

Distances and angles at the K^+ ion are given in Table 3.

The arrangement of the water molecules in space group *Pbcn* requires half-atom positions $Ow(3)$ and $Ow(4)$ for one water molecule, which cause a few forbidden $O \cdots O$ distances of less than 2.3 Å. Besides this, many possible hydrogen bonds can be recognized which determine the three-dimensional arrangement of the structure. Possibly a reduction of the symmetry may lead to an ordered structure of the water molecules.

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Bis(trichlorovinyl)mercury–3,4,7,8-Tetramethyl-1,10-phenanthroline

BY N. A. BELL AND I. W. NOWELL

Department of Chemistry, Sheffield City Polytechnic, Pond Street, Sheffield S1 1WB, England

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Abstract. $[Hg(C_2Cl_3)_2] \cdot C_{16}H_{16}N_2 \cdot C_4Cl_6Hg \cdot C_{16}H_{16}N_2$, $M_r = 697.68$, monoclinic, $C2/c$, $a = 18.976$ (6), $b = 13.876$ (4), $c = 18.401$ (5) Å, $\beta = 107.32$ (6)°, $U = 4625.5$ Å³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $Z = 8$, $D_m = 1.98$ (2), $D_c = 2.00$ Mg m⁻³, $\mu = 7.06$ mm⁻¹, $F(000) = 2656$. The structure was refined to $R = 0.052$ for 4158 counter reflections. There is a weak but significant donor–acceptor interaction between the 3,4,7,8-tetramethyl-1,10-phenanthroline ligand and bis(trichlorovinyl)mercury. Long Hg–N distances [2.687 (6), 2.629 (9) Å] are accompanied by a small N–Hg–N angle of 61.4 (2)° and distortion of the C–Hg–C skeleton from linearity [164.8 (3)°].

Introduction. In contrast to dialkylmercurials for which no addition complexes have been isolated, dialkynylmercurials give rise to stable adducts with, for example, nitrogenous bases (Cano-Esquivel, Santos-Macias & Ballester-Reventos, 1977). Dialkenylmercurials might also be expected to form stable complexes and while the coordination chemistry of the dialkenylmercurials themselves has not been reported, bis(trichlorovinyl)mercury is known (Bell, Nowell & Reynolds, 1979) to form isolable adducts of the type $[Hg(Cl_2C=CCl)_2] \cdot L$ [$L = 1,10$ -phenanthroline (phen); 2,9-dimethyl-1,10-phenanthroline; 3,4,7,8-tetramethyl-1,10-phenanthroline (tmp); 2,2'-bipyridyl; 1,2-bis(diphenyl-

phosphino)ethane]. To investigate the strength and nature of the donor–acceptor interactions in these complexes, the crystal structure of $[Hg(Cl_2C=CCl)_2] \cdot tmp$ has been determined.

A crystal $0.33 \times 0.36 \times 0.16$ mm was mounted with b coincident with the rotation (ω) axis of a Stöe Stadi 2 two-circle diffractometer. With monochromated Mo $K\alpha$ radiation and the background- ω scan-background technique, 6154 unique reflections were measured of which 4158 had $I > 3\sigma(I)$ and were considered to be observed. [The net intensity $I = T - B$, where $T =$ scan count, $B =$ mean background count over the scan width; $\sigma(I) = (T + Bc/2t)^{1/2}$, where $c =$ scan time, $t =$ time for background measurements at each end of the scan.] Corrections for Lorentz, polarization and absorption effects were made. The Hg atom position was determined from a Patterson function and the remaining atoms were located from successive electron-density maps. H atoms were included in calculated positions (C–H = 1.08 Å). Common isotropic temperature factors were applied to methyl and phenyl H atoms and refined to final values of $U = 0.233$ (40) and 0.068 (15) Å² respectively. Scattering factors were calculated with an analytical approximation (*International Tables for X-ray Crystallography*, 1974) and the weighting scheme was $w = 1.0000 [\sigma^2(F_o) + 0.0075(F_o)^2]$. Full-matrix refinement with anisotropic temperature factors for all non-H

Table 1. Fractional coordinates (Hg × 10⁵; remaining atoms × 10⁴)

E.s.d.'s are in parentheses.

	x	y	z
Hg	31303 (2)	57413 (3)	43985 (2)
Cl(1)	1557 (1)	6203 (3)	4748 (2)
Cl(2)	3742 (1)	6393 (2)	6263 (1)
Cl(3)	2309 (2)	6761 (3)	6433 (2)
Cl(4)	3858 (2)	4707 (3)	3170 (2)
Cl(5)	5290 (2)	5972 (4)	3674 (2)
Cl(6)	4748 (2)	6909 (3)	4790 (2)
N(1)	1950 (4)	5355 (7)	3202 (4)
N(2)	2734 (4)	3934 (6)	4100 (4)
C(1)	1741 (4)	4437 (6)	3029 (4)
C(2)	1544 (6)	6049 (8)	2782 (5)
C(3)	920 (5)	5900 (9)	2159 (5)
C(31)	521 (8)	6783 (12)	1752 (7)
C(4)	720 (5)	4959 (9)	1961 (5)
C(41)	62 (6)	4714 (13)	1266 (6)
C(5)	1115 (5)	4199 (7)	2398 (4)
C(6)	950 (5)	3205 (9)	2265 (5)
C(7)	1362 (6)	2503 (9)	2674 (6)
C(8)	1998 (5)	2704 (7)	3307 (5)
C(9)	2457 (6)	1991 (8)	3760 (6)
C(91)	2310 (9)	942 (9)	3557 (8)
C(10)	3039 (5)	2241 (8)	4371 (5)
C(101)	3553 (7)	1566 (10)	4896 (8)
C(11)	3144 (5)	3272 (8)	4522 (5)
C(12)	2170 (4)	3681 (7)	3489 (4)
C(13)	2507 (4)	6162 (8)	5104 (5)
C(14)	2802 (5)	6411 (7)	5824 (5)
C(15)	3967 (5)	5551 (7)	3885 (5)
C(16)	4589 (5)	6046 (9)	4085 (5)
H(2)	1709	6784	2935
H(6)	466	3006	1808
H(7)	1214	1762	2524
H(11)	3586	3497	5012
H(311)	54	6561	1285
H(312)	894	7205	1536
H(313)	332	7213	2146
H(411)	-180	5373	992
H(412)	-344	4316	1448
H(413)	249	4283	870
H(911)	1832	880	3062
H(912)	2208	565	4029
H(913)	2783	630	3434
H(101)	3408	834	4713
H(102)	3513	1662	5465
H(103)	4111	1713	4892

atoms gave $R = 0.052$ and $R' = 0.056$. Atomic parameters are given in Table 1.*

Discussion. Bond lengths and angles are given in Table 2 and mean planes in Table 3. The tmp ligand is weakly coordinated to Hg (Fig. 1), with long Hg–N distances [2.687 (6), 2.629 (9) Å] and a small N–Hg–N angle

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34880 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (Å) and angles (°) with e.s.d.'s in parentheses

Symmetry code: none x, y, z ; (i) $0.5 - x, 1.5 - y, 1.0 - z$; (ii) $1.0 - x, 1.0 - y, 1.0 - z$.

Hg–N(1)	2.687 (6)	C(2)–C(3)	1.397 (12)
Hg–N(2)	2.629 (9)	C(3)–C(31)	1.516 (18)
Hg–C(13)	2.082 (10)	C(3)–C(4)	1.377 (17)
Hg–C(15)	2.091 (10)	C(4)–C(41)	1.536 (13)
Cl(1)–C(13)	1.727 (8)	C(4)–C(5)	1.402 (14)
Cl(2)–C(14)	1.725 (9)	C(5)–C(6)	1.419 (16)
Cl(3)–C(14)	1.729 (11)	C(6)–C(7)	1.333 (15)
Cl(4)–C(15)	1.728 (10)	C(7)–C(8)	1.432 (12)
Cl(5)–C(16)	1.718 (12)	C(8)–C(9)	1.415 (13)
Cl(6)–C(16)	1.725 (12)	C(8)–C(12)	1.412 (14)
N(1)–C(1)	1.343 (12)	C(9)–C(91)	1.507 (16)
N(1)–C(2)	1.327 (13)	C(9)–C(10)	1.366 (12)
N(2)–C(11)	1.300 (12)	C(10)–C(101)	1.483 (15)
N(2)–C(12)	1.347 (9)	C(10)–C(11)	1.459 (16)
C(1)–C(5)	1.431 (10)	C(13)–C(14)	1.322 (12)
C(1)–C(12)	1.438 (11)	C(15)–C(16)	1.320 (14)
Hg...Cl(1)	3.299	Hg...Cl(3 ⁱ)	3.780
Hg...Cl(2)	3.401	Hg...Cl(1 ⁱ)	4.499
Hg...Cl(4)	3.308	Hg...Cl(5 ⁱⁱ)	4.571
Hg...Cl(6)	3.355		
N(1)–Hg–N(2)	61.4 (2)	C(6)–C(7)–C(8)	121.9 (11)
N(1)–Hg–C(13)	94.4 (3)	C(7)–C(8)–C(9)	124.4 (10)
N(1)–Hg–C(15)	99.5 (3)	C(7)–C(8)–C(12)	117.4 (9)
N(2)–Hg–C(13)	102.6 (3)	C(9)–C(8)–C(12)	118.2 (7)
N(2)–Hg–C(15)	89.6 (3)	C(8)–C(9)–C(91)	119.5 (9)
C(13)–Hg–C(15)	164.8 (3)	C(8)–C(9)–C(10)	120.8 (10)
Hg–N(1)–C(1)	119.9 (5)	C(91)–C(9)–C(10)	119.7 (10)
Hg–N(1)–C(2)	122.0 (7)	C(9)–C(10)–C(101)	126.0 (10)
C(1)–N(1)–C(2)	118.1 (7)	C(9)–C(10)–C(11)	115.9 (8)
Hg–N(2)–C(11)	117.8 (6)	C(101)–C(10)–C(11)	118.0 (8)
Hg–N(2)–C(12)	121.9 (6)	N(2)–C(11)–C(10)	123.8 (7)
C(11)–N(2)–C(12)	120.0 (9)	N(2)–C(12)–C(1)	118.1 (8)
N(1)–C(1)–C(5)	121.7 (7)	N(2)–C(12)–C(8)	121.2 (8)
N(1)–C(1)–C(12)	118.5 (6)	C(1)–C(12)–C(8)	120.6 (6)
C(5)–C(1)–C(12)	119.8 (8)	Hg–C(13)–Cl(1)	119.7 (5)
N(1)–C(2)–C(3)	125.0 (10)	Hg–C(13)–C(14)	123.3 (6)
C(2)–C(3)–C(31)	117.5 (10)	Cl(1)–C(13)–C(14)	117.0 (8)
C(2)–C(3)–C(4)	117.2 (9)	Cl(2)–C(14)–Cl(3)	112.6 (5)
C(31)–C(3)–C(4)	125.3 (8)	Cl(2)–C(14)–C(13)	122.4 (8)
C(3)–C(4)–C(41)	121.5 (10)	Cl(3)–C(14)–C(13)	125.0 (7)
C(3)–C(4)–C(5)	120.2 (8)	Hg–C(15)–Cl(4)	119.7 (5)
C(41)–C(4)–C(5)	118.3 (11)	Hg–C(15)–C(16)	123.0 (8)
C(1)–C(5)–C(4)	117.7 (9)	Cl(4)–C(15)–C(16)	117.3 (8)
C(1)–C(5)–C(6)	116.9 (8)	Cl(5)–C(16)–Cl(6)	112.8 (6)
C(4)–C(5)–C(6)	125.4 (8)	Cl(5)–C(16)–C(15)	126.3 (9)
C(5)–C(6)–C(7)	123.3 (8)	Cl(6)–C(16)–C(15)	120.9 (9)

of 61.4 (2)°. The C–Hg–C skeleton is significantly distorted from linearity [164.8 (3)°] and although the Hg–C distances are longer in the present complex (2.087 Å average), the remaining N₂HgC₂ geometry is similar to that found in [Hg(C₆H₅)(CN)].phen [Hg–C 2.063 (14), 2.067 (12); Hg–N 2.660 (8), 2.680 (9) Å; C–Hg–C 167.5 (4); N–Hg–N 61.8 (2)° (Ruiz-Amil, Martínez-Carrera & García-Blanco, 1978)] and [Hg{(C₆H₅)C≡C}₂].phen [Hg–C 2.03, 2.05 (1); Hg–N 2.67, 2.69 (1) Å; C–Hg–C 165.6 (4); N–Hg–N 61.2 (4)° (Gutiérrez-Puebla, Vegas & García-Blanco, 1978)].

The phenanthroline ring system is almost planar with the mean planes associated with the outer fused rings inclined at an angle of 3.35° to each other. The phenanthroline molecule is inclined at an angle of 78.00°

Table 3. Equations of least-squares planes referred to orthogonal axes with distances (Å) of atoms from the planes in square brackets

Plane A: N(1), C(1), C(2), C(3), C(4), C(5)

$$0.7989X - 0.0286Y - 0.6008Z - 5.7285 = 0.0000$$

[N(1), 0.013 (8); C(1), -0.009 (8); C(2), -0.003 (11); C(3), -0.013 (10); C(4), 0.017 (10); C(5), -0.006 (9); C(31), -0.045 (15); C(41), 0.067 (11); N(2), -0.082 (8); Hg, 0.001 (4)]

Plane B: N(2), C(8), C(9), C(10), C(11), C(12)

$$0.8126X + 0.0252Y - 0.5823Z - 5.8724 = 0.0000$$

[N(2), -0.002 (8); C(8), -0.016 (10); C(9), 0.002 (11); C(10), 0.011 (10); C(11), -0.012 (10); C(12), 0.016 (8); C(91), 0.037 (17); C(101), 0.010 (14); N(1), 0.157 (8); Hg, 0.234 (0)]

Plane C: C(1), C(5), C(6), C(7), C(8), C(12)

$$0.7961X - 0.0146Y - 0.6050Z - 5.6490 = 0.0000$$

[C(1), 0.007 (8); C(5), 0.012 (9); C(6), -0.018 (10); C(7), 0.006 (11); C(8), 0.014 (10); C(12), -0.019 (8); N(1), 0.045 (8); N(2), -0.088 (8); Hg, 0.027 (0)]

Plane D: Hg, C(13), C(15)

$$-0.1742X + 0.7873Y - 0.5914Z + 6.0276 = 0.0000$$

[N(1), 1.097 (9); N(2), -1.562 (8); C(6), -0.037 (11)]

Plane E: Cl(1), Cl(2), Cl(3), C(13), C(14)

$$0.1425X + 0.9516Y - 0.2724Z - 0.7223 = 0.0000$$

[Cl(1), -0.004 (4); Cl(2), -0.005 (3); Cl(3), -0.001 (4); C(13), 0.001 (11); C(14), 0.009 (10); Hg, 0.007 (0)]

Plane F: Cl(4), Cl(5), Cl(6), C(15), C(16)

$$-0.2517X + 0.6951Y - 0.6734Z + 7.8236 = 0.0000$$

[Cl(4), -0.003 (4); Cl(5), 0.006 (5); Cl(6), 0.003 (4); C(15), 0.012 (9); C(16), -0.017 (11); Hg, 0.058 (0)]

Angles between planes (°), e.s.d.'s 1.1–1.7°

A-B	3.35	B-C	2.79	C-E	74.67
A-C	0.85	B-D	77.14	C-F	78.65
A-D	78.84	B-E	72.64	D-E	27.69
A-E	75.50	B-F	78.17	D-F	8.36
A-F	79.42	C-D	78.02	E-F	36.00

(mean values for the three rings) to the plane containing Hg, C(13), C(15) and at angles of 74.27 and 78.75° to the trichlorovinyl groups. The latter are effectively planar and twisted with respect to each other by 36.0°. The α -Cl and two of the four β -Cl atoms lie at average distances of 3.304 and 3.378 Å respectively from Hg. While both of these values are within the upper limit

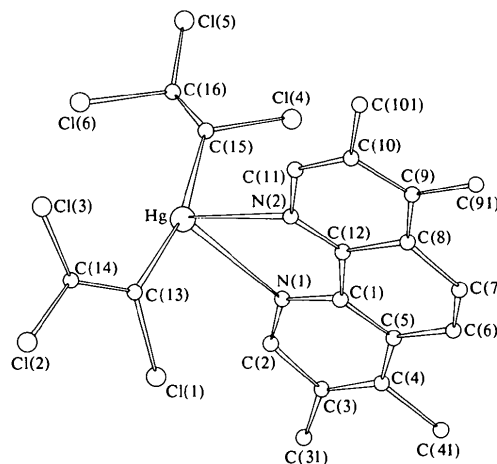


Fig. 1. Structure and atom-numbering scheme for [Hg(Cl₂C=CCl₂)].tmp. H atoms have been omitted for clarity.

suggested by Grdenić (1965) for the sum of the van der Waals radii (3.53 Å), bond distances and angles within the trichlorovinyl groups are as expected and it is difficult to evaluate the importance of intramolecular Hg...Cl interactions within the present complex. Intermolecular Hg...Cl interactions appear to be of little significance, the shortest intermolecular Hg...Cl contact being 3.78 Å.

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