

The Mo—N distances 2.156 (3) and 2.169 (2) Å are in the range observed for other molybdenum complexes, such as [Mo(H₂NO)₂(NO)(bpy)], (Mo—N)_{av} = 2.189 Å (Weighardt, Holzback, Weiss, Nuker & Prikner, 1979) and [Mo(OPrⁱ)₂(bpy)₂], (Mo—N)_{av} = 2.118 Å (Chisholm, Huffman, Rothwell, Bradley, Kress & Woodruff, 1981). The bipyridine ligand parameters in [MoCl₄(bpy)]⁻ do not vary significantly from those in the free ligand, which contains *trans*-nitrogen atoms.

The cation is found as a regular tetrahedron with an average P—C(phenyl) of 1.796 (2) Å. In the phenyl rings the average C—C distance is 1.383 (10) Å in a range 1.360–1.395 Å. Two close distances are to be noted in this structure arising from C(8) and C(9) across the centre of symmetry. They are C(8)—C(8) 3.37 and C(8)—C(9) 3.38 Å.

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Structure of a Dipotassium Tetracyanomercurate(II) Salt with Dibenzo-18-crown-6

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(Received 12 May 1987; accepted 15 June 1987)

Abstract. Bis[(dibenzo[*b,k*][1,4,7,10,13,16]hexaoxacyclooctadeca-2,11-diene)potassium] tetracyanomercurate(II) dihydrate, [K(C₂₀H₂₄O₆)₂][Hg(CN)₄].2H₂O, *M_r* = 1139.7, triclinic, *P* $\bar{1}$, *a* = 14.050 (4), *b* = 14.111 (4), *c* = 14.750 (4) Å, α = 99.49 (2), β = 100.98 (2), γ = 115.87 (2)°, *U* = 2479 (1) Å³, *Z* = 2, *D_m* = 1.50 (3), *D_x* = 1.53 g cm⁻³, λ (Mo *K* α) = 0.7107 Å, μ = 33.27 cm⁻¹, *F*(000) = 1148, room temperature, final *R* = 0.048 for 6479 [*I* > 3 σ (*I*)] independent observed reflections. The structure of the molecule essentially consists of two moieties: one has the tetrahedral tetracyanomercurate(II) anion linked through a cyanide group to a potassium-crown-ether cation [K...N 2.759 (8) Å] while the other is an independent (dibenzo-18-crown-6)potassium complex cation in which the central metal ion is also seven-

coordinate (six O atoms from the macrocycle and the seventh from a water molecule). The average K...O distances are 2.741 and 2.759 Å respectively, excluding the distance to the water molecule for the second fragment which is 2.672 (7) Å.

Introduction. It is well known that the K ion is strongly coordinated to the six oxygen atoms of dibenzo-18-crown-6 and forms the most stable complex amongst Group Ia cations (Izatt, Eatough & Christensen, 1973). Although many compounds containing the [K-DB18C6]⁺ cation have been prepared and characterized there have been relatively few structure reports except for the iodide (Aldoshin, D'yachenko, Tkachev & Atovmyen, 1981; Hilgenfeld & Saenger, 1981), a series of aluminates (Atwood, Hunter, Rogers &

Weeks, 1985; Zaworotko, Reid & Atwood, 1985), a complex chromium anion (Wink, Fox & Cooper, 1985) and a thiocyanatocobaltate(II) (Fan, Zhang, Wang, Zhang & Han, 1985). We report here the structure of the tetracyanomercurate(II).

Experimental. Compound prepared by reacting an aqueous alcohol solution of dipotassium tetracyanomercurate(II) with a solution of dibenzo-18-crown-6 in chloroform. Density by flotation in CCl₄/petroleum ether. Crystal size approximately 0.3 × 0.3 × 0.5 mm. Nicolet R3m four-circle diffractometer using graphite-monochromated Mo K α radiation; cell dimensions derived by least-squares calculations from angular settings of 25 reflections measured at 5 < 2 θ < 35°; ω -scan mode with scan rate between 2.93 and 29.30° min⁻¹. 9410 reflections measured, 2 θ _{max} = 50°, h -16 to 16, k -16 to 16, l 0 to 16; data reduction gave 9119 unique reflections; statistical analysis suggested the space group $P\bar{1}$. Three standards measured for every 97 reflections; slight degradation in intensity observed initially. Empirical absorption correction applied using *SHELXTL* Revision 4.1 (Sheldrick, 1984), max. and min. transmission factors 0.893, 0.530. Mercury atom located by direct methods and all non-H atoms from difference Fourier maps. 6479 data with $I > 3\sigma(I)$ used in subsequent calculations. All non-H atoms refined anisotropically. H atoms placed at fixed distance (0.96 Å) from C or O atoms; their thermal parameters were assigned the final isotropic thermal parameters of their carrier atoms and were not refined. Weighting scheme $w = [\sigma^2(F) + 0.002F^2]^{-1}$. Final $R(F) = 0.048$, $wR = 0.051$, $S = 1.01$ and mean shift/e.s.d. < 0.008. In final difference map, maximum and minimum heights 0.68 and -1.00 e Å⁻³. Calculations and refinements performed on Nova 4X computer; final refinement on Data General 30 computer using *SHELXTL* Revision 5.1 (Sheldrick, 1985). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic coordinates are given in Table 1, details of selected interatomic distances and angles in Table 2.*

Discussion. The structure of the molecule is shown in Fig. 1 and a packing diagram in Fig. 2. The tetracyanomercurate ion is almost a regular tetrahedron with C-Hg-C angles in the range of 106.4 (3) to 115.0 (3)° for an average of 109.4 (3)°. The Hg-C-N atoms are nearly collinear and the angles are within 2° from linearity except for the Hg-C(1)-N(1) angle at 173.3 (5)°. There is no systematic variation in

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

	x	y	z	U_{eq} *
Hg	6383.6 (2)	2064.8 (2)	3558.8 (2)	68 (1)
C(1)	6570 (6)	1494 (5)	4835 (5)	74 (4)
N(1)	6732 (6)	1303 (5)	5539 (5)	90 (4)
C(2)	5297 (6)	787 (6)	2238 (5)	72 (3)
N(2)	4750 (7)	104 (6)	1573 (5)	112 (4)
C(3)	8051 (6)	3015 (5)	3459 (4)	69 (3)
N(3)	8933 (5)	3504 (5)	3415 (5)	91 (4)
C(4)	5729 (6)	3216 (5)	3836 (5)	75 (3)
N(4)	5400 (6)	3790 (6)	3995 (5)	106 (4)
K(1)	9806 (1)	3385 (1)	1902 (1)	72 (1)
K(2)	7094 (1)	7283 (1)	3679 (1)	78 (1)
O(11)	8398 (3)	3793 (3)	664 (3)	72 (2)
C(12)	7428 (5)	2843 (6)	25 (5)	80 (3)
C(13)	7155 (5)	1940 (6)	492 (5)	80 (3)
O(14)	7993 (4)	1638 (3)	603 (3)	74 (2)
C(15)	7762 (6)	782 (6)	1029 (6)	88 (4)
C(16)	8630 (7)	438 (6)	1033 (5)	94 (4)
O(17)	9648 (4)	1328 (4)	1619 (3)	87 (3)
C(18)	10561 (7)	1162 (7)	1720 (5)	88 (5)
C(19)	10570 (9)	233 (9)	1325 (6)	113 (7)
C(20)	11576 (13)	217 (13)	1507 (8)	143 (10)
C(21)	12512 (11)	1069 (12)	2032 (8)	135 (10)
C(22)	12549 (8)	2042 (10)	2457 (7)	128 (7)
C(23)	11549 (8)	2079 (8)	2287 (6)	96 (6)
O(24)	11494 (4)	3010 (5)	2700 (4)	96 (3)
C(25)	12443 (7)	3902 (8)	3350 (6)	119 (6)
C(26)	12136 (7)	4723 (7)	3773 (5)	119 (5)
O(27)	11760 (4)	5137 (4)	3029 (3)	89 (3)
C(28)	11418 (7)	5892 (6)	3379 (5)	100 (4)
C(29)	11092 (6)	6334 (6)	2615 (5)	90 (4)
O(30)	10106 (4)	5451 (4)	1898 (3)	76 (2)
C(31)	9646 (5)	5682 (5)	1118 (5)	68 (3)
C(32)	10030 (7)	6690 (7)	945 (6)	96 (5)
C(33)	9480 (8)	6813 (8)	133 (6)	101 (5)
C(34)	8572 (8)	5960 (9)	-506 (6)	101 (6)
C(35)	8179 (7)	4907 (7)	-385 (5)	86 (4)
C(36)	8719 (5)	4792 (6)	442 (4)	69 (3)
O(41)	5679 (4)	7466 (4)	2215 (3)	76 (2)
C(42)	5177 (6)	8107 (7)	2510 (5)	89 (4)
C(43)	5141 (6)	8099 (7)	3509 (6)	92 (4)
O(44)	6257 (4)	8630 (4)	4112 (3)	82 (3)
C(45)	6346 (6)	8780 (6)	5095 (5)	78 (4)
C(46)	7537 (7)	9440 (6)	5643 (5)	84 (4)
O(47)	8085 (4)	8818 (4)	5442 (3)	81 (3)
C(48)	9212 (6)	9313 (6)	5873 (4)	75 (4)
C(49)	9815 (7)	10315 (6)	6520 (5)	90 (4)
C(50)	10941 (8)	10746 (8)	6932 (6)	110 (5)
C(51)	11441 (7)	10167 (7)	6684 (6)	115 (5)
C(52)	10849 (7)	9108 (7)	6018 (6)	99 (5)
C(53)	9718 (6)	8702 (7)	5619 (5)	78 (4)
O(54)	9033 (4)	7670 (4)	4954 (3)	83 (3)
C(55)	9538 (7)	7059 (7)	4602 (6)	93 (5)
C(56)	8646 (7)	6004 (7)	3897 (6)	92 (5)
O(57)	8193 (4)	6228 (4)	3060 (3)	81 (3)
C(58)	7375 (7)	5270 (6)	2338 (5)	85 (4)
C(59)	7037 (7)	5584 (6)	1472 (5)	87 (4)
O(60)	6487 (4)	6203 (4)	1698 (3)	80 (3)
C(61)	6162 (5)	6631 (5)	1001 (4)	70 (3)
C(62)	6272 (7)	6463 (7)	106 (5)	96 (5)
C(63)	5974 (8)	6979 (7)	-540 (6)	107 (5)
C(64)	5573 (7)	7688 (7)	-242 (6)	98 (5)
C(65)	5479 (5)	7856 (6)	654 (5)	78 (4)
C(66)	5765 (5)	7351 (5)	1293 (5)	69 (3)
O(71)	5528 (6)	5872 (5)	4290 (5)	117 (4)
O(72)	2884 (5)	8664 (4)	2418 (4)	107 (3)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

the Hg-C distances which range from 2.164 (6) to 2.209 (9) Å for an average of 2.184 (8) Å. However, the C-N distances appear to reflect which N atom is coordinated to the K ion; the C(3)-N(3) bond length is 1.147 (10) Å compared to the range from 1.109 (13) to 1.122 (11) Å with an average of 1.114 (11) Å for the other three C-N distances. The observed bond angles and distances are in general agreement with those

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44150 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

reported for this ion in other compounds (Brodersen, Beck, Beck, Hummel & Liehr, 1984; Kluefers, Fuess & Haussuehl, 1981).

Table 2. Bond lengths (Å) and angles (°)

Hg—C(1)	2.181 (8)	Hg—C(2)	2.164 (6)
Hg—C(3)	2.182 (8)	Hg—C(4)	2.209 (9)
C(1)—N(1)	1.122 (11)	C(2)—N(2)	1.112 (8)
C(3)—N(3)	1.147 (10)	N(3)—K(1)	2.759 (8)
C(4)—N(4)	1.109 (13)	K(1)—O(11)	2.747 (5)
K(1)—O(24)	2.744 (7)	K(1)—O(14)	2.709 (3)
K(1)—O(27)	2.720 (4)	K(1)—O(17)	2.766 (7)
K(1)—O(30)	2.759 (5)	K(2)—O(41)	2.771 (6)
K(2)—O(54)	2.769 (6)	K(2)—O(44)	2.691 (7)
K(2)—O(57)	2.744 (7)	K(2)—O(47)	2.743 (4)
K(2)—O(60)	2.835 (5)	K(2)—O(71)	2.672 (7)
O(11)—C(12)	1.430 (6)	O(11)—C(36)	1.397 (9)
O(12)—C(13)	1.485 (12)	C(13)—O(14)	1.409 (11)
O(14)—C(15)	1.394 (10)	C(15)—C(16)	1.496 (15)
C(16)—O(17)	1.405 (7)	O(17)—C(18)	1.387 (14)
C(18)—C(19)	1.351 (17)	C(18)—C(23)	1.391 (10)
C(19)—C(20)	1.397 (24)	C(20)—C(21)	1.304 (16)
C(21)—C(22)	1.386 (23)	C(22)—C(23)	1.405 (19)
C(23)—O(24)	1.395 (14)	O(24)—C(25)	1.390 (8)
C(25)—C(26)	1.489 (17)	C(26)—O(27)	1.444 (12)
O(27)—C(28)	1.411 (13)	C(28)—C(29)	1.468 (13)
C(29)—O(30)	1.446 (6)	O(30)—C(31)	1.368 (9)
C(31)—C(32)	1.373 (12)	C(31)—C(36)	1.390 (7)
C(32)—C(33)	1.375 (14)	C(33)—C(34)	1.339 (10)
O(41)—C(42)	1.431 (12)	C(34)—C(35)	1.398 (15)
C(42)—C(43)	1.485 (12)	C(35)—C(36)	1.382 (12)
O(44)—C(45)	1.404 (9)	O(41)—C(66)	1.378 (9)
C(46)—O(47)	1.429 (13)	C(43)—O(44)	1.426 (8)
C(48)—C(49)	1.353 (9)	C(45)—C(46)	1.483 (10)
C(49)—C(50)	1.384 (13)	O(47)—C(48)	1.380 (9)
C(53)—O(54)	1.399 (8)	C(48)—C(53)	1.384 (14)
C(55)—C(56)	1.494 (9)	C(50)—C(51)	1.339 (18)
O(57)—C(58)	1.409 (7)	C(51)—C(52)	1.418 (11)
C(59)—O(60)	1.435 (13)	O(52)—C(53)	1.392 (11)
C(61)—C(62)	1.351 (11)	O(54)—C(55)	1.429 (13)
C(62)—C(63)	1.397 (15)	C(56)—O(57)	1.427 (11)
C(64)—C(65)	1.346 (12)	C(58)—C(59)	1.482 (12)
		O(60)—C(61)	1.378 (10)
		C(61)—C(66)	1.403 (12)
		C(63)—C(64)	1.399 (17)
		C(65)—C(66)	1.373 (12)
C(1)—Hg—C(2)	115.0 (3)	C(1)—Hg—C(3)	106.4 (3)
C(2)—Hg—C(3)	111.9 (3)	C(1)—Hg—C(4)	107.3 (3)
C(2)—Hg—C(4)	108.8 (3)	C(3)—Hg—C(4)	107.1 (3)
Hg—C(1)—N(1)	173.3 (5)	Hg—C(2)—N(2)	177.4 (9)
Hg—C(3)—N(3)	178.8 (8)	C(3)—N(3)—K(1)	130.7 (5)
Hg—C(4)—N(4)	178.4 (7)	N(3)—K(1)—O(11)	96.4 (2)
N(3)—K(1)—O(14)	98.5 (2)	O(11)—K(1)—O(14)	62.3 (1)
O(11)—K(1)—O(17)	121.7 (1)	N(3)—K(1)—O(17)	102.2 (2)
O(14)—K(1)—O(17)	60.5 (2)	O(11)—K(1)—O(24)	162.8 (2)
N(3)—K(1)—O(24)	100.7 (2)	O(14)—K(1)—O(24)	116.2 (2)
O(11)—K(1)—O(27)	117.3 (2)	O(17)—K(1)—O(24)	56.1 (2)
O(14)—K(1)—O(27)	172.6 (2)	N(3)—K(1)—O(27)	88.9 (2)
O(17)—K(1)—O(27)	117.8 (2)	O(24)—K(1)—O(27)	61.7 (2)
N(3)—K(1)—O(30)	88.2 (2)	O(11)—K(1)—O(30)	55.5 (1)
O(24)—K(1)—O(30)	123.1 (1)	O(14)—K(1)—O(30)	117.8 (2)
O(27)—K(1)—O(30)	62.4 (2)	O(17)—K(1)—O(30)	169.6 (2)
O(41)—K(2)—O(44)	60.8 (2)	O(41)—K(2)—O(47)	120.7 (2)
O(41)—K(2)—O(57)	114.5 (2)	O(44)—K(2)—O(47)	62.0 (2)
O(44)—K(2)—O(57)	168.0 (2)	O(41)—K(2)—O(54)	160.2 (2)
O(47)—K(2)—O(57)	118.2 (2)	O(44)—K(2)—O(54)	118.0 (2)
O(54)—K(2)—O(60)	121.3 (2)	O(47)—K(2)—O(54)	56.4 (2)
O(57)—K(2)—O(60)	60.3 (2)	O(41)—K(2)—O(60)	55.0 (2)
O(41)—K(2)—O(71)	96.8 (2)	O(44)—K(2)—O(60)	115.7 (2)
O(44)—K(2)—O(71)	85.4 (2)	O(47)—K(2)—O(60)	163.6 (2)
O(47)—K(2)—O(71)	92.9 (2)	O(54)—K(2)—O(71)	102.8 (2)
K(1)—O(11)—C(36)	123.4 (3)	O(54)—K(2)—O(57)	62.2 (2)
O(11)—C(12)—C(13)	108.2 (5)	O(57)—K(2)—O(71)	106.4 (2)
K(1)—C(13)—O(14)	50.7 (2)	O(60)—K(2)—O(71)	103.3 (2)
K(1)—O(14)—C(13)	105.6 (3)	K(1)—O(11)—C(12)	115.5 (5)
C(13)—O(14)—C(15)	112.2 (6)	C(12)—O(11)—C(36)	118.4 (5)
C(15)—C(16)—O(17)	107.8 (7)	C(12)—C(13)—O(14)	110.4 (6)
K(1)—O(17)—C(18)	123.2 (4)	K(1)—O(14)—C(15)	104.9 (3)
O(17)—C(18)—C(19)	126.9 (7)	O(14)—C(15)—C(16)	108.8 (7)
C(19)—C(18)—C(23)	119.4 (11)	K(1)—O(17)—C(16)	116.6 (6)
C(19)—C(20)—C(21)	122.8 (17)	C(16)—O(17)—C(18)	116.3 (7)
C(21)—C(22)—C(23)	117.6 (9)	O(17)—C(18)—C(23)	113.7 (10)
C(18)—C(23)—O(24)	117.3 (10)	C(18)—C(19)—C(20)	118.8 (9)
K(1)—O(24)—C(23)	121.5 (4)	C(20)—C(21)—C(22)	120.8 (17)

Table 2 (cont.)

C(23)—O(24)—C(25)	118.9 (8)	C(18)—C(23)—C(22)	120.6 (11)
C(25)—C(26)—O(27)	109.6 (7)	C(22)—C(23)—O(24)	122.1 (7)
K(1)—O(27)—C(28)	101.9 (4)	K(1)—O(24)—C(25)	117.1 (7)
O(27)—C(28)—C(29)	110.7 (7)	O(24)—C(25)—C(26)	108.4 (8)
K(1)—O(30)—C(29)	114.1 (5)	K(1)—O(27)—C(28)	112.3 (6)
C(29)—O(30)—C(31)	118.3 (5)	K(1)—O(27)—C(26)	103.7 (4)
O(30)—C(31)—C(36)	115.5 (6)	C(26)—O(27)—C(28)	112.3 (6)
C(31)—C(32)—C(33)	120.1 (6)	C(28)—C(29)—O(30)	108.3 (6)
C(33)—C(34)—C(35)	120.7 (10)	K(11)—O(30)—C(31)	123.7 (3)
O(11)—C(36)—C(31)	114.8 (6)	O(30)—C(31)—C(32)	126.0 (5)
C(31)—C(36)—C(35)	121.5 (7)	C(31)—C(36)—C(36)	118.5 (7)
K(2)—O(41)—C(66)	122.2 (5)	C(32)—C(33)—C(34)	121.3 (10)
O(41)—C(42)—C(43)	108.3 (8)	C(34)—C(35)—C(36)	117.7 (6)
K(2)—O(44)—C(43)	106.9 (5)	O(11)—C(36)—C(35)	123.7 (5)
C(43)—O(44)—C(45)	113.4 (6)	K(2)—O(41)—C(42)	116.0 (4)
C(45)—C(46)—O(47)	109.3 (6)	C(42)—O(41)—C(66)	117.2 (6)
K(2)—O(47)—C(48)	121.4 (5)	C(42)—C(43)—O(44)	107.4 (6)
O(47)—C(48)—C(49)	124.3 (9)	K(2)—O(44)—C(45)	105.7 (5)
C(49)—C(48)—C(53)	120.1 (7)	O(44)—C(45)—C(46)	107.9 (6)
C(49)—C(50)—C(51)	119.6 (8)	K(2)—O(47)—C(46)	114.3 (4)
C(51)—C(52)—C(53)	116.6 (10)	C(46)—O(47)—C(48)	117.5 (5)
C(48)—C(53)—O(54)	116.5 (6)	O(47)—C(48)—C(53)	115.5 (5)
K(2)—O(54)—C(53)	119.5 (6)	C(48)—C(49)—C(50)	120.7 (10)
C(53)—O(54)—C(55)	118.2 (6)	C(50)—C(51)—C(52)	122.1 (8)
C(55)—C(56)—O(57)	109.2 (7)	C(48)—C(53)—C(52)	120.8 (7)
K(2)—O(57)—C(58)	105.4 (5)	C(52)—C(53)—O(54)	122.7 (9)
O(57)—C(58)—C(59)	108.6 (6)	K(2)—O(54)—C(55)	115.0 (4)
K(2)—O(60)—C(59)	114.8 (4)	O(54)—C(55)—C(56)	108.1 (8)
C(59)—O(60)—C(61)	117.3 (6)	K(2)—O(57)—C(56)	104.1 (5)
O(60)—C(61)—C(66)	115.5 (6)	C(56)—O(57)—C(58)	112.9 (6)
C(61)—C(62)—C(63)	121.0 (10)	C(58)—C(59)—O(60)	108.1 (7)
C(63)—C(64)—C(65)	119.5 (9)	K(2)—O(60)—C(61)	120.9 (4)
O(41)—C(66)—C(61)	115.3 (7)	O(60)—C(61)—C(62)	124.8 (9)
C(61)—C(66)—C(65)	119.0 (7)	C(62)—C(61)—C(66)	119.5 (8)
		C(62)—C(63)—C(64)	118.9 (9)
		C(64)—C(65)—C(66)	122.1 (9)
		O(41)—C(66)—C(65)	125.6 (8)

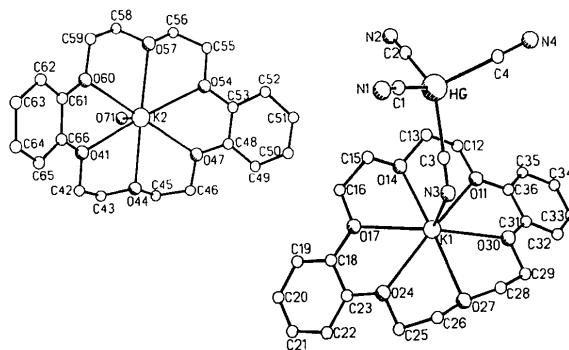


Fig. 1. Diagram showing the two fragments of the molecule; these are unconnected and have been orientated separately for clarity.

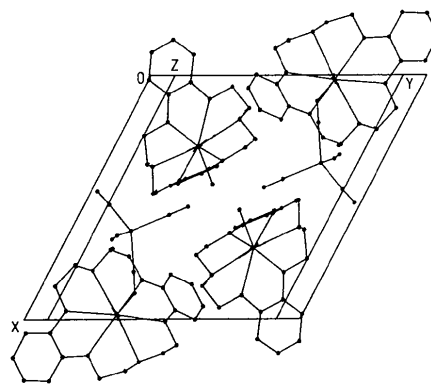


Fig. 2. Packing diagram showing the main components in the unit cell.

A coordination number greater than six is commonly observed for the K ion in crown-ether complexes. Thus both K ions in this molecule are seven coordinated. In addition to the six O atoms from the macrocycle, the seventh coordinating atom is the N atom of a cyanide group in one case and the O atom of a water molecule in the other. The K(1)···N(3) and K(2)···O(71) distances are 2.759 (8) and 2.672 (7) Å respectively.

In both the [K-DB18C6]⁺ ions the K is nearly coplanar with the six O atoms of the crown ether. All the O···K···O angles are close to 60°, ranging from 56.1 (2) to 62.3 (1)° in one case and from 56.4 (2) to 62.0 (2)° in the other. K(1) is 0.28 (1) Å from the O₆ plane while K(2) is -0.38 (1) Å. K···O distances vary from 2.709 (3) to 2.766 (7) Å for an average of 2.741 (6) Å for K(1) while the corresponding values are 2.691 (7), 2.835 (5) and 2.759 (6) Å respectively for K(2). Both the range and the average of the K···O distances are in agreement with values reported elsewhere (Hilgenfeld & Saenger, 1981; Poonia & Bajaj, 1979; Rogers & Atwood, 1984; Wink *et al.*, 1985).

Within the polyether macrocycle the aliphatic C—C distances average 1.484 (14) Å for the K(1) complex ion and 1.487 (11) Å for K(2) with a spread of less than 0.02 Å. These values are slightly shorter than that expected for single C—C bonds but are within the range usually observed in crown-ether complexes (Bush & Truter, 1971). Bond distances and angles within the benzene rings are normal. Average O—C(aliphatic) distances are 1.416 (9) and 1.423 (11) Å for the two macrocycles while average O—C(aromatic) distances are 1.386 (11) and 1.383 (10) Å respectively, again in good agreement with reported values.

The second water molecule was located from the difference Fourier map after all the other non-H atoms were refined anisotropically. It is not bonded to the K atoms.

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Acta Cryst. (1987). **C43**, 2096–2099

Two Crystalline Phases of Chlorotris(2-pyridyl)phosphinegold(I)

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(Received 5 May 1987; accepted 30 June 1987)

Abstract. The title compound, [AuCl(C₅H₄N)₃P], *M_r* = 497.68, crystallizes in two different crystalline phases *A* and *B*. *A* is triclinic, *P* $\bar{1}$, *a* = 13.050 (1), *b* = 14.276 (2), *c* = 11.263 (2) Å, α = 115.86 (1), β = 92.10 (1), γ = 118.80 (1)°, *V* = 1571.5 Å³, *Z* = 4, *D_m* = 2.13 (2), *D_x* = 2.10 g cm⁻³, graphite-monochromated Mo *K* α radiation, λ = 0.71069 Å, μ =

99.4 cm⁻¹, *F*(000) = 966.8, *T* = 298 K, *R* = 0.0584 for 3861 unique reflections and 379 parameters. *B* is triclinic, *P* $\bar{1}$, *a* = 8.611 (2), *b* = 9.118 (2), *c* = 11.186 (3) Å, α = 94.86 (2), β = 112.22 (2), γ = 94.96 (2)°, *V* = 803.4 Å³, *Z* = 2, *D_m* = 2.13 (2), *D_x* = 2.06 g cm⁻³, graphite-monochromated Mo *K* α radiation, λ = 0.71069 Å, μ = 97.4 cm⁻¹, *F*(000) = 483.4,