman (1986).

The perchlorate oxygens have high thermal motion, which leads to Cl–O distances [mean 1.322 (15) Å, range 1.296–1.371 Å,  $\sigma = 0.018$  Å (supplementary material)] artificially shorter than normal. They have the expected tetrahedral geometry (O–Cl–O angles ranging from 103.1 to 114.0°,  $\sigma = 0.9^\circ$ ).

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## N(8)-Coordinating Allopurinol: Structure of Bis(allopurinol)diaquadichlorometal(II) (Metal = Co, Ni)

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**Abstract.** [CoCl<sub>2</sub>(C<sub>5</sub>H<sub>4</sub>N<sub>4</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>],  $M_r = 438.10$ , triclinic,  $P\bar{1}$ ,  $a = 6.459$  (2),  $b = 6.538$  (1),  $c =$

$9.631$  (1) Å,  $\alpha = 105.50$  (1),  $\beta = 96.60$  (2),  $\gamma = 105.56$  (2)°,  $V = 369.8$  (3) Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 1.97$ ,  $D_m = 1.97$  g cm<sup>-3</sup>. [NiCl<sub>2</sub>(C<sub>5</sub>H<sub>4</sub>N<sub>4</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>],  $M_r = 437.86$ , triclinic,  $P\bar{1}$ ,  $a = 6.438$  (4),  $b = 6.516$  (1),

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