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A Second Example of Rupture and Realignment of the Phosphine Bridge Orientation in Polynuclear Complexes. The Formation and Structure of Bis(μ-2,6-bis(diphenylphosphino)pyridine)(μ-iodo)(μ-carbonyl)dirhodium Tetraphenylborate

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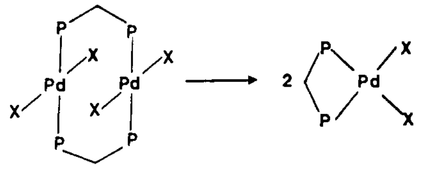
Treatment of yellow $Rh_2\{\mu\text{-}(Ph_2P)_2py\}_2(CO)_2Cl_2$ ($(Ph_2P)_2py$ is 2,6-bis(diphenylphosphino)pyridine), which has the phosphine bridges arranged to give two *trans*-$Rh(CO)ClP_2$ units and uncoordinated pyridine nitrogens, with sodium iodide and sodium tetraphenylborate produces orange-brown $[Rh_2\{\mu\text{-}(Ph_2P)_2py\}_2(\mu\text{-}CO)(\mu\text{-}I)][BPh_4]$. In the latter complex the rhodium centers are bridged by $(Ph_2P)_2py$ ligands that are arranged so that one diphenylphosphino group is trans to a pyridine nitrogen while the other is part of a four-membered chelate ring. $[Rh_2(C_{29}H_{23}P_2N)_2(CO)I][BC_{24}H_{20}]\cdot 2CH_2Cl_2$ crystallizes in the triclinic space group $P\bar{1}$ (No. 2) with $a = 14.646$ (5) Å, $b = 15.210$ (5) Å, $c = 19.705$ (6) Å, $\alpha = 85.63$ (2)°, $\beta = 68.92$ (2)°, $\gamma = 72.12$ (2)°, $Z = 2$, and $V = 3895$ (2) Å^3 at 140 K. Refinement yielded $R = 0.079$ and $R_w = 0.088$ for 7593 reflections and 450 parameters.

Introduction

There has been considerable recent interest in the chemical reactivity and structural characterization of bi- and polynuclear complexes that are held together through various phosphine-containing bridges.[1,2] Many such dinuclear complexes, particularly those with the *trans*-$M_2(dpm)_2$ core (dpm is bis(diphenylphosphino)methane), undergo chemical transformations in which the core remains intact while the other ligands and/or the metal–metal interactions undergo major changes. However, a few observations of alteration of the arrangement of the phosphine bridges have been discovered. Some binuclear complexes have been found to undergo dissociation into corresponding monomeric units. An example is shown in eq 1[3] (the phosphine is bis(diphenylphosphino)methane; X is Cl, Br, or I), and very similar results regarding dissociation have been obtained on related platinum complexes.[4] Fortunately for the development of novel binuclear chemistry, such dissociation is relatively rare. Another alteration of bridges involves the head-to-head (HH)/head-to-tail (HT) isomerization found for 2-(diphenylphosphino)pyridine (Ph_2Ppy) complexes. One of several known examples is shown in equation 2.[5,6] The reversible rupture and realignment occurring in eq 3 offers a more dramatic example of the extent to which the bridging arrangement can be altered.[7] In this reaction not only are two Rh(I) units reversibly lost from the tetranuclear cluster, but the phosphine ligands undergo major reorientation. In this article we report a related reaction in which a $\{(Ph_2P)_2py\}_2Rh_2$ core undergoes rupture and realignment under mild conditions.



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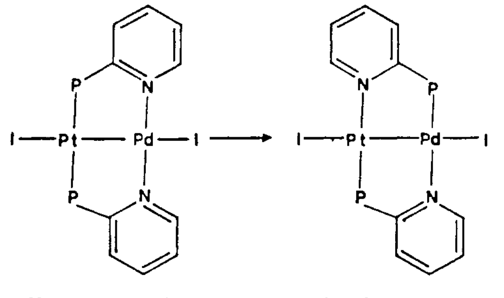

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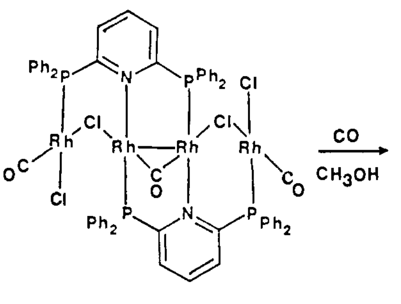

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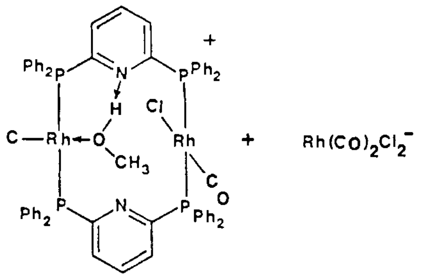

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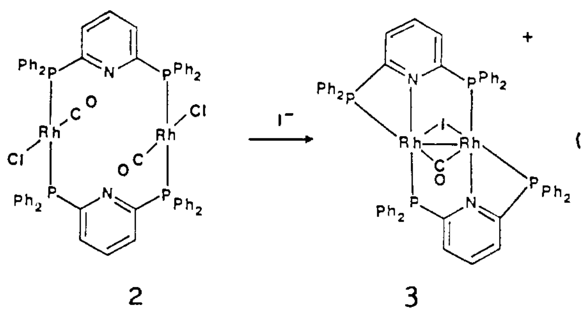
Results

Preparation. As shown in eq 4, treatment of dichloromethane solutions of yellow $Rh_2\{\mu\text{-}(Ph_2P)_2py\}_2(CO)_2Cl_2$ (**2**)[8] successively



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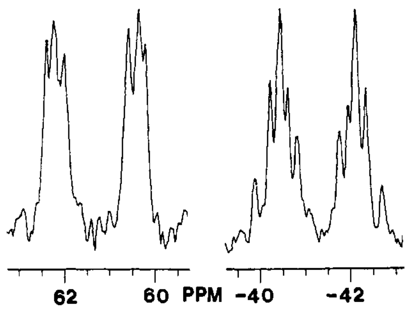

Figure 1. 81-MHz ^{31}P NMR spectrum of [Rh$_2${μ-(Ph$_2$P)$_2$py}$_2$(μ-CO)(μ-I)[BPh$_4$] in dichloromethane at 25 °C.

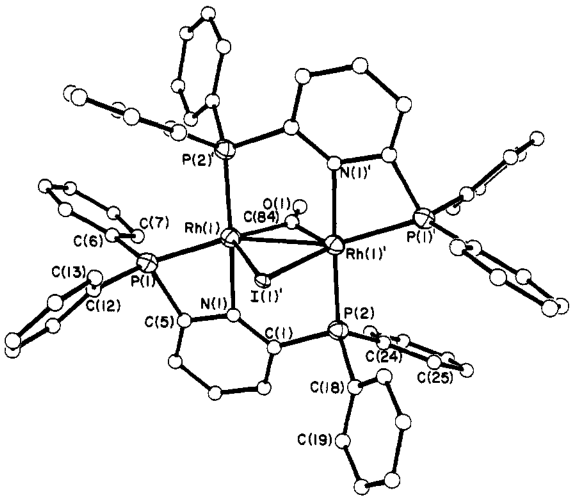

Figure 2. Perspective drawing of cation 1 of [Rh$_2${μ-(Ph$_2$P)$_2$py}$_2$(μ-CO)(μ-I)][BPh$_4$].

with sodium iodide and sodium tetraphenylborate converts the complex into orange-brown [Rh$_2${μ-(Ph$_2$P)$_2$py}$_2$(μ-CO)(μ-I)][BPh$_4$] (**3**), which has been isolated in 60% yield. The reaction occurs rapidly at room temperature. The infrared spectrum of **3** as a mineral oil mull shows an intense absorption at 1825 cm^{-1}, which is ascribed to the presence of bridging carbon monoxide ligand. No evidence for terminal carbon monoxide absorption was found. The ^{31}P NMR spectrum is shown in Figure 1. Two multiplets ($\delta_1 = 61.3$; $\delta_2 = -41.3$ ppm) are present, each with major splitting due to one-bond coupling to rhodium (^1J(Rh,P) = 153 Hz; ^1J(Rh,P) = 135 Hz). The low-frequency multiplet is assigned to the phosphorus atom in the four-membered chelating ring portion of the cation, since related, chelated Ph$_2$Ppy complexes show resonances in a similar region.[5,9] The lack of any P,P coupling greater than 25 Hz is consistent with the relative orientations of the phosphine ligands.

X-ray Crystal Structure of [Rh$_2${μ-(Ph$_2$P)$_2$py}$_2$(μ-CO)(μ-I)][BPh$_4$]·2CH$_2$Cl$_2$. The complex crystallized with two half-cations, one tetraphenylborate, and two dichloromethane molecules in the asymmetric unit. There are no unusual contacts between these constituents. Final atomic positional and thermal parameters are given in Table I. Table II contains selected interatomic distances, and Table III lists selected interatomic angles within the cations.

Perspective views that give the numbering scheme of the two independent cations are shown in Figures 2 and 3. These complexes have a double A-frame structure. Comparison of the two figures and of the structural parameters in Tables II and III reveals that the two cations have very similar structures. Each cation is located at a center of symmetry in the crystal. There is, consequently, disorder in the positions of the bridging carbon monoxide and iodide ligands. The drawings in Figures 2 and 3 show

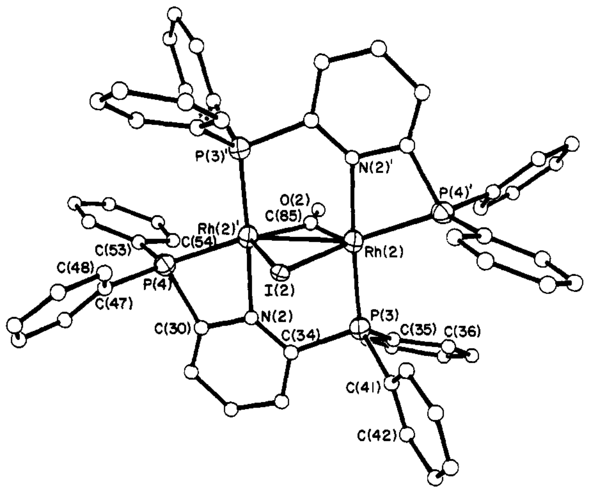

Figure 3. Perspective drawing of cation 2 of [Rh$_2${μ-(Ph$_2$P)$_2$py}$_2$(μ-CO)(μ-I)][BPh$_4$].

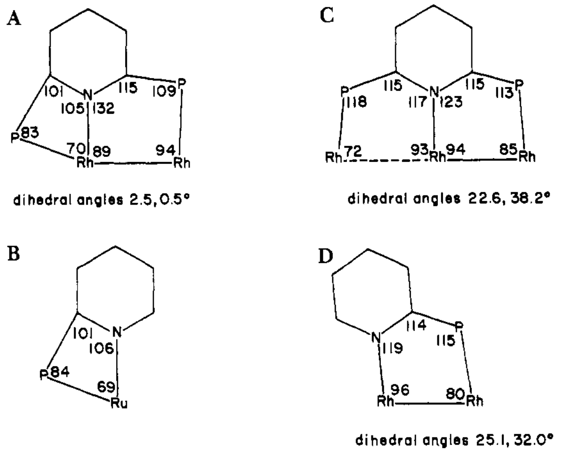

Figure 4. Comparison of the internal angles in bridging and chelating phosphinopyridine units in (A) [Rh$_2${μ-(Ph$_2$P)$_2$py}$_2$(μ-CO)(μ-I)]$^+$ (**3**), (B) Ru(Ph$_2$Ppy)(CO)$_2$Cl$_2$ (**5**), (C) Rh$_4${μ-(Ph$_2$P)$_2$py}$_2$(μ-CO)(CO)$_2$Cl$_4$ (**1**), and (D) Rh$_2${μ-(Ph$_2$P)$_2$py}$_2$(μ-CO)Cl$_2$ (**6**).

only one of the two possible orientations of the carbon monoxide and iodide ligands. The positions of the iodide and the oxygen of the carbon monoxide coincide, with each having 0.5 occupancy. Such disorder involving interchange of carbon monoxide and halide ligands has been encountered in other related complexes.[10,11] In the present case the Rh$_2$(μ-CO)(μ-I) unit resembles a related fragment found in Rh$_3$(μ-dpmp)$_2$(μ-CO)(μ-I)$_2$(CO)$_2$$^+$ (**4**).[12] The Rh- - -Rh separations of 2.568 (2) and 2.577 (2) Å in **3** and 2.760 (2) Å in **4** are similar, as are the Rh–I distances (2.866 (2), 2.924 (2) Å in **3**; 2.801 (3), 2.865 (3) Å in **4**) and the Rh–I–Rh angles (53.3 (2), 53.0 (2)° in **3**; 58.3 (3)° in **4**). The somewhat larger Rh–Rh distance and Rh–I–Rh angle can be attributed to the greater flexibility of the methylene-bridged triphosphine, which does not constrain the metal–metal separation as greatly as does

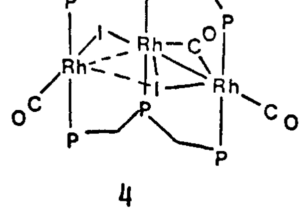

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Table I. Atom Coordinates (×10^4) and Thermal Parameters (Å^2 × 10^3) for [Rh$_2${μ-(Ph$_2$P)py}$_2$(μ-CO)(μ-I)][BPh$_4$]·2CH$_2$Cl$_2$

atom	*x*	*y*	*z*	*U*
Rh(1)	4896 (1)	4698 (1)	5641 (1)	26 (1)[a]
Rh(2)	4683 (1)	597 (1)	528 (1)	30 (1)[a]
I(1)	4777 (1)	3531 (1)	4626 (1)	13 (1)[a]
I(2)	3314 (1)	640 (1)	−254 (1)	19 (1)[a]
O(1)	4760	3440	4604	30
O(2)	3185	730	−233	30
Cl(1)	2226 (5)	4812 (4)	4058 (4)	105 (3)[a]
Cl(2)	1023 (10)	3592 (8)	4503 (4)	237 (9)[a]
P(1)	5339 (2)	3958 (2)	6586 (2)	27 (1)[a]
P(2)	6809 (2)	4670 (2)	3942 (2)	26 (1)[a]
P(3)	5355 (3)	1644 (2)	−175 (2)	31 (1)[a]
P(4)	6180 (3)	−1253 (2)	−1687 (2)	31 (1)[a]
N(1)	6467 (7)	4030 (6)	5292 (5)	26 (2)
N(2)	5973 (7)	332 (7)	−1236 (5)	29 (2)
C(1)	7209 (9)	3911 (8)	4619 (6)	27 (3)
C(2)	8146 (9)	3222 (8)	4521 (6)	29 (3)
C(3)	8292 (9)	2711 (8)	5106 (6)	32 (3)
C(4)	7545 (9)	2848 (8)	5776 (6)	30 (3)
C(5)	6616 (9)	3499 (8)	5858 (7)	32 (3)
C(6)	5007 (8)	2955 (7)	7061 (6)	23 (3)
C(7)	5281 (10)	2131 (8)	6646 (7)	35 (3)
C(8)	4943 (10)	1389 (10)	7000 (7)	46 (4)
C(9)	4321 (10)	1479 (10)	7718 (7)	44 (3)
C(10)	4063 (10)	2285 (9)	8105 (8)	42 (3)
C(11)	4391 (10)	3023 (9)	7781 (7)	38 (3)
C(12)	5521 (9)	4613 (8)	7252 (6)	28 (3)
C(13)	5061 (9)	5561 (8)	7335 (7)	34 (3)
C(14)	5251 (10)	6074 (10)	7778 (7)	42 (3)
C(15)	5902 (10)	5644 (9)	8143 (7)	41 (3)
C(16)	6341 (9)	4706 (8)	8082 (7)	34 (3)
C(17)	6167 (9)	4202 (8)	7633 (6)	28 (3)
C(18)	7562 (9)	5487 (8)	3730 (6)	25 (3)
C(19)	8577 (11)	5216 (10)	3682 (8)	49 (4)
C(20)	9157 (12)	5814 (11)	3450 (8)	58 (4)
C(21)	8740 (11)	6701 (10)	3285 (8)	49 (4)
C(22)	7701 (12)	6970 (11)	3356 (8)	57 (4)
C(23)	7128 (11)	6381 (9)	3584 (7)	45 (3)
C(24)	7397 (10)	3919 (8)	3125 (7)	35 (3)
C(25)	7763 (10)	4315 (10)	2446 (7)	43 (3)
C(26)	8206 (11)	3720 (10)	1809 (9)	55 (4)
C(27)	8261 (12)	2800 (11)	1879 (9)	63 (4)
C(28)	7919 (11)	2450 (11)	2503 (8)	51 (4)
C(29)	7484 (10)	3006 (9)	3140 (7)	41 (3)
C(30)	6504 (9)	−152 (8)	−1892 (6)	31 (3)
C(31)	7078 (9)	217 (8)	−2466 (7)	29 (3)
C(32)	7141 (9)	1106 (8)	−2384 (6)	28 (3)
C(33)	6576 (9)	1613 (8)	−1720 (6)	31 (3)
C(34)	5989 (9)	1168 (8)	−1135 (7)	30 (3)
C(35)	6408 (10)	1929 (8)	−50 (7)	35 (3)
C(36)	6179 (11)	2629 (9)	480 (7)	45 (3)
C(37)	6944 (11)	2888 (10)	583 (8)	47 (4)
C(38)	7959 (11)	2453 (10)	180 (8)	51 (4)
C(39)	8183 (11)	1731 (10)	−325 (8)	50 (4)
C(40)	7430 (10)	1486 (9)	−432 (7)	41 (3)
C(41)	4463 (9)	2796 (8)	−206 (7)	34 (3)
C(42)	4805 (11)	3538 (9)	−515 (7)	47 (4)
C(43)	4119 (11)	4421 (10)	−491 (8)	53 (4)
C(44)	3094 (13)	4538 (12)	−147 (9)	63 (4)
C(45)	2744 (13)	3806 (11)	166 (9)	64 (4)
C(46)	3435 (11)	2941 (10)	150 (8)	52 (4)
C(47)	5554 (10)	−1323 (8)	−2333 (7)	35 (3)
C(48)	4513 (11)	−1216 (9)	−2036 (8)	45 (3)
C(49)	4008 (11)	−1223 (9)	−2492 (7)	45 (3)
C(50)	4493 (12)	−1341 (11)	−3217 (9)	64 (4)
C(51)	5579 (12)	−1451 (10)	−3538 (9)	56 (4)
C(52)	6071 (11)	−1434 (9)	−3066 (7)	45 (4)
C(53)	7392 (10)	−2160 (8)	−1961 (7)	35 (3)
C(54)	8150 (10)	−2059 (9)	−1717 (7)	40 (3)
C(55)	9085 (11)	−2761 (10)	−1847 (8)	52 (4)
C(56)	9235 (12)	−3573 (11)	−2240 (8)	55 (4)
C(57)	8480 (11)	−3682 (10)	−2468 (8)	48 (4)
C(58)	7584 (11)	−2983 (9)	−2330 (7)	44 (3)
C(59)	8420 (9)	−37 (8)	3799 (6)	31 (3)
C(60)	7837 (10)	−669 (10)	3968 (7)	44 (3)
C(61)	6839 (12)	−451 (11)	4443 (8)	58 (4)
C(62)	6367 (11)	399 (10)	4778 (8)	50 (4)
C(63)	6876 (10)	1063 (10)	4627 (7)	42 (3)
C(64)	7889 (9)	812 (8)	4154 (7)	34 (3)
C(65)	10193 (9)	−1399 (8)	3127 (7)	33 (3)
C(66)	10821 (10)	−1862 (9)	2459 (7)	38 (3)
C(67)	11330 (10)	−2806 (9)	2392 (7)	41 (3)
C(68)	11232 (10)	−3332 (9)	2991 (7)	40 (3)
C(69)	10638 (10)	−2910 (9)	3673 (7)	42 (3)
C(70)	10125 (10)	−1974 (8)	3737 (7)	36 (3)
C(71)	10229 (10)	255 (9)	3521 (7)	35 (3)
C(72)	10231 (9)	1178 (8)	3358 (6)	31 (3)
C(73)	10692 (10)	1636 (9)	3664 (7)	37 (3)
C(74)	11157 (9)	1211 (8)	4133 (7)	33 (3)
C(75)	11192 (10)	320 (9)	4314 (7)	39 (3)
C(76)	10719 (9)	−151 (9)	4000 (6)	33 (3)
C(77)	9603 (10)	55 (8)	2401 (7)	34 (3)
C(78)	10371 (10)	358 (9)	1886 (7)	39 (3)
C(79)	10394 (11)	588 (10)	1171 (8)	46 (4)
C(80)	9642 (11)	500 (10)	956 (9)	55 (4)
C(81)	8861 (11)	200 (10)	1433 (8)	52 (4)
C(82)	8847 (10)	−24 (9)	2149 (7)	40 (3)
C(83)	2090 (21)	3830 (14)	4485 (15)	124 (16)[a]
C(84)	4871	4098	4770	30
C(85)	3947	429	−134	30
C(86)	682 (37)	2768 (33)	8643 (26)	70
Cl(3)	932 (9)	2318 (8)	9382 (7)	70
Cl(4)	−311 (9)	3957 (8)	8784 (7)	70
B	9599 (11)	−283 (9)	3210 (7)	29 (3)

[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Table II. Selected Interatomic Distances (Å) for [Rh$_2${μ-(Ph$_2$P)$_2$py}$_2$(μ-CO)(μ-I)]$^+$

cation 1		cation 2	
Rh(1)–Rh(1)′	2.568 (2)	Rh(2)–Rh(2)′	2.577 (2)
Rh(1)–C(84)	2.02	Rh(2)–C(85)	2.04
Rh(1)–I(1)	1.859 (2)	Rh(2)–I(2)	2.919 (2)
Rh(1)–P(1)	2.292 (4)	Rh(2)–P(4)′	2.303 (3)
Rh(1)–P(2)′	2.238 (3)	Rh(2)–P(3)	2.251 (4)
Rh(1)–N(1)	2.081 (9)	Rh(2)–N(2)′	2.106 (10)
C(1)–P(2)	1.834 (13)	C(30)–P(4)	1.854 (14)
C(5)–P(1)	1.861 (10)	C(34)–P(3)	1.868 (12)

Table III. Selected Interatomic Angles in [Rh$_2${μ-(Ph$_2$P)$_2$py}$_2$(μ-CO)(μ-I)]$^+$

cation 1		cation 2	
Rn(1)′-Rh(1)-P(1)	159.3 (1)	Rh(2)′-Rh(2)-P(4)′	158.3 (1)
Rh(1)′-Rh(1)-P(2)′	93.3 (1)	Rh(2)′-Rh(2)-P(3)	94.4 (1)
Rh(1)′-Rh(1)-N(1)	89.4 (3)	Rh(2)′-Rh(2)-N(2)′	88.8 (3)
P(2)′-Rh(1)-N(1)	176.1 (3)	P(3)-Rh(2)-N(2)′	176.8 (3)
P(2)′-Rh(1)-P(1)	107.4 (1)	P(3)–Rh(2)–P(4)′	107.1 (1)
P(2)′-Rh(1)-I(1)′	91.9 (1)	P(3)-Rh(3)-I(2)	93.8 (1)
N(1)-Rh(1)-P(1)	69.8 (3)	N(2)′-Rh(2)-P(4)′	69.7 (3)

(Ph$_2$P)$_2$py. These structural similarities, along with the observation of an infrared absorption due to a bridging carbonyl, give compelling evidence for this bridging arrangement. Notice also that with a bridging carbon monoxide and iodide the cation **3** achieves an 18-electron count at each rhodium.

The (Ph$_2$P)$_2$py ligands form a chelate ring about one rhodium and serve as bridges to the second rhodium. This is the first observation of this geometrical arrangement for (Ph$_2$P)$_2$py. The chelate ring portion resembles a similar unit found in Ru(PPh$_2$py)(CO)$_2$Cl$_2$ (**5**),[9] while the central, bridging portion resembles similar sections found in Rh$_4${(μ-Ph$_2$P)$_2$py}$_2$(μ-CO)(CO)$_2$Cl$_4$ (**1**)[13] and in Rh$_2$(μ-Ph$_2$Ppy)(μ-CO)Cl$_2$ (**6**).[14] The bond

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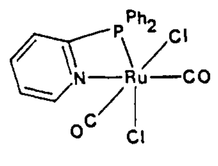

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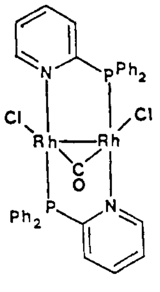

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lengths within the $Rh_2\{\mu\text{-}(Ph_2P)_2py\}_2$ core all appear normal. The lengthening of the Rh(1)–P(1) and Rh(2)–P(4)′ bonds relative to the Rh(1)–P(2)′ and Rh(2)–P(3) bonds can be largely ascribed to the trans effect of the Rh–Rh bond, since the longer bonds lie nearly opposite the Rh–Rh bond. Ring strain may also contribute to the lengthening of these bonds. However, the problems encountered in accomodating the fused four- and five-membered rings in **3** result in some angular distortions in the ligands. These can be seen and compared to related situations in Figure 4, which shows planar projections of the cores of the complexes **1**, **3**, **5**, and **6**. The angles within the four-membered chelate rings in **3** and **5** are very similar; all are significantly less than their ideal values. As a consequence phosphorus atoms P(1) and P(4) lie out of the plane of the $Rh_2(\mu\text{-}I)(\mu\text{-}CO)$ core, whereas in nonstrained complexes, ligands occupying these positions would be anticipated to lie in that plane. The presence of the four-membered rings in **3** produces noticeable changes in the bridging portion of the cation. Note that the bridging portions of **1** and **6**, which lack the fused four-membered ring, are very similar in both the internal angles and the NRhRhP dihedral angles. In **3** however, the Rh–N–C angle has been forced open by the bending on the opposite side, and the N–Rh–Rh–P dihedral angle is substantially reduced. As a result of these angular distortions, the nonbonded N···P separations are smaller in the chelate ring portions than in the bridging section.

Experimental Section

Preparation of Compounds. $(Ph_2P)_2py$[15] and $Rh_2\{\mu\text{-}(Ph_2P)_2py\}_2(CO)_2Cl_2$[8] were prepared as described previously.

$[Rh_2\{\mu\text{-}(Ph_2P)_2py\}_2(\mu\text{-}CO)(\mu\text{-}I)][BPh_4]$. A solution of 220 mg (1.50 mmol) of sodium iodide in 5 mL of methanol was added dropwise (to avoid the precipitation of an unidentified yellow solid) to a yellow solution of 250 mg (0.204 mmol) of $Rh_2\{\mu\text{-}(Ph_2P)_2py\}_2(CO)_2Cl_2$ in 20 mL of dichloromethane. After addition was complete, a solution of 210 mg (0.610 mmol) of sodium tetraphenylborate in 5 mL of methanol was added. The mixture was filtered and its volume reduced by half by the use of a rotary evaporator. The orange-brown crystalline product was collected by filtration and washed with methanol and ethyl ether. Purification was achieved by dissolving the product in dichloromethane, filtering, and adding a solution of 100 mg of sodium tetraphenylborate in 20 mL of methanol. The orange-brown product was collected by filtration, washed with methanol and ethyl ether, and vacuum-dried: yield 194 mg, 60%.

X-ray Crystallography. Well-formed crystals were obtained by slow diffusion of ether into a dichloromethane solution of the complex. Crystal data, data collection procedures and refinement procedures are summarized in Table IV. The lattice was found to be triclinic by standard procedures using the $P2_1$ software. No decay of the check reflections occurred during data collection. The structure was solved by the Patterson method. The asymmetric unit contains two half-molecules of the cation, $[Rh_2\{\mu\text{-}(Ph_2P)_2py\}_2(\mu\text{-}CO)(\mu\text{-}I)]^+$. A crystallographic center of inversion generates the other half of each molecule. There is an apparent statistical disorder in the positions of the CO and I groups. Although the iodine dominates the scattering, and carbon and oxygen do not appear on a difference map, their presence is strongly inferred. On the basis of the geometry of bridging CO in a related structure,[12] the oxygen and iodine positions can be expected to nearly coincide. If the iodine is assigned full occupancy, its thermal parameter is much larger than the average, while if it is assigned 0.5 occupancy, it is smaller than average. Therefore, a disordered model with I(1), I(2), O(1), O(2), C(84), and C(85) at 0.5 occupancy was used. These C and O atoms were given fixed U_{iso}'s of 0.03 Å^2. The C–O and Rh–O distances were fixed at 1.15 and 3.01 Å, respectively. The thermal parameters for I(1) and I(2) were refined and assumed normal values with this model. In addition to the crystallographic evidence, there was good spectroscopic evidence for a bridging CO in this compound. In the final cycles of refinement the Rh, I, and P atoms and one of the molecules of CH_2Cl_2 were assigned anisotropic thermal parameters. The other molecule of CH_2Cl_2 was more disordered, and was allowed to refine with a fixed isotropic U value of 0.07 Å^2 and a variable occupancy, which converged at 35.5 (6)%. In the final cycle of refinement, the mean shift/esd was 0.04. A few large peaks (ca. 3 e Å^{-3}) remained in the final difference map. These can be attributed to failure to fully correct for absorption, as they are in the vicinity of the Rh and I atoms. Crystallographic software was SHELXTL, version 4, installed on a Data General Eclipse S/230. Scattering factors and corrections for anomalous scattering were taken from ref 16. Corrections for extinction and absorption were applied to the data.[17] Positional parameters are presented in Table I.

Table IV. Crystal Data for $[Rh_2\{\mu\text{-}(Ph_2P)_2py\}_2(\mu\text{-}CO)(\mu\text{-}I)][BPh_4]\cdot 2CH_2Cl_2$

formula	$Rh_2ICl_4P_4ON_2C_{85}BH_{70}$
fw dimer + solvent	1744.75
cryst system	triclinic
space group	$P\bar{1}$
based on conditns	no conditns
cryst dimens, mm	0.28 × 0.37 × 0.125
color and habit	brown plates
unit cell dimens[a]	
a, Å	14.646 (5)
b, Å	15.210 (5)
c, Å	19.705 (6)
α, deg	85.63 (2)
β, deg	68.92 (2)
γ, deg	72.12 (2)
V, Å^3	3895 (2)
d(calcd),[a] g cm^{-3}	1.49
Z	2
radiatn; λ,[b] Å	Mo Kα; 0.071 069
μ(Mo Kα), cm^{-1}	10.7
range of abs cor factors	1.12–1.32
scan type; $2\theta_{max}$, deg	ω; 45
scan range, deg	1.5
background offset, deg	1.2
octants	$h,\pm k,\pm l$
scan speed, deg min^{-1}	60
check reflcn and interval no.	2 measured every 200 reflcns
no. of unique data	10 117
no. of data $I > 3\sigma(I)$	7593
R	0.079
R_w	0.088
no. of params	450

[a] At 130 K. [b] Graphite monochromator.

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Supplementary Material Available: Tables of hydrogen atom coordinates, bond lengths, bond angles, and atomic anisotropic thermal parameters for $[Rh_2\{\mu\text{-}(Ph_2P)_2py\}_2(\mu\text{-}CO)(\mu\text{-}I)][BPh_4]\cdot 2CH_2Cl_2$ (7 pages); a table of structure factors for $[Rh_2\{\mu\text{-}(Ph_2P)_2py\}_2(\mu\text{-}CO)(\mu\text{-}I)][BPh_4]\cdot 2CH_2Cl_2$ (45 pages). Ordering information is given on any current masthead page.

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(16) *International Tables for X-ray Crystallography*; Kynoch: Birmingham, England, 1974; Vol. 4.

(17) The method obtains an empirical absorption tensor from an expression relating F_o and F_c: Hope, H.; Moezzi, B. Department of Chemistry, University of California, Davis, CA.