skewing of the z axis is evident in the pp but not in the nn complex, where both *trans* CO groups are pushed away from the ligating atoms.

The P—C(sp^3) distance of 1·839 (4) Å can be compared with values of 1·85 (Doedens, Robinson & Ibers, 1967), 1·865 (Churchill & O'Brien, 1970), 1·88 (Cheung, Lai & Mok, 1971) and 1·846 (7) and 1·844 (8) Å (Bernal, Reisner, Dobson & Dobson, 1986). The average of 1·834 (6) Å for the two P—C(sp^2) distances is in good agreement with other reported values of 1·827 (Churchill & O'Brien, 1970), 1·83 (Cheung, Lai & Lam, 1970; Doedens, Robinson & Ibers, 1967), 1·828 (Daly, 1964), 1·81 (La Placa & Ibers, 1965) and 1·838 Å (Bernal, Reisner, Dobson & Dobson, 1986). The mean C—C bond length and C—C—C bond angle for the two phenyl groups are 1·377 (7) Å and 118·3 (5)°, respectively.

Least-squares planes were calculated for each of the three rings. The two phenyl rings (rings 1 and 2) are essentially planar, while the pyridine ring (ring 3) has two out-of-plane atoms [N and C(9)]. For ring 1 [C(18)—C(23)], the maximum and minimum deviations are 0·0051 and −0·0064 Å; the maximum and minimum deviations for ring 2 [C(12)—C(17)] are 0·0060 and −0·0066 Å. The dihedral angle between these two rings is 76·54 (7)°. The maximum deviation from planarity for ring 3 is 0·127 and the minimum is −0·0105 Å. The dihedral angle between rings 1 and 3 is 42·27 (7)° and that between rings 2 and 3 is 84·41 (6)°.

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Tris(3-ethylpyridinium) Decavanadate Monohydrate

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Abstract. $[C_7H_{10}N]_3[V_{10}H_3O_{28}].H_2O$, $M_r = 1302·8$, monoclinic, $P2_1/n$, $a = 7·294$ (2), $b = 23·083$ (7), $c = 24·403$ (3) Å, $\beta = 96·92$ (1)°, $V = 4079$ (1) Å^3, $Z = 4$, $D_m = 2·1$ (1) (by flotation in $Br_3CH—CCl_4$), $D_x = 2·12$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $\mu = 1·77$ mm^{-1}, $F(000) = 2576$, room temperature, $R = 0·042$ for 2242 observed reflections. The molecule is very similar to other triply protonated decavanadates recently described. The three protons link the decavanadate anion in layers parallel to (100), and these layers are held together by hydrogen bonding through the water molecule.

Introduction. The structures of several decavanadates have previously been described, for example $K_2Zn_2V_{10}O_{28}.16H_2O$ (Evans, 1966), $Ca_3V_{10}O_{28}.17H_2O$

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Table 1. *Atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and* B_{eq} *values* (Å^2)

	x	y	z	B_{eq}*
V(1)	9637 (4)	2368 (1)	1862 (1)	1·97
V(2)	10151 (4)	3386 (1)	1020 (1)	2·31
V(3)	10009 (4)	2134 (1)	601 (1)	2·19
V(4)	6092 (4)	2944 (1)	2224 (1)	2·07
V(5)	5950 (4)	1658 (1)	1809 (1)	2·03
V(6)	6343 (4)	2722 (1)	898 (1)	1·93
V(7)	6625 (4)	3969 (1)	1342 (1)	2·73
V(8)	6336 (4)	1459 (1)	494 (1)	2·28
V(9)	9751 (4)	3644 (1)	2252 (1)	2·53
V(10)	9510 (4)	1130 (1)	1411 (1)	2·24
O(11)	4844 (13)	2477 (4)	1521 (4)	1·72
O(12)	7864 (14)	1998 (5)	1214 (4)	2·47
O(13)	5228 (15)	2267 (5)	424 (5)	2·80
O(14)	8184 (16)	1754 (5)	150 (4)	2·61
O(15)	8470 (14)	2863 (4)	525 (4)	2·35
O(16)	10564 (14)	1706 (5)	1975 (4)	2·42
O(17)	7962 (13)	3076 (4)	1565 (4)	2·04
O(18)	7786 (15)	3329 (5)	2634 (5)	2·94
O(19)	7520 (14)	1102 (4)	1891 (5)	2·62
O(20)	10999 (14)	2585 (5)	1257 (5)	2·38
O(21)	4518 (15)	1500 (5)	2221 (4)	2·81
O(22)	11376 (15)	2292 (5)	173 (4)	3·03
O(23)	7463 (14)	2226 (5)	2238 (4)	2·11
O(24)	10964 (14)	1471 (5)	939 (4)	2·26
O(25)	7803 (15)	888 (5)	847 (5)	3·05
O(26)	10647 (13)	2806 (5)	2344 (4)	2·44
O(27)	5374 (15)	3378 (5)	790 (5)	2·86
O(28)	11546 (17)	3538 (5)	602 (5)	3·80
O(29)	4741 (13)	1381 (4)	1112 (4)	2·30
O(30)	5067 (16)	1121 (5)	34 (4)	3·27
O(31)	4955 (16)	3625 (4)	1846 (5)	2·90
O(32)	4586 (15)	2787 (5)	2622 (5)	3·52
O(33)	11187 (15)	3680 (5)	1681 (5)	3·00
O(34)	8163 (14)	4193 (4)	1942 (5)	3·20
O(35)	10622 (15)	557 (5)	1612 (5)	3·17
O(36)	5461 (17)	4528 (5)	1160 (6)	4·25
O(37)	8408 (14)	3969 (5)	895 (5)	3·12
O(38)	10933 (15)	3967 (5)	2736 (5)	3·77
N(39)	8387 (21)	1769 (8)	4489 (7)	4·09
C(40)	8191 (27)	2196 (10)	4136 (9)	4·49
C(41)	8207 (27)	1506 (9)	3393 (8)	3·96
C(42)	8131 (31)	2078 (9)	3572 (9)	4·90
C(43)	8383 (29)	1065 (10)	3770 (9)	5·03
C(44)	7731 (37)	239 (11)	3059 (11)	7·07
C(45)	8539 (43)	392 (12)	3653 (11)	8·10
C(46)	8533 (28)	1218 (10)	4347 (8)	4·57
C(47)	2851 (29)	1797 (9)	3329 (9)	4·82
C(48)	2449 (31)	2807 (9)	3759 (12)	6·64
N(49)	3142 (22)	1215 (7)	3398 (8)	4·40
C(50)	3338 (25)	1890 (8)	4338 (7)	3·33
C(51)	2897 (25)	2141 (8)	3809 (8)	3·80
C(52)	3627 (33)	1303 (9)	4398 (10)	5·38
C(53)	3501 (26)	978 (8)	3906 (10)	4·22
C(54)	4214 (36)	3131 (10)	3893 (9)	6·32
C(55)	6141 (34)	4959 (9)	4153 (11)	5·70
C(56)	9081 (34)	4483 (13)	4460 (11)	8·18
C(57)	4224 (31)	4823 (8)	3282 (10)	4·88
C(58)	5465 (35)	4401 (9)	3123 (9)	5·25
C(59)	7071 (32)	4271 (9)	3486 (8)	4·45
N(60)	4609 (25)	5053 (7)	3764 (9)	5·19
C(61)	7396 (27)	4551 (9)	4009 (9)	4·32
C(62)	−702 (41)	−726 (19)	716 (13)	15·52
O*W*(63)	2575 (20)	328 (6)	2673 (6)	5·24

* Calculated according to Willis & Pryor (1975).

(Swallow, Ahmed & Barnes, 1966), $Y_2V_{10}O_{28}.24H_2O$, $La_2V_{10}O_{28}.20H_2O$ and $Nd_2V_{10}O_{28}.28H_2O$ (Saf'yanov, Kuz'min & Belov, 1979), $Na_6V_{10}O_{28}.18H_2O$ (Durif, Averbuch-Pouchot & Guitel, 1980), $(C_7H_9NH)_4H_2V_{10}O_{28}$ (Debaerdemaeker, Arrieta & Amigó, 1982), $Er_2V_{10}O_{28}.25H_2O$ (Rivero, Rigotti, Punte & Navaza, 1984), and $(C_6H_7NH_3)_3H_3V_{10}O_{28}.H_2O$ (Santiago, Arnaiz, Lorente, Arrieta & Germain, 1988). As part of a general study of the crystal chemistry of the polyanions of organic bases (Arrieta, Gili & Lorente, 1984, and references therein), we have solved the structure of the title compound in order to eliminate several uncertainties arising from the disordered organic molecule in tris(3-methylpyridinium) decavanadate (Santiago *et al.*, 1988).

Experimental. Yellow–orange transparent prismatic crystals have been synthesized from acidic aqueous media following the method described by Arrieta, Gili & Lorente (1984) and Arrieta, Gili, Lorente & Germain (1985). The chemical analysis for V, C, H and N gave the following calculated (experimental) results: V_2O_5 69·80 (68·86); C 19·34 (19·26); H 2·69 (2·66); N 3·22 (3·52%). Vanadium was analysed as V_2O_5 after calcination at 823 K for 2 h; data collected from crystal approximately 0·1 × 0·1 × 0·05 mm; cell parameters determined by least squares from the setting angles of 23 reflections ($18 \leq 2\theta \leq 30°$); 7238 independent reflections measured; Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω–2θ scans ($\Delta 2\theta = 2{\cdot}5° + 0{\cdot}5°\tan\theta$) up to $2\theta = 52°$; one standard reflection (291) measured every 70, and a linear decay correction was made to allow for an overall 19% decrease in diffracting power caused by decomposition; Lp corrections applied but no correction for absorption; 2242 reflections [$I \geq 2{\cdot}5\sigma(I)$] considered observed and included in refinement; index range h 0→8, k 0→27, l −25→26; structure solved with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); an E map showed the decavanadate anion. All remaining (non-H) atoms from three ΔF syntheses; refinement by least squares with *SHELX*76 (Sheldrick, 1976), unit weights, $\sum(\Delta F)^2$ minimized, completed with anisotropic thermal parameters; the H atoms were not taken into account; final $R = 0{\cdot}042$; scattering factors from *International Tables for X-ray Crystallography* (1974) and the anomalous-scattering factors from Cromer & Liberman (1970); max. $\Delta/\sigma = 2{\cdot}65$ [for x, C(56), a secondary C atom of the ethyl group]; mean Δ/σ ratio on the final cycle 0·021; max. and min. electron densities in final difference map +1·09 and −0·39 e Å^{-3}.

Discussion. Table 1 gives the final atomic parameters with their B_{eq} values (Willis & Pryor, 1975).* Fig. 1 shows the numbering of the decavanadate anion and the arrangement of different V [V(I)–V(III)] and O (O*a*–O*g*) atom types according to Evans (1966). Fig. 2 shows the distances between V atoms. Fig. 3 shows a stereoscopic view of the unit cell. Distances given in Table 2 and angles (deposited*) are similar to others

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* Lists of structure factors, anisotropic thermal parameters, angles of the decavanadate anion, angles of the organic molecules, intermolecular distances and least-squares-planes' data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44780 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

previously described and cited in the *Introduction*. The severe distortions in the VO_6 octahedra are mainly due to internal Coulombic repulsions (Evans, 1966), and can be interpreted by the methods proposed by Baur (1970) and by Brown & Shannon (1973). The correlation between distortion (mean-square relative deviation of bond length from the average) and average V—O bond length within each VO_6 octahedron in our compound is slightly worse compared to that obtained from the data of Evans (1966), Rivero *et al.* (1984) and Santiago *et al.* (1988).

Table 3(*a*) shows extreme and average values of the V—O distances for each type of V and O atom. The ranges spanned by each type are a measure of the distortion from *mmm* symmetry (Rivero *et al.*, 1984). Although these ranges correspond to deviations of no more than 0·06 Å from the distances reported by Evans (1966), there are some significant variations as can be seen from the V—V distances given in Fig. 2, or observed in V(4)—O(18)/V(4)—O(31), V(5)—O(29)/V(5)—O(19) [V(I)—O*d* type] and V(4)—O(11)/V(4)—O(23), V(5)—O(11)/V(5)—O(23) [V(I)—O*b* type], where the first distance is lengthened by 0·22, 0·21, 0·21 and 0·20 Å (Table 2) respectively.

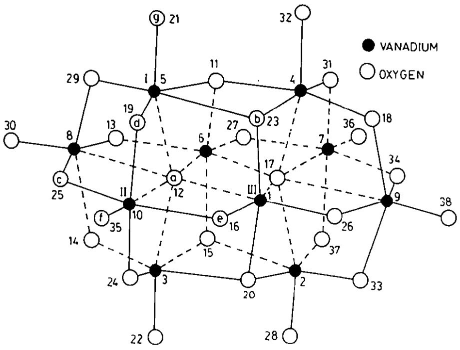

Fig. 1. View of the decavanadate group.

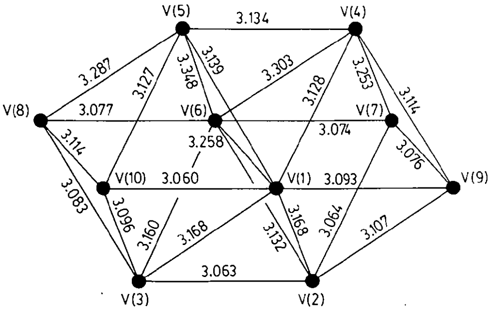

Fig. 2. Distances (Å) between vanadium atoms (e.s.d.'s 0·004 Å).

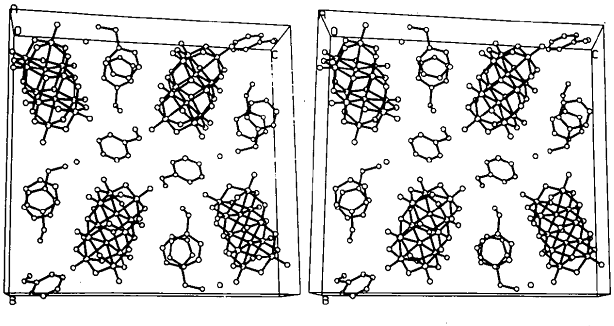

Fig. 3. Stereoscopic view of the molecular packing drawn with *PLUTO* (Motherwell & Clegg, 1978).

Table 2. *Bond lengths* (Å) *with e.s.d.'s* (0·01 Å) *and the bond numbers s of the decavanadate anion*

V(III)	O*a*	O*e*	O*a*	O*b*	O*b*	O*e*	Σ*s*(V)
V(I)	O(12)	O(16)	O(17)	O(20)	O(23)	O(26)	
	2·10	1·68	2·12	1·94	1·95	1·65	
s	0·44	1·39	0·42	0·67	0·65	1·528	5·09
Σ*s*(O)	1·96	1·96	1·99	1·85	2·00	2·02	
V(I)	O*b*	O*a*	O*b*	O*g*	O*d*	O*d*	
V(2)	O(15)	O(17)	O(20)	O(28)	O(33)	O(37)	
	2·02	2·31	2·01	1·57	1·83	1·85	
s	0·54	0·27	0·56	1·96	0·90	0·85	5·07
Σ*s*(O)	1·78	1·99	1·85	1·96	1·77	1·82	
V(I)	O*a*	O*d*	O*b*	O*b*	O*g*	O*d*	
V(3)	O(12)	O(14)	O(15)	O(20)	O(22)	O(24)	
	2·31	1·84	2·02	1·97	1·57	1·83	
s	0·27	0·87	0·54	0·62	1·96	0·90	5·15
Σ*s*(O)	1·96	1·82	1·78	1·85	1·96	1·80	
V(I)	O*b*	O*a*	O*d*	O*b*	O*d*	O*g*	
V(4)	O(11)	O(17)	O(18)	O(23)	O(31)	O(32)	
	2·14	2·25	1·74	1·93	1·96	1·59	
s	0·40	0·31	1·16	0·68	0·63	1·84	5·02
Σ*s*(O)	1·29	1·99	1·83	2·00	1·20	1·84	
V(I)	O*b*	O*a*	O*d*	O*g*	O*b*	O*d*	
V(5)	O(11)	O(12)	O(19)	O(21)	O(23)	O(29)	
	2·14	2·27	1·72	1·58	1·94	1·93	
s	0·40	0·30	1·23	1·84	0·67	0·68	5·11
Σ*s*(O)	1·29	1·96	1·85	1·84	2·00	1·22	
V(III)	O*b*	O*a*	O*e*	O*b*	O*a*	O*e*	
V(6)	O(11)	O(12)	O(13)	O(15)	O(17)	O(27)	
	2·06	2·10	1·70	1·92	2·06	1·68	
s	0·49	0·44	1·30	0·70	0·49	1·39	4·82
Σ*s*(O)	1·29	1·96	1·83	1·78	1·99	1·89	
V(II)	O*a*	O*e*	O*d*	O*c*	O*f*	O*d*	
V(7)	O(17)	O(27)	O(31)	O(34)	O(36)	O(37)	
	2·32	2·05	2·00	1·81	1·58	1·80	
s	0·27	0·50	0·57	0·95	1·90	0·97	5·16
Σ*s*(O)	1·99	1·89	1·20	1·87	1·90	1·82	
V(II)	O*a*	O*e*	O*d*	O*c*	O*d*	O*f*	
V(8)	O(12)	O(13)	O(14)	O(25)	O(29)	O(30)	
	2·32	2·03	1·81	1·85	2·02	1·58	
s	0·27	0·53	0·95	0·85	0·54	1·90	5·03
Σ*s*(O)	1·96	1·83	1·82	1·75	1·22	1·90	
V(II)	O*a*	O*d*	O*e*	O*d*	O*c*	O*f*	
V(9)	O(17)	O(18)	O(26)	O(33)	O(34)	O(38)	
	2·39	1·94	2·05	1·84	1·82	1·57	
s	0·23	0·67	0·5	0·87	0·92	1·96	5·15
Σ*s*(O)	1·99	1·83	2·02	1·77	1·87	1·96	
V(II)	O*a*	O*e*	O*d*	O*d*	O*c*	O*f*	
V(10)	O(12)	O(16)	O(19)	O(24)	O(25)	O(35)	
	2·36	2·00	1·97	1·83	1·83	1·60	
s	0·24	0·57	0·62	0·90	0·90	1·78	5·00
Σ*s*(O)	1·96	1·96	1·85	1·80	1·75	1·78	

Other parameters of interest, such as intermolecular contacts and the three ideal mean planes corresponding to the symmetry planes were calculated with *PARST* (Nardelli, 1983). The values of Ω^2 [$\Omega^2 =$

Table 3. *Some additional distances*

(*a*) Average bond distances (Å) and ranges in the decavanadate ion. The number of averaged values is given in parentheses

	Mean	Range
V(I)–O*g*	1·58	1·57–1·59 (4)
V(I)–O*a*	2·29	2·25–2·31 (4)
V(I)–O*b*	2·02	1·93–2·14 (8)
V(I)–O*d*	1·84	1·72–1·96 (8)
V(II)–O*f*	1·58	1·57–1·60 (4)
V(II)–O*a*	2·35	2·32–2·39 (4)
V(II)–O*c*	1·83	1·81–1·85 (4)
V(II)–O*d*	1·90	1·80–2·02 (8)
V(II)–O*e*	2·03	2·00–2·05 (4)
V(III)–O*e*	1·68	1·65–1·70 (4)
V(III)–O*a*	2·08	2·06–2·12 (4)
V(III)–O*b*	1·97	1·92–2·06 (4)

(*b*) Distances (Å) in the molecules of 3-ethylpyridine with e.s.d.'s in parentheses

C(40)–N(39)	1·30 (3)	C(53)–N(49)	1·35 (3)
C(46)–N(39)	1·33 (3)	C(51)–C(50)	1·42 (3)
C(42)–C(40)	1·40 (3)	C(52)–C(50)	1·38 (3)
C(42)–C(41)	1·39 (3)	C(53)–C(52)	1·41 (3)
C(43)–C(41)	1·37 (3)	N(60)–C(55)	1·39 (3)
C(45)–C(43)	1·59 (4)	C(61)–C(55)	1·39 (3)
C(46)–C(43)	1·44 (3)	C(61)–C(56)	1·56 (3)
C(45)–C(44)	1·54 (4)	C(58)–C(57)	1·42 (3)
N(49)–C(47)	1·37 (3)	N(60)–C(57)	1·29 (3)
C(51)–C(47)	1·41 (3)	C(59)–C(58)	1·41 (3)
C(51)–C(48)	1·57 (3)	C(61)–C(59)	1·42 (3)
C(54)–C(48)	1·49 (3)	C(56)–C(62)	1·41 (3)

$\sum(\Delta/\sigma)^2$, where $\Delta \equiv$ atomic deviation from the calculated mean plane and $\sigma \equiv$ standard deviation of Δ] are 830·9 (18 atoms), 325·4 (12 atoms) and 9·5 (6 atoms), indicating significant deviations from coplanarity in these three cases. Dihedral angles between these planes are 92·37 (4), 90·1 (1) and 89·5 (1)°.

In order to investigate the hydrogens of the polyanion we have used the empirical bond length/bond number calculation in an attempt to find valence-deficient O atoms, with the power function $s = (R/1{\cdot}791)^{-5{\cdot}1}$ (Brown, 1981), which relates the V–O distance R and the bond number s. Evans & Pope (1984) have made similar calculations for the $(C_7H_9NH)_4H_2V_{10}O_{28}$ structure (Debaerdemaeker *et al.*, 1982); they obtained strong evidence that the extra proton was attached at a double-linked O*d*-type atom ($\sum s = 1{\cdot}25$). In Table 2 we give the results of applying the same calculation to our molecule to obtain the $\sum s$ values for the O atoms: all except three are in the range 1·75–2·02. The exceptions are O(11), O(29) and O(31) whose $\sum s$ values are 1·29, 1·22 and 1·20 respectively; O(29) and O(31) are double-linked O*d*-type atoms and O(11) a triply linked O*b*-type atom. This situation was also identified in tris(3-methylpyridinium) decavanadate (Santiago *et al.*, 1988). The location of the acidic protons of the decavanadate ion on these three O atoms is also supported by the distances O(11)–O(20) 2·81 (1), O(29)–O(24) 2·74 (1) and O(31)–O(33) 2·73 (2) Å. These hydrogen bonds are parallel and link three adjacent O atoms in one ion to the corresponding oxygens of its neighbour along the *a* axis. Several other O atoms that are not hydrogen bonded are brought into close contact, O(21)–O(16) 2·91 (2), O(32)–O(26) 2·87 (1), O(27)–O(28) 2·80 (2), O(31)–O(33) 2·73 (2) Å. [All O–O distances cited in this paragraph have the relationship $O(X,Y,Z)$–$O(X-1,Y,Z)$.] These factors could also explain the deviations from *mmm* symmetry noted above.

The distances given in Table 3(*b*) and the deposited angles for the organic molecules show that they are not distorted. This is also confirmed by the values of Ω^2 for the pyridine rings, 3·0, 2·5 and 0·7 for [N(39)···C(46)], [N(49)···C(47)] and [N(60)···C(57)] rings respectively, indicating that they are planar.

Two pyridinium groups are bonded to the polyanion by hydrogen bonds, with N(39)–O(15) $[(X,\frac{1}{2}-Y,\frac{1}{2}+Z)$, (O*b* type)] 2·66 (2) and N(60)–O(25) $[(1-X,\frac{1}{2}+Y,\frac{1}{2}-Z)$, (O*c* type)] 2·85 (2) Å. The other organic group is H-bonded to the water molecule: N(49)–O*W*(63) = 2·71 (2) Å. The water molecule also links the polyanions as supported by the distances O*W*(63)–O(34) $[(1-X,Y-\frac{1}{2},\frac{1}{2}-Z)$, (O*c* type)] = 2·86 (2) and O*W*(63)–O(35) $[(X-1,Y,Z)$, (O*f* type)] = 2·85 (2) Å.

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Structure of Bis[1,2-bis(diphenylphosphinoethylene)]iodocobalt(II) Tetraphenylborate

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Abstract. $[CoI(C_{26}H_{22}P_2)_2][B(C_6H_5)_4]$, $M_r = 1297{\cdot}9$, monoclinic, $P2_1/c$, $a = 21{\cdot}014$ (3), $b = 13{\cdot}223$ (3), $c = 24{\cdot}696$ (5) Å, $\beta = 110{\cdot}9$ (2)°, $V = 6411$ (5) Å^3, $Z = 4$, D_m(by flotation) = 1·35, $D_x = 1{\cdot}345$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 8{\cdot}818$ cm^{-1}, $F(000) = 2660$, $T = 293$ K. Final $R = 0{\cdot}074$ for 3886 unique reflections. The geometry around cobalt is a distorted trigonal bipyramid in which each diphos ligand bridges an axial and an equatorial position and the iodine atom occupies an equatorial position. There is tetrahedral geometry around the B atom.

Introduction. The vacant 3*d* orbitals of the P atom are capable of interacting with filled non-bonding *d* orbitals of transition metals. Phosphines act as σ-bond donors and π-bond acceptors. The stabilizing effect of phosphine ligands has been utilized in the preparation of a wide range of stable organometallic compounds.

On the basis of ESR and electronic spectra, the geometry around cobalt in the cation of $[Co\{(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2\}_2Cl]SnCl_3.C_6H_5Cl$ (Horrocks, Hecke & Hall, 1967) was suggested as square pyramidal, but X-ray analysis showed a trigonal bipyramidal geometry with two of the P atoms occupying the axial positons and two P atoms and one Cl atom forming the equatorial plane (Stalick, Corfield & Meek, 1973).

Experimental. The compound was prepared by the procedure followed by McAuliffe & Meek (1969) for the analogous nickel(II) complex. Unit-cell parameters obtained by least-squares refinement of θ values of 25 high-angle reflections. Crystals 0·35 × 0·45 × 0·625 mm, three-dimensional intensity data collected on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo$K\alpha$ radiation, $\omega/2\theta$ scan mode, standard reflections (222, 133) showed no significant change. All reflections (h: −19→18, k: 0→13, l: 0→20) in the range $1 < \theta < 25°$ measured; maximum counting time for a reflection 60 s, 5683 reflections measured, 3886 unique reflections with $I > 3\sigma(I)$ considered observed; intensities not corrected for absorption. Structure was solved by heavy-atom method and refined by full-matrix least squares, minimizing $\sum w|\Delta F|^2$ with *SHELX*76 (Sheldrick, 1976), initially with isotropic and then anisotropic thermal parameters for non-H atoms of the cation. Refinements of 587 positional and thermal parameters including the scale factor converged to $R = 0{\cdot}074$. Maximum and average shift/e.s.d. are 0·38 and 0·27 respectively except for C(28), whose thermal parameter became non-positive-definite and the shift/e.s.d. was 0·87. Final difference Fourier maps revealed the positions of 15 of the 64 H atoms. These were refined for one cycle. Maximum peak in final difference Fourier map is 0·98 e Å^{-3}, $wR = 0{\cdot}074$, $w = 1{\cdot}00/[\sigma^2(F_o) + 0{\cdot}0007|F_o|^2]$.

Atomic scattering factors for non-H atoms from Cromer & Mann (1968), anomalous-dispersion correction factors from Cromer & Liberman (1970), H-atom scattering factors from Stewart, Davidson & Simpson (1965).

Discussion. Atomic coordinates with equivalent isotropic thermal parameters are listed in Table 1.* Table

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* Lists of structure factors, H-atom coordinates, anisotropic thermal parameters, bond distances and angles, and atom-to-plane distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44801 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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