Synthesis and Structural Characterization of the Solvent-Free Potassium Derivative of a Terphenyl-Substituted Primary Phosphane: [(2,6-Dimesitylphenyl)P(H)K]$_4$

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While numerous reports on molecular structures of lithium derivatives of both primary and secondary phosphanes have appeared,[1] still only relatively little information is available on solid-state structures of the corresponding derivatives of heavier alkali metals[2a−g] or molecular species containing M−P contacts (M = Na−Cs).[2h−k] Our previous work[3,4] has shown that heavier congeners of alkali metal salts (M = K, Rb, Cs) of 2,4,6-tri-*tert*-butylphenylphosphane (= "supermesitylphosphane")[5] exhibit infinitely extended M−P ladder-type connectivities. A common feature of these polymeric systems is π coordination of each supermesityl ring to an adjacent alkali metal cation.

Because of our interest in structural details of aryl pnictides of the heavier alkali metals, we were interested in investigating whether the use of extremely bulky primary phosphanes will moderate structure growth and limit nuclearity of the resulting complexes. Therefore, we continued our studies by employing the sterically demanding terphenyl ligand Dmp (Dmp = 2,6-dimesitylphenyl)[6] as a substituent on phosphorus. Our efforts produced crystal structure determinations of both the rubidium and the cesium derivative[7] of (2,6-dimesitylphenyl)phosphane.[8]

We here report the synthesis and X-ray crystal structure determination of KP(H)Dmp, thereby demonstrating that, by employing bulky terphenyl substituents on the phosphorus atom of a primary phosphane, extended ladder structures can be truncated to a four-rung structure. Also, we are presenting another novel structural motif in the area of heavier congeners of alkali metal derivatives of primary phosphanes.

KP(H)Dmp is obtained from the reaction of excess potassium hydride (or, alternatively, potassium metal) with DmpPH$_2$ in tetrahydrofuran at ambient temperature. Even in the presence of excess potassium hydride or metal, bismetalation of DmpPH$_2$ is not observed. Crystallization of KP(H)Dmp from toluene at −30 °C produces yellow crystals. The composition of these crystals was determined crystallographically as [KP(H)Dmp]$_4$ (**1**).[9] It is noteworthy that we were also able to obtain crystals of **1** by crystallization of KP(H)Dmp from hexanes in the presence of 18-crown-6.

The molecular structure of complex **1** is found to differ significantly from the solid-state structures of both the previously determined rubidium and cesium derivatives.[7] This observation is surprising, given the fact that the corresponding alkali metal phosphides (M = K−Cs) with a [P(H)^{t}Bu$_3$Mes] group *all* exhibit similar structural motifs, i.e. infinitely extended M−P ladder-type structures.[3,4]

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(9) Crystal data for **1**: $C_{96}H_{100}K_4P_4$, $M_r = 1534.04$, triclinic, $P\bar{1}$, $a = 11.5144(2)$ Å, $b = 14.3774(2)$ Å, $c = 15.1334(2)$ Å, $\alpha = 67.614(1)°$, $\beta = 89.469(1)°$, $\gamma = 69.255(1)°$, $V = 2143.32(6)$ Å^3, $Z = 1$, $D_{calc} = 1.189$ g cm^{-3}, $F(000) = 812$, Mo Kα radiation ($\lambda = 0.710\,73$ Å), $T = 203(2)$ K, μ(Mo Kα) = 0.327 mm^{-1}. A total of 11 533 reflections were collected on a Siemens SMART CCD diffractometer on a yellow crystal with approximate dimensions 0.10 × 0.10 × 0.10 mm^3 in the 2θ range from 2.94 to 42.00°. A total of 4589 reflections were independent and were considered observed and used for refinement. The structure was solved by direct methods and refined by full-matrix least-squares calculations on F^2 to final residuals of $R_1 = 0.0629$ and w$R_2 = 0.1737$ for 4589 observed data ($I > 2\sigma(I)$), and GOF = 1.040. The function minimized was $R(wF^2) = \sum[w(F_o^2 - F_c^2)^2]/\sum[(wF_o^2)^2]^{1/2}$; $R = \sum\Delta/\Sigma(F_o)$, $\Delta = |F_o - F_c|$. All hydrogen atoms excluding those on the phosphorus atoms, which were ignored, were treated as idealized contributions. Minimal/maximal residual electron density: +0.535/−0.504 e Å^{-3}. All software and sources of scattering factors are contained in the SHELXTL (5.03) program library (G. Sheldrick, Siemens XRD, Madison, WI).

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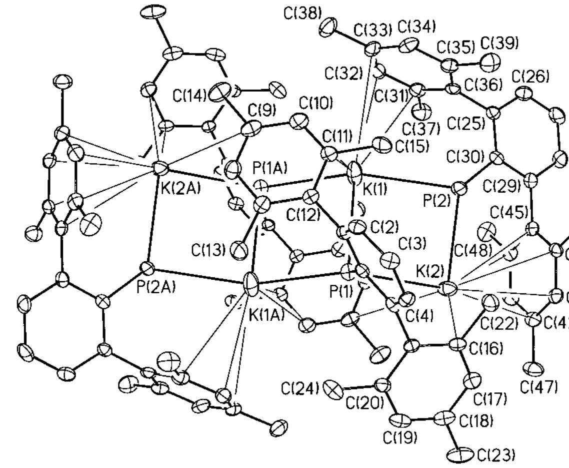

Figure 1. Molecular structure of [KP(H)Dmp]$_4$ (**1**) showing the four-step ladder-type arrangement in the solid state. Selected interatomic separations (Å) and angles (deg): K(1)−P(1) = 3.438(2), K(1)−P(1A) = 3.162(2), K(1)−P(2) = 3.189(2), K(2)−P(1) = 3.365(2), K(2)−P(2) = 3.043(2), K(1)···K(2) = 4.486(2), K(1)···K(1A) = 4.598(3), P(1)−C(6) = 1.812(5), P(2)−C(30) = 1.800(5), K(1)···C(7A) = 3.916(2), K(1)···C(10) = 3.484(2), K(1)···C(11) = 3.352(2), K(1)···C(12) = 3.989(2), K(1)···C(31) = 3.622(2), K(1)···C(32) = 3.487(2), K(1)···C(33) = 3.795(2), K(2)···C(8A) = 3.780(2), K(2)···C(9A) = 3.928(2), K(2)···C(16) = 3.103(2), K(2)···C(17) = 3.318(2), K(2)···C(21) = 3.722(2), K(2)···C(22) = 3.181(2), K(2)···C(40) = 3.529(2), K(2)···C(41) = 3.326(2), K(2)···C(42) = 3.208(2), K(2)···C(43) = 3.254(2), K(2)···C(44) = 3.464(2), K(2)···C(45) = 3.597(2), K(2)···C(47) = 3.927(2); P(1)−K(1)−P(1A) = 91.78(5), P(1)−K(1)−P(2) = 83.83(5), P(1)−K(2)−P(2) = 87.35(4), P(1A)−K(1)−P(2) = 156.06(7), C(6)−P(1)−K(1) = 117.0(2), C(6)−P(1)−K(1A) = 123.8(2), C(6)−P(1)−K(2) = 104.3(2), C(30)−P(2)−K(1) = 123.4(2), C(30)−P(2)−K(2) = 128.7(2).

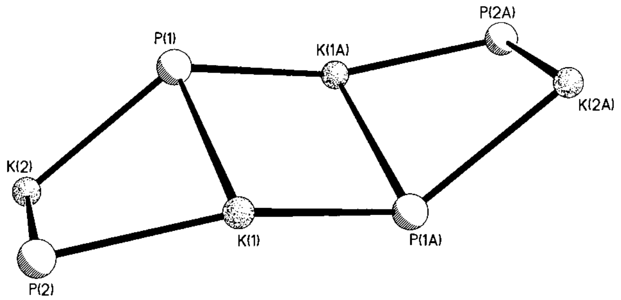

Figure 2. Ball-and-stick drawing of the K−P framework of complex **1**.

The molecular structure of **1** (Figure 1) features a centrosymmetric distorted transoid ladder-type arrangement of the ABB′A′ type which is composed of four K−P rungs in length (Figure 2). One of the mesityl rings of the Dmp ligand attached to phosphorus atom P(2) "solvates" potassium cation K(2) in an approximate η^6 fashion with K(2)···C distances ranging from 3.208(2) Å [C(42)] to 3.597(2) Å [C(45)]. The bulky substituents on phosphorus prevent further extension or association of the K−P ladder. Additionally, both mesityl rings of the Dmp ligand attached to phosphorus atom P(1) weakly "solvate" atom K(2) with different amounts of π interaction. Atom K(1) is complexed as well by one mesityl ring of each Dmp ligand with varying degrees of π interaction. The limiting effect of bulky substituents on phosphorus on the extension of M−P ladders was previously observed in the molecular structure of the lithium phosphide LiP(SiMe$_3$)$_2$.[1f] More specific details on the π bonding in complex **1** can be derived from the caption of Figure 1. The K···C distances in **1** can be compared, e.g., with the corresponding distances in η^3-bonded [KP(H)^{t}Bu$_3$Mes]$_x$ [2.884(4), 3.164(4), and 3.197(4) Å].[3] π interaction between alkali metal cations and aromatic systems has wide precedent in the literature.[2a,e−f,3,4,7,10]

The K−P distances in **1** range from 3.043(2) to 3.438(2) Å. These interatomic separations can be compared with the corresponding numbers in the stepped ladder framework of infinitely extended [KP(H)^{t}Bu$_3$Mes]$_x$ [3.181(2), 3.271(2) (in the rung position), and 3.357(2) Å][3] and can further be compared with reports on other molecular K−P species.[2a,d,e,g−k] As a matter of fact, the K−P distance of the outer rung position in complex **1** [K(2)−P(2) = 3.043(2) Å] is by far the shortest interatomic separation between these two elements observed so far in molecular K−P species. It is more than 0.1 Å shorter than all other K−P interatomic separations in complex **1**. This observation can be rationalized in terms of a low coordination number around the formally two-coordinate potassium atom K(2) with a P(1)−K(2)−P(2) angle which is smaller than rectangular [87.35(4)°]. The K(2)−P(2) distance is even shorter than the shortest K−P interatomic separation in solid-state compound KP [3.08(1) Å].[11] On the other hand, the K−P distance of the inner rung position [K(1)−P(1) = 3.438(2) Å] is the longest K−P distance in complex **1**. This result can most likely be interpreted as a consequence of steric factors, i.e. van der Waals repulsion between the Dmp ligands attached to phosphorus atom P(1).

Atom P(2) deviates 1.27(1) Å from the plane defined by atoms P(1), K(1), P(1A), and K(1A) with a dihedral angle of 23.6(2)° between the P(1)−K(1)−P(2) triangle and the P(1)−K(1)−P(1A)−K(1A) square. Atom K(2) deviates 2.55(1) Å from the P(1)−K(1)−P(1A)−K(1A) plane, and the dihedral angle between the K(1)−P(1)−K(2) triangle and the P(1)−K(1)−P(1A)−K(1A) square is 49.8(2)°.

The coordination geometry around phosphorus atom P(1) can best be described as distorted tetrahedral with K−P(1)−K and K−P(1)−C(6) angles ranging from 82.51(4) to 129.47(6)°, under neglect of the P−H proton atom. On the other hand, the coordination environment around phosphorus atom P(2) is distinctly trigonal-pyramidal with a sum of angles around phosphorus of 344.2°, again under neglect of the P−H proton atom. The proton atoms on the phosphorus atoms could not be located from a difference map and were therefore not included in the calculations. It is to be noted that the location of such atoms is ambiguous and their positions cannot be uniquely located by local stereochemistry.

The "ring-laddering" principle has precedent in both alkali

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metal phosphide[2a,g,3,4] and amide[12−17] chemistry. However, unsolvated stepped ladder frameworks are still rare and only very few examples containing M−P ladders without classical Lewis bases attached to the metal centers have been structurally characterized until today. As an example, the molecular structure of $LiP(SiMe_3)_2$ features a ladder-type arrangement with six Li−P rungs in length.[1f] Another example is previously reported polymeric $[KP(H)^tBu_3Mes]_x$, exhibiting infinitely extended stepped ladders in the solid state.[4] Among the Lewis base adducts of M−P ladders, the four-step Li−P ladders of general composition $Li_4(\mu_2\text{-}PR_2)_2(\mu_3\text{-}PR_2)_2(THF)_2$ (R = $SiMe_3$;[1j] R = ^tBu[1g,n]) need to be mentioned, as well as the infinitely extended K−P ladders of AA′BB′ type composed of different base adducts of $KP(^tBu)Ph$[2a] and the twisted ladder of polymeric $[(THF)LiP(H)Cy]_x$ (Cy = Cyclohexyl).[1o]

It is noteworthy that only one resonance is observed in the ^{31}P NMR spectrum of **1** in both benzene and tetrahydrofuran, indicating that the solution structure of **1** might be different from its structure in the solid state.

Our work demonstrates that the structural motifs found in molecular species of heavier congeners of alkali metal phosphides vary widely, depending both on the steric bulk of the phosphide ligand[3,4] employed and on the size of the alkali metal cation.[7] We can show that, in the case of the sterically congested potassium phosphide derivative **1**, oligomerization can be controlled by (a) use of a bulky R group on phosphorus and (b) saturating the remaining coordination sphere of the potassium cation by “solvating” through π interaction. As a consequence, the metal−ligand contacts are limited to four steps, thereby forming a structural motif that is a section of an extended K−P ladder, as opposed to a more extended connectivity in the case of polymeric $[KP(H)^tBu_3Mes]_x$. Finally, the molecular structure of the potassium derivative **1** demonstrates how a slight change of the size of the metal (from rubidium to potassium) causes a dramatic change of the overall molecular structure, employing the same phosphide ligand.

Experimental Section

The compounds described below were handled under nitrogen using Schlenk double-manifold, high-vacuum, and glovebox (M. Braun, Labmaster 130) techniques. Solvents were dried and physical measurements were obtained by following typical laboratory procedures. $DmpPH_2$ was prepared according to the literature.[8] Potassium hydride was purchased from Aldrich. Solution NMR spectra were recorded on a JEOL JMN-GX 400 instrument.

KP(H)Dmp (1). In the glovebox, addition of a solution of (2,6-dimesitylphenyl)phosphane (1.50 g, 4.3 mmol) in 20 mL of tetrahydrofuran to a colorless suspension of KH (0.70 g, 17.5 mmol) in 10 mL of tetrahydrofuran caused a slow color change to orange. The reaction mixture was stirred for 14 h and centrifuged. The solvent was removed, and the residues were extracted with toluene and centrifuged. The resulting orange-red solution was cooled to −30 °C, which resulted in almost complete crystallization of **1**. Removal of the mother liquor followed by drying under vacuum gave **1** as yellow-orange microcrystalline material (0.83 g, 50%). Alternatively, KP(H)Dmp can be prepared in similar yield using potassium metal instead of potassium hydride. Analytically pure complex **1** is completely insoluble in hexanes but soluble in aromatic solvents and in tetrahydrofuran. Anal. Calcd for $C_{24}H_{26}KP$: C, 74.96; H, 6.81. Found: C, 75.21; H, 7.08. ^1H NMR (C_6D_6, 400 MHz, 25 °C): δ 1.63 (d, $^1J_{P-H}$ = 182 Hz, 1H), 2.21 (s, 12H), 2.24 (s, 6H), 6.80 (d, $^3J_{H-H}$ = 7 Hz, 2H), 6.83 (s, 4H), 6.94 (t, $^3J_{H-H}$ = 7 Hz, 1H). ^1H NMR (C_4D_8O, 400 MHz, 25 °C): δ 1.61 (d, $^1J_{P-H}$ = 178 Hz, 1H), 2.13 (s, 12H), 2.25 (s, 6H), 6.27 (d, $^3J_{H-H}$ = 7 Hz, 2H), 6.35 (t, $^3J_{H-H}$ = 6 Hz, 1H), 6.78 (s, 4H). $^{31}P\{^1H\}$ NMR (C_6D_6, 161.9 MHz, 25 °C): δ −130.5 (d, $^1J_{P-H}$ = 182 Hz). $^{31}P\{^1H\}$ NMR (C_4D_8O, 161.9 MHz, 25 °C): δ −123.8 (d, $^1J_{P-H}$ = 178 Hz). IR (Nujol): 2310 m, 2244 m, 1732 w, 1722 w, 1605 m, 1574 m, 1249 m, 1176 w, 1109 w, 1090 m, 1040 s, 880 w, 847 s, 785 s, 721 s, 590 m, 575 m, 548 w cm^{-1}.

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Supporting Information Available: Tables listing detailed crystallographic data, atomic positional parameters, bond lengths and angles, and thermal parameters (9 pages). An X-ray crystallographic file, in CIF format, is available on the Internet only. Ordering and access information is given on any current masthead page.

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