C14—O15—C16	117.1 (4)	O36—K1—O37*A*	122.9 (5)
O15—C16—C17	125.3 (6)	O36—K1—O37A^{i}	102.3 (4)
C21—O22—C23	113.3 (4)	K1—O37*A*—K1^{i}	95.1 (7)
O22—C23—C24	112.6 (5)	K1—O37*B*—K1^{i}	96.9 (4)

Symmetry codes: (i) $-x, y, \frac{3}{2}-z$; (ii) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (iii) $\frac{1}{2}+x, y-\frac{1}{2}, z$; (iv) $1-x, y, \frac{3}{2}-z$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1158). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Alkyne Coupling Induced by a Trinuclear Ruthenium Cluster: Synthesis and Structure of Di-μ-carbonyl-hexacarbonyl-μ_3-(1,2,3,4-tetraphenyl-1,3-butadiene-1,4-diyl)-triruthenium(2 *Ru*—*Ru*), [$Ru_3(CO)_8(C_4Ph_4)$]

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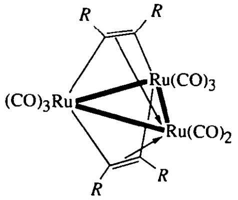
Abstract

The reaction of PhC≡CPh with [$Ru_3(CO)_{10}(MeCN)_2$] affords two [$Ru_3(CO)_8(C_2Ph_2)_2$] complexes, one red–violet containing only terminal carbonyl groups and the other the orange–yellow title compound, di-μ-carbonyl-1:2κ^2*C*;1:3κ^2*C*-hexacarbonyl-1κ^2*C*,2κ^2*C*,3κ^2*C*-μ_3-[1,2,3,4-tetraphenyl-2(η^4):3(η^4)-1,3-butadiene-1,4-diyl-1κ^2*C*]-triruthenium(2 *Ru*—*Ru*). The structure of the latter isomer consists of an open Ru_3 cluster [Ru—Ru 2.6696 (8) and 2.6717 (6), Ru···Ru 3.7461 (7) Å] coordinated to the C_4Ph_4 ligand, formed by the dimerization of PhC≡CPh, and to eight CO groups, six terminal and two bridging. The central Ru atom is σ-bonded to the butadiene moiety to form an RuC_4 metallacyclopentadiene ring [Ru—C 2.219 (4) and 2.216 (4); mean C—C 1.461 Å]. Each of the other two metal atoms is η^4-bonded to the C atoms of the RuC_4 ring [mean Ru—C 2.322 Å] to form a pentagonal bipyramidal Ru_3C_4 core.

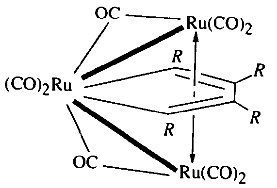
Comment

Dodecacarbonyltriruthenium has been shown to react with alkynes to give good yields of complexes containing a metallacyclopentadiene ring, RuC_4, with σ metal–carbon bonds (Sears & Stone, 1968; Cetini, Gambino, Sappa & Valle, 1969; Sappa, Manotti Lanfredi & Tiripicchio, 1980; Rosenberg *et al.*, 1981), where two or three alkynes join to form the heterocyclic five-membered ring.

Dodge & Schomaker (1965) reported the X-ray crystal structures of two isomers of the general formula [$Fe_3(CO)_8(C_2Ph_2)_2$] obtained by the reaction of [$Fe_3(CO)_{12}$] with diphenylacetylene, PhC≡CPh. The direct reaction produced a rather unstable violet complex, which readily underwent rearrangement to a more stable black isomer. Cetini *et al.* (1969) found that the direct reaction of [$Ru_3(CO)_{12}$] with PhC≡CPh affords several products, among them two isomers analogous to the iron complexes studied by Dodge & Schomaker (1965): a red–violet compound containing only terminal carbonyl groups and an orange–yellow compound also containing bridging carbonyl groups. From IR and X-ray powder data, they concluded that the structures of the violet Fe isomer and the red–violet Ru compound (I) were similar, as were the structures of the black Fe complex and the orange–yellow Ru product (II). Foulds, Johnson & Lewis (1985) reported a synthesis of the red–violet isomer (I) with 90% yield by the reaction of [$Ru_3(CO)_{11}(MeCN)$] and PhC≡CPh at room temperature. Here, we report a facile synthesis and the structure of the orange–yellow isomer (II).



(I) *R* = Ph



(II) *R* = Ph

The structure analysis of (II) showed that the crystal contains discrete molecules of [$Ru_3(CO)_8(C_4Ph_4)$] (Fig. 1) and is isostructural with the black isomer of the Fe analogue (Dodge & Schomaker, 1965). The open triruthenium unit is coordinated to the organic ligand C_4Ph_4, formed by the dimerization of diphenylacetylene, and to eight carbonyl groups (six terminal and two bridging). The central Ru(1) atom is σ-bonded to the butadiene moiety to form an RuC_4 metallacyclopentadiene (*M*Cp) ring. Each of the other two metal atoms is η^4-bonded to the C atoms of the *M*Cp ring, so that the Ru_3C_4 core forms a pentagonal bipyramidal arrangement, with the two terminal metal atoms, Ru(2) and Ru(3), in the axial positions. Overall, the molecule displays an approximate twofold axis [defined by Ru(1) and the midpoint of C(5)—C(6)], while the $Ru_3C_4(CO)_8$ fragment (*i.e.* excluding the Ph rings) has approximate *mm*2 (C_{2v}) point symmetry. This $Ru_3C_4(CO)_8$ moiety is analagous to those found in the structures of [$Ru_3(HC_2{}^tBu)_3(CO)_8$], (III) (Sappa *et al.*, 1980), and [$Ru_3(C_{12}H_{18})(CO)_8$], (IV) (Rosenberg *et al.*, 1981).

Within experimental error the two metal–metal bonds are equal and almost perpendicular to each other. These values are similar to those found in (III) and (IV); the rather short Ru—Ru distances were attributed to the presence of the bridging CO groups (Sappa *et al.*, 1980; Rosenberg *et al.*, 1981). The non-bonded Ru(2)···Ru(3) distance is 3.7461 (7) Å.

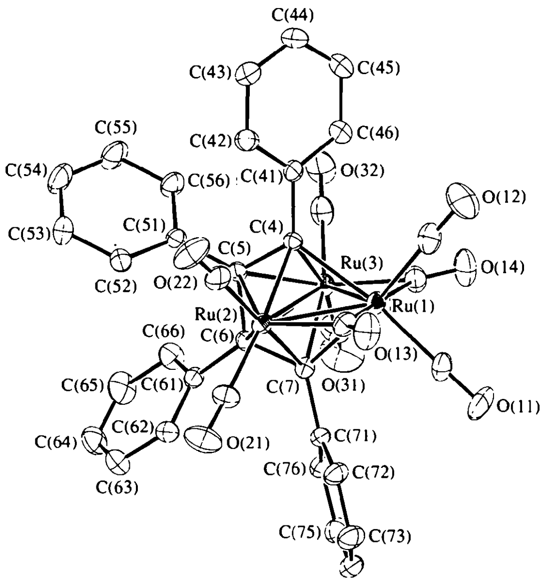


Fig. 1. Molecular structure of the title compound showing the displacement ellipsoids drawn at the 30% probability level. In the CO groups, both atoms are identically numbered; only O atoms are labelled.

The *M*Cp ring is planar (mean deviation from the plane 0.013 Å) and its geometric parameters are comparable with those observed in (III) and (IV). The double bonds of the butadiene moiety are delocalized over the three C—C bonds and, although the central one is longer, the difference is of marginal statistical significance. The Ru(2)—C(7) and Ru(3)—C(4) distances are significantly shorter than the other six Ru—C π bonds, which have a mean length of 2.334 Å. The same feature was present in the black isomer of the Fe analogue (Dodge & Schomaker, 1965), but it was less noticeable since in that study the e.s.d.'s were *ca* five times larger than in the present one. It is difficult to envisage the origin of this asymmetry since (*a*) the *M*Cp ring is quite symmetrical and perpendicular to the Ru_3 plane (dihedral angle 89.4°), (*b*) both terminal metal atoms are equidistant from the plane of the *M*Cp ring [Ru(2) 1.8718, Ru(3) 1.8741 Å], and (*c*) the bonds opposite to Ru(2)—C(7) and Ru(3)—C(4) [*i.e.* Ru(2)—C(4) and Ru(3)—C(7)] do not show any elongation.

The C—C distances between the *M*Cp and Ph rings (mean 1.491 Å) reveal partial double-bond character and, therefore, the existence of an extended conjugated system involving the *M*Cp and Ph rings. The *M*Cp/Ph inter-ring twist angles are in the range 53.2–60.3° and are all in the same sense. This arrangement is a compromise between coplanarity (*i.e.* dihedral angles 0°), which would maximize the π overlap between rings, and perpendicularity (*i.e.* dihedral angles 90°), which would minimize intramolecular non-bonded contacts

There are two clearly different groups of terminal Ru—C(CO) bond lengths: those involving C(11) and C(12) (average 1.920 Å) and the other four, which are significantly shorter (average 1.883 Å). The latter values are comparable with the mean of 1.896 Å reported by Orpen *et al.* (1989), whereas the longer values are affected by the *trans* influence of the alkyl ligand (Appleton, Clark & Manzer, 1973; Shustorovich, Porai-Koshits & Buslaev, 1975). Similar differences were observed in (III) and (IV) and in other Ru complexes containing *trans*-influenced CO groups, *e.g.* $[Ru_3(C_5H_4)(CO)_{10}]$ (Arce, De Sanctis, Manzur & Capparelli, 1994) and $[Ru_2(C_5H_4CHCH_3)(CO)_6]$ (Capparelli, De Sanctis & Arce, 1995). The terminal carbonyl ligands show little variability in their geometry: the six C—O distances (average 1.135 Å) vary by no more than 0.030 Å (*i.e. ca* five e.s.d.'s), while the corresponding Ru—C—O angles are within 3.1° of 180° (*i.e. ca* six e.s.d.'s). The bridging CO groups are approximately coplanar with the Ru_3 moiety, and the O atoms are bent towards Ru(1) [O(13) 2.9, O(14) 3.9° from the bisectors of the corresponding Ru—C(O)—Ru angles].

Experimental

To a solution of PhC≡CPh (80 mg, 0.45 mmol, *i.e.* threefold excess) in tetrahydrofuran (50 cm^3) was added $[Ru_3(CO)_{10}(MeCN)_2]$ (100 mg, 0.15 mmol). This mixture was refluxed under nitrogen for 4 h, after which time a red–orange solution was obtained. Removal of the solvent *in vacuo* and TLC separation (SiO_2, *n*-hexane) gave two bands, which produced orange–yellow (II) (98 mg, 75% yield, R_f = 0.7) and red–violet (I) (16 mg, 12% yield, R_f = 0.3). Crystals of (I) could not be obtained due to the low yield of the product. For (II), crystals suitable for X-ray analysis were obtained by slow evaporation of a cyclohexane solution. Spectroscopic data for (II): IR [cyclohexane, ν(CO), cm^{-1}]: 2070*m*, 2025*vs*, 2015*s*, 1975*s*, 1880*m*, 1859*m*; ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 7.3*m* (4H), 7.0*m* (6H), 6.8*m* (6H), 6.1*m* (4H).

Crystal data

$[Ru_3(C_{28}H_{20})(CO)_8]$
$M_r = 883.76$
Monoclinic
$P2_1/c$
$a = 9.556\,(3)$ Å
$b = 18.694\,(4)$ Å
$c = 18.465\,(2)$ Å
$\beta = 95.77\,(1)°$
$V = 3281\,(1)$ Å^3
$Z = 4$
$D_x = 1.789$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 16.9–21.1°$
$\mu = 1.415$ mm^{-1}
$T = 295\,(1)$ K
Plate
0.47 × 0.28 × 0.12 mm
Orange–yellow

Data collection

Rigaku AFC-7*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.72$, $T_{max} = 1.00$
6368 measured reflections
6037 independent reflections
4924 observed reflections $[I > 3\sigma(I)]$

$R_{int} = 0.037$
$\theta_{max} = 25.0°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 22$
$l = -21 \rightarrow 21$
3 standard reflections monitored every 150 reflections intensity decay: 9.85%

Refinement

Refinement on *F*
$R = 0.0336$
$wR = 0.0490$
$S = 2.012$
4924 reflections
444 parameters
Only H-atom *U*'s refined
Weighting scheme based on measured e.s.d.'s

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.79$ e Å^{-3}
$\Delta\rho_{min} = -0.87$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Ru(1)	0.14602 (4)	0.16040 (2)	0.69780 (2)	0.0349 (1)
Ru(2)	0.42374 (4)	0.17918 (2)	0.70264 (2)	0.03102 (9)
Ru(3)	0.18562 (4)	0.08807 (2)	0.82299 (2)	0.0347 (1)
O(11)	−0.0196 (5)	0.0886 (3)	0.5683 (2)	0.085 (2)
O(12)	−0.0550 (6)	0.2872 (3)	0.6832 (3)	0.103 (2)
O(13)	0.2577 (4)	0.2467 (2)	0.5721 (2)	0.066 (1)
O(14)	−0.1185 (4)	0.1057 (3)	0.7641 (2)	0.070 (1)
O(21)	0.6374 (5)	0.1303 (3)	0.6036 (2)	0.080 (2)
O(22)	0.5946 (5)	0.3158 (2)	0.7048 (2)	0.077 (2)
O(31)	0.1132 (7)	−0.0633 (3)	0.8633 (3)	0.107 (2)
O(32)	0.0908 (5)	0.1208 (3)	0.9706 (2)	0.076 (2)
C(4)	0.2822 (4)	0.1953 (2)	0.7958 (2)	0.028 (1)
C(5)	0.3993 (4)	0.1493 (2)	0.8231 (2)	0.029 (1)
C(6)	0.4086 (4)	0.0819 (2)	0.7826 (2)	0.031 (1)
C(7)	0.3059 (4)	0.0752 (2)	0.7195 (2)	0.031 (1)
C(11)	0.0428 (6)	0.1156 (3)	0.6149 (3)	0.051 (2)
C(12)	0.0215 (6)	0.2407 (3)	0.6890 (3)	0.060 (2)
C(13)	0.2742 (5)	0.2150 (3)	0.6254 (3)	0.044 (1)
C(14)	0.0018 (5)	0.1108 (3)	0.7651 (3)	0.048 (2)
C(21)	0.5553 (6)	0.1479 (3)	0.6408 (3)	0.045 (1)
C(22)	0.5300 (6)	0.2647 (3)	0.7061 (2)	0.047 (2)
C(31)	0.1420 (6)	−0.0065 (3)	0.8466 (3)	0.061 (2)
C(32)	0.1261 (5)	0.1091 (3)	0.9150 (3)	0.049 (2)
C(41)	0.2545 (5)	0.2636 (2)	0.8337 (2)	0.033 (1)
C(42)	0.3569 (5)	0.3144 (3)	0.8505 (3)	0.043 (1)
C(43)	0.3274 (6)	0.3768 (3)	0.8880 (3)	0.055 (2)
C(44)	0.1975 (7)	0.3884 (3)	0.9090 (3)	0.062 (2)
C(45)	0.0941 (6)	0.3385 (4)	0.8936 (4)	0.065 (2)
C(46)	0.1225 (5)	0.2771 (3)	0.8557 (3)	0.049 (2)
C(51)	0.5055 (5)	0.1670 (2)	0.8850 (2)	0.031 (1)
C(52)	0.6474 (5)	0.1698 (3)	0.8747 (3)	0.043 (1)
C(53)	0.7472 (6)	0.1848 (3)	0.9322 (3)	0.058 (2)
C(54)	0.7060 (7)	0.1966 (4)	1.0007 (3)	0.066 (2)
C(55)	0.5673 (7)	0.1955 (4)	1.0116 (3)	0.062 (2)
C(56)	0.4658 (6)	0.1803 (3)	0.9547 (3)	0.046 (1)
C(61)	0.5171 (5)	0.0268 (2)	0.8064 (2)	0.034 (1)
C(62)	0.6130 (5)	0.0019 (3)	0.7615 (3)	0.042 (1)
C(63)	0.7157 (6)	−0.0470 (3)	0.7874 (3)	0.055 (2)
C(64)	0.7233 (6)	−0.0720 (3)	0.8571 (4)	0.060 (2)
C(65)	0.6251 (7)	−0.0493 (3)	0.9017 (3)	0.063 (2)
C(66)	0.5219 (6)	−0.0005 (3)	0.8772 (3)	0.047 (1)
C(71)	0.3022 (5)	0.0124 (2)	0.6700 (2)	0.034 (1)
C(72)	0.3158 (6)	0.0219 (3)	0.5961 (3)	0.046 (1)
C(73)	0.3122 (6)	−0.0352 (3)	0.5487 (3)	0.053 (2)

C(74)	0.2930 (6)	−0.1036 (3)	0.5737 (3)	0.054 (2)
C(75)	0.2783 (6)	−0.1142 (3)	0.6464 (3)	0.049 (2)
C(76)	0.2838 (5)	−0.0570 (3)	0.6943 (3)	0.041 (1)

Table 2. *Selected geometric parameters* (Å, °)

Ru(1)—Ru(2)	2.6696 (8)	Ru(3)—C(4)	2.283 (4)
Ru(1)—Ru(3)	2.6717 (6)	Ru(3)—C(5)	2.341 (4)
Ru(1)—C(4)	2.219 (4)	Ru(3)—C(6)	2.329 (4)
Ru(1)—C(7)	2.216 (4)	Ru(3)—C(7)	2.339 (4)
Ru(1)—C(11)	1.928 (5)	Ru(3)—C(14)	2.008 (5)
Ru(1)—C(12)	1.912 (6)	Ru(3)—C(31)	1.878 (6)
Ru(1)—C(13)	2.159 (5)	Ru(3)—C(32)	1.887 (6)
Ru(1)—C(14)	2.155 (5)	C(4)—C(5)	1.459 (6)
Ru(2)—C(4)	2.312 (4)	C(4)—C(41)	1.492 (6)
Ru(2)—C(5)	2.327 (4)	C(5)—C(6)	1.472 (6)
Ru(2)—C(6)	2.356 (4)	C(5)—C(51)	1.488 (6)
Ru(2)—C(7)	2.284 (4)	C(6)—C(7)	1.452 (6)
Ru(2)—C(13)	2.029 (5)	C(6)—C(61)	1.496 (6)
Ru(2)—C(21)	1.875 (5)	C(7)—C(71)	1.486 (6)
Ru(2)—C(22)	1.892 (6)		
Ru(2)—Ru(1)—Ru(3)	89.07 (2)	C(13)—Ru(2)—C(22)	94.7 (2)
Ru(2)—Ru(1)—C(11)	120.7 (2)	C(21)—Ru(2)—C(22)	83.9 (2)
Ru(2)—Ru(1)—C(12)	120.5 (2)	Ru(1)—Ru(3)—C(31)	131.5 (2)
Ru(3)—Ru(1)—C(11)	118.9 (2)	Ru(1)—Ru(3)—C(32)	130.1 (2)
Ru(3)—Ru(1)—C(12)	120.2 (2)	C(4)—Ru(3)—C(31)	169.0 (2)
C(4)—Ru(1)—C(7)	74.1 (2)	C(7)—Ru(3)—C(32)	167.0 (2)
C(4)—Ru(1)—C(11)	170.8 (2)	Ru(1)—C(4)—C(5)	118.4 (3)
C(7)—Ru(1)—C(12)	172.6 (2)	C(4)—C(5)—C(6)	114.3 (4)
C(11)—Ru(1)—C(12)	90.5 (2)	C(5)—C(6)—C(7)	114.3 (4)
C(11)—Ru(1)—C(13)	89.5 (2)	Ru(1)—C(7)—C(6)	118.8 (3)
C(11)—Ru(1)—C(14)	88.0 (2)	Ru(1)—C(13)—Ru(2)	79.1 (2)
C(12)—Ru(1)—C(13)	87.9 (2)	Ru(1)—C(13)—O(13)	137.6 (4)
C(12)—Ru(1)—C(14)	87.6 (2)	Ru(2)—C(13)—O(13)	143.3 (5)
C(13)—Ru(1)—C(14)	174.8 (2)	Ru(1)—C(14)—Ru(3)	79.8 (2)
Ru(1)—Ru(2)—C(21)	131.6 (2)	Ru(1)—C(14)—O(14)	136.2 (4)
Ru(1)—Ru(2)—C(22)	129.8 (2)	Ru(3)—C(14)—O(14)	144.0 (5)
C(13)—Ru(2)—C(21)	98.2 (2)		

The structure was solved by Patterson methods (*DIRDIF*92; Beurskens *et al.*, 1992). H atoms were placed in calculated positions (C—H 0.96 Å) and assigned fixed coordinates and refined isotropic displacement parameters.

MSC/AFC Difractometer Control Software (Molecular Structure Corporation, 1993*a*) was used for data collection and cell refinement. All other calculations, drawings and publication material were made using the *TEXSAN* software package (Molecular Structure Corporation, 1993*b*).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1098). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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