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"Structure of Trimethylplatinum (IV) with a Tripod Ligand"

by

R. E. Marsh, W. P. Schaefer, D. K. Lyon, J. A. Labinger and J. E. Bercaw

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13. ABSTRACT (Maximum 200 words) [Cyclopentadienylcobalt tris(bis-methoxyphosphito)]trimethylplatinum(IV), PtCoP3O9C14H32; Mr = 691.35; triclinic, $P\bar{1}$; $a = 9.106(3)$, $b = 14.803(3)$, $c = 15.147(3)$ Å, $\alpha = 112.95(2)$, $\beta = 103.68(2)$, $\gamma = 95.10(2)^\circ$, $V = 1788.9(9)$ Å ³ , $Z = 3$, $D_x = 1.93$ g cm ⁻³ , Mo K α , 0.71073 Å, $\mu = 68.69$ cm ⁻¹ , $F(000) = 1014$, room temperature, $R = 0.038$ for 4620 reflections with $F_o^2 > 3\sigma(F_o^2)$. The trimethylplatinum(IV) completes octahedral coordination by bonding to three oxygen atoms of the tripod-shaped methoxy Kläui ligand. There are two independent molecules in the cell, one disordered about a center of symmetry. The ordered molecule has normal bond distances and angles; Pt-c = 2.001[11]Å and Pt-O = 2.173[5] Å. Many distances in the disordered molecule are uncertain, particularly in the areas of the Cp carbon atoms and the CH ₃ groups, which overlap in the two orientations.				
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Structure of Trimethylplatinum(IV) with a Tripod Ligand

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Abstract. [Cyclopentadienylcobalt tris(bis-methoxyphosphito)]trimethylplatinum(IV), $\text{PtCoP}_3\text{O}_9\text{C}_{14}\text{H}_{32}$; $M_r = 691.35$; triclinic, $P\bar{1}$; $a = 9.106(3)$, $b = 14.803(3)$, $c = 15.147(3)$ Å, $\alpha = 112.95(2)$, $\beta = 103.68(2)$, $\gamma = 95.10(2)^\circ$, $V = 1788.9(9)\text{Å}^3$, $Z = 3$, $D_x = 1.93 \text{ g cm}^{-3}$, $\text{MoK}\alpha$, 0.71073Å , $\mu = 68.69 \text{ cm}^{-1}$, $F(000) = 1014$, room temperature, $R = 0.038$ for 4620 reflections with $F_o^2 > 3\sigma(F_o^2)$. The trimethylplatinum(IV) completes octahedral coordination by bonding to three oxygen atoms of the tripod-shaped methoxy Kläui ligand. There are two independent molecules in the cell, one disordered about a center of symmetry. The ordered molecule has normal bond distances and angles; $\text{Pt-C} = 2.001[11]\text{Å}$ and $\text{Pt-O} = 2.173[5] \text{Å}$. Many distances in the disordered molecule are uncertain, particularly in the areas of the Cp carbon atoms and the CH_3 groups, which overlap in the two orientations.

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Introduction. Interest in the activation and functionalization of saturated hydrocarbons by aqueous platinum chloride solutions (Shilov, 1984 and 1989; Labinger, Herring and Bercaw, 1990 and 1990b) has led to our synthesizing model platinum complexes containing "hard" ligands (Herring, Henling, Labinger and Bercaw, 1991). Model complexes analogous to the putative $\text{Pt}(\text{R})\text{Cl}_4(\text{H}_2\text{O})^-$ species that have been proposed as intermediates in the activation/functionalization of alkanes by platinum (Shul'pin, Shilov, Kitaigorodskii and Zeile Krevor, 1980; Shibaera, Rozenberg, Lobkovskaya, Shilov and Shul'pin, 1981; Shul'pin, Nizova, Nikitaev, 1984; Khrushch, Laurushki, Misharin, Moravsky and Shilov, 1983) are thus attractive targets, but few examples of structurally well-characterized complexes are available. The tripod ligand $(\eta^5\text{-C}_5\text{H}_5)\text{Co}[\text{P}(\text{O})(\text{OR})_2]_3^-$ (henceforth abbreviated L_{OR}) behaves as a hard ligand, with field strengths approximately equal to that of three F^- ligands (Kläui, 1990). Recently the synthesis of $\text{Pt}(\text{CH}_3)_3\text{L}_{\text{OR}}$, where $\text{R} = \text{Me}$ and Et , as well as the crystal and molecular structure of the latter, have been reported (Nettle, Valderrama, Contreras, Scotti, Peters, Von Schnering and Werner, 1988). In the course of preparing these complexes for study of their chemical behavior, we obtained crystals of $\text{Pt}(\text{CH}_3)_3\text{L}_{\text{OMe}}$, and decided to determine its structure for purposes of comparison. Formation of methanol during aqueous decomposition of $\text{Pt}(\text{CH}_3)_3\text{L}_{\text{OR}}$ will be reported elsewhere.

Experimental. Crystal a truncated bipyramid, $0.34 \times 0.30 \times 0.30$ mm; CAD-4 diffractometer, $\theta - 2\theta$ scans; 25 reflections with $36^\circ < 2\theta < 38^\circ$ used for cell dimensions; no absorption correction (psi scans showed relative transmissions of 0.97 to 1.03, and the uncorrected data merged with a goodness of fit of 1.04); $(\sin\theta/\lambda)_{\text{max}} = 0.60$; h from -10 to 10 , k from -17 to 17 , l from -18 to 18 ; three standard reflections ($\bar{1}24$, $\bar{3}31$ and $0\bar{1}4$) showed a slight isotropic decay that we corrected for; 12,652 reflections measured, 6284 independent; R_{int} for 5449 reflections with exactly two observations, 0.030; all reflections used in solution and refinement of the structure; Pt, Co atom locations deduced from a Patterson map, locations of remaining heavy atoms found from successive structure factor-Fourier

calculations; F^2 values used in refinement, hydrogen atoms on ordered molecule placed by calculation (C-H, 0.95 Å) assuming staggered geometry; hydrogen atom parameters not refined, but included as constant contributions to the structure factors; no hydrogen atoms introduced on disordered molecule; coordinates and anisotropic displacement parameters of all heavy atoms of the ordered molecule, a scale factor and a secondary extinction parameter (Larson, 1967) were refined. In the disordered molecule the Co and Pt atoms, which are separated by 0.65 Å, were refined satisfactorily as half-weight, anisotropic atoms. The three P's were also refined as half-weight, anisotropic atoms, and the nine O's as half-weight isotropic atoms. On the other hand, the six C atoms of the methoxy groups appeared to be closely overlapped in the two orientations, and were refined as three full-weight, anisotropic atoms. Overlap of the five atoms of the Cp ring with the three Pt-coordinated methyl groups was complicated; these atoms were placed in idealized, half-populated sites with isotropic B's and not refined. The final difference map clearly showed that the modeling in this area was incomplete, presumably because of the neglect of the large anisotropies in the U_{ij} 's expected for such atoms. Final R on F for all 5754 reflections with $F_o^2 > 0$, 0.051, wR (on F^2), 0.008, S = 1.81; weights taken as $1/\sigma^2(F_o^2)$; variances ($\sigma^2(F_o^2)$) derived from counting statistics plus an additional term, $(0.014I)^2$; variances of the merged data by propagation of error plus another additional term, $(0.014\bar{I})^2$; ratio of maximum shift to standard deviation in the final cycle, 0.03; maximum and minimum peaks in final difference map, 1.33 eÅ^{-3} (near Cp ring on disordered molecule – the five largest peaks are in this region) and -1.01 eÅ^{-3} (0.7 Å from P3); secondary extinction parameter refined to $0.94(3) \times 10^{-6}$; atomic scattering factors from Cromer and Waber (1974) and dispersion corrections from Cromer (1974); programs used were those of the CRYM Crystallographic Computing System (Duchamp, 1964) and ORTEP (Johnson, 1976). Final refined parameters are listed in Table 1 with selected distances and angles in Table 2. The two independent molecules are shown in Figs. 1 and 2; Fig. 3 shows the packing.*

* Lists of assigned hydrogen parameters, anisotropic displacement parameters, complete

Discussion. The molecular structure of $\text{Pt}(\text{CH}_3)_3\text{LOMe}$ is quite similar to that previously reported for the ethyl analog, which in turn displays structural parameters closely resembling those of other complexes containing the LOR tripod ligand as well as those with the PtMe_3 moiety (Nettle, *et al.*, 1988). Comparing bond distances and angles between the two $\text{Pt}(\text{CH}_3)_3\text{LOR}$ structures, nearly all agree to within experimental uncertainty. The only apparently significant differences are found in the $\text{P}=\text{O}-\text{Pt}$ linkages: The average values for $\text{Pt}-\text{O}$, $\text{O}-\text{P}$, and $\text{Pt}-\text{O}-\text{P}$ are 2.196(1) Å, 1.507(8) Å and 125.1(9)° for $\text{Pt}(\text{CH}_3)_3\text{LOEt}$; and 2.173(5) Å, 1.492(6) Å and 129.2(3)° for the ordered molecule of $\text{Pt}(\text{CH}_3)_3\text{LOMe}$, respectively. (Values for the disordered molecule are intermediate, but the uncertainties in these parameters are considerably larger, and their significance is dubious.) Increased steric crowding around the set of $\text{P}=\text{O}$ ligands could result in a greater separation between the tripod ligand and the Pt center and the observed increase in bond lengths for $\text{Pt}(\text{CH}_3)_3\text{LOEt}$, but this interpretation predicts an increase in $\text{Pt}-\text{O}-\text{P}$ angles, whereas a decrease is found.

The crystal structure of $\text{Pt}(\text{CH}_3)_3\text{LOMe}$ exhibits an interesting feature: of the two independent molecules in the unit cell, one is disordered about a center of symmetry (Fig. 2). It may be noteworthy that each of the six C atoms of the methoxy groups virtually overlaps its partner in the other orientation of the disordered molecule; perhaps the breaking of this pseudosymmetry element by additional substitution is responsible for the fact that no such disorder was found for the ethyl analog.

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distances and angles, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP XXXXX (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, ENGLAND.

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Legends to Figures

- Figure 1. An ORTEP drawing of the ordered cation with 50% probability ellipsoids showing the numbering system. Hydrogen atoms are not shown.
- Figure 2. An ORTEP drawing of the disordered cation with 50% probability ellipsoids showing the numbering system. Primed atoms are related to unprimed ones by a center of symmetry.
- Figure 3. An ORTEP packing drawing projected approximately down a showing the contents of a unit cell, with a unit cell outlined. The c axis is horizontal. Atoms are shown as 50% probability ellipsoids, with Co and Pt atoms of the ordered cation shaded; hydrogen atoms are not shown. Only one orientation of the disordered cation is shown.

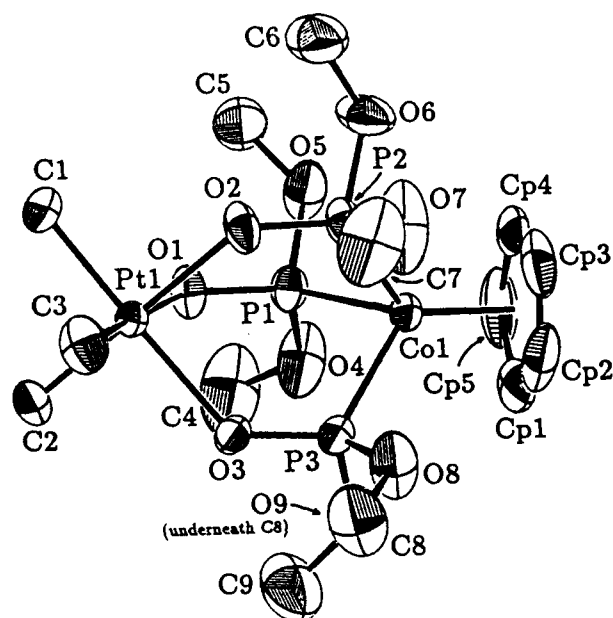


Figure 1. Marsh, Schaefer, Lyon & Bercau

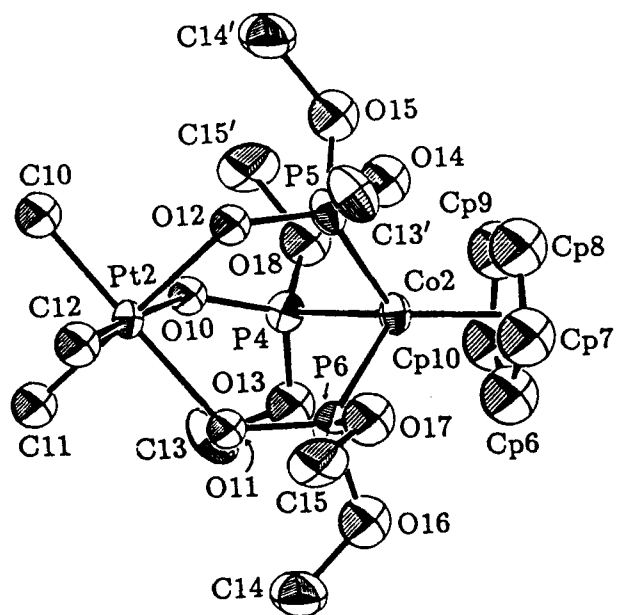


Figure 2. Marsh, Schrofer, Lyon & Bercau

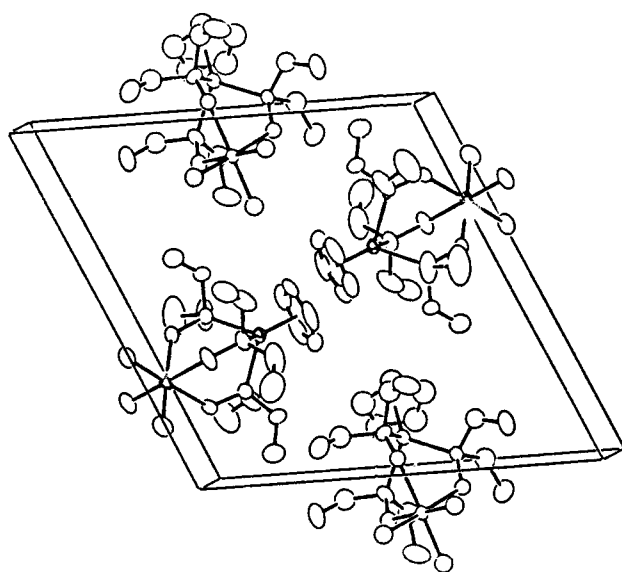


Figure 3. Marsh, Schaefer, Lyon, & Decca

Table 1. Final Refined Parameters for
Platinum(IV) Trimethyl · Klauī.

x, y, z and $U_{eq}^a \times 10^4$				
Atom	x	y	z	U_{eq} or B
Pt1	2511(.4)	83(.2)	2764(.3)	480(1)
Co1	1646(1)	2860(1)	3835(1)	461(2)
P1	1247(3)	1786(2)	4439(2)	562(5)
P2	3894(3)	2538(2)	3770(2)	637(6)
P3	563(3)	1736(2)	2313(2)	637(6)
O1	1582(7)	757(4)	4009(4)	660(15)
O2	4151(6)	1493(4)	3355(5)	768(19)
O3	893(7)	700(4)	1966(4)	757(19)
C1	3991(12)	-443(7)	3551(8)	958(34)
C2	988(11)	-1194(6)	2237(8)	853(33)
C3	3368(12)	-498(7)	1599(8)	913(31)
O4	-508(8)	1675(6)	4433(7)	1252(26)
O5	2122(10)	2288(5)	5607(5)	1122(29)
O6	5049(7)	3125(5)	4892(6)	1172(29)
O7	4580(11)	3103(6)	3232(8)	1516(31)
O8	918(11)	2203(5)	1579(5)	1254(31)
O9	-1258(8)	1631(5)	2068(6)	1168(29)
Cp1	7(14)	3730(8)	3829(18)	1157(53)
Cp2	1163(32)	4061(11)	3524(10)	1188(51)
Cp3	2464(17)	4364(8)	4272(19)	1140(52)

Table 1. (Cont.)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} or <i>B</i>
Cp4	2172(24)	4224(9)	5059(10)	1102(52)
Cp5	615(27)	3823(10)	4782(17)	1122(46)
C4	-1368(17)	858(12)	4427(16)	1977(70)
C5	2962(16)	1859(9)	6117(9)	1235(48)
C6	6446(14)	2898(10)	5192(9)	1239(45)
C7	5176(21)	2723(13)	2487(14)	1764(61)
C8	841(19)	1643(10)	591(9)	1464(55)
C9	-2308(16)	774(11)	1611(14)	1742(69)
Pt2	6392(1)	4794(1)	-1149(1)	432(2)
Co2	4044(4)	5141(3)	853(3)	496(7)
P4	4576(5)	6252(3)	315(3)	500(10)
P5	3303(5)	3954(3)	-666(4)	628(12)
P6	6382(5)	4890(4)	1072(3)	586(11)
O10	5174(12)	6014(7)	-570(8)	4.4(2) *
O11	7285(12)	4954(8)	383(8)	4.5(2) *
O12	4351(12)	3774(8)	-1325(8)	4.8(2) *
O13	5742(15)	7213(10)	1257(10)	6.6(3) *
O14	2668(16)	2880(11)	-738(11)	7.8(3) *
O15	1678(16)	4155(11)	-1246(11)	7.7(3) *
O16	7445(18)	5615(11)	2199(11)	8.1(4) *

Table 1. (Cont.)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} or <i>B</i>
O17	6316(17)	3796(11)	1048(11)	8.1(4) *
O18	3124(16)	6774(10)	94(11)	7.6(3) *
C13	6636(12)	7956(7)	1095(9)	955(34)
C14	8953(13)	6202(9)	2375(8)	1157(43)
C15	7574(12)	3243(9)	834(9)	1049(34)

$$^a U_{eq} = \frac{1}{3} \sum_i \sum_j [U_{ij} (a_i^* a_j^*) (\vec{a}_i \cdot \vec{a}_j)]$$

* Isotropic displacement parameter, *B*

Table 2. Selected Distances and Angles for
Platinum(IV) Trimethyl · Klau.

Distance(Å)		Distance(Å)	
Pt1 -O1	2.168(6)	P3 -O3	1.498(7)
Pt1 -O2	2.178(6)	P3 -O8	1.596(9)
Pt1 -O3	2.174(6)	P3 -O9	1.591(8)
Pt1 -C1	1.998(11)	O4 -C4	1.37(2)
Pt1 -C2	1.991(10)	O5 -C5	1.331(16)
Pt1 -C3	2.013(11)	O6 -C6	1.365(15)
Co1 -P1	2.166(3)	O7 -C7	1.32(2)
Co1 -P2	2.159(3)	O8 -C8	1.380(18)
Co1 -P3	2.168(3)	O9 -C9	1.337(19)
Co1 -Cp1	2.057(17)	Cp1 -Cp2	1.37(3)
Co1 -Cp2	2.07(2)	Cp1 -Cp5	1.37(3)
Co1 -Cp3	2.066(19)	Cp2 -Cp3	1.33(3)
Co1 -Cp4	2.057(17)	Cp3 -Cp4	1.37(3)
Co1 -Cp5	2.06(2)	Cp4 -Cp5	1.38(3)
P1 -O1	1.497(6)	Pt2 -O10	2.201(11)
P1 -O4	1.592(9)	Pt2 -O11	2.178(11)
P1 -O5	1.587(8)	Pt2 -O12	2.181(11)
P2 -O2	1.489(7)	Pt2 -C10	2.018
P2 -O6	1.609(8)	Pt2 -C11	2.020
P2 -O7	1.560(11)	Pt2 -C12	2.016

Table 2. (Cont.)

Distance(Å)		Distance(Å)	
Co2 -P4	2.167(6)	O16 -C14	1.47(2)
Co2 -P5	2.177(6)	O17 -C15	1.49(2)
Co2 -P6	2.164(6)	O18 -C15'	1.39(2)
Co2 -Cp6	2.089		
Co2 -Cp7	2.089		
Co2 -Cp8	2.089		
Co2 -Cp9	2.090		
Co2 -Cp10	2.089		
P4 -O10	1.492(12)		
P4 -O13	1.619(15)		
P4 -O18	1.622(16)		
P5 -O12	1.505(12)		
P5 -O14	1.594(16)		
P5 -O15	1.649(17)		
P6 -O11	1.498(12)		
P6 -O16	1.615(17)		
P6 -O17	1.601(17)		
O13 -C13	1.441(18)		
O14 -C13'	1.417(19)		
O15 -C14'	1.52(2)		

Table 2. (Cont.)

Angle(°)		Angle(°)	
O2 -Pt1 -O1	88.1(2)	O5 -P1 -Co1	108.6(3)
O3 -Pt1 -O1	86.5(2)	O4 -P1 -O1	108.0(4)
C1 -Pt1 -O1	91.3(4)	O5 -P1 -O1	107.6(4)
C2 -Pt1 -O1	91.0(3)	O5 -P1 -O4	101.3(5)
C3 -Pt1 -O1	177.9(4)	O2 -P2 -Co1	122.0(3)
O3 -Pt1 -O2	87.8(2)	O6 -P2 -Co1	106.9(3)
C1 -Pt1 -O2	91.3(4)	O7 -P2 -Co1	109.5(4)
C2 -Pt1 -O2	179.0(3)	O6 -P2 -O2	108.0(4)
C3 -Pt1 -O2	90.6(4)	O7 -P2 -O2	107.9(5)
C1 -Pt1 -O3	177.7(4)	O7 -P2 -O6	100.4(5)
C2 -Pt1 -O3	91.8(3)	O3 -P3 -Co1	121.6(3)
C3 -Pt1 -O3	91.9(4)	O8 -P3 -Co1	107.1(3)
C2 -Pt1 -C1	89.1(4)	O9 -P3 -Co1	108.9(3)
C3 -Pt1 -C1	90.3(4)	O8 -P3 -O3	108.5(4)
C3 -Pt1 -C2	90.3(4)	O9 -P3 -O3	107.6(4)
P2 -Co1 -P1	91.7(1)	O9 -P3 -O8	101.3(4)
P3 -Co1 -P1	91.4(1)	P1 -O1 -Pt1	128.9(4)
P3 -Co1 -P2	91.7(1)	P2 -O2 -Pt1	129.4(4)
O1 -P1 -Co1	122.2(3)	P3 -O3 -Pt1	129.3(4)
O4 -P1 -Co1	107.1(3)	C4 -O4 -P1	125.2(10)

Table 2. (Cont.)

Angle(°)				Angle(°)			
C5	-O5	-P1	126.1(8)	C11	-Pt2	-O12	177.2
C6	-O6	-P2	123.0(8)	C12	-Pt2	-O12	92.7
C7	-O7	-P2	127.1(11)	C11	-Pt2	-C10	90.1
C8	-O8	-P3	124.1(9)	C12	-Pt2	-C10	90.2
C9	-O9	-P3	126.2(9)	C12	-Pt2	-C11	90.1
Cp5	-Cp1	-Cp2	108.8(17)	P5	-Co2	-P4	91.4(2)
Cp3	-Cp2	-Cp1	108.0(18)	P6	-Co2	-P4	91.3(2)
Cp4	-Cp3	-Cp2	109.2(17)	P6	-Co2	-P5	91.6(2)
Cp5	-Cp4	-Cp3	107.7(16)	O10	-P4	-Co2	122.1(5)
Cp4	-Cp5	-Cp1	106.3(18)	O13	-P4	-Co2	107.5(6)
O11	-Pt2	-O10	87.9(4)	O18	-P4	-Co2	111.8(6)
O12	-Pt2	-O10	86.8(4)	O13	-P4	-O10	108.8(7)
C10	-Pt2	-O10	92.2	O18	-P4	-O10	105.4(7)
C11	-Pt2	-O10	90.4	O18	-P4	-O13	98.8(8)
C12	-Pt2	-O10	177.6	O12	-P5	-Co2	121.1(5)
O12	-Pt2	-O11	88.5(4)	O14	-P5	-Co2	114.1(6)
C10	-Pt2	-O11	178.3	O15	-P5	-Co2	106.2(6)
C11	-Pt2	-O11	91.6	O14	-P5	-O12	105.5(8)
C12	-Pt2	-O11	89.7	O15	-P5	-O12	108.8(8)
C10	-Pt2	-O12	89.8	O15	-P5	-O14	98.9(8)

Table 2. (Cont.)

Angle(°)			
O11	-P6	-Co2	121.2(5)
O16	-P6	-Co2	110.6(6)
O17	-P6	-Co2	106.7(6)
O16	-P6	-O11	106.6(8)
O17	-P6	-O11	107.2(8)
O17	-P6	-O16	103.1(9)
P4	-O10	-Pt2	126.2(7)
P6	-O11	-Pt2	127.7(7)
P5	-O12	-Pt2	127.9(7)
C13	-O13	-P4	120.5(10)
C13'	-O14	-P5	123.3(12)
C14'	-O15	-P5	125.1(11)
C14	-O16	-P6	120.6(12)
C15	-O17	-P6	120.7(11)
C15'	-O18	-P4	124.1(12)