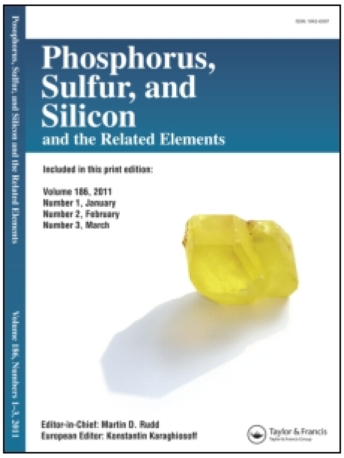

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CLEAVAGE OF *cyclo*-(<i>t</i>BuSb)$_4$ WITH Li, Na OR K AND FORMATION OF Sb$_7^{3-}$ BY REACTIONS OF PRIMARY STIBINES WITH BuLi OR K

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CLEAVAGE OF *cyclo*-(^{t}BuSb)$_4$ WITH Li, Na OR K AND FORMATION OF Sb_7^{3-} BY REACTIONS OF PRIMARY STIBINES WITH BuLi OR K

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Novel reactions of cyclo-*(^{t}BuSb)$_4$ and RSbH$_2$ (R=C$_6$H$_5$, 2,4,6-(CH$_3$)$_3$C$_6$H$_2$ or [(CH$_3$)$_3$Si]$_2$CH) with alkali metals or n-BuLi are described.*

Keywords: Antimony; lithium; potassium; sodium; Zintl anions

(^{t}BuSb)$_4$ is known to react with Na or K and pentamethyldiethylenetriamine (L) with formation of [M(L)$_n$(^{t}Bu$_4$Sb$_3$)] (M = Na, K; n = 1, 2) or [K(L)(^{t}Bu$_3$Sb$_2$].[1,2] Red crystals of the novel antimonide [(^{t}Bu$_2$Sb)K(L)]$_n$ (**1**) are obtained in 15% yield by reacting (^{t}BuSb)$_4$ with K in boiling tetrahydrofuran (thf) for 2.5 h and addition of L. The complexes [Li(L′)$_2$][*t*-Bu$_4$Sb$_3$] (**2**), [Na(L′)(thf)(^{t}Bu$_4$Sb$_3$)] (**3**), [Na(L′)$_2$(^{t}Bu$_4$Sb$_3$)] (**4**) [L′ = tetramethylethylenediamine] form under similar conditions.

The stibines RSbH$_2$ (R = Ph, 2,4,6-Me$_3$C$_6$H$_2$, (Me$_3$Si)$_2$CH) react with BuLi in thf and L′ or with Na or K and L in liquid NH$_3$ to give [Sb$_7$Li$_3$(L′)$_3$] (**5**) or [Sb$_7$M$_3$(L)$_3$] [M = Na, (**6**), K(**7**)] in 50–60% yield. **1**–**7** were characterized by single crystal x-ray diffraction. Crystals of **1** contain a zigzag chain of alternating Sb and K atoms. The ^{t}Bu$_4$Sb$_3^-$ ion is noncoordinating in **2**. It is coordinated to the Na centers through the terminal Sb atoms in **3** and through the central Sb atom in **4**. The Zintl compounds **5**, **6**, and **7** contain the well known nortricyclane framework of the Sb_7^{3-} ion.[3,4]

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