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# A second solid-state isomer of $[(\text{PPh}_3)_2\text{N}][\text{Ru}_6\text{C}(\text{CO})_{15}(\text{C}_3\text{H}_5)]$ : shift of the carbonyl and allyl positions on large cluster cores by crystal-packing force

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## Abstract

Treatment of  $[(\text{PPh}_3)_2\text{N}][\text{Ru}_6\text{C}(\text{CO})_{15}(\text{C}_3\text{H}_5)]$  in hot methanol gave crystals in the triclinic space group  $P\bar{1}$  (**2**), which is a solid-state isomer of the same compound as previously obtained from  $\text{CH}_2\text{Cl}_2$ –methanol at  $-20^\circ\text{C}$  in the monoclinic space group  $P2_1/a$  (**1**). In solutions, **1** and **2** have the same IR and variable-temperature  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra. In the solid state, the carbonyl stereochemistries are different: **1** possesses one bridging and one semi-bridging carbonyl ligand, while **2** has a single bridging carbonyl ligand. Taking the carbonyl bridge as standard, the  $\mu, \eta^3$ -allyl group occupies the same edge of the Ru-octahedron in both isomers, but its position in **2** is shifted significantly from that of **1**, so that the three carbon atoms of the allyl group are on the same side of the equatorial plane which contains that edge.

**Keywords:** Ruthenium; Cluster; Isomer; Carbonyl; Allyl; Bridging ligand

## 1. Introduction

In recent years, isomeric structures of organometallic molecules in crystals have been recognized as important in many instances. They reflect flexibility or ‘softness’ of a molecule since the crystal-packing force, which brings about a solid-state isomerism, should be fairly weak in general and a ‘harder’ molecule would have less tendency for such isomerization. In other words, crystal forces can stabilize less stable conformers by compensating intramolecular energy, and this can happen only in the region where the intramolecular potential energy surface is flat enough. Of particular interest are the ligand positions in cluster complexes, since solid-state isomerism provides visual information about the ease of ligand movement in the cluster core, which is difficult to obtain by other spectroscopic techniques. Most of the examples of solid-state isomerization in clusters are those concerning carbonyl ligands. Different positions and directions of terminal, bridging, and

semi-bridging carbonyls have been found in two or more solid-state isomers of  $[\text{Fe}_5\text{N}(\text{CO})_{14}]^-$  [1],  $\text{HRu}_6\text{-B}(\text{CO})_{17}$  [2],  $\text{Ru}_6\text{C}(\text{CO})_{17}$  [3],  $[\text{Rh}_{11}(\text{CO})_{23}]^{3-}$  [4],  $\text{Ru}_5\text{-S}(\text{CO})_{15}$  [5],  $[\text{Fe}_4(\text{CO})_{13}]^{2-}$  [6],  $[\text{Ru}_6\text{C}(\text{CO})_{16}]^{2-}$  [7], and  $[\text{HRu}_6\text{C}(\text{CO})_{16}]^-$  [8]. The case of  $\text{Ir}_6(\text{CO})_{16}$  is exceptional in that the two isomers, one with four face-bridging CO and the other four edge-bridging CO, do not interconvert in solution even at  $100^\circ\text{C}$  [9]. In  $[\text{H}_3\text{Ru}_4(\text{CO})_{12}]^-$  the two isomers differ in their hydride bridges positions [10].

In a previous paper we reported the synthesis and molecular structure of the allyl complex of monoanionic hexaruthenium carbonyl cluster as a PPN salt,  $[\text{PPN}][\text{Ru}_6\text{C}(\text{CO})_{13}(\mu\text{-CO})_2(\mu, \eta^3\text{-C}_3\text{-H}_5)]$  (**1**) [11], which was crystallized from  $\text{CH}_2\text{Cl}_2$ –MeOH at  $-20^\circ\text{C}$ . On crystallizing the same complex from hot methanol, we have observed that apparently different crystals (**2**) grew. Though **1** and **2** gave the same IR and NMR spectra in solutions, we decided to undertake X-ray diffraction study of this isomer in order to examine if the carbonyl and allyl coordination could differ from **1** in any way. The change of allyl coordination in high-nuclearity clusters seemed particularly in-

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teresting since it might be relevant to the mobility of small hydrocarbon molecules on metal surfaces.

## 2. Results and discussion

On cooling a concentrated solution of  $[\text{PPN}][\text{Ru}_6\text{C}(\text{CO})_{15}(\text{C}_3\text{H}_5)]$  in hot methanol to room temperature, rhombic reddish–brown crystals (**2**) were obtained. Our previous isomer (**1**), reddish–brown rectangular crystals, had been grown from  $\text{CH}_2\text{Cl}$ –methanol at  $-20^\circ\text{C}$ . While the solid-state IR spectra of **1** and **2** in the CO stretching region show small differences, the  $^1\text{H}$  NMR and IR spectra of **2** in solutions were completely identical to those of **1**. Moreover, the  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra at low ( $-90^\circ\text{C}$ ) and high ( $+70^\circ\text{C}$ ) temperatures confirmed the presence of a single species at different temperatures.

A crystal of **2** was subjected to a single crystal X-ray analysis, showing that the space group ( $P\bar{1}$ ) was different from that of **1** ( $P2_1/a$ ). Complex **2** has been revealed to be  $[\text{PPN}][\text{Ru}_6\text{C}(\text{CO})_{14}(\mu\text{-CO})(\mu,\eta^3\text{-C}_3\text{H}_5)]$ . The cluster anion structures of **1** and **2** are compared in Fig. 1. Selected intra- and intermolecular distances and bond angles for the two complexes are listed in Table 1.

The Ru–Ru bond lengths differ in the range 2.843(1)–2.954(1) Å in **1** and 2.818(1)–2.979(1) Å in **2**, though the average values happen to be the same (2.897 Å) in both complexes. The cluster framework, therefore, is more distorted in **2** than in **1**. The ‘soft’ nature of transition metal cluster skeletons has already been pointed out [12].

The most obvious difference in the CO coordination is observed at the Ru(4)–Ru(6) edge. In **1**, moderate interaction exists between Ru(4) and the carbonyl carbon C(64) (2.497(10) Å), so that the CO may be assigned as semi-bridging (Ru(6)–C(64) 1.924(10) Å,

Ru(6)–C(64)–O(64)  $154.9(9)^\circ$ ), while in **2** the corresponding CO ligand coordinates only to Ru(6) as a terminal carbonyl (Ru(6)–C(63)–O(63)  $175.8(7)^\circ$ ). Both complexes have a typical bridging CO at the Ru(3)–Ru(4) edge. This strongly suggests that the interconversion of the terminal and bridging coordination modes of CO between Ru(4) and Ru(6) is a low energy process. Since the cluster molecule which has bridging CO at Ru(3)–Ru(5) in place of Ru(3)–Ru(4) must present equal probability, bridging and unbridging of CO at Ru(5)–Ru(6) should be equally facile. Considering the recent report on an easy rotameric isomerism of the tricarbonyl ruthenium units in  $\text{Ru}_6\text{C}(\text{CO})_{17}$ , which is induced even by crystal packing force [3], the present observation may be relevant to the process of CO ligand scrambling in carbonyl clusters. The  $^{13}\text{C}$  NMR spectrum showed only a singlet in the carbonyl region at  $-90^\circ\text{C}$ .

Interestingly, the allyl ligand bridging Ru(1) and Ru(2) in each complex differs significantly in its position. The coordination in **1** appears to be more normal: the distances to the equatorial plane Ru(1)–Ru(2)–Ru(4)–Ru(5) are C(1) +0.465(9), C(2) –0.134(9), and C(3) +0.587(9) Å. In contrast, the corresponding values in **2** are C(1) +0.629(8), C(2) +0.065(8), and C(3) +0.773(8) Å, meaning that the three allyl carbons are located on the same side of this equatorial plane. The dihedral angles between the allyl plane and the same equatorial plane are  $90.5(9)^\circ$  in **1** and  $82.2(8)^\circ$  in **2**. The distances to the center C(0) of the cluster frame in **1** are: C(1) 3.71(1), C(2) 3.49(1), C(3) 3.81(1) Å and in **2**: C(1) 3.70(1), C(2) 3.57(1), C(3) 3.83(1) Å. Therefore, only C(2) moves a little away from the cluster center on going from **1** to **2**. All of these data indicate clearly that the change of the allyl moiety is the shift towards the apical metal atom Ru(6) and simultaneous tilting, movement which has a slight tendency from an edge-bridging allyl ( $\mu_2$ -allyl) towards a face-capping allyl ( $\mu_3$ -allyl).

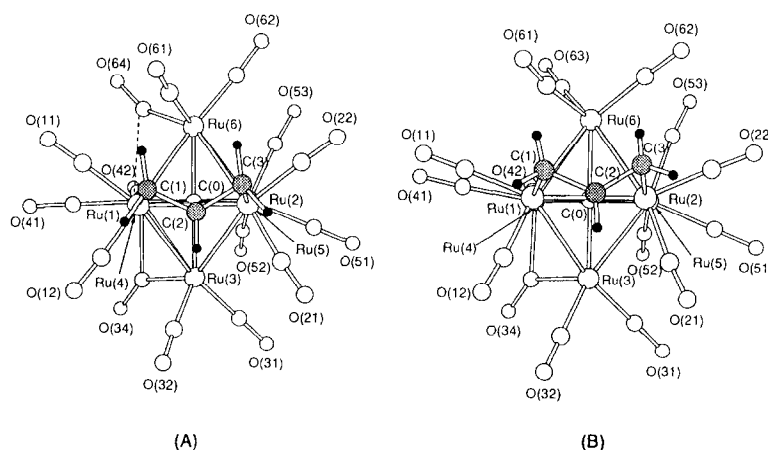


Fig. 1. Perspective view of (A)  $[\text{Ru}_6\text{C}(\text{CO})_{13}(\mu\text{-CO})_2(\mu,\eta^3\text{-C}_3\text{H}_5)]$  (anion of **1**) and (B)  $[\text{Ru}_6\text{C}(\text{CO})_{14}(\mu\text{-CO})(\mu,\eta^3\text{-C}_3\text{H}_5)]$  (anion of **2**). The first digit of each oxygen number is the number of the ruthenium atom to which the carbonyl is attached. The carbonyl carbons have the same numbering as their oxygen counterparts.

Table 1

Selected bond lengths (Å) and angles (°) for [PPN][Ru<sub>6</sub>C(CO)<sub>13</sub>(μ-CO)<sub>2</sub>(μ<sup>3</sup>-C<sub>3</sub>H<sub>5</sub>)] (**1**) and [PPN][Ru<sub>6</sub>C(CO)<sub>14</sub>(μ-CO)(μ<sup>3</sup>-C<sub>3</sub>H<sub>5</sub>)] (**2**)

	<b>1</b>	<b>2</b>
<i>(a) Intramolecular distances and angles</i>		
Ru(1)–Ru(2)	2.9539(11)	2.8926(9)
Ru(1)–Ru(3)	2.9253(11)	2.9387(8)
Ru(1)–Ru(4)	2.8676(11)	2.9204(9)
Ru(1)–Ru(6)	2.9043(12)	2.9228(9)
Ru(2)–Ru(3)	2.9416(12)	2.9794(9)
Ru(2)–Ru(5)	2.8536(11)	2.8413(9)
Ru(2)–Ru(6)	2.9102(11)	2.8940(8)
Ru(3)–Ru(4)	2.8428(12)	2.8181(7)
Ru(3)–Ru(5)	2.8456(12)	2.8631(9)
Ru(4)–Ru(5)	2.9227(11)	2.9311(9)
Ru(4)–Ru(6)	2.8950(12)	2.8922(9)
Ru(5)–Ru(6)	2.8943(11)	2.8662(9)
Ru(1)–C(1)	2.178(8)	2.161(9)
Ru(1)–C(2)	2.644(9)	2.695(7)
Ru(2)–C(2)	2.514(8)	2.488(9)
Ru(2)–C(3)	2.202(8)	2.209(9)
Ru(3)–C(1)	4.552(8)	4.628(9)
Ru(3)–C(2)	4.090(9)	4.279(8)
Ru(3)–C(3)	4.689(9)	4.817(8)
Ru(6)–C(1)	4.057(8)	3.969(9)
Ru(6)–C(2)	4.165(8)	4.132(8)
Ru(6)–C(3)	4.086(9)	4.002(8)
Ru(3)–C(34)	2.015(9)	2.087(6)
Ru(4)–C(34)	2.130(10)	2.048(6)
Ru(4)–C(63)	—	2.987(7)
Ru(4)–C(64)	2.497(10)	—
Ru(6)–C(63)	—	1.899(11)
Ru(6)–C(64)	1.924(10)	—
O(34)–C(34)	1.174(12)	1.164(7)
O(63)–C(63)	—	1.163(13)
O(64)–C(64)	1.173(12)	—
C(0)–C(1)	3.705(11)	3.700(12)
C(0)–C(2)	3.490(12)	3.574(11)
C(0)–C(3)	3.805(12)	3.834(10)
C(1)–C(2)	1.432(12)	1.438(10)
C(2)–C(3)	1.451(13)	1.385(9)
C(1)–H(11)	1.18(8)	0.98(6)
C(1)–H(12)	1.08(8)	0.93(5)
C(2)–H(2)	1.04(8)	0.94(6)
C(3)–H(31)	1.09(8)	0.94(6)
C(3)–H(32)	1.08(9)	0.83(6)
Ru(2)–Ru(1)–C(1)	82.3(2)	81.7(2)
Ru(2)–Ru(1)–C(2)	53.0(2)	52.7(2)
C(1)–Ru(1)–C(2)	32.8(3)	32.1(3)
Ru(1)–Ru(2)–C(2)	57.2(2)	59.6(2)
Ru(1)–Ru(2)–C(3)	87.3(2)	88.1(2)
C(2)–Ru(2)–C(3)	35.1(3)	33.6(2)
Ru(4)–Ru(3)–C(34)	48.4(3)	46.5(2)
Ru(3)–Ru(4)–C(34)	45.0(3)	47.6(2)
C(34)–Ru(4)–C(63)	—	168.2(3)
C(34)–Ru(4)–C(64)	176.8(3)	—
Ru(6)–Ru(4)–C(63)	—	37.7(2)
Ru(6)–Ru(4)–C(64)	41.0(2)	—
Ru(4)–Ru(6)–C(63)	—	73.9(2)
Ru(4)–Ru(6)–C(64)	58.4(3)	—
Ru(1)–C(1)–C(2)	91.8(5)	94.9(6)
Ru(1)–C(1)–H(11)	103(4)	112(3)
Ru(1)–C(1)–H(12)	114(4)	110(4)
C(2)–C(1)–H(11)	101(5)	116(3)

Table 1 (continued)

	<b>1</b>	<b>2</b>
<i>(a) Intramolecular distances and angles</i>		
C(2)–C(1)–H(12)	122(4)	124(3)
H(11)–C(1)–H(12)	121(6)	100(5)
Ru(1)–C(2)–Ru(2)	69.8(2)	67.7(2)
Ru(1)–C(2)–C(1)	55.4(4)	53.0(4)
Ru(1)–C(2)–C(3)	120.3(5)	119.2(6)
Ru(1)–C(2)–H(2)	107(4)	104(4)
Ru(2)–C(2)–C(1)	118.0(6)	114.5(6)
Ru(2)–C(2)–C(3)	60.7(4)	62.1(5)
Ru(2)–C(2)–H(2)	107(4)	104(5)
C(1)–C(2)–C(3)	125.5(8)	125.6(6)
C(1)–C(2)–H(2)	114(4)	115(4)
C(3)–C(2)–H(2)	117(4)	118(4)
Ru(2)–C(3)–C(2)	84.3(5)	84.3(5)
Ru(2)–C(3)–H(31)	101(4)	105(5)
Ru(2)–C(3)–H(32)	111(4)	117(6)
C(2)–C(3)–H(31)	103(4)	124(4)
C(2)–C(3)–H(32)	113(5)	116(5)
H(31)–C(3)–H(32)	133(6)	108(6)
Ru(3)–C(34)–Ru(4)	86.6(4)	85.9(2)
Ru(3)–C(34)–O(34)	139.5(8)	135.3(5)
Ru(4)–C(34)–O(34)	133.9(7)	138.8(5)
Ru(4)–C(63)–Ru(6)	—	68.5(2)
Ru(4)–C(64)–Ru(6)	80.7(4)	—
Ru(4)–C(63)–O(63)	—	115.7(6)
Ru(4)–C(64)–O(64)	124.4(8)	—
Ru(6)–C(63)–O(63)	—	175.8(7)
Ru(6)–C(64)–O(64)	154.9(9)	—
<i>(b) Intermolecular distances</i>		
C(1)–O(31)	3.120(11)	6.556(12)
C(1)–O(32)	6.678(11)	3.354(9)
C(2)–O(31)	3.232(10)	6.669(12)
C(2)–O(32)	6.799(10)	3.266(9)
C(3)–O(21)	7.155(11)	3.810(10)
C(3)–O(31)	3.253(10)	6.876(12)

The crystal structures of both **1** and **2** consist basically of columns of cluster anions surrounded by cation columns, as have been observed for the rhodium carbonyl cluster salts [12]. The packing is illustrated in Fig. 2 for a pair of closest neighbors. In the anion column, the distance between adjacent cluster centers C(0) is 10.46 Å in **1** and 9.63 Å in **2**. As far as the allyl moiety is concerned, it faces directly the neighboring cluster anion in **1**, but not so in **2**. The shortest intermolecular distance from the allyl group is 3.12(1) Å to oxygen (a) in **1** and 3.266(9) Å from the allyl central carbon to oxygen (b) in **2** (see Fig. 2). By inspecting the packing shown in Fig. 2, it appears reasonable that the allyl group is pushed upwards in **2** compared with that in **1**, albeit with a weak intermolecular repulsive force.

The present work demonstrates that a  $\pi$ -coordinated hydrocarbon ligand in a large cluster core can shift its position, at least to the extent of 0.1–0.2 Å or so, by small intermolecular driving forces (e.g. crystal-packing force). Care should be taken in discussing crystal structures of clusters not to relate the small difference below

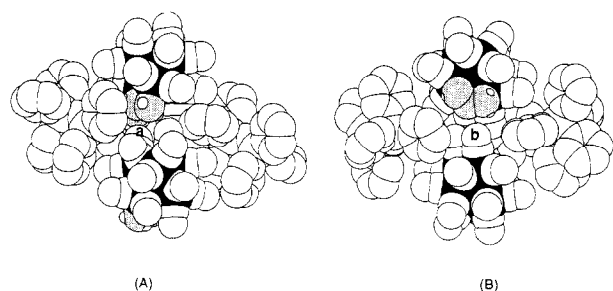


Fig. 2. Packing of two closest pairs of cation (PPN) and anion (ruthenium, black; allyl, grey) in solid-state. (A)  $[\text{Ru}_6\text{C}(\text{CO})_{13}(\mu\text{-CO})_2(\mu, \eta^3\text{-C}_3\text{H}_5)]$  (crystal 1) and (B)  $[\text{Ru}_6\text{C}(\text{CO})_{14}(\mu\text{-CO})(\mu, \eta^3\text{-C}_3\text{H}_5)]$  (crystal 2).

this range to intramolecular energy minimization. It might well be that a positional fluctuation of a hydrocarbon ligand on cluster surfaces takes place in solutions too, particularly at high temperatures.

### 3. Experimental details

#### 3.1. Preparation of $[\text{PPN}][\text{Ru}_6\text{C}(\text{CO})_{14}(\mu\text{-CO})(\text{C}_3\text{H}_5)]$ (2)

Complex  $[\text{PPN}][\text{Ru}_6\text{C}(\text{CO})_{15}(\text{C}_3\text{H}_5)]$  was prepared according to the reported method [11]. On standing the saturated, hot (ca. 50°C) methanol solution at room temperature, rhombic reddish–brown crystals of **2** were obtained. IR and NMR spectra were recorded on a Perkin-Elmer 1600 FT-IR spectrophotometer and a Jeol JNM- $\alpha$ 400 spectrometer. IR ( $\text{CH}_2\text{Cl}_2$ ) 2057w, 2014w, 2000vs, 1944w(br), 1793w(br); (KBr) 2054m, 1998s, 1991w(sh), 1975w, 1964w, 1941w, 1931w, 1922w, 1799m  $\text{cm}^{-1}$ .  $^{13}\text{C}$  NMR (acetone- $d_6$ ) 204.395 (s, CO), 133.748–126.352 (m, Ph), 49.073 (s, allyl), 18.082 (s, allyl).  $^1\text{H}$  NMR (acetone- $d_6$ ) 7.73–7.53 (30H, m, Ph), 3.91(2H, ddd,  $J = 7.02, 1.83,$  and  $1.22$  Hz, *syn*-H), 1.44 (1H, tt,  $J = 12.21$  and  $7.02$  Hz, central-H), 0.43 (2H, ddd,  $J = 12.21, 1.83,$  and  $1.22$  Hz, *anti*-H). Anal. Found: C, 40.81; H, 2.21; N, 0.91.  $\text{C}_{55}\text{H}_{35}\text{NO}_{15}\text{Ru}_6$  Calc.: C, 40.82; H, 2.18; N, 0.87%.

Rectangular reddish–brown crystals of  $[\text{PPN}][\text{Ru}_6\text{C}(\text{CO})_{13}(\mu\text{-CO})_2(\text{C}_3\text{H}_5)]$  (**1**) could best be obtained by addition of methanol to the  $\text{CH}_2\text{Cl}_2$  solution at  $-20^\circ\text{C}$ . IR of **1** (KBr) 2054m, 1997s, 1991w(sh), 1974w, 1965w, 1941w, 1932w, 1921w, 1799m  $\text{cm}^{-1}$ .

#### 3.2. Structure determination

A reddish–brown crystal sample was mounted in a glass capillary filled with argon. Intensity data were collected by use of an Enraf-Nonius CAD4 automated diffractometer with graphite monochromated Mo K $\alpha$

radiation. Data collection was performed by the  $\omega$ -scan method. Lorentz and polarization corrections were applied to the data; there was no evidence of decay during the course of the data collection. An empirical absorption correction was applied [13]. Crystal data and experimental details are given in Table 2. The cell constants allowed the space group to be either  $P1$  or  $P\bar{1}$ ; the latter was confirmed by the successful refinement. The coordinates of the Ru atoms were obtained by direct methods (MULTAN [14]) and all the remaining non-hydrogen atoms and 29 hydrogen atoms were located from subsequent Fourier difference syntheses. Refinement was by block-diagonal least-squares methods with anisotropic thermal parameters for all non-hydrogen atoms and isotropic thermal parameters for hydrogen atoms using the program UNICS-III [15]. The final model converged with  $R = 0.040$ . The final Fourier difference synthesis showed no chemically significant features, with the highest peak being within the covalent radius of the ruthenium atom ( $0.98 \text{ \AA}$  from Ru(3)). The analytical form of the scattering factor [16] for the appropriate neutral atom was corrected for both real ( $\Delta f'$ ) and imaginary ( $\Delta f''$ ) components of anomalous dispersions [17]. Final positional and thermal parameters are listed in Table 3.

Table 2  
Crystallographic data for  $[\text{PPN}][\text{Ru}_6\text{C}(\text{CO})_{14}(\mu\text{-CO})(\mu, \eta^3\text{-C}_3\text{H}_5)]$  (2)

Formula	$\text{C}_{55}\text{H}_{35}\text{NO}_{15}\text{P}_2\text{Ru}_6$
$M$	1618.2
Crystal system	triclinic
Space group (No.)	$P\bar{1}$ (2)
$a$ (Å)	14.239(1)
$b$ (Å)	17.319(3)
$c$ (Å)	12.871(2)
$\alpha$ (°)	109.664(14)
$\beta$ (°)	96.757(10)
$\gamma$ (°)	72.511(13)
$V$ (Å <sup>3</sup> )	2848.4
$Z$	2
$D_c$ (g $\text{cm}^{-3}$ )	1.887
$T$ (°C)	21
$\mu$ (cm <sup>-1</sup> )	16.42
$F(000)$	1572
Crystal size (mm <sup>3</sup> )	$0.35 \times 0.32 \times 0.32$
$\lambda$ (Mo K $\alpha$ ) (Å)	0.71073
$2\theta_{\text{max}}$ (°)	55
No. of data measured	13992
No. of unique data used	9716 [ $F \geq 5\sigma$ ]
No. of parameters	829
Correction made	$\psi$ -scan
Transmission coefficients	0.923–0.998
Final $R$	0.042
Final $R'$	0.040 [ $w = 1$ ]
Goodness of fit	2.18
$\Delta\rho$ (e Å <sup>-3</sup> )	1.50

Table 3

Atomic coordinates<sup>a</sup> and equivalent temperature factors ( $\text{\AA}^2$ ) for  $\text{Ru}_6\text{C}(\text{CO})_{14}(\mu\text{-CO})\chi(\mu,\eta^3\text{-C}_3\text{H}_5)_2$  (2)

Ru(1)	7806.4(3)	6657.9(3)	4332.1(4)	3.7
Ru(2)	8645.6(4)	7958.7(3)	5833.8(4)	3.9
Ru(3)	9682.3(3)	6783.2(3)	3762.8(4)	3.8
Ru(4)	7971.8(3)	7268.2(3)	2524.3(4)	3.7
Ru(5)	8819.3(4)	8568.3(3)	4094.6(4)	3.9
Ru(6)	6936.5(4)	8452.6(3)	4496.7(5)	4.3
P(1)	14550.0(11)	6723.4(9)	8090.8(12)	3.4
P(2)	12856.5(11)	7461.2(9)	9585.6(12)	3.3
O(11)	6239(4)	5833(4)	3107(5)	7.2
O(12)	9036(4)	4844(3)	3983(5)	6.8
O(21)	10709(4)	7182(4)	6540(5)	7.3
O(22)	8689(6)	9657(4)	7486(6)	10.6
O(31)	11558(4)	7169(4)	3595(7)	9.4
O(32)	10968(4)	5016(3)	3576(5)	6.9
O(34)	9743(4)	5955(4)	1281(4)	6.5
O(41)	6706(5)	6189(4)	1104(5)	8.4
O(42)	7779(5)	8009(4)	681(5)	8.5
O(51)	10420(5)	9225(5)	5561(6)	9.9
O(52)	9799(4)	8506(4)	2102(5)	7.6
O(53)	7450(4)	10329(3)	4276(5)	7.7
O(61)	5311(3)	7888(4)	5075(4)	6.0
O(62)	6374(6)	10017(5)	6553(7)	12.0
O(63)	5736(4)	9004(5)	2606(6)	9.1
N	13764(4)	6787(3)	8910(4)	5.7
C(0)	8304(4)	7598(3)	4193(5)	3.2
C(1)	7360(5)	6634(5)	5866(6)	5.1
C(2)	8119(5)	6977(4)	6525(6)	4.8
C(3)	7990(6)	7813(5)	7206(6)	5.7
C(11)	6820(5)	6161(4)	3569(6)	4.7
C(12)	8614(5)	5548(4)	4120(6)	5.0
C(21)	9938(5)	7448(5)	6196(6)	5.3
C(22)	8661(8)	9005(5)	6844(7)	7.7
C(31)	10835(5)	7035(5)	3663(8)	6.5
C(32)	10462(5)	5685(4)	3673(6)	4.6
C(34)	9329(5)	6425(4)	2079(5)	4.5
C(41)	7198(5)	6579(5)	1642(6)	5.4
C(42)	7854(5)	7732(5)	1383(7)	5.7
C(51)	9790(7)	8967(6)	5023(8)	7.4
C(52)	9411(5)	8523(4)	2836(6)	5.2
C(53)	7898(6)	9657(5)	4240(6)	5.6
C(61)	5975(5)	8030(4)	4830(5)	4.8
C(62)	6574(7)	9425(6)	5688(9)	8.7
C(63)	6218(5)	8771(6)	3295(8)	7.5
C(A1)	14840(4)	7705(4)	8285(5)	3.8
C(A2)	15381(5)	8024(4)	9215(6)	4.9
C(A3)	15628(6)	8780(5)	9412(7)	6.2
C(A4)	15315(7)	9204(5)	8655(7)	6.7
C(A5)	14750(6)	8916(4)	7743(6)	5.9
C(A6)	14522(5)	8161(4)	7555(5)	4.8
C(B1)	15669(4)	5966(4)	8293(4)	3.8
C(B2)	15626(5)	5258(5)	8532(6)	5.5
C(B3)	16479(7)	4641(5)	8695(7)	6.9
C(B4)	17360(6)	4771(6)	8604(7)	7.6
C(B5)	17455(6)	5441(6)	8361(6)	6.5
C(B6)	16597(5)	6024(5)	8190(6)	5.4
C(C1)	14139(4)	6348(3)	6678(4)	3.3
C(C2)	13241(5)	6187(4)	6442(5)	4.3
C(C3)	12901(5)	5929(5)	5375(6)	5.5
C(C4)	13474(6)	5828(5)	4530(6)	5.6
C(C5)	14385(5)	5976(5)	4726(5)	5.2
C(C6)	14709(5)	6228(4)	5802(5)	4.6
C(D1)	12479(4)	8469(4)	9327(5)	4.0
C(D2)	12677(5)	9191(4)	10074(6)	5.2

Table 3 (continued)

C(D3)	12419(7)	9935(5)	9814(9)	8.1
C(D4)	11972(8)	9988(6)	8848(9)	8.9
C(D5)	11774(8)	9277(7)	8104(9)	9.3
C(D6)	12017(6)	8507(5)	8329(6)	5.9
C(E1)	13118(4)	7671(4)	11037(4)	3.5
C(E2)	14058(5)	7378(5)	11405(5)	5.0
C(E3)	14242(6)	7531(5)	12529(6)	6.4
C(E4)	13505(6)	7975(5)	13257(6)	6.2
C(E5)	12584(5)	8273(5)	12900(6)	5.5
C(E6)	12382(5)	8125(5)	11796(5)	4.8
C(F1)	11820(4)	7020(4)	9292(5)	4.0
C(F2)	11994(5)	6149(5)	8989(6)	5.4
C(F3)	11213(7)	5807(6)	8866(7)	7.4
C(F4)	10294(7)	6313(7)	9039(7)	8.1
C(F5)	10071(6)	7193(6)	9332(6)	6.9
C(F6)	10875(5)	7528(5)	9462(6)	5.6
H(11)	738(4)	606(3)	584(4)	3.3
H(12)	669(4)	690(4)	597(5)	4.4
H(2)	871(4)	657(4)	659(5)	5.3
H(31)	839(5)	799(4)	783(5)	6.3
H(32)	741(5)	810(4)	736(5)	6.4
H(CA2)	1564(4)	773(3)	971(4)	3.8
H(CA4)	1541(4)	965(4)	872(5)	5.0
H(CA5)	1451(5)	917(4)	729(5)	5.8
H(CA6)	1417(4)	797(4)	698(5)	4.5
H(CB2)	1510(3)	523(3)	870(4)	2.3
H(CB4)	1770(6)	447(5)	866(7)	9.2
H(CB6)	1659(3)	643(3)	807(4)	2.2
H(CC2)	1293(5)	629(4)	696(5)	5.9
H(CC3)	1230(5)	585(4)	528(5)	5.5
H(CC4)	1330(5)	572(5)	396(6)	7.3
H(CC6)	1525(4)	629(3)	597(4)	3.8
H(CD2)	1299(4)	914(3)	1069(4)	4.1
H(CD3)	1246(5)	1036(4)	1026(6)	7.1
H(CD5)	1150(6)	927(5)	760(6)	8.1
H(CD6)	1198(4)	805(4)	785(5)	4.3
H(CE2)	1452(4)	713(4)	1096(5)	4.5
H(CE3)	1485(5)	732(4)	1270(5)	5.5
H(CE4)	1357(5)	806(4)	1390(5)	6.1
H(CE5)	1210(5)	855(4)	1332(5)	5.9
H(CE6)	1178(4)	824(4)	1152(5)	4.9
H(CF2)	1257(4)	585(3)	892(4)	4.0
H(CF3)	1139(5)	529(4)	861(5)	6.4
H(CF4)	985(6)	618(6)	896(7)	10.3
H(CF6)	1073(3)	801(3)	956(4)	2.4

<sup>a</sup> Fractional  $\times 10^4$  for non-hydrogen atoms and  $\times 10^3$  for hydrogen atoms.<sup>b</sup>  $B_{\text{eq}} = 4/3(\sum_i \sum_j B_{ij} a_i b_j)$  for non-hydrogen atoms. Hydrogen atoms were refined isotropically.

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