

[Mo₂Cl₄O₄]²⁻ anion. The only non-bonding interactions involving the hydrogen chloride solvate species are those indicative of hydrogen bonding between symmetry-related HCl molecules [Cl...Cl' = 2.87 (1) Å]. This situation is different from that in other HCl solvates of transition-metal complexes; recent structural studies (Endres & Schlicksupp, 1979, 1980) have revealed that HCl in a crystal lattice is usually hydrogen-bonded to O and/or N atoms of the metal-containing moiety.

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1,1,1,2,2,2,3,3-Octacarbonyl-2,3;3,1-di- μ -hydrido- μ_3 -phenylphosphido-3-triphenylphosphine-triangulo-triruthenium

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Abstract. C₃₂H₂₂O₈P₂Ru₃, [H₂Ru₃(CO)₈(PPh₃)₂(μ_3 -PPh)], *M_r* = 899.68, monoclinic, *P*2₁/*c*, *a* = 12.094 (3), *b* = 15.044 (4), *c* = 18.727 (4) Å, β = 94.00 (3)°, *U* = 3398.9 Å³, *D_c* = 1.758 Mg m⁻³, *Z* = 4, μ (Mo *K* α) = 1.31 mm⁻¹, *F*(000) = 1760. Final *R* and *R_w* are 0.027 and 0.030 respectively for 4964 unique observed reflections [*I* ≥ 3 σ (*I*); 3 ≤ θ ≤ 25°]. The three Ru atoms define an isosceles triangle with the triphenylphosphine group bonded *trans* to the μ_3 -PPh group onto the Ru atom associated with two μ -H bridges, P–Ru–P = 164.8 (1)°.

Introduction. Hydrido clusters involving μ_3 -PR bridging units are of interest because of the presence of reactive H atoms attached to an intact metal triangle and many clusters of this type have been prepared (*e.g.* Natarajan, Scheidsteger & Huttner, 1981; Natarajan, Zsolnai & Huttner, 1981; Huttner, Schneider, Mohr & von Seyerl, 1980; Iwasaki, Mays, Raithby, Taylor & Wheatley, 1981). However, relatively little attention has been given to the effect of substitution on this type of cluster and the influence of such a substitution on the metal–metal bonding and the cluster geometry.

The title compound is obtained as a minor product from the reaction of [HRu₃(CO)₉(μ₃-PPh)]⁻ with [Rh(CO)₃(PPh₃)₂]⁺ (Mays, Raithby, Taylor & Henrick, 1982). Recrystallization from hexane gave yellow crystals. Intensities were recorded on a Philips PW 1100 diffractometer with graphite-monochromated Mo Kα radiation, θ–2θ scans, from a crystal 0.21 × 0.23 × 0.21 mm. Lp corrections, and a semi-empirical absorption correction based on a pseudo-ellipsoid model and 426 azimuthal scan data for eight independent reflections were applied; transmission factors ranged from 0.931 to 0.972 for the full data set.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²)

$$U_{\text{eq}} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U _{eq}
Ru(1)	0.02818 (2)	0.12025 (2)	0.19608 (2)	0.0343 (2)
Ru(2)	0.23074 (2)	0.18598 (2)	0.13355 (2)	0.0355 (2)
Ru(3)	0.10242 (2)	0.29552 (2)	0.22973 (2)	0.0337 (2)
C(11)	0.0632 (3)	0.0698 (2)	0.2915 (2)	0.046 (2)
O(11)	0.0816 (3)	0.0423 (2)	0.3470 (2)	0.069 (2)
C(12)	-0.0056 (4)	0.0183 (3)	0.1370 (2)	0.056 (3)
O(12)	-0.0242 (4)	-0.0417 (2)	0.1030 (2)	0.098 (3)
C(13)	-0.1226 (3)	0.1461 (3)	0.2104 (2)	0.052 (2)
O(13)	-0.2119 (2)	0.1617 (2)	0.2180 (2)	0.081 (2)
C(21)	0.2258 (3)	0.1131 (3)	0.0510 (2)	0.059 (3)
O(21)	0.2199 (3)	0.0689 (3)	0.0025 (2)	0.102 (3)
C(22)	0.2892 (3)	0.2841 (3)	0.0853 (2)	0.060 (3)
O(22)	0.3208 (3)	0.3460 (3)	0.0587 (2)	0.096 (3)
C(31)	0.1462 (3)	0.2725 (3)	0.3304 (2)	0.044 (2)
O(31)	0.1689 (3)	0.2598 (2)	0.3889 (2)	0.068 (2)
C(32)	-0.0400 (3)	0.3394 (3)	0.2466 (2)	0.046 (2)
O(32)	-0.1262 (2)	0.3650 (2)	0.2561 (2)	0.068 (2)
C(33)	0.1654 (3)	0.4103 (3)	0.2140 (2)	0.052 (2)
O(33)	0.2051 (3)	0.4764 (2)	0.2020 (2)	0.084 (2)
P(1)	0.05072 (7)	0.23667 (6)	0.12017 (5)	0.0343 (5)
C(111)	-0.0272 (3)	0.2773 (3)	0.0411 (2)	0.042 (2)
C(112)	-0.0628 (3)	0.2192 (3)	-0.0120 (2)	0.062 (3)
C(113)	-0.1200 (4)	0.2524 (5)	-0.0744 (3)	0.082 (4)
C(114)	-0.1411 (4)	0.3395 (5)	-0.0824 (3)	0.086 (4)
C(115)	-0.1084 (4)	0.3950 (4)	-0.0299 (3)	0.087 (4)
C(116)	-0.0509 (4)	0.3671 (3)	0.0326 (3)	0.063 (3)
P(2)	0.40931 (7)	0.13344 (6)	0.18048 (5)	0.0360 (5)
C(211)	0.4537 (3)	0.0236 (3)	0.1499 (2)	0.044 (2)
C(212)	0.5650 (4)	0.0022 (3)	0.1460 (3)	0.062 (3)
C(213)	0.5954 (4)	-0.0822 (4)	0.1271 (3)	0.074 (3)
C(214)	0.5169 (5)	-0.1463 (3)	0.1124 (3)	0.074 (3)
C(215)	0.4097 (5)	-0.1270 (3)	0.1164 (3)	0.075 (4)
C(216)	0.3761 (4)	-0.0418 (3)	0.1351 (2)	0.056 (3)
C(221)	0.4362 (3)	0.1211 (2)	0.2775 (2)	0.039 (2)
C(222)	0.5431 (3)	0.1128 (3)	0.3092 (2)	0.064 (3)
C(223)	0.5574 (4)	0.0955 (4)	0.3817 (3)	0.076 (3)
C(224)	0.4689 (4)	0.0896 (3)	0.4240 (2)	0.056 (3)
C(225)	0.3644 (3)	0.0978 (3)	0.3925 (2)	0.054 (2)
C(226)	0.3476 (3)	0.1132 (3)	0.3201 (2)	0.048 (2)
C(231)	0.5158 (3)	0.2109 (2)	0.1538 (2)	0.040 (2)
C(232)	0.5633 (3)	0.2025 (3)	0.0891 (2)	0.054 (2)
C(233)	0.6334 (4)	0.2681 (4)	0.0673 (3)	0.071 (3)
C(234)	0.6553 (4)	0.3420 (4)	0.1090 (3)	0.075 (3)
C(235)	0.6081 (4)	0.3513 (3)	0.1727 (3)	0.075 (3)
C(236)	0.5383 (4)	0.2859 (3)	0.1953 (2)	0.057 (3)

Table 2. Selected bond lengths (Å) and angles (°)

Bond lengths within the phenyl rings have been deposited.

Ru(1)—Ru(2)	2.958 (1)	Ru(1)—Ru(3)	2.842 (1)
Ru(1)—C(11)	1.961 (4)	Ru(1)—C(12)	1.919 (4)
Ru(1)—C(13)	1.901 (4)	Ru(1)—P(1)	2.284 (1)
Ru(2)—Ru(3)	2.959 (1)	Ru(2)—C(21)	1.892 (5)
Ru(2)—C(22)	1.894 (5)	Ru(2)—P(1)	2.304 (1)
Ru(2)—P(2)	2.408 (1)	Ru(3)—C(31)	1.953 (4)
Ru(3)—C(32)	1.892 (4)	Ru(3)—C(33)	1.919 (4)
Ru(3)—P(1)	2.282 (1)	C(11)—O(11)	1.126 (5)
C(12)—O(12)	1.118 (6)	C(13)—O(13)	1.124 (5)
C(21)—O(21)	1.125 (6)	C(22)—O(22)	1.133 (6)
C(31)—O(31)	1.127 (5)	C(32)—O(32)	1.135 (5)
C(33)—O(33)	1.132 (5)	P(1)—C(111)	1.806 (4)
P(2)—C(211)	1.841 (4)	P(2)—C(221)	1.833 (4)
P(2)—C(231)	1.832 (4)		
Ru(3)—Ru(1)—Ru(2)	61.3 (1)	C(11)—Ru(1)—Ru(2)	111.0 (1)
C(11)—Ru(1)—Ru(3)	96.4 (1)	C(12)—Ru(1)—Ru(2)	100.8 (1)
C(12)—Ru(1)—Ru(3)	157.0 (1)	C(12)—Ru(1)—C(11)	103.9 (2)
C(13)—Ru(1)—Ru(2)	144.7 (1)	C(13)—Ru(1)—Ru(3)	94.0 (1)
C(13)—Ru(1)—C(11)	95.7 (2)	C(13)—Ru(1)—C(12)	94.4 (2)
P(1)—Ru(1)—Ru(2)	50.1 (1)	P(1)—Ru(1)—Ru(3)	51.5 (1)
P(1)—Ru(1)—C(11)	146.8 (1)	P(1)—Ru(1)—C(12)	106.5 (1)
P(1)—Ru(1)—C(13)	95.0 (1)	Ru(3)—Ru(2)—Ru(1)	57.4 (1)
C(21)—Ru(2)—Ru(1)	98.6 (1)	C(21)—Ru(2)—Ru(3)	146.3 (1)
C(22)—Ru(2)—Ru(1)	142.8 (1)	C(22)—Ru(2)—Ru(3)	94.9 (1)
C(22)—Ru(2)—C(21)	93.0 (2)	P(1)—Ru(2)—Ru(1)	49.6 (1)
P(1)—Ru(2)—Ru(3)	49.5 (1)	P(1)—Ru(2)—C(21)	97.3 (1)
P(1)—Ru(2)—C(22)	94.1 (1)	P(2)—Ru(2)—Ru(1)	119.5 (1)
P(2)—Ru(2)—Ru(3)	117.1 (1)	P(2)—Ru(2)—C(21)	94.8 (1)
P(2)—Ru(2)—C(22)	94.3 (1)	P(2)—Ru(2)—P(1)	164.8 (1)
Ru(2)—Ru(3)—Ru(1)	61.3 (1)	C(31)—Ru(3)—Ru(1)	96.3 (1)
C(31)—Ru(3)—Ru(2)	111.7 (1)	C(32)—Ru(3)—Ru(1)	94.8 (1)
C(32)—Ru(3)—Ru(2)	144.9 (1)	C(32)—Ru(3)—C(31)	95.1 (2)
C(33)—Ru(3)—Ru(1)	157.5 (1)	C(33)—Ru(3)—Ru(2)	100.2 (1)
C(33)—Ru(3)—C(31)	103.0 (2)	C(33)—Ru(3)—C(32)	95.0 (2)
P(1)—Ru(3)—Ru(1)	51.5 (1)	P(1)—Ru(3)—Ru(2)	50.1 (1)
P(1)—Ru(3)—C(31)	146.9 (1)	P(1)—Ru(3)—C(32)	95.2 (1)
P(1)—Ru(3)—C(33)	107.3 (1)	O(11)—C(11)—Ru(1)	178.4 (4)
O(12)—C(12)—Ru(1)	179.1 (4)	O(13)—C(13)—Ru(1)	179.2 (4)
O(21)—C(21)—Ru(2)	178.0 (4)	O(22)—C(22)—Ru(2)	176.1 (4)
O(31)—C(31)—Ru(3)	178.3 (4)	O(32)—C(32)—Ru(3)	179.0 (4)
O(33)—C(33)—Ru(3)	176.7 (4)	Ru(2)—P(1)—Ru(1)	80.3 (1)
Ru(3)—P(1)—Ru(1)	77.0 (1)	Ru(3)—P(1)—Ru(2)	80.4 (1)
C(111)—P(1)—Ru(1)	133.8 (1)	C(111)—P(1)—Ru(2)	129.5 (1)
C(111)—P(1)—Ru(3)	134.5 (1)	C(112)—C(111)—P(1)	119.9 (3)
C(116)—C(111)—P(1)	121.0 (3)		
C(211)—P(2)—Ru(2)	117.1 (1)		
C(221)—P(2)—Ru(2)	119.2 (1)	C(221)—P(2)—C(211)	100.5 (2)
C(231)—P(2)—Ru(2)	108.7 (1)	C(231)—P(2)—C(211)	105.1 (2)
C(231)—P(2)—C(221)	104.8 (2)	C(212)—C(211)—P(2)	121.9 (3)
C(216)—C(211)—P(2)	119.7 (3)		
C(222)—C(221)—P(2)	121.9 (3)		
C(226)—C(221)—P(2)	119.3 (3)		
C(232)—C(231)—P(2)	121.6 (3)		
C(236)—C(231)—P(2)	118.6 (3)		

Equivalents were averaged to give 4964 unique observed intensities. Cell dimensions were derived from the angular measurements of 25 strong reflections (10.0 ≤ θ ≤ 15.0°).

The Ru atoms were located from a Patterson map; positions of all the other non-hydrogen atoms were found from a subsequent difference synthesis. The structure was refined by full-matrix least squares using SHELEX (Sheldrick, 1976), with complex neutral-atom scattering factors (*International Tables for X-ray Crystallography*, 1974) and weights $w = 1/\sigma^2(F)$. The parameters refined included anisotropic thermal param-

eters for the non-hydrogen atoms and a common isotropic temperature factor for the phenyl H atoms. The C—H atoms were estimated geometrically (C—H 1.08 Å, C—C—H 120.0°) and the positions of the hydrido H atoms were located from a final difference map. The refinement converged to $R = 0.027$ and $R_w = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o| = 0.030$. The final atomic coordinates for the non-hydrogen atoms are listed in Table 1 and selected bond lengths and angles in Table 2.*

Discussion. The molecular structure of [H₂Ru₃(CO)₈(PPh₃)(μ₃-PPh)] is shown in Fig. 1. The overall geometry of the structure is a trigonal pyramid with the three Ru atoms lying at the vertices of an isosceles triangle as the base of the pyramid and the μ₃-phenylphosphido ligand as the apex. The triphenylphosphine ligand bonds to Ru(2), *trans* to the μ₃-PPh group, with P(1)—Ru(2)—P(2) = 164.8 (1)°. The eight carbonyl groups are terminal. Two of the Ru—Ru bonds are bridged by H atoms, and these Ru—Ru distances are longer (average 2.959 Å) than the unbridged one [Ru(1)—Ru(3) = 2.842 (1) Å]. The Ru—H distances are in the range 1.79–1.85 Å with angles at the H atoms Ru(1)—H(1)—Ru(2) = 109 and Ru(2)—H(2)—Ru(3) = 110°. The PPh₃ ligand is bonded to Ru(2), the Ru atom associated with the two hydrido ligands.†

* Lists of structure factors, H-atom coordinates, complete lists of bond lengths and angles, and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36828 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† Note that the crystallographic numbering differs from the systematic chemical nomenclature.

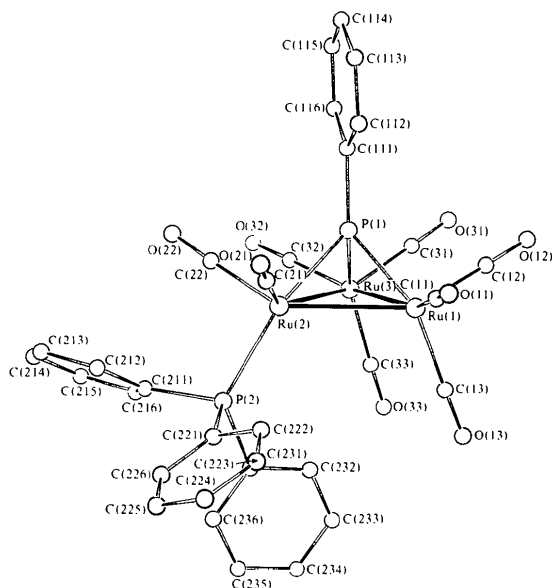


Fig. 1. An ORTEP (Johnson, 1965) drawing of [H₂Ru₃(CO)₈(PPh₃)(μ₃-PPh)].

The short bond Ru(1)—Ru(3) appears to be unaffected by the presence of the phosphine group and its length is not significantly different from the 2.844 (2) Å in [H₂Ru₃(CO)₉{μ₃-P(*p*-C₆H₄OMe)}] (Natarajan, Scheidsteger & Huttner, 1981). However, both of the hydrido-bridged bonds in [H₂Ru₃(CO)₈(PPh₃)(μ₃-PPh)] are significantly longer than those found in [H₂Ru₃(CO)₉{μ₃-P(*p*-C₆H₄OMe)}] [2.937, 2.928 (2) Å].

The triphenylphosphine—Ru(2) bond length [2.408 (1) Å] is longer than the values found for [H₄Ru₄(CO)₁₀(PPh₃)₂] (Wilson, Wu, Love & Bau, 1978) of 2.360, 2.359 (4) Å and for [Ru₃(CO)₁₁(PPh₃)] (Forbes, Goodhand, Jones & Hamor, 1979) of 2.380 (6) Å. In the latter complex the introduction of the phosphine ligand resulted in an expansion of the Ru₃ triangle, particularly the Ru—Ru distance *cis* to the equatorially bound PPh₃ ligand, compared to the parent compound [Ru₃(CO)₁₂] (Mason & Rae, 1968), with Ru—Ru distances *cis* 2.907 (3), *trans* 2.876 (3), diagonal 2.875 (3) Å, compared to 2.837, 2.849, and 2.859 Å in [Ru₃(CO)₁₂]. In the comparison of [H₄Ru₄(CO)₁₂] and [H₄Ru₄(CO)₁₀(PPh₃)₂] (Wilson *et al.*, 1978) the effect of the replacement of the carbonyl ligands by PPh₃ gave only subtle changes in the geometry of the molecule. The PPh₃ ligands are bonded *transoid* to the unbridged Ru—Ru bonds and result in a slight decrease in the Ru—Ru bonds from 2.786 (1) to 2.772 (2) Å with an increase in the Ru—Ru(μ-H) bonds from 2.950 (1) to 2.966 (2) Å.

The μ₃-PPh—Ru bond distances found here [Ru(1), 2.284 (1); Ru(2), 2.304 (1); Ru(3), 2.282 (1) Å] show a similar pattern in lengths to that found for [H₂Ru₃(CO)₉{μ₃-P(*p*-C₆H₄OMe)}] where the corresponding distances of 2.275, 2.320 and 2.273 (4) Å were observed with the longer Ru—P distance in both cases associated with the Ru atom bonded to both hydride ligands.

The average Ru(1),Ru(3)—carbonyl distance is 1.924 Å, longer than the average Ru(2)—carbonyl distance of 1.893 (5) Å. This is the reverse to that in [H₂Ru₃(CO)₉{μ₃-P(*p*-C₆H₄OMe)}] where mean values of 1.927 and 1.968 Å respectively were found. This is the most noticeable effect of substitution by the PPh₃ ligand between these two complexes. The shortening of *M*—CO distances where the metal is also attached to a PPh₃ ligand was also found in [H₂Os₃(CO)₉(PPh₃)] (Benfield, Johnson, Lewis, Raithby, Zuccaro & Henrick, 1979) and this may be caused by the increased electron density on Ru(2), as a result of the σ-donor properties of the PPh₃ ligand, being taken up by back bonding to the carbonyls.

The carbonyls are all approximately linear with a mean Ru—C—O angle of 178.1 (4)°; the average C—O distance is 1.127 (5) Å.

The μ₃-P—C distance [1.806 (4) Å] is shorter than the triphenylphosphine P—C distances of 1.832–

1.841 (4) Å [cf. the mean aryl C—P distance of 1.828 (1) Å for a wide range of structures (Domenicano, Vaciago & Coulson, 1975)], while the C(116)—C(111)—C(112) angle in the phosphido ligand [119.1 (4)°] is only slightly larger than the corresponding angles in the PPh₃ ligand, mean = 118.7° [cf. the mean α angle in the PPh₃ group of 118.5 (1)° (Domenicano *et al.*, 1975)].

The μ₃-P atom lies −1.55 (1) Å from the Ru₃ plane with both the triphenylphosphine P atom and the hydride ligands on the opposite side of this plane at distances of 2.02 (2), 0.86 (7) and 0.82 (7) Å [for P(2), H(1) and H(2) respectively].

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Structure of Bromo(ethyl)[(−)-α-isosparteine]magnesium(II)

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Abstract. MgBr(C₂H₅)(C₁₅H₂₆N₂), C₁₇H₃₁BrMgN₂, *M_r* = 367.7, orthorhombic, *P*2₁2₁2₁, *a* = 10.646 (4), *b* = 14.491 (4), *c* = 11.857 (3) Å, *Z* = 4, *D_x* = 1.34 Mg m^{−3}, *R* = 0.074 for 1235 non-zero reflexions. The Mg atom is tetrahedrally coordinated by the C atom of the ethyl group, the Br atom and the two N atoms of the (−)-α-isosparteine. The distortion from tetrahedral geometry of the Mg atom results from repulsion between (−)-α-isosparteine and the other ligands.

Introduction. During the study of the asymmetric selective polymerization of racemic methacrylates by Grignard reagent-(−)-sparteine complexes, catalyti-

cally active EtMgBr-(−)-sparteine and unreactive EtMgBr-(−)-α-isosparteine were isolated as single crystals (Okamoto, Suzuki & Yuki, 1980; Okamoto, Suzuki, Yuki, Kageyama, Miki, Tanaka & Kasai, 1980). A prismatic colourless crystal of EtMgBr-(−)-α-isosparteine, 0.55 × 0.38 × 0.30 mm, was sealed in a glass capillary tube under nitrogen atmosphere. A Rigaku automated four-circle diffractometer was used with graphite-monochromatized Mo *K*α radiation (*λ* = 0.7107 Å). Systematic absences of *h*00 for *h* = 2*n* + 1, 0*k*0 for *k* = 2*n* + 1, and 00*l* for *l* = 2*n* + 1 indicated the space group to be *P*2₁2₁2₁. Reflexion intensities (2θ ≤ 50.5°) were measured by the θ–2θ scan technique, the 2θ scan rate being 4° min^{−1} and the scan width Δ2θ = (2.4 + 0.7 tan θ)°. Backgrounds were counted for

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