P−H-Functionalized Phosphanyl Alcohols: $RHPCH_2CHMeOH$ and 2-PHR-1-OH-cyclo-C_6H_{10} (R = Ph, 2,4,6-$Me_3C_6H_2$, 2,4,6-iPr$_3C_6H_2$) and Molecular Structures of (C_R,C_R,P_R/C_S,C_S,P_S)-2-PH(2,4,6-iPr$_3C_6H_2$)-1-OH-cyclo-C_6H_{10} and its Dilithio Salt $[Li_2(THF)_{0.5}\{(C_R,C_R/C_S,C_S)$-2-P(2,4,6-$iPr_3C_6H_2$)-1-O-cyclo-$C_6H_{10}\}]_4$

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The P–H-functionalized phosphanyl alcohols $RHPCH_2CHMeOH$ [R = Ph (**1**), 2,4,6-$Me_3C_6H_2$ (Mes) (**2**), 2,4,6-iPr$_3C_6H_2$ (Tipp) (**3**)] and 2-PHR-1-OH-cyclo-C_6H_{10} [(R = Ph (**4**), Mes (**5**), Tipp (**6**)] have been prepared by ring-opening of propene oxide or cyclohexene oxide, respectively, with the appropriate LiPHR species, followed by hydrolytic workup and distillation (**1**–**4**) or recrystallization (**5**, **6**). **1**–**6**, which were obtained as diastereomeric mixtures, have been characterized spectroscopically (^1H, ^{13}C, ^{31}P NMR, IR, MS). Only in the case of **6** could separation of the diastereomers be achieved by fractional crystallization and the molecular structure of the racemic isomer (C_R,C_R,P_R/C_S,C_S,P_S) has been determined. In situ double deprotonation of **1**–**6** with BuLi gave the corresponding dianions **1a**–**6a**. The molecular structure of the dilithio salt $[Li_2(THF)_{0.5}\{(C_R,C_R/C_S,C_S)$-2-P(2,4,6-$iPr_3C_6H_2$)-1-O-cyclo-$C_6H_{10}\}]_4$ (**6a**) has been determined and shows that it is tetrameric in the solid state with a central Li_8 cluster.

Introduction

While tertiary phosphanyl alcohols are employed as ligands in catalytically active transition metal complexes,[1] the corresponding secondary P−H-functionalized phosphanyl alcohols have received only little attention. As early as 1965, Issleib et al. reported the ring-opening of oxiranes by alkali metal phosphanides, which, after hydrolytic workup, gave tertiary or secondary phosphanyl alcohols. The products were, however, only characterized by elemental analysis and IR spectroscopy.[2] In 1991, Heinicke et al. reported the formation of the primary phosphanyl alcohols $H_2PCH_2CHR'OH$ (R′ = H, Me, Ph) by ring-opening of ethene, propene, or styrene oxides with sodium phosphanide, $NaPH_2$.[3] In 1997, Thiel et al. used the stereoselective ring-opening of cyclohexene oxide by lithium diphenylphosphanide to prepare chiral phosphanyl alcohols.[4]

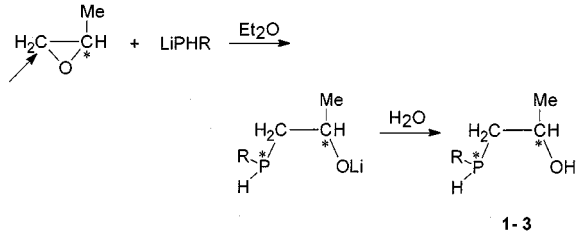
Primary[3,5] and secondary[6,7] phosphanyl alcohols have hitherto been used for the preparation of organic P heterocycles. Their ligation properties are, however, largely unstudied.

We now report the synthesis and spectroscopic characterization of the P−H-functionalized phosphanyl alcohols $RHPCH_2CHMeOH$ [R = Ph (**1**), 2,4,6-$Me_3C_6H_2$ (Mes) (**2**), 2,4,6-iPr$_3C_6H_2$ (Tipp) (**3**)] and 2-PHR-1-OH-cyclo-C_6H_{10} [(R = Ph (**4**), Mes (**5**), Tipp (**6**)], as well as the molecular structures of the diastereoisomerically pure phosphanyl alcohol (C_S,C_S,P_S/C_R,C_R,P_R)-2-PH(2,4,6-iPr$_3C_6H_2$)-1-OH-cyclo-C_6H_{10} (**6**) and its dilithio salt $[Li_2(THF)_{0.5}(C_S,C_S/C_R,C_R)$-2-P(2,4,6-$iPr_3C_6H_2$)-1-O-cyclo-$C_6H_{10}]_4$ (**6a**). Syntheses of **1**[2,7] and **4**[2] have been reported previously, but both compounds were only characterized by elemental analysis and IR spectroscopy.[2] Although a ^1H NMR spectrum has been reported for **1**, no diastereomers were observed.[7]

Results and Discussion

Synthesis of $RHPCH_2CHMeOH$ [R = Ph (1), 2,4,6-$Me_3C_6H_2$ (Mes) (2), 2,4,6-iPr$_3C_6H_2$ (Tipp) (3)] and 2-PHR-1-OH-cyclo-C_6H_{10} [(R = Ph (4), Mes (5), Tipp (6)] and their Dilithio Salts

The P−H-functionalized phosphanyl alcohols $RHPCH_2CHMeOH$ [R = Ph (**1**),[2,7] Mes (**2**), Tipp (**3**)] were obtained by ring-opening of propene oxide with LiPHR, followed by hydrolytic workup and distillation [Equation (1)].



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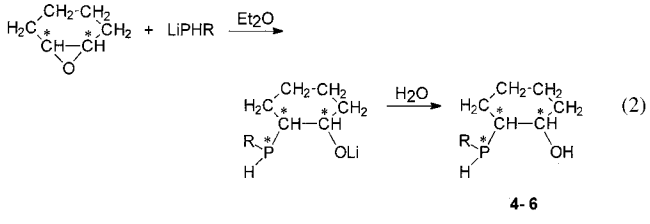
As in base-catalyzed ring-opening reactions, one C−O bond of the oxirane ring is selectively cleaved by the nucleophilic phosphanide anion, which attacks the sterically less hindered C atom.

As **1**−**3** have two chiral centres, two pairs of diastereomers are formed [*meso* (C_S,P_R/C_R,P_S) and *rac* (C_S,P_S/C_R,P_R)], which are obtained in a 1:1 ratio. Subsequent distillation furnished **1**−**3** as colourless, viscous oils. **3** was found to solidify at room temperature (m.p. 35 °C). The diastereomers could not be separated either by distillation or by fractional crystallization.

In their proton-coupled ^{31}P NMR spectra, **1**−**3** give rise to two doublets due to the two diastereomers. The spectrum of the Ph derivative **1** features a signal at low field compared to those of **2** and **3** [**1**: δ = −63.4 ($^1J_{P-H}$ = 210.6 Hz), −64.1 ($^1J_{P-H}$ = 210.6 Hz); **2**: δ = −97.7 (d, $^1J_{P-H}$ = 217.0 Hz), −98.9 (d, $^1J_{P-H}$ = 218.7 Hz); **3**: δ = −105.2 ($^1J_{P-H}$ = 215.5 Hz), −105.9 ($^1J_{P-H}$ = 217.1 Hz)].

Analogously to **1**−**3**, the phosphanyl alcohols 2-PHR-1-OH-cyclo-C_6H_{10} [(R = Ph (**4**),[2] Mes (**5**), Tipp (**6**)], which contain three chiral centres (C, C, P), could be prepared by selective ring opening of cyclohexene oxide with LiPHR, followed by hydrolytic workup [Equation (2)]. **4** was obtained as a colourless oil, whereas **5** and **6** were recovered as colourless solids. The diastereoselective ring-opening of the oxirane by the nucleophilic phosphanide anion yielded only two pairs of diastereomers (ratio 2:3 for **4** and **6**, 1:1 for **5**), which could not be separated by distillation. However, in the case of **6**, it was found that the diastereomers could be separated by fractional crystallization.

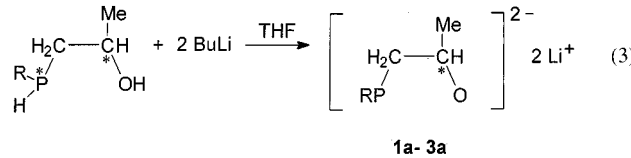


(2)

4-**6**

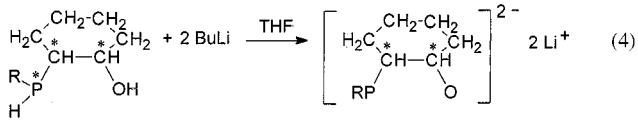
Compounds **4**−**6** give rise to two doublets in their proton-coupled ^{31}P NMR spectra due to the two pairs of diastereomers with C_S,C_S,P_R/C_R,C_R,P_S and C_S,C_S,P_S/C_R,C_R,P_R configurations. The signals of **5** (δ = −105.3 and −106.0) are shifted to high field relative to those of **4** (δ = −40.9 and −45.5) and **6** (δ = −77.6 and −89.9).

The bifunctionalized phosphanyl alcohols **1**−**6** could be doubly deprotonated with BuLi in THF to give the dianions Li_2[RPCH$_2$CHMeO] [R = Ph (**1a**), Mes (**2a**), Tipp (**3a**)] [Equation (3)] and Li_2[2-RP-1-O-cyclo-C_6H_{10}] [R = Ph (**4a**), Mes (**5a**), Tipp (**6a**)] [Equation (4)], which were characterized by ^{31}P NMR spectroscopy. The spectra of the dianions **2a**, **4a**, **5a**, and **6a** in THF feature two broad signals, indicating dynamic processes involving ligand exchange in solution. In the case of **6a**, this was further verified by a crystal structure determination.



(3)

1a-**3a**



(4)

4a-**6a**

Molecular Structures of (C_R,C_R,P_R/C_S,C_S,P_S)- 2-PH(2,4,6-*i*Pr$_3$C$_6$H$_2$)-1-OH-cyclo-C_6H_{10} (6) and Its Dilithio Salt [Li_2(THF)$_{0.5}${(C_R,C_R/C_S,C_S)-2-P(2,4,6-*i*Pr$_3$C$_6$H$_2$)-1-O-cyclo-C_6H_{10}}]$_4$ (6a)

Colourless crystals of **6** were obtained by repeated recrystallization from pentane. **6** crystallizes in the centrosymmetric monoclinic space group $P2_1/c$. Only the diastereomer with C_R,C_R,P_R/C_S,C_S,P_S configuration (OH and PHR in equatorial positions) was found to be present in the unit cell (Figure 1). A section of the three-dimensional structure shows that molecules of one enantiomer are stacked along the *b*-axis. Two such stacks are related by a 2_1 axis, and the molecules of these two stacks of the two enantiomers are connected through hydrogen (OH) bonds (Figure 2) resulting in a helical arrangement. The inversion centre generates the other enantiomer.

The excellent ligation properties of the 2-P(2,4,6-*i*Pr$_3$C$_6$H$_2$)-1-O-cyclo-C_6H_{10} dianion are exemplified by the solid-state structure of **6a**. This salt could be obtained from pentane in the form of small, pale-yellow, very air-sensitive crystals of rather poor quality (monoclinic centrosymmetric space group $P2_1/c$). In the solid state, the compound forms tetrameric molecules composed of eight lithium atoms and four phosphanidoalkoxides. Two independent molecules are present in the asymmetric unit. In both of these, the eight lithium atoms form a polyhedron in which two trigonal

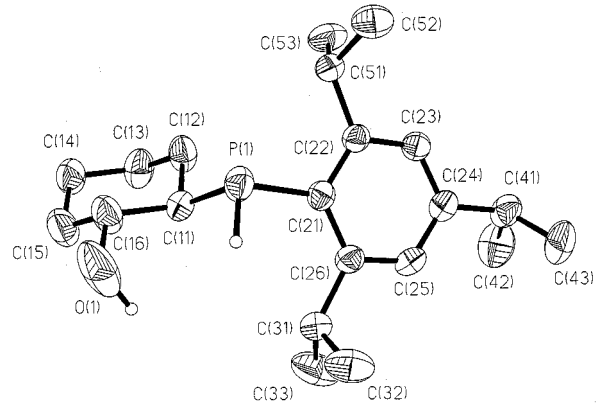


Figure 1. Molecular structure of **6**; only one enantiomer (C_R,C_R,P_R configuration) is shown; selected bond lengths [Å] and angles [°]: P(1)−C(21) 1.845(2), P(1)−C(11) 1.869(2), O(1)−C(16) 1.429(3), O(1)−HO(1) 1.19(4), P(1)−H(1) 1.27(2); C(21)−P(1)−C(11) 102.70(9), C(12)−C(11)−P(1) 110.9(2), C(16)−C(11)−P(1) 110.0(2), C(22)−C(21)−P(1) 118.3(1), C(26)−C(21)−P(1) 122.8(1), O(1)−C(16)−C(15) 111.0(2), O(1)−C(16)−C(11) 111.5(2)

prisms share one square face and are twisted by 90° (Figure 3). The Li−Li distances range from 2.47(2) to 2.83(2) Å. This polyhedron has four triangular faces and four square faces. The four phosphorus atoms of the phosphanidoalkoxide cover the triangular faces, while the four oxygen atoms of the ligand are located above the square faces in such a way that the oxygen and phosphorus atoms belonging to one phosphanidoalkoxide ligand are located on adjacent faces of the Li_8 polyhedron [P(1) and O(1), P(2) and O(2), P(3) and O(3), and P(4) and O(4) are located next to each other (Figure 4 and Figure 5); Li−O 1.962(16)−2.096(14) Å, Li−P 2.490(15)−2.699(16) Å for both molecules]. Thus, the four oxygen atoms and the four phosphorus atoms form distorted tetrahedra. In addition, four THF molecules are coordinated to four of the eight Li

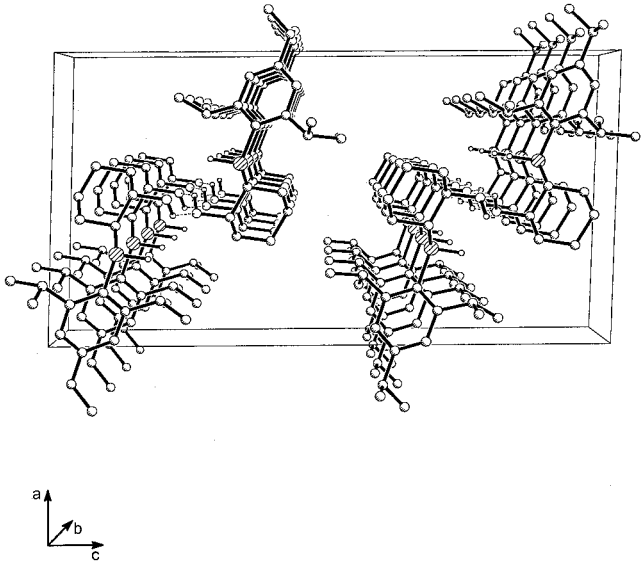


Figure 2. Molecular structure of **6**; section of the three-dimensional arrangement

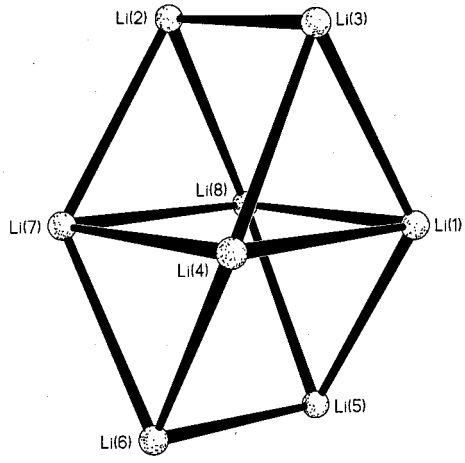


Figure 3. Central Li_8 cluster in **6a** (only one of the two independent molecules is shown); Li(1)−Li(5) 2.66(2), Li(1)−Li(3) 2.67(2), Li(1)−Li(8) 2.79(2), Li(1)−Li(4) 2.80(2), Li(2)−Li(3) 2.47(2), Li(2)−Li(8) 2.67(2), Li(2)−Li(7) 2.72(2), Li(3)−Li(4) 2.654(19), Li(4)−Li(6) 2.66(2), Li(4)−Li(7) 2.83(2), Li(5)−Li(6) 2.47(2), Li(5)−Li(8) 2.67(2), Li(6)−Li(7) 2.63(2), Li(7)−Li(8) 2.80(2)

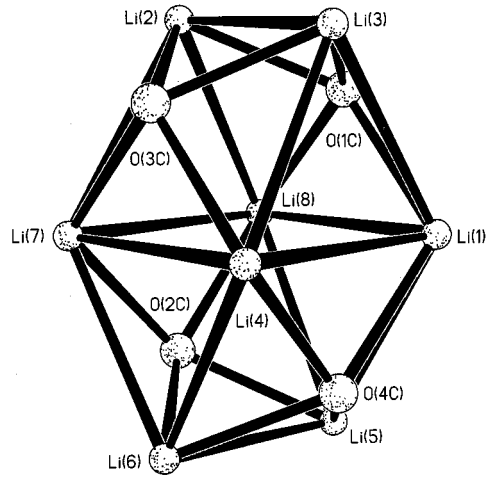


Figure 4. Central Li_8O_4 cluster in **6a** (only one of the two independent molecules is shown); O(1C)−Li(3) 1.980(15), O(1C)−Li(2) 1.997(16), O(1C)−Li(1) 2.037(15), O(1C)−Li(8) 2.085(16), O(2C)−Li(5) 1.962(16), O(2C)−Li(8) 1.986(15), O(2C)−Li(6) 1.997(17), O(2C)−Li(7) 2.072(15), O(3C)−Li(3) 1.998(15), O(3C)−Li(2) 2.008(15), O(3C)−Li(7) 2.020(16), O(3C)−Li(4) 2.083(15), O(4C)−Li(6) 1.965(16), O(4C)−Li(5) 2.011(17), O(4C)−Li(4) 2.012(15), O(4C)−Li(1) 2.096(14)

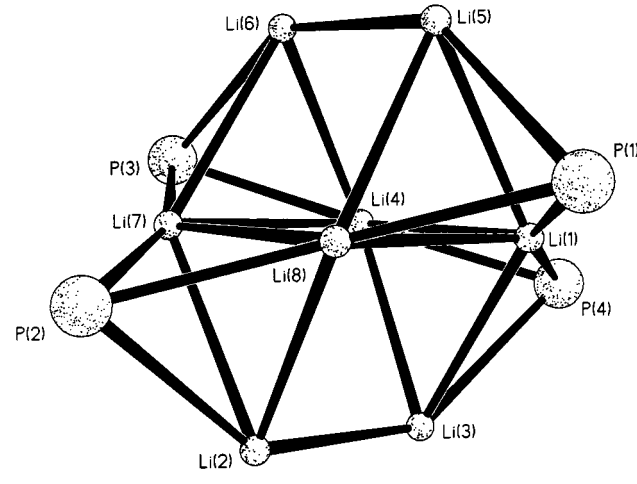


Figure 5. Central Li_8P_4 cluster in **6a** (only one of the two independent molecules is shown); P(1)−Li(8) 2.514(13), P(1)−Li(1) 2.563(15), P(1)−Li(5) 2.685(15), P(2)−Li(7) 2.523(15), P(2)−Li(8) 2.558(13), P(2)−Li(2) 2.699(16), P(3)−Li(4) 2.524(14), P(3)−Li(7) 2.589(15), P(3)−Li(6) 2.686(15), P(4)−Li(1) 2.490(15), P(4)−Li(4) 2.549(14), P(4)−Li(3) 2.680(14)

atoms, Li(2), Li(3), Li(5), and Li(6). A view of the whole molecule is given in Figure 6. The molecule has C_2 symmetry [the noncrystallographic C_2 axis bisects Li(2)−Li(3) and Li(5)−Li(6)], and the four phosphanidoalkoxide ligands have alternatingly *R*/*R*, *S*/*S*, *R*/*R*, and *S*/*S* configuration at the chiral C atoms. Thus, the formation of this cluster can be regarded as a self-assembly of chiral molecules.

Although Li_8 clusters are known,[9] the topology of the Li_8 core in **6a** is very unusual. While lithium clusters have been observed in electron-deficient alkyl or aryllithium compounds,[10] polyhedral arrangements of lithium phosphanides have been observed only recently. In 1995, Drieß et al. reported the formation of clusters of the di- and trimetallated compounds $R^1R^2Si(PR^3Li)_2$ (R^1 = *t*Bu, R^2 = Tipp,

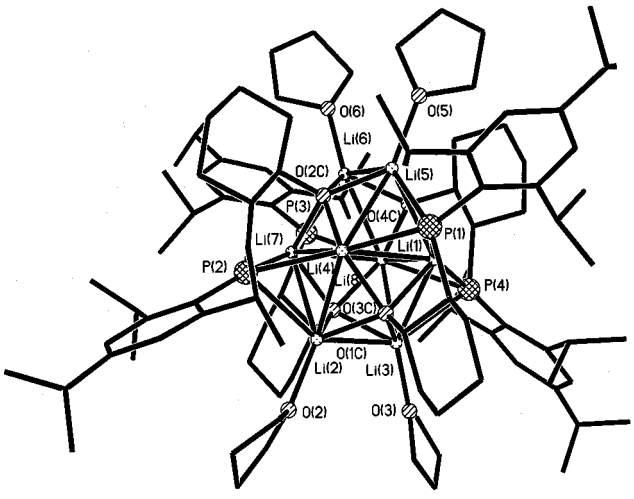


Figure 6. Molecular structure of $[Li_2(THF)_{0.5}\{(C_R,C_R/C_S,C_S)$-2-P(2,4,6-*i*Pr$_3C_6H_2$)-1-O-cyclo-C$_6H_{10}\}]_4$ (**6a**) (only one of the two independent molecules is shown)

R^3 = $SiPh_3$) and $EtSi(PR^3Li)_3$ (R^3 = Si*i*Pr$_3$).[11] In the former, two dimeric molecules $[R^1R^2Si(PR^3Li)_2]_2$ are connected through an Li_2Cl_2 square. In the latter, a dimer of $EtSi(PR^3Li)_3$ is observed, in which the $Li_6P_6Si_2$ fragment forms a distorted rhombododecahedron. The Li−P distances range from 2.55(3) to 2.67(3) Å in the dimetallated compound and from 2.449(5) to 2.562(5) Å in the trimetallated compound and are thus comparable with those in **6a**.

We have already shown that the phosphanylalkoxides reported here can be employed as ligands in mono- and dinuclear complexes.[12] Further studies of the ligation properties of **1**−**6** and their dianions **1a**−**6a** in transition metal complexes are currently in progress.

Experimental Section

General Remarks: All experiments were carried out under purified dry argon. Solvents were dried and freshly distilled under argon. The NMR spectra were recorded with a Bruker AVANCE DRX 400 spectrometer. ^{1}H NMR: internal standard solvent, external standard TMS; ^{13}C NMR (100.6 MHz): internal standard solvent, external standard TMS; ^{31}P NMR (162 MHz): external standard 85% H_3PO_4; ^{7}Li NMR (155.5 MHz): external standard 1 M LiCl in D_2O. The ^{13}C NMR spectra were assigned with the aid of 2D $^{13}C\{^1H\}$ NMR spectra and $^{13}C\{^{31}P,^1H\}$ NMR spectra. − IR spectra were recorded on a Perkin−Elmer System 2000 FT-IR spectrometer in the range 350−4000 cm^{-1}. − Melting points were determined in sealed capillaries under argon and are uncorrected. − Propene oxide and cyclohexene oxide are commercially available. RPH_2 (R = Ph,[13] Mes,[14] Tipp[14]) were prepared according to literature procedures.

General Procedure for the Synthesis of the Phosphanyl Alcohols 1−6: The primary phosphane RPH_2 was dissolved in Et_2O (200 mL) and an equimolar amount of a BuLi solution in hexane was added at −50 °C. The mixture was allowed to warm to room temp. and stirred for a further 2 h. The pale-yellow solution was then cooled to −50 °C once more, whereupon an equimolar amount of propene oxide or cyclohexene oxide was slowly added. The resulting mixture was allowed to warm to room temp. once more, stirred for 2 h, and then hydrolyzed with 50 mL of a 4 M NH_4Cl solution. After stirring for 12 h, the ethereal phase was separated, the aqueous phase was washed with Et_2O (3 × 20 mL), and the combined ethereal phases were dried over Na_2SO_4. The solution was filtered and the solvent was removed in vacuo. The resulting oil was distilled under reduced pressure.

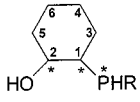
1-(Phenylphosphanyl)-2-propanol (1): Prepared from $PhPH_2$ (12.7 g, 115 mmol), 1.5 M BuLi solution in hexane (80 mL, 120 mmol), and propene oxide (6.8 mL, 140 mmol). Yield: 9.8 g (51%); colourless oil (b.p. 91 °C, 1 × 10^{-1} Torr). − ^{1}H NMR (C_6D_6): δ = 7.40 (m, 4 H, 2 isomers, Ph), 7.07 (m, 6 H, 2 isomers, Ph), 4.24 (d of t, $^1J_{P-H}$ = 210.5 Hz, $^3J_{H-H}$ = 1.7 Hz, 1 H, 1 isomer, PH), 4.18 (d of t, $^1J_{P-H}$ = 210.5 Hz, $^3J_{H-H}$ = 1.7 Hz, 1 H, 1 isomer, PH), 3.75 (sext., $^3J_{H-H}$ = 6.5 Hz, 1 H, 1 isomer, C*H*Me), 3.69 (sext., $^3J_{H-H}$ = 6.4 Hz, 1 H, 1 isomer, C*H*Me), 2.22 (br. s, 2 H, 2 isomers, OH), 2.1−1.73 (m, 4 H, 2 isomers, P−CH_2), 1.25−1.12 (m, 6 H, 2 isomers, CH_3). − $^{13}C\{^1H\}$ NMR (C_6D_6): δ = 136.7 (d, $^3J_{P-C}$ = 10.5 Hz, 4 C, 2 isomers, *m*-CH of Ph), 134.6 (d, $^2J_{P-C}$ = 15.9 Hz, 4 C, 2 isomers, *o*-CH of Ph), 130.9 (2 C, 2 isomers, *p*-CH of Ph), 67.4 (d, $^2J_{P-C}$ = 9.5 Hz, 1 C, 1 isomer, O−CH), 66.9 (d, $^2J_{P-C}$ = 10.1 Hz, 1 C, 1 isomer, O−CH), 35.0 (d, $^1J_{P-C}$ = 25.5 Hz, 1 C, 1 isomer, P−CH_2), 34.8 (d, $^1J_{P-C}$ = 27.5 Hz, 1 C, 1 isomer, P−CH_2), 25.3 (d, $^3J_{P-C}$ = 9.8 Hz, 1 C, 1 isomer, CH_3), 25.2 (d, $^3J_{P-C}$ = 10.6 Hz, 1 C, 1 isomer, CH_3); *ipso*-C of Ph not observed. − ^{31}P NMR (C_6D_6): δ = −63.4 (d, $^1J_{P-H}$ = 210.6 Hz), −64.1 (d, $^1J_{P-H}$ = 210.6 Hz). − $C_9H_{13}OP$ (168.17): calcd. C 64.3, H 7.8, P 18.4; found C 64.0, H 8.1, P 18.0.

1-Mesitylphosphanyl-2-propanol (2): Prepared from $MesPH_2$ (8.0 g, 53.0 mmol), 1.5 M BuLi solution in hexane (35 mL, 53.0 mmol), and propene oxide (3.7 mL, 53.0 mmol). Yield: 6.2 g (56%); colourless oil (b.p. 100−115 °C, 8 × 10^{-3} Torr). − ^{1}H NMR (C_6D_6): δ = 6.75 (s, 4 H, 2 isomers, *m*-CH of Mes), 4.39 (d of t, $^1J_{P-H}$ = 218.5 Hz, $^3J_{H-H}$ = 7.3 Hz, 1 H, 1 isomer, PH), 4.33 (d of t, $^1J_{P-H}$ = 218.0 Hz, $^3J_{H-H}$ = 7.5 Hz, 1 H, 1 isomer, PH), 3.76 (sext., $^3J_{H-H}$ = 6.2 Hz, 1 H, 1 isomer, C*H*Me), 3.69 (sext., $^3J_{H-H}$ = 6.3 Hz, 1 H, 1 isomer, C*H*Me), 2.51 (br. s, 2 H, 2 isomers, OH), 2.43 (s, 12 H, 2 isomers, *o*-CH_3), 2.10 (s, 6 H, 2 isomers, *p*-CH_3), 2.05−1.85 (m, 2 H, 1 isomer, P−CH_2), 1.65−1.50 (m, 2 H, 1 isomer, P−CH_2), 1.11 (d, $^3J_{H-H}$ = 6.2 Hz, 3 H, 1 isomer, CH_3), 1.09 (d, $^3J_{H-H}$ = 6.1 Hz, 3 H, 1 isomer, CH_3). − $^{13}C\{^1H\}$ NMR (C_6D_6): δ = 142.7 (d, $^2J_{P-C}$ = 12.0 Hz, 2 C, 1 isomer, *o*-C of Mes), 142.7 (d, $^2J_{P-C}$ = 12.0 Hz, 2 C, 1 isomer, *o*-C of Mes), 138.6 (d, $^4J_{P-C}$ = 11.0 Hz, 2 C, 2 isomers, *p*-C of Mes), 131.0 (d, $^1J_{P-C}$ = 14.2 Hz, 1 C, 1 isomer, *ipso*-C of Mes), 131.0 (d, $^1J_{P-C}$ = 14.2 Hz, 1 C, 1 isomer, *ipso*-C of Mes), 130.0 (4 C, 2 isomers, *m*-C of Mes), 68.2 (d, $^2J_{P-C}$ = 9.6 Hz, 1 C, 1 isomer, O−CH), 67.6 (d, $^2J_{P-C}$ = 10.7 Hz, 1 C, 1 isomer, O−CH), 32.9 (d, $^1J_{P-C}$ = 14.4 Hz, 1 C, 1 isomer, P−CH_2), 32.9 (d, $^1J_{P-C}$ = 14.9 Hz, 1 C, 1 isomer, P−CH_2), 25.3 (1 C, 1 isomer, CH_3), 25.2 (1 C, 1 isomer, CH_3), 23.9 (2 C, 1 isomer, *o*-CH_3 of Mes), 23.8 (2 C, 1 isomer, *o*-CH_3 of Mes), 21.7 (2 C, 2 isomers, *p*-CH_3 of Mes). − ^{31}P NMR (C_6D_6): δ = −97.7 (d, $^1J_{P-H}$ = 217.0 Hz), −98.9 (d, $^1J_{P-H}$ = 218.7 Hz). − EI MS: *m*/*z* (%) = 210 (78) [M^+], 150 (100) [$PHMes^+$], 119 (88) [Mes^+], and fragmentation products thereof. − IR (KBr): $\tilde{\nu}$ = 3356 cm^{-1} [br, s, ν(OH)], 2335 [s, ν(PH)]. − $C_{12}H_{19}OP$ (210.25): calcd. C 68.6, H 9.1, P 14.7; found C 68.1, H 9.3, P 13.9.

1-(Triisopropylphenylphosphanyl)-2-propanol (3): Prepared from $TippPH_2$ (9.0 g, 38.6 mmol), 1.5 M BuLi solution in hexane (26 mL, 39.0 mmol), and propene oxide (3 mL, 43.0 mmol). Yield: 7.0 g (62%); colourless oil (b.p. 110−140 °C, 2 × 10^{-2} Torr), which was found to solidify at room temp. (m.p. 35 °C). − ^{1}H NMR (C_6D_6): δ = 7.16 (s, 4 H, 2 isomers, *m*-H of Tipp), 4.50 (d of t, $^1J_{P-H}$ =

216.1 Hz, $^3J_{H-H}$ = 7.3 Hz, 2 H, 2 isomers, PH), 3.84 (sept., $^3J_{H-H}$ = 7.0 Hz, 4 H, 2 isomers, *o*-C*H*Me$_2$ of Tipp), 3.74 (m, $^3J_{H-H}$ = 5.8 Hz, 2 H, 2 isomers, C*H*Me), 2.78 (sept., $^3J_{H-H}$ = 7.0 Hz, 2 H, 2 isomers, *p*-C*H*Me$_2$ of Tipp), 2.03 and 1.65 (m, 4 H, 2 isomers, P−CH$_2$), 1.34 [d, $^3J_{H-H}$ = 6.7 Hz, 12 H, 2 isomers, *o*-CH(CH_3)$_2$ of Tipp], 1.26 [d, $^3J_{H-H}$ = 6.8 Hz, 12 H, 2 isomers, *p*-CH(CH_3)$_2$ of Tipp], 1.20 [d, $^3J_{H-H}$ = 6.7 Hz, 12 H, 2 isomers, *o*-CH(CH_3)$_2$ of Tipp], 1.07 (d, $^3J_{H-H}$ = 6.1 Hz, 6 H, 2 isomers, CHCH_3); OH not observed. − ^{13}C{H} NMR (C_6D_6): δ = 154.0 (d, $^2J_{P-C}$ = 10.4 Hz, 4 C, 2 isomers, *o*-C of Tipp), 150.6 (d, $^4J_{P-C}$ = 10.1 Hz, 2 C, 2 isomers, *p*-C of Tipp), 122.4 (s, 4 C, 2 isomers, *m*-C of Tipp), 67.9 (d, $^2J_{P-C}$ = 12.1 Hz, 1 C, 1 isomer, O−CH), 67.6 (d, $^2J_{P-C}$ = 12.9 Hz, 1 C, 1 isomer, O−CH), 35.6 (d, $^1J_{P-C}$ = 13.7 Hz, 1 C, P−CH$_2$), 35.6 (d, $^1J_{P-C}$ = 13.9 Hz, 1 C, P−CH$_2$), 35.4 (s, 2 C, 2 isomers, *p*-C*H*Me$_2$ of Tipp), 33.7 (s, 2 C, 1 isomer, *o*-C*H*Me$_2$ of Tipp), 33.6 (s, 2 C, 1 isomer, *o*-C*H*Me$_2$ of Tipp), 25.8 [s, 4 C, 2 isomers, *p*-CH(CH_3)$_2$ of Tipp], 25.6 (s, 2 C, 2 isomers, CHCH_3), 25.2 [d, $^4J_{P-C}$ = 3.1 Hz, 4 C, 1 isomer, *o*-CH(CH_3)$_2$ of Tipp], 24.8 [d, $^4J_{P-C}$ = 3.6 Hz, 4 C, 1 isomer, *o*-CH(CH_3)$_2$ of Tipp]; *ipso*-C of Tipp not observed. − ^{31}P NMR (C_6D_6): δ = −105.2 (d, 1 isomer, $^1J_{P-H}$ = 215.5 Hz), −105.9 (d, 1 isomer, $^1J_{P-H}$ = 217.1 Hz). − EI MS: *m*/*z* (%) = 294 (79) [M$^+$], 235 (50) [PHTipp$^+$], 203 (100) [Tipp$^+$], and fragmentation products thereof. − IR (KBr): $\tilde{\nu}$ = 3377 cm^{-1} [br, s, ν(OH)], 2330 [m, ν(PH)]. − $C_{18}H_{31}OP$ (294.41): calcd. C 73.4, H 10.6, P 10.5; found C 73.0, H 10.5, P 10.3.

Numbering scheme for the cyclohexyl ring:



2-(Phenylphosphanyl)cyclohexan-1-ol (4): Prepared from $PhPH_2$ (5.0 g, 46.0 mmol), 1.5 M BuLi solution in hexane (30 mL, 46.0 mmol), and cyclohexene oxide (4.6 mL, 46.0 mmol). Yield: 4.8 g (51%); colourless oil (b.p. 70−75 °C, 3×10^{-2} Torr). − ^{1}H NMR (C_6D_6): δ = 7.52 (m, 4 H, 2 isomers, Ph), 7.11 (m, 6 H, 2 isomers, Ph), 4.57 (d of d, $^1J_{P-H}$ = 216.6 Hz, $^3J_{H-H}$ = 3.8 Hz, 1 H, 1 isomer, PH), 4.21 (d of d, $^1J_{P-H}$ = 213.1 Hz, $^3J_{H-H}$ = 5.0 Hz, 1 H, 1 isomer, PH), 3.40 (m, $^3J_{H-H}$ = 4.1 Hz, 1 H, 1 isomer, O−CH), 3.23 (m, $^3J_{H-H}$ = 4.2 Hz, 1 H, 1 isomer, O−CH), 2.40 (br. s, 2 H, 2 isomers, OH), 1.96−0.85 (m, 18 H, 2 isomers, Cy). − ^{13}C{H} NMR (C_6D_6): δ = 136.4 (d, $^2J_{P-C}$ = 15.9 Hz, 2 C, 1 isomer, *o*-C of Ph), 135.5 (d, $^2J_{P-C}$ = 15.1 Hz, 2 C, 1 isomer, *o*-C of Ph), 135.2 (d, $^4J_{P-C}$ = 12.4 Hz, 1 C, 1 isomer, *p*-C of Ph), 134.8 (d, $^4J_{P-C}$ = 12.1 Hz, 1 C, 1 isomer, *p*-C of Ph), 129.6 (d, 2 C, 2 isomers, *ipso*-C, partially obscured by solvent signal), 129.2 (d, $^3J_{P-C}$ = 6.2 Hz, 2 C, 1 isomer, *m*-C of Ph), 129.2 (d, $^3J_{P-C}$ = 5.4 Hz, 2 C, 1 isomer, *m*-C of Ph), 73.7 (d, $^2J_{P-C}$ = 9.1 Hz, 1 C, 1 isomer, O−CH), 73.1 (d, $^2J_{P-C}$ = 8.8 Hz, 1 C, 1 isomer, O−CH), 44.4 (d, $^1J_{P-C}$ = 12.7 Hz, 1 C, 1 isomer, P−CH), 43.2 (d, $^1J_{P-C}$ = 10.3 Hz, 1 C, 1 isomer, P−CH), 37.1 (d, $^3J_{P-C}$ = 6.3 Hz, 1 C, 1 isomer, C3 of Cy), 36.9 (d, $^3J_{P-C}$ = 5.1 Hz, 1 C, 1 isomer, C3 of Cy), 32.0 (d, $^2J_{P-C}$ = 3.8 Hz, 1 C, 1 isomer, C6 of Cy), 30.7 (d, $^2J_{P-C}$ = 10.0 Hz, 1 C, 1 isomer, C6 of Cy), 27.1 (d, $^3J_{P-C}$ = 6.4 Hz, 1 C, 1 isomer, C5 of Cy), 26.6 (d, $^3J_{P-C}$ = 7.9 Hz, 1 C, 1 isomer, C5 of Cy), 25.6 (1 C, 1 isomer, C4 of Cy), 25.5 (1 C, 1 isomer, C4 of Cy). − ^{31}P NMR (C_6D_6): δ = −40.9 (d, $^1J_{P-H}$ = 213.8 Hz), −45.5 (d, $^1J_{P-H}$ = 217.1 Hz). − EI MS: *m*/*z* (%) = 208 (47) [M$^+$], 190 (83) [M$^+$ − H_2O], 109 (58) [PHPh$^+$], and fragmentation products thereof. − IR (KBr): $\tilde{\nu}$ = 3385 cm^{-1} [br, s, ν(OH)], 2286 [s, ν(PH)]. − $C_{12}H_{17}OP$ (208.24): calcd. C 69.2, H 8.2, P 14.9; found C 68.7, H 8.0, P 14.5.

2-(Mesitylphosphanyl)cyclohexan-1-ol (5): Prepared from $MesPH_2$ (10.0 g, 66.0 mmol), 1.5 M BuLi solution in hexane (47 mL, 70.0 mmol), and cyclohexene oxide (7.4 mL, 77.0 mmol). Yield: 7.1 g (43%); white solid (m.p. 110−115 °C). − ^{1}H NMR (C_6D_6): δ = 6.79 (s, 4 H, 2 isomers, *m*-C*H* of Mes), 4.79 (d of d, $^1J_{P-H}$ = 222.3 Hz, $^3J_{H-H}$ = 4.8 Hz, 1 H, 1 isomer, PH), 4.08 (d of d, $^1J_{P-H}$ = 216.6 Hz, $^3J_{H-H}$ = 6.8 Hz, 1 H, 1 isomer, PH), 3.48 (m, 1 H, 1 isomer, O−CH), 3.38 (m, 1 H, 1 isomer, O−CH), 2.55 (s, 6 H, 2 isomers, *o*-CH_3 of Mes), 2.45 (s, 6 H, 2 isomers, *o*-CH_3 of Mes), 2.09 (s, 6 H, 2 isomers, *p*-CH_3 of Mes), 2.0−0.87 (m, 18 H, 2 isomers, Cy); OH not observed. − ^{13}C{^{1}H} NMR (C_6D_6): δ = 144.1 (d, $^2J_{P-C}$ = 12.0 Hz, 2 C, 1 isomer, *o*-C of Mes), 142.9 (d, $^2J_{P-C}$ = 11.8 Hz, 2 C, 1 isomer, *o*-C of Mes), 139.0 (1 C, 1 isomer, *p*-C of Mes), 138.4 (1 C, 1 isomer, *p*-C of Mes), 130.1 (4 C, 2 isomers, *m*-C of Mes), 130.0 (d, $^1J_{P-C}$ = 20.4 Hz, 2 C, 2 isomers, *ipso*-C of Mes), 75.6 (d, $^2J_{P-C}$ = 9.5 Hz, 1 C, 1 isomer, O−CH), 75.1 (d, $^2J_{P-C}$ = 13.2 Hz, 1 C, 1 isomer, O−CH), 43.9 (d, $^1J_{P-C}$ = 12.0 Hz, 1 C, 1 isomer, P−CH), 42.2 (d, $^1J_{P-C}$ = 12.3 Hz, 1 C, 1 isomer, P−CH), 37.1 (d, $^3J_{P-C}$ = 10.0 Hz, 2 C, 2 isomers, C3 of Cy), 30.3 (d, $^2J_{P-C}$ = 15.6 Hz, 2 C, 2 isomers, C6 of Cy), 27.0 (d, $^3J_{P-C}$ = 10.7 Hz, 2 C, 2 isomers, C5 of Cy), 25.7 (d, $^4J_{P-C}$ = 3.9 Hz, 2 C, 2 isomers, C4 of Cy), 24.4 (d, $^3J_{P-C}$ = 12.0 Hz, 2 C, 2 isomers, *o*-CH_3 of Mes), 24.4 (d, $^3J_{P-C}$ = 10.9 Hz, 2 C, 2 isomers, *o*-CH_3 of Mes), 21.7 (d, $^5J_{P-C}$ = 2.5 Hz, 2 C, 2 isomers, *p*-CH_3 of Mes). − ^{31}P NMR (C_6D_6): δ = −105.3 (d, $^1J_{P-H}$ = 215.5 Hz), −106.0 (d, $^1J_{P-H}$ = 215.5 Hz). − EI MS: *m*/*z* (%) = 250 (50) [M$^+$], 151 (67) [PHMes$^+$], and fragmentation products thereof. − IR (KBr): $\tilde{\nu}$ = 3335 cm^{-1} [br, s, ν(OH)], 2339 [s, ν(PH)]. − $C_{15}H_{23}OP$ (250.32): calcd. C 72.0, H 9.3, P 12.4; found C 71.5, H 8.9, P 13.0.

2-(Triisopropylphenylphosphanyl)cyclohexan-1-ol (6): Prepared from $TippPH_2$ (12.3 g, 53.0 mmol), 1.5 M BuLi solution in hexane (38 mL, 57.0 mmol), and cyclohexene oxide (5.4 mL, 53.0 mmol). Yield: 12.7 g (72%); colourless oil (b.p. 170 °C, 5×10^{-3} Torr), which was found to solidify at room temp. Recrystallization from pentane yielded single crystals (m.p. 156 °C). − ^{1}H NMR (C_6D_6): δ = 7.18 (s, 4 H, 2 isomers, *m*-CH of Tipp), 5.04 (d of d, $^1J_{P-H}$ = 220.0 Hz, $^3J_{H-H}$ = 5.7 Hz, 1 H, 1 isomer, PH), 4.38 (d of d, $^1J_{P-H}$ = 216.0 Hz, $^3J_{H-H}$ = 6.2 Hz, 1 H, 1 isomer, PH), 4.09 (sept., $^3J_{H-H}$ = 4.5 Hz, 2 H, 1 isomer, *o*-C*H*Me$_2$ of Tipp), 3.85 (sept., $^3J_{H-H}$ = 3.5 Hz, 2 H, 1 isomer, *o*-C*H*Me$_2$ of Tipp), 3.46 (m, 2 H, 2 isomers, O−CH), 2.76 (sept., $^3J_{H-H}$ = 5.0 Hz, 2 H, 2 isomers, *p*-C*H*Me$_2$ of Tipp), 2.15−1.08 (m, 18 H, 2 isomers, Cy), 1.35 [d, $^3J_{H-H}$ = 6.7 Hz, 12 H, 1 isomer, *o*-CH(CH_3)$_2$ of Tipp], 1.30 [d, $^3J_{H-H}$ = 6.8 Hz, 6 H, 1 isomer, *p*-CH(CH_3)$_2$ of Tipp], 1.27 [d, $^3J_{H-H}$ = 6.9 Hz, 6 H, 1 isomer, *p*-CH(CH_3)$_2$ of Tipp], 1.21 [d, $^3J_{H-H}$ = 6.8 Hz, 12 H, 1 isomer, *o*-CH(CH_3)$_2$ of Tipp]; OH not observed. − ^{13}C{^{1}H} NMR (C_6D_6): δ = 155.0 (d, $^2J_{P-C}$ = 11.1 Hz, 2 C, 1 isomer, *o*-C of Tipp), 153.9 (d, $^2J_{P-C}$ = 11.0 Hz, 2 C, 1 isomer, *o*-C of Tipp), 150.6 (1 C, 1 isomer, *p*-C of Tipp), 150.2 (1 C, 1 isomer, *p*-C of Tipp), 127.4 (d, $^1J_{P-C}$ = 15.9 Hz, 2 C, 2 isomers, *ipso*-C of Tipp), 122.3 (t, 4 C, 2 isomers, *m*-C of Tipp), 75.6 (d, $^2J_{P-C}$ = 10.6 Hz, 1 C, 1 isomer, O−CH), 75.1 (d, $^2J_{P-C}$ = 13.9 Hz, 1 C, 1 isomer, O−CH), 45.7 (d, $^1J_{P-C}$ = 12.0 Hz, 1 C, 1 isomer, P−CH), 43.3 (d, $^1J_{P-C}$ = 12.9 Hz, 1 C, 1 isomer, P−CH), 37.3 (d, $^3J_{P-C}$ = 6.2 Hz, 1 C, 1 isomer, C3 of Cy), 36.9 (d, $^3J_{P-C}$ = 6.6 Hz, 1 C, 1 isomer, C3 of Cy), 35.3 (s, 4 C, 2 isomers, *o*-C*H*Me$_2$ of Tipp), 33.7 (d, $^2J_{P-C}$ = 14.6 Hz, 1 C, 1 isomer, C6 of Cy), 33.6 (d, $^2J_{P-C}$ = 14.9 Hz, 1 C, 1 isomer, C6 of Cy), 32.7 (s, 2 C, 2 isomers, *p*-C*H*Me$_2$ of Tipp), 29.3 (d, $^3J_{P-C}$ = 15.2 Hz, 1 C, 1 isomer, C5 of Cy), 27.5 (d, $^4J_{P-C}$ = 2.8 Hz, 1 C, 1 isomer, C4 of Cy), 27.0 (d, $^3J_{P-C}$ = 10.4 Hz, 1 C, 1 isomer, C5 of Cy), 25.7, 25.6, 25.5, 25.3, 25.2, 25.1 [12 C, 2 isomers, CH(CH_3)$_2$ of Tipp], 24.6 (d, $^4J_{P-C}$ = 4.8 Hz, 1 C, 1 isomer, C4 of Cy). − ^{31}P NMR (C_6D_6): δ = −77.6 (d, $^1J_{P-H}$ = 216.0 Hz), −89.9 (d,

$^1J_{P-H}$ = 220.0 Hz). − EI MS: *m/z* (%)= 334 (63) [M^+], 235 (52) [$PHTipp^+$], 203 (100) [$Tipp^+$], and fragmentation products thereof. − IR (KBr): $\tilde{\nu}$ = 3452 cm^{-1} [br, s, ν(OH)], 2359 [s, ν(PH)]. − $C_{21}H_{35}OP$ (334.48): calcd. C 75.4, H 10.5, P 9.3; found C 73.2, H 10.6, P 9.0.

Dianions 1a−6a: 1−6 were treated with two equivalents of BuLi in THF at −50 °C. Subsequent warming to room temp. gave the dilithio salts Li_2[$RPCH_2CHMeO$] [R = Ph (**1a**), Mes (**2a**), Tipp (**3a**)] [Equation (3)] and Li_2[2-RP-1-O-cyclo-C_6H_{10}] [R = Ph (**4a**), Mes (**5a**), Tipp (**6a**)] [Equation (4)], of which only **6a** was isolated. The deprotonation could be monitored by ^{31}P NMR and ^7Li NMR spectroscopy (solvent THF/C_6D_6). − **1a**: ^{31}P NMR: δ = −76.9 (br. s). − ^7Li NMR: δ = 1.5 (s), 1.3 (s), 1.2 (s), 0.9 (s). − **2a**: ^{31}P NMR: δ = −82.0 (br. s), −87.2 (br. s). − ^7Li NMR: δ = 1.4 (s), 1.3 (s), 1.0 (s). − **3a**: ^{31}P NMR: δ = −42.9 (br. s). − ^7Li NMR: δ = 4.6 (br. s), 3.9 (br. s). − **4a**: ^{31}P NMR: δ = −18.3 (br. s), −45.9 (br. s). − ^7Li NMR: δ = 5.6 (br. s), 5.0 (br. s), 4.5 (br. s). − **5a**: ^{31}P NMR: δ = −81.5 (br. s), −96.0 (br. s). − ^7Li NMR: δ = 4.3 (br. s), 3.9 (br. s). − **6a**: ^{31}P NMR: δ = −81.4 (br. s), −95.6 (br. s). − ^7Li NMR: δ = 4.3 (br. s), 3.9 (br. s), 3.7 (br. s). Crystals of **6a** were obtained from pentane.

Data Collection and Structure Refinement of 6 and 6a:

6: Experimental data (a total of 13072 reflections) were collected at *T* = 220(2) K on a Siemens CCD (SMART) diffractometer in the range 1.5 < Θ < 26.2° using monochromated Mo-K_α radiation (λ = 0.71073 Å). Of these, a total of 3740 reflections were considered as unique (R_{int} = 0.0637). All observed reflections were used in the determination of the unit cell parameters. The studied specimen was a white single crystal of dimensions 0.4 × 0.2 × 0.1 mm. Crystal data for **6** are as follows: $C_{21}H_{35}OP$, M_r = 334.46, space group $P2_1/c$ (no. 14), *a* = 13.755(1), *b* = 5.955(1), *c* = 25.444(1) Å, β = 91.529(1)°, *V* = 2083.3(2) $Å^3$ at *T* = 220(2) K, *Z* = 4, $D_{calcd.}$ = 1.066 Mg m^{-3}, μ(Mo-K_α) = 0.136 mm^{-1}; absorption correction with SADABS.[15]

The positions of the P, O, and C atoms were located using direct methods (SHELXTL-PLUS).[16] Subsequent least-squares refinement and difference electron density map calculations revealed the positions of the H atoms. Final full-matrix least-squares refinement of 348 parameters with a unit weighting scheme (P, O, and C atoms anisotropic approximation; H atoms isotropic approximation) converged to *R*1 = 0.0497, *wR*2 = 0.1282 [for reflections with $I > 2\sigma(I)$], *R*1 = 0.0606, *wR*2 = 0.1363 (all data).

6a: Experimental data (a total of 160515 reflections) were collected at *T* = 210(2) K on a Siemens CCD (SMART) diffractometer in the range 1.0 < Θ < 25.6° using monochromated Mo-K_α (λ = 0.71073 Å) radiation. Of these, a total of 40798 reflections were considered unique (R_{int} = 0.1687). All observed reflections were used in the determination of the unit cell parameters. The studied specimen was a white single crystal of dimensions 0.4 × 0.3 × 0.1 mm. Crystal data for **6a** are as follows: $C_{100}H_{164}Li_8O_8P_4$, M_r = 1673.71, space group $P2_1/c$ (no. 14), monoclinic, *a* = 27.929(1), *b* = 36.492(1), *c* = 24.992(1) Å, β = 110.668(1)°, *V* = 23833(2) $Å^3$ at *T* = 210(2) K, *Z* = 8, $D_{calcd.}$ = 0.933 Mg m^{-3}, μ(Mo-K_α) = 0.106 mm^{-1}; absorption correction with SADABS.[15]

The positions of the Li, P, O, and C atoms were located using direct methods (SHELXTL-PLUS);[16] H atoms were placed in calculated positions. Final full-matrix least-squares refinement of 2161 parameters with a unit weighting scheme (Li, P, O, and C atoms anisotropic approximation; H atoms isotropic approximation) converged to *R*1 = 0.1559, *wR*2 = 0.3746 [for reflections with $I > 2\sigma(I)$], *R*1 = 0.3221, *wR*2 = 0.4570 (all data).

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre (**6:** CCDC-141876, **6a:** CCDC-141875). Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, U.K. [Fax: (internat.) +44 (0)1223 336033; E-mail: deposit@ccdc.cam.ac.uk].

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