

## Supersilylverbindungen des Phosphors, V [1].

### Darstellung, Struktur und Reaktivität des Pentaphosphids $(t\text{Bu}_3\text{Si})_3\text{P}_5\text{Na}_2$ und des Pentaphosphans $(t\text{Bu}_3\text{Si})_3\text{P}_5$ [2]

Supersilyl Compounds of Phosphorus, V [1].

Preparation, Structure, and Reactivity of the Pentaphosphide  $(t\text{Bu}_3\text{Si})_3\text{P}_5\text{Na}_2$  and the Pentaphosphane  $(t\text{Bu}_3\text{Si})_3\text{P}_5$  [2]

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The orange THF adduct  $(t\text{Bu}_3\text{Si})_3\text{P}_5\text{Na}_2(\text{THF})_4$  of the pentaphosphide  $(t\text{Bu}_3\text{Si})_3\text{P}_5\text{Na}_2$  (**3**) has been prepared, (i) by protolysis of the tetraphosphide  $(t\text{Bu}_3\text{Si})_2\text{P}_4\text{Na}_2(\text{THF})_n$  (**2**) with  $\text{CF}_3\text{CO}_2\text{H}$  in THF (molar ratio 2 : 1), (ii) by dissolving crystalline **2** in toluene, and (iii) by the reaction of  $\text{P}_4$  with  $t\text{Bu}_3\text{SiNa}(\text{THF})_2$  in benzene (molar ratio 1 : 4). According to an X-ray structural analysis, the THF adduct of **3** contains a  $\text{P}_3$  ring with two  $\text{PNa}(\text{Si}t\text{Bu}_3)$  substituents in *cis* position and one  $\text{Si}t\text{Bu}_3$  substituent in *trans* position to the former groups. The protolysis of **3** with  $\text{CF}_3\text{CO}_2\text{H}$  leads to the pentaphosphane  $\text{P}_5\text{H}_2(\text{Si}t\text{Bu}_3)_3$  (**9**), the silylation of **3** with  $\text{Me}_2\text{SiCl}_2$  to the pentaphosphane  $\text{P}_5(\text{SiMe}_2)(\text{Si}t\text{Bu}_3)_3$  (**10**), and the oxidation of **3** with  $\text{C}_2(\text{CN})_4$  to the pentaphosphane  $\text{P}_5(\text{Si}t\text{Bu}_3)_3$  (**5**). The structures of **3**, **5**, **9**, and **10** have been assigned from  $^{31}\text{P}$  and  $^{29}\text{Si}$  NMR data. The pentaphosphane **5** contains a hitherto unknown  $\text{P}_5$  backbone of a  $\text{P}_3$  ring anellated with a  $\text{P}_4$  ring.

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