that in either TPPO or its previously known complexes.

The $AuCl_4^-$ ion possesses approximate 4/*mmm* symmetry, as found in its other salts, *e.g.* $Ph_4AsAuCl_4$ (Jones, Guy & Sheldrick, 1975).

We thank the SRC for a contribution towards the cost of the diffractometer. The figures were drawn with *PLUTO* written by Dr W. D. S. Motherwell; all other crystallographic programs were written by GMS.

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Bis(acetylacetonato)bis(6-methylquinoline)cobalt(II)

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Abstract. $C_{30}H_{32}CoN_2O_4$, $M_r = 543{\cdot}53$, monoclinic, $a = 7{\cdot}543\ (1)$, $b = 10{\cdot}961\ (1)$, $c = 16{\cdot}082\ (1)$ Å, $\beta = 97{\cdot}15\ (1)°$, $U = 1319{\cdot}3$ Å^3, $Z = 2$, $D_x = 1{\cdot}370$, $D_c = 1{\cdot}368$ g cm^{-3}, space group $P2_1/c$, $F(000) = 566$, μ(Mo $K\alpha$) = 6·4 cm^{-1}, λ(Mo $K\alpha$) = 0·71069 Å. The structure was refined to $R = 0{\cdot}0375$ for 3564 diffractometer data. The complex is centrosymmetric. The Co atom has an octahedral coordination with Co—N and mean Co—O distances of 2·262 (4) and 2·036 (3) Å respectively.

Introduction. The structure of the title compound has been determined as a preliminary to interpreting its magnetic susceptibility in terms of the model outlined by Gerloch & McMeeking (1975).

The complex was prepared by adding the stoichiometric amount of 6-methylquinoline to a solution of Co acetylacetonate dihydrate in toluene. Suitable crystals were obtained by recrystallizing from CH_2Cl_2.

The crystal system was determined from oscillation and Weissenberg photographs. Cell dimensions were obtained by least squares from the setting angles for fifteen reflexions automatically centred on a Nonius CAD-4 diffractometer. Intensities for 5616 reflexions ($1{\cdot}5 < \theta < 33°$) were recorded with graphite-monochromated Mo $K\alpha$ radiation, the $\omega/2\theta$ scan and a crystal 0·55 × 0·35 × 0·25 mm. All data were

Table 1. *Atom coordinates* ($\times 10^4$)

	x	y	z
Co	0	0	0
N(1)	1124 (2)	−1880 (1)	317 (1)
C(2)	1718 (2)	−2016 (2)	1117 (1)
C(3)	2505 (3)	−3091 (2)	1467 (1)
C(4)	2709 (3)	−4050 (2)	952 (1)
C(5)	2313 (3)	−4912 (2)	−487 (1)
C(6)	1731 (3)	−4790 (2)	−1319 (1)
C(7)	919 (2)	−3683 (2)	−1602 (1)
C(8)	707 (2)	−2734 (2)	−1072 (1)
C(9)	1317 (2)	−2847 (1)	−209 (1)
C(10)	2115 (2)	−3957 (1)	87 (1)
C(11)	1935 (5)	−5789 (2)	−1943 (2)
O(12)	2104 (1)	746 (1)	734 (1)
C(13)	3304 (2)	1391 (1)	469 (1)
C(14)	3655 (2)	1453 (2)	−364 (1)
C(15)	2774 (2)	793 (1)	−1034 (1)
O(16)	1393 (2)	144 (1)	−1007 (1)
C(17)	4442 (1)	2149 (2)	1115 (1)
C(18)	3496 (3)	848 (2)	−1867 (1)

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corrected for Lp factors and variable measuring time but not for absorption. Averaging equivalent reflexions and omitting those for which $I < 1{\cdot}5\sigma(I)$ yielded 3564 unique data. The structure was solved by the heavy-atom method and refined by full-matrix least squares to a final $R = 0{\cdot}0375$. The H atoms were located from difference maps and refined with isotropic temperature factors; C–H distances vary between 0·71 and 1·27 Å. All other atoms were refined anisotropically. In the final refinement an isotropic extinction parameter, x, was varied where $F' = F[1 - (xF^2/\sin\theta)]$ for F_c; x refined to a value of 0·0055 (4). The weighting scheme $w = 1/[\sigma^2(F_o) + 0{\cdot}0008F_o^2]$ was used to give constancy of $w\Delta F^2$ with $\sin\theta$ and $(F_o/F_{\max})^{1/2}$. Neutral-atom scattering factors were taken from Stewart, Davidson & Simpson (1965) for H and from Cromer & Mann (1968) for Co, C, N and O atoms. The final atomic coordinates are presented in Tables 1 and 2 and the interatomic distances and angles in Tables 3 and 4.* The molecule is illustrated in Fig. 1.

Table 2. *Hydrogen atom coordinates* ($\times 10^3$) *and isotropic temperature factors* (Å$^2 \times 10^3$)

	x	*y*	*z*	*U*
H(2)	156 (3)	−131 (2)	143 (1)	50 (5)
H(3)	296 (3)	−314 (2)	203 (1)	58 (6)
H(4)	335 (4)	−487 (2)	118 (2)	62 (7)
H(5)	295 (4)	−571 (3)	−28 (2)	80 (8)
H(7)	48 (3)	−361 (2)	−213 (2)	64 (6)
H(8)	11 (3)	−207 (2)	−128 (1)	56 (6)
H(11*a*)	192 (5)	−653 (3)	−173 (2)	90 (10)
H(11*b*)	181 (12)	−582 (8)	−243 (4)	282 (26)
H(11*c*)	320 (6)	−605 (4)	−182 (2)	122 (14)
H(14)	474 (3)	201 (2)	−49 (2)	71 (7)
H(17*a*)	503 (3)	287 (2)	86 (2)	72 (7)
H(17*b*)	403 (5)	226 (4)	148 (2)	130 (14)
H(17*c*)	603 (6)	174 (4)	125 (3)	162 (15)
H(18*a*)	403 (5)	142 (3)	−197 (2)	102 (11)
H(18*b*)	487 (5)	64 (3)	−189 (2)	103 (10)
H(18*c*)	275 (5)	22 (3)	−228 (2)	94 (10)

Table 3. *Interatomic distances* (Å)

Co–O(12)	2·028 (3)	Co–N(1)	2·262 (4)
Co–O(16)	2·043 (3)	N(1)–C(2)	1·318 (3)
N(1)–C(9)	1·375 (3)	C(2)–C(3)	1·404 (3)
C(3)–C(4)	1·359 (4)	C(4)–C(10)	1·412 (3)
C(5)–C(10)	1·417 (3)	C(5)–C(6)	1·361 (4)
C(6)–C(7)	1·409 (4)	C(7)–C(8)	1·367 (3)
C(8)–C(9)	1·412 (3)	C(9)–C(10)	1·414 (3)
C(13)–C(14)	1·399 (3)	C(14)–C(15)	1·395 (3)
O(12)–C(13)	1·263 (3)	C(15)–O(16)	1·267 (3)
C(6)–C(11)	1·507 (5)	C(13)–C(17)	1·511 (4)
C(15)–C(18)	1·509 (4)	N(1)–O(12)	3·026 (6)
N(1)–O(16)	3·099 (6)	N(1)–O(12)	3·050 (6)
N(1)–O(16)	2·997 (6)	O(12)–O(16)	2·861 (6)
O(16)–O(16*)	2·897 (6)		

Table 4. *Bond angles* (°)

O(12)–Co–N(1)	89·5 (2)	O(16)–Co–N(1)	91·9 (2)
O(16)–Co–O(12)	89·3 (2)	C(9)–N(1)–C(2)	117·6 (2)
C(3)–C(2)–N(1)	124·2 (2)	C(4)–C(3)–C(2)	118·7 (3)
C(10)–C(4)–C(3)	119·9 (3)	C(10)–C(5)–C(6)	121·4 (3)
C(7)–C(6)–C(5)	118·5 (3)	C(11)–C(6)–C(5)	122·4 (3)
C(11)–C(6)–C(7)	119·1 (3)	C(8)–C(7)–C(6)	122·2 (3)
C(9)–C(8)–C(7)	119·8 (2)	C(8)–C(9)–N(1)	119·4 (2)
C(10)–C(9)–N(1)	122·0 (2)	C(10)–C(9)–C(8)	118·6 (2)
C(5)–C(10)–C(4)	122·9 (3)	C(9)–C(10)–C(4)	117·7 (2)
C(9)–C(10)–C(5)	119·4 (2)	C(14)–C(13)–O(12)	125·4 (2)
C(17)–C(13)–O(12)	116·5 (2)	C(17)–C(13)–C(14)	118·2 (2)
C(15)–C(14)–C(13)	125·8 (2)	O(16)–C(15)–C(14)	125·5 (2)
C(18)–C(15)–C(14)	118·3 (2)	C(18)–C(15)–O(16)	116·2 (2)
C(13)–O(12)–Co	124·8 (2)	C(15)–O(16)–Co	125·2 (2)
C(2)–N(1)–Co	113·3 (2)	C(9)–N(1)–Co	129·0 (2)

Discussion. The metal coordination is octahedral with a small tetragonal distortion along the N–Co–N axis. The Co–N distance is slightly longer [2·262 (4) *vs* 2·187 (5) Å] than the corresponding bond in the analogous pyridine complex (Elder, 1968) and this may be a reflexion of the greater bulk of the quinoline ligand. The Co atom lies 0·41 Å from the least-squares plane of the acetylacetonate group corresponding to a fold about the O···O vector of 16·3°. The deviation of the Co atom from the quinoline plane is 0·09 Å.

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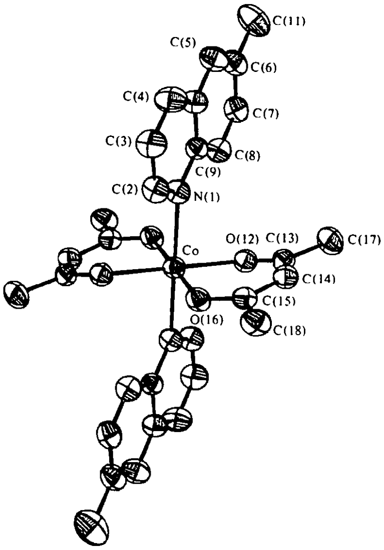

Fig. 1. The molecular configuration.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33284 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

University IBM 370/165 computers with programs written by Dr G. M. Sheldrick (Cambridge University). The diagram was drawn with *ORTEP* (Johnson, 1965).

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Bis(5,5-Dimethyl-2-oxo-1,3,2-dioxaphosphorinanyl) Oxide

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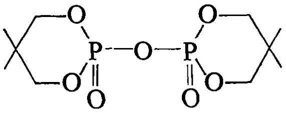
Abstract. $C_{10}H_{20}O_7P_2$, orthorhombic, *Pbca*, $a = 26{\cdot}88$ (2), $b = 9{\cdot}99$ (1), $c = 11{\cdot}13$ (1) Å; $V = 2988{\cdot}8$ Å^3; $M_r = 314{\cdot}22$; $D_x = 1{\cdot}39$, $D_m = 1{\cdot}39$ g cm^{-3}; $Z = 8$; $F(000) = 1328{\cdot}0$; $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å; $\mu r(\text{Cu } K\alpha) = 0{\cdot}6$. The structure was solved by direct methods and refined by least-squares methods with anisotropic temperature factors to an *R* value of 0·121 for 1179 observed reflections. The symmetry of the bicyclic molecules is C_1. The conformation around the bridge bonds P—O—P is staggered. The two structurally independent dioxaphosphorinane rings have partially flattened chair conformations with the double-bonded O atom in an equatorial and the linking O atom in an axial position. An analysis of the causes of the flattening observed in the 1,3,2-dioxaphosphorinane rings was carried out and the structural results are compared with those for bis(5,5-dimethyl-2-oxo-1,3,2-dioxaphosphorinanyl) sulphide.

Introduction. We are studying several structures of bicyclic organic pyro- and thiopyrophosphates (Bukowska-Strzyżewska, Dobrowolska, Michalski, Młotkowska & Skoweranda, 1976; Bukowska-Strzyżewska, Michalski, Młotkowska & Skoweranda, 1976).

The organic phosphates have a special interest because they are the components of many biologically important molecules such as nucleic acids and some enzymes or vitamins, performing an essential function in important life processes.

Crystals of the title compound have been prepared by B. Młotkowska and crystallized by us from an ethyl acetate solution.



A preliminary report on this work has been published elsewhere (Bukowska-Strzyżewska & Dobrowolska, 1976).

Equi-inclination Weissenberg film data were collected with the multiple-film technique on the 0 to 8 layers around both the *b* and *c* crystal axes. The intensities were visually estimated with a calibrated intensity scale. In total, 1179 reflections were collected. No absorption correction was made.

The structure was solved by direct methods using the *SIGMA* 2 and *PHASE* programs of the XRAY 70 system (Stewart, Kundell & Baldwin, 1970) on an

Table 1. *Final atomic positions* ($\times 10^4$) *with e.s.d.'s in parentheses*

	x	*y*	*z*
P(2)	1194 (1)	4589 (3)	325 (4)
P(12)	1178 (1)	7530 (4)	−74 (4)
O(1)	623 (4)	4332 (9)	277 (9)
O(11)	1710 (3)	7871 (8)	−499 (9)
O(2)	1448 (5)	4208 (11)	−719 (12)
O(12)	814 (4)	7459 (13)	−1002 (11)
O(3)	1383 (4)	3879 (9)	1470 (10)
O(13)	1072 (3)	8566 (10)	918 (10)
O(4)	1253 (4)	6176 (8)	691 (11)
C(4)	1091 (6)	3978 (16)	2546 (16)
C(14)	1459 (6)	8868 (16)	1777 (14)
C(5)	562 (6)	3529 (14)	2294 (15)
C(15)	1945 (5)	9299 (13)	1182 (13)
C(6)	339 (6)	4436 (17)	1375 (15)
C(16)	2109 (5)	8166 (16)	339 (17)
C(7)	515 (6)	2009 (15)	1949 (16)
C(17)	1863 (8)	10621 (15)	471 (17)
C(8)	243 (9)	3758 (20)	3452 (17)
C(18)	2346 (7)	9526 (22)	2135 (17)