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[BH_3·$PPh_2CH_2C_6H_4CH_2PPh_2$·Ru(*p*-cym)Cl_2]: A New Bifunctional Compound and Prototype of a Linked Borane/Metal Cluster Species

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Introduction

Bidentate phosphine ligands have been used extensively in coordination and organometallic chemistry.[1] They are best known as chelating ligands and as such have seen many applications in catalysis;[2] however, they can also be found in bridging situations. Thus they have been used to bridge simple metal-containing moieties[3] and metal clusters.[4] There are also many examples wherein the bidentate ligand is coordinated at one end only so that the other end is "dangling".[5] Such chemistry has been described in the literature recently, as have examples of such ligands bridging various groups. In this report we describe the use of the phosphine with a rigid backbone, α,α′-bis(diphenylphosphino)-*p*-xylene ($PPh_2CH_2C_6H_4CH_2PPh_2$, **1**), in linking a main group moiety with a transition metal-containing moiety.

Results and Discussion

Treatment of the rigid backbone base $PPh_2CH_2C_6H_4CH_2PPh_2$ (**1**) with [Ru(*p*-cym)Cl_2]$_2$ in CH_2Cl_2 at −78 °C, followed by warming to room temperature and purification using radial chromatography, allows separation of a brick-red solid powder, [$PPh_2CH_2C_6H_4CH_2Ph_2P$·Ru(*p*-cym)Cl_2] (**2**), in 62% yield. The species was identified by NMR spectroscopy and by elemental analysis. Treatment of **1** with BH_3·thf under essentially the same conditions followed by similar workup allowed the isolation of the brick-red crystalline solid [BH_3·$PPh_2CH_2C_6H_4CH_2PPh_2$·Ru(*p*-cym)Cl_2] (**3**) in 75% yield. In both cases the use of simple chromatography was not adequate for the separation of the products, but careful use of a chromatotron allowed purification. NMR spectral data are given for compounds **2** and **3** in the Experimental Section.

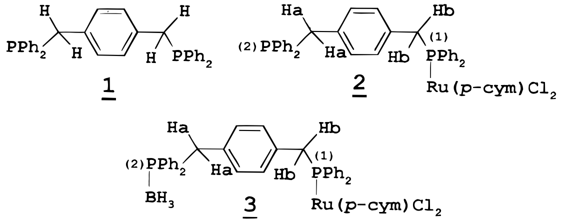

Figure 1. Diagrammatic representation of compounds **1**−**3**.

Drawings of the structures of **1**−**3** are given in Figure 1 and they are used to compare the NMR spectral data. In compound **1** the methylene protons of the *p*-xylene are observed as a singlet at 3.37 ppm (4H). Two-dimensional [$^1H-^{31}P$]-COSY experiments on compound **1** showed no evidence of $^2J_{H-P}$ coupling, so the coupling constant here can be assumed to be approximately zero. In compound **2** the methylene protons H_a exist as a doublet at 3.86 ppm with $^2J_{H-P} = 8.87$ Hz, while the methylene protons H_b are seen as a singlet at 3.18 ppm. Thus there is coupling between P(1) and H_a, but approximately zero coupling between P(2) and H_b. This is again confirmed in 2D [$^1H-^{31}P$]-COSY experiments on compound **2**. Another interesting feature of this compound is that long-range coupling between P(1) and P(2) with $^7J_{P-P} = 4.10$ Hz is observed. This coupling is confirmed in 2D [$^{31}P-^{31}P$]-COSY experiments. Whether this represents through bond coupling involving seven bonds is unclear, and most likely there is delocalization through the π-cloud of the benzene ring, which assists coupling. However, there are examples of seven-bond coupling to ^{31}P in the literature,[6a] and we have observed other relatively long-range $^{31}P-^{31}P$ coupling in related systems.[6b,c] In compound **3** the methylene protons H_a exist as a doublet at 3.86 ppm ($^2J_{H-P} = 8.85$ Hz), while the methylene protons H_b are also observed as a doublet at 3.36 ppm ($^2J_{H-P} = 11.94$ Hz). Again, we were able to confirm this pattern in 2D [$^1H-^{31}P$]-COSY experiments on **3**. As in the case with **2** there is long-range coupling observed between P(1) and P(2) ($^7J_{P-P} = 5.58$ Hz). The ^{31}P resonance for P(1) is a doublet at $\delta = 29.84$ ppm, while P(2) is observed a broad singlet at 17.94 ppm, as expected for a P atom bonded to boron.

Presumably the differences observed in the $^1H-^{31}P$ coupling for **2** and **3** can be attributed to the hybridization of the phosphorus atom. When the phosphorus is trigonal pyramidal (PR_3), the hybridization of phosphorus is p^3, with the s orbital primarily located on the lone pair. However, when the phos-

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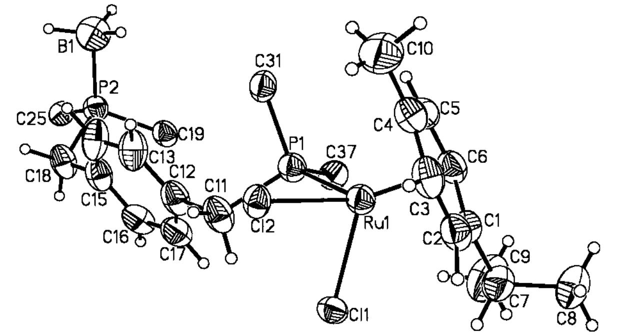

Figure 2. Projection of the structure of [(*p*-cym)$RuCl_2PPh_2CH_2C_6H_4CH_2Ph_2P\cdot BH_3$], **3**, with 50% thermal ellipsoids.

Table 1. Summary of Crystallographic Data for **3**

formula	$C_{44}H_{49}BCl_6P_2Ru$	fw	964.35
temp, K	217(2)	radiation (λ, Å)	Mo Kα (0.71073)
cryst syst	monoclinic	space group	$P2_1/n$
a, Å	11.7101(2)	*V*, Å^3	4520.75(12)
b, Å	15.8196(2)	*Z*	2
c, Å	24.4240(4)	D_{calcd}	1.417 Mg/m^3
α, deg	90	cryst size	0.1 × 0.1 × 0.05 mm^3
β, deg	92.339(1)	abs coeff	0.803 mm^{-1}
γ, deg	90	refl measured	8262 (±h, ±j, ±l)
no. variables	494	refl used	8262 with $I > 2\sigma(I)$
R	0.078	w*R*2^a	0.1504

[a] $wR2 = [\sum\omega(F_o - F_c)^2/\sum\omega F_o^2]^{1/2}$.

Table 2. Selected Bond Lengths (Å) and Angles (deg) for **3**

P(2)−B(1)	1.923(9)	P(2)−C(18)	1.833(8)
P(2)−C(25)	1.813(5)	P(2)−C(19)	1.808(5)
P(1)−Ru(1)	2.3619(13)	P(1)−C(11)	1.847(5)
P(1)−C(37)	1.820(5)	P(1)−C(31)	1.828(5)
C(25)−P(2)−C(18)	106.4(2)	C(11)−P(1)−Ru(1)	113.39(17)
C(25)−P(2)−C(19)	107.7(2)	C(37)−P(1)−Ru(1)	110.82(16)
C(19)−P(2)−C(18)	103.2(2)	C(37)−P(1)−C(11)	107.0(2)
C(19)−P(2)−B(1)	112.8(3)	P(1)−Ru(1)−Cl(1)	85.18(4)
C(25)−P(2)−B(1)	111.6(3)	P(1)−Ru(1)−Cl(2)	87.29(4)
C(18)−P(2)−B(1)	114.4(3)	Cl(1)−Ru(1)−Cl(2)	87.79(4)
C(31)−P(1)−C(37)	103.7(2)	P(1)−Ru(1)−cen^a	130.6
C(31)−P(1)−C(11)	103.2(3)	Cl(1)−Ru(1)−cen	125.8
C(31)−P(1)−Ru(1)	117.68(17)	Cl(2)−Ru(1)−cen	126.1

[a] cen is the centroid of the C_6 ring in *p*-cymene.

phorus forms an adduct (i.e., becomes pseudotetrahedral) the hybridization changes to essentially sp^3. Thus, since coupling information is communicated via the s electrons, the more s character a bond has, the higher the coupling between the atoms. This is demonstrated in **1**, where both the P atoms may be considered to be p^3 hybridized, and as a result there is approximately no coupling to the methylene protons. In **2**, P(1) becomes sp^3 hybridized and shows coupling to protons H_a, while P(2) remains p^3 hybridized and shows no coupling to protons H_b. In **3**, both P(1) and P(2) are sp^3 hybridized and show coupling to H_a and H_b, respectively. Similar observations of very low or effectively zero $^1H-^{31}P$ coupling have been made previously and accounted for in terms of the hybridization of the P atoms.[7]

Bright red rectangular crystals of **3**, containing 2 mol of CH_2Cl_2 solvent, were grown from a toluene/CH_2Cl_2/Et_2O solution at −5 °C. A projection of the structure is given in Figure 2 and a summary of crystallographic data is given in Table 1. Selected bond angles and distances are given in Table 2. The two P atoms in **3** are essentially syn to each other and the $C_{C_6H_4}-C_{methylene}-P$ planes have a dihedral angle of 4.5°, indicating two effectively coplanar linkages perpendicular to the connecting C_6H_4 ring. The actual angles between the interconnecting C_6H_4 ring and the planes C(15)−C(18)−P(2) and C(12)−C(11)−P(1) are 95.9° and 100.7°, respectively. This conformation appears to be the least sterically hindered, and it is illustrated in the alternative view, generated from the structural data, given in Figure 3. Thus on P(1) the largest substituent, $RuCl_2$(*p-cym*), points away from the basketlike arrangement of the $PCH_2C_6H_4CH_2P$ skeleton in a pseudo-exo orientation and the less sterically demanding phenyl groups are in equivalent pseudo-endo positions, twisted so as to reduce interaction. Similarly, the groups on P(2) adopt the positions of minimum steric interaction with the small BH_3 moiety and one of the phenyl groups in endo-type positions. Thus the BH_3 group interacts least with the C_6H_5 groups on P(2). On the other hand, the mutual arrangement of the groups around P(2) reflects that the C_6H_5 planes are able to twist to minimize interaction with each other, whereas the steric influence of the BH_3 group on the proximal phenyl groups is more important. Thus the ipso-carbon or methylene−P(2)−B(1) angles are in the range 111.6°−114.4°, whereas the corresponding angles between the C atoms on P(2) are less than the tetrahedral angle and fall in the range 103.2°−107.7°. The B(1)−P(2) distance is 1.923(9) Å and is essentially the same as the value for $PPh_3\cdot BH_3$ (1.933 Å).[8] Adding to the steric demands of the Ru moiety is the fact that the metal is effectively octahedral, assuming that the *p*-cymene ligand occupies three coordination positions with the two Cl atoms and the P atom occupying the others. Thus the angles to the Cl atoms and P are approximately 90°, their values ranging from ca. 85°−88°, reflecting the steric bulk of the *p*-cymene ligand. It is informative to compare the structural features around the Ru atom to those for such species as [(*p*-cym)$RuCl_2P(CH_2Ph)_3$], in which the angles between the P and Cl atoms are somewhat smaller, reflecting less steric hindrance in **3**.[9]

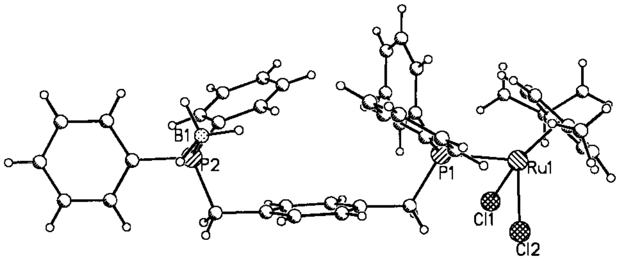

Figure 3. Alternative view illustrating the syn-orientation of the P atoms of **3**.

The species **3** is novel and may be considered as prototypical of a new type of linked borane/metal cluster system. The RuC_5 system is a cluster of atoms and the BH_3 group, or a phosphine−borane group, may be incorporated into a larger borane cluster system. Linked borane cluster to metal cluster systems are quite rare, to our knowledge; the only ones which have appeared in the literature are the $Os_3C-B_5H_8$ and $-C-C_2B_{10}$ systems reported by Shore et al. several years ago.[10] There are, however, several examples of clusters involving carboranes linked by a variety of organic groups, which we are not addressing herein.[11] Of related interest to the chemistry we describe is the use of

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the dppf (1,1′-bis(diphenylphosphino)ferrocene) ligand, which has been shown to chelate metals coordinating to boron atoms in B_3H_7[12] and, in studies pertinent to this one, link a series of identical borane species ranging from BH_3 to systems such as SB_9H_{11} and $C_2B_{10}H_{12}$.[13] Our formation of **3** relates to other work in our laboratory involving the formation of linked cluster systems,[14,15] and we intend to report on these soon.

Experimental Section

General. Reactions were carried out under a dry nitrogen atmosphere on a high vacuum line. Solvents used were reagent grade and dried before use. PPh_2Cl (Strem) and BH_3•thf solution (Aldrich) was used as received, while α,α′dibromo-*p*-xylene (Eastman Organic) was sublimed before use. The compounds $PPh_2CH_2C_6H_4CH_2PPh_2$[4c] and [(*p*-cym)$RuCl_2$]$_2$[16] were prepared according to published procedures. Products were purified on a radial chromatograph using a 25 cm diameter circular plate coated with 0.1 cm of silica gel (EM Science). ^{31}P NMR spectra (referenced to 85% H_3PO_4), ^{11}B spectra (referenced to Et_2O•BF_3), and ^1H spectra (referenced to $SiMe_4$) were carried out on a Brucker ARX 500 spectrometer. Mass spectra were recorded by the Washington University Mass Spectrometry Resource, an NIH Research Resource (Grant No. P41RR0954), and elemental analyses were carried out by Atlantic Microlabs Inc., Norcross, GA.

Synthesis of [$\mathbf{PPh_2CH_2C_6H_4CH_2PPh_2}$•Ru(*p*-cym)$\mathbf{Cl_2}$]. In a 50 mL two-necked round-bottom flask was placed 500 mg (1.05 mmol) of $PPh_2CH_2C_6H_4CH_2PPh_2$ together with a stir bar. A tipper tube charged with 60 mg (0.100 mmol) of [(*p*-cym)$RuCl_2$]$_2$ was then attached. The flask was then connected to the high vacuum line and evacuated, at which point it was cooled to −78 °C and 20 mL of CH_2Cl_2 was condensed in and the [(*p*-cym)$RuCl_2$]$_2$ added. The mixture was stirred for 30 min, warmed slowly to room temperature, filled with dry nitrogen, and then exposed to the air. The solution was then reduced to 1 mL in volume and applied to the radial chromatograph using CH_2Cl_2 as eluent. A colorless band was observed which was shown to be unreacted phosphine, then CH_3CN was slowly added to the eluent until a red band was detected, which was later characterized as [$PPh_2CH_2C_6H_4CH_2PPh_2$•Ru(*p*-cym)Cl_2]. The solvent was then removed, leaving 97 mg (62.1%) of [$PPh_2CH_2C_6H_4CH_2PPh_2$•Ru(*p*-cym)Cl_2]. ^1H NMR ($CDCl_3$): *p*-cymene, δ 0.88 (d, $J = 6.95$ Hz, 6H, $\mathrm{CH}\mathit{Me}_2$), 1.81 (s, 3H, Me), 2.47 (sept, $J = 6.95$ Hz, 1H, $\mathrm{C}\mathit{H}\mathrm{Me}_2$), 5.10 (d, $J = 5.80$ Hz, 2H, $\mathrm{Me}\mathit{C_6H_4}\mathrm{CHMe_2}$), 5.23 (d, $J = 5.80$ Hz, 2H, $\mathrm{Me}\mathit{C_6H_4}\mathrm{CHMe_2}$); $PPh_2CH_2C_6H_4CH_2PPh_2$, δ 3.18 (s, 2H, $\mathrm{PPh_2}\mathit{CH_2}$), 3.86 (d, $^2J_{\mathrm{H-P}} = 8.87$ Hz, 2H, $\mathit{CH_2}\mathrm{PPh_2}$•Ru(*p*-cym)$Cl_2$), 6.19 (d, $J = 7.24$ Hz, 2H, $\mathrm{CH_2}\mathit{C_6H_4}\mathrm{CH_2}$), 6.51 (d, $J = 7.24$ Hz, 2H, $\mathrm{CH_2}\mathit{C_6H_4}\mathrm{CH_2}$), 7.15−7.77 (m, 20H, Ph). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ −9.57 (d, $^7J_{\mathrm{P-P}} = 4.10$ Hz, $\mathit{PPh_2}$CH2), 29.78 (d, $^7J_{\mathrm{P-P}} = 4.10$ Hz, $\mathrm{CH_2}\mathit{PPh_2}$.Ru(*p*-cym)$Cl_2$). Anal. Calcd for $C_{42}H_{42}P_2Cl_2Ru$: C, 64.61; H, 5.43. Found: C, 64.07; H, 5.44.

Synthesis of [$\mathbf{BH_3}$•$\mathbf{PPh_2CH_2C_6H_4CH_2PPh_2}$•Ru(*p*-cym)$\mathbf{Cl_2}$]. In a 50 mL two-necked round-bottom flask was placed 60 mg (0.077 mmol) of $PPh_2CH_2C_6H_4CH_2PPh_2$•Ru(*p*-cym)Cl_2 together with a stir bar. The flask was connected to the high vacuum line and evacuated. The vessel was then cooled to −78 °C, 20 mL of CH_2Cl_2 was condensed in, and the vessel was filled with dry N_2 at that temperature. Then BH_3•thf solution (0.3 mmol) was introduced via a syringe, under a positive N_2 flow. The solution was stirred at this temperature for 15 min, warmed slowly to room temperature, exposed to air, reduced to 1 mL in volume, and applied to the radial chromatograph using CH_2Cl_2 as the mobile phase. CH_3CN was then slowly added to the eluent until a red band was detected. The bright red solution obtained from this band was then evacuated to dryness, giving 46 mg (75.3%) of a brick-red solid which was characterized as [BH_3•$PPh_2CH_2C_6H_4CH_2PPh_2$•Ru(*p*-cym)Cl_2], **3**. Crystals suitable for X-ray diffraction were grown from a toluene/CH_2Cl_2/Et_2O solution at −5 °C. ^1H NMR ($CDCl_3$): *p*-cymene, δ 0.90 (d, $J = 7.20$ Hz, 6H, $\mathrm{CH}\mathit{Me}_2$), 1.80 (s, 3H, Me), 2.47 (sept, $J = 7.20$ Hz, 1H, $\mathrm{C}\mathit{H}\mathrm{Me}_2$), 5.10 (d, $J = 5.06$ Hz, 2H, $\mathrm{Me}\mathit{C_6H_4}\mathrm{CHMe_2}$), 5.23 (d, $J = 5.06$ Hz, 2H, $\mathrm{Me}\mathit{C_6H_4}\mathrm{CHMe_2}$); $PPh_2CH_2C_6H_4CH_2PPh_2$, δ 3.36 (d, $^2J_{\mathrm{H-P}} = 11.9$ Hz, 2H, $\mathrm{BH_3PPh_2}\mathit{CH_2}$), 3.86 (d, $^2J_{\mathrm{H-P}} = 8.8$ Hz, 2H, $\mathit{CH_2}\mathrm{PPh_2}$•Ru(*p*-cym)$Cl_2$), 6.17 (d, $J = 7.58$ Hz, 2H, $\mathrm{CH_2}\mathit{C_6H_4}\mathrm{CH_2}$), 6.39 (d, $J = 7.58$ Hz, 2H, $\mathrm{CH_2}\mathit{C_6H_4}\mathrm{CH_2}$), 7.14−7.80 (m, 20H, Ph); BH_3, δ 0.83 (d, $^2J_{\mathrm{H-P}} =15.9$, 3H, $\mathrm{B}\mathit{H_3}$). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ 17.94 (br, BH_3•$\mathit{PPh_2}\mathrm{CH_2}$), 29.84 (d, $^7J_{\mathrm{P-P}} = 5.58$ Hz, $\mathrm{CH_2}\mathit{PPh_2}$•Ru(p-cym)$Cl_2$). $^{11}B\{^1H\}$ NMR ($CDCl_3$): δ −38.81 (s, $\mathit{B}\mathrm{H_3}$). HRMS: FAB (NBA-Li) $^{12}C_{42}{}^1H_{45}{}^{31}P_2{}^{37}Cl_2{}^{11}B_1Ru_1{}^7Li$, 801.1670 (calcd), 801.1705 (obsd), standard deviation 4.3 ppm.

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Supporting Information Available: CIF file of crystallographic data. This material is available free of charge via the Internet at http://pubs.acs.org.

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