(Lawton, Hoh, Johnson & Knisely, 1973, and references cited therein) is 2·54 Å.

The closest distances between the tribromide anion and the decamethylferrocenium cation are 3·447 (8) Å for Br(1)···C(3), and 3·30–3·36 Å for Br(1) and H atoms at the methyl C atoms C(4) and C(5). This rules out stronger bonding interactions between the two moieties.

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Structure of 1,1-Dicarbonyl-1,1-bis(dimethylphenylphosphine)-2,3-bis(methoxycarbonyl)-1-ruthenaindene, a Ruthenium(II) Complex Containing a Five-Membered Metallacycle

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Abstract. $[\overline{\mathrm{Ru(CO)_2\{C(CO_2Me){=}C(CO_2Me)C_6H_4}}\}(\mathrm{PMe_2Ph})_2]$, $[\mathrm{Ru(C_{12}H_{10}O_4)(C_8H_{11}P)_2(CO)_2}]$, $M_r = 651{\cdot}6$, triclinic, $P\bar{1}$, $a = 11{\cdot}858\ (1)$, $b = 11{\cdot}958\ (1)$, $c = 12{\cdot}970\ (2)$ Å, $\alpha = 99{\cdot}41\ (1)$, $\beta = 82{\cdot}42\ (1)$, $\gamma = 60{\cdot}93\ (1)°$, $V = 1537{\cdot}4$ Å^3, $Z = 2$, $D_x = 1{\cdot}41\ (1)$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}64$ mm^{-1}, $F(000) = 668$, $T = 290\ (2)$ K, $R = 0{\cdot}044$ for 4556 observed reflections with $F_o > 3\sigma(F_o)$ and 352 parameters. In the solid state the complex exists as discrete monomeric units, with slightly irregular octahedral geometry about the Ru atom. Within the planar five-membered metallacycle the Ru—C dis-

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tances are 2·123 (5) and 2·099 (4) Å. The average Ru—P and Ru—C(carbonyl) distances are 2·363 (1) and 1·916 (5) Å, respectively.

Introduction. We have shown that treatment of the ruthenium(II) complex $[\mathrm{RuCl_2(CO)_2(PMe_2Ph)_2}]$ with the alkyne $\mathrm{MeO_2CC{\equiv}CCO_2Me}$ results in the formation of an alkenyl complex $[\mathrm{Ru(CO)_2\{C(CO_2Me){=}C(CO_2Me)Cl\}Cl(PMe_2Ph)_2}]$. A study of the structure of the product by X-ray diffraction revealed that *cis*-addition of the Ru—Cl bond to the alkyne had occurred and we suggested a mechanism involving substitution of a carbonyl ligand by the alkyne, intramolecular rearrangement to give five-coordinate $[\mathrm{Ru(CO)\{C(CO_2Me){=}C(CO_2Me)Cl\}Cl(PMe_2Ph)_2}]$, and reintroduction of the carbonyl ligand into the vacant coordination site (Holland, Howard & Mawby, 1983).

We have also found that aryl complexes of ruthenium(II), $[\mathrm{Ru(CO)_2(C_6H_4}X\mathrm{\text{-}4)Cl(PMe_2Ph)_2}]$ (X = H, Me, OMe or Cl) react with $\mathrm{MeO_2CC{\equiv}CCO_2Me}$, but in these reactions the alkyne is inserted into the metal—aryl rather than the metal—chlorine bond. When heated in $\mathrm{CHCl_3}$ solution, the products $[\mathrm{Ru(CO)_2\{C(CO_2Me){=}C(CO_2Me)(C_6H_4}X\mathrm{\text{-}4)\}Cl(PMe_2Ph)_2}]$ yielded several species: in each case, elemental analysis suggested that one of the reactions which took place involved the elimination of HCl. Assuming that the addition of the ruthenium—aryl bond to the alkyne was *cis*, we suggested on the basis of NMR evidence that HCl elimination might result from attack by the metal on the aryl group, yielding five-membered metallacycles $[\overline{\mathrm{Ru(CO)_2\{C(CO_2Me){=}C(CO_2Me)C_6H_3}}X\}\mathrm{(PMe_2Ph)_2}]$ (Crook, Chamberlain & Mawby, 1989). Here we report the structure of one of these complexes (X = H) by X-ray diffraction.

Experimental. The complex was prepared as described previously (Crook *et al.*, 1989). Crystals suitable for X-ray analysis were obtained by recrystallization from a mixture of ethanol and water. A needle-shaped single crystal of dimensions 0·09 × 0·14 × 0·28 mm was selected for crystal structure determination. Intensity data were collected at room temperature on an Enraf–Nonius CAD-4 diffractometer using Zr-filtered Mo $K\alpha$ radiation (λ = 0·71073 Å). Unit-cell dimensions were obtained by least-squares methods for 25 reflections in the range $8 < \theta < 15°$. Data were collected to $\theta_{\max} = 29°$ using the $\omega/2\theta$ scan method and were corrected for Lorentz and polarization effects. Corrections for absorption were not applied. The range for h was -16 to 16, for k -16 to 16 and for l -17 to 0. The intensity variation over the period of data collection was less than 3%. Of the 8174 independent reflections measured, 4556 with $F_o > 3\sigma(F_o)$ were used in the refinement, $R_{\rm int} = 0{\cdot}025$. The structure was solved by standard Patterson techniques and refined using full-matrix least-squares refinement based on F with weights $w = [\sigma^2(F_o)]^{-1}$. Anisotropic thermal parameters for non-H atoms were included in final cycles. The H positions were estimated geometrically; H atoms were given isotropic thermal factors derived from those of the parent atoms and were not refined. Highest peak in the final difference Fourier synthesis: 0·41 e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974); shift-to-e.s.d. ratio in final refinement cycle $< 0{\cdot}01$. Final $R = 0{\cdot}044$, $wR = 0{\cdot}062$, $S = 1{\cdot}082$.

Calculations were performed using the *SDP* system of computer programs (Enraf–Nonius, 1979).

Discussion. The structure consists of discrete molecules with no intermolecular contacts between non-H atoms shorter than 3·35 Å. As shown in Fig. 1, the complex is indeed the metallacycle $[\overline{\mathrm{Ru(CO)_2\{C(CO_2Me){=}C(CO_2Me)C_6H_4}}\}\mathrm{(PMe_2Ph)_2}]$. Atomic coordinates are listed in Table 1,* and selected bond lengths and angles in Table 2.

The ligand arrangement around the Ru atom is not greatly distorted from regular octahedral. The most notable departure from regular geometry is the angle C(17)—Ru—C(26) [77·5 (2)°], which forms part of the five-membered ring. The remaining angles in the ring, which is essentially planar and also coplanar with the six-membered ring to which it is

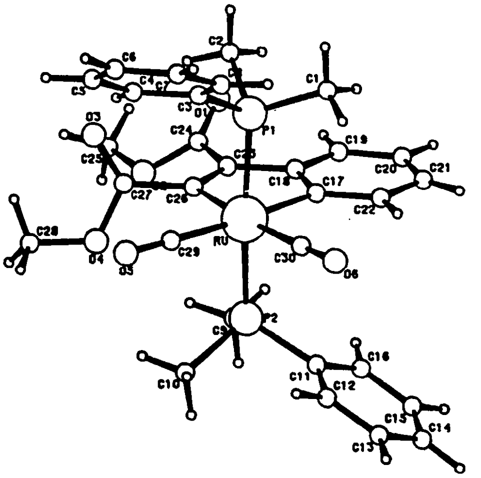

Fig. 1. The molecular structure of $[\overline{\mathrm{Ru(CO)_2\{C(CO_2Me){=}C(CO_2Me)C_6H_4}}\}\mathrm{(PMe_2Ph)_2}]$ showing the atomic numbering scheme.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52199 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates and B_{eq} values for the non-*H *atoms*

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq} (Å^2)
Ru	−0·26965 (3)	−0·13149 (3)	−0·23729 (3)	2·507 (6)
P(1)	−0·2006 (1)	−0·3572 (1)	−0·3005 (1)	2·92 (3)
P(2)	−0·3140 (1)	0·0883 (1)	−0·1984 (1)	3·13 (3)
O(1)	0·2354 (3)	−0·3183 (4)	−0·4336 (4)	5·9 (1)
O(2)	0·1723 (3)	−0·1970 (4)	−0·2636 (4)	5·9 (1)
O(3)	0·0515 (4)	−0·3294 (3)	−0·1364 (3)	5·1 (1)
O(4)	−0·0658 (3)	−0·1086 (3)	−0·0661 (3)	4·35 (9)
O(5)	−0·3378 (4)	−0·1212 (4)	0·0014 (3)	5·5 (1)
O(6)	−0·5481 (3)	−0·0479 (4)	−0·2666 (4)	6·3 (1)
C(1)	−0·2731 (5)	−0·3893 (5)	−0·4084 (4)	4·2 (1)
C(2)	−0·0243 (5)	−0·4718 (5)	−0·3548 (4)	4·2 (1)
C(3)	−0·2415 (4)	−0·4307 (4)	−0·1998 (4)	3·2 (1)
C(4)	−0·1699 (6)	−0·4541 (5)	−0·1193 (5)	4·9 (1)
C(5)	−0·1998 (7)	−0·5086 (5)	−0·0403 (5)	6·0 (2)
C(6)	−0·2943 (7)	−0·5394 (5)	−0·0405 (5)	6·6 (2)
C(7)	−0·3643 (6)	−0·5144 (5)	−0·1159 (6)	6·0 (2)
C(8)	−0·3371 (5)	−0·4639 (5)	−0·1993 (5)	4·4 (1)
C(9)	−0·1740 (5)	0·1105 (4)	−0·2361 (5)	4·3 (1)
C(10)	−0·3753 (5)	0·1717 (5)	−0·0549 (4)	4·5 (1)
C(11)	−0·4408 (4)	0·2023 (4)	−0·2559 (4)	3·4 (1)
C(12)	−0·5724 (5)	0·2577 (6)	−0·2100 (5)	5·1 (2)
C(13)	−0·6711 (6)	0·3489 (7)	−0·2498 (5)	6·3 (2)
C(14)	−0·6334 (7)	0·3863 (6)	−0·3387 (5)	6·7 (2)
C(15)	−0·5034 (7)	0·3308 (6)	−0·3843 (5)	6·6 (2)
C(16)	−0·4050 (6)	0·2387 (6)	−0·3470 (5)	5·6 (2)
C(17)	−0·1947 (4)	−0·1426 (4)	−0·3989 (4)	3·3 (1)
C(18)	−0·0605 (4)	−0·1809 (4)	−0·4256 (4)	3·4 (1)
C(19)	0·0003 (5)	−0·1884 (5)	−0·5301 (4)	4·8 (1)
C(20)	−0·0725 (6)	−0·1611 (6)	−0·6075 (4)	5·9 (2)
C(21)	−0·2006 (6)	−0·1253 (6)	−0·5842 (5)	5·7 (2)
C(22)	−0·2649 (5)	−0·1156 (5)	−0·4798 (4)	4·5 (1)
C(23)	0·0058 (4)	−0·2057 (4)	−0·3366 (4)	3·4 (1)
C(24)	0·1481 (4)	−0·2476 (5)	−0·3531 (5)	4·2 (1)
C(25)	0·3101 (6)	−0·2435 (9)	−0·2677 (8)	10·4 (3)
C(26)	−0·0708 (4)	−0·1897 (4)	−0·2420 (3)	2·76 (9)
C(27)	−0·0177 (4)	−0·2190 (4)	−0·1466 (4)	3·5 (1)
C(28)	−0·0309 (6)	−0·1281 (7)	0·0337 (5)	6·7 (2)
C(29)	−0·3136 (4)	−0·1255 (4)	−0·0880 (4)	3·3 (1)
C(30)	−0·4451 (4)	−0·0781 (5)	−0·2545 (4)	4·0 (1)

Table 2. *Selected bond distances* (Å) *and angles* (°) *for the non-*H *atoms*

Ru—P(1)	2·362 (1)	O(3)—C(27)	1·211 (6)
Ru—P(2)	2·364 (1)	O(4)—C(27)	1·348 (6)
Ru—C(17)	2·123 (5)	O(4)—C(28)	1·432 (7)
Ru—C(26)	2·099 (4)	O(5)—C(29)	1·143 (6)
Ru—C(29)	1·918 (5)	O(6)—C(30)	1·134 (6)
Ru—C(30)	1·914 (5)	C(17)—C(18)	1·398 (7)
P(1)—C(1)	1·819 (5)	C(17)—C(22)	1·406 (7)
P(1)—C(2)	1·805 (5)	C(18)—C(19)	1·417 (7)
P(1)—C(3)	1·826 (5)	C(18)—C(23)	1·470 (7)
P(2)—C(9)	1·823 (5)	C(19)—C(20)	1·386 (9)
P(2)—C(10)	1·821 (6)	C(20)—C(21)	1·337 (9)
P(2)—C(11)	1·820 (5)	C(21)—C(22)	1·421 (8)
O(1)—C(24)	1·205 (7)	C(23)—C(24)	1·481 (7)
O(2)—C(24)	1·350 (7)	C(23)—C(26)	1·352 (7)
O(2)—C(25)	1·437 (8)	C(26)—C(27)	1·478 (6)

Phenyl rings: C(3)—C(8) 1·339 (11)–1·410 (8), average 1·376 (9)
C(11)—C(16) 1·342 (11)–1·408 (8), average 1·379 (9)

P(1)—Ru—P(2)	171·0 (1)	P(1)—C(3)—C(4)	117·5 (4)
P(1)—Ru—C(17)	86·3 (1)	P(1)—C(3)—C(8)	123·3 (4)
P(1)—Ru—C(26)	88·8 (1)	P(2)—C(11)—C(12)	120·9 (4)
P(1)—Ru—C(29)	94·6 (2)	P(2)—C(11)—C(16)	120·7 (4)
P(1)—Ru—C(30)	90·9 (2)	Ru—C(17)—C(18)	115·0 (4)
P(2)—Ru—C(17)	85·3 (1)	Ru—C(17)—C(22)	127·2 (4)
P(2)—Ru—C(26)	86·1 (1)	C(18)—C(17)—C(22)	117·8 (5)
P(2)—Ru—C(29)	93·2 (2)	C(17)—C(18)—C(19)	120·4 (5)
P(2)—Ru—C(30)	93·0 (2)	C(17)—C(18)—C(23)	114·7 (4)
C(17)—Ru—C(26)	77·5 (2)	C(19)—C(18)—C(23)	124·8 (5)
C(17)—Ru—C(29)	171·9 (2)	C(18)—C(19)—C(20)	119·9 (6)
C(17)—Ru—C(30)	94·1 (2)	C(19)—C(20)—C(21)	120·8 (6)
C(26)—Ru—C(29)	94·4 (2)	C(20)—C(21)—C(22)	120·7 (6)
C(26)—Ru—C(30)	171·6 (2)	C(17)—C(22)—C(21)	120·3 (5)
C(29)—Ru—C(30)	93·9 (2)	C(18)—C(23)—C(24)	120·8 (5)
Ru—P(1)—C(1)	113·7 (2)	C(18)—C(23)—C(26)	115·7 (4)
Ru—P(1)—C(2)	116·5 (2)	C(24)—C(23)—C(26)	123·5 (5)
Ru—P(1)—C(3)	115·3 (2)	O(1)—C(24)—O(2)	121·8 (5)
C(1)—P(1)—C(2)	103·5 (3)	O(1)—C(24)—C(23)	126·9 (5)
C(1)—P(1)—C(3)	103·8 (3)	O(2)—C(24)—C(23)	111·3 (5)
C(2)—P(1)—C(3)	102·3 (3)	Ru—C(26)—C(23)	117·1 (3)
Ru—P(2)—C(9)	117·1 (2)	Ru—C(26)—C(27)	120·1 (3)
Ru—P(2)—C(10)	113·3 (2)	C(23)—C(26)—C(27)	122·7 (4)
Ru—P(2)—C(11)	116·0 (2)	O(3)—C(27)—O(4)	122·7 (5)
C(9)—P(2)—C(10)	101·9 (3)	O(3)—C(27)—C(26)	124·7 (5)
C(9)—P(2)—C(11)	103·7 (3)	O(4)—C(27)—C(26)	112·3 (4)
C(10)—P(2)—C(11)	102·8 (3)	Ru—C(29)—O(5)	178·5 (5)
C(24)—O(2)—C(25)	115·4 (6)	Ru—C(30)—O(6)	178·8 (6)
C(27)—O(4)—C(28)	116·4 (5)		

Phenyl rings: C(3)—C(8) 118·6 (6)–122·3 (7), average 120·0 (6)
C(11)—C(16) 118·3 (5)–122·7 (7), average 120·0 (6)

fused, range from 114·7 (4) to 117·1 (3)°. Delocalization evidently does not extend to the carboxylate O atoms, which are twisted out of the plane of the rings, presumably for steric reasons. A similar effect was observed for the alkenyl complex $[Ru(CO)_2\{C(CO_2Me){=}C(CO_2Me)Cl\}Cl(PMe_2Ph)_2]$ (Holland *et al.*, 1983).

The lengths of the bonds between the metal and the sp^2-hybridized C atoms C(17) and C(26) do not differ greatly. The bond to the alkene C(26) atom [2·099 (4) Å] is shorter than that in $[Ru(CO)_2\{C(CO_2Me){=}C(CO_2Me)Cl\}Cl(PMe_2Ph)_2]$ [2·16 (2) Å] (Holland *et al.*, 1983) where the organic ligand is monodentate, but a little longer than those in two other ruthenium(II) complexes, $[Ru\{CH{=}C(CO_2Bu)Me\}H(PPh_3)_3]$ [2·061 (10) Å] (Komiya, Ito, Cowie, Yamamoto & Ibers, 1976) and $[Ru\{C(CO_2Me){=}C(CO_2Me)H\}\{\eta^5\text{-}C_5H_4C(CF_3)_2OH\}(PPh_3)]$ [2·035 (4) Å] (Raghavan & Davis, 1975), in which the organic ligand is chelated to the metal (in these instances through the carbonyl O atom of a carboxylate group). The length of the alkene C=C bond C(23)—C(26) [1·352 (7) Å] compares with 1·141 (3) and 1·368 (15) Å for the corresponding bonds in $[Ru(CO)_2\{C(CO_2Me){=}C(CO_2Me)Cl\}Cl(PMe_2Ph)_2]$ (Holland *et al.*, 1983) and $[Ru\{CH{=}C(CO_2Bu)Me\}H(PPh_3)_3]$ (Komiya *et al.*, 1976), and the 'standard' C=C bond length of 1·33 Å.

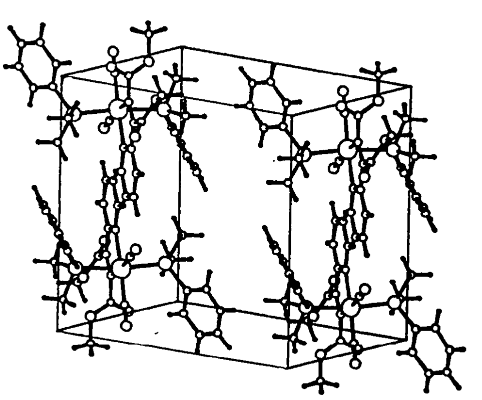

Fig. 2. View of the packing in the unit cell of $[Ru(CO)_2\{C(CO_2Me){=}C(CO_2Me)C_6H_4\}(PMe_2Ph)_2]$ (**b** horizontal, **c** vertical, **a** into the page).

The similarity in length of the Ru—C(17) and Ru—C(26) bonds is reflected in the bonding to the carbonyl ligands [Ru—C(29), 1·918 (5); Ru—C(30), 1·914 (5) Å]. These bond lengths are very close to that [1·91 (2) Å] to the carbonyl ligand *trans* to the alkenyl ligand in [Ru(CO)$_2${C(CO_2Me)═C(CO_2Me)-Cl}Cl(PMe_2Ph)$_2$] (Holland *et al.*, 1983).

The Ru—P bond lengths [Ru—P(1), 2·362 (1); Ru—P(2), 2·364 (1) Å] are in good agreement with the values observed for other ruthenium(II) complexes containing a pair of mutally *trans* PMe_2Ph ligands (Ashworth, Nolte, Singleton & Laing, 1977; Dauter, Mawby, Reynolds & Saunders, 1983).

The packing within the unit cell is shown in Fig. 2. The crystal packing is stabilized largely by van der Waals forces; the closest intermolecular contact involving the non-H atoms is 3·362 (8) Å [O(3)···C(5), $-x$, $-1-y$, $-z$] and between H atoms is 2·21 (4) Å [H(C2*B*)···H(C2*B*), $-x$, $-1-y$, $-1-z$].

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Structure of Bis(glycinato)zinc(II) Monohydrate, a Five-Coordinate Zinc(II) Complex

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Abstract. [Zn($C_2H_4NO_2$)$_2$].H_2O, $M_r = 231·5$, triclinic, $P\bar{1}$, $a = 9·165$ (7), $b = 9·571$ (7), $c = 10·438$ (9) Å, $\alpha = 105·97$ (4), $\beta = 106·19$ (4), $\gamma = 107·12$ (4)°, $V = 774$ (2) Å^3, $Z = 4$, $D_m = 1·97$ (2), $D_x = 1·99$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 32·01$ cm^{-1}, $F(000) = 472$, $T = 294$ K. Final $R = 0·028$ for 2056 observed reflections. There are two formula units in the asymmetric unit. Each of the two crystallographically independent Zn atoms is five-coordinate, is chelated by two glycinate molecules, and forms a fifth bond to a carboxyl O atom in a neighbouring bis(glycinato)-zinc complex. The coordination is approximately square-pyramidal with a significant distortion towards trigonal-bipyramidal geometry. About 10% of the Zn atoms of each type occupy alternative sites in which the chelating ligands and the coordination geometry are retained, but in which the fifth donor atom belongs to a different neighbouring complex. If all the Zn atoms occupied positions half-way between their major and minor sites, the coordination would be octahedral, as originally proposed by Low, Hirshfeld & Richards [*J. Am. Chem. Soc.* (1959), **81**, 4412–4416]. The complexes are linked into polymeric sheets. The water molecules occupy interstitial positions.

Introduction. The structure of the glycinate complex of zinc was originally reported by Low, Hirshfeld & Richards (1959) as part of a study of the coordination of zinc by amino acids. The complex was described as a 'most unpromising' candidate for a complete structure analysis under the conditions at that time. Not only were there eight bis(glycinato)-zinc(II) monohydrate formula units in the triclinic unit cell, but all reflections described as having odd values of $[h + \frac{1}{2}(k - l)]$ were extremely weak. The corresponding cadmium complex, bis(glycinato)-cadmium(II) monohydrate, crystallized in a space group of higher symmetry and was amenable to crystallographic analysis by two-dimensional

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