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LETTERS TO THE EDITOR

METHANOLYSIS OF 2-FURYLSILANE

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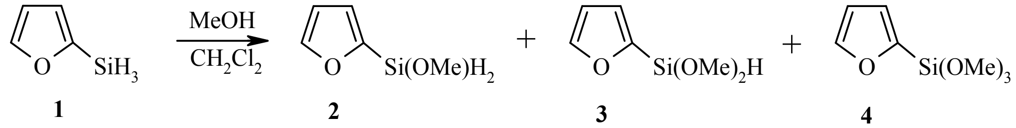
Many organic silicon derivatives [1], in particular, furylsilanes substituted at position 2 [2, 3], have shown significant biological activity. Furylsilanes with therapeutic properties have been synthesized and described [4].

2-Furylhydrosilane is the simplest furylsilane derivative. It was used as a model compound in theoretical (physicochemical) studies of different types of furylsilane derivatives [5–11]. The first seven-coordinate organic silicon compound, too, was obtained from 2-furylsilane [12–14].

This paper describes a new, mild and efficient method of obtaining furylsilane derivatives. The autocatalytic reaction of hydrolysis of 2-furylsilane is known and has been described [8]. For the first time, the reaction of methanolysis of (2-furyl)trihydrosilane (**1**) was performed under new conditions: without catalyst, at room temperature, in dichloromethane solution (100 mg compound **1** in 2 ml CH_2Cl_2 for all molar proportions). Heretofore unavailable new derivatives of 2-furylsilane were obtained: dihydro(2-furyl)methoxysilane (**2**), dimethoxy-(2-furyl)hydrosilane (**3**), and (2-furyl)trimethoxysilane (**4**). The reaction scheme is given below:



Reactions were performed in three different molar proportions of reagents (silane **1**:methanol): (*a*) 1:3.46; (*b*) 1:3.22; (*c*) 1:3.01. The obtained methoxy derivatives **2-4** were analyzed by GC/MS (EI, 70 eV). The results are shown in Table 1. It is interesting that derivatives with unsubstituted hydrogen atoms were obtained even under excess of methanol.

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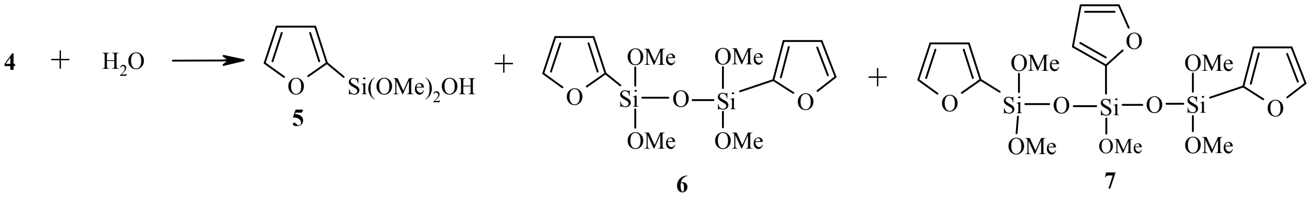
TABLE 1. Results of 2-Furylsilane Methanolysis Reaction

Com-pound	MS, *m/z* (I_{rel}, %)	Yield, %		
		a	*b*	*c*
2	128 $[M]^+$ (26), 97 (57), 68 (5), 59 (100), 53 (18), 45 (35)	—	10.0	18.0
3	158 $[M]^+$ (44), 127 (68), 97 (44), 91 (25), 59 (100)	8.5	17.0	13.0
4	188 $[M]^+$ (62), 173 (46), 161 (39), 127 (75), 90 (100)	91.0	73.0	69.0

Under conditions *a*, tetramethoxysilane (0.5 %), the product of substitution of the furyl part by a molecule of methanol, was obtained.

(2-Furyl)trimethoxysilane (**4**) easily underwent hydrolysis reaction (20 mg compound **4** in 0.2 ml CH_2Cl_2, 100 sec, surface of contact 1.25 cm^2). The reaction did not stop at 2-furyldimethoxysilanol (**5**) and produced mostly 1,1,3,3-tetramethoxy-1,3-di(furyl-2)disiloxane (**6**) and 1,1,3,5,5-pentamethoxy-1,3,5-tri(furyl-2)-trisiloxane (**7**).



GC/MS was performed with a GC-17A (Shimadzu) gas chromatograph equipped with a BPX5 (Phenomenex) column (30 m × 0.25 mm × 0.25 μm) and a QP5050 (Shimadzu) mass spectrometric detector. Helium was used as the carrier gas at a flow rate of 0.7 $ml \cdot min^{-1}$. The injection temperature was 180°C. The column was held at 80°C for 5 min after injection, then programmed at $5°C \cdot min^{-1}$ to 280°C, which was held for 30 min. The detector temperature was 290°C, and the potential difference was 1.5 V. The MS was operated in full-scan (TIC) and selected ion monitoring (SIM) modes with electron impact ionization.

TABLE 2. Results of hydrolysis of 2-(furyl)trimethoxysilane (**4**)

Com-pound	MS, *m*/z (I_{rel}, %)	Yield, %
5	174 $[M]^+$ (6), 173 (9), 77 (10), 76 (13), 45 (13), 36 (37), 32 (51), 28 (100)	0.1
6	330 $[M]^+$ (27), 230 (40), 200 (27), 167 (25), 151 (31), 137 (33), 118 (100)	17.5
7	472 $[M]^+$ (32), 440 (10), 389 (3), 257 (26), 241 (72), 227 (35), 81 (100)	3.5

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