# Reduction of Bis(phosphonio)isophosphindolides to Phosphane-Functionalized Benzo[*c*]phospholides

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*Dedicated to Professor Dr. Edgar Niecke on the occasion of his 60th birthday*

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Bis(phosphonio)benzo[*c*]phospholides (isophosphindolides) **1a**,**b** have been found to react with magnesium or alkali metal naphthalenides $MC_{10}H_8$ (M = Li, Na, K) with reduction of one or both of the phosphonio groups. The main products are the mono- or bis(phosphanyl)-substituted heterocycles **2**, **3**, **6**, **7**, which have been characterized by in situ NMR studies and in some cases isolated. Subsequent reaction of **2** with excess $[Ni(CO)_4]$ gave the complex **4**, which has been characterized by X-ray diffractometry. Reactions of **1a** with alkali metals followed a more complicated course and gave a mixture of **2**, **3**, and the substitution product **8**, which was further reduced to the phosphanido-substituted benzo[*c*]phospholide **9**. The latter could be formed selectively by first converting **1a** into **8** by treatment with PhNa and then treating this with lithium. NMR data of the phosphane-substituted benzo[*c*]phospholides and the structural features of **4** are discussed.

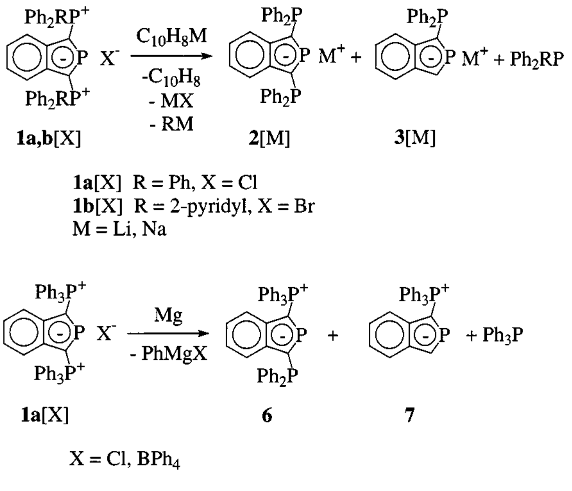
## Introduction

Phospholides[1] are hetero analogues of cyclopentadienide anions and are distinguished by both high aromatic character[2] and versatile ligand properties. The phospholide moiety is not only capable of acting as a 6e π-donor ligand similar to an η5-coordinated cyclopentadienide, but may also coordinate as a terminal 2e donor through the phosphorus lone pair, or, through a combination of σ and π coordination, as a bridging 4e or 8e ligand.[1] Whereas recent developments in the field of cyclopentadienides have been focused on derivatives bearing additional ligand groups attached either directly to the ring or on pendant side chains,[3] reports on analogous phospholide derivatives are scarce.[4][5] In view of the fact that a general route to tertiary phosphanes involves reductive cleavage of phosphonium salts,[6] we have examined the potential of reduction of bis(phosphonio)-substituted isophosphindolides incorporating a fused benzo[*c*]phospholide ring system.[7] We found that this reaction gives access to previously unknown[8] neutral and anionic benzo[*c*]phospholides bearing exocyclic phosphane functionalities.

## Results

### Reactions

Reactions of the bis(triphenylphosphonio)isophosphindolide salts **1a**[Cl][9] and **1b**[Br] (R = 2-pyridyl)[10] with four equivalents of lithium naphthalenide in THF were found to proceed with selective cleavage of one P−C bond in each exocyclic phosphonium moiety, thereby affording a mixture of the phosphane-substituted benzophospholide anions **2** and **3**. The by-products produced included the cleavage products $C_6H_5Li$ or $C_5H_4NLi$ along with triphenylphosphane or diphenylpyridylphosphane, respectively, as well as some $LiPPh_2$ arising from further reduction of the latter (Scheme 1).



Scheme 1. X = Cl, Br, $BPh_4$; M = Li, Na; R = Ph (**1a**), 2-pyridyl (**1b**)

The anions **2**, **3** were generated in a ratio of approximately 3:1 almost irrespective of the reaction temperature. Mixtures of similar composition were also obtained in other solvents (DME, liq. $NH_3$) or when sodium naphthalenide was used as the reducing agent. No further intermediates could be detected; only mixtures of **2**, **3** and unreacted starting material were produced when the reactions were performed with less than four equivalents of the metal naphthalenide.

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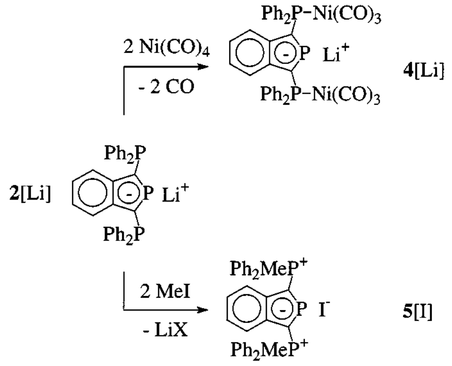
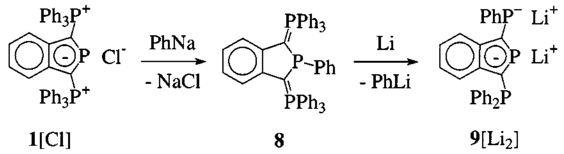
All reaction products were identified in situ by multinuclear 1D- and 2D-NMR studies (see Experimental Section). A salt **2**[Li(THF)$_x$] was isolated in crude form (approximately 95% pure on the basis of $^{31}$P- and $^{1}$H-NMR data) after quenching the remaining lithiated species with a stoichiometric amount of FeCl$_2$, filtering, and precipitation of the product with diethyl ether/hexane. Attempted purification of the product by recrystallization was unsuccessful, but a crystalline derivative could be obtained after reaction of either crude **2**[Li], or the reaction mixture containing both anions **2** and **3**, with excess Ni(CO)$_4$, followed by repeated recrystallization of the formed complex **4**[Li(THF)$_4$] from THF/diethyl ether. Likewise, reaction of crude **2**[Li] with excess MeI afforded the quaternization product **5**[I], which could be isolated in pure form after recrystallization from CH$_2$Cl$_2$/THF (Scheme 2).



Scheme 2

Only one phosphonium moiety was reduced in reactions of **1a**[X] (X = Cl, BPh$_4$) with magnesium. As in the case of the naphthalenide reduction, two main products were formed, which were identified by $^{31}$P-NMR spectroscopy as the neutral species **6** and **7** (Scheme 1; the generation of **7** was accompanied by the formation of Ph$_3$P). The exact reaction conditions and the product ratio varied with the specific physical state of the employed metal: The use of magnesium turnings required activation by treatment with I$_2$ or CCl$_4$ and produced some 25−50% of the cleavage product **7**; in contrast, application of a suspension of magnesium powder, freshly prepared by thermal decomposition of magnesium−anthracene,[11] resulted in shorter overall reaction times and a higher selectivity, giving up to 80% of the phosphane-substituted heterocycle **6**. Isolation and further spectroscopic characterization of this product was feasible after solvent extraction of the reaction mixture and recrystallization from toluene. The by-product **7** decomposed during the workup procedure and could not be further characterized.

A different and more complicated reaction pathway was observed when alkali metals were used instead of their naphthalenides as reducing agents. NMR-spectroscopic monitoring of the early stages of the reactions of **1a**[Cl] with Li or Na revealed, besides the presence of the anions **2**, **3** [and concomitant formation of the cleavage products C$_6$H$_5$Li(Na) and Ph$_3$P], the formation of the substitution product **8** (Scheme 3). The concentration of the latter decreased during the course of the reaction as a new species appeared, which was later identified as the phosphanide-substituted benzophospholide **9**[M$_2$] (M = Li, Na). In total, **2**, **3**, and **9** constituted approximately 90% of all the phosphorus-containing species; a higher percentage was prevented by unspecified side reactions and slow cleavage to give MPPh$_2$. Prolonged reaction times or elevated temperatures led to the decomposition of all other species, leaving the phosphanides MPPh$_2$ as the only well-defined phosphorus-containing reaction products.



Scheme 3

The formation of **8** by substitution of the cation **1a** by C$_6$H$_5$M (which was generated in the initial reduction of **1a** to give **2** and **3**) was proven by its independent synthesis from **1a**[Cl] and one equivalent of C$_6$H$_5$Na. Since a control experiment revealed that lithium reduction of an isolated sample of **2** under similar conditions did not yield any detectable amount of **9**[M$_2$], formation of this product can be assumed to occur exclusively through the intermediate **8**. This was confirmed by an investigation of the reaction of pure **8** with lithium under ultrasound sonification, which gave **9**[M$_2$] together with a small amount (10−15%) of LiPPh$_2$ as the only reaction products detectable by $^{31}$P-NMR spectroscopy. Even though all attempts to isolate **9**[M$_2$] from any of the reaction mixtures have hitherto been unsuccessful, it could be characterized in situ by multinuclear NMR spectroscopy (see below).

## NMR-Spectroscopic Investigations

The identities of the neutral and anionic benzophospholides **2**−**4**, **6**, **7**, and **9**, the benzophosphole **8**, and the cation **5** were unequivocally established from their $^{1}$H-, $^{13}$C-, and $^{31}$P-NMR data. Substitution patterns at the fused ring system were easily assigned from the habit of the $^{31}$P{$^{1}$H}-NMR spectra, which featured characteristic A$_2$X (**2**, **4**, **5**), A$_2$B (**8**), AMX (**6**, **9**), or AX patterns (**3**, **7**). Consequently, the $^{1}$H-NMR spectra displayed ABCD (**3**, **6**, **7**, **9**) or AA′BB′ patterns (**2**, **4**, **5**, **8**) for the four protons of the benzenoid fragment of the fused ring system. In addition, the spectra of **3** and **7** also featured a resonance for the 2-H proton, showing characteristic splitting due to coupling with the two phosphorus atoms. Discrimination between phosphonio (Ph$_3$P−), phosphane (Ph$_2$P−), and phosphanido [Ph(M)P−] substituents was feasible on the basis of characteristic $^{31}$P chemical shift differences (Table 1). Signals for the first two types of substituents appear in common spectral regions, while the resonance of the phosphanide group in **9** appears at much higher field than in simple diarylphosphanides [$\delta^{31}$P = −22.5, −36 for Ph$_2$P(Na, Li)[12]]. The resonances of the Ph(M)P and Ph$_2$P

Table 1. $^{31}$P- and selected $^{13}$C-NMR data of **2−9**

| | $\delta^{31}$P | $J_{\mathrm{P,P}}$ [Hz] | $\delta^{13}$C | $J_{\mathrm{P,C}}$ [Hz] |
|---|---|---|---|---|
| **2**[Li] | $-20.0$ ($\mathrm{P^APh_2}$)<br>216.4 ($-\mathrm{P^X}-$) | 130 | 124.4 ($\mathrm{P^X{-}C{-}P^A}$) | 51 ($J_{\mathrm{P(X),C}}$), 13 ($J_{\mathrm{P(A),C}}$), 10 ($J_{\mathrm{P(A'),C}}$) |
| **3**[Li] | $-21.9$ ($\mathrm{P^APh_2}$)<br>174.0 ($-\mathrm{P^X}-$) | 219 | –[a] | –[a] |
| **4**[Li] | 10.3 ($\mathrm{P^APh_2}$)<br>237.3 ($-\mathrm{P^X}-$) | 164 | –[a] | –[a] |
| **5**[I] | 12.5 ($\mathrm{P^APh_3}$)<br>229.0 ($-\mathrm{P^X}-$) | 92 | 109.4 ($\mathrm{P^X{-}C{-}P^A}$) | 54 ($J_{\mathrm{P(X),C}}$), 96 ($J_{\mathrm{P(A),C}}$), 14 ($J_{\mathrm{P(A'),C}}$) |
| **6** | $-21.4$ ($\mathrm{P^APh_2}$)<br>14.7 ($\mathrm{P^MPh_3}^+$)<br>218.4 ($-\mathrm{P^X}-$) | 76 ($J_{\mathrm{P(A),P(X)}}$)<br>82 ($J_{\mathrm{P(M),P(X)}}$) | 92.8 ($\mathrm{P^X{-}C{-}P^M}$)<br>143.2 ($\mathrm{P^X{-}C{-}P^A}$) | 99 ($J_{\mathrm{P(M),C}}$), 60 ($J_{\mathrm{P(X),C}}$), 10 ($J_{\mathrm{P(A),C}}$)<br>53 ($J_{\mathrm{P(M),C}}$), 14 ($J_{\mathrm{P(A),C}}$), 12 ($J_{\mathrm{P(X),C}}$) |
| **7** | 15.1 ($\mathrm{P^APh_3}$)<br>190.2 ($-\mathrm{P^X}-$) | 88 | –[a] | –[a] |
| **8** | 8.0 ($\mathrm{P^APh_3}^+$)<br>14.0 ($\mathrm{P^BPh}$) | 122 | 33.3 ($\mathrm{P^A{-}C{-}P^B}$) | 119.2 ($J_{\mathrm{P(A),C}}$), 11.4 ($J_{\mathrm{P(B),C}}$),<br>$-9.3$ ($J_{\mathrm{P(A)',C}}$)[b] |
| **9**[Li$_2$] | $-87.1$ ($\mathrm{P^APhLi}$)<br>$-20.8$ ($\mathrm{P^MPh_2}$)<br>194.2 ($-\mathrm{P^X}-$) | 108 ($J_{\mathrm{P(A),P(X)}}$)<br>61 ($J_{\mathrm{P(M),P(X)}}$) | 116.0 ($\mathrm{P^X{-}C{-}P^A}$)<br>139.2 ($\mathrm{P^X{-}C{-}P^M}$) | 52 ($J_{\mathrm{P(X),C}}$), 8 ($J_{\mathrm{P(A),C}}$), 5 ($J_{\mathrm{P(M),C}}$)<br>46 ($J_{\mathrm{P(X),C}}$), 30 ($J_{\mathrm{P(M),C}}$), 6 ($J_{\mathrm{P(A),C}}$) |
| **9**[Na$_2$] | $-86.2$ ($\mathrm{P^APhNa}$)<br>$-20.8$ ($\mathrm{P^MPh_2}$)<br>193.2 ($-\mathrm{P^X}-$) | 106 ($J_{\mathrm{P(A),P(X)}}$)<br>66 ($J_{\mathrm{P(M),P(X)}}$) | –[a] | –[a] |

[a] Not determined. – [b] Spectral analysis as $[\mathrm{A}]_2\mathrm{BX}$ system gave $J_{\mathrm{P(B),C}}/J_{\mathrm{P(B'),C}} < 0$ and $^4J_{\mathrm{P(B),P(B')}} = 5.5$ Hz.

fragments in **9** are further distinguished by different multiplicities in $^1$H non-decoupled $^{31}$P spectra, showing coupling to two or four *ortho* protons, respectively. The chemical shifts of the phospholide P atoms in **2**, **4**, **6**, and **9** are seen to be somewhat more shielded as in **1a** ($\delta^{31}\mathrm{P} = 241.8$[9]), but significantly less shielded than in monocyclic phospholides ($\delta^{31}\mathrm{P} = 55{-}142$[1b]). This trend could be confirmed by a comparison with calculated[13] $\delta^{31}$P values of model compounds (Table 2). This revealed that the deshielding of the resonance of **1a** (as well as of **2** and **4**) compared to those of the monocyclic phospholides can be attributed to similar contributions from both the benzanellation and the influence of the phosphonio (phosphane) substituents. Inspection of the results of calculations suggests that the deshielding shows a correlation with both a continuously decreasing energy gap between the phosphorus lone pair and the lowest $\pi^*$ orbital, and an increasing shift of $\pi$ electron density from the two-coordinate phosphorus atom to the adjacent carbon atoms, which tends to impose some "ylidic" character on the P−C bond.

Comparison of $\delta^{31}$P values for the corresponding nuclei in the lithium and sodium salts of the anions **2**, **3**, and **9** indicates some influence of the counterion, which is less pronounced for the monoanions **2**, **3** than for **9** and suggests some degree of ionic association (contact ion pair formation) in solution. Of diagnostic relevance in the $^{13}$C-NMR spectra are the signals of the ring carbon atoms adjacent to the two-coordinate phosphorus atoms: Carbon atoms bearing exocyclic phosphonio groups display similar chemical shifts and exocyclic $^1J$(P,C) couplings as seen in **1a**, whereas those bearing phosphanyl groups exhibit lower $^1J$(P,C) values and chemical shifts appear further downfield resembling those due to the α-carbon atoms in phospholides.[1]

Table 2. Calculated [GIAO-B3LYP/6−311+g(2d,d,p)//B3LYP/6−31+g(d,p)] $^{31}$P chemical shifts of phospholides and of the parent and 1,3-disubstituted benzo[*c*]phospholides[12]

| | $\delta^{31}$P |
|---|---|
| $[\mathrm{C_4H_4P}]^-$ | 139.4 |
| $[\mathrm{C_8H_6P}]^-$ (benzo[*c*]phospholide) | 200.5 |
| $[1,3\text{-}(\mathrm{H_2P})_2\text{-}\mathrm{C_8H_4P}]^-$ | 306.0 |
| $[1,3\text{-}(\mathrm{H_3P})_2\text{-}\mathrm{C_8H_4P}]^+$ | 311.5 |

## Crystal Structure of Compound 4

The results of an X-ray crystal structure analysis of **4**[Li(THF)$_4$] revealed a salt-like composition with two independent, only slightly different pairs of $[\mathrm{Li(THF)_4}]^+$ cations and benzophospholide anions per asymmetric unit (Figure 1). The bonding parameters in the fused ring systems of the anions do not differ significantly from those in the phosphonio-substituted cation **1a**.[14] On the other hand, the exocyclic P−C distances (1.774−1.789 Å) are longer than the corresponding bonds in **1a** (1.747−1.749 Å[14]) but shorter than the peripheral P−C(phenyl) distances (1.825−1.847 Å). The P−Ni distances in **4** are comparable with typical values in $\mathrm{PPh_3{-}Ni}$ complexes (2.225 ± 0.070 Å).[15] The remaining bond parameters display no peculiarities. On the whole, these findings suggest that electronic perturbation of the 10π system in **4** by hyperconjugation with the substituents[7] is lower than that in **1a**. In view of the rather low degree of electronic interaction between the five- and six-membered rings in a structurally characterized benzo[*b*]phospholide,[8] the anion **4** would seem to represent the first example of a benzophospholide with a unique 10π electron system having an electronic structure comparable to that found in heteronaphthalenic benzodithiaphospholium cations.[16]

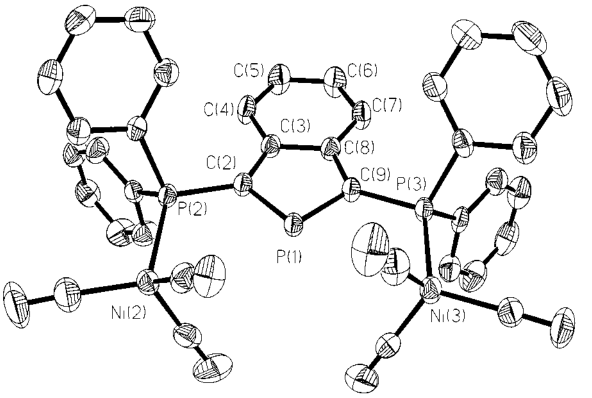


Figure 1. Representation of one of the two crystallographically independent anions in crystalline **4**[Li(THF)$_4$] (50% thermal ellipsoids); selected bond lengths [Å] and angles [°] (data of the second anion in brackets): P(1)−C(2) 1.732(4) [1.747(4)], P(1)−C(9) 1.736(4) [1.744(4)], C(2)−C(3) 1.441(5) [1.423(5)], C(2)−P(2) 1.789(4) [1.777(4)], C(3)−C(4) 1.416(5) [1.416(5)], C(3)−C(8) 1.432(5) [1.440(5)], C(4)−C(5) 1.367(5) [1.371(5)], C(5)−C(6) 1.399(6) [1.401(5)], C(6)−C(7) 1.372(5) [1.380(5)], C(7)−C(8) 1.413(5) [1.400(5)], C(8)−C(9) 1.440(5) [1.431(5)], C(9)−P(3) 1.780(4) [1.774(4)], P(2)−Ni(2) 2.241(1) [2.238(1)], P(3)−Ni(3) 2.241(1) [2.240(1)]; C(2)−P(1)−C(9) 93.0(2) [92.6(2)]

## Discussion

The reported reactions of the bis(phosphonio)isophosphindolide cations **1a**, **b** conform to the general scheme of the reduction of phosphonium salts through cleavage of P−C bonds to give phosphanes and carbanions. The latter may be protonated to give the corresponding hydrocarbons during workup.[6] For the bis(phosphonium) cations studied here, the extent of the reduction appears to be controlled by the reductive power of the metal: The reaction with magnesium stops after reduction of the first phosphonium functionality, while the stronger reducing agent alkali metal/naphthalene attacks both substituents. The failure to observe the neutral species **6**, **7** as intermediates under these conditions, which would be expected if a stepwise reaction is assumed, may be attributed to kinetic factors: The second reaction step may be faster, presumably due to the low solubility of the starting material in the solvents employed.

The formation of product mixtures in the observed reactions reflects the presence of different types of P−C single bonds: Starting from the cation **1a**, cleavage of a peripheral phenyl group affords the phosphane-substituted phospholide **6** and the corresponding phenylmetal compound, both of which were observed in the reaction mixtures, while fission of a P−C(ring) bond should liberate Ph$_3$P and an α-deprotonated benzophospholide anion. The latter was not directly detected and is most likely quenched by abstraction of a proton (presumably from a solvent molecule) to afford the neutral compound **7**, which could be isolated in the course of the reaction with magnesium. Further reduction of **6** and **7** with alkali metals can be expected to afford a mixture of **2** and **3**, together with Ph$_3$P and the parent benzophospholide anion. The latter was not observed, suggesting that the second reduction step occurs either with selective P−C(phenyl) bond fission, or that this product decomposes under the reaction conditions. If one considers that the ease of reductive P−C cleavage in phosphonium salts generally parallels the ability of the leaving group to stabilize a negative charge,[6] the observed product ratio in the reduction of **1a** suggests a slightly higher charge-stabilizing capacity of a triphenylphosphoniobenzophospholide as compared to a phenyl moiety, whereas selective cleavage of pyridyl instead of phenyl groups in the reduction of **1b** suggests that the pyridyl group is superior to both.

The outcome of the reduction of **1a** with alkali metals in the absence of naphthalene is consistently explained in terms of two competing reaction channels, viz. reduction of the cation **1a** and its substitution by the formed metal phenyl to give **8**. The successful competition of the two different pathways under these conditions is again attributable to kinetic factors: the reduction proceeds at a much lower rate when solid metals are used rather than soluble metal naphthalenides, so that the slower substitution of the formed phenylmetal compound with still present starting material to give **8** may take place. Further conversion of **8** to the dimetallated species **9**[M$_2$] requires the reductive cleavage of phenyl groups from both the two exocyclic ylide moieties and the endocyclic phosphorus atom. Both types of reactions are known in principle. Reductive cleavage of triphenylphosphonium ylides has been reported, and even though this process is not fully understood, it is considered to follow a different mechanism than the reduction of phosphonium salts,[17] thus accounting for the different nature of the obtained product. Reductive cleavage of P−Ph bonds in phospholes has been extensively studied, in particular by Mathey and co-workers.[1] Since no further intermediates were identified, the exact mechanism of the reaction from **8** to **9** cannot be further elucidated. Nonetheless, it appears reasonable to assume reduction of one of the phosphonium moieties as the first step.

## Conclusion

Our investigations have shown that bis(phosphonio)isophosphindolides can be reduced with preservation of the fused aromatic ring system to give phosphane-substituted benzo[*c*]phospholides. Despite the formation of mixtures of products, which arise from the presence of different P−C bonds in the starting materials that may possibly be cleaved, the novel phosphane-substituted benzophospholide **6** and the anionic nickel complex **4** could be isolated. Further investigations into the syntheses and reactivities of other phosphane-substituted (poly)phospholide derivatives, in particular with respect to a side-on π coordination to transition metal ions, are currently in progress.

## Experimental Section

**General Remarks:** NMR spectra were recorded at 30 °C with a Bruker AMX 300 spectrometer (300.13, 121.50, and 75.46 MHz for $^{1}$H, $^{31}$P, and $^{13}$C). Chemical shifts are referenced to external TMS ($^{1}$H, $^{13}$C) or 85% H$_3$PO$_4$. $^{13}$C,$^{31}$P{$^{1}$H}INEPT spectra were recorded with a 5-mm triple-resonance probe head equipped with an inner tunable coil adjusted to $^{13}$C and a second coil double-tuned to $^{1}$H and $^{31}$P. The spectra of **3** and **6** were recorded directly from the reaction mixtures with suppression of the solvent signals by presaturation. Signal assignments were made with the aid of (semi)selective 1D $^{13}$C,$^{31}$P{$^{1}$H}-INEPT and 2D $^{1}$H,$^{1}$H-COSY/TOCSY, $^{1}$H,$^{13}$C-HMQC, and $^{1}$H,$^{31}$P-HMQC-TOCSY spectra. The atoms of the benzophospholide moiety are denoted by C-1 to C-7a and 1-H to 7-H, the carbon atoms of the phenyl groups by C$_{para}$, C$_{meta}$ etc., and the designation of the phosphorus nuclei is as given in Table 1. Resonances in the $^{13}$C spectra forming part of [A]$_2$MY or [A]$_2$Y spin systems (A, M = $^{31}$P) were in some cases analyzed using the program WINDAISY; in the remaining cases the given values of $\Sigma J$ and $J_{\mathrm{M}}$ denote the sum of the couplings to the isochronous $^{31}$P nuclei of the [A]$_2$ fragments and the coupling to the endocyclic phosphorus atom, respectively. − IR spectra: Nicolet Magna 550 FT-IR spectrometer.

**Reduction of 1a[Cl] with Lithium Naphthalenide:** A freshly prepared solution of lithium napththalenide [obtained from 180 mg (30 mmol) of Li and 3.84 g (30 mmol) of $C_{10}H_8$] in 150 mL of THF was slowly added to a stirred suspension of **1a**[Cl] (5.45 g, 7.5 mmol) in 50 mL of THF at 0°C. Stirring was continued for 1 h. Anions **2**, **3**, $Ph_3P$, and PhLi were identified in situ in the dark-red mixture by multinuclear NMR analyses. A stoichiometric amount of $FeCl_2$ was then added to quench the PhLi, and the resulting mixture was stirred for a further 2 h. After addition of 10 mL of $Et_2O$, stirring was continued for another 30 min, then the mixture was filtered. The filtrate was concentrated in vacuo to a volume of 15 mL and 100 mL of a $Et_2O$/hexane (2:1) mixture was added. The resulting precipitate was collected by filtration, redissolved in a little THF, and reprecipitated with $Et_2O$/hexane. $^{31}$P- and $^{1}$H-NMR analysis revealed that the product consisted of approximately 95% **2**[Li(THF)$_x$]; yield 3.00 g (50%). The same protocol was used for the reduction of **1a**[Cl] with $C_{10}H_8Li$ and for that of **1a**,**b**[Cl] with $C_{10}H_8Na$. − **2**[Li]: $^{1}$H NMR (THF): $\delta$ = 7.53 (m, 8 H, H$_{ortho}$), 7.45 (m, 2 H, 4-H/7-H), 7.08 (m, 8 H, H$_{meta}$), 7.04 (m, 4 H, H$_{para}$), 6.49 (m, 2 H, 5-H/6-H). − $^{13}$C{$^{1}$H}NMR (THF): $\delta$ = 145.2 (m, $\Sigma J$ = 18.2 Hz, $J_{\mathrm{M}}$ = 2.2 Hz, C$_{ipso}$), 144.1 (m, $\Sigma J$ = 15.1 Hz, $J_{\mathrm{M}}$ = 7.6 Hz, C-3a/C-7a), 132.9 (m, $^{3}J_{\mathrm{P(A),C}}$ = 18.7 Hz, $^{5}J_{\mathrm{P(M),C}}$ = 1 Hz, C$_{meta}$), 126.9 (m, spectral analysis gave $^{4}J_{\mathrm{P(A),P(A)'}}$ = 1.8 Hz, $^{2}J_{\mathrm{P(A),C}}$ = 5.5 Hz, C$_{ortho}$), 125.6 (s, C$_{para}$), 124.4 (ddd, $^{1}J_{\mathrm{P(M),C}}$ = 51.0 Hz, $J_{\mathrm{P(A)/A',C}}$ = 13, 10 Hz, C-1/C-3), 119.4 (m, $\Sigma J$ = 10 Hz, $J_{\mathrm{M}}$ = 6.8 Hz, C-4/C-7), 115.1 (s, C-5/C-6). − **3**[Li]: $^{1}$H NMR (THF): $\delta$ = 7.58 (dd, $^{2}J_{\mathrm{H,P(X)}}$ = 39.3 Hz, $^{4}J_{\mathrm{H,P(A)}}$ = 2.9 Hz, 1 H, 3-H), 7.50 (m, 4 H, H$_{meta}$), 7.47 (m, 1 H, 4-H), 7.34 (m, 1 H, 7-H), 7.07 (m, 4 H, H$_{ortho}$), 7.02 (m, 2 H, H$_{para}$), 6.52 (m, 1 H, 6-H), 6.40 (m, 1 H, 5-H).

**Reduction of 1b[Br] with Lithium Naphthalenide:** The reduction of **1b**[Br] (1.94 g, 2.5 mmol) with lithium naphthalenide [obtained from 60 mg (10 mmol) of Li and 1.28 g (10 mmol) of $C_{10}H_8$] was carried out under analogous conditions as described above. Anions **2**, **3**, (2-py)$Ph_2P$, and (2-py)Li were identified in situ in the dark-red solution by $^{31}$P- and $^{1}$H-NMR analyses. No further workup of the reaction mixture was attempted.

**Nickel Complex 4[Li(THF)$_4$]**: A solution of 1.00 g (1.25 mmol) of crude **2**[Li(THF)$_x$] in 10 mL of THF was added dropwise with stirring to a freshly prepared solution of 1.2 mL of $Ni(CO)_4$ in 10 mL of THF. After stirring for a further 2 h, the mixture was concentrated to a volume of 5 mL, treated with 5 mL of $Et_2O$, and filtered. Storage of the filtrate at 2°C for several weeks led to the deposition of a colorless precipitate, which was recrystallized once from THF to give 540 mg (40%) of complex **4**[Li(THF)$_4$], m.p. 150°C (dec.), as colorless cubic crystals. − $^{1}$H NMR (CDCl$_3$): $\delta$ = 7.56 [m, 8 H, H$_{meta}$(PPh$_2$)], 7.20−7.05 [m, 12 H, H$_{ortho/para}$(PPh$_2$)], 6.96 (m, 2 H, 4-H/7-H), 6.28 (m, 2 H, 5-H/6-H). − IR (THF): $\tilde{\nu}$ (CO) = 2056 (s), 1982 (vs), 1972 (vs) cm$^{-1}$.

**Bis(methyldiphenylphosphonio)isophosphindolide Iodide (5[I]):** A solution of 0.50 g (3.5 mmol) of methyl iodide in 5 mL of THF was added dropwise to a solution of 1.00 g (1.25 mmol) of crude **2**[Li(THF)$_x$] in 10 mL of THF. The mixture was refluxed for 10 min, allowed to cool to room temperature, and then concentrated to dryness. The residue was extracted with 10 mL of $CH_2Cl_2$, the extract was filtered, and the filtrate was concentrated to a volume of 5 mL. Addition of 15 mL of THF led to the deposition of 620 mg (63%) of an off-white precipitate of **5**[I], m.p. 185°C, which was collected by filtration and dried in vacuo. − $^{1}$H NMR (CDCl$_3$): $\delta$ = 7.75−7.53 (m, 20 H, $C_6H_5$), 7.08 (m, 2 H) and 6.93 (m, 2 H, $C_6H_4$), 2.77 (dd, 6 H, $^{2}J_{\mathrm{H,P}}$ = 12.8 Hz, $^{4}J_{\mathrm{H,P}}$ = 1.4 Hz, CH$_3$). − $^{13}$C{$^{1}$H}NMR (CH$_2$Cl$_2$): $\delta$ = 143.5 (m, $\Sigma J$ = 25.4 Hz, $J_{\mathrm{M}}$ = 2.2 Hz, C-3a/C-7a), 133.7 (s, C$_{para}$), 132.4 (m, $\Sigma J$ = 10.7 Hz, C$_{ortho}$), 129.4 (m, $\Sigma J$ = 13.1 Hz, C$_{meta}$), 122.8 (dd, $J_{\mathrm{P(A),C}}$ = 89.0 Hz, $J_{\mathrm{P(X),C}}$ = 2.9 Hz, C$_{ipso}$), 120.8 (s) and 119.8 (m) (C-4/C-7 and C-5/C-6), 109.4 (ddd, $J_{\mathrm{P,C}}$ = 95.5, 54.3 and 14.3 Hz, C-1/C-3), 12.6 (dd, $J_{\mathrm{P(A),C}}$ = 61.2 Hz, $J_{\mathrm{P(X),C}}$ = 7.9 Hz, CH$_3$). − $C_{34}H_{30}I_2P_3$ (785.34): calcd. C 52.00, H 3.85; found C 51.5, H 3.8.

**Reduction of 1a[Cl] with Magnesium:** To a stirred suspension of 3.0 g (12.3 mmol) of Mg powder (freshly prepared by decomposition of $C_{14}H_{10}Mg$[11]) in 150 mL of THF was added 6.91 g (10 mmol) of **1a**[Cl]. Once the reaction had commenced, stirring was continued overnight. $^{31}$P-NMR-spectroscopic assay of the dark-red mixture confirmed the formation of **6** (ca. 80%) and **7**/Ph$_3$P (ca. 20%). The mixture was subsequently treated with 40 mL of $Et_2O$, filtered, and the filtrate was concentrated in vacuo. The residue was suspended in 120 mL of DME/$Et_2O$ (5:1), stirred for 1 h, and then filtered. The volume of the filtrate was reduced to 40 mL, and the product was precipitated by addition of 100 mL of MeOH. Recrystallization from toluene afforded 3.35 g (58%) of **6** as a yellow powder, m.p. 150°C. − $^{1}$H NMR (CDCl$_3$): $\delta$ = 7.67 (m, 1 H, 4-H), 7.60 [m, 6 H, H$_{ortho}$(PPh$_3$)], 7.48 [m, 4 H, H$_{ortho}$(PPh$_2$)], 7.35−7.45 [m, 9 H, H$_{meta/para}$(PPh$_3$)], 7.15−7.25 [m, 6 H, H$_{meta/para}$(PPh$_2$)], 6.91 (m, 1 H, 7-H), 6.85 (m, 1 H, 6-H), 6.74 (m, 1 H, 5-H). − $^{13}$C{$^{1}$H}NMR (CDCl$_3$): $\delta$ = 146.3 (ddd, $^{3}J_{\mathrm{P(A),C}}$ = 17.2 Hz, $^{2}J_{\mathrm{P(M),C}}$ = 15.3 Hz, $^{2}J_{\mathrm{P(X),C}}$ = 6.5 Hz, C-7a), 144.3 (ddd, $^{2}J_{\mathrm{P(A),C}}$ = 10.2 Hz, $^{2}J_{\mathrm{P(X),C}}$ = 5.4 Hz, $^{3}J_{\mathrm{P(M),C}}$ = 1.2 Hz, C-3a), 143.2 (ddd, $^{1}J_{\mathrm{P(X),C}}$ = 53.4 Hz, $^{1}J_{\mathrm{P(A),C}}$ = 14.1 Hz, $^{3}J_{\mathrm{P(M),C}}$ = 11.9 Hz, C-3), 140.4 [dd, $^{1}J_{\mathrm{P(A),C}}$ = 10.7 Hz, $^{3}J_{\mathrm{P(X),C}}$ = 8.8 Hz, C$_{ipso}$(PPh$_2$)], 134.0 [dd, $^{2}J_{\mathrm{P(M),C}}$ = 10.3 Hz, $^{4}\mathrm{J}_{\mathrm{P(X),C}}$ = 1.1 Hz, C$_{ortho}$(PPh$_3$)], 133.3 [dd, $^{3}J_{\mathrm{P(A),C}}$ = 18.3 Hz, $^{5}J_{\mathrm{P(X),C}}$ = 1.2 Hz, C$_{meta}$(PPh$_2$)], 132.9 [d, $^{4}J_{\mathrm{P(M),C}}$ = 3.1 Hz, C$_{para}$(PPh$_3$)], 129.1 [d, $^{3}J_{\mathrm{P(M),C}}$ = 12.8 Hz, C$_{meta}$(PPh$_3$)], 127.8 [d, $^{2}J_{\mathrm{P(A),C}}$ = 6.9 Hz, C$_{ortho}$(PPh$_2$)], 127.4 [s, C$_{para}$(PPh$_2$)], 125.2 [dd, $^{1}J_{\mathrm{P(M),C}}$ = 89.7 Hz, $^{3}J_{\mathrm{P(X),C}}$ = 1.9 Hz, C$_{ipso}$(PPh$_3$)], 120.7 (dd, $^{3}J_{\mathrm{P(A),C}}$ = 12.1 Hz, $^{3}J_{\mathrm{P(X),C}}$ = 7.5 Hz, C-4), 120.0 (d, $J_{\mathrm{P,C}}$ = 4.2 Hz, C-7), 119.4 (d, $J_{\mathrm{P,C}}$ = 3.1 Hz, C-5), 118.3 (s, C-6), 92.8 (ddd, $^{1}J_{\mathrm{P(X),C}}$ = 98.8 Hz, $^{1}J_{\mathrm{P(M),C}}$ = 60.0 Hz, $^{3}J_{\mathrm{P(A),C}}$ = 10.1 Hz, C-1). − $C_{38}H_{29}P_3$ (578.57): calcd. C 78.89, H 5.05; found C 78.0, H 4.9.

**Reduction of 1a[Cl] with Lithium:** To a stirred suspension of **1a**[Cl] (1.45 g, 2 mmol) in 15 mL of THF was added approximately 60 mg (10 mmol) of freshly cut lithium wire. After 30 min, $^{31}$P-NMR-spectroscopic monitoring revealed, besides the presence of unreacted **1a**, the formation of **2**, **3**, Ph$_3$P, Ph$_2$PLi (from cleavage of Ph$_3$P), as well as of the substitution product **8**. Prolonged reaction times led to a decay of the signals due to **8**, while those due to a new product **9** intensified. Continuation of the reaction for several days, or heating, led to the disappearance of all resonances other than that due to $Ph_2PLi$. Similar results were obtained when the reduction of **1a**[Cl] was performed with sodium.

**Benzophosphole 8:** A freshly prepared solution of phenyllithium (26.6 mmol) in 100 mL of THF was added dropwise to a suspension of **1a**[Cl] (15.7 g, 21.7 mmol) in 80 mL of THF at 0°C. The mixture was stirred for 5 h at 0°C, then treated with 20 mL of $Et_2O$, and filtered. The filtrate was concentrated to dryness, the residue was refluxed for 1 h with 250 mL of toluene, and the hot solution was filtered. Slow cooling of the filtrate resulted in the deposition of a crystalline precipitate, which was collected by filtration and dried in vacuo to give 14.3 g (90%) of **8** as black needles; m.p. 160°C. – $^{1}H$ NMR ($CDCl_3$): $\delta$ = 7.55 [m, 12 H, $H_{meta}(PPh_3)$], 7.38 [m, 6 H, $H_{para}(PPh_3)$], 7.27 [m, 12 H, $H_{ortho}(PPh_3)$], 7.21–7.15 [m, 3 H, $H_{ortho/para}(PPh)$], 6.91 [m, 2 H, $H_{meta}(PPh)$], 6.08 (m, 2 H, 5-H/6-H), 5.94 (m, 2 H, 4-H/7-H). – $^{13}C\{^{1}H\}$ NMR ($CDCl_3$):[18] $\delta$ = 154.6 [m, $^{1}J_{P(B),C}$ = 58.7 Hz, $\Sigma J$ = 2.6 Hz, $C_{ipso}(PPh)$], 148.3 (m, $\Sigma J$ = 29 Hz, $^{2}J_{P(B),C}$ = 0.5 Hz, C-3a/C-7a), 133.9 [m, $^{2}J_{P(A),C}$ = 9.6 Hz, $^{4}J_{P(B),C}$ = 2.0 Hz, $C_{ortho}(PPh_3)$], 130.8 [m, $^{4}J_{P(A),C}$ = 2.3 Hz, $C_{para}(PPh_3)$], 129.7 [m, $^{1}J_{P(A),C}$ = 86.7 Hz, $^{3}J_{P(B),C}$ = +3.1 Hz, $C_{ipso}(PPh_3)$], 128.2 [d, $^{3}J_{P(B),C}$ = 6.9 Hz, $C_{meta}(PPh)$], 127.8 [m, $^{3}J_{P(A),C}$ = 11.1 Hz, $C_{meta}(PPh_3)$], 126.4 [d, $^{2}J_{P(B),C}$ = 4.6 Hz, $C_{ortho}(PPh)$], 125.7 [s, $C_{para}(PPh)$], 116.6 (m, $\Sigma J$ = 1.9 Hz, C-4/C-7), 116.3 (s, C-5/C-6), 33.3 (m, $^{1}J_{P(A),C}$ = 119.2 Hz, $^{3}J_{P(A)',C}$ = –9.3 Hz, $^{1}J_{P(B),C}$ = 11.4 Hz, C-1/C-3). – $C_{50}H_{39}P_3$ (732.78): calcd. C 81.95, H 5.36; found C 81.0, H 5.1.

**Reduction of Benzophosphole 8 with Lithium:** To a solution of 1.00 g (1.37 mmol) of **8** in 25 mL of THF was added an excess of lithium powder, and the resulting mixture was allowed to react for 30 min under sonification with ultrasound. $^{1}H$- and $^{31}P$-NMR-spectroscopic assays confirmed the formation of **9**[$Li_2$] (accounting for ca. 85–90% of the phosphorus-containing material), some $Ph_2PLi$ (ca. 10–15%), and phenyllithium. Characterization of the major product **9**[$Li_2$] was achieved in situ by multinuclear ($^{1}H$-, $^{13}C$-, $^{31}P$-)NMR analyses. All attempts to isolate and further purify the product were unsuccessful, invariably resulting in partial or complete decomposition. – **9**[$Li_2$]: $^{1}H$ NMR ($CDCl_3$): $\delta$ = 7.60 (m, 1 H, 6-H), 7.44 [m, 4 H, $H_{meta}(PPh_2)$], 7.38 (m, 1 H, 4-H/5-H), 7.22 (m, 1 H, 5-H/4-H), 7.13 [m, 2 H, $H_{meta}(PPh)$], 6.91 [m, 4 H, $H_{ortho}(PPh_2)$], 6.80 (m, 1 H, 7-H), 6.68 [m, 2 H, $H_{ortho}(PPh)$], 6.40 [m, 2 H, $H_{para}(PPh_2)$], 6.28 [m, 1 H, $H_{para}(PPh)$]. – $^{13}C\{^{1}H\}$NMR ($CDCl_3$): $\delta$ = 160.2 [dd, $^{1}J_{P(A),C}$ = 45.8 Hz, $^{3}J_{P(X),C}$ = 7.6 Hz, $C_{ipso}(PPh)$], 146.6 [dd, $^{1}J_{P(M),C}$ = 14.3 Hz, $^{3}J_{P(X),C}$ = 5.0 Hz, $C_{ipso}(PPh_2)$], 144.8 (dd, $^{1}J_{P(M)/A,C}$ = 15.0 Hz, $^{3}J_{P(X),C}$ = 7.2 Hz, C-3a/C-7a), 144.5 (dd, $^{1}J_{P(M)/A,C}$ = 7.5, 1 Hz, $^{3}J_{P(X),C}$ = 1 Hz, C-7a/C-3a), 139.2 (ddd, $^{1}J_{P(X),C}$ = 46.1 Hz, $^{1}J_{P(M),C}$ = 30.3, $^{3}J_{P(A),C}$ = 6.3 Hz, C-3), 133.6 [dd, $^{3}J_{P(M),C}$ = 18.7 Hz, $^{5}J_{P(X),C}$ = 1.5 Hz, $C_{meta}(PPh_2)$], 128.2 [dd, $^{3}J_{P(A),C}$ = 17.8 Hz, $^{5}J_{P(X),C}$ = 2.0 Hz, $C_{meta}(PPh)$], 126.9 [d, $^{3}J_{P(M),C}$ = 5.7 Hz, $C_{ortho}(PPh_2)$], 126.1 [d, $^{3}J_{P(M),C}$ = 5.7 Hz, $C_{ortho}(PPh)$], 120.6 (dd, $^{3}J_{P(A),C}$ = 7 Hz, $^{3}J_{P(X),C}$ = 6 Hz, C-7), 120.4 (dd, $^{4}J_{P(A)/M,C}$ = 4 Hz, $^{4}J_{P(X),C}$ = 6 Hz, C-4/C-5), 119.7 (ddd, $^{4}J_{P(A)/M,C}$ = 5.5, 1.7 Hz, $^{4}J_{P(X),C}$ = 12.0 Hz, C-5/C-4), 118.6 (dd, $^{3}J_{P(M),C}$ = 15.7 Hz, $^{4}J_{P(X),C}$ = 4.8 Hz, C-6), 116.5 [s, $C_{para}$ $(PPh_2)$], 116.0 [s, $C_{para}(PPh)$], 116.0 (ddd, $^{1}J_{P(X),C}$ = 52 Hz, $^{1}J_{P(A),C}$ = 8 Hz, $^{3}J_{P(M),C}$ = 5 Hz).

**X-ray Crystal Structure Determination of 4[Li(THF)$_4$]:** $C_{38}H_{24}Ni_2O_6P_3 \cdot Li(THF)_4 \cdot Et_2O$, colorless, $M_r$ = 1156.4; monoclinic, space group $P2_1/c$ (No. 14), $a$ = 32.3511(7) Å, $b$ = 13.8186(5) Å, $c$ = 29.4593(8) Å, $\beta$ = 116.262(2)°, $V$ = 11810.3(6) Å$^3$, $Z$ = 8, $\mu(\text{Mo-}K_\alpha)$ = 0.78 mm$^{-1}$, $T$ = 123(2) K, 68443 reflections measured ($2\theta_{max}$ = 50°), 19310 independent reflections used for structure solution (direct methods) and refinement (on $F^2$ anisotropic with SHELXL-93), 1348 parameters, 2915 restraints, H atoms as a riding model, $wR2(F^2)$ = 0.145, $R[F, I > 2\sigma(I)]$ = 0.049. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-101915. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, U.K. [Fax: (internat.) + 44-1223/336033; E-mail: deposit@ccdc.cam.ac.uk].

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