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Letters

On the interaction of silyl triflates with enamines: iminium ion formation versus silylation

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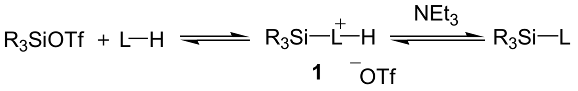
Abstract—The interaction of Me_3SiOTf and $(C_6F_5)_3SiOTf$ with enamines generating α-silyl-substituted iminium ions is investigated. A trimethylsilyl iminium cation is formed as a long-lived species observable by NMR spectroscopy, whilst the tris(pentafluorophenyl)silyl analogue is very labile and prone to the loss of a proton. On the basis of the latter phenomenon, a method for the synthesis of β-silyl enamines is proposed.

Silyl triflates have found wide application in synthetic chemistry as silylating reagents.[1] Silylation of functional groups usually affords *O*-silyl derivatives, while direct silylation at carbon is quite rarely encountered.[2] The general scheme of *C*-silylation requires transfer of a silyl group from the triflate to the substrate followed by deprotonation of the positively charged complex **1** by a nitrogen base (Eq. 1).



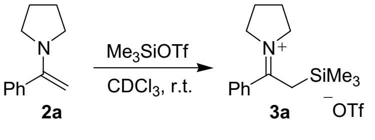
$$R_3SiOTf + L{-}H \rightleftharpoons \underset{\mathbf{1}\quad {}^{-}OTf}{R_3Si{-}\overset{+}{L}{-}H} \xrightleftharpoons{NEt_3} R_3Si{-}L \qquad (1)$$

To compensate for the energy loss upon exchange of Si–O to Si–C, the positive charge in complex **1** is highly stabilized.[3] On the other hand, if cation **1** is too stable, its deprotonation by the amine base may become difficult.

The examples of direct *C*-silylation described in the literature involve the interaction of silyl triflates with 1,1-dialkoxyalkenes[1b,4] and electron rich aromatics[1b,5] (e.g., pyrroles, indoles). In these cases, the silylation is reversible and the use of triethylamine as co-solvent may be required to shift the equilibrium to the products. When extremely electron rich alkenes such as 1-alkoxy-1-dialkylaminoalkenes and 1,1-bis(dialkylamino)alkenes are silylated, the strongly basic sodium hydride is necessary to effect deprotonation.[4b]

Enamines are known to be the strongest, neutral, synthetically useful, π-nucleophiles[6] and it would be expected that their interaction with silyl triflates would afford very stable silyl iminium cations reluctant to undergo further transformations. Herein, we report that by varying the electron releasing ability of the silyl group, the reactivity of silyl iminium ions may be altered significantly. Two reagents exhibiting different electronic effects, namely trimethylsilyl triflate (Me_3SiOTf) and the recently introduced tris(pentafluorophenyl)silyl triflate[7] ($(C_6F_5)_3SiOTf$), were selected for the present study.

The interaction of Me_3SiOTf with enamine **2a** in $CDCl_3$ gave rise to the silyl iminium ion **3a**, which was identified by ^1H, ^{13}C, and ^{29}Si NMR spectroscopy[8] (Scheme 1). As expected, subsequent addition of triethylamine did not effect deprotonation of **3a**. This observation testifies in favor of the considerable conjugative stabilization of the iminium cation by the adjacent trimethylsilyl group.[9]



Scheme 1.

Keywords: Silyl triflates; Silylation; Enamines; Iminium cations.

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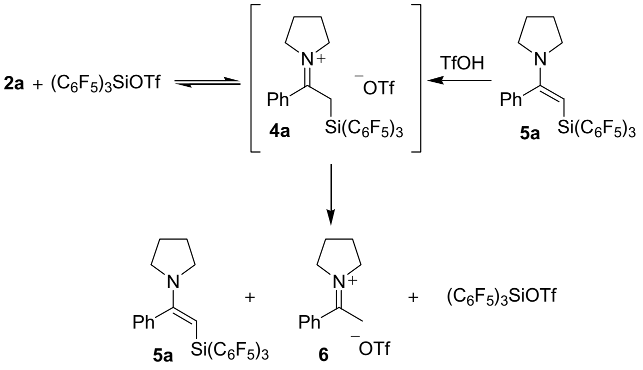
In contrast, the reaction of $(C_6F_5)_3SiOTf$ with enamine **2a** under the same conditions proceeded in a completely different way (Scheme 2). Instead of the anticipated silyl iminium ion **4a**, only silyl enamine **5a** and non-silylated iminium ion **6** were observed, along with half of the equivalent amount of unreacted silyl triflate (^1H and ^{19}F NMR control). Interestingly, attempted generation of cation **4a** by addition of triflic acid to the independently synthesized silyl enamine **5a** also afforded the mixture of **5a**, **6**, and $(C_6F_5)_3SiOTf$, thereby pointing to facile disproportionation of **4a**.[10] From these experiments, we can conclude that the formation of iminium ion **4a** from the enamine and silyl triflate is reversible and that **4a** can be easily deprotonated by enamine **2a**.



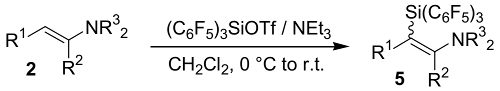
Scheme 2.

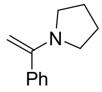
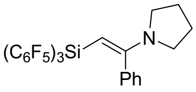
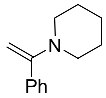
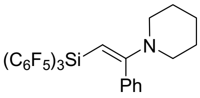
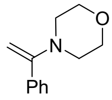
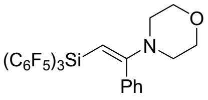
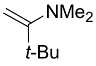
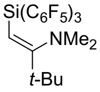
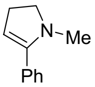
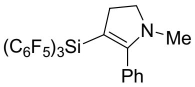
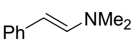
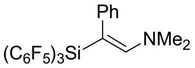
Presumably, owing to the influence of the three pentafluorophenyl rings the $(C_6F_5)_3Si$-fragment plays the role of electron acceptor and destabilizes the neighboring cationic center. While salt **4a** is very labile and prone to disproportionation, the presence of a tertiary amine base may favor the controlled formation of the silyl enamine. Indeed, the addition of an enamine to a mixture of $(C_6F_5)_3SiOTf$ and triethylamine smoothly furnished silyl enamines **5** in good yields (Table 1).[11,12]

In all cases, the silyl enamines were produced as single isomers with the sterically hindered tris(pentafluorophenyl)silyl group in the *trans* orientation with respect to the bulkier substituent. It is likely that steric effects influence the course of silylations of substrates bearing a substituent at the β-carbon atom. Thus, silylation of enamines derived from cyclopentanone, cyclohexanone, and propiophenone afforded complex mixtures that may be associated with difficulties in formation of tetrasubstituted double bonds. At the same time, silylation of enamine **2e**, in which the double bond is confined within a five-membered ring, proceeded quite efficiently. Furthermore, under standard conditions, enamine **2f** did not undergo the reaction even after prolonged periods of time (<5%, 20 h). However, addition of 10 mol % of pyridine allowed the reaction to occur cleanly affording the desired product.

Single crystals of **5a** and **5f** were investigated by X-ray diffraction analysis (Figs. 1 and 2).[13] In both structures,

Table 1. Synthesis of silyl enamines



Entry	Enamine		Silyl enamine		Reaction time	Yield of **5**, %[a]	Mp, °C
1		**2a**		**5a**[b]	15 min	81	149–151
2		**2b**		**5b**[c]	15 min	83	160–161
3		**2c**		**5c**[c]	15 min	92	156–158
4		**2d**		**5d**[c]	15 min	78	141–142
5		**2e**		**5e**	30 min	77	154–155
6		**2f**[d]		**5f**[b]	48 h	68	113–115

[a] In all cases, the yields of crude products were close to quantitative. The yields given in the table correspond to recrystallized material.

[b] Configuration was determined by X-ray diffraction analysis.

[c] Configuration was determined by NOE experiments.

[d] 10 mol % of pyridine was added upon silylation, see text.

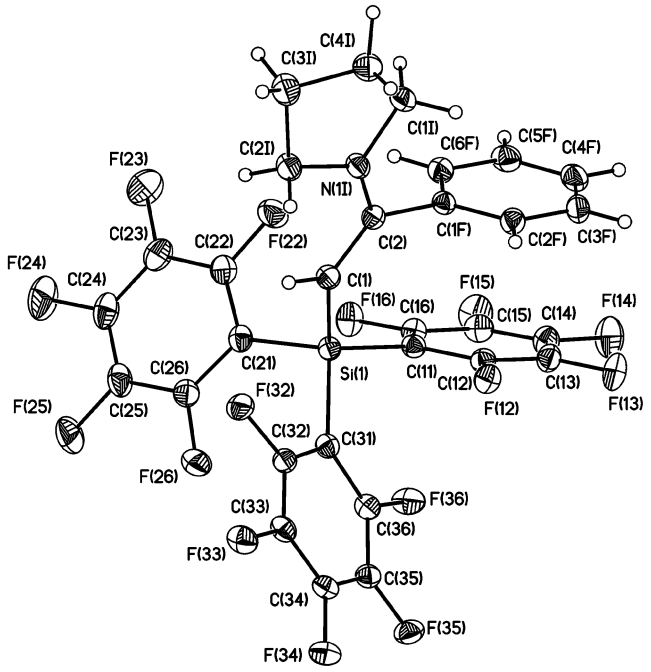


Figure 1. The molecular structure of **5a**. Atoms are presented as thermal ellipsoids at 50% probability. Selected bond lengths and angles (Å and °): Si(1)–C(1) 1.823(1), Si(1)–C(11) 1.897(1), Si(1)–C(21) 1.910(1), Si(1)–C(31) 1.915(1), C(1)–C(2) 1.375(2), C(2)–N(1I) 1.360(2); C(2)C(1)Si(1) 132.2(1), N(1I)C(2)C(1) 112.1(1), C(1)Si(1)C(11) 114.90(6), C(1)Si(1)C(21) 107.94(6), C(1)Si(1)C(31) 110.00(6).

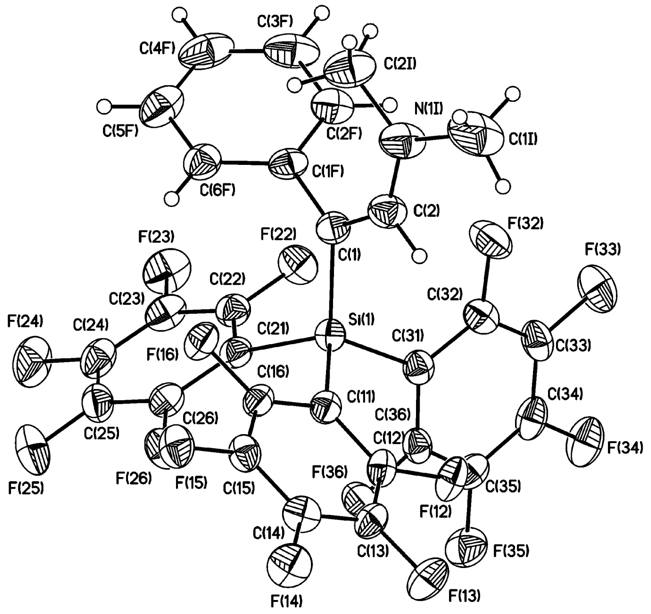


Figure 2. The molecular structure of **5f**. Atoms are presented as thermal ellipsoids at 50% probability. Selected bond lengths and angles (Å and °): Si(1)–C(1) 1.829(3), Si(1)–C(11) 1.898(3), Si(1)–C(21) 1.912(3), Si(1)–C(31) 1.902(3), C(1)–C(2) 1.360(4), C(2)–N(1I) 1.349(4); C(2)C(1)Si(1) 115.9(2), N(1I)C(2)C(1) 130.6(3), C(1)Si(1)C(11) 109.7(1), C(1)Si(1)C(21) 110.1(1), C(1)Si(1)C(31) 115.5(1).

the Si–C bond distance of 1.826 Å in the fragment $(C_6F_5)_3$*Si–C*=C is somewhat shorter than in an analogous vinyl silane (1.849 Å in $(C_6F_5)_3$*Si–C*H=CH_2[14]) and this may be associated with attractive interactions between the electron poor $(C_6F_5)_3$Si-group and the electron rich enamine system. The latter effect enforces steric interactions within the molecule leading to the elongation of other $Si–C_{C_6F_5}$ bonds up to 1.915 Å (vs the average of 1.886 Å for many other $RSi(C_6F_5)_3$ derivatives[14]) and distortion of the tetrahedral arrangement around silicon, the C–Si–C angles measuring up to 115° versus the conventional 109°. In addition, in the structure of **5a** the phenyl and one of the pentafluorophenyl rings have near parallel orientation with an interplanar angle and C(11)···C(1F) distance of 12.1° and 3.256 Å, respectively.

In summary, the interaction of silyl triflates with enamines leads to the generation of silyl iminium carbocations whose properties strongly depend on the nature of the silyl group. Iminium ions having trimethylsilyl substituents are stable, long-lived species, which can be observed spectroscopically. On the contrary, the tris(pentafluorophenyl)silyl-containing counterparts are labile intermediates prone to loss of a proton and produce silyl enamines in the presence of a base.

Acknowledgements

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2. Silylation of esters may furnish α-*C*-silyl esters, which are produced not directly, but through rearrangement of the initially formed silyl ketene acetals, that is, *O*-silylated compounds, see: Emde, H.; Simchen, G. *Liebigs Ann. Chem.* **1983**, 816.
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8. Compound **3a**: ^{1}H NMR (CDCl$_3$, 250.1 MHz), δ: −0.07 (s, 9H, SiMe$_3$), 1.97 (quint, 2H, $J = 6.6$, CH$_2$), 2.20 (quint, 2H, $J = 6.6$, CH$_2$), 2.78 (s, 2H, CH$_2$-Si), 3.82 (t, 2H, $J = 6.9$, NCH$_2$), 3.95 (t, 2H, $J = 6.9$, NCH$_2$), 7.35–7.62 (m, 5H, Ph). ^{13}C (62.9 MHz), δ: −0.9, 24.3, 24.6, 35.7, 54.0, 56.8, 127.0, 129.2, 132.4, 134.3, 184.9. ^{29}Si (INEPT, 59.6 MHz), δ: 9.33.
9. The hyperconjugative effect in *N*-methyl-2-(trialkylsilyl)-methylpyridinium cations possessing a silyl iminium fragment has been discussed: Hassall, K.; Lobachevsky, S.; White, J. M. *J. Org. Chem.* **2005**, *70*, 1993.
10. (a) Similar observations concerning the instability of Cl$_3$Si-substituted iminium ions were reported for the interaction of SiCl$_4$ with α-dimethylaminostyrene and 1,1-bis(dimethylamino)ethylene, see: Weingarten, H.; Wager, J. S. *J. Chem. Soc., Chem. Commun.* **1970**, 854; (b) Weingarten, H.; Wager, J. S. *Synth. React. Inorg. Met.-Org. Chem.* **1971**, *1*, 123.
11. General procedure: Under an argon atmosphere to solid tris(pentafluorophenyl)silyl triflate (678 mg, 1 mmol) were successively added dichloromethame (1.4 mL) and triethylamine (207 μL, 1.5 mmol). The mixture was cooled to 0 °C and the enamine was added in one portion with stirring. The cooling bath was removed and the mixture was stirred at room temperature for the time shown in Table 1. The solvent was evaporated, the residue was extracted with hot hexane (3 × 5 mL). The combined hexane solution was concentrated to half of the original volume, the residue was brought to reflux followed by slow cooling to −23 °C. The cold solvent was decanted, and the crystals were washed with 1 mL of cold hexane and then dried in vacuum.

    Compound **5a**:^{1}H NMR (CDCl$_3$, 250.1 MHz), δ:1.91 (br s, 4H, $\mathrm{C}\mathit{H}_2\mathrm{CH_2N}$), 3.14 (br s, 4H, $\mathrm{CH_2C}\mathit{H}_2\mathrm{N}$), 4.19 (s, 1H, =CH), 7.01–7.19 (m, 5H, Ph). ^{13}C (50.3 MHz), δ:25.7, 49.1 (br), 73.9, 108.2 (tm, $^2J_{\mathrm{C–F}} = 31.2$, i-$\mathrm{C_{C_6F_5}}$), 127.7, 128.2, 128.3, 137.3 (dm, $^1J_{\mathrm{C–F}} = 255.5$, $\mathrm{C_{C_6F_5}}$), 139.0, 142.7 (dm, $^1J_{\mathrm{C–F}} = 256.9$, $\mathrm{C_{C_6F_5}}$), 149.0 (dm, $^1J_{\mathrm{C–F}} = 245.6$, $\mathrm{C_{C_6F_5}}$), 163.0. ^{19}F (188.3 MHz), δ:−162.63 (m, *meta*), −151.73 (tt, $J_{\mathrm{F–F}} = 20.1$, 4.2, *para*), −127.56 (dm, $J_{\mathrm{F–F}} = 18.0$, *ortho*). Anal. Calcd for $\mathrm{C_{30}H_{14}F_{15}NSi}$:C, 51.36; H, 2.01%. Found:C, 51.26; H, 2.00%.

    Compound **5b**:^{1}H NMR (CDCl$_3$, 250.1 MHz), δ:1.50–1.71 (m, 6H, (CH$_2$)$_3$) 3.00–3.16 (m, 4H, (CH$_2$)$_2$N), 4.62 (s, 1H, =CH), 7.01–7.19 (m, 5H, Ph). ^{13}C (62.9 MHz), δ:24.7, 25.8, 49.4, 79.9, 107.8 (tm, $^2J_{\mathrm{C–F}} = 29.6$, i-$\mathrm{C_{C_6F_5}}$), 128.3, 128.7, 137.3 (dm, $^1J_{\mathrm{C–F}} = 254.9$, $\mathrm{C_{C_6F_5}}$), 139.5, 142.8 (dm, $^1J_{\mathrm{C–F}} = 256.7$,$\mathrm{C_{C_6F_5}}$), 149.0 (dm, $^1J_{\mathrm{C–F}} = 245.6$, $\mathrm{C_{C_6F_5}}$), 166.6. ^{19}F (188.3 MHz), δ: −162.36 (m, *meta*), −151.27 (t, $J_{\mathrm{F–F}} = 20.1$, 4.2, *para*), −127.47 (dm, $^3J_{\mathrm{F–F}} = 18.03$, *ortho*). Anal. Calcd for $\mathrm{C_{31}H_{16}F_{15}NSi}$: C, 52.04; H, 2.25%. Found: C, 52.04; H, 2.22%.

    Compound **5c**: ^{1}H NMR (CDCl$_3$, 250.1 MHz), δ: 3.02 (t, 4H, $J = 4.6$, (CH$_2$)$_2$N), 3.69 (t, 4H, $J = 4.6$, (CH$_2$)$_2$O), 4.71 (s, 1H, =CH), 7.06–7.19 (m, 5H, Ph). ^{13}C (75.5 MHz), δ: 48.5, 66.7, 82.7, 107.2 (tm, $^2J_{\mathrm{C–F}} = 28.4$, i-$\mathrm{C_{C_6F_5}}$), 128.5, 129.0, 137.3 (dm, $^1J_{\mathrm{C–F}} = 252.6$), 138.4; 142.9 (dm, $^1J_{\mathrm{C–F}} = 256.8$), 148.9 (dm, $^1J_{\mathrm{C–F}} = 244.1$), 166.6. ^{19}F (188.3 MHz), δ: −161.98 (m, *meta*), −150.59 (tt, $J_{\mathrm{F–F}} = 20.1$, 4.2, *para*), −127.29 (dm, $J_{\mathrm{F–F}} = 18.3$, *ortho*). Anal. Calcd for $\mathrm{C_{30}H_{14}F_{15}NOSi}$: C, 50.22; H, 1.97%. Found: C, 50.39; H, 1.99%.

    Compound **5d**: ^{1}H NMR (CDCl$_3$, 250.1 MHz), δ: 1.23 (s, 9H, *t*-Bu), 2.30 (s, 6H, NMe$_2$), 5.79 (s, 1H, =CH). ^{13}C (62.9 MHz), δ: 31.2, 41.3, 44.0, 107.8 (tm, $^2J_{\mathrm{C–F}} = 28.7$, i-$\mathrm{C_{C_6F_5}}$), 111.8, 137.5 (dm, $^1J_{\mathrm{C–F}} = 249.5$, $\mathrm{C_{C_6F_5}}$), 142.8 (dm, $^1J_{\mathrm{C–F}} = 256.4$, $\mathrm{C_{C_6F_5}}$), 149.1 (dm, $^1J_{\mathrm{C–F}} = 243.8$, $\mathrm{C_{C_6F_5}}$), 180.5. ^{19}F (188.3 MHz), δ: −161.90 (m, *meta*), −151.02 (tt, $J_{\mathrm{F–F}} = 20.1$, 3.8, *para*), −127.40 (dm, $J_{\mathrm{F–F}} = 18.0$, *ortho*). Anal. Calcd for $\mathrm{C_{26}H_{16}F_{15}NSi}$: C, 47.64; H, 2.46%. Found: C, 47.35; H, 2.44%.

    Compound **5e**: ^{1}H NMR (CDCl$_3$, 250.1 MHz), δ: 2.45 (s, 3H, NMe), 2.80 (t, 2H, $J = 9.5$, $\mathrm{C}\mathit{H}_2\mathrm{CH_2N}$), 3.44 (t, 2H, $J = 9.5$, $\mathrm{CH_2C}\mathit{H}_2\mathrm{N}$), 6.92–7.23 (m, 5H, Ph). ^{13}C (62.9 MHz), δ: 32.1, 36.4, 55.8, 90.8, 107.1 (tm, $^2J_{\mathrm{C–F}} = 26.9$, i-$\mathrm{C_{C_6F_5}}$), 128.2, 128.3, 128.7, 133.7, 137.3 (dm, $^1J_{\mathrm{C–F}} = 252.8$, $\mathrm{C_{C_6F_5}}$), 143.0 (dm, $^1J_{\mathrm{C–F}} = 256.4$, $\mathrm{C_{C_6F_5}}$), 149.1 (dm, $^1J_{\mathrm{C–F}} = 242.0$, $\mathrm{C_{C_6F_5}}$), 166.6. ^{19}F (188.3 MHz), δ: −162.22 (m, *meta*), −150.67 (t, $J_{\mathrm{F–F}} = 20.1$, *para*), −127.51 (dm, $J_{\mathrm{F–F}} = 18.7$, *ortho*). Anal. Calcd for $\mathrm{C_{29}H_{12}F_{15}NSi}$: C, 50.67; H, 1.76%. Found: C, 50.35; H, 1.76%. Enamine **5f** was obtained using the general procedure but with the addition of 10% of pyridine to the reaction mixture. ^{1}H NMR (CDCl$_3$, 250.1 MHz), δ: 2.67 (s, 6H, NMe$_2$), 6.38 (s, 1H, =CH), 6.96–7.13 (m, 5H, Ph). ^{13}C (50.3 MHz), δ: 43.1, 89.2, 107.5 (tm, $^2J_{\mathrm{C–F}} = 27.7$, i-$\mathrm{C_{C_6F_5}}$), 125.8, 127.6, 130.9, 137.5 (dm, $^1J_{\mathrm{C–F}} = 254.1$, $\mathrm{C_{C_6F_5}}$), 139.1, 143.2 (dm, $^1J_{\mathrm{C–F}} = 256.9$, $\mathrm{C_{C_6F_5}}$), 148.9, 149.2 (dm, $^1J_{\mathrm{C–F}} = 245.6$, $\mathrm{C_{C_6F_5}}$). ^{19}F (188.3 MHz), δ: −162.14 (m, *meta*), −150.50 (tt, $J_{\mathrm{F–F}} = 20.1$, 4.2, *para*), −126.81 (dm, $J_{\mathrm{F–F}} = 18.0$, *ortho*). Anal. Calcd for $\mathrm{C_{28}H_{12}F_{15}NSi}$: C, 49.79; H, 1.79%. Found: C, 49.72; H, 1.72%.
12. For other synthetic approaches to β-silyl enamines, see: (a) Kuno, S.; Sato, Y. *J. Organomet. Chem.* **1981**, *218*, 309; (b) Capella, L.; Capperucci, A.; Curotto, G.; Lazzari, D.; Dembech, P.; Reginato, G.; Ricci, A. *Tetrahedron Lett.* **1993**, *34*, 3311; (c) Marciniec, B.; Chadyniak, D.; Krompiec, S. *Tetrahedron Lett.* **2004**, *45*, 4065; (d) Timbart, L.; Cintrat, J.-C. *Chem. Eur. J.* **2002**, *8*, 1637.
13. Compound **5a**: Crystals of $\mathrm{C_{30}H_{14}F_{15}NSi}$ are triclinic at 120 K, space group $P\bar{1}$, $a = 9.873(3)$, $b = 12.026(4)$, $c = 13.288(4)$ Å, $\alpha = 63.190(5)$, $\beta = 75.250(6)$, $\gamma = 76.718(6)°$, $V = 1349.6(7)$ Å^3, $Z = 2$, $M = 701.51$, $d_{\mathrm{calcd}} = 1.726$ g cm^{-3}, μ(Mo Kα) = 2.16 cm^{-1}, $F(000) = 700$. Reflections (12,364) were collected, 7238 reflections were unique. $R_1 = 0.0427$ was calculated for 6143 reflections with $I > 2\sigma(I)$. Compound **5f**: Crystals of $\mathrm{C_{28}H_{12}F_{15}NSi}$ are monoclinic at 120 K, space group $P2_1/n$, $a = 11.820(4)$, $b = 19.156(7)$, $c = 12.160(4)$ Å, $\beta = 103.634(8)°$, $V = 2675.6(16)$ Å^3, $Z = 4$, $M = 675.48$, $d_{\mathrm{calcd}} = 1.677$ g cm^{-3}, μ(Mo Kα) = 2.14 cm^{-1}, $F(000) = 1344$. Reflections (12,371) were collected, 3833 reflections were unique. $R_1 = 0.0418$ was calculated for 3281 reflections with $I > 2\sigma(I)$. Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre (CCDC 263625 for **5a**, 263624 for **5f**) and are available free of charge at CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223 336033 or e-mail: deposit@ccdc.cam.ac.uk).
14. Dilman, A. D.; Arkhipov, D. E.; Korlyukov, A. A.; Ananikov, V. P.; Danilenko, V. M.; Tartakovsky V. A. *J. Organomet. Chem.*, submitted for publication.