Journal of Organometallic Chemistry 695 (2010) 663–666

Contents lists available at ScienceDirect

Journal of Organometallic Chemistry

journal homepage: www.elsevier.com/locate/jorganchem

Preparation of sila-functional 3-sila-1-thiacyclohexanes

Svetlana V. Kirpichenko *, Aleksander I. Albanov

Irkutsk Institute of Chemistry, Siberian Branch of the Russian Academy of Sciences, 1 Favorsky Str., 664033 Irkutsk, Russia

ARTICLE INFO

Article history:
Received 25 April 2009
Received in revised form 7 July 2009
Accepted 26 November 2009
Available online 2 December 2009

Keywords:
Silicon
Silathiacyclohexanes
Synthesis
Protecting group

ABSTRACT

First sila-functional heterocycles **7** and **9** with Si and S atoms in 1,3-position bearing the reactive X group at silicon (X = *i*-PrO, F) were readily prepared from the corresponding phenyl-protected cycle **6** in good yields *via* Ph–Si bond cleavage by electrophilic reagents. Treatment of *i*-propoxy derivative **7** with $LiAlH_4$ gave heterocycle **8** with Si–H bond.

1. Introduction

Over many years there has been a considerable interest in silathiacycloalkanes, organosilicon heterocyclic compounds containing the Si–$(CH_2)_n$–S (n = 1–3) moiety [1]. Most significant results in this field including some of our own results are given in recent review [1a]. Due to the presence of both elements these compounds exhibit the peculiar and complementary reactivity. To date, all researches have focused on the chemistry of 1,3 or 1,4-silathiacyclohexanes bearing simple alkyl groups at the silicon atom [1a]. Previously we have studied their S-functionalization including conversions into sulfoxides [2a,b], sulfones [2a] and sulfimines [2c,d]. Along the line of our studies, we became interested in a new type of 1,3-silathiacyclohexanes having a labile Si–X bond (X – hydrogen, halogen, etc). They offer many opportunities for nucleophilic substitution reactions at the silicon atom and are the interesting objects for conformational analysis. Several routes to 1,3-silathiacycloalkanes with different ring size are available [1a], with the most recent one involving the photochemical addition of thioacetic acid to dimethyl(chloromethyl)allyl or vinylsilanes followed by solvolysis of the thioacetate adducts and ring forming stage [2a,3a,b]. The considerable drawback of this method for preparation of related sila-functional 3-sila-1-thiacyclohexanes appears to be difficult preparation of the starting silanes. Besides, the presence of a labile functional group such as RO, Si–H or Si–F in the intermediate thioacetates complicates their purification and subsequent transformations under basic conditions. Recently we tried to synthesize 3-fluoro-3-methyl-3-sila-1-thiacyclohexane **9** *via* a common approach from (chloromethyl)methylallylfluorosilane. The latter was prepared from commercially available (chloromethyl)methyldichlorosilane in low overall yield in the two steps [4]. Although the target thioacetate was formed in high yield (78%), its solvolysis was problematic due to sensitivity of the Si–F bond to bases. As a mild base, pyrrolidine was proposed for cleavage of thioacetates [5]. Indeed, reaction of 3-[(chloromethyl)methylfluorosilyl]propylthioacetate with pyrrolidine in acetonitrile gave desired heterocycle **9**, however, in low isolated yield (10%) [6].

Therefore, we chose an alternative protection protocol for such substrates which involves the preparation of precursor stable to synthetic procedure and purification techniques. In organosilicon chemistry one of the most exploited protecting groups is an aryl moiety, especially in synthesis of silanols and silanediols. Removal of the protecting groups by electrophilic reagents allows to remain other Si–C bonds intact [7,8].

Here we described a simple and versatile route to the first 3-sila-1-thiacyclohexanes with reactive X–Si groups based on Si–C(Ph) bond cleavage of phenyl-protected heterocycle **6**.

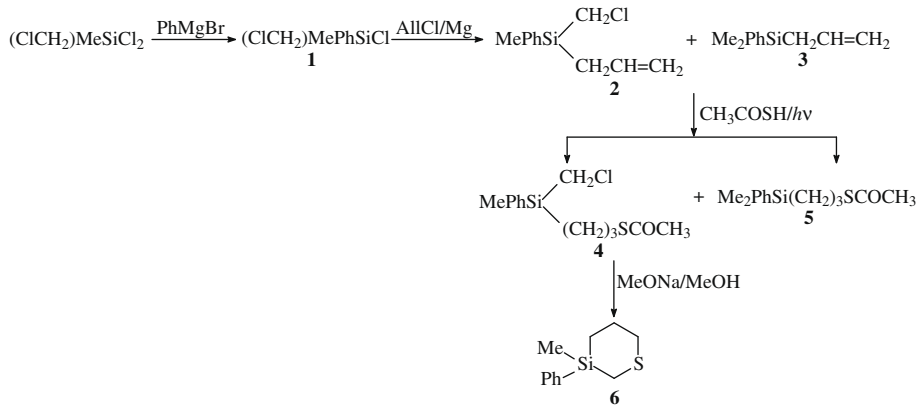
2. Results and discussion

Cyclic compound **6** was prepared in four-step synthesis according to Scheme 1. Thus, treatment of starting (chloromethyl)methyldichlorosilane with phenylmagnesium bromide afforded silane **1** the reaction of which with allylmagnesium chloride resulted in allylsilane **2**.

After vacuum distillation of a poor separable mixture of **2** and **3**, their ratio of 94%:6% was achieved. This mixture was subjected to the addition of thioacetic acid to give thioacetates **4** and **5** sepa-

* Corresponding author. Tel.: +7 3952 426345; fax: +7 3952 419346.
E-mail address: svk@irioch.irk.ru (S.V. Kirpichenko).

0022-328X/$ - see front matter © 2009 Elsevier B.V. All rights reserved.
doi:10.1016/j.jorganchem.2009.11.038



Scheme 1.

rated by column chromatography. Methanolysis of thioacetate **4** followed by the intramolecular ring closure provided phenyl-protected cycle **6** in high yield (74%).

By the action of triflic acid arylsilanes are readily converted into the highly reactive silyl triflates [9] which are useful for further transformations into Si–halogen [10], Si–H [11] and Si–OR [10b,11b] derivatives. In fact, removal of the phenyl group from **6** with triflic acid and subsequent treatment with *i*-propanol/triethylamine gave **7** in good yield (53%). The resulting compound was reduced with lithium aluminum hydride to generate heterocyclic compound **8** with Si–H bond (Scheme 2).

Replacing phenyl group with fluoride can easily be accomplished by treatment with triflic acid/fluorinating agent (HF [12], NaF [10c]). One-step conversion of the Ph–Si bond into a F–Si moiety with use of boron trifluoride-acetic acid complex seems to be more attractive procedure [13]. Reaction of this complex with **6** under mild conditions was successful, producing desired fluoro containing cycle **9** in 35% isolated yield.

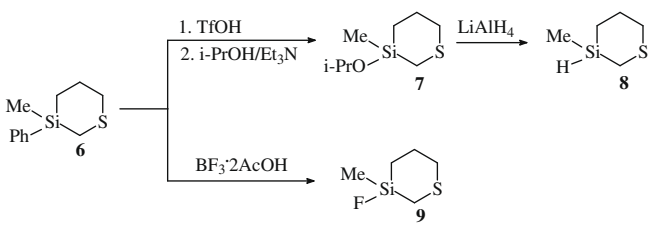
Compounds **7**–**9** were isolated as colorless liquids, whereas cycle **6** was obtained as a clear oil slowly crystallizing on keeping. Compounds **2**–**9** are stable to a wide range of workup, reaction conditions and purification by column chromatography on silica gel (except compound **7**) and can be handled without any precautions.

In conclusion, the above reactions offer the preparative route approach to a wide variety of new types of sila-functional heterocycles, useful for studies of their reactivity and conformational behavior. These two aspects are under current investigations.

3. Experimental

3.1. General comments

All reactions were carried out under an argon atmosphere with magnetic stirring. Triflic acid was purchased from Alfa Aesar and used without further purification. Diethyl ether was distilled from sodium benzophenone ketyl prior to use; *n*-pentane, CH_2Cl_2, *i*-propanol, triethylamine were distilled from CaH_2. Analytical thin layer chromatography and column chromatography were carried out using silica gel plates (60 F-254, Merck) and silica gel 60 (0.063–0.200 mm, ICN Biomedical Inc.), respectively. The ^1H, ^{13}C, ^{19}F and ^{29}Si NMR spectra were recorded on Bruker DPX-400 spectrometer (^1H, 400.1 MHz; ^{13}C, 100.6 MHz; ^{19}F, 376.5 MHz; ^{29}Si, 79.5 MHz). Chemical shifts (ppm) were determined relative to residual $CHCl_3$ (^1H, δ 7.27, $CDCl_3$), internal $CDCl_3$ (^{13}C, δ 77.0, $CDCl_3$), external $CFCl_3$ (^{19}F, δ 0.00, $CDCl_3$), and external TMS (^{29}Si, δ 0.00, $CDCl_3$). Analysis and assignment of the ^1H NMR data were supported by homonuclear (COSY) and heteronuclear (HSQC ^{13}C–^1H, HMBC ^{13}C–^1H) 2D correlation experiments.



Scheme 2.

3.2. Synthesis of compounds ***1***–***9***

3.2.1. (Chloromethyl)methylphenylchlorosilane (***1***)

(Chloromethyl)methylphenylchlorosilane (**1**) was synthesized following the general procedure described in Ref. [14]. Phenylmagnesium bromide, prepared from bromobenzene (39.26 g, 0.25 mol) and powder magnesium (7.20 g, 0.30 g-a) in Et_2O (100 mL), was added to (chloromethyl)methyldichlorosilane (45.79 g, 0.28 mol) in Et_2O (50 mL). The mixture was refluxed 6 h, cooled, diluted with hexane (100 mL) and filtered. Concentration in vacuo followed by rapid distillation from the remaining magnesium salts afforded a liquid product (31.01 g, b.p. 80–123 °C/3 mm Hg) which was redistilled to give **1** (27.14 g, 0.13 mol, 53%), b.p. 78–82 °C/2 mm Hg. Lit.: [12]: b.p. 106–108 °C/8 mm Hg. ^1H NMR ($CDCl_3$): δ 0.87 (s, 3H, *Me*), 3.18 (s, 2H, CH_2Cl), 7.45–7.54 (m, 3H, H_{m+p}), 7.72 (m, 2H, H_o). ^{13}C NMR ($CDCl_3$): δ −1.65 (*Me*), 29.78 (CH_2Cl), 128.23 (C_m), 131.11 (C_p), 133.64 (C_o), 133.91 (C_i). ^{29}Si NMR ($CDCl_3$): δ 11.91.

3.2.2. (Chloromethyl)allylmethylphenylsilane (***2***)

(Chloromethyl)allylmethylphenylsilane (**2**) was prepared by a modified procedure [15]. (Chloromethyl)methylphenylchlorosilane (**1**) (25.10 g, 0.12 mol) in Et_2O (40 mL) was added to a vigorously stirred Grignard reagent, prepared from allyl chloride (13.01 g, 0.17 mol), magnesium powder (4.09 g, 0.17 g-a) in Et_2O (20 mL) at 0 °C. The reaction mixture was then refluxed for 6 h. Hexane (40 mL) was added before quenching with a saturated aqueous NH_4Cl solution at 0 °C. The layers were separated and the aqueous layer was extracted with Et_2O (2 × 20 mL). The combined organic phase was dried ($MgSO_4$), filtered and concentrated under reduced pressure to give the crude product (17.95 g) containing allylsilanes **2** and **3** in a 9:1 ratio (by ^1H NMR data). Vacuum distillation gave **3** (1.27 g, 0.7% yield, 90% purity by ^1H NMR, b.p.

76–77 °C/1 mm Hg) and target allylsilane **2** (15.33 g, 43% yield, 94% purity by ^{1}H NMR), b.p. 87–88 °C/1 mm Hg used without further purification in the next step.

Analytically pure samples of **2** and **3** were obtained by column chromatography (silica gel, hexane).

*Data for **2**:* a colorless oil, R_f = 0.54 (hexane). ^{1}H NMR ($CDCl_3$): δ 0.43 (s, 3H, *Me*Si), 1.94 (ddt, 1H, J_{AB} = 13.8, J = 8.2 and J = 1.3 Hz, Si-C*H*$_A$C), 1.98 (ddt, 1H, SiC*H*$_B$C), 3.00 (d, 1H, J_{AB} = 13.8 Hz, SiC*H*$_A$Cl), 3.09 (d, 1H, SiC*H*$_B$Cl), 4.93 (ddt, 1H, ^{cis}J = 9.9, J = 1.8 and J = 1.3 Hz, =C*H*$_A$), 4.97 (ddt, 1H, $^{trans}J$ = 16,9 Hz, =C*H*$_B$), 5.81 (ddt, 1H, C*H*=), 7.41 (m, 3H, H_{m+p}), 7.56 (m, 2H, H_o). ^{13}C NMR ($CDCl_3$): δ −6.56 (*Me*Si), 20.16 (SiCH_2C), 28.58 (SiCH_2Cl), 114.72 (=CH_2), 128.03 (C_m), 129.89 (C_p), 133.21 (*C*H=), 134.07 (C_o), 134.59 (C_i). ^{29}Si NMR ($CDCl_3$): δ −4.97. Anal. Calc. for $C_{11}H_{15}SiCl$: C, 62.68; H, 7.17; Si, 13.33. Found: C, 62.88; H, 7.27; Si, 13.24%.

*Data for **3**:* a colorless oil, R_f = 0.34 (hexane). ^{1}H NMR ($CDCl_3$): δ 0.37 (s, 6H, *Me*Si), 1.84 (dt, 2H, J = 8.2 and J = 1.9 Hz, SiCH_2C), 4.93 (ddt, 1H, ^{cis}J = 10.1, J = 2.1 and J = 1.3 Hz, =C*H*$_A$), 4.95 (ddt, 1H, $^{trans}J$ = 16.8 Hz, =C*H*$_B$), 5.86 (ddt, 1H, C*H*=), 7.43 (m, 3H, H_{m+p}), 7.60 (m, 2H, H_o). ^{13}C NMR ($CDCl_3$): δ −3.51 (*Me*Si), 23.66 (SiCH_2C), 113.38 (=CH_2), 127.72 (C_m), 128.97 (C_p), 133.55 (C_o), 134.57 (*C*H=), 138.61 (C_i). ^{29}Si NMR ($CDCl_3$): δ −4.59. The ^{1}H and ^{13}C NMR data are consistent with those reported in Ref. [16].

3.2.3. 3-[(Chloromethyl)methylphenylsilyl]propylthioacetate (**4**)

Freshly distilled thioacetic acid (1.01 g, 1.0 mL, 13.2 mmol) was added dropwise to allylsilane **2** (1.86 g, 8.8 mmol, containing 6% of **3**) and irradiated for 3 h using a DRT-400 mercury lamp. The reaction temperature was maintained at 40 °C. Excess of thioacetic acid was removed under reduced pressure and the crude product (2.24 g) was purified by column chromatography (silica gel, hexane-ether, increasing polarity from 25:1 to 9:1) gave **4** (1.69 g, 67%) and **5** (0.15 g).

*Data for **4**:* a colorless oil, R_f = 0.32 (silica gel, hexane/ether, 9:1). ^{1}H NMR ($CDCl_3$): δ 0.41 (s, 3H, *Me*Si), 1.01 (m, 2H, SiCH_2C), 1.64 (m, 2H, CCH_2C), 2.31 (s, 3H, CH_3CO), 2.87 (t, 2H, J 7.17 Hz, CH_2S), 2.97 (d, 1H, J_{AB} = 14.08 Hz, SiC*H*$_A$Cl,), 3.03 (d, 1H, SiC*H*$_B$Cl) 7.39 (m, 3H, H_{m+p}), 7.51 (m, 2H, H_o). ^{13}C NMR ($CDCl_3$): δ −6.40 (*Me*Si), 12.03 (SiCH_2C), 23.95 (CCH_2C), 28.99 (*C*H$_2$Cl), 30.64 (*C*H$_3$CO), 32.28 (CCH_2S), 128.03 (C_m), 129.79 (C_p), 133.87 (C_o), 134.73 (C_i), 195.80 (*C*O). ^{29}Si NMR ($CDCl_3$): δ −2.92. Anal. Calc. for $C_{13}H_{19}SiClOS$: C, 54.43; H, 6.68; Si, 9.79. Found: C 54.54; H 6.50; Si 9.82%.

*Data for **5**:* a colorless oil, R_f = 0.57 (hexane/ether, 9:1). ^{1}H NMR ($CDCl_3$): δ 0.35 (s, 6H, Me_2Si), 0.90 (m, 2H, SiH_2), 1.66 (m, 2H, CCH_2C), 2.36 (s, 3H, CH_3CO), 2.94 (t, 2H, J = 7.3 Hz, SCH_2), 7.42 (m, 3H, H_{m+p}), 7.56 (m, 2H, H_o). ^{13}C NMR ($CDCl_3$): δ −3.10 (*Me*Si), 15.41 (SiCH_2C), 24.42 (CCH_2C), 30.62 (*C*H$_3$CO), 32.51 (CCH_2S), 127.81 (C_o), 128.97 (C_p), 133.53 (C_m), 138.85 (C_i), 195.79 (*C*=O). ^{29}Si NMR ($CDCl_3$): δ −2.98.

Thioacetate (**5**) was obtained from pure allyldimethylphenylsilane previously in a similar manner [17].

3.2.4. 3-Methyl-3-phenyl-3-sila-1-thiacyclohexane (**6**)

A solution of **4** (1.77 g, 6.20 mmol) in methanol (2 mL) was added to a solution of sodium methoxide (5 mL) prepared from sodium (0.14 g, 6.20 g-a). After stirring at room temperature for 3 h, the reaction mixture was diluted with hexane (10 mL) and quenched with an aqueous ammonium chloride solution and water. The aqueous layer was extracted with hexane (2 × 10 mL), the combined organic layer was dried ($MgSO_4$), filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (silica gel, hexane/ether, 9:1) to afford cycle (**6**) as a colorless oil (0.67 g, 74% yield).

*Data for **6**:* R_f = 0.70 (hexane/ether, 9:1). ^{1}H NMR ($CDCl_3$): δ 0.46 (s, 3H, *Me*Si), 0.98 (m, 2H, SiCH_2C), 1.82 (d, 1H, J_{AB} = 14.8 Hz, Si-C*H*$_A$S), 2.11 (d, 1H, SiC*H*$_B$S), 2.07 (m, 1H, CC*H*$_A$C), 2.24 (m, 1H, CC*H*$_B$C), 2.57 (m, 2H, CCH_2S), 7.39 (m, 3H, H_{m+p}), 7.57 (m, 2H, H_o). ^{13}C NMR ($CDCl_3$): δ −4.86 (*Me*Si), 12.69 (SiCH_2C), 13.40 (SiCH_2S), 26.99 (CCH_2C), 32.30 (CCH_2S), 127.93 (C_o), 129.38 (C_p), 133.65 (C_m), 137.72 (C_i). ^{29}Si NMR ($CDCl_3$): δ −16.23. Anal. Calc. for $C_{11}H_{16}SiS$: C, 63.40; H, 7.74; Si, 13.48. Found: C, 62.99; H, 7.45; Si, 13.10%.

3.2.5. 3-i-Propoxy-3-methyl-3-sila-1-thiacyclohexane (**7**)

Triflic acid (1.90 mL, 22.0 mmol) was added dropwise to a stirred solution of **6** (4.00 g, 19.2 mmol) in *n*-pentane (10 mL) at room temperature, and the mixture was then heated under reflux for 1 h. After cooling a mixture of *i*-propanol (1.38 g, 23.0 mmol) and triethylamine (2.33 g, 23.0 mmol) was added at 0 °C. After 10 min., the reaction mixture was warmed to room temperature and stirred further for 2 h. The upper organic phase was separated, and the lower phase was washed with *n*-pentane (2 × 10 mL). The organic phases were combined, the solvent was removed by distillation under atmospheric pressure, and the residue was distilled in vacuo to give **7** in 53% yield (1.92 g) as a colorless liquid, b.p. 95–96 °C/14 mm Hg.

*Data for **7**:* ^{1}H NMR ($CDCl_3$): δ 0.28 (s, 3H, *Me*Si), 0.73 (ddd, 1H, J_{AB} = 14.55, J = 11.37, J = 4.77 Hz, SiC*H*$_A$C), 0.80 (m, 1H, SiC*H*$_B$C), 1.15 (d, 6H, Me_2CHO, J = 6.11 Hz), 1.60 (d, 1H, J_{AB} = 14.43 Hz, Si-C*H*$_A$S), 1.93 (d, 1H, SiC*H*$_B$S), 1.92 (m, 1H, CC*H*$_A$C), 2.25 (m, 1H, CC*H*$_B$C), 2.43 (m, 2H, CCH_2S), 4.05 (heptet, 1H, *CH*O). ^{13}C NMR ($CDCl_3$): δ −2.76 (*Me*Si), 15.82 (SiCH_2C), 16.27 (SiCH_2S), 27.16 (*Me*-CHO), 30.07 (CCH_2C), 33.53 (CCH_2S), 66.58 (*C*HO). ^{29}Si NMR ($CDCl_3$): δ −1.64. Anal. Calc. for $C_8H_{18}Si0S$: C, 50.57; H, 9.54; Si, 14.75. Found: C, 50.38; H, 9.58; Si, 14.75%.

3.2.6. 3-Methyl-3-sila-1-thiacyclohexane (**8**)

Compound **7** (1.92 g, 10.1 mmol) was added to a stirred suspension of lithium aluminum hydride (0.3 g, 7.1 mmol) in diethyl ether (5 mL) at room temperature. The resulting mixture was heated under reflux for 4 h and allowed to cool to room temperature. After adding pentane (10 mL) the reaction mixture was added to a stirred mixture of concentrated hydrochloric acid (25 mL), *n*-pentane (20 mL), water (25 mL) and ice (30 g). The organic layer was separated and the aqueous phase was extracted with pentane (2 × 10 mL). The combined organic extracts were dried over $MgSO_4$. The solvents were removed under atmospheric pressure to give the crude product **8** (1.27 g), vacuum distillation of which afforded compound **8** (0.55 g, 41% yield).

The crude product can be also purified by column chromatography on silica gel using *n*-pentane as the eluent.

*Data for **8**:* a clear colorless liquid, b.p. 53–55 °C/43 mm Hg. ^{1}H NMR ($CDCl_3$): δ 0.21 (d, 3H, J = 3.30 Hz, *Me*Si), 0.69 (m, 1H, SiC*H*$_A$C), 0.92 (m, 1H, SiC*H*$_B$C), 1.69 (dd, 1H, J_{AB} = 14.43, J = 3.91 Hz, SiC*H*$_A$S), 1.88 (d, 1H, SiC*H*$_B$S), 2.06 (m, 2H, CCH_2C), 2.48 (m, 2H, CCH_2S), 4.03 (m, 1H, Si*H*). ^{13}C NMR ($CDCl_3$): δ −5.59 (*Me*Si), 11.35 (SiCH_2C), 11.97 (SiCH_2S), 27.17 (CCH_2C), 32.21 (CCH_2S). ^{29}Si NMR ($CDCl_3$): δ −23.67. Anal. Calc. for $C_5H_{12}SiS$: C, 45.39; H, 9.15; Si, 21.23. Found: C, 45.17; H, 9.09; Si, 21.14%.

3.2.7. 3-Fluoro-3-methyl-3-sila-1-thiacyclohexane (**9**)

$BF_3{\cdot}2CH_3COOH$ (0.5 mL, 3.5 mmol) was added to a stirred solution of **6** (1.46 g, 7.0 mmol) in CH_2Cl_2 (5 mL) at room temperature. The reaction mixture was heated under reflux for 6 h, cooled to room temperature, and aqueous saturated solution of Na_2CO_3 (3 mL) was added dropwise until the solution was neutral pH. The mixture was extracted with CH_2Cl_2, the combined organic extracts were dried ($MgSO_4$), filtered and the solvent was removed by distillation at atmospheric pressure to give **9** (1.34 g, 75% purity, by ^{1}H NMR data). Compound **9** was obtained in 35% yield (0.36 g) by vacuum distillation.

Data for ***9****:* a colorless liquid, b.p. 73 °C/26 mm Hg. ^{1}H NMR ($CDCl_3$): δ 0.41 (d, 3H, J_{H-F} = 6.9 Hz, *Me*Si), 0.81 (ddd, 1H, J_{AB} = 14.85, J = 10.50, J = 4.61 Hz, SiCH_AC), 0.88 (m, 1H, SiCH_BC), 1.72 (d, 1H, J_{AB} = 14.8 Hz, SiCH_AS), 1.96 (d, 1H, SiCH_BS) 2.00 (m, 1H, CCH_AC), 2.27 (m, 1H, CCH_BC), 2.43 (m, 2H, CCH_2S). ^{13}C NMR ($CDCl_3$): δ −3.93 (d, J_{C-F} = 14.86 Hz, *Me*Si), 14.07 (d, J_{C-F} = 12.2 Hz, SiCH_2C), 14.54 (d, J_{C-F} = 14.78 Hz, SiCH_2S), 28.79 (d, CCH_2C, J_{C-F} = 2.15 Hz), 32.15 (CCH_2S). ^{19}F NMR ($CDCl_3$): δ −159.45 (d). ^{29}Si NMR ($CDCl_3$): δ −13.35 (d, J_{Si-F} 292.6 Hz). Anal. Calc. for $C_5H_{11}SiFS$: H, 7.38; Si, 18.69. Found: H, 7.29; Si, 18.12%.

Acknowledgment

This work was supported by the Russian Foundation for Basic Research and Deutsche Forschungsgemeinschaft (Grant RFBR-DFG No. 08-03-91954).

References

[1] (a) For reviews on silathiacycloalkanes see: G. Rousseau, L. Blanco, Tetrahedron 62 (2006) 7951–7993;
(b) E. Block, M. Aslam, Tetrahedron 44 (1988) 281–324;
(c) E. Block, Rev. Heteroat. Chem. 1 (1988) 163–177.
[2] (a) S.V. Kirpichenko, E.N. Suslova, L.L. Tolstikova, A.I. Albanov, B.A. Shainyan, Zh. Obshch. Khim. 67 (1997) 1542–1547. Russ. J. Gen. Chem. (Engl. Trans.) 67 (1997) 1449–1454;
(b) S.V. Kirpichenko, A.I. Albanov, V.A. Pestunovich, J. Sulfur Chem. 25 (2004) 21–27;
(c) S.V. Kirpichenko, E.N. Suslova, A.I. Albanov, B.A. Shainyan, Sulfur Lett. 22 (1999) 245–248;
(d) E.N. Suslova, A.I. Albanov, B.A. Shainyan, J. Organomet. Chem. 667 (2003) 73–79.
[3] (a) K.B. Borisenko, S. Samdal, E.N. Suslova, V.A. Sipachev, I.F. Shishkov, L.V. Vilkov, Acta Chem. Scand. 52 (1998) 975;
(b) M.G. Voronkov, S.V. Kirpichenko, E.N. Suslova, L.L. Tolstikova, Zh. Obshch. Khim. 60 (1990) 2630–2632. J. Gen. Chem. USSR (Engl. Transl.) 60 (1990) 2358–2359.
[4] E.N. Suslova, A.I. Albanov, B.A. Shainyan, J. Organomet. Chem. 694 (2009) 420–426.
[5] K.E. Yelm, Tetrahedron Lett. 40 (1999) 1101–1102.
[6] S.V. Kirpichenko, unpublished results.
[7] For review see: S.McN. Sieburth, C.-A. Chen, Eur. J. Org. Chem. (2006) 311–322.
[8] (a) L. Nielsen, K.B. Lindsay, J. Faber, N.C. Nielsen, T. Skrydstrup, J. Org. Chem. 72 (2007) 10035–10044;
(b) L. Nielsen, T. Skrydstrup, J. Am. Chem. Soc. 130 (2008) 13145–13151.
[9] (a) A.R. Bassindale, T. Stout, J. Organomet. Chem. 271 (1984) C1–C3;
(b) W. Uhlig, J. Organomet. Chem. 452 (1993) 29–32.
[10] (a) R. Tacke, T. Heinrich, R. Bertermann, C. Burschka, A. Hamacher, M.U. Kassack, Organometallics 23 (2004) 4468–4477;
(b) J.O. Daiss, K.A. Barth, C. Burschka, P. Hey, R. Ilg, K. Klemm, I. Richter, S.A. Wagner, R. Tacke, Organometallics 23 (2004) 5193–5197;
(c) A.D. Dilman, V.V. Levin, M. Karni, Y. Apeloig, J. Org. Chem. 71 (2006) 7214–7222.
[11] (a) J.Y. Corey, D.M. Kraichely, J.L. Huhmann, J. Braddock-Wilking, A. Lindeberg, Organometallics 14 (1995) 2704–2717;
(b) E. Hengge, P. Gspalt, E. Pinter, J. Organomet. Chem. 521 (1996) 145–155;
(c) P.I. Gudnason, I. Arnason, Org. Lett. 11 (2009) 2015–2017.
[12] J. Kim, S.McN. Sieburth, J. Org. Chem. 69 (2004) 3008–3014.
[13] (a) I. Fleming, J.D. Kilburn, J. Chem. Soc., Perkin Trans. 1 (1998) 2663–2671;
(b) H.-J. Knolker, G. Baum, O. Schmitt, G. Wanzl, Chem. Commun. (1999) 1737–1738;
(c) M.G. Organ, C. Buon, C.P. Decicco, A.P. Combs, Org. Lett. 4 (2002) 2683–2685;
(d) M.J. Sanganee, P.G. Steel, D.K. Whelligan, Org. Biomol. Chem. 2 (2004) 2393–2402.
[14] Z.V. Belyakova, S.A. Golubtsov, Zh. Obshch. Khim. 31 (1961) 3178–3181. J. Gen. Chem. USSR (Engl. Transl.) 31 (1961) 2966–2968.
[15] J. Yoshida, T. Maekawa, S. Matsunaga, S. Isoe, J. Am. Chem. Soc. 112 (1990) 1962–1970.
[16] Z. Li, X. Cao, G. Lai, J. Liu, Y. Ni, J. Wu, H. Qiu, J. Organomet. Chem. 691 (2006) 4740–4746.
[17] V.F. Mironov, N.A. Pogonkina, Izv. Akad. Nauk USSR, Ser. Khim. (1959) 85–90.