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Acta Cryst. (1993). C**49**, 1923–1926

Isostructural Mixed Ligand Complexes of $[VO]^{2+}$ and $[InCl]^{2+}$ with Hydrotris(3,5-dimethylpyrazolyl)borato and 1,3-Diphenyl-1,3-propanedionato Ligands

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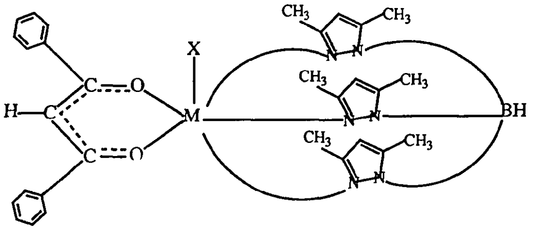
(*Received* 5 *November* 1992; *accepted* 18 *May* 1993)

Abstract

The crystal and molecular structures of (1,3-diphenyl-1,3-propanedionato-κ^2O,O')oxo[tris(3,5-dimethyl-1-pyrazolyl-κN^2)hydroborato]vanadium(IV) (1) and chloro(1,3-diphenyl-1,3-propanedionato-κ^2O,O')[tris(3,5-dimethyl-1-pyrazolyl-κN^2)hydroborato]indium(III) methyl cyanide (2) have been determined. The crystals of both (1) and (2) consist of discrete six-coordinate molecules which have distorted octahedral geometries. As expected, there is a lengthening of the V—N bond *trans* to the terminal oxo group in the vanadium complex with a similar, but less pronounced, lengthening of the In—N bond *trans* to the chloro group in the indium complex.

Comment

We have an ongoing interest in the electron paramagnetic resonance (EPR) spectroscopy of oxovanadium(IV) species. In order to maximize the information available from single-crystal measurements it is necessary to dilute the paramagnetic compound in a diamagnetic isostructural host. We have found that in some cases compounds containing the $[M\mathrm{Cl}]^{2+}$ moiety, where M = Ga or In, act as suitable diluents for compounds containing $[VO]^{2+}$ (Collison, Gahan & Mabbs, 1987; Collison, Mabbs & Temperley, 1991). As part of these studies we now report the crystal structures of [LVO(dbzac)] (1) and [LInCl(dbzac)].CH_3CN (2), where L = tris(3,5-dimethylpyrazolyl)hydroborato and dbzac = 1,3-diphenyl-1,3-propanedionato. The structures of these two complexes are sufficiently similar for [LInCl(dbzac)] to act as a suitable diamagnetic host for [LVO(dbzac)]. The single-crystal EPR spectroscopy of [LInCl(VO)(dbzac)] will be reported elsewhere.



(1) M = V, X = O
(2) M = In, X = Cl

The molecular geometry in each compound is similar (Figs. 1 and 2) and consists of a distorted octahedron. As expected there is a significant lengthening of the V—N bond *trans* to the terminal oxo group [V(1)—N(11) = 2.326 (5) Å compared with V(1)—N(21) = 2.067 (10) and V(1)—N(31) = 2.121 (10) Å]. There is a similar, but less pronounced, lengthening of the In—N bond *trans* to Cl in [LInCl(dbzac)] [In(1)—N(11) = 2.304 (4) Å compared with In(1)—N(21) = 2.213 (4) and In(1)—N(31) = 2.223 (3) Å]. The atoms within the six-membered rings comprising the metal and the O(41)—C(41)—C(42)—C(43)—O(42) part of the dbzac ligand are not coplanar. There is a significant tilting about the O(41)—O(42) direction towards the terminal monodentate ligand. The dihedral angle

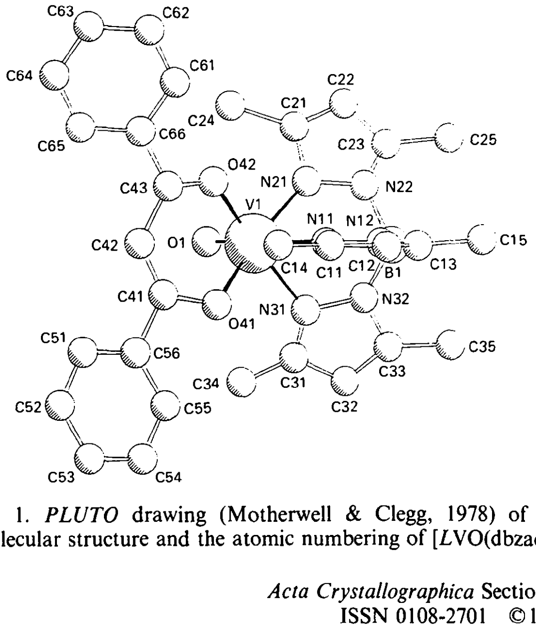


Fig. 1. *PLUTO* drawing (Motherwell & Clegg, 1978) of the molecular structure and the atomic numbering of [LVO(dbzac)].

between the best plane formed by V, O(41) and O(42) and the best plane through O(41), C(41), C(42), C(43), O(42), C(56) and C(66) is 23.2°; the dihedral angle between the best plane formed by In, O(41) and O(42) and that through O(41), C(41), C(42), C(43) and O(42) is 19.2°. These tilts are similar to that found in [*L*VO(acac)], where acac = 2,4-pentanedionato (Beddoes, Collison, Mabbs & Passand, 1990), and as suggested previously they can be attributed to an interaction with the methyl group [C(14)] on the pyrazolyl ring which is *trans* to the terminal oxo group or the chloro group. Apart from some large standard deviations in the bond lengths and angles in the vanadium complex, no unusual features were observed in the dimensions of the ligand *L*.

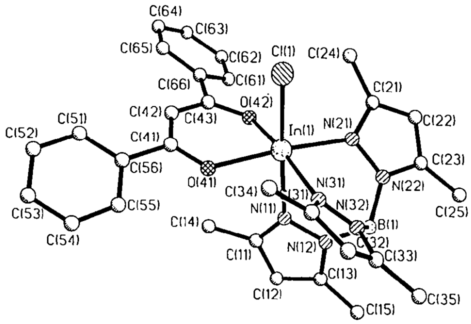


Fig. 2. Molecular structure and atomic numbering of [*L*InCl(dbzac)].

Experimental

Compound (1)

Crystal data

$[VO(C_{15}H_{22}BN_6)(C_{15}H_{11}O_2)]$
$M_r = 587.5$
Orthorhombic
$Pna2_1$
$a = 15.960\ (1)$ Å
$b = 12.447\ (1)$ Å
$c = 17.044\ (1)$ Å
$V = 3385.8\ (4)$ Å^3
$Z = 4$
$D_x = 1.15$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 100 reflections
$\theta = 11.1\text{–}11.9°$
$\mu = 0.34$ mm^{-1}
$T = 293$ K
Prismatic
0.45 × 0.32 × 0.26 mm
Pale green

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3832 measured reflections
3832 independent reflections
2467 observed reflections [$F > 3\sigma(F)$]
$\theta_{\max} = 25°$
$h = 0 \rightarrow 22$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 24$
1 standard reflection monitored every 150 reflections
intensity variation: ±3%

Refinement

Refinement on F^2
Final $R = 0.074$
$wR = 0.084$
$S = 1.07$
2440 reflections
286 parameters
H-atom parameters not refined
$w = (3.03 - 0.058F + 0.0043F^2)^{-1/2}$
$(\Delta/\sigma)_{\max} = 0.3$
$\Delta\rho_{\max} = 0.6$ e Å^{-3}
$\Delta\rho_{\min} = -0.5$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (2)

Crystal data

$[InCl(C_{15}H_{22}BN_6)(C_{15}H_{11}O_2)].CH_3CN$
$M_r = 711.8$
Monoclinic
$P2_1$
$a = 8.169\ (2)$ Å
$b = 19.599\ (7)$ Å
$c = 10.737\ (3)$ Å
$\beta = 99.18\ (2)°$
$V = 1696.9\ (9)$ Å^3
$Z = 2$
$D_x = 1.39$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 39 reflections
$\theta = 2.3\text{–}17.8°$
$\mu = 0.80$ mm^{-1}
$T = 293$ K
Prismatic
0.50 × 0.45 × 0.30 mm
Colourless

Data collection

Nicolet $R3m/V$ diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.37$, $T_{\max} = 0.42$
6489 measured reflections
6022 independent reflections
5486 observed reflections [$F > 4\sigma(F)$]
$R_{\rm int} = 0.015$
$\theta_{\max} = 25°$
$h = -9 \rightarrow 9$
$k = -23 \rightarrow 23$
$l = 0 \rightarrow 12$
3 standard reflections monitored every 100 reflections
intensity variation: −4.5%

Refinement

Refinement on F^2
Final $R = 0.034$
$wR = 0.043$
$S = 1.01$
5486 reflections
399 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.001F^2]$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.8$ e Å^{-3}
$\Delta\rho_{\min} = -0.8$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	$U_{\rm eq}$
Compound (1)				
V(1)	0.08982 (7)	0.14938 (9)	0.0000	0.033 (1)
O(1)	0.0159 (3)	0.0631 (4)	0.0028 (8)	0.053 (3)
N(11)	0.1890 (3)	0.2863 (4)	0.0003 (8)	0.035 (3)
N(12)	0.1601 (4)	0.3904 (4)	−0.0001 (9)	0.038 (3)
N(21)	0.0324 (7)	0.2492 (8)	0.0806 (6)	0.045 (6)
N(22)	0.0271 (7)	0.3591 (10)	0.0751 (7)	0.049 (6)
N(31)	0.0368 (6)	0.2480 (8)	−0.0885 (5)	0.033 (5)
N(32)	0.0294 (7)	0.3575 (6)	−0.0724 (6)	0.033 (5)

B(1)	0.0656 (5)	0.4105 (6)	−0.0013 (13)	0.039 (4)
C(11)	0.2725 (4)	0.2930 (6)	−0.0041 (8)	0.042 (4)
C(12)	0.2975 (5)	0.4016 (7)	−0.0042 (10)	0.051 (4)
C(13)	0.2252 (5)	0.4595 (6)	−0.0041 (9)	0.049 (4)
C(14)	0.3281 (5)	0.1966 (8)	−0.0021 (12)	0.068 (7)
C(15)	0.2144 (7)	0.5796 (8)	0.0035 (14)	0.078 (7)
C(21)	−0.0075 (10)	0.2280 (14)	0.1539 (10)	0.062 (10)
C(22)	−0.0371 (12)	0.3208 (13)	0.1842 (8)	0.061 (10)
C(23)	−0.0145 (10)	0.4007 (11)	0.1301 (6)	0.051 (8)
C(24)	−0.0119 (13)	0.1098 (10)	0.1820 (9)	0.081 (10)
C(25)	−0.0309 (12)	0.5206 (14)	0.1370 (11)	0.072 (12)
C(31)	−0.0034 (8)	0.2255 (11)	−0.1512 (6)	0.040 (7)
C(32)	−0.0387 (10)	0.3215 (15)	−0.1830 (8)	0.067 (9)
C(33)	−0.0170 (7)	0.4038 (12)	−0.1375 (8)	0.047 (8)
C(34)	−0.0113 (9)	0.1242 (14)	−0.1836 (11)	0.083 (12)
C(35)	−0.0381 (12)	0.5178 (17)	−0.1364 (12)	0.079 (12)
O(41)	0.1625 (6)	0.0819 (7)	−0.0807 (6)	0.050 (6)
C(41)	0.2001 (9)	−0.0166 (9)	−0.0681 (8)	0.054 (8)
C(42)	0.2149 (5)	−0.0599 (6)	−0.0010 (9)	0.051 (4)
C(43)	0.1976 (7)	−0.0066 (8)	0.0744 (5)	0.030 (5)
O(42)	0.1617 (6)	0.0761 (6)	0.0805 (4)	0.036 (5)
N(3)	0.099 (2)	0.240 (3)	0.489 (3)	0.141 (11)
C(1)	0.0445 (17)	0.211 (2)	0.4687 (14)	0.067 (6)
C(2)	−0.013 (2)	0.188 (3)	0.419 (2)	0.098 (9)
C(51)	0.2736	−0.1650	−0.1438	0.052 (2)
C(52)	0.3031	−0.2081	−0.2142	0.069 (3)
C(53)	0.2885	−0.1543	−0.2847	0.066 (4)
C(54)	0.2444	−0.0575	−0.2848	0.078 (4)
C(55)	0.2150	−0.0144	−0.2145	0.060 (3)
C(56)	0.2296	−0.0682	−0.1440	0.049 (3)
C(61)	0.2425	0.0080	0.2133	0.057 (3)
C(62)	0.2701	−0.0375	0.2835	0.088 (4)
C(63)	0.2836	−0.1481	0.2883	0.123 (9)
C(64)	0.2694	−0.2131	0.2230	0.095 (4)
C(65)	0.2418	−0.1676	0.1528	0.071 (3)
C(66)	0.2284	−0.0571	0.1480	0.039 (3)
Compound (2)				
In(1)	0.38236 (3)	0.5000	0.41553 (2)	0.044 (1)
Cl(1)	0.5373 (2)	0.6047 (1)	0.4711 (2)	0.080 (1)
N(11)	0.2336 (4)	0.4002 (3)	0.3780 (3)	0.049 (1)
N(12)	0.2463 (4)	0.3539 (2)	0.4759 (3)	0.048 (1)
N(21)	0.5779 (4)	0.4305 (2)	0.5087 (3)	0.044 (1)
N(22)	0.5335 (5)	0.3793 (2)	0.5852 (3)	0.046 (1)
N(31)	0.2741 (4)	0.4954 (3)	0.5927 (3)	0.047 (1)
N(32)	0.2837 (5)	0.4346 (2)	0.6562 (3)	0.045 (1)
B(1)	0.3511 (6)	0.3698 (3)	0.6030 (5)	0.046 (2)
C(11)	0.1279 (6)	0.3729 (3)	0.2835 (4)	0.060 (2)
C(12)	0.0717 (7)	0.3113 (3)	0.3176 (6)	0.074 (2)
C(13)	0.1497 (6)	0.2995 (3)	0.4396 (5)	0.062 (2)
C(14)	0.0783 (9)	0.4100 (5)	0.1630 (5)	0.088 (3)
C(15)	0.1324 (9)	0.2398 (3)	0.5236 (8)	0.091 (3)
C(21)	0.7397 (5)	0.4241 (3)	0.5070 (5)	0.055 (2)
C(22)	0.8005 (6)	0.3688 (3)	0.5821 (5)	0.067 (2)
C(23)	0.6679 (6)	0.3417 (3)	0.6298 (5)	0.062 (2)
C(24)	0.8285 (6)	0.4712 (3)	0.4340 (5)	0.072 (2)
C(25)	0.6626 (9)	0.2811 (3)	0.7126 (7)	0.091 (3)
C(31)	0.1934 (7)	0.5398 (3)	0.6546 (5)	0.058 (2)
C(32)	0.1502 (6)	0.5077 (4)	0.7586 (5)	0.065 (2)
C(33)	0.2064 (6)	0.4422 (3)	0.7580 (4)	0.057 (2)
C(34)	0.1590 (10)	0.6097 (3)	0.6116 (7)	0.093 (3)
C(35)	0.1936 (9)	0.3864 (4)	0.8484 (5)	0.088 (3)
O(41)	0.1804 (4)	0.5540 (2)	0.3094 (3)	0.059 (1)
C(41)	0.1782 (7)	0.5841 (3)	0.2016 (5)	0.060 (2)
C(42)	0.2945 (7)	0.5722 (3)	0.1221 (5)	0.065 (2)
C(43)	0.4238 (6)	0.5255 (3)	0.1380 (4)	0.055 (2)
O(42)	0.4650 (4)	0.4876 (2)	0.2362 (3)	0.061 (1)
C(51)	0.0229 (8)	0.6734 (3)	0.0612 (5)	0.072 (2)
C(52)	−0.1170 (10)	0.7112 (3)	0.0230 (6)	0.087 (3)
C(53)	−0.2546 (9)	0.7034 (3)	0.0812 (6)	0.079 (2)
C(54)	−0.2480 (7)	0.6592 (3)	0.1799 (6)	0.070 (2)
C(55)	−0.1071 (6)	0.6220 (3)	0.2176 (5)	0.061 (2)
C(56)	0.0300 (6)	0.6267 (3)	0.1575 (5)	0.054 (2)
C(61)	0.6072 (8)	0.4539 (4)	0.0313 (6)	0.081 (3)
C(62)	0.6964 (8)	0.4401 (5)	−0.0661 (6)	0.099 (3)
C(63)	0.6956 (8)	0.4857 (5)	−0.1619 (6)	0.090 (3)
C(64)	0.6140 (9)	0.5451 (5)	−0.1602 (6)	0.092 (3)
C(65)	0.5230 (8)	0.5602 (4)	−0.0639 (5)	0.076 (2)
C(66)	0.5218 (6)	0.5140 (3)	0.0341 (4)	0.057 (2)
C(1)	−0.3925 (9)	0.2627 (4)	0.1942 (8)	0.102 (3)
N(1)	−0.3281 (12)	0.2505 (6)	0.1153 (9)	0.152 (5)
C(2)	−0.4684 (13)	0.2799 (6)	0.2992 (10)	0.154 (6)

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

Compound (1)			
V(1)—O(1)	1.596 (5)	C(41)—C(42)	1.287 (18)
V(1)—O(41)	1.986 (10)	C(42)—C(43)	1.472 (17)
V(1)—O(42)	2.007 (8)	N(11)—C(11)	1.336 (9)
V(1)—N(11)	2.326 (5)	N(11)—N(12)	1.376 (7)
V(1)—N(21)	2.067 (10)	N(21)—C(21)	1.43 (2)
V(1)—N(31)	2.121 (10)	N(21)—N(22)	1.373 (14)
O(42)—C(43)	1.183 (12)	N(31)—C(31)	1.279 (14)
O(41)—C(41)	1.382 (14)	N(31)—N(32)	1.395 (12)
O(1)—V(1)—N(11)	174.9 (2)	O(41)—V(1)—O(42)	87.0 (3)
O(1)—V(1)—N(21)	93.3 (4)	O(41)—V(1)—N(31)	89.1 (4)
O(1)—V(1)—N(31)	96.7 (4)	O(42)—V(1)—N(21)	94.1 (5)
O(1)—V(1)—O(41)	99.7 (4)	N(21)—V(1)—N(31)	87.0 (2)
O(1)—V(1)—O(42)	95.5 (4)		
Compound (2)			
In(1)—Cl(1)	2.434 (2)	C(41)—C(42)	1.394 (8)
In(1)—O(41)	2.131 (3)	C(42)—C(43)	1.388 (8)
In(1)—O(42)	2.154 (3)	N(11)—C(11)	1.335 (6)
In(1)—N(11)	2.304 (4)	N(11)—N(12)	1.380 (5)
In(1)—N(21)	2.213 (4)	N(21)—C(21)	1.331 (6)
In(1)—N(31)	2.223 (3)	N(21)—N(22)	1.381 (5)
O(42)—C(43)	1.289 (6)	N(31)—C(31)	1.332 (8)
O(41)—C(41)	1.296 (6)	N(31)—N(32)	1.369 (7)
Cl(1)—In(1)—N(11)	175.9 (1)	O(41)—In(1)—O(42)	84.5 (1)
Cl(1)—In(1)—N(21)	95.7 (1)	O(41)—In(1)—N(31)	95.6 (1)
Cl(1)—In(1)—N(31)	95.0 (2)	O(42)—In(1)—N(21)	91.6 (1)
Cl(1)—In(1)—O(41)	92.3 (1)	N(21)—In(1)—N(31)	86.8 (2)
Cl(1)—In(1)—O(42)	95.1 (1)		

Compound (1) was prepared as reported previously (Beddoes, Collison, Mabbs & Passand, 1990) and crystals suitable for X-ray analysis were grown from methyl cyanide solution. Because there were some suspect bond lengths in the regions of the phenyl groups, data were collected on two crystals, each from separate preparations. The data were the same within experimental accuracy. The solution and refinement of the structure was by Patterson and Fourier techniques, using *SHELX*76 (Sheldrick, 1976). H atoms were placed in calculated positions and during the refinement the phenyl rings were treated as rigid groups. All atoms except the H atoms and the phenyl C atoms were refined with anisotropic thermal parameters. There is significant departure from idealized mirror symmetry both by the phenyl rings, and by the appearance of a solvent molecule (in the difference Fourier maps) with occupancy of *ca* 0.5. Intensity statistics also favour the non-centrosymmetric space group $Pna2_1$ rather than *Pnma* which would require the molecule to be on the mirror plane. The final difference Fourier map revealed small 'ghost' peaks near the phenyl rings, indicating a small amount of disorder.

Compound (2) was prepared by stirring a stoichiometric mixture of $InCl_3$ (Fluka), K*L* (Trofimenko, 1967), dbzacH and triethylamine in dried CH_3CN for 24 h. The solution was evaporated to dryness and then extracted with dry CH_2Cl_2. The resulting solid was recrystallized from dried CH_3CN. Solution and refinement of the structure were accomplished by Patterson and full-matrix least-squares techniques using a Nicolet *SHELXTL-Plus* (Sheldrick, 1987) MicroVAX II system. All non-H-atom parameters were refined anisotropically, while a riding model with calculated positions and fixed isotropic *U* was used for H atoms. The absolute structure was determined by the method of Rogers (1981); η = 1.07 (5).

Atomic scattering factors from Cromer & Waber (1974) and anomalous-dispersion factors from Cromer (1974) were used for both structure solutions.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry for compounds (1) and (2) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71341 (55 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1033]

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Acta Cryst. (1993). C**49**, 1926–1929

Structure of the Heteronuclear Cluster $[Au_2Ru_4(\mu_3\text{-}H)(\mu\text{-}H)(\mu\text{-}Ph_2PCH{=}CHPPh_2)(CO)_{12}]$

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(*Received* 4 *February* 1993; *accepted* 19 *May* 1993)

Abstract

The X-ray structure analysis shows that the digold tetraruthenium cluster [μ-bis(diphenylphosphino)ethene-5κP:6$\kappa P'$]-dodecacarbonyl-1κ^3C,2κ^2C,3κ^3C,4κ^3C-μ_3-hydrido-1:2:3κ^3H-μ-hydrido-1:4κ^2H-digoldtetraruthenium (6 *Ru—Ru*)(4 *Ru—Au*)(*Au—Au*), $[Au_2Ru_4(\mu_3\text{-}H)(\mu\text{-}H)(\mu\text{-}Ph_2PCH{=}CHPPh_2)(CO)_{12}]$ (1), adopts a capped square-based pyramid geometry. The metal core consists of a square-based pyramid defined by two Au atoms and two Ru atoms in the basal plane with an Ru atom at the apex. The Ru_3 face of this Au_2Ru_3 unit is capped by another Ru atom [Au—Au 2.861 (2), Au—Ru 2.707 (2)–2.827(2), Ru—Ru 2.796 (3)–3.017 (3) Å]. The bis(diphenylphosphino)ethene ligand, *cis*-$Ph_2PCH{=}CHPPh_2$, bridges the two Au atoms. One of the two hydrido ligands caps an Ru_3 face and the other bridges an Ru—Ru edge of an adjacent Ru_3 face of the metal polyhedron. Each Ru atom is bonded to three terminal carbonyl groups.

Comment

A particular class of cluster compounds which are attracting a great deal of current investigation are those in which two or more MPR_3 (M = Cu, Ag, Au; R = alkyl or aryl) units form part of the overall metal polyhedron (Freeman, Orpen & Salter, 1987; Brown, Salter & Toupet, 1988; Adatia, McPartlin & Salter, 1988; Bates *et al.*, 1989; Blaxill, Brown, Frankland, Salter & Sǐk, 1989). From the numerous studies that have been reported, the most interesting feature of these heteronuclear clusters is the degree of flexibility their metal skeletons exhibit in solution when the nature of the attached phosphine ligand is varied. Variable temperature ^{1}H and ^{31}P-{^{1}H} NMR studies show that the metal frameworks of these compounds are stereochemically non-rigid, fluctuating between capped trigonal bipyramidal and capped square-based pyramidal geometries (Freeman *et al.*, 1987; Bates *et al.*, 1989). X-ray studies confirm that the type of metal-core geometry adopted by these clusters in the solid state is critically governed by the nature of the attached phosphine ligand (Freeman *et al.*, 1987; Bates *et al.*, 1989).

The X-ray structure analysis confirms that the heteronuclear cluster $[Au_2Ru_4(\mu_3\text{-}H)(\mu\text{-}H)(\mu\text{-}Ph_2PCH{=}CHPPh_2)(CO)_{12}]$ (1) adopts a capped square-based pyramidal geometry (Fig. 1), as previously proposed from IR and NMR spectroscopic studies (Blaxill *et al.*, 1989).

The metal-core geometry established for (1) resembles the metal frameworks previously characterized for the monoclinic and orthorhombic crystalline forms of the cluster $[Au_2Ru_4(\mu_3\text{-}H)(\mu\text{-}H)(\mu\text{-}Ph_2PCH_2PPh_2)(CO)_{12}]$ (2) and for the compound $[Au_2Ru_4(\mu_3\text{-}H)(\mu\text{-}H)(\mu\text{-}Ph_2PCH_2CH_2PPh_2)(CO)_{12}]$ (3) (Bates *et al.*, 1989). For a comparative study, the bond lengths associated with the capped square-based pyramidal metal frameworks in (1), (2) and (3) are displayed in Fig. 2. The range of $M—M$ distances around the metal framework in (1) are similar to those observed in clusters (2) and (3) (Fig. 2).

Acta Crystallographica Section C
ISSN 0108-2701 ©1993