

This article was downloaded by:

On: 28 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



## 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>

### Cyclic Silylhydroxylamines and 1,3-Diaza-2-Silacyclopentane - Lithium Derivatives and Reactions -

Friedhelm Diedrich; Christina Ebker; Uwe Klingebiel

**To cite this Article** Diedrich, Friedhelm , Ebker, Christina and Klingebiel, Uwe(2001) 'Cyclic Silylhydroxylamines and 1,3-Diaza-2-Silacyclopentane - Lithium Derivatives and Reactions -', *Phosphorus, Sulfur, and Silicon and the Related Elements*, 169: 1, 253 – 256

**To link to this Article:** DOI: 10.1080/10426500108546636

**URL:** <http://dx.doi.org/10.1080/10426500108546636>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

## Cyclic Silylhydroxylamines and 1,3-Diaza-2-Silacyclopentane – Lithium Derivatives and Reactions –

FRIEDHELM DIEDRICH, CHRISTINA EBKER  
and UWE KLINGEBIEL\*

*Institute of Inorganic Chemistry, Georg-August-University Goettingen,  
Tammannstr. 4, D-37077 Goettingen, Germany*

Lithium derivatives of silylhydroxylamines are used for more than thirty years. Now we are able to present the first crystal structures. Lithium is bonded side on and end on in these silylhydroxylamides. Depending on the reaction conditions and the bulkiness of the substituents dimeric, trimeric, and tetrameric oligomers are found. Fluoro-functional bis(silyl)hydroxylamines are excellent precursors for rings. By-products of the syntheses of bis(silyl)hydroxylamines are N, bis(silyl)ethylendiamines, which are easily cyclized forming 1,2-diaza-2-silacyclopentanes. Reactions and X-ray analyses are discussed.

**Keywords:** Silylhydroxylamines; Lithium silylhydroxylamides; Diaza-silacyclopentanes; X-ray structures

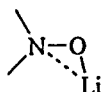
### Lithium Derivatives of Bis(silyl)hydroxylamines

O-lithium derivatives of silylhydroxylamines are obtained in the reaction of BuLi with N,O-bis(silyl)hydroxylamines.<sup>[1]</sup> This includes an anionic 1,2-silyl group migration from oxygen to the nitrogen atom. Lithium is bonded side on and end on in these compounds. Depending

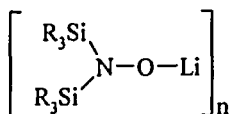
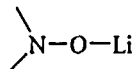
\* Corresponding author. Tel.: 0551/39-3052 – Fax: 0551/39-3373 – E-Mail: uklinge@gwdg.de

on the reaction conditions and the bulkiness of the substituents dimeric, trimeric, and tetrameric oligomers are found, e.g.<sup>[2,3]</sup>

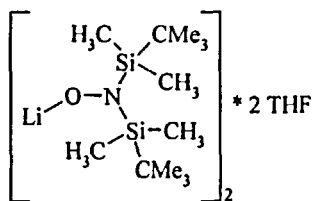
side on



end on



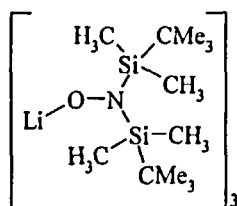
$$n = 2, 3, 4$$



$$\text{O-N} = 147.4 \text{ pm}$$

$$\Sigma \text{N} = 359.9^\circ$$

$$\Sigma \text{Li} = 360.0^\circ$$

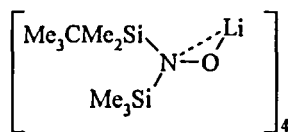


$$\text{Si-N} = 173.0 - 174.5 \text{ pm}$$

$$\Sigma \text{N} = 359^\circ - 359.6^\circ$$

$$\Sigma \text{O} = 360.0^\circ$$

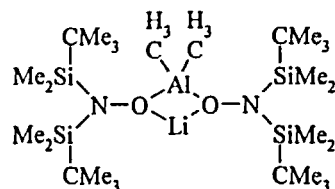
The first aluminium containing silylhydroxylamine was isolated in the reaction of O-lithium-N,N-bis(tert.-butyldimethylsilyl)hydroxylamide with chlorodimethylalan.



$$\text{O-N} = 150.0 \text{ pm}$$

$$\Sigma \text{N} = 352.1^\circ$$

$$\Sigma \text{Li} = 356.5^\circ$$



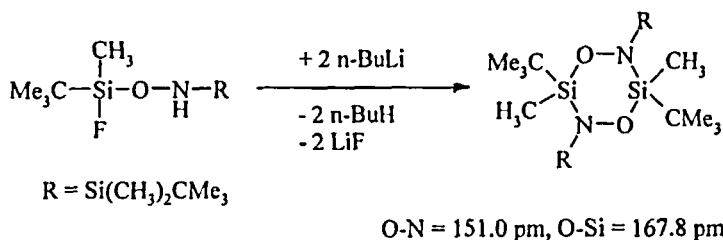
$$\text{Si-N: } 173.8-174.6 \text{ pm}$$

$$\text{O-N: } 148 \text{ pm}$$

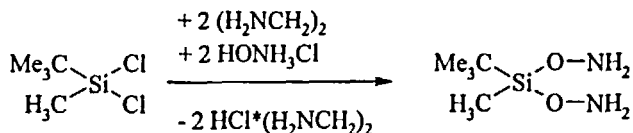
$$\Sigma \text{N} = 355.19^\circ - 375.75^\circ$$

Cyclic Silylhydroxylamines

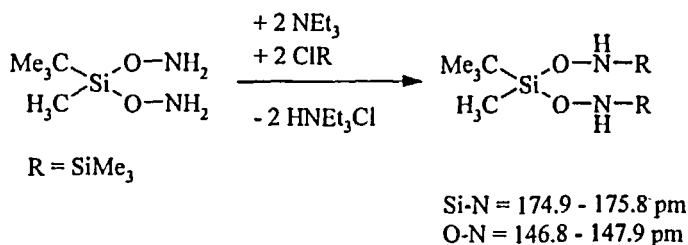
Fluorofunctional bis(silyl)hydroxylamines are precursors for rings, e.g.

Bis(hydroxylamino)silane

The first bis(hydroxylamino)silanes, obtained in the following reaction.<sup>[4]</sup>

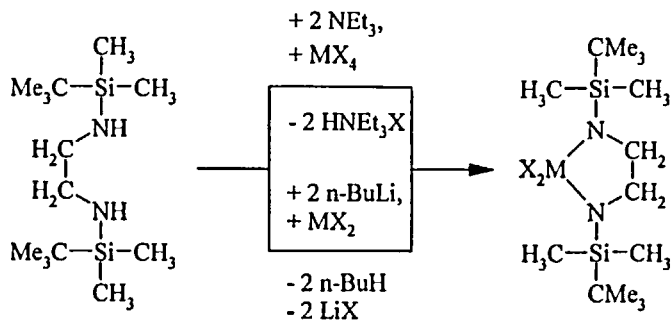


The bis(hydroxylamino)silanes can be used for the synthesis of cyclic and acyclic O,O-bis(silylhydroxylamino)silanes, e.g.



1,3-Diaza-2-silacyclopentanes

By-products of the syntheses of bis(silyl)hydroxylamines are N,N-bis(silyl)ethyldiamines, which are easily cyclized forming 1,2-diaza-2-silacyclopentanes.<sup>[4]</sup>



The following derivatives were isolated:  $\text{MX}_2$ :  $\text{SiBr}_2$ ;  $\text{SiCl}_2$ ;  $\text{SiF}_2$ ;  $\text{Si(H)Cl}$ ;  $\text{Si(OMe)}_2$ ;  $\text{Si(NH}_2)_2$ ;  $\text{Si(F)OSiR}_3$ ;  $\text{Sn}$ .

**ACKNOWLEDGMENTS**

This work has been supported by the *Deutsche Forschungsgemeinschaft* and the *Fonds der Chemischen Industrie*.

**References**

- [1] P. Boudjouk and R. West, *Intra-Sci. Chem Rep* **1973**, 7, 65–82.
- [2] F. Diedrich, U. Klingebiel, and M. Schäfer, *J. Organomet. Chem.* **588**, 242 (1999).
- [3] F. Diedrich, U. Klingebiel, F. Dall'Antonia, Ch. Lehmann, M. Noltemeyer, and T. R. Schneider, in preparation.
- [4] F. Diedrich, U. Klingebiel, M. Noltemeyer, and T. R. Schneider, in preparation.