ISSN 1070-3632, Russian Journal of General Chemistry, 2009, Vol. 79, No. 5, pp. 1045–1046. © Pleiades Publishing, Ltd., 2009.
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LETTERS TO THE EDITOR

4,4-Dimethyl-4-silathiane-1-*N*-(trifluoromethanesulfonyl)-sulfimide, the First Representative of the SiS-Cycles Triflamide Derivatives

E. N. Suslova and B. A. Shainyan

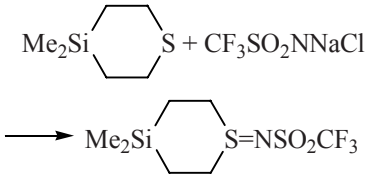
Favorskii Irkutsk Institute of Chemistry, Siberian Branch, Russian Academy of Sciences, ul. Favorskogo 1, Irkutsk, 664033 Russia
e-mail: svk@irioch.irk.ru

Received January 12, 2009

DOI: 10.1134/S1070363209050326

N-Triflyl derivatives of organosilicon sulfimides are of interest from the viewpoint of possible stabilization of these compounds. Earlier, we succeeded in preparation of the six-membered cyclic organosilicon sulfimides [1, 2] whereas their five-membered analogs are unstable and decompose with the ring opening at the Si–CH_2S bond [3]. Linear organosilicon sulfimides are stable provided that the silicon and sulfur atoms are separated by two or three CH_2 groups and are unstable when the Si–CH_2–S group is present in the molecule [1, 4]. It is presumable that the introduction of the electron-acceptor trifluoromethanesulfonyl group would decrease the reactivity of these compounds with respect to electrophiles and increase their stability. Therefore, we attempted to synthesize *N*-trifluoromethanesulfonylimide by the example of the earlier obtained 4,4-dimethyl-4-silathiane [5].

The target 4,4-dimethyl-4-silathiane-1-*N*-(trifluoromethanesulfonyl)sulfimide was formed by the reaction of sodium salt of *N*-chlorotrifluoromethane-sulfon0-amide prepared as desribed in [6] with 4,4-dimethyl-4-silathiane in CH_2Cl_2 in 80% yield.



The structure of the product was proved by the ^{1}H, ^{13}C, ^{19}F, and ^{29}Si NMR spectroscopy, mass-spectrometry, and elemental analysis. In the mass spectrum obtained by direct admission mode, the peak of molecular ion with *m/z* 293 was absent but it was registered (with 2.5% intensity) in the GC–MS analysis.

Therefore, the first representative of the *N*-trifluoromethanesulfonyl-substituted imides of thiasilacycloalkanes is obtained that makes it possible to use this method for the synthesis of other trifluoromethanesulfonyl derivatives of this class of compounds as well as of their linear analogs.

4,4-Dimethyl-4-silathiane-1-*N*-(trifluoromethanesulfonyl)sulfimide. To the solution of 0.584 g of 4,4-dimethyl-4-silathiane and 0.044 g of triethylbenzylammonium chloride in 10 ml of anhydrous CH_2Cl_2, 1.2 g of sodium salt of *N*-chlorotrifluoromethanesulfonamide prepared by the reflux of suspension of disodium salt of triflamide (prepared from triflamide with sodium methoxide) with *N,N*-dichlorotriflamide (prepared by chlorination of triflamide with chlorine in aqueous alkaline solution) in dry CCl_4 was added by portions in the course of 20 min. The reaction mixture was stirred for 1 h at room temperature and 3 h at 38°C, washed with cold water (2×5 ml), dried over $MgSO_4$, solvent was removed in a vacuum to obtain 0.952 g (80%) of the crude product which was purified by column chromatography on silica (Merck) in the system hexane–ether from 8:1 to 1:7. 0.392 g (34%) of pure product was isolated as lightweight white crystals, mp 73.4°C . IR spectrum, ν, cm^{-1}: 2950 (C–H), 1420 ($SiCH_2$), 1315, 1130, 600 (SO_2), 1250 (SiMe), 1200, 1170, (CF_3), 1000, 780 (S=N), 980, 830. ^{1}H NMR, δ, ppm: 0.18, 0.21 s (6H, $SiMe_2$), 1.04 d.d.d (2H, $SiCH^{ax}$, *J* 15.6, 9.2, 3.3 Hz), 1.56 d.d.d (2H, $SiCH^{eq}$, *J* 15.6,

10.2, 3.8 Hz), 3.24 d.d.d (2H, SCH^{ax}, *J* 13.8, 10.2, 3.3 Hz), 3.40 d.d.d (2H, SCH^{eq}, *J* 13.8, 9.5, 3.8 Hz). ^{13}C NMR, δ, ppm: –4.40, –3.86 (SiMe), 6.83 ($SiCH_2$), 44.70 (SCH_2), 120.29 q (CF_3, *J* 322.8 Hz). ^{19}F NMR, δ_F, ppm: –78.18. ^{29}Si NMR, δ_{Si}, ppm: –6.97. Mass spectrum (direct admission), *m/z* ($I_{rel.}$, %) ion: 265 (6) [$M - C_2H_4$], 191 (13) [$CF_3SO_2SiMe_2$], 141 (23) [265 – SO_2 – Me_2SiH_2], 132 (54) [265 – SO_2CF_3], 116 (10) [$Me_2SiCH{=}CHS$], 104 (100) [Me_2SiSN], 90 (23) [Me_2SiS], 77 (66) [Me_2SiF], 75 (32) [MeSiS], 69 (31) [CF_3] 59 (37) [Me_2SiH], 58 (38) [Me_2Si], 43 (65) [MeSi]. Found, %: C 28.81; H 4.81; F 19.95; N 4.88; S 22.67. $C_7H_{14}F_3NO_2S_2Si$. Calculated, %: C 28.66; H 4.81; F 19.43; N 4.77; S 21.85.

IR spectrum was taken on a Specord IR 75 spectrometer from KBr pellets. ^1H, ^{13}C, ^{19}F, and ^{29}Si NMR spectra were registered from solutions in $CDCl_3$ on a Bruker DPX 400 spectrometer (400, 100, 376, and 79.5 MHz, respectively), chemical shifts are given relative to TMS (^1H, ^{13}C, ^{29}Si) and CCl_3F (^{19}F). Electron impact mass spectra (70 eV) were recorded on a GCMS-QP5050A Shimadzu instrument (quadrupole mass analyzer, capillary column, liquid phase SPB-5).

ACKNOWLEDGMENTS

This work was financially supported by the Russian Foundation for Basic Research (grants nos. 07-03-00425 and RFBR-DFG 08-03-91954).

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