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Structure of *trans*-[Bis(2,2′-bipyridyl)bis(methyldiphenylphosphine)ruthenium(II)] Perchlorate Tetrahydrofuran Solvate

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Abstract. $[Ru(C_{10}H_8N_2)_2(C_{13}H_{13}P)_2](ClO_4)_2.2C_4H_8O$, $M_r = 1156{\cdot}9$, monoclinic, $P2_1/c$, $a = 11{\cdot}387\,(3)$, $b = 10{\cdot}940\,(5)$, $c = 21{\cdot}633\,(6)$ Å, $\beta = 105{\cdot}503\,(10)°$, $V = 2597$ Å^3, $Z = 2$ (implying that each molecule sits on a twofold special position), $D_x = 1{\cdot}479$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}520$ mm^{-1}, $F(000) = 1196$, $T = 293$ K, $R = 0{\cdot}0379$ for 3535 unique observed reflections. The Ru lies on a crystallographic inversion centre and is coordinated by two *trans* methyldiphenylphosphines [Ru—P = 2·4023 (9) Å] and two mutually *trans* bipyridyl ligands [Ru—N = 2·090 (3), 2·096 (3) Å]. The structure results in very close H···H non-bonded interactions of 1·95 Å between H(6) of one bipyridyl and H(6′) of the other in the complex and in a hinge angle of 158·7 (3)° between the two rings of each bipyridyl ligand, which is in a bowed (rather than a twisted) configuration. Although the H···H interactions are stereochemically unfavourable, the *trans* isomer is the main product of the preparative reaction, presumably because the *cis* form would involve unacceptably close contacts between the bulky phosphine ligands.

Experimental. Compound prepared as described previously (Sullivan, Salmon & Meyer, 1978) but using tetrahydrofuran instead of acetone. Orange columnar crystal, 0·20 × 0·18 × 0·80 mm, Stadi-2 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, cell parameters from 13 reflections with $3{\cdot}8 < \theta < 17{\cdot}6°$. For data collection, ω scans with scan width $1{\cdot}0 + 0{\cdot}5 \times (\sin\mu/\tan\theta)°$, $\theta_{\max} = 25°$, h −14→14, k 0→12, l 0→26, no significant variation in intensities of three standard reflections, no absorption correction, 4642 reflections, 4490 unique ($R_{\text{int}} = 0{\cdot}008$), giving 3535 with $F > 6\sigma(F)$ for structure solution [from the inferred Ru position using *DIRDIF* (Beurskens *et al.*, 1983)] and refinement [using full-matrix least squares on F (Sheldrick, 1976)]. Anisotropic thermal parameters for Ru, Cl, P, N, O and C, phenyl rings refined as rigid groups and H atoms in calculated positions. At convergence, $R = 0{\cdot}0379$, $wR = 0{\cdot}0480$ [$w^{-1} = \sigma^2(F) + 0{\cdot}000326F^2$], $S = 1{\cdot}310$ for 307 parameters, max. shift/e.s.d. in final cycle = 0·034, max. and min. residues in final difference Fourier synthesis 0·59 and −0·73 e Å^{-3} respectively. Scattering factors were inlaid (Sheldrick, 1976) except for Ru (Cromer & Mann, 1968). Atom coordinates and equivalent isotropic thermal parameters are given in Table 1 while selected bond lengths and angles appear in Table 2.† The atom-numbering scheme for the cation is shown in Fig. 1, which was generated using *ORTEP* (Mallinson & Muir, 1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Table 1. *Fractional coordinates of atoms with e.s.d.'s*

$$U_{\text{eq}} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
Ru(1)	0·5	0·0	0·0	0·0252 (2)
N(1)	0·44312 (24)	0·17675 (24)	0·01459 (13)	0·0311 (15)
C(2)	0·3662 (3)	0·2290 (3)	−0·03788 (16)	0·0326 (19)
C(3)	0·3337 (4)	0·3514 (3)	−0·03862 (19)	0·0449 (23)
C(4)	0·3742 (4)	0·4195 (4)	0·01640 (22)	0·055 (3)
C(5)	0·4444 (4)	0·3646 (4)	0·07138 (20)	0·0503 (24)
C(6)	0·4773 (3)	0·2441 (3)	0·06855 (17)	0·0383 (20)
N(1′)	0·34546 (24)	0·02517 (23)	−0·07722 (12)	0·0297 (15)
C(2′)	0·3104 (3)	0·1439 (3)	−0·08944 (15)	0·0314 (18)
C(3′)	0·2180 (3)	0·1763 (4)	−0·14260 (17)	0·0416 (22)
C(4′)	0·1536 (4)	0·0870 (4)	−0·18308 (19)	0·0475 (23)
C(5′)	0·1839 (4)	−0·0333 (4)	−0·16868 (18)	0·0437 (23)
C(6′)	0·2800 (3)	−0·0603 (3)	−0·11672 (16)	0·0355 (20)
P(1)	0·62633 (8)	0·08677 (8)	−0·06160 (4)	0·0305 (5)
C(12)	0·82313 (22)	−0·0722 (3)	−0·02992 (9)	0·054 (3)
C(13)	0·91900 (22)	−0·1412 (3)	−0·04048 (9)	0·067 (3)
C(14)	0·93979 (22)	−0·1432 (3)	−0·10116 (9)	0·062 (3)
C(15)	0·86470 (22)	−0·0764 (3)	−0·15129 (9)	0·060 (3)
C(16)	0·76884 (22)	−0·0075 (3)	−0·14073 (9)	0·0468 (22)
C(11)	0·74805 (22)	−0·0054 (3)	−0·08005 (9)	0·0365 (18)
C(22)	0·48597 (23)	0·06200 (16)	−0·18747 (11)	0·0414 (21)
C(23)	0·41336 (23)	0·10364 (16)	−0·24625 (11)	0·053 (3)
C(24)	0·39414 (23)	0·22870 (16)	−0·25695 (11)	0·060 (3)
C(25)	0·44755 (23)	0·31211 (16)	−0·20888 (11)	0·061 (3)
C(26)	0·52016 (23)	0·27047 (16)	−0·15010 (11)	0·0468 (23)
C(21)	0·53938 (23)	0·14542 (16)	−0·13940 (11)	0·0352 (19)
C(1*M*)	0·7137 (4)	0·2172 (3)	−0·02127 (17)	0·0455 (23)

Related literature. Analogous compounds containing *trans*-[Ru(bipyridyl)$_2XY$]$^{x+}$ where $X,Y = R_3$P, NO$^+$, Cl$^-$, pyridine, H$_2$O, CH$_3$CN *etc.* have been generated

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† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44720 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. *Bond lengths* (Å), *angles* (°) *and selected torsion angles* (°) *with e.s.d.'s*

Ru(1)—N(1)	2·090 (3)	N(1′)—C(2′)	1·365 (4)
Ru(1)—N(1′)	2·096 (3)	N(1′)—C(6′)	1·349 (4)
Ru(1)—P(1)	2·4023 (9)	C(2′)—C(3′)	1·381 (5)
N(1)—C(2)	1·361 (4)	C(3′)—C(4′)	1·384 (6)
N(1)—C(6)	1·347 (5)	C(4′)—C(5′)	1·375 (6)
C(2)—C(3)	1·388 (5)	C(5′)—C(6′)	1·375 (5)
C(2)—C(2′)	1·462 (5)	P(1)—C(11)	1·842 (3)
C(3)—C(4)	1·375 (6)	P(1)—C(21)	1·8259 (24)
C(4)—C(5)	1·380 (6)	P(1)—C(1M)	1·823 (4)
C(5)—C(6)	1·377 (5)		

N(1)—Ru(1)—N(1′)	76·74 (10)	C(2)—C(2′)—C(3′)	123·5 (3)
N(1)—Ru(1)—P(1)	88·50 (8)	N(1′)—C(2′)—C(3′)	121·6 (3)
N(1′)—Ru(1)—P(1)	90·32 (7)	C(2′)—C(3′)—C(4′)	120·2 (3)
Ru(1)—N(1)—C(2)	114·79 (21)	C(3′)—C(4′)—C(5′)	118·3 (4)
Ru(1)—N(1)—C(6)	127·42 (23)	C(4′)—C(5′)—C(6′)	119·2 (4)
C(2)—N(1)—C(6)	117·7 (3)	N(1′)—C(6′)—C(5′)	123·6 (3)
N(1)—C(2)—C(3)	121·7 (3)	Ru(1)—P(1)—C(11)	120·06 (9)
N(1)—C(2)—C(2′)	114·9 (3)	Ru(1)—P(1)—C(21)	113·12 (8)
C(3)—C(2)—C(2′)	123·0 (3)	Ru(1)—P(1)—C(1*M*)	112·01 (13)
C(2)—C(3)—C(4)	119·1 (4)	C(11)—P(1)—C(21)	104·28 (11)
C(3)—C(4)—C(5)	119·6 (4)	C(11)—P(1)—C(1*M*)	100·73 (15)
C(4)—C(5)—C(6)	118·7 (4)	C(21)—P(1)—C(1*M*)	104·96 (15)
N(1)—C(6)—C(5)	122·9 (3)	P(1)—C(11)—C(12)	117·31 (19)
Ru(1)—N(1′)—C(2′)	114·82 (21)	P(1)—C(11)—C(16)	122·67 (19)
Ru(1)—N(1′)—C(6′)	128·16 (22)	P(1)—C(21)—C(22)	118·57 (17)
C(2′)—N(1′)—C(6′)	117·0 (3)	P(1)—C(21)—C(26)	121·33 (17)
C(2)—C(2′)—N(1′)	114·4 (3)		

N(1′)—Ru(1)—N(1)—C(2)	−18·04 (22)	C(4)—C(5)—C(6)—N(1)	−0·6 (6)
N(1′)—Ru(1)—N(1)—C(6)	164·5 (3)	Ru(1)—N(1′)—C(2′)—C(2)	−14·6 (4)
N(1)—Ru(1)—N(1′)—C(2′)	17·57 (22)	Ru(1)—N(1′)—C(2′)—C(3′)	173·3 (3)
N(1)—Ru(1)—N(1′)—C(6′)	−164·6 (3)	C(6′)—N(1′)—C(2′)—C(2)	167·3 (3)
Ru(1)—N(1)—C(2)—C(3)	−171·0 (3)	C(6′)—N(1′)—C(2′)—C(3′)	−4·8 (5)
Ru(1)—N(1)—C(2)—C(2′)	16·0 (4)	Ru(1)—N(1′)—C(6′)—C(5′)	−175·9 (3)
C(6)—N(1)—C(2)—C(3)	6·8 (5)	C(2′)—N(1′)—C(6′)—C(5′)	2·0 (5)
C(6)—N(1)—C(2)—C(2′)	−166·3 (3)	C(2)—C(2′)—C(3′)—C(4′)	−167·5 (3)
Ru(1)—N(1)—C(6)—C(5)	173·0 (3)	N(1′)—C(2′)—C(3′)—C(4′)	3·8 (5)
C(2)—N(1)—C(6)—C(5)	−4·4 (5)	C(2′)—C(3′)—C(4′)—C(5′)	0·2 (6)
N(1)—C(2)—C(3)—C(4)	−4·1 (6)	C(3′)—C(4′)—C(5′)—C(6′)	−3·0 (6)
C(2′)—C(2)—C(3)—C(4)	168·3 (4)	C(4′)—C(5′)—C(6′)—N(1′)	1·9 (6)
N(1)—C(2)—C(2′)—N(1′)	−0·9 (4)		
N(1)—C(2)—C(2′)—C(3′)	171·0 (3)		
C(3)—C(2)—C(2′)—N(1′)	−173·8 (3)		
C(3)—C(2)—C(2′)—C(3′)	−1·9 (5)		
C(2)—C(3)—C(4)—C(5)	−1·1 (6)		
C(3)—C(4)—C(5)—C(6)	3·4 (6)		

by direct reaction of *cis*-$[Ru(bipyridyl)_2Cl_2]$ or by photoisomerization of corresponding *cis* complexes (Walsh & Durham, 1982). The crystal structures of *trans*-$[Ru(bipyridyl)_2(OH)(OH_2)]^{2+}$ (Durham, Wilson, Hodgson & Meyer, 1980), *trans*-$[Ru(4,4'\text{-dimethyl-}2,2'\text{-bipyridyl})_2(pyridine)_2]^{2+}$ and *trans*-$[Ru(bipyridyl)_2(PPh_3)_2]^{2+}$ have been reported and conformations of the distorted bipyridyl ligands analysed (Cordes, Durham, Swepston, Pennington, Condren, Jensen & Walsh, 1982; Cordes, Swepston, Pennington, Condren & Durham, 1981).

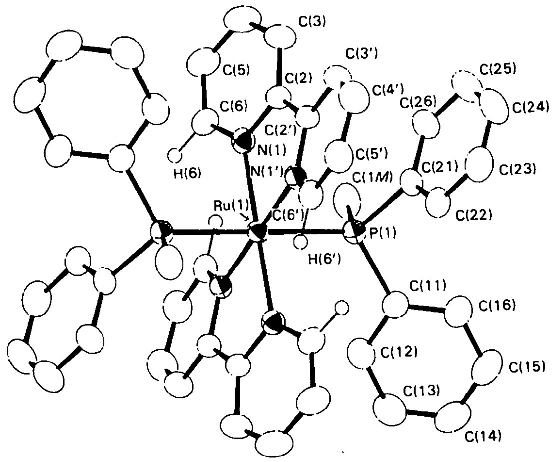

Fig. 1. *ORTEP* plot of a single molecule showing atom-labelling scheme. H atoms are omitted for clarity, except H(6) and H(6′) which have artificial radii of 0·10 Å.

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