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[8] The MS/MS spectra of $\mathbf{Mo_3}$ (*m*/*z* 690) exhibit peaks at *m*/*z* 448 and 432, which correspond to the negative ions of $[Mo_3O_{10}]$ and $[Mo_3O_9]$, respectively, suggesting that the proposed structure of $\mathbf{Mo_3}$, $[\{Mo(O)_2\}_3(\mu_3\text{-}O)(\mu_2\text{-}O)_3]^{2-}$, is appropriate.

[9] ^{95}Mo NMR experiments of **3** have been performed both in CD_3OD and CD_2Cl_2 at room temperature. Although the spectrum in CD_2Cl_2 shows three signals with nearly equal intensity at $\delta = 30.0$ ($I = 78\,\%$), 86.6 ($I = 100\,\%$), and 167.2 ($I = 89\,\%$)—supporting preservation of the solid-state structure—the spectrum in CD_3OD is much more complicated and gives five broad signals with different intensities at $\delta = -9.0$ ($I = 53\,\%$), 50.8 ($I = 100\,\%$), 95.5 ($I = 56\,\%$), 113.5 ($I = 51\,\%$), and 155.3 ($I = 43\,\%$). The results of ^{95}Mo NMR and ESI-MS experiments (the observation of the envelopes for $\mathbf{3_{im}}$ (*m*/*z* 809) and $\mathbf{3_{OMe}}^+$ (*m*/*z* 953)) suggest that in MeOH, cluster **3** exists as equilibrium mixtures [Eqs. (1), (2)]. These equilibria, which lie far to the right, may be reached very quickly. The species $[(Cp^*Rh)_2Mo_4O_{15}]^{2-}$ in Equation (2), which was not detected by ESI-MS, may react with further MeOH to establish a new equilibrium.

[10] The MS/MS spectra of $\mathbf{3_{im}}$ (*m*/*z* 809) exhibit peaks at *m*/*z* 778, 685, 587, 399, and 368, which correspond to the negative ions of $[Cp^*RhMo_3O_8(OMe)_4]$, $[Cp^*RhMo_3O_8(OMe)]$, $[Cp^*RhMo_2O_4(OMe)_3]$, $[Cp^*RhMoO_2(OMe)]$, and $[Cp^*RhMoO_2]$, respectively. The MS/MS spectra of $\mathbf{3_{OMe}}^+$ (*m*/*z* 953) exhibit peaks at *m*/*z* 763, 732, 588, 541, and 540, which correspond to the negative ions of $[Cp^*RhMo_3O_9(OMe)_3]$, $[Cp^*RhMo_3O_9(OMe)_2]$, $[Cp^*RhMo_2O_6(OMe)_2]$, $[Cp^*RhMo_2O_5(OMe)]$, and $[Cp^*RhMo_2O_3(OMe)_2]$, respectively. These results indicate that the proposed structures of $\mathbf{3_{im}}$ and $\mathbf{3_{OMe}}^+$ shown in Scheme 1 are appropriate.

[11] Scan range: 674.9 – 705.1, 794.9 – 825.1, and 936.9 – 967.1 amu (atomic mass unit); step size: 0.2 amu; dwell time per step: 0.001 s; scan time: 0.47 s. The envelope of the molecular ion of **3** at *m*/*z* 1677 was not selected in the rapid scanning experiments because the intensity was too weak.

[12] We have conducted a reaction of **2** with **3** in MeOH at room temperature in order to check whether there are routes for the formation of $\mathbf{3_{OMe}}^+$ through $\mathbf{3_{im}}$, which would account for the reduction in intensity of $\mathbf{3_{im}}$ between ca. 8 – 14 s (Figure 2e). However, $\mathbf{3_{im}}$ does not react with the dimolybdate or any heavier species produced from **2** in MeOH; intensity changes in the envelopes at *m*/*z* 809 and 953 are not observed during the reaction of **2** with one equivalent of **3**. Furthermore, the fragmentation patterns of $\mathbf{3_{im}}$ are different from that of $\mathbf{3_{OMe}}^+$,[10] suggesting that there is no relationship between $\mathbf{3_{im}}$ and $\mathbf{3_{OMe}}^+$. Therefore, we believe that the existence of the intermediate $\mathbf{3_{im}}$ is essential.

[13] The spectrum after 18 s of reaction is essentially identical to that of **3** (Figure 1b).

The First Bismuth Phosphide Complex: $[Li(thf)_4]^+[\{(tBuP)_3\}_2Bi]^-$**

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Although amide[1] and imido[2] complexes of the heavier Group 15 elements (Sb and Bi) have been reported in recent years, for the related phosphorus compounds (containing R_2P^- or RP^{2-} groups) only those of Sb have appeared in the literature.[3] Moreover, the only examples of complexes containing Bi–P bonds to be structurally characterized are Bi^{III} halides in which neutral phosphane ligands coordinate the metal centers.[4] In recent studies of the reactions of $[E(NMe_2)_3]$ (E = As, Sb) with primary phosphido alkali metal complexes ([RPHM]; M = Li, Na), we showed that heterocyclic anions of the type $[(RP)_nE]^-$ are generated[5] prior to the ultimate formation of Zintl compounds containing E_7^{3-} ions.[6] Prompted by the absence of structurally characterized Bi phosphides, which would have the potential for the formation of elusive polyatomic Bi_5^{3-} and Bi_3^{3-} ions by this low-temperature route,[7] we decided to investigate the

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analogous reactions of $[Bi(NMe_2)_3]$. We report here the synthesis and structural characterization of the first bismuth phosphide complex $[Li(THF)_4][Bi\{(tBuP)_3\}_2]$ (**1**).

The reaction of $[Bi(NMe_2)_3]$ with $[tBuPHLi]$ (ratio 1:3) in THF/hexane gives metallic Bi and a yellow solution at room temperature. Prolonged storage of the filtrate (−30 °C) gives yellow crystals of **1** (see Experimental Section). Although moderately stable in solution, the complex proved to be too thermally unstable to isolate and therefore could not be characterized satisfactorily by standard spectroscopic and analytical means. Room-temperature ^{31}P NMR spectra of fresh and aged reaction solutions show that **1** and $[tBuP]_4$ (s, $\delta = -57.0$) are the major products. The formation of the latter suggests that the reaction involved is similar to that occurring between $[E(NMe_2)_3]$ (E = As, Sb) and primary phosphidolithium complexes.[5, 6] A surprising feature of the $[tBuP]_3^{2-}$ ligands of **1** is that they appear as two double doublets at $\delta = -69.8$ and -108.9 (ratio 2:1) rather than the anticipated doublet and triplet (as is observed for $[\{(tBuP)_3As\}]^-$ [5]).[8] This ABX pattern is consistent with an essentially static structure for the $[Bi\{(tBuP)_3\}_2]^-$ ion in solution, in which the axial and equatorial P centers [P(A,B)] are rendered marginally inequivalent and couple separately to the central P atom [P(X)]. The lack of Berry pseudorotation in the $[Bi\{(tBuP)_3\}_2]^-$ ion (even up to 60 °C in THF) presumably stems from the steric congestion of the *t*Bu groups of the terminal P centers which would arise in a square-base pyramidal intermediate (Figure 1).[9]

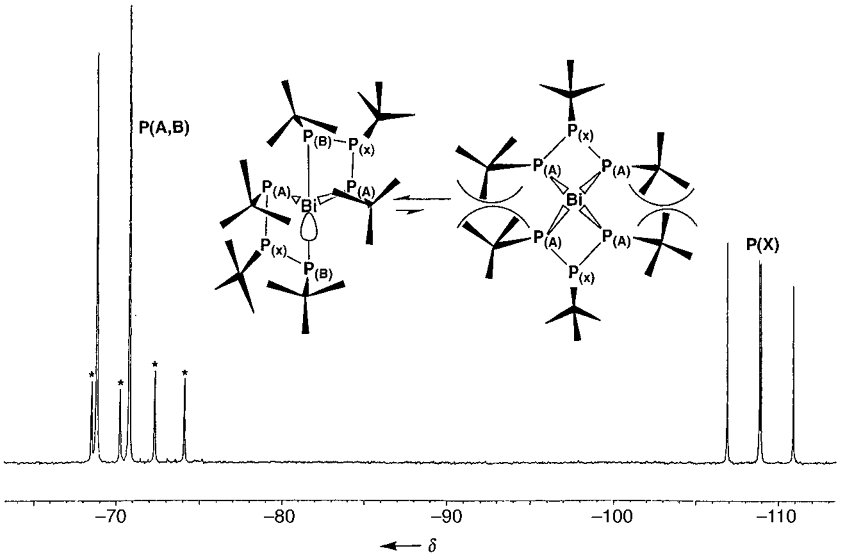

Figure 1. ^{31}P NMR spectrum of fresh and aged reactions mixtures from $[Bi(NMe_2)_3]$ and $[tBuPHLi]$ (50:50 in $[D_8]$THF, +25 °C), as well as a representation of the Berry pseudorotation (see text for details).

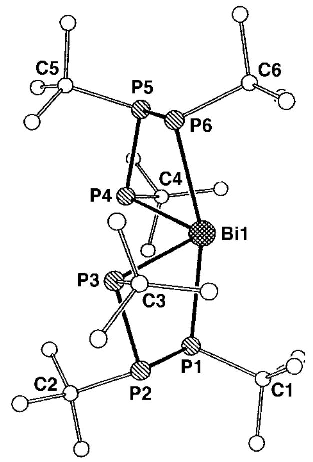

Figure 2. Structure of the anion of **1**. H atoms are omitted for clarity. Key bond lengths [Å] and angles [°]: Bi1–P1 2.911(3), Bi1–P6 2.885(3), Bi1–P3 2.630(4), Bi1–P4 2.651(3), P1–P2 2.177(5), P5–P6 2.174(5), P2–P3 2.240(5), P4–P5 2.236(5); P1-Bi1-P3 76.12(10), P4-Bi1-P6 75.78(11), P1-Bi1-P6 162.10(11), P1-Bi1-P4 91.50(11), P3-Bi1-P6 90.67(10), P1-P2-P3 101.6(2), P4-P5-P6 101.1(2).

The low-temperature X-ray crystal structure of **1**[10] reveals that the complex has an ion-separated structure, $[Li(thf)_4]^+[Bi\{(tBuP)_3\}_2]^-$. The anion of **1** consists of a Bi^{III} center which is coordinated by two crystallographically independent $[tBuP]_3^{2-}$ ions (Figure 2). The resulting pseudo-trigonal bipyramidal geometry of the metal center (10e), whose vacant coordination site is characteristic of a stereochemically active metal lone pair, is comparatively rare for Bi^{III}; here the high Lewis acidity of the metal center almost always leads to an increase in coordination number above four,[11] and only a few examples of discrete Bi^{III} anions having a pseudo-trigonal bipyramidal geometry have been structurally characterized.[12] More significantly, it was only relatively recently that the synthesis of the first binary phosphide of Bi^{III} itself (BiP) was realized[13] and there are no reported examples of molecular species containing bonds between anionic P centers and Bi. This situation presumably reflects the generally high thermal instability of these species (as is the case with solid **1**).

Within the anion of **1**, the axial P–Bi bonds (Bi1–P1 2.911(3), Bi1–P6 2.885(3) Å) are considerably longer than the equatorial ones (Bi1–P3 2.630(4), Bi1–P4 2.651(3) Å). Overall, these bond lengths are similar to those found in structurally characterized complexes between Bi^{III} halides and neutral phosphanes (range 2.654(8)–3.090(3) Å), in which the Bi cations are all six-coordinate.[4] The adoption of a pseudo-trigonal bipyramidal geometry for the $[Bi\{(tBuP)_3\}_2]^-$ ion and the large ionic radius of Bi^{III} results in considerable strain in the heterocyclic P_3Bi ring units. This is most apparent in the significant expansion of the angles at the central P atom of the $[tBuP]_3^{2-}$ ligands (mean 101.4°) compared to that occurring in the $[(tBuP)_3As]^-$ ring of $[\{(tBuP)_3As\}Li(tmeda)(thf)]$ (91.4(2)°),[5] and in the irregular pattern of P–P bond lengths found in these units. These bond lengths mirror the extent of interaction of the terminal P centers of the $[tBuP]_3^{2-}$ ligands with the Bi atom; the P–P bonds to the axial P centers (P2,5–P1,6, mean 2.176(5) Å) are significantly shorter than those made with the equatorial P atoms (P2,5–P3,4, mean 2.238(5) Å; cf. ca. 2.20 Å for both the P–P bonds in $[(t\mathrm{BuP})_3\mathrm{As}]^-$[5]).

In summary, studies of the reaction of $[\mathrm{Bi(NMe_2)_3}]$ with $[t\mathrm{BuPHLi}]$ have led to the structural characterization of the first bismuth phosphide complex. We have found no evidence so far for the formation of Zintl compounds containing Bi_n^{x-} ions in this system. However, by using appropriate ligands (such as cryptands) we hope to stabilize these potential products in future.

Experimental Section

1: To a solution of *t*BuPHLi (6.0 mmol) in THF (4 mL)/hexane (5 mL) at −78 °C was added dropwise a solution of $\mathrm{Bi(NMe_2)_3}$ (2.0 mmol, 0.8 mL, 2.5 mol L^{-1} in THF). The mixture was stirred (3 h), giving a deep red solution and considerable deposition of Bi metal. The mixture was allowed to warm slowly to room temperature. At about −20 °C the color began to change and a yellow solution was formed at room temperature. The mixture was stirred (15 min) and then filtered slowly through a thick bed of celite. Storage of the solution for up to four months gave **1** in low yield as large yellow crystals. Attempts to isolate the complex resulted in decomposition into a black solid. However, ^{31}P NMR spectra (101.256 MHz +25 °C, rel. to 80% $\mathrm{H_3PO_4/D_2O}$) obtained on several fresh and aged reaction solutions (50:50 [D$_8$]THF) show that **1** is always formed as the major product together with $[t\mathrm{BuP}]_4$ (only minor by-products are observed in the range $\delta = 0$ to -50). $J_{\mathrm{AX}} = 195.9$ Hz, $J_{\mathrm{BX}} = 205.8$ Hz (error = ±1.7 Hz). The interdependence of the two resonances was confirmed by ^{31}P COSY and *J*-spectrum NMR experiments. Although heating of the solution results in broadening of the resonances, no coalescence occurs in the range 25–60 °C.

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[10] Crystal data for **1**; $\mathrm{C_{40}H_{86}BiLiO_4P_6}$, $M_r = 1032.83$, monoclinic, space group $P2_1/n$, $Z = 4$, $a = 11.727(2)$, $b = 17.710(3)$, $c = 26.131(4)$ Å, $\beta = 102.268(4)°$, $V = 5303.2(15)$ Å^3, $\rho_{\mathrm{calcd}} = 1.292$ Mg m^{-3}, $F(000) = 2136$, $\mu(\mathrm{Mo_{K\alpha}}) = 3.538$ mm^{-1}, $T = 223(2)$ K. The crystal ($0.30 \times 0.30 \times 0.15$ mm), mounted in inert oil, diffracted weakly at high angle; 7316 data were collected on a Siemens *P*4 diffractometer (θ range 1.79–21.00°). The structure was solved by direct methods. Relatively high displacement parameters for the atoms of the THF ligands indicated some conformational disorder and three carbon atoms were each resolved into two components of about 0.5 site occupancy; chemically equivalent bonds within the ligands were constrained to be equal within an esd of 0.01 Å. Hydrogen atoms of the methylene and methyl carbon atoms were included in idealized positions and assigned $U_{\mathrm{iso}} =$ 1.2 and 1.5 U_{eq} of the parent carbon atom, respectively. Anisotropic thermal parameters were applied to all the fully occupied non-hydrogen atoms. Full-matrix least-squares refinement with 5696 independent absorption corrected data ($R_{\mathrm{int}} = 0.0884$, $T_{\mathrm{max}} = 0.756$, $T_{\mathrm{min}} = 0.327$) on F^2 with 467 parameters. Final $R1 = 0.0654$ $[I > 2\sigma(I)]$ and $wR2 = 0.1423$ (all data). Largest peak and hole in the final difference map 1.063 and −1.270 e Å^{-3}. Programs used see reference [14]. 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-114097. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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