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Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713618290>

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Online publication date: 12 August 2010

To cite this Article Breunig, Hans J. and Ghesner, Mihaiela E. (2004) 'CLEAVAGE OF $\text{cyclo}-(\text{BuSb})_4$ WITH Li, Na OR K AND FORMATION OF Sb_7^{3-} BY REACTIONS OF PRIMARY STIBINES WITH BuLi OR K', *Phosphorus, Sulfur, and Silicon and the Related Elements*, 179: 4, 971 – 972

To link to this Article: DOI: 10.1080/10426500490429293

URL: <http://dx.doi.org/10.1080/10426500490429293>

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CLEAVAGE OF *cyclo*-(*t*BuSb)₄ WITH Li, Na OR K AND FORMATION OF Sb₇³⁻ BY REACTIONS OF PRIMARY STIBINES WITH BuLi OR K

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(Received August 18, 2003; accepted October 3, 2003)

*Novel reactions of cyclo-(*t*BuSb)₄ and RSbH₂ (R=C₆H₅, 2,4,6-(CH₃)₃C₆H₂ or [(CH₃)₃Si]₂CH) 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*Bu₄Sb₃)] (M = Na, K; n = 1, 2) or [K(L)(*t*Bu₃Sb₂)]^{1,2}. Red crystals of the novel antimonide [(*t*Bu₂Sb)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*-Bu₄Sb₃] (**2**), [Na(L')(thf)(*t*Bu₄Sb₃)] (**3**), [Na(L')₂(*t*Bu₄Sb₃)] (**4**) [L' = tetramethylethylenediamine] form under similar conditions.

The stibines RSbH₂ (R = Ph, 2,4,6-Me₃C₆H₂, (Me₃Si)₂CH) react with BuLi in thf and L' or with Na or K and L in liquid NH₃ to give [Sb₇Li₃(L')₃] (**5**) or [Sb₇M₃(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*Bu₄Sb₃⁻ 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 Sb₇³⁻ ion.^{3,4}

We thank Dr. Enno Lork for the supervision of the x-ray structure analyses and the Deutsche Forschungsgemeinschaft for financial support.

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