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Synthesis and Crystal Structure of Undecacarbonyl(triphenylphosphine)-triangulo-triruthenium, $Ru_3(CO)_{11}Ph_3P$

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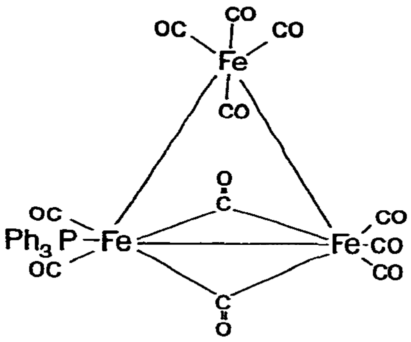
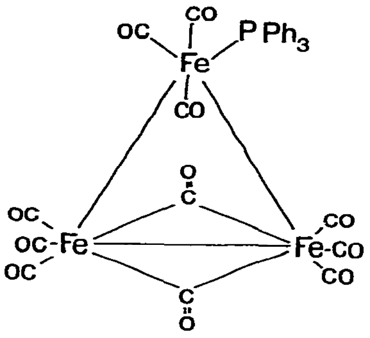
Summary

Irradiation of $Ru_3(CO)_{12}$ in hexane in the presence of triphenylphosphine gives undecacarbonyl(triphenylphosphine)-*triangulo*-triruthenium in addition to the previously obtained $Ru(CO)_4PPh_3$ and $Ru(CO)_3(PPh_3)_2$. X-Ray structure determination of the cluster compound reveals a single isomer in which there is a triangle of ruthenium atoms with the triphenylphosphine equatorially substituted and all the carbonyl groups terminally sited. The Ru-Ru distances are 2.907(3), 2.876(3) and 2.875(3)Å, with the longest distance *cis* to the triphenylphosphine ligand. The crystals are monoclinic space group C2/c with *a* 22.30(2)Å, *b* 16.34(1)Å, *c* 17.42(1)Å, β = 103.84(4)° and *Z* = 8. The structure was refined to R 7.2% for 1773 observed counter amplitudes.

There is a dearth of structural information concerning triangulo trinuclear ruthenium complexes which are mono-substituted with a group V ligand. A number of such iron and osmium complexes have, however, been studied by X-ray crystallography, eg. $Fe_3(CO)_{11}PPh_3$ [1] and $Os_3(CO)_{11}P(OCH_3)_3$ [2]. The study of the osmium complex revealed only one isomer in which the carbonyl groups were all terminal. More interestingly, the study of the iron complex revealed the existence of two isomers (I) and (II), in both of which the triphenylphosphine is equatorially substituted. The existence of two isomers in the solid phase was thought to reflect the dynamic nature of the molecule in solution with the carbonyl bridges opening and reforming between any two iron atoms in the molecule [3]. This exchange of carbonyl groups (similar to that in $Fe_3(CO)_{12}$ itself) was confirmed by ^{13}C n.m.r. spectroscopy [4-6]. Exchange of carbonyl groups has also been shown to occur in $Ru_3(CO)_{12}$, but since carbonyl bridges have not been found either in the solid phase or in solution it is considered that this exchange results from the interconversion of axial and equatorial groups on the same metal atom [5,6]. This type of exchange has been shown by ^{13}C n.m.r. spectroscopy to occur in the triangulo nitrosyl complex $Ru_3(CO)_{10}(NO)_2$ (III) [5] and in the acetylenic complexes $HM_3(CO)_9[C_2C(CH_3)_3]$(M = Ru and Os) (IV) [6]. The possibility that a



(I)



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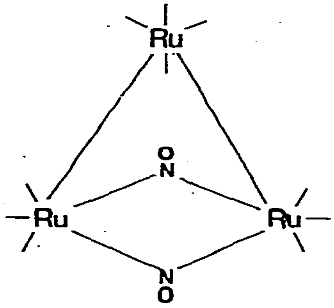

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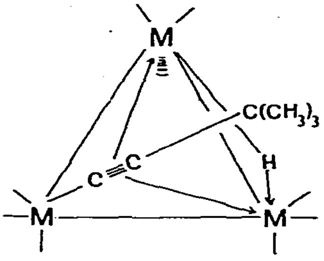

(IV)

mono-substituted triangulo-ruthenium complex could exist in two isomeric forms prompted the current X-ray structure determination of the title compound.

Experimental

Preparation of Undecacarbonyl(triphenylphosphine)-triangulo-triruthenium.* A solution of $Ru_3(CO)_{12}$ (0.128 g, 0.2 mmoles) and triphenylphosphine (0.157 g, 0.6 mmoles) in n-hexane (150 cm^3) under oxygen-free nitrogen was irradiated with a tungsten-halogen lamp at $\lambda > 390$ nm (2M $NaNO_2$ as filter) at 25°. When the reaction was complete (3 hr.) the solution was filtered to give $Ru(CO)_3(PPh_3)_2$ as a pale-yellow solid (0.085 g), m.p. 209-210°. The filtrate was evaporated to dryness and the residue chromatographed on silica. Elution with n-hexane-ether (4:1) yielded, after evaporation of the solvents, two compounds (a) $Ru(CO)_4PPh_3$ (0.19 g), m.p. 118-122° and (b) undecacarbonyl(triphenylphosphine)-triangulo-triruthenium (0.016 g) as dark red needles, m.p. 130-132° (lit. [7] cites 131-133°). ν_{max}(n-hexane) 2097 m, 2046 s, 2030 sh, 2023 sh, 2014 s, 1996 sh, 1986 m, 1972 sh, and 1960 sh, identical with lit. values [7]. The compound is soluble in hydrocarbon solvents, but its solutions are light and air sensitive.

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*The title compound has been previously prepared by the thermal reaction of Ph_3P and $Ru_3(CO)_{12}$ [7]. From the photochemical reaction between these latter two compounds only $Ru(CO)_4PPh_3$ and $Ru(CO)_3(PPh_3)_2$ were obtained [8].

Crystallographic measurements. After initial examination by oscillation and Weissenberg photographs, final cell dimensions and intensity data were measured with a Stoe two-circle computer-controlled diffractometer using graphite-monochromated Mo $K\alpha$ radiation. The crystal used for all X-ray measurements had dimensions 1.0 x 0.2 x 0.01 mm and was mounted about the direction of elongation (z) inside a Pantak capillary tube. The ω-scan technique was employed with a scan speed of 0.6° min^{-1} and 30 s stationary background measurements at each end of the scan. For layers $hk0$ – $hk5$ the scan-width was 1.4° and for $hk6$ – $hk18$ it was calculated from $(1.0 + 0.5 \sin\mu/\tan\theta')°$, where μ is the equi-inclination angle and $2\theta'$ is the azimuth angle. Reflections were scanned within the range $0.1 < \sin\theta/\lambda < 0.65\ \text{Å}^{-1}$, and of these 1773 $[I > 2.5\sigma(I)]$ were considered to be observed.

In the conversion of intensities to structure amplitudes absorption corrections were applied.

Crystal Data – $C_{29}H_{15}O_{11}PRu_3$, M = 873.6, monoclinic, a = 22.30(2), b = 16.34(1), c = 17.42(1) Å, β = 103.84(4)°, U = 6163 Å^3, Z = 8, D_c = 1.883 g cm^{-3}. Absorption coefficient (Mo $K\alpha$ radiation, λ = 0.71069 Å) = 14.1 cm^{-1}. Space group Cc or $C2/c$ from systematic absences, $hk\ell$ when $h + k$ is odd, $h0\ell$ when ℓ is odd. $C2/c$ established as a result of the analysis.

Structure Determination – Initially, the lower space group Cc was assumed, and the structure was solved by Patterson and Fourier methods. It soon became obvious that the two molecules in the asymmetric unit were not independent, and were related by a centre of symmetry in the higher space group $C2/c$. Atomic coordinates were averaged accordingly and the structure refined in $C2/c$ by least-squares calculations. In the final cycles of refinement, coordinates and anisotropic thermal parameters were adjusted for the phosphorus and ruthenium atoms and coordinates and isotropic temperature factors for carbon and oxygen atoms. Hydrogen atoms were included in calculated positions, but their parameters were not refined. The calculations were terminated when all calculated shifts were $< 0.1\sigma$ and R was 7.17% for the 1773 observed amplitudes.* The weighting scheme was $\underline{w} = 1/\sigma^2(F)$ where $\sigma(F)$ is the standard deviation in the observed amplitudes based on counting statistics. Final atomic coordinates and temperature factors are in Table 1. Computations were carried out on the Birmingham University 1906 A computer and the CDC 7600 at the University of Manchester Regional Computer Centre with SHELX [9].

Results and Discussion

The overall geometry of the molecule is illustrated in Fig.1, which also shows the atomic numbering. Bond lengths and angles are listed in Table 2.

The crystallographic analysis reveals that there is only one isomer of the complex in which the ruthenium atoms are arranged in a nearly equilateral triangle with the triphenylphosphine group equatorially substituted. There are no carbonyl bridges and each of the ruthenium atoms has a distorted octahedral coordination geometry. Equatorial substituents deviate by up to ± 0.2 Å from the plane of the ruthenium triangle.

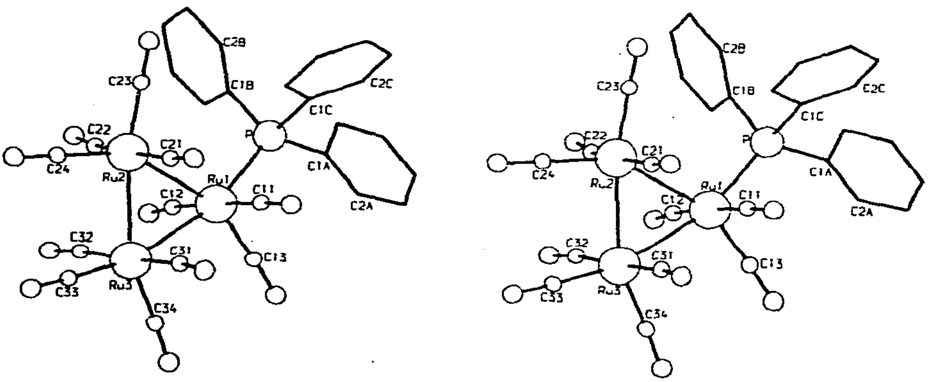

Fig. 1. Stereoscopic drawing of the $Ru_3(CO)_{11}PPh_3$ molecule showing the atomic numbering.

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*A list of observed and calculated structure factors can be obtained from the authors.

TABLE 1.

FRACTIONAL ATOMIC COORDINATES (x 10^4) AND THERMAL PARAMETERS ($Å^2$ x 10^3) WITH ESD'S IN PARENTHESES

	x	y	z	U
Ru(1)	2334(1)	-515(1)	2380(1)	†
Ru(2)	1876(1)	518(2)	1006(1)	†
Ru(3)	1037(1)	-160(1)	1858(1)	†
P	3412(3)	-592(4)	2457(3)	†
C(11)	2465(10)	451(16)	3055(12)	34(6)
C(12)	2184(11)	-1457(17)	1738(14)	41(7)
C(13)	2248(11)	-1170(16)	3222(14)	42(7)
C(21)	1936(11)	1434(16)	1707(13)	34(7)
C(22)	1918(13)	-484(21)	412(17)	69(9)
C(23)	2547(14)	969(19)	636(16)	63(9)
C(24)	1193(13)	940(17)	227(16)	55(8)
C(31)	1171(11)	718(17)	2618(13)	44(7)
C(32)	935(12)	-1019(17)	1042(15)	45(7)
C(33)	269(14)	286(19)	1316(17)	69(9)
C(34)	805(11)	-858(16)	2589(14)	42(7)
C(1A)	3855(10)	-1321(15)	3199(13)	33(6)
C(2A)	3742(11)	-1333(15)	3936(12)	33(6)
C(3A)	4120(11)	-1812(15)	4521(13)	39(7)
C(4A)	4588(13)	-2276(18)	4331(16)	61(8)
C(5A)	4693(12)	-2246(16)	3617(14)	47(7)
C(6A)	4346(10)	-1755(14)	3024(12)	30(6)
C(1B)	3582(10)	-882(14)	1529(11)	24(5)
C(2B)	3861(11)	-352(17)	1099(13)	46(7)
C(3B)	3972(11)	-564(18)	360(13)	50(7)
C(4B)	3816(10)	-1340(16)	95(13)	39(7)
C(5B)	3574(11)	-1905(17)	527(14)	48(7)
C(6B)	3457(11)	-1672(17)	1238(14)	47(7)
C(1C)	3847(9)	369(14)	2766(11)	25(5)
C(2C)	4445(12)	361(19)	3253(14)	56(8)
C(3C)	4755(12)	1068(18)	3463(14)	47(7)
C(4C)	4517(12)	1820(18)	3173(14)	46(7)
C(5C)	3956(12)	1843(18)	2695(14)	51(8)
C(6C)	3615(12)	1118(17)	2465(14)	45(7)

(continued)

TABLE 1 (continued)

	x	y	z	U
O(11)	2565(8)	938(11)	3511(10)	53(5)
O(12)	2089(8)	−2089(12)	1410(10)	55(5)
O(13)	2186(8)	−1585(12)	3741(11)	68(6)
O(21)	1978(9)	2023(13)	2073(11)	62(6)
O(22)	1975(9)	−998(13)	−3(12)	75(6)
O(23)	2942(9)	1247(13)	426(11)	72(6)
O(24)	787(10)	1144(14)	−262(12)	79(6)
O(31)	1173(9)	1262(13)	3052(11)	74(6)
O(32)	818(8)	−1511(12)	590(10)	58(5)
O(33)	−187(10)	582(14)	982(11)	81(6)
O(34)	658(8)	−1301(12)	3025(10)	58(5).

† Anisotropic temperature factors in the form

$$T = \exp[-2\pi^2(U_{11}h^2a^{*2} + \ldots. + 2U_{12}hka^*b^*)]$$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ru(1)	26(1)	39(1)	38(1)	1(1)	3(1)	−1(1)
Ru(2)	44(1)	49(2)	42(1)	4(1)	10(1)	2(1)
Ru(3)	27(1)	48(2)	50(1)	−3(1)	6(1)	0(1)
P	19(3)	21(4)	35(3)	−5(3)	6(3)	2(4)

The Ru–Ru distances average 2.886 Å which is some 0.04 Å greater than the average in $Ru_3(CO)_{12}$ [10]. A somewhat smaller (0.02 Å) increase in Os–Os distances had been noted [2] for the comparable undecacarbonyl (trimethylphosphite)triosmium in comparison with the metal–metal distances in $Os_3(CO)_{12}$ [11]. A similar elongation (0.02 Å) of the metal–metal bonds is also seen in the triangulo-iron complex (II) [1] when compared with the average metal–metal distance in $Fe_3(CO)_{12}$ [12]. This general increase in metal–metal distance can in part be ascribed to the effect of substituting a good π-acceptor ligand with a lesser one (Ph_3P or $P(OCH_3)_3$), thus promoting a higher electron density in the metal framework of the molecule which is relieved by expansion.

TABLE 2.

INTERATOMIC DISTANCES (Å) AND BOND ANGLES (°) WITH ESD'S IN PARENTHESES

(a) Bonded distances

Ru(1) - Ru(2)	2.907(3)
Ru(1) - Ru(3)	2.876(3)
Ru(2) - Ru(3)	2.875(3)
P - Ru(1)	2.380(6)
C(11) - Ru(1)	1.95(3)
C(12) - Ru(1)	1.89(3)
C(13) - Ru(1)	1.86(3)
C(21) - Ru(2)	1.92(3)
C(22) - Ru(2)	1.95(4)
C(23) - Ru(2)	1.91(3)
C(24) - Ru(2)	1.91(3)
C(31) - Ru(3)	1.93(3)
C(32) - Ru(3)	1.97(3)
C(33) - Ru(3)	1.89(3)
C(34) - Ru(3)	1.87(3)
C(1A) - P	1.86(2)
C(1B) - P	1.81(2)
C(1C) - P	1.86(2)
C(1A) - C(2A)	1.37(3)
C(2A) - C(3A)	1.40(3)
C(3A) - C(4A)	1.39(3)
C(4A) - C(5A)	1.32(3)

C(5A) - C(6A)	1.39(3)
C(6A) - C(1A)	1.40(3)
C(1B) - C(2B)	1.39(3)
C(2B) - C(3B)	1.41(3)
C(3B) - C(4B)	1.37(3)
C(4B) - C(5B)	1.38(3)
C(5B) - C(6B)	1.38(3)
C(6B) - C(1B)	1.39(3)
C(1C) - C(2C)	1.40(3)
C(2C) - C(3C)	1.35(3)
C(3C) - C(4C)	1.39(3)
C(4C) - C(5C)	1.33(3)
C(5C) - C(6C)	1.41(3)
C(6C) - C(1C)	1.38(3)
O(11) - C(11)	1.11(2)
O(12) - C(12)	1.17(3)
O(13) - C(13)	1.17(3)
O(21) - C(21)	1.15(3)
O(22) - C(22)	1.14(3)
O(23) - C(23)	1.13(3)
O(24) - C(24)	1.13(3)
O(31) - C(31)	1.17(3)
O(32) - C(32)	1.11(3)
O(33) - C(33)	1.15(3)
O(34) - C(34)	1.15(3)

(b) Selected non-bonded distances

O(11) ... Ru(1)	3.05
O(12) ... Ru(1)	3.05
O(13) ... Ru(1)	3.03
O(21) ... Ru(2)	3.06
O(22) ... Ru(2)	3.08

C(33) ... C(34)	2.93
C(34) ... C(13)	3.18
C(11) ... C(21)	2.86
C(12) ... C(22)	2.75
C(11) ... C(31)	2.84

(continued)

TABLE 2 (continued)

O(23) ... Ru(2)	3.04
O(24) ... Ru(2)	3.04
O(31) ... Ru(3)	3.09
O(32) ... Ru(3)	3.08
O(33) ... Ru(3)	3.04
O(34) ... Ru(3)	3.02
C(13) ... P	3.33
C(23) ... P	4.17
C(23) ... C(24)	2.93
C(24) ... C(33)	3.29

C(12) ... C(32)	2.85
C(21) ... C(31)	2.85
C(22) ... C(32)	2.82
O(11) ... O(21)	3.09
O(12) ... O(22)	3.00
O(11) ... O(31)	3.06
O(12) ... O(32)	3.01
O(21) ... O(31)	3.02
O(22) ... O(32)	3.11

(c) Bond angles

Ru(2) - Ru(1) - Ru(3)	59.6(1)
C(11) - Ru(1) - Ru(2)	89.9(7)
- Ru(3)	91.2(6)
- C(12)	178.1(10)
- C(13)	91.1(10)
- P	90.3(7)
C(12) - Ru(1) - Ru(2)	90.5(7)
- Ru(3)	87.4(8)
- C(13)	87.8(10)
- P	91.4(8)
C(13) - Ru(1) - Ru(2)	154.2(7)
- Ru(3)	94.6(7)
- P	102.6(8)
P - Ru(1) - Ru(2)	103.2(2)
- Ru(3)	162.7(2)
Ru(1) - Ru(2) - Ru(3)	59.7(1)
C(21) - Ru(2) - Ru(1)	88.4(7)
- Ru(3)	85.8(7)
- C(22)	171.3(11)
- C(23)	88.4(11)
- C(24)	95.3(11)
C(22) - Ru(2) - Ru(1)	84.1(9)
- Ru(3)	94.0(8)
- C(23)	90.2(12)
- C(24)	93.4(12)
C(23) - Ru(2) - Ru(1)	110.6(8)
- Ru(3)	168.8(8)
- C(24)	100.2(12)
C(24) - Ru(2) - Ru(1)	149.1(8)
- Ru(3)	89.9(8)
Ru(1) - Ru(3) - Ru(2)	60.7(1)

C(34) - Ru(3) - Ru(1)	94.7(7)
- Ru(2)	154.9(7)
C(1A) - P - Ru(1)	115.8(7)
- C(1B)	105.2(10)
- C(1C)	100.8(10)
C(1B) - P - Ru(1)	112.9(7)
- C(1C)	105.9(9)
C(1C) - P - Ru(1)	115.0(7)
O(11) - C(11) - Ru(1)	172(2)
O(12) - C(12) - Ru(1)	173(2)
O(13) - C(13) - Ru(1)	179(2)
O(21) - C(21) - Ru(2)	174(2)
O(22) - C(22) - Ru(2)	170(3)
O(23) - C(23) - Ru(2)	179(3)
O(24) - C(24) - Ru(2)	176(3)
O(31) - C(31) - Ru(3)	172(2)
O(32) - C(32) - Ru(3)	173(2)
O(33) - C(33) - Ru(3)	178(3)
O(34) - C(34) - Ru(3)	178(2)
P - C(1A) - C(2A)	118(2)
- C(6A)	119(2)
C(6A) - C(1A) - C(2A)	122(2)
C(1A) - C(2A) - C(3A)	119(2)
C(2A) - C(3A) - C(4A)	119(2)
C(3A) - C(4A) - C(5A)	121(3)
C(4A) - C(5A) - C(6A)	122(3)
C(5A) - C(6A) - C(1A)	117(2)
P - C(1B) - C(2B)	122(2)
- C(6B)	120(2)
C(6B) - C(1B) - C(2B)	117(2)
C(1B) - C(2B) - C(3B)	123(3)

(continued)

TABLE 2 (continued)

C(31) - Ru(3) - Ru(1)	87.4(7)	C(2B) - C(3B) - C(4B)	117(3)
- Ru(2)	93.1(7)	C(3B) - C(4B) - C(5B)	123(2)
- C(32)	176.2(10)	C(4B) - C(5B) - C(6B)	119(3)
- C(33)	91.7(11)	C(5B) - C(6B) - C(1B)	121(2)
- C(34)	90.5(10)	P - C(1C) - C(2C)	122(2)
C(32) - Ru(3) - Ru(1)	91.0(7)	- C(6C)	121(2)
- Ru(2)	83.1(7)	C(6C) - C(1C) - C(2C)	117(2)
- C(33)	88.8(11)	C(1C) - C(2C) - C(3C)	121(3)
- C(34)	93.1(10)	C(2C) - C(3C) - C(4C)	122(3)
C(33) - Ru(3) - Ru(1)	163.1(9)	C(3C) - C(4C) - C(5C)	118(3)
- Ru(2)	102.5(8)	C(4C) - C(5C) - C(6C)	121(3)
- C(34)	102.1(11)	C(5C) - C(6C) - C(1C)	121(2)

The Ru-Ru bond lengths in our structure fall into two categories; a long bond, Ru(1)-Ru(2) of 2.907 Å, cis to the triphenylphosphine ligand and two shorter bonds, Ru(1)-Ru(3) of 2.876 Å and Ru(2)-Ru(3) of 2.875 Å. Estimated standard deviations are 0.003 Å for Ru-Ru distances, so that this difference of 0.03 Å is significant. A similar, but somewhat smaller distortion of the metal triangle occurs in $Os_3(CO)_{11}P(OCH_3)_3$. Here the Os-Os distance cis to the trimethylphosphite group is 2.908(4) Å, while the two other Os-Os bond lengths are 2.892(4) and 2.890(4) Å [2]. These uneven increases in the lengths of the metal-metal bonds in comparison with those in the parent carbonyls may be due not only to an electronic effect, as mentioned earlier, but also to a steric effect induced by the bulky substituent, since the largest increase occurs in the metal-metal bond cis to the substituent.

The Ru-C bond lengths are 1.86 - 1.97, mean 1.91 Å, in good agreement with expected values [10,13,14]. The axial bonds, mean length 1.935(11) Å are slightly longer than equatorial ones, mean length 1.888(10) Å. Similar differences between axial and equatorial Metal-CO bonds occur in $Os_3(CO)_{11}P(OCH_3)_3$ [2] and $Os_3(CO)_{12}$ [11] and have been rationalised on theoretical grounds [11]. In $Ru_3(CO)_{12}$ [10], however, the trend is reversed though the accuracy of this determination has been questioned [11].

The non-bonded C···C and O···O contacts for the axial carbonyl groups are 2.75 - 2.86, mean 2.83 Å, and 3.00 - 3.11, mean 3.05 Å. The

C···C distances are thus all smaller than the Ru–Ru distances while the O···O distances are greater. This increase in the O···O distances relative to the C···C distances is brought about by a bending of the carbonyl groups. Angles Ru – C ≡ O are 170 – 174, mean 172° for axial carbonyls. Equatorially substituted carbonyls are more nearly linear with Ru – C ≡ O angles of 176 – 179, mean 178°. The same dichotomy between non-linear axial CO groups and almost linear equatorial groups is to be seen also in $Os_3(CO)_{11}P(OCH_3)_3$ [2] and in $Os_3(CO)_{12}$ [11]. Deviations from linearity of M – C ≡ O fragments now seem a well-established feature; the smaller deviations are expected on electronic grounds [15], the larger ones in axial groups may well have a steric component.

The geometry around the phosphorus atom is essentially tetrahedral, but distorted with the phenyl groups bent away from the ruthenium atom to which it is bonded (angles C – P – C are in the range 100.8 – 105.9, mean 104.0°, whereas those of type C – P – Ru are 112.9 – 115.8, mean 114.6°). A similar effect occurs in $Os_3(CO)_{11}P(OCH_3)_3$ [2] and $\mu - H_4(CO)_{10}(PPh_3)_2Ru_4$ [14], and was also noted for the antimony atom in $Ru(CO)_4SbPh_3$ [13]. Our Ru–P bond length at 2.380 Å compares well with previously determined values [14].

The ruthenium complex, unlike the corresponding iron complex exists in only one isomeric form. The existence of a second isomer would require it to contain either bridging carbonyl groups (a less favourable situation than with iron) or an axially sited ligand (a thermodynamically less favourable situation). It is tempting to think that our compound is isomerically different from that prepared previously [7] by the thermal reaction between triphenylphosphine and $Ru_3(CO)_{12}$ which gave a yellow compound. Our photochemical preparation gave a deep-red compound. However, the two specimens have the same melting point and infra-red spectrum so that they must be regarded as dimorphic, or as merely differing in particle size.

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