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Formation of a Cyclic Bis(iminophosphorane) from a 2,2′-Bis(phosphino)azobenzene via N=N Bond Cleavage

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ABSTRACT: *An eight-member cyclic bis(iminophosphorane) was synthesized by treatment of 2,2′-bis(phosphino)azobenzene with pentachlorophenol via N=N bond cleavage of the azobenzene. The crystal structure of the cyclic bis(iminophosphorane) was determined by the X-ray crystallographic analysis. A study on the reaction mechanism showed that both protonation of the azo moiety and intramolecular nucleophilic attacks of the phosphino groups played a key role in the formation of the bis(iminophosphorane) from the azo compound bearing two phosphino groups.* © 2011 Wiley Periodicals, Inc. Heteroatom Chem 22:301–306, 2011; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.20680

INTRODUCTION

Iminophosphoranes are useful both as reagents for aza-Wittig reactions and as ligands for transition metal complexes [1]. They are usually prepared by the Staudinger reaction [2] of an azide with a phosphine or a phosphite. Considering the explosive potential of several azide reagents and sodium azide, a precursor of many organic azides, other nitrogen-containing precursors are desired for the synthesis of iminophosphoranes. Azobenzene is a stable N=N double-bond compound and is considered as a candidate for such nitrogen-containing precursors because its combination with 2 equiv of phosphine is expected to form two iminophosphoranes. However, azobenzene has rarely been converted to an iminophosphorane because bond fission of the N=N double bond by a phosphorus reagent is difficult [3]. In contrast, we previously reported the synthesis of 2-phosphinoazobenzene **1** and its unique properties and reactivity based on an equilibrium with inner phosphonium salt **2** [4] that corresponds to the betaine intermediate of the Mitsunobu reaction (Scheme 1) [5]. The formation of **2** encouraged us to study reactivities of an azobenzene bearing two phosphino groups; in particular, its double P–N bond formation reaction via the N=N bond fission reaction. We report here the synthesis of 2,2′-bis(phosphino)azobenzene, and its reactivity to form a cyclic bis(iminophosphorane).

RESULTS AND DISCUSSION

4,4′-Dibutyl-2,2′-diiodoazobenzene (**3**) [6] was lithiated with butyllithium (2.2 equiv), and then the reaction mixture was treated with diphenylthiophosphinoyl chloride (2.3 equiv) to give the corresponding 2,2′-bis(diphenylthiophosphinoyl)azobenzene **4**

Dedicated to Prof. Kin-ya Akiba on the occasion of his 75th birthday.

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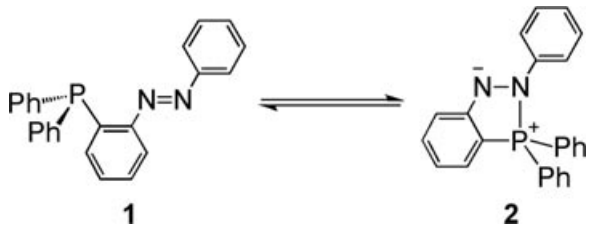


1 **2**

SCHEME 1

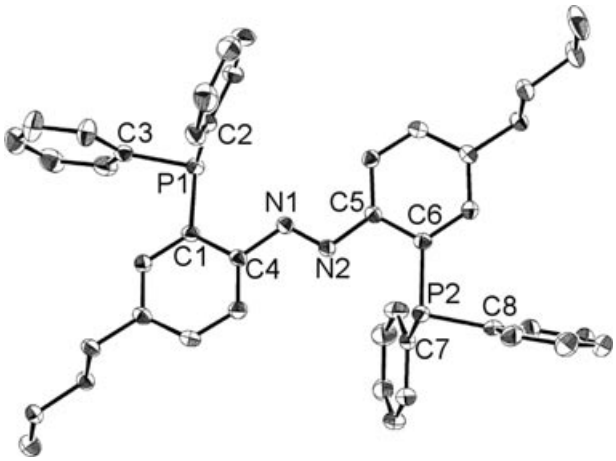


FIGURE 1 ORTEP drawing (50% probability) of **5**. Hydrogen atoms are omitted for clarity. Selected bond lengths (Å), angles (°), and torsion angles (°): P1—C1, 1.8419(15); P1—C2, 1.8353(18); P1—C3, 1.8309(16); N1—N2, 1.2556(16); P2—C6, 1.8437(15); P2—C7, 1.8292 (17); P2—C8, 1.8342(15); C6—P2—C7, 103.35(6); C6—P2—C8, 100.38(7); C7—P2—C8, 101.79(7); C1—C4—N1—N2, 166.06(11); C4—N1—N2—C5, 176.97(10) ; N1—N2—C5—C6, 178.88(11).

(72%) (Scheme 2). Desulfurization of **4** with hexamethylphosphorous triamide (excess) in toluene at 100°C in a sealed tube and successive recrystallization from toluene/hexane gave red crystals of 2,2′-bis(diphenylphosphino)azobenzene **5** (74%). X-ray crystallographic analysis of **5** revealed an almost C_i symmetric structure with a planar azobenzene moiety (Fig. 1). The C—P—C bond angles (100.38°–103.37°) and the N=N bond length (1.2556(16) Å)

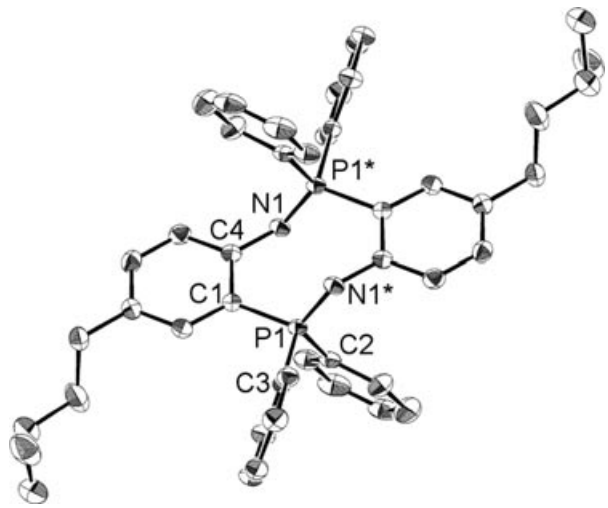
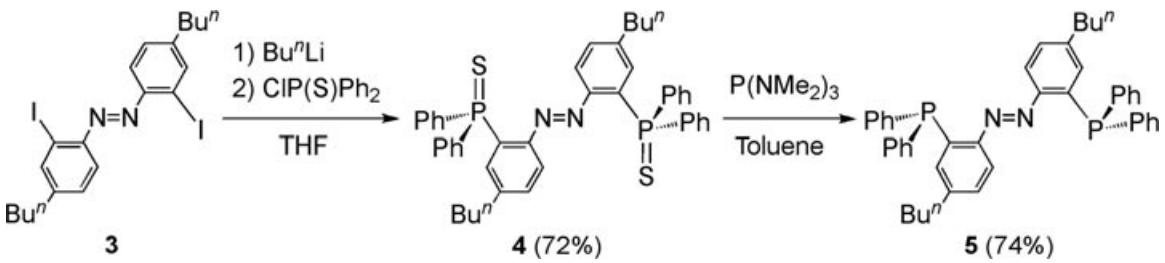


FIGURE 2 ORTEP drawing (50% probability) of **8**. Hydrogen atoms are omitted for clarity. Selected bond lengths (Å), angles (°), and torsion angles (°): P1—N1*, 1.5650(15); P1—C1, 1.8034(18); P1—C2, 1.8097(18); P1—C3, 1.8146(17); C4—N1, 1.394(2); N1*—P1—C1, 115.81(8); N1*—P1—C2, 115.96(8); N1*—P1—C3, 107.63(8); C1—P1—C2, 106.29(8); C1—P1—C3, 105.28(8); C2—P1—C3, 104.83(8); P1—C1—C4, 117.80(12); C1—C4—N1, 121.68(15); C4—N1—P1*, 128.82(12); C1—C4—N1—P1*, 83.30(19).

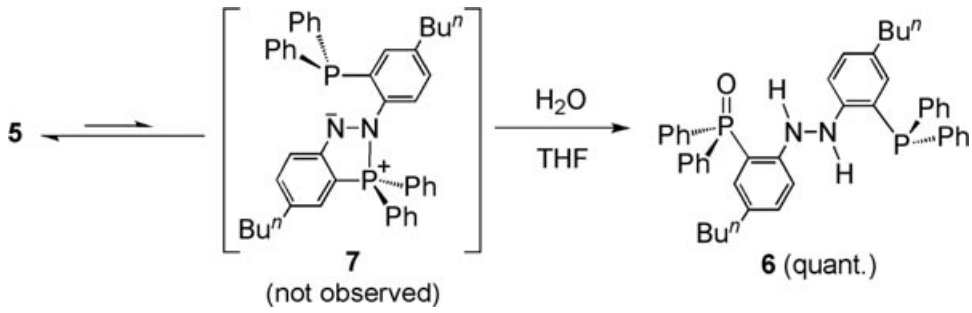
were almost the same as those of triphenylphosphine and azobenzene, respectively. These results show no interaction between the phosphorus atom and the azo group of **5** in the crystalline state.

Azobenzene **5** is moisture sensitive, and hydrolysis of **5** in THF gave hydrazobenzene **6** quantitatively (Scheme 3), indicating the existence of an equilibrium with **7** in the solution state, similarly to **1** [4]. Addition of pentachlorophenol (1.0 equiv) to a solution of **5** in toluene resulted in a color change from red of the azo group to pale yellow. After heating at 100°C for 6 h and the usual workup, cyclic iminophosphorane **8** was obtained in 84% yield as a colorless solid (Scheme 4). The structure of **8** was identified by ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy, MS, and X-ray analysis. The crystal structure of **8** is shown in Fig. 2. The molecular structure of **8** has a

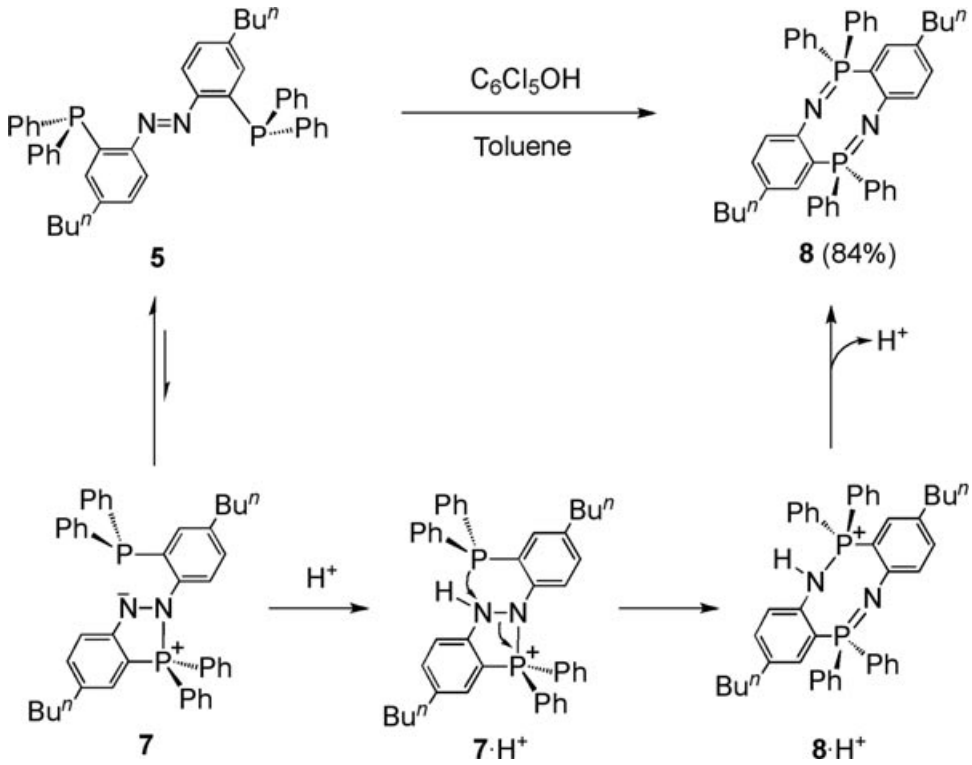


3 **4** (72%) **5** (74%)

SCHEME 2



SCHEME 3



SCHEME 4

center of symmetry (C_i symmetry). The interatomic N···N distance was 3.2 Å, clearly showing the N=N bond cleavage of the azo group. The short P—N distances 1.5650(15) Å are almost the same as the P—N bond lengths of usual iminophosphoranes [7] also implying P—N bond formation. The central eight-membered ring has a chair-like conformation. Despite C_i symmetry in the crystal structure, the four phenyl groups bound to the phosphorus atoms are equivalent in the ^{1}H and ^{13}C NMR spectra in C_6D_6 because of the fast conformational change of the eight-membered ring. While many iminophosphoranes such as $PhN{=}PPh_3$ usually undergo the aza-Wittig reaction with aldehydes to give the corresponding imine and phosphine oxide quantitatively, the reaction of **8** with benzaldehyde in C_6D_6 at 60°C for 48 h resulted in only little conversion (<10%), indicating the low reactivity of **8**.

To the best of our knowledge, there have been only a few reports of formation of an iminophosphorane by the reaction of a phosphine with an azo compound instead of azides [3,8]. Reactions of allenylphosphonates with dialkyl azodicarboxylate in the presence of triphenylphosphine were reported to give imonophosphoranes. Detailed mechanism is, however, not described there [8]. To investigate the reaction mechanism, the VT-^{31}P NMR spectra of the reaction solution of **5** after the addition of pentachlorophenol were measured and it was found that two singlets appeared at −15 and 48 ppm, which can

be assigned to the signals due to a phosphine and a phosphonium salt, respectively. Considering that protonation of **1** formed a nitrogen-protonated intermediate via equilibrium with **2** [4], these results indicate the formation of intermediate **7**·H^+ by protonation with pentachlorophenol. Attempted isolation of the intermediate **7**·H^+ failed because of its instability. Furthermore when no acid or the less acidic *p*-Bu^tC_6H_4OH was used, the signal due to the phosphonium salt was not observed at all, even at low temperature, indicating the presence of the acidity-dependent equilibration of **5** with **7**·H^+. In addition, heating **5** for 30 days under no acid or less acidic conditions resulted in no formation of **8**. These results indicate the formation of iminophosphorane **8** via **7**·H^+.

A plausible reaction mechanism is suggested as follows: first,**7**·H^+ is formed by the protonation of **7** equilibrated with **5**. Then the phosphine moiety of **7**·H^+ attacks the nitrogen atom of the NH group to form iminophosphorane **8**·H^+ with an aminophosphonium moiety via the N—N bond cleavage, which undergoes deprotonation to give **8** finally (Scheme 4). A similar intermolecular reaction of triphenylphosphine and the protonated phosphonium salt **2**·H^+ that was prepared from 2-phosphinoazobenzene **1** resulted in recovery of the starting materials. These results suggest that intramolecular nucleophilic attack of the phosphino group and formation of the P=N bonds are the driving forces in the present reaction.

In summary, we have shown the synthesis of 2,2′-bis(phosphino)azobenzene and its treatment with an acid to form the cyclic bis(iminophosphorane). Bond fission of the N=N double bond of the azobenzene derivative is an interesting approach for the synthesis of the iminophosphoranes although it is limited to this azobenzene at present. These results suggest the necessity of both an acid and an intramolecular nucleophile for N=N double bond cleavage via an inner phosphonium salt.

EXPERIMENTAL

General Procedure

All chemicals were reagent grade and used without further purification. Solvents were purified before use. All reactions were carried out under argon atmosphere unless otherwise noted. The ^{1}H NMR (500 MHz), ^{13}C NMR (126 MHz), and ^{31}P NMR (162 MHz) spectra were measured at 293 K with a JEOL A500 spectrometer using the signals of residual nondeuterated solvents and tributylphosphine ($\delta_P = -31.8$) as internal standards. Mass spectra were recorded with a JEOL JMX-SX 102 mass spectrometer. Elemental analyses were performed by the Microanalytical Laboratory of Department of Chemistry, Faculty of Science, The University of Tokyo. 2,2′-Diiodoazobenzene **3** was prepared according to a procedure mentioned in the literature [6].

Synthesis of 2,2′-Bis(diphenylthiophosphinoyl)-azobenzene **4**. To a THF solution (150 mL) of 2,2′-diiodoazobenzene **3** (1.00 g, 1.83 mmol) at −105°C, *n*-BuLi (1.6 M in hexane, 2.5 mL, 4.0 mmol) was added rapidly. After the reaction solution was stirred further at −105°C for 5 min, diphenylthiophosphinoyl chloride prepared from chlorodiphenylphosphine (0.80 mL, 4.20 mmol) and elemental sulfur (134 mg, 4.20 mmol) were added and the reaction mixture was stirred at room temperature for 3 h. After evaporation of the solvent, separation by silica-gel chromatography (eluent: $CHCl_3$) gave red crystals of **4** (958 mg, 72%).

4: ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, J = 7.5 Hz, 6H), 1.29 (sext, J = 7.5 Hz, 4H), 1.51 (quint, J = 7.5 Hz, 4H), 2.56 (t, J = 7.5 Hz, 4H), 6.41 (dd, J = 7.5 Hz, J = 5.0 Hz, 2H), 7.07 (d, J = 7.5 Hz, 2H), 7.30 (dd, J = 7.5 Hz, J = 3.0 Hz, 8H), 7.36–7.38 (m, 4H), 7.61 (dd, J = 16.0 Hz, J = 1.5 Hz, 2H), 7.72 (dd, J = 13.7 Hz, J = 7.5 Hz, 8H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 13.91 (s, CH_3), 22.27 (s, CH_2), 32.96 (s, CH_2), 35.49 (s, CH_2), 116.53 (d, J_{CP} = 7.2 Hz, *C*H), 128.12 (d, J_{CP} = 12.9 Hz, *C*H), 130.91 (d, J_{CP} = 2.4 Hz, *C*H), 131.72 (d, J_{CP} = 10.8 Hz, *C*H), 132.31 (d, J_{CP} = 80.3 Hz, *C*P), 132.73 (s, *C*H), 134.15 (d, J_{CP} = 88.1 Hz, *C*P), 134.25 (d, J_{CP} = 10.3 Hz, *C*H), 146.57 (d, J_{CP} = 10.8 Hz, *C*Bu^n), 149.85 (d, J_{CP} = 2.8 Hz, *C*N); ^{31}P{^{1}H} NMR (162 MHz, $CDCl_3$) δ 43.5 (s).

Synthesis of 2,2′-Bis(diphenyphosphino)azobenzene **5**. To a toluene solution (0.5 mL) of phosphine sulfide **4** (100 mg, 138 μmol), hexamethylphosphorous triamide (0.10 mL, 5.45 mmol) was added and the reaction solution was sealed. After the solution was heated at 100°C for 24 h, evaporation of the solvent and recrystallization of the residue from toluene/hexane gave red single crystals of **5** (67.7 mg, 74%).

5: ^{1}H NMR (500 MHz, C_6D_6) δ 0.75 (t, J = 7.5 Hz, 6H), 1.05 (sext, J = 7.5 Hz, 4H), 1.48–1.53 (m, 4H), 2.24 (t, J = 7.5 Hz, 4H), 6.88 (dd, J = 7.5 Hz, J = 4.8 Hz, 2H), 6.89 (s, 2H), 7.04–7.12 (m, 12H), 7.51 (td, J = 7.5 Hz, J = 1.5 Hz, 8H), 7.54 (dd, J = 7.5 Hz, J = 3.6 Hz, 2H); ^{13}C{^{1}H} NMR (126 MHz, C_6D_6) δ 13.99 (s, CH_3), 22.41 (s, CH_2), 33.30 (s, CH_2), 35.72 (s, CH_2), 116.61 (s, *C*H), 128.66 (d, J_{CP} = 14.9 Hz, *C*H), 128.72 (s, *C*H), 129.74 (s, *C*Bu^n), 133.17

(s, *C*H), 134.53 (d, J_{CP} = 20.6 Hz, *C*H), 138.80 (d, J_{CP} = 12.4 Hz, *C*P), 139.27 (d, J_{CP} = 17.3 Hz, *C*P), 146.21 (s, *C*H), 152.59 (d, J_{CP} = 14.0 Hz, *C*N); $^{31}P\{^1H\}$ NMR (162 MHz, C_6D_6) δ −12.2 (s).

Reaction of 2,2′-Bis(diphenyphosphino)azobenzene **5** *with Water.* To a THF solution of **5** (10 mg, 15 μmol), a 1:1 mixture of THF and water was added. After the reaction solution was stirred at room temperature for 1 min, the solvent was evaporated. Quantitative formation of colorless solid of **6** was confirmed by the ^1H and ^{31}P NMR spectra in $CDCl_3$.

6: ^1H NMR (500 MHz, $CDCl_3$) δ 0.80 (t, J = 7.5 Hz, 3H), 0.81 (t, J = 7.5 Hz, 3H), 1.15–1.22 (m, 4H), 1.29–1.38 (m, 4H), 2.32 (t, J = 7.5 Hz, 2H), 2.33 (t, J = 7.5 Hz, 2H), 6.36 (brs, 1H), 6.48 (dd, J = 8.0 Hz, J = 5.0 Hz, 1H), 6.54 (dd, J = 8.0 Hz, J = 1.5 Hz, 1H), 6.56 (d, J = 1.5 Hz, 1H), 6.70 (dd, J = 8.0 Hz, J = 5.0 Hz, 1H), 6.82 (dd, J = 8.0 Hz, J = 1.5 Hz, 1H), 7.00 (d, J = 8.0 Hz, 1H), 7.29–7.36 (m, 10H), 7.44–7.52 (m, 4H), 7.53–7.61 (m, 2H), 7.62–7.68 (m, 4H), 8.22 (brs, 8H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 13.77 (s, CH_3), 13.80 (s, CH_3), 21.83 (s, CH_2), 21.89 (s, CH_2), 33.34 (s, CH_2), 33.41 (s, CH_2), 34.22 (s, CH_2), 34.42 (s, CH_2), 111.14 (d, J_{CP} = 103.7 Hz, *C*P), 111.34 (d, J_{CP} = 3.3 Hz, *C*H), 112.14 (d, J_{CP} = 8.3 Hz, *C*H), 117.88 (d, J_{CP} = 10.8 Hz, *C*P), 128.26 (d, J_{CP} = 11.6 Hz, *C*H), 128.32 (d, J_{CP} = 6.6 Hz, *C*H), 128.55 (s, *C*H), 130.35 (s, *C*H), 131.28 (d, J_{CP} = 12.4 Hz, *C*H), 131.75 (d, J_{CP} = 2.5 Hz, *C*H), 131.88 (d, J_{CP} = 10.0 Hz, *C*H), 132.07 (d, J_{CP} = 103.7 Hz, *C*P), 132.24 (d, J_{CP} = 10.8 Hz, *C*H), 133.20 (d, J_{CP} = 2.5 Hz, *C*H), 133.37 (s, *C*H), 133.46 (d, J_{CP} = 3.3 Hz, *C*H), 133.56 (s, *C*H), 135.11 (d, J_{CP} = 8.3 Hz, *C*Bu^n), 148.63 (d, J_{CP} = 13.5 Hz, *C*Bu^n), 151.91 (s, *C*N), 151.94 (d, J_{CP} = 3.8 Hz, CN); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$) δ −20.9 (s), 36.0 (s). HRMS (FAB) m/z calcd for $C_{44}H_{46}N_2OP_2$ $[M]^+$ 680.3085, found: 680.3080.

Synthesis of Cyclic Bis(iminophosphorane) **8**. To a toluene solution (0.5 mL) of phosphine **5** (12.6 mg, 19 μmol), pentachlorophenol (5.3 mg, 20 μmol) was added and the reaction solution was sealed. After the solution was heated at 100°C for 6 h, evaporation of the solvent followed by separation using GPC (eluent: chloroform) gave colorless crystals of **8** (10.8 mg, 84%).

8: ^1H NMR (500 MHz, $CDCl_3$) δ 0.80 (t, J = 7.5 Hz, 6H), 1.22 (sext, J = 7.5 Hz, 4H), 1.35 (quint, J = 7.5 Hz, 4H), 2.30 (t, J = 7.5 Hz, 4H), 6.43 (d, J = 13.5 Hz, 2H), 7.16 (s, 4H), 7.21–7.29 (m, 6H), 7.35–7.40 (m, 14H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 13.82 (s, CH_3), 22.08 (s, CH_2), 33.18 (s, CH_2), 34.53 (s, CH_2), 121.14 (d, J_{CP} = 87.2 Hz, *C*P), 127.92–128.01 (m, *C*H), 128.35–128.51 (m, *C*H), 130.16 (s, *C*H), 131.63–131.70 (m, *C*H), 131.88–131.97 (m, *C*Bu^n), 133.40 (s, *C*H), 133.62 (d, J_{CP} = 114.8 Hz, *C*P), 134.51–134.56 (m, *C*H), 156.45 (s, *C*N); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$) δ 9.9 (s). HRMS (FAB) m/z calcd for $C_{44}H_{44}N_2P_2$ $[M]^+$ 662.2980, found: 662.3000. Elemental analysis calc. for $C_{44}H_{44}N_2P_2 \cdot H_2O$: C, 77.63; H, 6.81; N, 4.11. Found: C, 77.24; H, 6.69; N, 3.80%.

Treatment of 2,2′-Bis(diphenylphosphino) azobenzene **5** *with Phenols.* To a solution of (*E*)-**6** (12.6 mg, 20 μmol) in toluene (0.5 mL), pentachlorophenol or 4-*tert*-butylphenol (3.1 mg, 20 μmol) was added and the VT-^{31}P NMR spectra of the reaction mixture were measured.

X-ray Crystallographic Analysis

X-ray diffraction data were collected on a Rigaku Mercury CCD diffractometer with graphite-monochromated Mo Kα adiation. The structures of **5** and **8** were solved by direct methods (SIR97) and expanded using Fourier techniques [9]. The non-hydrogen atoms were refined anisotropically, and hydrogen atoms were refined isotropically with SHELX-97 [10]. Crystal data for **5**: $C_{44}H_{44}N_2P_2$, triclinic, $P\bar{1}$, a = 12.699(6), b = 12.795(6), c = 12.953(6) Å, α = 104.787(4), β = 94.500(4), γ = 113.723(4)° V = 1823.7(16) Å^3, MW = 662.75, Z = 2, D = 1.207 g/cm^3, 11556 measured, 6224 independent, GOF = 1.041, $R_1(I > 2\sigma(I))$ = 0.0335, wR_2(all data) = 0.0921. Crystal data for **8**, monoclinic, $P2_1/n$, a = 10.999(4), b = 8.703(3), c = 18.993(8) Å, β = 93.5044(13)°, V = 1814.6(12) Å^3, MW = 662.75, Z = 2, D = 1.213 g/cm^3, 11254 measured, 3191 independent, GOF = 1.064, $R_1(I > 2\sigma(I))$ = 0.0398, wR_2(all data) = 0.1130. CCDC 781725 (**5**) and 781726 (**8**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

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