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# Novel α,β-unsaturated imine derivatives of 3-aminopropylsilatrane

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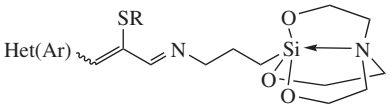
**Novel highly functionalized and pharmacologically promising active silatranes equipped with an α,β-unsaturated imine group, Het(Ar)CH=C(SR)C(H)=N–, in the axial chain have been synthesized by the reaction of 3-aminopropylsilatrane and (*Z*)-2-alkylthio-3-(het)arylpropenals. The structures of these compounds were characterized by IR and $^{1}$H, $^{13}$C, $^{15}$N, $^{29}$Si, 1D and 2D NMR spectroscopy.**



Over many decades, the chelate organosilicon ethers of biogenic alkanolamines, silatranes $R^1$–Si(OCH$R^2$C$H_2$)$_3$N ($R^1$ = Hal, Alk, OAlk, Ar, *etc.*), attract widespread attention.[1] Owing to intramolecular transannular coordinate bond Si←N, the silatrane molecules have original tricyclic structure. Such a structure with pentacoordinated silicon atom imparts specific physical-chemical and unique biological properties to the silatranes.

The presence of the Si←N bond ensures high dipole moment of these molecules, which facilitates their transportation through biological membranes. A strong electron-donating effect of the silatrane skeleton significantly increases electron density in the substituent $R^1$ and Si(O–)$_3$ moiety thus enhancing the degree of silatranes coupling with receptors. Thorough investigations of biological activity of silatranes show that they can be successfully employed in agriculture, microbiology, pharmacology and medicine.[2]

The most facile method for the synthesis of silatranes is transetherification of trialkoxysilanes. For instance, the reaction of available (but hydrolytically unstable) (3-aminopropyl)triethoxysilane $H_2N(CH_2)_3Si(OEt)_3$ **1a** with triethanolamine affords water-soluble and stable 3-aminopropylsilatrane $H_2N(CH_2)_3$-Si(OC$H_2$C$H_2$)$_3$N **1b**. This compound represents a convenient building block for design of numerous new modern materials (Si-organic polyimide films, catalysts, optical probes, aghesive layers to fix gold nanoparticles, biosensors, gas separation membranes)[3–8] and pharmacologically active compounds (antiparasitic, antibacterial, antihepatitis, immuno-modulating, and antitumor agents).[9–13] On the other hand, aldehydes are known to react with primary amines to deliver imines which can also be employed as precursors of drugs including antitumor ones.[14]

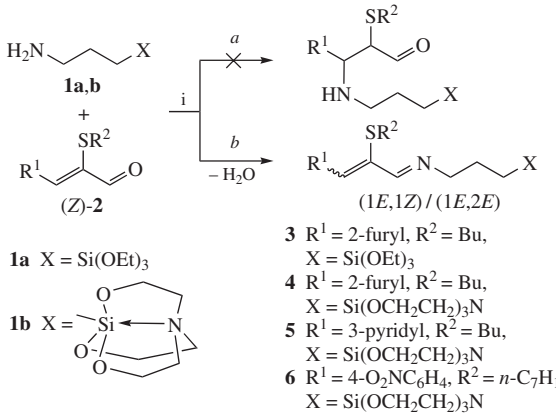
The data on reactions of 3-aminopropylsilane **1b** with unsaturated aldehydes are scanty.[15] The target silatranes with the substituent at the silicon atom should contain the imine moiety C=N. Such functionalized silatranes turn out to be selective adsorbents of copper ions[15(*c*)] as well as show growth-stimulating[16] and anticancer activity.[17]

We have developed an expedient stereoselective method for the synthesis of original α,β-unsaturated aldehydes, (*Z*)-2-alkoxy- and (*Z*)-2-alkylthio-3-(het)arylpropenals Het(Ar)CH=C(YR)C(H)=O (Ar = phenyl, furyl, pyridinyl, *etc.*; Y = O, S), by the condensation of (het)aryl-containing aldehydes with 2-alkoxy- and 2-alkylthioacetaldehydes.[18] The thus obtained α,β-unsaturated aldehydes are highly reactive compounds attracting significant theoretical[19] and practical[20] interest. For example, they were used in the synthesis of various heterocycles and drugs.[18(*b*),(*c*)]

It has been shown in our[12(*b*)–(*d*)] and other works[12(*a*),13] that the integration of two or three biologically active components in one molecule can lead to synergetic effect of their activity.

To synthesize novel pharmacologically active hybrids, we have implemented herein the reaction of 3-aminopropyltriethoxysilane **1a** and 3-aminopropylsilatrane **1b** with the previously obtained (*Z*)-2-alkylthio-3-(het)arylpropenals **2** (Scheme 1).

The reaction course is not obvious. The process could follow the routes (*a*) or (*b*) or it could occur in a competitive fashion (*a* and *b*) to furnish a mixture of 1,2- and 1,4-adducts. However, according to the data of elemental analysis, $^{1}$H, $^{13}$C, $^{15}$N, $^{29}$Si NMR and IR spectroscopy,[†] the reaction between silanes **1a**,**b**




**Scheme 1** *Reagents and conditions*: i, THF, $CHCl_3$ or $CH_2Cl_2$, 25 °C, 24 h, filtered sieves 4 Å or $MgSO_4$.

[†] The $^{1}$H, $^{13}$C, $^{29}$Si and $^{15}$N NMR spectra were recorded in $CDCl_3$ at room temperature on Bruker DPX-400 and AV-400 spectrometers (400.13, 100.61, 79.46 and 40.56 MHz, respectively). Chemical shifts were referred to TMS ($^{1}$H, $^{13}$C, $^{29}$Si) and nitromethane ($^{15}$N). IR spectra were recorded on a Bruker IFS25 spectrophotometer. Melting points were determined on a Micro-Hot-StagePolyTermA (Warner and Muzn) instrument. Elemental analysis was performed on a Thermo Finning 1112ser analyzer.

and the corresponding propenals affords viscous (**3**–**5**) and solid (**6**) imines following thus the route *b* exclusively. To increase the product yields, sieves 4 Å or $MgSO_4$ have been employed.

The analysis of the $^1H$ and $^{13}C$ NMR spectra has shown that the products are formed as a mixtures of (1*E*,2*Z*) and (1*E*,2*E*) stereoisomers in ~2 : 1 ratio. In all the cases, the 1*E*-configuration of the imine fragment remains the same.

Signals in the $^1H$ and $^{13}C$ NMR spectra have been assigned using 2D homo- and heteronuclear experiments (COSY, NOESY, HSQC and HMBC). The values of chemical shifts and the observed NOE correlations in 2D NOESY spectra have allowed us to establish conformational peculiarities of the compounds, which are most brightly demonstrated in products **3** and **4** (Figure 1). The (het)aryl substituent in the (1*E*,2*Z*) isomer is effectively conjugated to the CH=CS bond that facilitates the formation of the intramolecular hydrogen bond CH···S.[21] In case of the (1*E*,2*E*) isomers **5** and **6**, the imine proton being under the anisotropic effect of the heterocycle is shifted downfield to 8.12–8.14 ppm. For compounds **3**, **4** this resonance is even greater downfield shifted (8.76 ppm) due to the formation of intramolecular hydrogen bond CH···O[21] (see Figure 1).

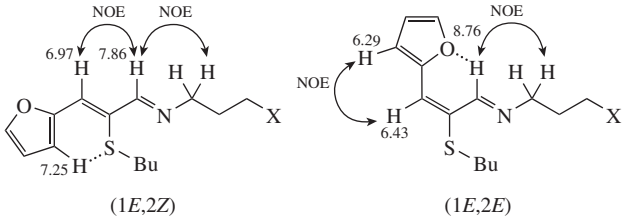



**Figure 1** Main NOE correlations and selected $^1H$ chemical shifts for isomers of compounds **3** and **4**.

Note that compounds **3**–**6** are generated in good yields which promote their use *in situ* without isolation. For instance, they can be employed for the preparation of sorbents containing Schiff base, metal complexes (catalysts), ionic liquids[4] and hitherto unknown silatranyl derivatives of pyrroles *via* the original method for the synthesis of tetrasubstituted pyrroles developed by us.[18(d)]

In conclusion, a novel family of silatranes has been synthesized from 3-aminopropylsilatrane and 2-alkylthio-3-(het)arylpropenals.

*General procedure for the synthesis of compounds* **3**–**6**. A mixture of amino silane **1a** or **1b** (1 mmol) and the corresponding propenals **2** (in 1 : 1 ratio) in THF, $CHCl_3$ or $CH_2Cl_2$ in the presence of sieve 4 Å or $MgSO_4$ was stirred at room temperature for 24 h. The drying agent was filtered off, the solvent was distilled off and the residue was washed with diethyl ether and dried *in vacuo* to obtain pure oils **3**–**5** or powder **6**.

*[6-Butylthio-7-(2-furyl)-4-azahepta-4,6-dien-1-yl]triethoxysilane* **3**: colourless oil, yield 85%. IR ($\nu$/cm$^{-1}$): 1028 (Si–O), 1589 (C=C), 1627 (C=N). 1*E*,2*Z* (major isomer). $^1H$ NMR ($CDCl_3$) $\delta$: 0.62 (m, 2H, $CH_2Si$), 0.85 (t, 3H, Me, *J* 7.0 Hz), 1.21 (t, 9H, Me, *J* 7.0 Hz), 1.36 (m, 2H, $CH_2$ in SBu), 1.47 (m, 2H, $CH_2$ in SBu), 1.80 (m, 2H, C*H*$_2$CH$_2$Si), 2.97 (t, 2H, $SCH_2$, *J* 7.4 Hz), 3.67 (t, 2H, $NCH_2$, *J* 6.7 Hz), 3.81 (q, 6H, $OCH_2$, *J* 7.0 Hz), 6.51 (dd, 1H, H$^4$, $^3J$ 3.2 Hz, $^4J$ 1.6 Hz), 7.04 (s, 1H, CH=), 7.32 (d, 1H, H$^3$, $^3J$ 3.2 Hz,), 7.47 (d, 1H, H$^5$, $^4J$ 1.6 Hz), 7.90 (s, 1H, CH=N). 1*E*,2*E* (minor isomer). $^1H$ NMR ($CDCl_3$) $\delta$: 0.64 (m, 2H, $CH_2Si$), 0.92 (t, 3H, Me, *J* 7.0 Hz), 1.21 (t, 9H, Me, *J* 7.0 Hz), 1.45 (m, 2H, $CH_2$ in SBu), 1.65 (m, 2H, $CH_2$ in SBu), 1.65 (m, 2H, C*H*$_2$CH$_2$Si), 2.78 (t, 2H, $SCH_2$, *J* 7.4 Hz), 3.62 (t, 2H, $NCH_2$, *J* 6.7 Hz), 3.81 (q, 6H, $OCH_2$, *J* 7.0 Hz), 6.32 (d, 1H, H$^3$, $^3J$ 3.4 Hz), 6.41 (dd, 1H, H$^4$, $^3J$ 3.4 Hz, $^4J$ 1.8 Hz), 6.47 (s, 1H, CH=), 7.44 (d, 1H, H$^5$, $^4J$ 1.8 Hz), 8.83 (s, 1H, CH=N). Found (%): C, 58.18; H, 8.43; N, 3.30; Si, 6.73. Calc. for $C_{20}H_{35}NSO_4Si$ (%): C, 58.08; H, 8.53; N, 3.38; Si, 6.79.

*1-[6-Butylthio-7-(2-furyl)-4-azahepta-4,6-dien-1-yl]-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane* **4**: light yellow oil, yield 79%. IR ($\nu$/m$^{-1}$): 580 (N→Si), 1050 (Si–O), 1591 (C=C), 1643 (C=N). (1*E*,2*Z*) isomer, $^1H$ NMR ($CDCl_3$) $\delta$: 0.37 (m, 2H, $CH_2Si$), 0.85 (t, 3H, Me, *J* 7.3 Hz), 1.36 (m, 2H, $CH_2$ in SBu), 1.47 (m, 2H, $CH_2$ in SBu), 1.74 (m, 2H, C*H*$_2$CH$_2$Si), 2.77 (t, 6H, $CH_2N$, *J* 5.6 Hz), 2.99 (t, 2H, $SCH_2$, *J* 7.4 Hz), 3.54 (t, 2H, $NCH_2$, *J* 7.0 Hz), 3.73 (t, 6H, $OCH_2$, *J* 5.6 Hz), 6.48 (dd, 1H, H$^4$, $^3J$ 3.2 Hz, $^4J$ 1.6 Hz), 6.97 (s, 1H, CH=), 7.25 (d, 1H, H$^3$, $^3J$ 3.2 Hz), 7.42 (d, 1H, H$^5$, $^4J$ 1.6 Hz), 7.87 (s, 1H, CH=N). $^{13}C$ NMR ($CDCl_3$) $\delta$: 13.58 ($CH_2Si$), 13.65 (Me), 21.84 ($CH_2$ in SBu), 26.51 (*C*H$_2$CH$_2$Si), 32.27 ($CH_2$ in SBu), 32.40 ($SCH_2$), 51.16 ($CH_2N$), 57.81 ($OCH_2$), 65.00 ($NCH_2$), 112.20 (C$^4$), 113.95 (C$^3$), 128.51 (CH=), 131.35 (=C–S), 142.68 (C$^5$), 151.94 (C$^2$), 160.61 (CH=N). $^{15}N$ NMR ($CDCl_3$) $\delta$: –359.2 ($CH_2N$), –42.3 (CH=N). $^{29}Si$ NMR ($CDCl_3$) $\delta$: –66.1. (1*E*,2*E*) isomer, $^1H$ NMR ($CDCl_3$) $\delta$: 0.37 (m, 2H, $CH_2Si$), 0.90 (t, 3H, Me, *J* 7.3 Hz), 1.45 (m, 2H, $CH_2$ in SBu), 1.65 (m, 2H, $CH_2$ in SBu), 1.76 (m, 2H, C*H*$_2$CH$_2$Si), 2.76 (t, 6H, $CH_2N$, *J* 5.6 Hz), 2.75 (t, 2H, $SCH_2$, *J* 7.4 Hz), 3.60 (t, 2H, $NCH_2$, *J* 7.0 Hz), 3.73 (t, 6H, $OCH_2$, *J* 5.6 Hz), 6.29 (d, 1H, H$^3$, $^3J$ 3.4 Hz), 6.39 (dd, 1H, H$^4$, $^3J$ 3.4 Hz, $^4J$ 1.8 Hz), 6.43 (s, 1H, CH=), 7.41 (d, 1H, H$^5$, $^4J$ 1.8 Hz), 8.76 (s, 1H, CH=N). $^{13}C$ NMR ($CDCl_3$) $\delta$: 13.72 ($CH_2Si$), 13.77 (Me), 22.19 ($CH_2$ in SBu), 26.56 (*C*H$_2$CH$_2$Si), 30.38 ($CH_2$ in SBu), 30.99 ($SCH_2$), 51.16 ($CH_2N$), 57.81 ($OCH_2$), 65.30 ($NCH_2$), 111.40 (C$^3$), 111.64 (C$^4$), 117.90 (CH=), 135.12 (=C–S), 142.97 (C$^5$), 151.92 (C$^2$), 157.86 (CH=N). $^{15}N$ NMR ($CDCl_3$) $\delta$: –359.2 ($CH_2N$), –37.2 (CH=N). $^{29}Si$ NMR ($CDCl_3$) $\delta$: –66.2. Found (%): C, 56.42; H, 7.71; N, 6.65. Calc. for $C_{20}H_{32}N_2SO_4Si$ (%): C, 56.57; H, 7.60; N, 6.60.

*Product* **5**: transparent yellow oil, yield 81%. IR ($\nu$/cm$^{-1}$): 576 (N→Si), 1053 (Si–O), (C=C), 1628 (C=N). (1*E*,2*Z*) isomer. $^1H$ NMR ($CDCl_3$) $\delta$: 0.34 (m, 2H, $CH_2Si$), 0.82 (t, 3H, Me, *J* 7.3 Hz), 1.35 (m, 2H, $CH_2$ in SBu), 1.47 (m, 2H, $CH_2$ in SBu), 1.75 (m, 2H, C*H*$_2$CH$_2$Si), 2.79 (t, 6H, $CH_2N$, *J* 5.8 Hz), 2.86 (t, 2H, $SCH_2$, *J* 7.4 Hz), 3.54 (t, 2H, $NCH_2$, *J* 7.4 Hz), 3.69 (t, 6H, $OCH_2$, *J* 5.8 Hz), 7.07 (s, 1H, CH=), 7.26 (m, 1H, H$^5$), 7.93 (s, 1H, CH=N), 8.23 (m, 1H, H$^4$), 8.44 (m, 1H, H$^6$), 8.78 (m, 1H, H$^2$). $^{13}C$ NMR ($CDCl_3$) $\delta$: 13.59 ($CH_2Si$), 13.66 (Me), 21.56 ($CH_2$ in SBu), 26.21 (*C*H$_2$CH$_2$Si), 31.90 ($CH_2$ in SBu), 32.57 ($SCH_2$), 50.95 ($CH_2N$), 57.62 ($OCH_2$), 64.78 ($NCH_2$), 122.92 (C$^5$), 131.82 (C$^3$), 136.05 (C$^4$), 136.65 (CH=), 136.90 (=C–S), 148.54 (C$^6$), 151.16 (C$^2$), 160.84 (CH=N). $^{15}N$ NMR ($CDCl_3$) $\delta$: –359.2 ($CH_2N$), –71.7 (N$^1$), –38.6 (CH=N). $^{29}Si$ NMR ($CDCl_3$) $\delta$: –66.7. (1*E*,2*E*) isomer. $^1H$ NMR ($CDCl_3$) $\delta$: 0.33 (m, 2H, $CH_2Si$), 0.89 (t, 3H, Me, *J* 7.3 Hz), 1.42 (m, 2H, $CH_2$ in SBu), 1.60 (m, 2H, $CH_2$ in SBu), 1.70 (m, 2H, C*H*$_2$CH$_2$Si), 2.77 (t, 2H, $SCH_2$, *J* 7.4 Hz), 2.79 (t, 6H, $CH_2N$, *J* 5.8 Hz), 3.48 (t, 2H, $NCH_2$, *J* 7.4 Hz), 3.69 (t, 6H, $OCH_2$, *J* 5.8 Hz), 6.65 (s, 1H, CH=), 7.24 (m, 1H, H$^5$), 7.50 (m, 1H, H$^4$), 8.12 (s, 1H, CH=N), 8.44 (m, 1H, H$^6$), 8.45 (m, 1H, H$^2$). $^{13}C$ NMR ($CDCl_3$) $\delta$: 13.69 ($CH_2Si$), 13.73 (Me), 22.12 ($CH_2$ in SBu), 26.27 (*C*H$_2$CH$_2$Si), 30.08 ($CH_2$ in SBu), 30.61 ($SCH_2$), 50.97 ($CH_2N$), 57.62 ($OCH_2$), 64.91 ($NCH_2$), 123.05 (C$^5$), 125.58 (CH=), 136.17 (C$^3$), 136.65 (C$^4$), 140.22 (=C–S), 147.91 (C$^6$), 149.74 (C$^2$), 156.27 (CH=N). $^{15}N$ NMR ($CDCl_3$) $\delta$: –359.2 ($CH_2N$), –71.2 (N$^1$), –35.7 (CH=N). $^{29}Si$ NMR ($CDCl_3$) $\delta$: –66.8. Found (%): C, 57.87; H, 7.65; N, 9.65. Calc. for $C_{21}H_{33}N_3SO_3Si$ (%): C, 57.89; H, 7.64; N, 9.65.

*Product* **6**: light yellow powder, yield 79%, mp 77 °C. IR ($\nu$/cm$^{-1}$): 585 (N→Si), 1085 (Si–O), 1340 $\nu_s(NO_2)$, 1516 $\nu_{as}(NO_2)$, 1595 (C=C), 1648 (C=N). (1*E*,2*Z*) isomer. $^1H$ NMR ($CDCl_3$) $\delta$: 0.34 (m, 2H, $CH_2Si$), 0.83 (t, 3H, Me, *J* 7.2 Hz), 1.14–1.36 (m, 8H, $CH_2$ in SHept), 1.50 (m, 2H, $CH_2$ in SHept), 1.78 (m, 2H, C*H*$_2$CH$_2$Si), 2.79 (t, 6H, $CH_2N$, *J* 5.8 Hz), 2.91 (t, 2H, $SCH_2$, *J* 7.4 Hz), 3.58 (t, 2H, $NCH_2$, *J* 7.4 Hz), 3.74 (t, 6H, $OCH_2$, *J* 5.8 Hz), 7.15 (s, 1H, CH=), 7.89 (m, 2H, H$^{2,6}$), 7.96 (s, 1H, CH=N), 8.20 (m, 2H, H$^{3,5}$). $^{13}C$ NMR ($CDCl_3$) $\delta$: 13.48 ($CH_2Si$), 13.88 (Me), 21.61–23.12 ($CH_2$ in SHept), 26.18 (*C*H$_2$CH$_2$Si), 29.70 ($CH_2$ in SHept), 32.62 ($SCH_2$), 50.98 ($CH_2N$), 57.62 ($OCH_2$), 64.85 ($NCH_2$), 123.15 (C$^{3,5}$), 130.40 (C$^{2,6}$), 136.13 (CH=), 138.67 (=C–S), 142.38 (C$^1$), 146.56 (C$^4$), 160.54 (CH=N). $^{15}N$ NMR ($CDCl_3$) $\delta$: –359.1 ($CH_2N$), –39.1 (CH=N), –10.7 ($NO_2$). $^{29}Si$ NMR ($CDCl_3$) $\delta$: –66.5. (1*E*,2*E*) isomer. $^1H$ NMR ($CDCl_3$) $\delta$: 0.39 (m, 2H, $CH_2Si$), 0.84 (t, 3H, Me, *J* 7.2 Hz), 1.14–1.36 (m, 8H, $CH_2$ in SHept), 1.52 (m, 2H, $CH_2$ in SHept), 1.75 (m, 2H, C*H*$_2$CH$_2$Si), 2.79 (t, 6H, $CH_2N$, *J* 5.8 Hz), 2.81 (t, 2H, $SCH_2$, *J* 7.4 Hz), 3.55 (t, 2H, $NCH_2$, *J* 7.4 Hz), 3.74 (t, 6H, $OCH_2$, *J* 5.8 Hz), 6.66 (s, 1H, CH=), 7.37 (m, 2H, H$^{2,6}$), 8.14 (s, 1H, CH=N), 8.19 (m, 2H, H$^{3,5}$). $^{13}C$ NMR ($CDCl_3$) $\delta$: 13.69 ($CH_2Si$), 13.85 (Me), 21.56–23.75 ($CH_2$ in SHept), 26.27 (*C*H$_2$CH$_2$Si), 27.78 ($CH_2$ in SHept), 28.72 ($CH_2$ in SHept), 32.97 ($SCH_2$), 50.98 ($CH_2N$), 57.62 ($OCH_2$), 64.66 ($NCH_2$), 123.52 (C$^{3,5}$), 125.87 (CH=), 129.71 (C$^{2,6}$), 142.31 (C$^1$), 142.38 (=C–S), 145.97 (C$^4$), 156.47 (CH=N). $^{15}N$ NMR ($CDCl_3$) $\delta$: –359.1 ($CH_2N$), –36.5 (CH=N), –11.2 ($NO_2$). $^{29}Si$ NMR ($CDCl_3$) $\delta$: –66.8. Found (%): C, 57.74; H, 7.62; N, 8.15; Si, 5.18. Calc. for $C_{25}H_{39}N_3SO_5Si$ (%): C, 57.55; H, 7.53; N, 8.05; Si, 5.38.

These hybrid inorganic-organic compounds of pentacoordinated silicon contain α,β-unsaturated imine group attached to Si atom through a propylene linker. The highly functionalized target products represent promising building blocks for design of advanced materials and pharmaceuticals.

The main results were obtained using the equipment of the Baikal Analytical Center of Collective Use of the Siberian Branch of the Russian Academy of Sciences.

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