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PREPARATION AND REACTIONS OF PERMETHYLATED CYCLOOLIGOSILANE ALKALI METAL DERIVATIVES

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Abstract A simple way for the synthesis of potassium undecamethylcyclohexasilane is described and discussed.

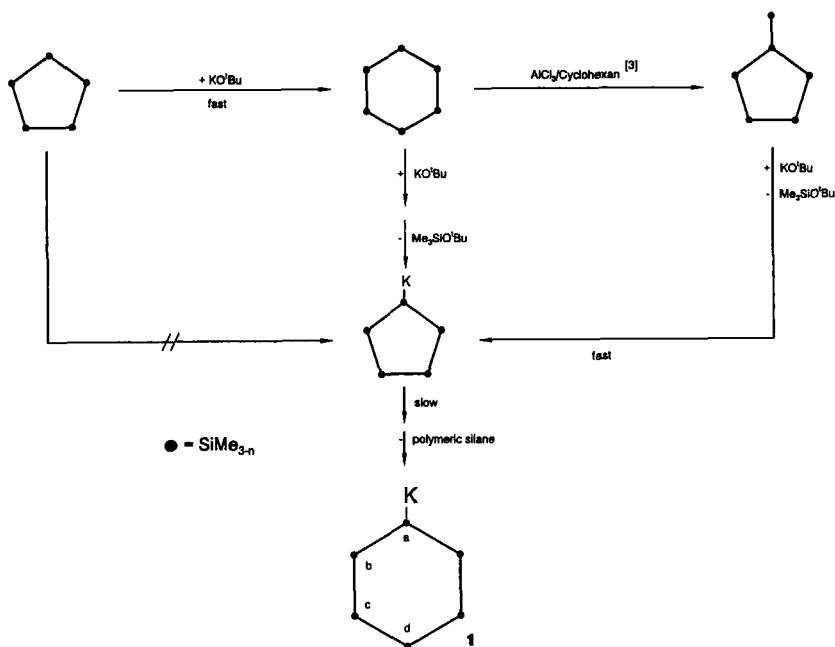
Introduction

The highly reactive potassium undecamethylcyclohexasilane is a suitable starting material for the synthesis of several cyclic silanes. Potassium undecamethylcyclohexasilane **1** has been synthesized by two methods. Both methods have some disadvantages^[1a-b]. On the basis of these facts we tried to develop a simple way to synthesize potassium undecamethylcyclohexasilane.

Synthesis

Potassium undecamethylcyclohexasilane is formed in the reaction of the permethylated cyclohexasilane with potassium t-butanolate in glyme solvents at room temperature over a period of two weeks. This provides a simple access to **1**.

The observed reaction products can be rationalized by a complicated ring-chain-ring-equilibrium. The first step is a Si-Si-bond cleavage followed by a methyl group shift. Trimethyl(t-butoxy)silane is the byproduct. The resulting potassium nonamethylcyclopentasilane reacts slowly to **1**. The byproduct of this step is a polymeric silane. The theoretical yield is nearly 80% of the starting cyclosilane. Practically the reaction of **1**, e.g., with propylchloride yields 65 - 75% propylundecamethylcyclohexasilane. Conversions of decamethylcyclopentasilane and trimethylsilylnonamethylcyclopentasilane with potassium t-butanolate leads only to **1** in the described way. Potassium nonamethylcyclopentasilane is only an intermediate which can not be isolated.



1 reacts with zirconocenedichloride at -78°C to a dark red colored product (mp: $188\text{--}190^{\circ}\text{C}$), which is the first group IV derivative of a cyclic silane. A second substitution step does not occur even in the presence of two equivalents of potassium undecamethylcyclohexasilane.

Si-Si-bonds are formed when **1** is reacted with phenylthiooligosilanes. Alkali metal thiophenolates, which are byproducts in the reaction can be easily removed. Also reaction of triethyl- or tributylstannylundecamethylcyclohexasilane with alkali metal silanes leads to the formation of Si-Si-bonds. In contrast to the related alkali metal halide elimination reactions of organohalooligosilanes, transmetalations are never observed in these reactions. Examples are described in [2].

References

- [1] a) A.L.Allred, R.T.Smart, D.A.Van Beek; *Organometallics* **11**(1992)4225
 b) E.Hengge, P.K.Jenkner; *J.Organomet.Chem.* **314**(1986)1;
 E.Hengge, P.K.Jenkner, P.Gspaltl, A.Spielberger; *Z.Anorg.Allg.Chem.* **560**(1988)17
- [2] F.Uhlig, B.Stadelmann, A.Zechmann, P.Lassacher, H.Stüger, E.Hengge;
Phosphorus&Sulfur 1994 in press
- [3] M.Ishikawa, M.Kumada; *J.C.S.,Chem.Comm.* (1969)567