Russian Journal of General Chemistry, Vol. 75, No. 10, 2005, pp. 1674–1675. Translated from Zhurnal Obshchei Khimii, Vol. 75, No. 10, 2005, pp. 1753–1754.

LETTERS TO THE EDITOR

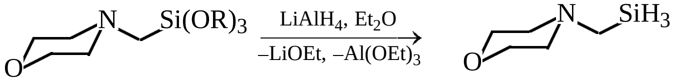
A Procedure for Preparing Morpholinomethylhydrosilane

K. Pypowski*, T. A. Koval', and N. P. Erchak***

* *Universit of Podlasie, Siedlce, Poland*
** *Pushkin Brest State University, Brest, Belarus*

Received February 11, 2005

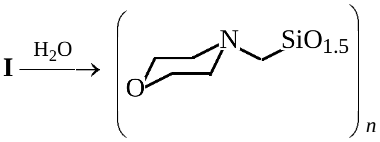
Morpholinomethylhydrosilane **I** is used in the synthesis of zwitterionic hypercoordinated organosilicon compounds with the Si–O–C and Si–S–C bonds [1–5]. It is prepared by reduction of morpholinomethyltrialkoxysilanes **II** with lithium aluminum hydride in diethyl ether.



IIa, IIb **I**

R = OMe (**a**), OEt (**b**).

Synthesis of **I** involves separation of the precipitate by filtration and isolation of the hydrosilane by distillation. The yield of **I** reaches 61% [5]. One of the major factors decreasing the yield of **I** may be its hydrolytic instability. Indeed, complete hydrolysis of the Si–H bonds of hydrosilane **I** with water vapor in the film occurs at room temperature within 5 min. The hydrolysis product of **I**, (morpholinomethyl)silsesquioxane **III**, according to the IR data, contains no Si–H bonds.



III

The hydrolysis of morpholinomethylhydrosilane in moist diethyl ether and THF is also very fast.

To exclude the hydrolysis of the Si–H bonds, we improved the step of the isolation of **I** and performed its distillation directly from the reaction mixture, without separating the precipitate. By so doing, the yield of **I** was increased to ~90%.

The starting (morpholinomethyl)triethoxysilane **IIb** was obtained according to [6].

(Morpholinomethyl)hydrosilane I. A 2.0-g portion of $LiAlH_4$ was added over a period of 30 min with vigorous stirring to a solution of 10.8 g of **IIb** in 100 ml of anhydrous diethyl ether. The mixture was gently refluxed for 8 h and allowed to stand for 16 h at room temperature. The solvent was removed at atmospheric pressure, after which product **I** was distilled at 52°C (18 mm Hg); yield 4.8 g (89%). ^1H NMR spectrum ($CDCl_3$), δ, ppm ($^3J_{HH}$, Hz): 2.16 q (2H, $SiCH_2$, 3.6), 2.44–2.48 m (4H, NCH_2), 3.58 t (3H, SiH_3, 3.6), 3.68–3.72 m (4H, OCH_2) {published data [5]: bp 35°C (11 mm Hg)}. ^1H NMR spectrum (C_6D_6), δ, ppm ($^3J_{HH}$, Hz): 1.92 q (2H, $SiCH_2$, 3.6), 2.21–2.29 m (4H, NCH_2), 3.61–3.67 m (4H, OCH_2), 3.71 t (3H, SiH_3, 3.6).

(Morpholinomethyl)silsesquioxane III. *a.* Three drops of **I** in 0.3 ml of anhydrous diethyl ether were placed on an aluminum foil to obtain as thin layer as possible; the layer was kept in air for 5 min. The resulting colorless amorphous powder was mixed on the foil surface in an inert atmosphere (Ar) with KBr and analyzed by IR spectroscopy. The absorption at 2300–2000 cm^{-1} was absent. The absorption of residual OH groups was observed at 3460 cm^{-1}.

b. A solution of 0.036 g of water in 5 ml of THF was added dropwise with stirring to a solution of

1070-3632/05/7510-1674 ©2005 Pleiades Publishing, Inc.

0.131 g of **I** in 5 ml of anhydrous THF. The solution became turbid, and a finely dispersed colorless precipitate formed. The mixture was allowed to stand at room temperature for 10 min. The solvent was withdrawn with a syringe, and the precipitate was washed with diethyl ether and vacuum-dried. Compound **III** was thus obtained in a quantitative yield (~0.153 g). Degree of polymerization n 6.3 × 10^4. No hydrosilyl groups were detected by IR spectroscopy. Found, %: C 39.05; H 6.68; N 9.02. $C_{10}H_{20}N_2O_5Si_2$. Calculated, %: C 39.47; H 6.58; N 9.21.

Hydrolysis of **I** in diethyl ether was performed similarly.

The IR spectrum (KBr) was recorded on a Nicolet MAGNA-760 spectrometer. The ^1H NMR spectrum was taken on a Varian Mercury-200B spectrometer (200 MHz) in $CDCl_3$, internal reference HMDS.

REFERENCES

1. Erchak, N.P. and Liepins, E.E., USSR Inventor's Certificate no. 1 710 562, 1992, *Byull. Izobret.*, 1992, no. 5.
2. Erchak, N.P. and Liepins, E.E., USSR Inventor's Certificate no. 1 696 440, 1991, *Byull. Izobret.*, 1991, no. 45.
3. Erchak, N. and Ancens, G., Abstracts of Papers, *XVI Int. Symp. on the Organic Chemistry of Sulfur (ISOCS)*, Merseburg, 1994, p. 182.
4. Erchak, N., Ancens, G., Kemme, A., and Lukevics, E., Abstracts of Papers, *XI Int. Symp. on Organosilicon Chemistry*, Montpellier, 1996, report PA 60.
5. Bertermann, R., Biller, A., Kaupp, M., Penka, M., Seiler, O., and Tacke, R., *Organometallics*, 2003, vol. 22, no. 20, p. 4104.
6. Lukevics, E.J., Pestunovich, A.E., and Voronkov, M.G., *Khim. Geterotsikl. Soedin.*, 1968, no. 5, p. 949.