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Cluster chemistry

LXX *. Preparation and X-ray structure of $Ru_3\{\mu_3\text{-}C_2(CO_2Me)_2\}(\mu\text{-}CO)(CO)_7(PMe_2Ph)_2$

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Abstract

The title complex has been obtained from a reaction between $Ru_3(CO)_{10}(PMe_2Ph)_2$ and $C_2(CO_2Me)_2$. The alkyne is attached to the closed triangular Ru_3 cluster in the usual μ_3-η^2-‖ mode, the two Ru atoms to which the alkyne is σ-bonded also bearing the PMe_2Ph ligands, and being bridged by a CO group. A minor product also isolated from this reaction was partially characterised as $Ru_3\{C_2(CO_2Me)_2\}_3(CO)_6(PMe_2Ph)_2$.

Introduction

Triruthenium clusters bearing μ_3-alkyne ligands have only recently been obtained from $[ppn][Ru_3(\mu\text{-}Cl)(CO)_{10}]$ [2] or $Ru_3(CO)_{10}(NCMe)_2$ [3] as precursors, the reactions with $Ru_3(CO)_{12}$ usually affording clusters containing oligomeric ligands [4]. The only structurally characterised complex of this type is $Ru_3\{\mu_3\text{-}HC_2CO_2Os_3(\mu\text{-}H)(CO)_{10}\}(\mu\text{-}CO)(CO)_{10}$ (**1**) [3]. Similarly, reactions between $Ru_3(CO)_{12}$ and $C_2(CO_2Me)_2$ have been reported to give products containing ligands formed by oligomerisation of the alkyne on the cluster; the binuclear complex $Ru_2\{\mu\text{-}C_4(CO_2Me)_4\}(CO)_6$ has also been described [5]. We describe herein the molecular structure of a further example of a complex containing a cluster-bound alkyne, which was obtained from the reaction between $Ru_3(CO)_{10}(PMe_2Ph)_2$ [6] and $C_2(CO_2Me)_2$.

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* For Part LXIX see ref. 1.

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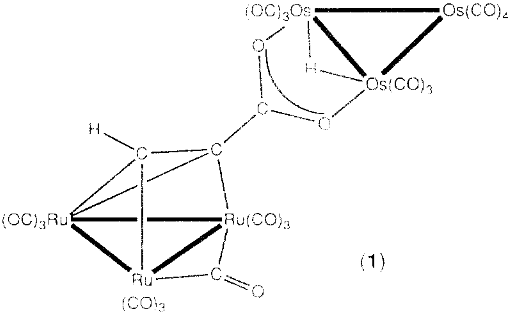
Results

A mixture of $Ru_3(CO)_{10}(PMe_2Ph)_2$ and $C_2(CO_2Me)_2$ was heated for 45 min in refluxing tetrahydrofuran. Separation by preparative TLC showed that a multitude of products had formed. One of these was crystallised from a mixture of dichloromethane and methanol to give red needles, which were characterised as the mono-alkyne complex $Ru_3\{\mu_3\text{-}C_2(CO_2Me)_2\}(\mu\text{-}CO)(CO)_7(PMe_2Ph)_2$ (**2**) by the usual analytical techniques and by a single crystal X-ray study. The solution IR spectrum showed only four terminal $\nu(CO)$ bands together with an absorption from a bridging CO ligand at 1802 cm^{-1}. The ^1H NMR spectrum of **2** contained a doublet at δ 1.92 for the P-Me groups and a singlet at δ 3.51 assigned to the OMe protons. The presence of single resonances for both types of proton indicated that the molecule was highly symmetrical. The phenyl protons appeared at δ ca. 7.43. The FAB mass spectrum contained a peak at m/z 946, assigned to $[M-H]^+$, which fragmented by sequential loss of the eight CO groups; competitive loss of OMe and CO occurred from the ion $[M-7CO]^+$. These data were consistent with a structure involving μ_3-η-$\|$ coordination of a $C_2(CO_2Me)_2$ ligand to the Ru_3 triangle, which was confirmed by an X-ray study.

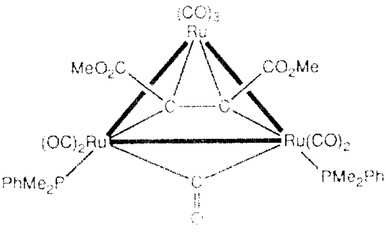
Molecular structure of $Ru_3\{\mu_3\text{-}C_2(CO_2Me)_2\}(\mu\text{-}CO)(CO)_7(PMe_2Ph)_2$ *(2)*

A molecule of 2 is shown in Figure 1, while selected bond parameters are given in Table 1. The three Ru atoms form an isosceles triangle, with two short edges [Ru(1)–Ru(3) 2.717, Ru(2)–Ru(3) 2.720(1) Å], while the Ru(1)–Ru(2) separation is somewhat longer, at 2.817(1) Å. The former distances are about 0.03 Å shorter than those found in **1**, whereas the longer distance is the same [3].

The alkyne ligand caps one face of the triangle, being σ-bonded to two Ru atoms [Ru(1)–C(31U′) 2.085(6), Ru(2)–C(31U) 2.087(5) Å] and π-bonded to the third [Ru(3)–C(31U) 2.225(6), Ru(3)–C(31U′) 2.235(5) Å]. As expected, the two CO_2Me substituents are bent up from the C(31U)–C(31U′) vector by ca 120° from the Ru_3 plane. The central C–C bond has lengthened to 1.379(7) Å. In **2**, the alkyne is symmetrically bonded to the Ru_3 triangle, in contrast with previous examples of Ru_3 and Os_3 complexes [3]. Similarly, the asymmetry in the Ru(3)–C(31U, 31U′) distances (difference, 0.01 Å) is much less than that found in **1**, where the difference is 0.22 Å [3].



(1)



(2)

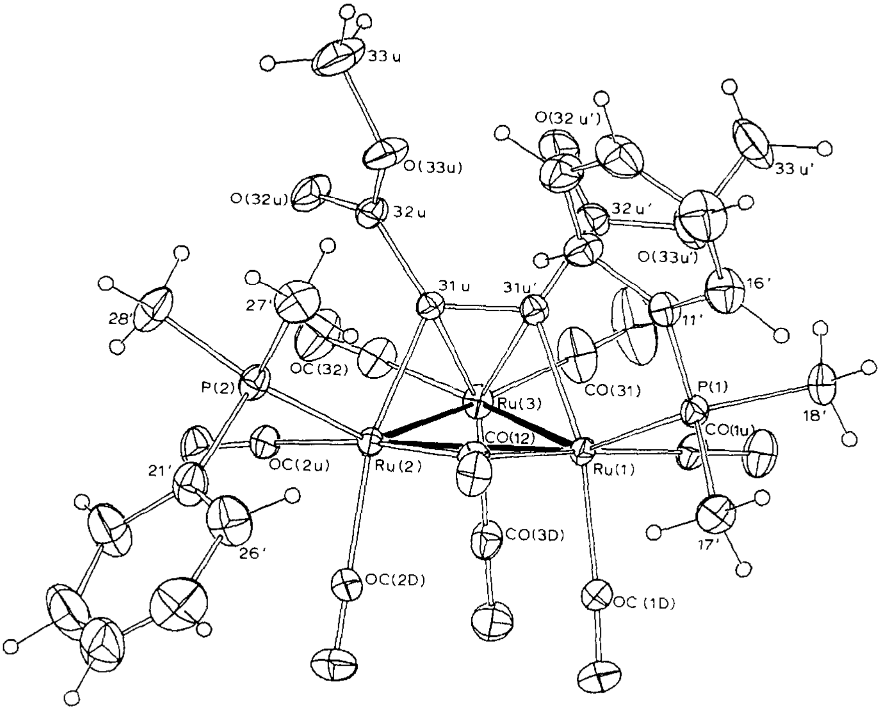


Fig. 1. Computer-generated plot of a molecule of $Ru_3\{\mu_3\text{-}C_2(CO_2Me)_2\}(\mu\text{-}CO)(CO)_7(PMe_2Ph)_2$ (**2**), showing atom numbering scheme. Non-hydrogen atoms are shown with 20% thermal ellipsoids; hydrogen atoms have arbitrary radii of 0.1 Å.

Table 1

Selected bond lengths (Å) and angles (deg.) for $Ru_3\{\mu_3\text{-}C_2(CO_2Me)_2\}(\mu\text{-}CO)(CO)_7(PMe_2Ph)_2$ (**2**)

Length		Angle	
Ru(1)-Ru(2)	2.817(1)	Ru(2)-Ru(1)-Ru(3)	58.85(3)
Ru(1)-Ru(3)	2.717(1)	Ru(1)-Ru(2)-Ru(3)	58.74(2)
Ru(2)-Ru(3)	2.720(1)	Ru(1)-Ru(3)-Ru(2)	62.41(2)
Ru(1)-P(1)	2.348(2)	Ru(2)-Ru(1)-P(1)	128.64(5)
Ru(2)-P(2)	2.346(2)	Ru(3)-Ru(1)-P(1)	151.29(5)
Ru(1)-C(12)	2.148(6)	Ru(1)-Ru(2)-P(2)	131.66(5)
Ru(2)-C(12)	2.110(6)	Ru(3)-Ru(2)-P(2)	146.66(5)
Ru(1)-C(31U′)	2.085(6)	Ru(2)-C(31U)-Ru(3)	78.2(2)
Ru(2)-C(31U)	2.087(5)	Ru(1)-C(31U′)-Ru(3)	77.9(2)
Ru(3)-C(31U)	2.225(5)	Ru(1)-C(31U′)-C(31U)	109.8(4)
Ru(3)-C(31U′)	2.235(6)	Ru(2)-C(31U)-C(31U′)	110.5(4)
		C(32U)-C(31U)-C(31U′)	123.3(5)
C(31U)-C(31U′)	1.379(7)	C(32U′)-C(31U′)-C(31U)	119.3(5)
Ru-CO		range 1.887(6)-1.955(7), av. 1.914 Å	
C-O		range 1.112(9)-1.146(9), av. 1.124 Å	
P-C(Me)		range 1.788(9)-1.813(7), av. 1.805 Å	
P-C(Ph)		1.818, 1.836(7), av. 1.827 Å	

The two Ru atoms to which the alkyne is σ-bonded are each bonded to one PMe_2Ph ligand [Ru(1)–P(1) 2.348(2), Ru(2)–P(2) 2.346(2) Å] and also bridged by the single μ-CO group [Ru(1)–C(12) 2.149(6), Ru(2)–C(12) 2.110(6) Å]. Three of the remaining CO groups are attached to Ru(3) and two each to Ru(1) and Ru(2). The two axial CO groups on Ru(1) and Ru(2) have Ru–C bonds [Ru(1)–C(1D) 1.934(7), Ru(2)–C(2D) 1.955(7)Å] which are significantly longer than those to the equatorial CO ligands which are *trans* to the Ru(1)–Ru(2) vector [Ru(1)–C(1U) 1.887(6), Ru(2)–C(2U) 1.906(6) Å] as a result of the strong *trans* influence of the Ru–C(alkyne) σ bonds.

Although both the tertiary phosphine ligands occupy equatorial sites, the two P atoms lie above the mean plane of the Ru_3 core, on the same side as the alkyne. One of the phenyl groups lies above this plane, and the other is below the plane. This arrangement minimises their mutual steric interactions. One Me group on each phosphine [C(17) and C(27)] lies respectively below and above the plane, while the other two [C(18) and C(28)] point away from the Ru_3 core.

A bright yellow minor product (**2**) isolated in small amounts from a yellow band was tentatively identified as $Ru_3\{C_2(CO_2Me)_2\}_3(CO)_6(PMe_2Ph)_2$ on the basis of its FAB mass spectrum, which containined an ion at m/z 1175, assigned to M^+. This ion fragmented by loss of six CO ligands, OMe and $C_2(CO_2Me)_2$ groups. The ^1H NMR spectrum contained several resonances in the Me and OMe regions, suggesting that all the latter were inequivalent. The molecular structure of this interesting complex could not be determined with the small amount of crystallographically non-suitable material which we obtained. However, the formula requires that the organic ligand donate either 8 e to a closed Ru_3 system, or 10 e to an open Ru_3 array. There are several ways in which this might occur (including coordination of one or more of the ester CO groups); three previous examples of $M_3(CO)_8\{(RC_2R')_3\}$ are $Fe_3(CO)_8(MeCCHCMeCHCCHMe)$ [7], $Ru_3(CO)_8\{Me_2CCCC(CH_2{}^tBu)C(CMe{=}CH_2)CH\}$ [8] and $Ru_3(CO)_8(MeCCHC^tBuCCH{=}CH^tBu)$ [9]. Further speculation is not warranted at this time.

Discussion

The reaction between $Ru_3(CO)_{10}(PMe_2Ph)_2$ and $C_2(CO_2Me)_2$, like that of $Ru_3(CO)_{12}$ [5], is complex, and only two pure compounds have been isolated from this initial study. Of these, complex **2** was isolated in 11% yield, and was characterised as containing the alkyne bonded in the usual μ_3-η^2-∥ mode to the Ru_3 cluster. This is the first occasion on which the $C_2(CO_2Me)_2$ molecule has been found on a ruthenium cluster. The coordination is symmetrical with respect to the two tertiary phosphine ligands and requires one CO to bridge an Ru–Ru bond, in this case that joining the two Ru atoms which are σ-bonded to the alkyne. Each Ru atom achieves an 18-electron count.

There are several examples of similar complexes which have been structurally characterised. These include $[ppn][Ru_3(\mu_3\text{-}C_2Ph_2)(\mu\text{-}Cl)(CO)_9]$ [2], $Ru_3(\mu\text{-}H)\{\mu_3\text{-}MeC_2(OMe)\}(CO)_9$ [10], $Ru_3(\mu\text{-}H)(\mu_3\text{-}MeC_2{}^iPr)(\mu\text{-}PPh_2)(CO)_8$ [11], $Ru_3(\mu_3\text{-}HC_2Ph)(\mu\text{-}PPh_2)_2(\mu\text{-}CO)(CO)_8$ [12] and $Ru_3(\mu_3\text{-}HC_2Ph)(\mu_3\text{-}S)(CO)_9$ [13]. All of the structures are undoubtedly influenced by the other bridging groups present, so that the present example is only the second 'simple' μ_3-alkyne derivative of $Ru_3(CO)_{12}$ to be studied structurally.

Complex **2** must be formed at an early stage in the reaction, and the plethora of products obtained with it suggests that it is highly reactive, undergoing ready modification and addition of further molecules of the alkyne. Previous studies with $Ru_3(CO)_{12}$ have demonstrated the formation of oligomeric products, and indeed, from the present reaction, a second complex was obtained in small yield, apparently containing a trimer of $C_2(CO_2Me)_2$. Further study of this system has been deferred pending the investigation of similar reactions with the related complex $Ru_3(\mu$-dppm)$(CO)_{10}$ [14].

Experimental

General experimental conditions have been described in an earlier paper [15]. A previously described method [6] was used to prepare $Ru_3(CO)_{10}(PMe_2Ph)_2$; $C_2(CO_2Me)_2$ was distilled before use.

Reaction between $Ru_3(CO)_{10}(PMe_2Ph)_2$ and $C_2(CO_2Me)_2$

A mixture of $Ru_3(CO)_{10}(PMe_2Ph)_2$ (200 mg, 0.233 mmol) and $C_2(CO_2Me)_2$ (1 ml of a 0.48 *M* solution in tetrahydrofuran) was heated in tetrahydrofuran (25 ml) at gentle reflux for 45 min. Separation by preparative TLC (acetone/light petroleum 3/7) developed fourteen bands. Band 1 (orange, R_f0.68) contained unreacted $Ru_3(CO)_{10}(PMe_2Ph)_2$ (34 mg, 17%), identified by its IR ν(CO) spectrum. Band 3 (orange, R_f0.57) was crystallised (CH_2Cl_2/MeOH) to give $Ru_3\{\mu_3\text{-}C_2(CO_2Me)_2\}(\mu\text{-}CO)(CO)_7(PMe_2Ph)_2$ (**2**) as red needles (25 mg, 11%), m.p. 154–157 °C. (Found: C, 38.12; H, 2.96; *M* (mass spectrometry), 946; $C_{30}H_{28}O_{12}P_2Ru_3$ calc.: C, 38.10; H, 2.98%; *M*, 947). IR (cyclohexane): ν(CO) 2064s, 2018vs, 1995m, 1884w, 1811(sh), 1802m cm^{-1}; IR (Nujol): ν(CO) 2064s, 2019vs, 2014vs, 1987m, 1981m, 1962m, 1807s; $\nu(C=O)$ 1707(sh), 1701m; ν(C–O) 1199m, 1190w, 1177m, 1162w cm^{-1}. ^1H NMR: δ ($CDCl_3$) 1.92 (d, *J*(PH) = 9.5, 12H, Me); 3.51 (s, 6H, OMe); 7.43 (m, 10H, Ph). FAB MS: 946, $[M-H]^+$, 4; 918, $[M-H-CO]^+$, 21; 890, $[M-H-2CO]^+$, 32; 862, $[M-H-3CO]^+$, 21; 834, $[M-H-4CO]^+$, 100; 806, $[M-H-5CO]^+$, 68; 778, $[M-H-6CO]^+$, 71; 751, $[M-H-7CO]^+$, 75; 720, $[M-7CO-OMe]^+$, 37; 692, $[M-8CO-OMe]^+$, 39. Band 10 (yellow, R_f 0.26) was crystallised (CH_2Cl_2/MeOH) to give yellow $Ru_3\{C_2(CO_2Me)_2\}_3(CO)_6(PMe_2Ph)_2 \cdot CH_2Cl_2$ (4 mg, 1%). (Found: C, 37.51; H, 3.19; *M* (mass spectrometry), 1175; $C_{40}H_{40}O_{18}P_2Ru_3$ calc.: C, 38.95; H, 3.17%; *M*, 1175). IR (CH_2Cl_2): ν(CO) 2043s, 2022s, 1987vs, 1975(sh), 1873w, 1864w cm^{-1}. ^1H NMR: δ ($CDCl_3$) 1.85–2.10 (7 × s, 12H, 4 × Me); 3.40–3.73 (5 × s, 18H, 6 × OMe); 5.29 (s, 2H, CH_2Cl_2); 7.26–7.78 (m, 10H, Ph). FAB MS: 1175, $[M]^+$, 11; 1147, $[M-CO]^+$, 100; 1116, $[M-CO-OMe]^+$, 83; 1091, $[M-3CO]^+$, 39; 1063, $[M-4CO]^+$, 56; 1033, $[M-C_2(CO_2Me)_2]^+$, 44; 1005, $[M-CO-C_2(CO_2Me)_2]^+$, 33; 977, $[M-2CO-C_2(CO_2Me)_2]^+$, 39; 949, $[M-3CO-C_2(CO_2Me)_2]^+$, 39; 918, $[M-3CO-C_2(CO_2Me)_2-OMe]^+$, 17; 890, $[M-4CO-C_2(CO_2Me)_2-OMe]^+$, 22.

The other bands contained only trace amounts of material and were not identified.

Crystallography

A unique data set was measured at ca 295 K within the limit $2\theta_{max} = 60°$ using an Enraf–Nonius CAD4 diffractometer ($2\theta/\theta$ scan mode; monochromatic Mo-K_α

Table 2

Non-hydrogen atomic coordinates and equivalent isotropic thermal parameters for $Ru_3\{\mu_3\text{-}C_2(CO_2Me)_2\}(\mu\text{-}CO)(CO)_7(PMe_2Ph)_2$ (**2**)

Atom	x	y	z	U_{eq} (Å^2)
Ru(1)	0.14798(2)	0.84565(3)	0.63566(4)	0.0340(2)
Ru(2)	0.15904(2)	0.73986(3)	0.43171(4)	0.0350(2)
Ru(3)	0.17049(3)	0.90497(3)	0.40571(4)	0.0415(2)
P(1)	0.08174(8)	0.8318(1)	0.8109(1)	0.0413(8)
P(2)	0.1013(1)	0.6212(1)	0.3770(2)	0.0507(9)
C(12)	0.1289(3)	0.7170(4)	0.6143(6)	0.046(3)
O(12)	0.1165(3)	0.6616(3)	0.6783(4)	0.061(3)
C(1U)	0.1636(3)	0.9558(4)	0.6795(6)	0.050(3)
O(1U)	0.1727(3)	1.0208(3)	0.7103(5)	0.077(3)
C(1D)	0.2281(3)	0.8145(4)	0.7177(6)	0.054(4)
O(1D)	0.2760(3)	0.7946(4)	0.7587(6)	0.092(4)
C(2U)	0.1930(3)	0.7456(4)	0.2684(5)	0.049(3)
O(2U)	0.2145(3)	0.7401(3)	0.1736(4)	0.068(3)
C(2D)	0.2401(3)	0.6860(4)	0.4819(6)	0.053(4)
O(2D)	0.2861(3)	0.6555(4)	0.5117(6)	0.095(4)
C(3D)	0.2610(3)	0.9031(4)	0.4463(6)	0.054(4)
O(3D)	0.3141(3)	0.9005(4)	0.4727(6)	0.078(4)
C(31)	0.1579(5)	1.0193(4)	0.4201(8)	0.083(5)
O(31)	0.1497(5)	1.0864(3)	0.4270(7)	0.146(7)
C(32)	0.1762(4)	0.9190(4)	0.2308(6)	0.060(4)
O(32)	0.1821(3)	0.9289(4)	0.1269(5)	0.092(4)
C(11′)	0.0009(3)	0.7869(4)	0.7954(6)	0.047(3)
C(12′)	−0.0248(3)	0.7678(5)	0.6825(6)	0.057(4)
C(13′)	−0.0865(4)	0.7323(6)	0.6730(8)	0.076(5)
C(14′)	−0.1219(4)	0.7165(6)	0.774(1)	0.088(6)
C(15′)	−0.0972(4)	0.7359(6)	0.8897(9)	0.094(7)
C(16′)	−0.0363(4)	0.7712(5)	0.8983(8)	0.076(5)
C(17′)	0.1186(4)	0.7695(5)	0.9304(6)	0.065(4)
C(18′)	0.0656(4)	0.9255(4)	0.8944(6)	0.059(4)
C(21′)	0.1503(4)	0.5284(4)	0.4011(7)	0.065(4)
C(22′)	0.1946(6)	0.5045(6)	0.3151(9)	0.103(7)
C(23′)	0.2341(7)	0.4368(8)	0.344(1)	0.14(1)
C(24′)	0.2293(8)	0.3959(7)	0.444(1)	0.13(1)
C(25′)	0.1877(7)	0.4175(6)	0.526(1)	0.126(9)
C(26′)	0.1452(5)	0.4864(5)	0.5123(9)	0.092(6)
C(27′)	0.0243(4)	0.6005(5)	0.4481(9)	0.081(6)
C(28′)	0.0792(5)	0.6171(5)	0.2158(7)	0.080(5)
C(31U)	0.0834(3)	0.8235(3)	0.4042(5)	0.035(2)
C(32U)	0.0348(3)	0.8207(4)	0.3015(5)	0.046(3)
O(32U)	0.0448(3)	0.8334(4)	0.1927(4)	0.070(3)
O(33U)	−0.0223(2)	0.7921(4)	0.3412(4)	0.070(3)
C(33U)	−0.0760(5)	0.7892(9)	0.2553(9)	0.111(8)
C(31U′)	0.0776(3)	0.8751(3)	0.5041(5)	0.038(3)
C(32U′)	0.0257(3)	0.9402(4)	0.5036(6)	0.051(3)
O(32U′)	−0.0035(3)	0.9621(4)	0.4153(5)	0.100(4)
C(33U′)	−0.0322(6)	1.0367(8)	0.623(1)	0.130(9)
O(33U′)	0.0172(3)	0.9734(4)	0.6147(5)	0.088(4)

radiation, λ 0.7107$_3$ Å); 5829 independent reflections were obtained, 5285 with $I > 3\sigma(I)$ being considered 'observed' and used in the block diagonal least squares refinement (with extinction) after Gaussian absorption correction. Anisotropic thermal parameters were refined for the non-hydrogen atoms; $(x, y, z, U_{iso})_H$ were included constrained at estimated values. Conventional residuals R, R' on $|F|$ are quoted, statistical weights derivative of $\sigma^2(I) = \sigma^2(I_{diff}) + 0.0004\sigma^4(I_{diff})$ being used. Computation used the XTAL 2.4 program system [16] implemented by S.R. Hall; neutral atom complex scattering factors were employed. Pertinent results are given in Fig.1 and Tables. Tables of structure factor amplitudes, thermal and hydrogen atom parameters and full non-hydrogen geometries are available from the authors.

Crystal and refinement data

$Ru_3\{\mu_3\text{-}C_2(CO_2Me)_2\}(\mu\text{-CO})(CO)_7(PMe_2Ph)_2 \equiv C_{30}H_{28}O_{12}P_2Ru_3, M = 945.7$, Orthorhombic, space group $P2_12_12_1$, $a = 20.472(4)$, $b = 16.326(4)$, $c = 10.858(6)$ Å, $V = 3629$ Å^3, $Z = 4$, $\rho_c = 1.73$ g cm^{-3}, $F(000) = 1864$. Crystal dimensions: 0.25 × 0.40 × 0.30 mm, $T = 295$ K, $\mu(\text{Mo-}K_\alpha) = 1.455$ cm^{-1}, $A^\star$ (min, max) = 1.38, 1.75, $R = 0.039$, $R_w = 0.046$ (preferred chirality).

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