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Notes

Synthesis of the Tetradentate Phosphine α,α,α′,α′-Tetrakis(diphenylphosphino)-*p*-xylene (dppx) and Crystal and Molecular Structure of $(CO)_3Fe(dppx)Fe(CO)_3$

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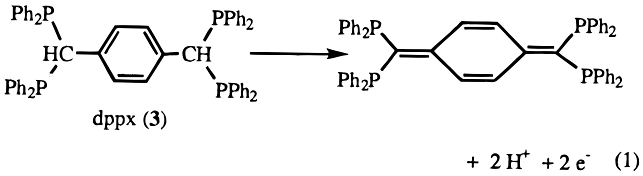
*Summary: The tetradentate phosphine α,α,α′,α′-tetrakis(diphenylphosphino)-p-xylene (**3**, dppx) was prepared in two steps from α,α′-bis(diphenylphosphino)-p-xylene (**1**) via the intermediate trisubstituted phosphine α,α,α′-tris(diphenylphosphino)-p-xylene (**2**). The ligand, dppx, reacts with $Fe_2(CO)_9$ to afford a bridging bischelated complex $Fe(CO)_3(dppx)Fe(CO)_3$ (**4**), which was characterized by X-ray diffraction. Complex **4** crystallized in the monoclinic space group $P2_1/n$, $a = 11.903(1)$ Å, $b = 15.295(2)$ Å, $c = 15.415(1)$ Å, $\beta = 96.306(9)°$, $V = 2789.5(8)$ Å^3, $Z = 2$, $R = 0.072$, and $R_w = 0.079$.*

Introduction

The use of polydentate phosphines as ligands in organometallic chemistry is pervasive[1−8] and has been reviewed recently.[9] The role of polydentate phosphines in promoting cooperative catalytic phenomena has also been reported.[10] The synthesis of novel polydentate phosphine ligands and the catalytic properties of organometallic compounds prepared from these ligands are areas of ongoing interest in our laboratory.[11−13] We report here a convenient synthesis of α,α,α′,α′-tetrakis(diphenylphosphino)-*p*-xylene (**3**, dppx). The new ligand dppx is the hydroquinone form of tetrakis(diphenylphosphino)quinodimethane, eq 1. The reaction of dppx with $Fe_2(CO)_9$ and the characterization of the diiron hexacarbonyl dimer $(CO)_3Fe(dppx)Fe(CO)_3$ (**4**) by X-ray crystallography are also reported.

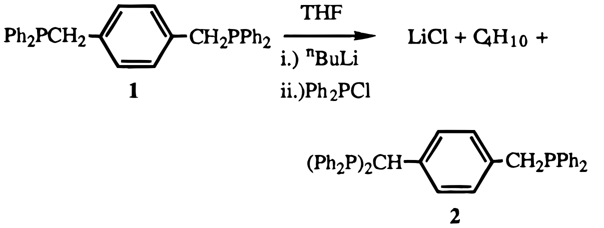


dppx (**3**)

(1)

Results and Discussion

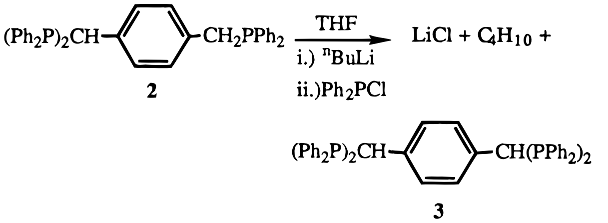
The synthesis of the tetradentate phosphine dppx is based on a stepwise approach starting from α,α′-bis(diphenylphosphino)-*p*-xylene (**1**).[14] Diphosphine **1** was reacted with 1 equiv of a strong non-nucleophilic base such as ^nBuLi at −40 °C. The subsequent addition of chlorodiphenylphosphine to the lithiated phosphine intermediate affords the trisubstituted α,α,α′-tris(diphenylphosphino)-*p*-xylene (**2**) as a white solid in 60% yield, eq 2. The same conditions can then be reapplied to **2** to yield the tetradentate phosphine α,α,α′,α′-tetrakis(diphenylphosphino)-*p*-xylene (dppx, **3**), eq 3.



1

2

(2)



2

3

(3)

Compound **3** is an air-sensitive white solid, which is soluble in methylene chloride, tetrahydrofuran, toluene, and benzene. As isolated, dppx (**3**) displays a single resonance in the $^{31}P\{^1H\}$ NMR at $\delta = -5.5$ ppm. The ^1H NMR spectrum shows singlets at $\delta = 4.2$ ($^2J_{PH} < 1$

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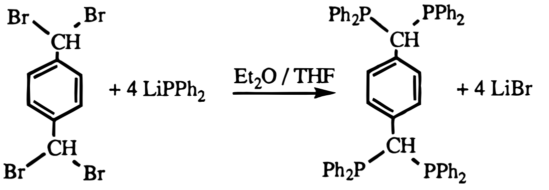
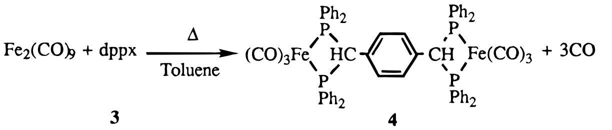
Hz) and 6.4 ppm that correspond to the methyne and aryl ring carbons of the xylene spacer, respectively. The molecular ion of **3** is observed by plasma desorption mass spectrometry (PDMS) ($m/z = 842.4$), along with the ions resulting from four stepwise oxygenations of the tetraphosphine, ($\mathbf{3} + n$O, $n = 1, 2, 3, 4$). In contrast, the molecular ion of **2** is observed by PDMS ($m/z = 658.5$) with ions resulting from three stepwise oxygenations of the triphosphine, ($\mathbf{2} + n$O, $n = 1, 2, 3$).

We find that the tetradentate phosphine (**3**) can also be prepared in a single step, via slow addition of lithium diphenylphosphide to a stirred solution of the α,α,α′,α′-tetrabromo-*p*-xylene precursor in a 10:1 diethyl ether/THF mixture (eq 4, method B in **Experimental Section**). Although the one step synthesis of dppx is viable, the yield is low (*ca.* 10%) and a large percentage of side products are also generated. This makes the isolation of **3**, as an analytically pure compound, difficult by the latter method. Thus, we prefer the stepwise synthesis of **3** (Method A in **Experimental Section**).



3 (4)

In order to study the properties of dppx as a ligand, it was reacted with $Fe_2(CO)_9$, eq 5. The resulting



3 **4** (5)

dinuclear iron complex, **4**, exhibits a single resonance in the $^{31}P\{^1H\}$ NMR at $\delta = 40.0$ ppm (CD_2Cl_2) and in a region similar to that observed for Fe(η^2-dppm)(CO)$_3$.[15,16] PDMS of **4** reveals the parent MH^+ ion, $m/z = 1124.1$, as well as fragment ions resulting from the loss of carbonyl groups $MH^+ - n(CO)$ ($n = 1, 2, 3, 6$). The infrared spectrum of **4** (KBr) shows three intense ν(CO) bands at 1982, 1911, and 1897 cm^{-1}. These compare very well with those reported for Fe(η^2-dppm)(CO)$_3$ (ν_{CO} = 1977, 1908, 1897 cm^{-1}).[16]

Crystals of **4** suitable for X-ray diffraction were obtained by the slow vapor diffusion of hexanes into a concentrated toluene solution of **4** under nitrogen. The overall structure is a bis(chelated) diiron carbonyl complex. An ORTEP drawing of **4** is presented in Figure 1. A summary of the crystallographic study is presented in Table 1. Selected bond distances and bond angles for compound **4** are given in Tables 2 and 3. The iron atoms are separated by 15.58 Å and related crystallographically by an inversion center within the bridging xylene group. The dppx ligand is nonplanar, with the diphosphinomethane groups at dihedral angles of approximately 60° with respect to the xylyl ring; the torsion angle P(1)−C(10)−C(11)−C(12) is 118(1)°. The overall structural aspects of compound **4** are similar to those found in the crystal structure of Fe(η^2-dppm)(CO)$_3$.[17] The geometry about the iron atoms differs from

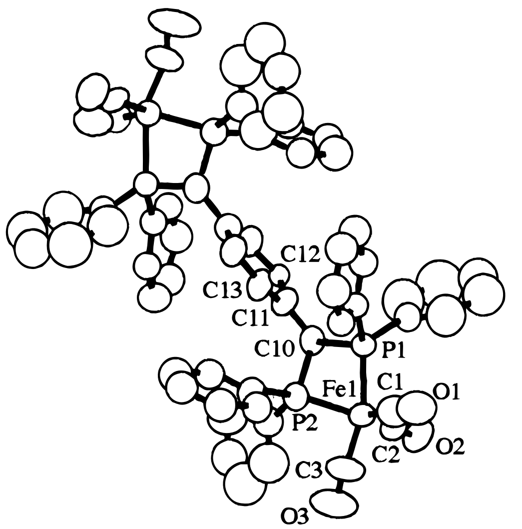


Figure 1. Molecular structure of **4** (ORTEP, 50% probability ellipsoids). Selected bond lengths (Å) and bond angles (deg) are given in Tables 2 and 3.

Table 1. Summary of Crystallographic Data for 4

formula	$Fe_2P_4O_6C_{62}H_{46}$	V, Å^3	2789.5(8)
fw	1122.64	Z	2
cryst size, mm	0.35 × 0.31 × 0.31	temp, °C	20
cryst syst	monoclinic	D_{calcd}, g cm^{-3}	1.337
space group	$P2_1/n$ (No. 14)	μ, cm^{-1}	6.81
a, Å	11.903(1)	radiation (λ, Å)	Mo Kα (0.710 73)
b, Å	15.295(2)	no. rflns measd	3802 ($\pm h,k,l$)
c, Å	15.415(1)	no. rflns used	1446 with $I > 3\sigma(I)$
α, deg	90.0	no. variables	214
β, deg	96.306(9)	R	0.072
γ, deg	90.0	R_w[a]	0.079

[a] $R_w = [\sum w(F_o - F_c)^2/\sum wF_o^2]^{1/2}$.

Table 2. Selected Bond Distances (Å) for 4

Fe(1)−P(1)	2.208(4)	O(2)−C(2)	1.18(2)
Fe(1)−P(2)	2.205(4)	O(3)−C(3)	1.16(2)
Fe(1)−C(1)	1.73(2)	C(10)−C(11)	1.52(2)
Fe(1)−C(2)	1.71(2)	C(11)−C(12)	1.37(2)
Fe(1)−C(3)	1.73(2)	C(11)−C(13)	1.40(2)
P(1)−C(10)	1.84(1)	C(12)−C(13)	1.38(2)
P(1)−C(111)	1.79(1)	C(111)−C(112)	1.31(2)
P(1)−C(121)	1.82(1)	C(111)−C(116)	1.32(2)
P(2)−C(10)	1.85(1)	C(112)−C(113)	1.43(3)
P(2)−C(211)	1.84(1)	C(113)−C(114)	1.32(3)
P(2)−C(221)	1.85(2)	C(114)−C(115)	1.28(3)
O(1)−C(1)	1.15(1)	C(115)−C(116)	1.44(3)

Table 3. Selected Bond Angles (deg) for 4

P(1)−Fe(1)−P(2)	73.3(2)	Fe(1)−P(2)−C(10)	95.3(4)
P(1)−Fe(1)−C(1)	95.8(5)	Fe(1)−P(2)−C(211)	120.5(5)
P(1)−Fe(1)−C(2)	90.7(6)	Fe(1)−P(2)−C(221)	124.0(6)
P(1)−Fe(1)−C(3)	162.1(6)	C(10)−P(2)−C(211)	109.9(7)
P(2)−Fe(1)−C(1)	109.1(5)	C(10)−P(2)−C(221)	103.6(7)
P(2)−Fe(1)−C(2)	137.3(6)	C(211)−P(2)−C(221)	101.7(7)
P(2)−Fe(1)−C(3)	92.6(6)	Fe(1)−C(1)−O(1)	178(2)
C(1)−Fe(1)−C(2)	111.8(8)	Fe(1)−C(2)−O(2)	179(2)
C(1)−Fe(1)−C(3)	99.2(8)	Fe(1)−C(3)−O(3)	177(2)
C(2)−Fe(1)−C(3)	92.6(8)	P(1)−C(10)−P(2)	91.0(6)
Fe(1)−P(1)−C(10)	95.5(5)	P(1)−C(10)−C(11)	122.8(9)
Fe(1)−P(1)−C(111)	120.3(5)	P(2)−C(10)−C(11)	125(1)
Fe(1)−P(1)−C(121)	121.3(5)	C(10)−C(11)−C(12)	120(1)
C(10)−P(1)−C(111)	108.6(7)	C(10)−C(11)−C(13)	121(1)
C(10)−P(1)−C(121)	106.4(6)	C(12)−C(11)−C(13)	118(1)
C(111)−P(1)−C(121)	103.2(6)	C(11)−C(12)−C(13)	122(1)
Fe(1)−P(2)−C(10)	95.3(4)	C(11)−C(13)−C(12)	120(1)

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an idealized trigonal bipyramid. The somewhat narrow P(1)−Fe−P(2) angle (73.3(2)°) is found for both dppx and dppm iron tricarbonyls and is typical of chelating methylene diphosphine ligands.[17,18] The two substituents occupying the axial sites on the iron atom in **4**, P(1) and C(3), are related by an angle of only 162.1(6)°. The atoms occupying the three sites of the "trigonal plane" are nearly coplanar, but form ligand−Fe−ligand angles of 109.1(5)°, 111.8(8)°, and 137.3(6)°. It is well-known that dppm has the ability to either chelate a single metal center to bridge two metal centers. Both of these bonding modes for dppm have been observed in complexes of iron carbonyls.[17,19] Formation of bridged metal−metal bonded complexes of dppx have not been isolated to date. It is likely that the presence of substituents on the diphosphinomethane carbon atom imposes steric limitations on the P−C−P bond angle that favor chelation over bridging.

In spite of considerable efforts to oxidize dppm to its quinone form, eq 1, tetra(diphenylphosphino)quinodimethane has not been isolated to date. In contrast, bromine oxidation of α,α,α′,α′-tetracyano-*p*-xylene is one method of preparing TCNQ.[20] The electrochemistry of dppx produced cyclic voltammograms in aprotic solvents that appeared similar to 1,4-hydroquinone. Under these conditions, **3** showed irreversible oxidations at ca. +1.1 V vs SCE. However, stable products could not be isolated from bulk electrolyses. Chemical oxidation of dppx to the quinone is complicated by oxidation of the four phosphorus(III) centers. The use of traditional oxidizing agents (i.e., peroxides, quinones, Sn_2Me_6, AIBN, Cl_2, Br_2, I_2, $(NH_4)_2Ce(NO_3)_6$, $NOBF_4$, $FeCl_3$, $AgNO_3$) resulted in either oxygenation of the phosphorus atoms or decomposition. Steric hindrance about the diphosphinomethane carbon atoms and the σ-donor and π-acceptor properties of the phosphine groups may also contribute to the difficulty of preparing tetra(diphenylphosphino)quinodimethane. A general lack of stability is observed in quinodimethanes when strong electron-withdrawing substituents are not present.[20−24] The oxidation of dppx, complexed to transition metal centers, may afford better routes to phosphinoquinodimethanes.

Conclusion

The synthesis of the new tetrakis(diphenylphosphino)-*p*-xylene, **3** (dppx), has been achieved. A novel trisubstituted α,α,α′-tris(diphenylphosphino)-*p*-xylene, **2**, was isolated and characterized *en route* to **3**. The dppx ligand was characterized by a crystal structure of its diiron hexacarbonyl complex, **4**. Although dppx may be viewed as the hydroquinone form of tetrakis(diphenylphosphino)quinodimethane, eq 1, its oxidation to the quinone form has not yet been realized.

Experimental Section

General Procedures. All manipulations were performed under N_2 atmosphere using standard Schlenk techniques. Organic solvents were dried and degassed using standard procedures. α,α′-bis(diphenylphosphino)-*p*-xylene (**1**) was freshly prepared from α,α′-dibromo-*p*-xylene purchased from Aldrich Chemical via a previously reported method.[14] ^{1}H NMR and $^{31}P\{^1H\}$ NMR spectra were recorded on a Varian XL-200 spectrometer and referenced to TMS and 85% H_3PO_4, respectively.

Synthesis of α,α,α′-Tris(diphenylphosphino)-*p*-xylene (2). A solution of α,α′-bis(diphenylphosphino)-*p*-xylene (**1**, 2.50 g, 5.27 mmol) in THF (500 mL) was cooled to −40 °C. One equivalent of ^{n}BuLi (0.34 mL, 5.3 mmol) in hexanes was slowly added to the stirred solution. The solution was stirred for 10 min at −40 °C and allowed to warm to 0 °C with continual stirring for 1 h. The solution turns from yellow to orange and finally to deep red. One equivalent of (1.16 g, 5.27 mmol) Ph_2PCl was then added dropwise at 0 °C. The solution immediately changed to pale yellow and was allowed to warm to room temperature. The phosphine α,α,α′-tris(diphenylphosphino)-*p*-xylene (**2**) was isolated as a pure white crystalline solid from slow evaporation of a saturated acetonitrile solution. The solid was washed with hexanes and dried under vacuum to give 2.08 g (60%) of **2**. ^{1}H NMR (CD_2Cl_2): δ 4.4 (s, 1H, $-CH(PPh_2)_2$, $^2J_{PH} < 1$ Hz), 3.2 (s, 2H, $-CH_2(PPh_2)$, $J_{PH} < 1$ Hz), 6.75 (dd, 4H, $-C_6H_4$), 7.5 (m, 30H, C_6H_5). $^{31}P\{^1H\}$ NMR (CD_2Cl_2): δ −4.5 (s, 2P, $-CH(PPh_2)_2$), −9.4 (s, 1P, $-CH_2(PPh_2)$). Anal. Calcd for $C_{44}H_{37}P_3$: C, 80.22; H, 5.66. Found C, 79.80; H, 5.71. The plasma desorption mass spectrum was measured, and the **2** + H^+ ion was observed at $m/z = 658.5$, along with the oxygenated fragment ions (**2** + O, 674.9; **2** + 2O, 690.7; and **2** + 3O, 707.0).

Synthesis of α,α,α,′α′-Tetrakis(diphenylphosphino)-*p*-xylene (3). Method A. A flask was charged with α,α,α′-tris(diphenylphosphino)-*p*-xylene (**2**, 1.00 g, 1.52 mmol) in 200 mL of THF. The solution was cooled to −40 °C. One equivalent of ^{n}BuLi (0.10 mL, 1.5 mmol) was added, as in the preparation of **2**. One equivalent of Ph_2PCl (0.335 g, 1.52 mmol) was added, and the solution was allowed to warm up slowly to room temperature. The solvent was then removed under vacuum to yield a white solid. The phosphine α,α,α′,α′-tetrakis(diphenylphosphino)-*p*-xylene (**3**, dppx) was isolated by washing the solid first with acetonitrile, followed by extraction with methylene chloride. Addition of acetonitrile to the filtrate gave a white solid, which was filtered,washed with acetonitrile, and dried under vacuum to give 0.333 g (26%) of **3**. ^{1}H NMR (CD_2Cl_2): δ 4.2 (s, 2H, $-CH(PPh_2)_2$, $J_{PH} < 1$ Hz), 6.4 (s, 4H, C_6H_4), 7.0 (m, 40H, C_6H_5). $^{31}P\{^1H\}$ NMR (CD_2Cl_2): δ −5.5 ppm. Anal. Calcd for $C_{56}H_{46}P_4$: C, 79.79; H, 5.50. Found C, 79.54; H, 5.47. The plasma desorption mass spectrum was measured, and the **3** + H^+ ion was observed at $m/z = 842.4$, along with the oxygenated fragment ions (**3** + O, 858.6; **3** + 2O, 875.5; **3** + 3O, 890.5; and **3** + 4O, 907.5).

Synthesis of α,α,α,′α′-Tetrakis(diphenylphosphino)-*p*-xylene (3). Method B. A 10:1 mixture of diethyl ether/THF (300 mL) was used to dissolve 2.50 g (5.93 mmol) of α,α,α,′α′-tetrabromo-*p*-xylene. The stirred solution was then cooled to 0 °C, and lithium diphenylphosphide (4.56 g, 23.7 mmol) in 100 mL of diethyl ether was added dropwise over 3 h. The solution turned orange in color. After addition was complete, distilled water (200 mL) was added. The layers were allowed to separate, and the organic layer was removed and evaporated to an oil. Addition of acetonitrile (100 mL) produced a light yellow solid. After the solid was stirred, the reaction solid was filtered, washed with acetonitrile, and dried. The solid can be recrystallized from methylene chloride to give 0.500 g (<10%) of **3**. ^{1}H NMR (CD_2Cl_2): δ 4.2 (s, 2H, $-CH(PPh_2)_2$), 6.4 (s, 4H, C_6H_4), 7.4 (m, 40H, C_6H_5). $^{31}P\{^1H\}$ NMR (CD_2Cl_2): δ −5.5. The plasma desorption mass spectrum was measured, and the **3** + H^+ ion was observed at $m/z = 842.4$, along with

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the oxygenated fragment ions (**3** + O, 858.6; **3** + 2O, 875.5; **3** + 3O, 890.5; and **3** + 4O, 907.5), and other fragments identical to those observed in the spectrum of **3** prepared by method A.

Synthesis of Diiron Hexacarbonyl α,α,α,′α′-Tetrakis(diphenylphosphino)-*p*-xylene (4). A flask was charged with dppx (**3**, 0.200 g, 0.237 mmol) and 100 mL toluene. Diiron nonacarbonyl (0.100 g, 0.275 mmol) was added, and the solution was heated at 80 °C for 12 h with periodic N_2 purges. The orange solution was cooled to room temperature, and hexane was added. The orange precipitate was filtered and washed with hexanes. The solid was recrystallized from methylene chloride/hexanes to yield compound **4** as an orange solid (0.173 g, 65%). ^{1}H NMR ($CDCl_3$): δ 5.6 (t, 2H, $-CH(PPh_2)_2$, $^2J_{PH} = 12.6$ Hz), 5.3 (s, 2H, CH_2Cl_2), 7.3 (m, 44H, C_6H_5). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ 40. Anal. Calcd for $C_{62}H_{46}O_6P_4Fe_2 \cdot 1.5(CH_2Cl_2)$: C, 61.01; H, 3.95. Found: C, 60.69; H, 3.96. The plasma desorption mass spectrum was measured, and the **4** + H^+ ion was observed at $m/z = 1124.1$, along with the fragment ions resulting from CO loss (**4** − CO, 1097.3; **4** − 2CO, 1066.9; **4** − 3CO, 1039.7; and **4** − 6CO, 956.0).

Crystal Data for 4. Crystals were grown slowly over 2 weeks by vapor diffusion of hexanes into a saturated toluene solution of **4**. The structure was solved by using the structure solution program SHELX-86. The remaining atoms were located in succeeding difference Fourier syntheses. The structure was refined to $R = 0.072$ and $R_w = 0.079$ for 1446 observations with $I > 3\sigma(I)$ with Mo Kα radiation. Two of the dppx phenyl rings (C(111)−C(116) and C(221)−C(226)) showed unusually high thermal motion ($B > 10$ Å^2) but could not be fit to a reasonable disorder model. Data were corrected for absorption empirically, $\mu = 6.81$ cm^{-1}. Data was collected using an Enraf-Nonius CAD4 computer axis diffractometer. The cell constants and orientation matrix for data collection were obtained from least-squares refinement. Crystal data are summarized in Table 1. A total of 3802 reflections were collected, of which 3802 were unique. Hydrogen atoms were located and added to the structure factor calculations, but their positions were not refined. Refinement was done using MolEN. Atomic coordinates, bond lengths and angles, thermal parameters, and observed and calculated structure factors are available as Supporting Information.

Acknowledgment is made to the NSF (CHE-9319173) and the Army Research Office URI program (DAAL03-92-G-0144) for support of this work. A.A.B. gratefully acknowledges a Purdue Research Foundation Graduate Fellowship.

Supporting Information Available: Description of crystallographic experimental procedures and tables of positional parameters, general temperature factor expressions, bond distances and angles, and torsion angles (16 pages). Ordering information is given on any current masthead page.

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