Crystal structure of *cis*-[Ru(bpy)₂{PMe(o-tol)₂}Cl⁺][ClO₄⁻]: Steric effects in some PMe_n(o-tol)_{3-n} (n = 1-3) derivatives of ruthenium(II)

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The complex *cis*-[Ru(bpy)₂{PMe(o-tol)₂}Cl⁺][ClO₄⁻] crystallizes in space group P2₁/c with a = 9.375(2) Å, b = 22.019(7) Å, c = 16.153(4) Å, $\beta = 90.83(2)^{\circ}$, V = 3333.9(16) Å³ and **D**(calc'd) = 1.547 g/cm³ for **Z** = 4. The Ru-PMe(o-tol)₂ bond length of 2.357(3) Å is significantly longer than distances of Ru-PMe₂(o-tol) = 2.324(2) Å and Ru-PMe₃ = 2.310(2) Å in analogous complexes. The corresponding Ru-P(o-tol)₃ complex has eluded synthesis, probably due to steric hindrance.

KEY WORDS: Steric effects; ruthenium(II).

Introduction

We have previously reported the crystal structures of *cis*-[Ru(bpy)₂(PMe₃)₂Cl⁺][ClO₄⁻]¹ and *cis*-[Ru(bpy)₂{PMe₂(o-tol)}Cl⁺][ClO₄⁻]¹ and now report the crystal structure of the more hindered species *cis*-[Ru(bpy)₂{PMe(o-tol)₂Cl⁺][ClO₄⁻]. We have been unable to synthesize the tri-(o-tolyl)phosphine derivative *cis*-[Ru(bpy)₂{P(o-tol)₃Cl⁺][ClO₄⁻], which would complete the series; this is, presumably, due to steric hindrance.

Experimental

Synthesis of PMe(o-tol)₂

In an inert atmosphere glovebox, α -bromotoluene (5 g, 29.2 mmol), magnesium (0.711 g, 29.3 mmol), tetrahydrofuran (30 mL) and a small crystal of I₂ were stirred together for 2 h to form α -bromomagnesium toluene. P(CH₃)Cl₂ (1.71 g, 14.6 mmol) was added slowly and the solution was stirred for a further 2 h.

The solution was washed with 10% aqueous NH₄Cl (3×25 mL) and the compound was extracted with methylene chloride and reduced to dryness on a rotary evaporator. The compound was purified by column chromatography on basic alumina with hexane being used as the eluent. The first band that eluted from the column was reduced to dryness using a rotary evaporator, producing 2.02 g of an off-white solid (60.7% yield), which was characterized by ¹H NMR (CDCl₃): δ 7.2 (m 8H-C₆H₄CH₃), 2.4 (s, 6H, -C₆H₄CH₃), 1.5 (d, 3H, -CH₃).

Synthesis of cis- $[Ru(bpy)_2 \{PMe(o-tol)_2\}Cl^+][ClO_4^-]$

This synthesis is based upon the method of Marmion and Takeuchi.² Ru(bpy)₂Cl₂·2H₂O (521 mg, 1 mmol) and PMe(o-tol)₂ (228 mg, 1 mmol) were added to 100 mL of a nitrogen-deaerated ethanol-water mixture (80/20 v/v) and refluxed for 6 h. The solution

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was reduced to dryness; 50 mL of water and an excess of sodium perchlorate were added to precipitate the perchlorate salt. The red solid was collected by suction filtration, air-dried and then dried in a vacuum oven overnight, without heating. Purification was achieved by column chromatography on basic alumina utilizing 1% methanol in methylene chloride as an eluent. The first red band to elute was collected and the solvent removed with a rotary evaporator. The product was then redissolved in a minimum amount of methylene chloride and added dropwise into hexane. The microcrystalline product was collected by suction-filtration, air-dried and dried in a vacuum oven overnight.

Yield 0.582 g (75.4%). Anal. Calcd. for $C_{35}H_{33}Cl_2N_4O_4PRu$: C 54.13, H 4.28%. Found: C 53.98, H 4.34%. UV-vis data (CH₂Cl₂) λ_{max} , nm (ϵ , M⁻¹ cm⁻¹): 476 (7300); 339 (7300); 295 (46000).

Crystals suitable for an x-ray study were prepared by recrystallization from CH₂Cl₂/toluene.

Attempted Synthesis of cis- $[Ru(bpy)_{2}\{P(o-tol)_{3}\}Cl^{+}]$ $[ClO_{4}^{-}]$

Attempts to synthesize this species from [Ru $(bpy)_2Cl_2$]·2H₂O and P(o-tol)₃, in a manner analogous to that described above, were not successful.^{3a}

Caution: Perchlorate salts of metal salts of organic ligands are potentially explosive. (See also detailed caution in ref 1.)

Crystallographic studies

Data were collected as described previously;⁴ experimental data are compiled in Table 1.

The crystals grew as thin plates; the largest we were able to produce $(0.3 \times 0.2 \times 0.05 \text{ mm}, \text{volume}$ approximately $3 \times 10^{-3} \text{ mm}^3$) was used for the singlecrystal x-ray diffraction study but gave rise to a rather weak diffraction pattern. Data were collected with a Siemens R3m/V diffractometer. The Laue symmetry (2/m) and the systematic absences (*hol* for l = 2n+1and 0k0 for k = 2n+1) uniquely define the ubiquitous centrosymmetric monoclinic space group $P2_1/c$. Data were collected for one hemisphere of reciprocal space (+*h*, $\pm k$, $\pm l$) to the limit of observable reflections ($2\theta = 35^\circ$; Mo $K\alpha$ radiation). The 4629 measured reflections were corrected for absorption and Lp factors and were reduced to a unique set of 2117 reflections ($R_{int} = 2.10\%$) of which 1495 were considered

	Fable 1	. Data	for	the	X-Ray	Diffraction	Study
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Compound	cis-[Ru(bpy) ₂ {PMe(o-tol) ₂ }Cl ⁺]
1	[ClO ₄]
CCDC deposit no.	CCDC-1003/5727
Color/shape	Red/plate
Chemical formula	$C_{35}H_{33}Cl_2N_4O_4PRu$
Formula weight	776.6
Temperature, K	295
Crystal system	Monoclinic
Space group	$P2_1/c$ (No. 14)
Unit cell dimensions	a = 9.375(2) Å
	b = 22.019(7) Å
	c = 16.153(4) Å
	$\beta = 90.83(2)$
Volume, Å ³	3333.9(16)
Ζ	4
Density (calculated), Mg/m ³	1.547
Absorption coefficient, mm ⁻¹	0.713
F(000)	1584
Diffractometer/scan	Siemens R3m/2 θ - θ
2θ range, deg./radiation	4.5 to 35.0/Mo <i>K</i> α(λ 0.71073Å)
Reflections measured	4629
Independent/observed re- flection	2117 ($R_{\text{int}} = 2.10\%$)/1495 [$I > 2\sigma(I)$]
Data/retraints/parameters	1495/0/249
Goodness-of-fit	1.42
Final <i>R</i> indices $[I > 2\sigma(I)]$	R1 = 0.0430, wR2 = 0.0361
<i>R</i> indices (all data)	R1 = 0.0731, wR2 = 0.0405
Largest diff. peak, hole (e^{-}/\mathring{A}^{3})	+0.39, -0.33

"observed" with $I > 2\sigma(I)$. Refinement led to R1 = 0.0430 for the observed data and RI = 0.0731 for all 2117 unique data. The limitations of the data set prompted us to refine anisotropically only the RuN₄ClP portion of the cation and atoms of the [ClO₄] anion. Carbon atoms were refined with isotropic thermal parameters and hydrogen atoms were included in calculated positions with $d(C-H) = 0.96\text{\AA}$.⁵ All calculations were performed under the SHELXTL PLUS (Release 4.11 (VMS))⁶ set of computer programs. The analytical scattering factors for neutral atoms^{7a} were corrected for both the real and imaginary components of anomalous dispersion.^{7b}

Discussion

The labeling of atoms is shown in Fig. 1, while the packing of *cis*-[Ru(bpy)₂{PMe(o-tol)₂}Cl⁺] cations and $[ClO_4^-]$ anions is illustrated in Fig. 2. The crystal



Fig. 1. Labeling of atoms for cis-[Ru(bpy)₂{PMe(o-tol)₂}Cl⁺] [ClO₄⁻]. This diagram shows 30% probability envelopes for thermal vibrations, with hydrogen atoms artificially reduced.

structure is stabilized by a series of weak $C-H\cdots O$ hydrogen bonds involving the aromatic C-H moieties of *o*-tolyl rings and perchlorate anions.

Final atomic coordinates and equivalent iso-



Fig. 2. Packing diagram for *cis*- $[Ru(bpy)_2\{PMe(o-tol)_2\}Cl^+]$ [ClO₄], projected on (100). (The *c*-axis is horizontal and the *b*-axis is vertical.) Note the C-H···O interactions.

tropic thermal parameters are given in Table 2. Selected interatomic distances and angles are provided in Table 3. The central ruthenium(II) cation has no elements of symmetry (point group C_1) and is chiral; the crystal as a whole contains an ordered racemic

Table 2. Final Atomic Coordinates $(\times 10^4)$ and Equivalent Isotropic Thermal Parameters $(\mathring{A}^2 \times 10^3)$ for *cis*-[Ru(bpy)₂{PMe (o-tol)₂}Cl⁺][ClO₄]

	x	у	z	U(eq)
(A) Atoms in	ruthenium(II) ca	tion		
Ru(1)	125(1)	2081(1)	526(1)	38(1)
P(1)	1011(3)	2993(1)	1098(2)	43(1)
Cl(1)	-622(3)	2519(1)	-787(2)	60(1)
N(11)	-640(10)	1265(3)	17(5)	43(4)
N(21)	1950(9)	1737(4)	-39(5)	42(4)
N(31)	-1746(8)	2272(3)	1133(5)	33(3)
N(41)	417(9)	1621(3)	1609(5)	33(3)
C(12)	308(11)	985(5)	-491(6)	39(3)
C(13)	-123(11)	451(5)	-914(6)	47(3)
C(14)	-1449(11)	228(5)	-806(6)	50(3)
C(15)	-2378(11)	505(5)	-289(6)	52(3)
C(16)	-1935(13)	1036(5)	134(7)	48(3)
C(22)	1707(11)	1241(5)	-529(6)	37(3)
C(23)	2835(12)	988(5)	-973(6)	54(4)
C(24)	4144(13)	1244(5)	-932(7)	63(4)
C(25)	4402(12)	1726(5)	-415(7)	61(4)
C(26)	3271(12)	1949(5)	23(6)	47(3)
C(32)	-1875(10)	2004(5)	1880(7)	39(3)
C(33)	-3040(11)	2125(5)	2392(6)	53(3)
C(34)	-4063(12)	2529(5)	2097(7)	64(4)
C(35)	-3967(11)	2785(5)	1337(6)	54(3)
C(36)	-2791(11)	2640(5)	861(6)	48(3)
C(42)	-702(12)	1610(5)	2127(7)	35(3)
C(43)	-724(11)	1226(5)	2818(6)	50(3)
C(44)	430(12)	875(5)	2988(7)	62(4)
C(45)	1598(12)	908(5)	2495(7)	51(3)
C(46)	1580(12)	1275(5)	1815(7)	47(3)
C(51)	2267(11)	3464(5)	528(6)	44(3)
C(52)	2951(12)	3984(5)	850(7)	59(4)
C(53)	3990(12)	4263(5)	367(7)	69(4)
C(54)	4304(13)	4059(6)	-425(8)	79(4)
C(55)	3573(12)	3571(5)	-747(8)	69(4)
C(56)	2534(11)	3286(5)	-291(6)	51(3)
C(57)	2678(12)	4272(5)	1670(7)	78(4)
C(61)	-363(11)	3527(4)	1479(6)	41(3)
C(62)	-1048(12)	3953(5)	973(7)	54(4)
C(63)	-2143(12)	4307(5)	1319(7)	66(4)
C(64)	-2553(13)	4209(5)	2109(8)	75(4)
C(65)	-1953(13)	3774(5)	2592(8)	74(4)
C(66)	-831(11)	3434(5)	2299(7)	54(4)
C(67)	-671(12)	4070(5)	87(7)	75(4)
C(71)	2033(10)	2809(5)	2040(5)	54(3)
(B) Atoms in	perchlorate anion	n		
C1(2A)	4470(4)	-559(2)	-1886(2)	59(2)
O(1A)	5286(10)	-50(5)	-1704(8)	139(5)
O(2A)	2993(9)	-436(3)	-1784(5)	78(4)
O(3A)	4707(11)	-716(5)	-2695(7)	146(6)
O(4A)	4878(10)	-1041(5)	-1380(7)	142(6)

$\begin{array}{c c c c c c c c c c c c c c c c c c c $					
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Ru(1) - P(1)	2.357(3)	Ru(1)-C1(1)	2.424(3)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Ru(1) - N(11)	2.098(8)	Ru(1) - N(21)	2.093(8)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Ru(1) - N(31)	2.065(8)	Ru(1) - N(41)	2.034(7)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	P(1) - C(51)	1.828(11)	P(1) - C(61)	1.855(10)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	P(1) - C(71)	1.831(9)	N(11) - C(12)	1.366(13)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	N(11) - C(16)	1.332(14)	N(21) - C(22)	1.366(13)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	N(21) - C(26)	1.326(14)	N(31) - C(32)	1.352(13)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	N(31) - C(36)	1.340(13)	N(41) - C(42)	1.352(14)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	N(41) - C(46)	1.368(14)	C(12) - C(13)	1.417(14)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(12) - C(22)	1.429(15)	C(13) - C(14)	1.350(15)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(14) - C(15)	1.360(15)	C(15) - C(16)	1.413(15)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(22) - C(23)	1.401(15)	C(23) - C(24)	1.351(16)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(24) - C(25)	1.369(15)	C(25) - C(26)	1.375(15)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(32) - C(33)	1.405(14)	C(32) - C(42)	1.452(15)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(33) - C(34)	1.389(15)	C(34) - C(35)	1.354(15)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(35) - C(36)	1.390(15)	C(42) - C(43)	1.400(15)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(43) - C(44)	1.354(15)	C(44) - C(45)	1.365(16)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(45) - C(46)	1.364(15)	C(51) - C(52)	1.409(15)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(51) - C(56)	1.405(14)	C(52) - C(53)	1.398(16)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(52) - C(57)	1.493(16)	C(53) - C(54)	1.392(17)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(54) - C(55)	1.373(17)	C(55) - C(56)	1.380(16)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(61) - C(62)	1.395(15)	C(61) - C(66)	1.416(15)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(62) - C(63)	1.411(16)	C(62) - C(67)	1.503(15)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C(63) - C(64)	1.354(17)	C(64) - C(65)	1.353(17)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(65) - C(66)	1.381(16)	C1(2A) - O(1A)	1.385(11)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C1(2A) - O(2A)	1.423(9)	C1(2A)-O(3A)	1.372(11)	
$\begin{array}{llllllllllllllllllllllllllllllllllll$	C1(2A) - O(4A)	1.390(11)			
$\begin{array}{llllllllllllllllllllllllllllllllllll$	P(1) - Ru(1) - Cl(1)	95.7(1)	P(1) - Ru(1) - N	J(11) 1	79.3(3)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Cl(1) - Ru(1) - N(11)) 84.5(2)	P(1)-Ru(1)-N	N(21) 1	01.2(2)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Cl(1) - Ru(1) - N(21)) 89.4(2)	N(11) - Ru(1) -	·N(21)	78.2(3)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	P(1) - Ru(1) - N(31)	86.4(2)	Cl(1) - Ru(1) - I	N(31)	95.6(2)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	N(11) - Ru(1) - N(3)	1) 94.2(3)	N(21) - Ru(1) -	·N(31) 1	70.5(3)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	P(1) - Ru(1) - N(41)	92.6(2)	Cl(1) - Ru(1) - I	N(41) 1	69.4(2)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	N(11) - Ru(1) - N(4)	1) 87.3(3)	N(21) - Ru(1) -	·N(41)	95.4(3)
Ru(1) - P(1) - C(61)115.3(3) $C(51) - P(1) - C(61)$ 105.3(5) $Ru(1) - P(1) - C(71)$ 108.3(3) $C(51) - P(1) - C(71)$ 102.2(5) $C(61) - P(1) - C(71)$ 102.9(4)102.9(4)	N(31) - Ru(1) - N(4)	1) 78.4(3)	Ru(1) - P(1) - C	2(51) 1	20.8(3)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Ru(1) - P(1) - C(61)	115.3(3)	C(51) - P(1) - C	2(61) 1	05.3(5)
C(61) - P(1) - C(71) 102 9(4)	Ru(1) - P(1) - C(71)	108.3(3)	C(51) - P(1) - C	2(71) 1	02.2(5)
C(01) = 1(1) = C(71) = 102.9(4)	C(61) - P(1) - C(71)	102.9(4)	· · · · ·	-	

 Table 3. Selected Interatomic Distances (Å) and Angles (°) for cis-[Ru(bpy)₂{PMe (o-tol)₂}Cl⁺][ClO₄⁻]

array of the two enantiomeric forms of this cation. The immediate coordination geometry about the metal atom may be regarded as a rather distorted octahedral arrangement. The bipyridyl ligands are associated with the reduced bite angles N(11)- $Ru(1)-N(21) = 78.2(3)^{\circ}$ and $N(31)-Ru(1)-N(41) = 78.4(3)^{\circ}$. The PMe(o-tol)₂ ligand is *trans* to one nitrogen atom, with $\angle P(1)-Ru(1)-N(11) = 179.3(3)^{\circ}$ and the other *trans* angles are reduced to $N(21)-Ru(1)-N(31) = 170.5(3)^{\circ}$ and $N(41)-Ru(1)-Cl(1) = 169.4(2)^{\circ}$.

The Ru-PMe(o-tol)₂ bond length is given by Ru(1)-P(1) = 2.357(3) Å and the *trans* Ru(1)-N(11) distance is 2.098(8) Å. The Ru-P-C(ipso) angles reflect the asymmetric nature of the PMe(o-tol)₂ ligand. Angles to the bulky o-tolyl groups $(\angle Ru(1) - P(1) - C(51) = 120.8(3)^{\circ}$ and $\angle Ru(1) - P(1) - C(61) = 115.3(3)^{\circ}$) are substantially larger than those to the sterically undemanding methyl group $(\angle Ru(1) - P(1) - C(71) = 108.3(3)^{\circ})$. The C-P-C angles behave analogously, albeit with smaller perturbations, with $\angle C(51) - P(1) - C(61) = 105.3(5)^{\circ}$ as compared to $\angle C(51) - P(1) - C(71) = 102.2(5)^{\circ}$ and $\angle C(61) - P(1) - C(71) = 102.9(4)^{\circ}$.

The Ru – P and Ru – N bond lengths in the present complex are compared to those in related Ru – $PMe_n(o-tol)_{3-n}$ (n = 1-3) complexes and to the related Ru – PPh_n(o-tol)_{3-n} (n = 1-3) complexes⁸⁻¹⁰ in Table 4. As can clearly be seen, the Ru – P distances increase steadily along the series Ru – PMe₃, Ru – PMe₂(o-

Cation	θ	Ru-P	trans-Ru-N	∠P-Ru-Cl	Ref
cis-[Ru(bpy) ₂ (PMe ₃)Cl ⁺]	118	2.310(2)	2.112(6)	86.4(1)	1
<i>cis</i> -[Ru(bpy) ₂ (PMe ₂ (o-tol))Cl ⁺]	141	2.324(2)	2.124(5)	90.1(1)	1
<i>cis</i> -[Ru(bpy) ₂ (PMe(o-tol) ₂]Cl ⁺]	169	2.357(3)	2.098(8)	95.7(1)	This work
<i>cis</i> -[Ru(bpy)(PPh ₃)Cl ⁺]	145	2.328(1)	2.109(4)	93.8(1)	8
cis -[Ru(bpy){PPh ₂ (o-tol)}Cl ⁺]	161	2.360(3)	2.092(7)	94.1(1)	9
cis-[Ru(bpy){PPh(o-tol) ₂ }Cl ⁺]	178	2.382(2)	2.088(7)	97.3(1)	10

Table 4. Ligand Cone Angles $(\theta^{\circ})^{a}$, Ru-P and the trans Ru-N distances and cis-P-Ru-Cl angles in some cis-[Ru(bpy)₂(phosphine)Cl⁺] cations

^a Cone angle of phosphine ligand (see refs 3b and 11).

tol), $Ru - PMe(o-tol)_2$ (for which the phosphine cone angles are respectively 118°, 143°, and 169°)¹¹ and along the series $Ru - PPh_3$, $Ru - PPh_2(o-tol)$, $Ru - PPh(o-tol)_2$ (for which the ligand cone angles are 145°, 161°, and 178°). The analogous complex with a $Ru - P(o-tol)_3$ ligand (cone angle 194°) has eluded synthesis, but, if it ever could be prepared, it would be expected to have an unusually long Ru - P linkage.

The steric, rather than electronic, cause of these results is confirmed by a study of the *cis*-P-Ru-Cl angles in these two series' of compounds. This angle expands steadily along each series: $86.4(1)^{\circ}$ for \angle Cl-Ru-PMe₃, $90.1(1)^{\circ}$ for \angle Cl-Ru-PMe₂(o-tol), $95.7(1)^{\circ}$ for \angle Cl-Ru-PMe(o-tol)₂; $93.8(1)^{\circ}$ for \angle Cl-Ru-PPh₃, $94.1(1)^{\circ}$ for \angle Cl-Ru-PPh₂(o-tol) and $97.3(1)^{\circ}$ for \angle Cl-Ru-PPh(o-tol)₂. This bond angle correlates strongly with θ , the cone angle of the phosphine ligand.

Although studies on the $Ru - PPh_n(o-tol)_{3-n}$ derivatives (n = 1-3) have been published previously, all three structures contained unusual combinations of solvent molecules of crystallization.⁸⁻¹⁰ The present $Ru - PMe_n(o-tol)_{3-n}$ derivatives confirm our findings for a series of complexes uncontaminated by solvent of crystallization.

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