



Fluoride ion catalyzed silylation of ketoximes by hydrosilanes

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Abstract

Silylation of aromatic and heteroaromatic oximes with hydrosilanes in the presence of system CsF/18-crown-6/benzene was studied. Silyl ethers were isolated in yields up to 78%. Mechanism of fluoride ion catalyzed silylation of oximes was proposed on the basis of quantum chemical calculations using the semi-empirical AM1 method.

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1. Introduction

Silylated derivatives of oximes are intensively studied as intermediates in organic synthesis and biologically active substances [1,2]. *O*-Trialkylsilyloximes were obtained in the reaction of corresponding oxime with trialkylchlorosilane/Et₃N [3], hydrosilane/ZnCl₂ [4], hydrosilane/H₂PtCl₆ [3], hydrosilane/piperidine [5], Et₃SiNH₂ [4], (Me₃Si)₂NH [6], and (Me₃Si)₂S [7]. Silyl ethers of oximes were also formed in the reaction of corresponding carbonyl compounds with H₂NOSiR₃ [8] or imines with mono-, di- or tri-silylated hydroxylamines [9].

The synthetic use of organosilicon compounds with catalysis by fluoride ion has been extensively studied [10–12]. The silylation of alcohols by R¹R²R³SiH in the presence of F[−] [13–17] or aminosilane/Bu₄NF [18] was described among these reactions. Re-

cently, we have performed silylation of oximes with Me₃SiC≡CH/CsF/18-crown-6 system [19]. However, fluoride ion mediated silylation of ketoximes by hydrosilanes was not investigated.

2. Experimental

2.1. Materials and methods

¹H and ¹³C NMR spectra were recorded on a “Varian Mercury” 200 spectrometer (200 MHz and 50 MHz correspondingly) using CDCl₃ as a solvent and TMS as internal standard. GC analysis was performed on a Chrom-5 instrument equipped with a flame-ionization detector using a glass column packed 5% OV-101/Chromosorb W-HP (80–100 mesh), 1.2 m × 3 mm. Ketoximes **1–6** were prepared from corresponding ketones according to the procedure described in the literature [20]. Hydrosilanes (Fluka) were used without additional purification. Benzene was distilled and dried over sodium. Cesium fluoride was calcined at 200 °C for 1 h.

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Table 1
Synthesis of silyl ethers of oximes in the PTC conditions at 50 °C

Oxime (<i>E:Z</i> ratio)	R	R ¹	Silane	Reaction Time (h)	Product (<i>E:Z</i> ratio)	Yield (%) ^a
1 (25:75)	Me	Et	PhMe ₂ SiH	10	1b (25:75)	77
1 (25:75)	Me	Et	Ph ₂ MeSiH	6	1c (25:75)	60
2 (100:0)	Me	Ph	Et ₃ SiH	10	2a^b (100:0)	12
2 (100:0)	Me	Ph	PhMe ₂ SiH	10	2b (100:0)	60
2 (100:0)	Me	Ph	Ph ₂ MeSiH	10	2c (100:0)	68
2 (100:0)	Me	Ph	Ph ₃ SiH	10	2d (100:0)	45
3 (100:0)	CHMe ₂	Ph	PhMe ₂ SiH	10	3b (100:0)	46
3 (100:0)	CHMe ₂	Ph	Ph ₂ MeSiH	10	3c (100:0)	78
4 (100:0)	Ph	Ph	Et ₃ SiH	10	4a^b (100:0)	32
4 (100:0)	Ph	Ph	PhMe ₂ SiH	10	4b (100:0)	58
5 (100:0)	Me	2-Furyl	PhMe ₂ SiH	10	5b (100:0)	16
6 (100:0)	Me	2-Pyridyl	PhMe ₂ SiH	6	6b (100:0)	23

Molar ratio of oxime:hydrosilane:CsF:18-crown-6 = 1:1:0.05:0.05.

^a Isolated yield.

^b [4,8].

2.2. Typical procedure for the fluoride mediated synthesis of silyl ethers of oximes **1b,c**, **2a–d**, **3b,c**, **4a,b**, **5b**, **6b**

Hydrosilane (1 mmol) was added to a suspension of oxime **1–6** (1 mmol), 18-crown-6 (0.013 g, 0.05 mmol), molecular sieves 4 Å (0.05 g) and freshly calcined CsF (0.0076 g, 0.05 mmol) in dry benzene (2 ml). The reaction mixture was stirred at 50 °C for 6–10 h. The solid substance was filtered off and the filtrate evaporated under reduced pressure to obtain *O*-silylethers of oximes. The results are shown in Table 1.

2.3. Calculations

All the calculations were carried out with the semi-empirical AM1 method [21] as implemented in MOPAC 6 program [22]. The equilibrium geometries were obtained with complete optimization at precise level [23]. All the optimized structures present minimum points on the potential energy surface as the frequency analysis has shown. To obtain the data on the change in geometry during the optimization process, calculations were performed using a keyword FLEPO.

Due to the absence in the MOPAC 6 program parameters of cesium we used potassium-like sparkle [22].

Post-processing computer visualization and animation was carried out with XMol [24] and JMol [25] programs.

3. Results and discussion

Synthesis of silyl ethers of oximes **1b,c**, **2a–d**, **3b,c**, **4a,b**, **5b**, **6b** were carried out using phase transfer catalytic (PTC) system oxime **1–6**/hydrosilane (R²R³R⁴SiH)/CsF/18-crown-6/benzene at 50 °C. It has been found that optimal amount of cesium fluoride is 5 mol% to oxime and hydrosilane. Increase of the amount of fluoride ion diminishes the yield of desired *O*-silylated oxime ethers. Silylated ketoximes were isolated in 13–78% yields (Tables 1–3). Experiments showed that silylation of ketoximes with aromatic hydrosilanes (Ph₃SiH, Ph₂MeSiH and PhMe₂SiH) afforded silylated oximes in considerably higher yields than in the presence of aliphatic one (Et₃SiH). Interestingly, the silyl ethers of aromatic ketoximes were isolated in higher yields than heteroaromatic ketoximes. Silylation of aldoximes under described conditions did not occur.

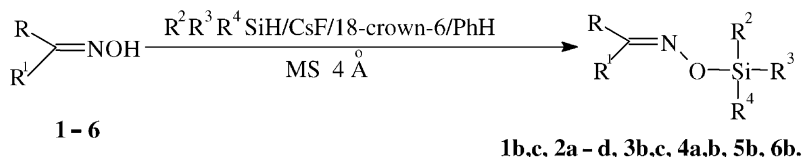


Table 2

¹H NMR and mass spectroscopic data for oxime *O*-silylethers **1b,c**, **2a–d**, **3b,c**, **4a,b**, **5b**, **6b**

Ether	M/z (relative intensity (%))	δ (ppm)
1b	221 (M ⁺ , 1), 206 (100), 147 (12), 137 (65), 121 (12), 105 (11), 91 (20), 77 (19), 70 (51)	<i>E</i> -Isomer: 0.18 (s, 6H, SiCH ₃), 0.76 (t, 3H, <i>J</i> = 7.8 Hz, CH ₂ CH ₃), 1.58 (s, 3H, CH ₃), 2.14 (q, 2H, <i>J</i> = 7.8 Hz, CH ₂ CH ₃), 7.1 and 7.34 (both m, 5H, Ph) <i>Z</i> -Isomer: 0.18 (s, 6H, SiCH ₃), 0.78 (t, 3H, <i>J</i> = 7.6 Hz, CH ₂ CH ₃), 1.61 (s, 3H, CH ₃), 1.92 (q, 2H, <i>J</i> = 7.6 Hz, CH ₂ CH ₃), 7.1 and 7.3 (both m, 5H, Ph)
1c	283 (M ⁺ , 1), 268 (100), 254 (13), 213 (14), 199 (82), 181 (13), 147 (45), 137 (39), 121 (17), 105 (21), 70 (40), 42 (57)	<i>E</i> -Isomer: 0.56 (s, 3H, SiCH ₃), 1.06 (t, 3H, <i>J</i> = 7.4 Hz, CH ₂ CH ₃), 1.84 (s, 3H, CH ₃), 2.47 (q, 2H, <i>J</i> = 7.8 Hz, CH ₂ CH ₃), 7.3 and 7.6 (both m, 10H, Ph) <i>Z</i> -Isomer: 0.72 (s, 3H, SiCH ₃), 1.03 (t, 3H, <i>J</i> = 7.4 Hz, CH ₂ CH ₃), 1.93 (s, 3H, CH ₃), 2.20 (q, 2H, <i>J</i> = 7.6 Hz, CH ₂ CH ₃), 7.3 and 7.6 (both m, 10H, Ph)
2a	249 (M ⁺ , 3), 220 (72), 192 (4), 151 (7), 118 (100), 103 (21), 77 (59), 59 (9), 45 (30)	0.77 (q, 6H, <i>J</i> = 8 Hz, SiCH ₂), 1.01 (t, 9H, <i>J</i> = 8 Hz, CH ₂ CH ₃), 2.57 (3H, s, CH ₃), 7.3–7.6 (m, 5H, Ph)
2b	269 (M ⁺ , 8), 254 (79), 213 (8), 195 (8), 137 (4), 118 (100), 91 (13), 77 (66), 51 (25)	0.31 (s, 6H, SiCH ₃), 2.30 (s, 3H, CH ₃), 7.3 (m, 10H, Ph)
2c	331 (M ⁺ , 7), 275 (2), 254 (17), 213 (8), 199 (73), 180 (12), 152 (13), 137 (26), 118 (84), 105 (17), 91 (16), 77 (100), 65 (12), 51 (34)	0.80 (s, 3H, SiCH ₃), 2.40 (s, 3H, CH ₃), 7.3 and 7.6 (m, 15H, Ph)
2d	393 (M ⁺ , 8), 378 (30), 316 (51), 259 (88), 199 (88), 181 (36), 152 (30), 118 (50), 105 (19), 77 (100), 65 (10), 51 (38)	2.41 (s, 3H, CH ₃), 7.68 (m, 20H, Ph)
3b	297 (M ⁺ , 23), 282 (34), 218 (18), 180 (15), 146 (82), 135 (59), 104 (100), 77 (74), 45 (30)	0.57 (s, 6H, SiCH ₃), 1.24 (d, 6H, <i>J</i> = 7.0 Hz, CH(CH ₃) ₂), 3.00 (heptet, 1H, <i>J</i> = 7.0 Hz, CH(CH ₃) ₂), 7.5 (m, 10H, Ph)
3c	347 (M ⁺ , 3), 344 (32), 280 (18), 197 (73), 180 (20), 146 (76), 104 (100), 77 (68)	0.77 (s, 3H, SiCH ₃), 1.22 (d, 6H, <i>J</i> = 7.2 Hz, CH(CH ₃) ₂), 2.84 (heptet, 1H, <i>J</i> = 7.2 Hz, CH(CH ₃) ₂), 7.3 and 7.6 (m, 15H, Ph)
4a	311 (M ⁺ , 2), 282 (24), 180 (100), 151 (8), 77 (43), 45 (15)	0.74 (q, 6H, <i>J</i> = 8.2 Hz, SiCH ₂), 0.97 (t, 9H, <i>J</i> = 8.2 Hz, CH ₂ CH ₃), 7.5 and 7.8 (both m, 10H, Ph)
4b	331 (M ⁺ , 7), 316 (20), 180 (100), 165 (10), 135 (14), 77 (50), 51 (16)	0.52 (s, 6H, SiCH ₃), 7.4 and 7.8 (both m, 15H, Ph)
5b	259 (M ⁺ , 51), 244 (100), 185 (22), 175 (79), 137 (81), 108 (62), 91 (23), 77 (25), 66 (19), 53 (20), 39 (50)	0.58 (s, 6H, SiCH ₃), 2.25 (s, 3H, CH ₃), 6.44 (dd, 1H, <i>J</i> _{3,4} = 3.6, <i>J</i> _{4,5} = 1.8 Hz, H ₄), 6.68 (dd, 1H, <i>J</i> _{3,4} = 3.6, <i>J</i> _{3,5} = 0.6 Hz, H ₃), 7.3 and 7.5 (both m, 5H, Ph), 7.55 (dd, 1H, <i>J</i> _{3,4} = 1.8, <i>J</i> _{3,5} = 0.6 Hz, H ₅)
6b	270 (M ⁺ , 2), 255 (100), 214 (15), 193 (63), 137 (52), 119 (58), 105 (18), 91 (16), 78 (75), 51 (33)	0.60 (s, 6H, SiCH ₃), 2.47 (s, 3H, CH ₃), 7.26 (m, 1H, H ₅), 7.4 and 7.7 (m, 5H, Ph), 7.61 (m, 1H, H ₃), 7.95 (m, 1H, H ₄), 8.63 (m, 1H, H ₆)

High efficiency of the PTC system CsF/18-crown-6/benzene system was recently demonstrated in the hydrosilylation [26], trimethylsilylcyanation [27], and trimethylsilylethynylation [28] reactions. Fluoride mediated silylation usually is selective. However, in some of the cases conversion of starting oxime was relatively low. The corresponding siloxanes (R²R³R⁴SiOSi R²R³R⁴) were isolated as minor products of the reac-

tion. The formation of such siloxanes is caused by the trace amount of water present in the reaction mixture. Experimental results showed that the formation of siloxanes (R²R³R⁴SiOSi R²R³R⁴) readily proceeds from oxime silyl ethers (RR¹C=NOSiR²R³R⁴) and hydrosilanes (HSiR²R³R⁴) in the presence of CsF and trace amounts of water. Our calculations confirm experimental result (as shown in Section 3.1).

Table 3
 ^{13}C NMR spectral data of oxime silyl ethers

Ether	δ (ppm)
1b	–1.99, 10.87, 13.97, 19.23, 29.12, 127.62, 129.41, 133.62, 137.91
1c	–2.99, 11.02, 13.99, 19.23, 29.10, 127.48, 129.48, 133.76, 137.56
2a	4.51, 6.78, 12.07, 126.06, 128.32, 128.919, 137.01
2b	–1.86, 12.31, 12.46, 126.02, 126.18, 127.67, 127.72, 132.96, 133.66, 136.47, 136.74
2c	–2.99, 12.30, 12.58, 126.25, 127.71, 129.17, 129.56, 133.93, 133.97, 135.73, 136.55
2d	12.35, 12.61, 126.29, 127.74, 129.20, 130.05, 133, 86, 134.94, 135.73, 136.37
3b	–2.00, 20.09, 28.64, 34.53, 127.53, 127.69, 128.08, 128.27, 132.94, 132.98, 136.12, 137.76
3c	–3.00, 12.58, 20.28, 34.55, 127.57, 127.60, 128.07, 128.10, 133.96, 134.55, 135.84, 135.89
4a	4.41, 6.70, 20.52, 127.64, 128.10, 132.33, 137.47
4b	–1.84, 20.83, 127.67, 128.05, 129.13, 129.22, 132.33, 132.93, 136.68, 137.35
6b	–1.86, 12.31, 29.68, 120.72, 127.77, 129.69, 132.96, 135.97, 137.31, 139.76, 154.67, 160.79

3.1. Mechanism of silylation

The quantum chemical study on silylation of acetophenone oxime with dimethylphenylsilane was carried out.

It is known that 18-crown-6 acts as “organic masking” agent for cesium ion. It provides “onium ion”-like entity that can be extracted or solubilized with the accompanying anion into organic solvent [29]. Thereby fluoride ion is transferred into benzene.

We suppose that the first reaction step is the interaction of F^- -ion with oxime group hydrogen atom carrying the largest positive charge (0.236 e) in molecule (Fig. 1, bond length is shown in Å unit).

The calculations have shown that the reaction proceeded without any activation barrier and leads

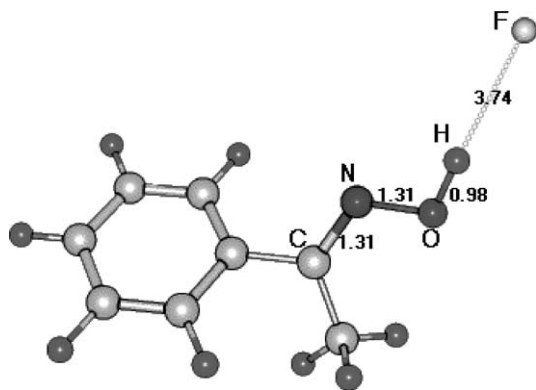


Fig. 1. The interaction of F^- -ion with oxime group hydrogen atom.

to the formation of oxime anion and complex of $\text{HF} \cdots \text{Cs}^+ \cdots 18\text{-crown-6}$ (Fig. 2). The reaction heat is -118.1 kcal/mol.

We suppose that oxime anion formed then interacted with dimethylphenylsilane. The calculations showed that heat of complex formation is equal to -5.9 kcal/mol. The formed complex is characterized by distance between O-atom in oxime and Si-atom (3.73 Å) (Fig. 3).

The positively charged complex $[\text{HF} \cdots \text{Cs} \cdots 18\text{-crown-6}]^+$ serves as source of proton. In our calculations, starting distance between hydrogen atom at silicon (H12) and the proton (H21) is equal to 2.16 Å (Fig. 4). Further reaction proceeds spontaneously.

Post-processing visualization showed that approaching of proton (H21) to the complex is accompanied

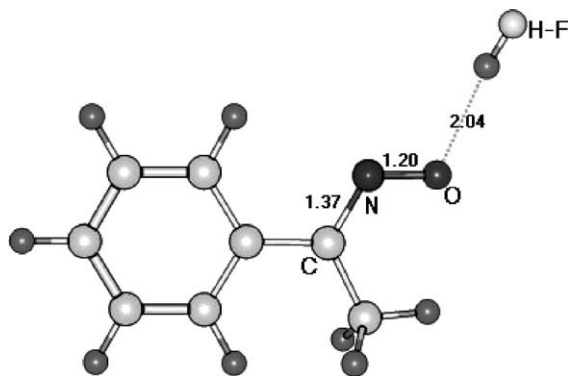


Fig. 2. The formation of oxime anion and HF.

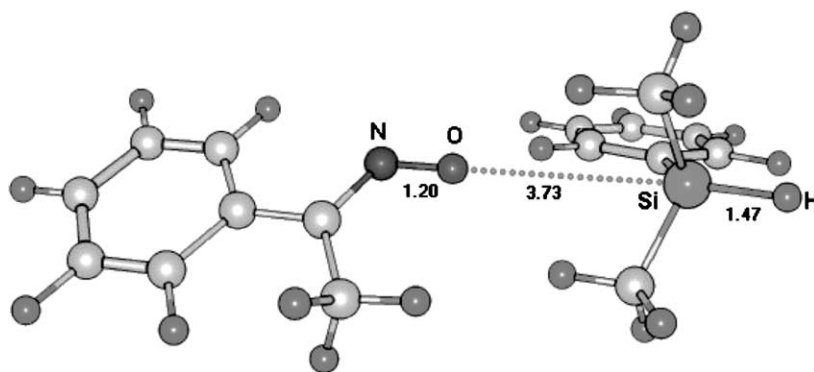


Fig. 3. The interaction of oxime anion with dimethylphenylsilane.

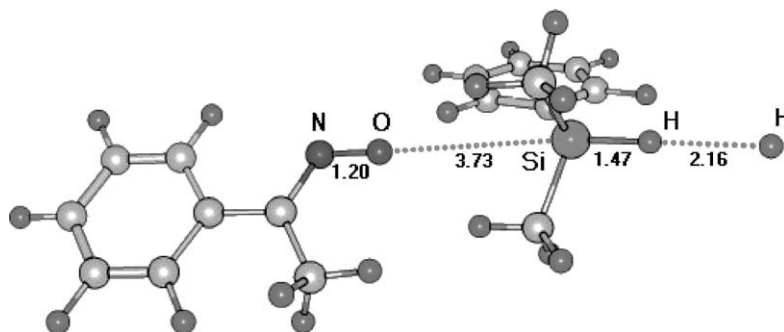


Fig. 4. The interaction between oxime anion and silicon complex and the proton.

by hydrogen atom possessing hydride properties (net charge is $-0.283 e$) take-off from silicon atom. In Fig. 5, it is shown a transition state where hydride atom (H12) position is near to symmetric to silicon and proton H21. After formation of hydrogen molecule the approaching of oxime anion and silicon cation takes place.

The reaction is completed with formation of bond between silicon and oxygen atoms (Fig. 6). The calculated reaction heat is -300.4 kcal/mol .

The experimental results show that in all the cases the corresponding siloxane ($R^2R^3R^4SiOSiR^2R^3R^4$) was isolated as minor product of the reaction. Obviously, the presence of siloxane formation in the

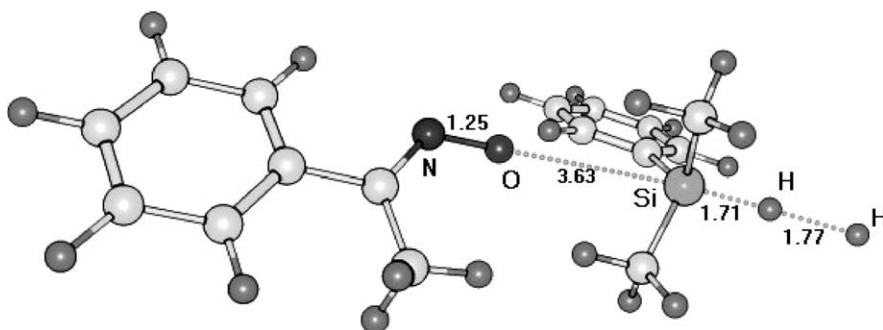


Fig. 5. The transition state forming hydrogen molecule.

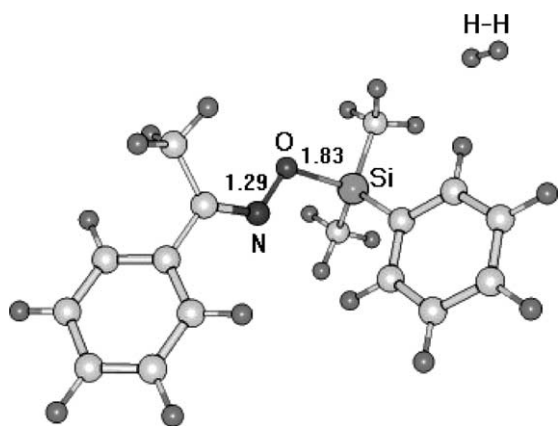


Fig. 6. The bond formation between silicon and oxygen atoms.

reaction mixture is a result of trace amounts of water. We suppose that the first reaction step of siloxane formation is hydration of cesium ion by interaction of Cs^+ with water. Fig. 7 shows the reaction initial state. Then spontaneously interaction between silylated oxime and hydrated cesium ion occurred. The calculations show that heat of reaction is equal to -103.5 kcal/mol. The bond order in silylated oximes between silicon and oxygen atoms changes from 0.718 to 0.320 (bond length increases from 1.833 to 2.044 Å; Fig. 8).

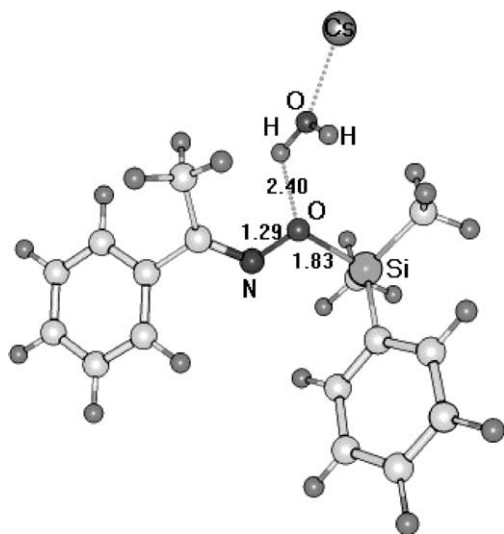


Fig. 7. The initial state of interaction of hydrated cesium with silylated oxime.

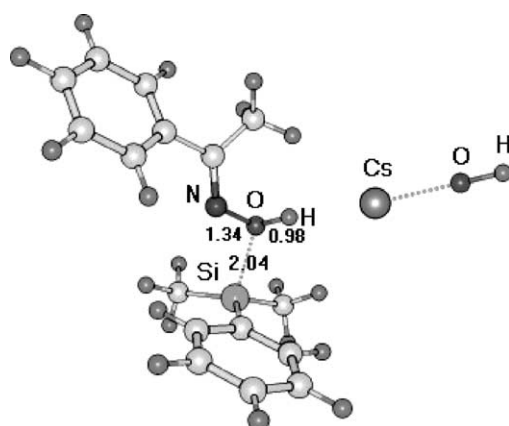


Fig. 8. The result of oxime protonation.

The next step is interaction of activated hydroxyl group with protonated silylated oxime intermediate (Fig. 9). The result of this process is breaking of the Si-O bond and formation of silanol and oxime (Fig. 10). The heat of reaction is equal to -205.7 kcal/mol.

We suppose that fluoride anion reacts with more positively charged hydrogen atom in hydroxyl group in silanol (Fig. 11) forming silanol anion and HF molecule (Fig. 12). The heat of reaction is equal to -83.0 kcal/mol. Negative charge of anion ($-0.937 e$) is located on the oxygen atom.

The next step of the reaction is the complex formation between silanol anion and hydrosilane (Fig. 13).

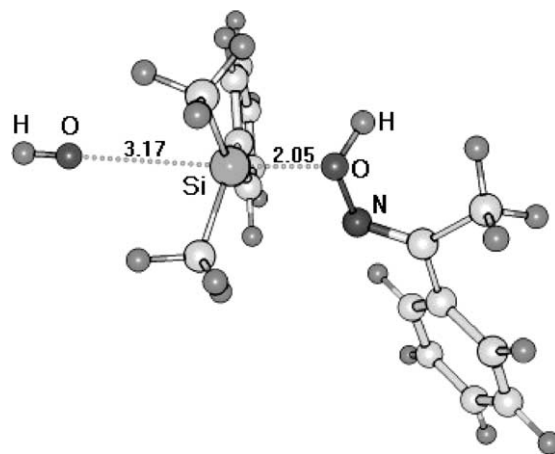


Fig. 9. The interaction of activated hydroxyl group with protonated silyl oxime intermediate.

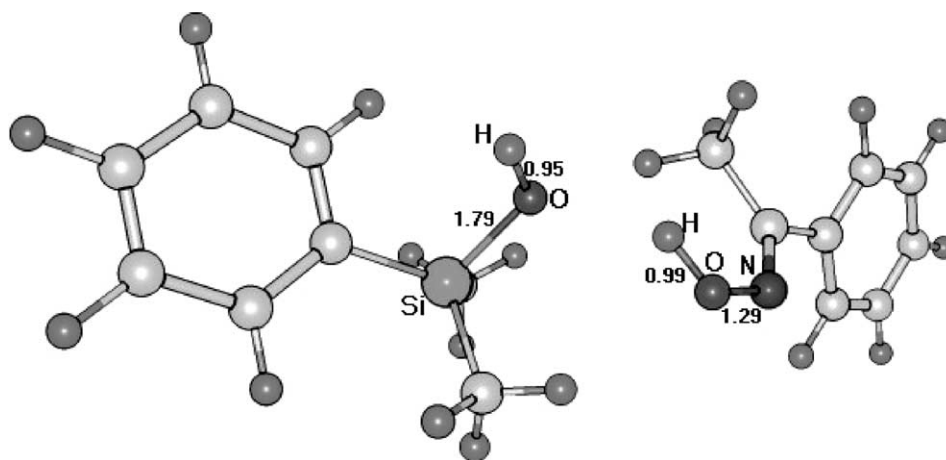


Fig. 10. The breaking of Si–O bond and formation of silanol and oxime.

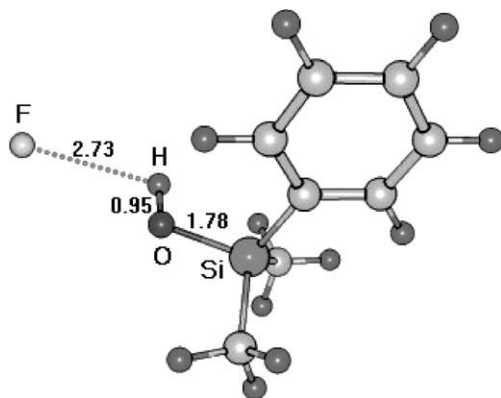


Fig. 11. The interaction of fluoride anion with hydrogen atom in silanol hydroxyl group.

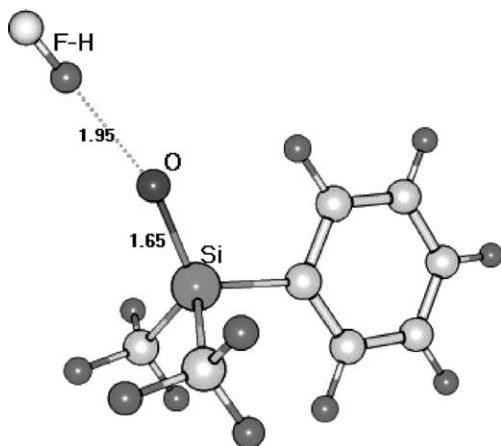


Fig. 12. The formation of silanol anion and HF molecule.

The energy of stabilization of this complex is equal to -9.7 kcal/mol.

The last step of the process is the interaction of earlier mentioned complex with hydrated cesium ion. Post-processing visualization showed that, at first, proton from complex $\text{HF} \cdots \text{Cs}^+ \cdots 18\text{-crown-6}$ interacts with negatively charged hydrogen atom in hydrosilane ($-0.297e$). The result of this interaction is the cleavage of hydrogen atom from hydrosilane

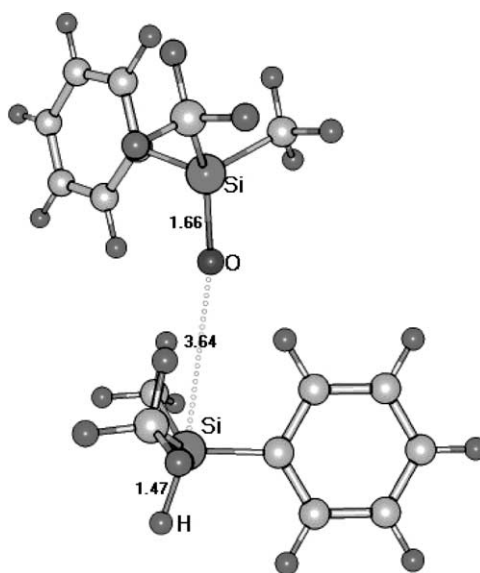


Fig. 13. The complex formation between silanol anion and hydrosilane.

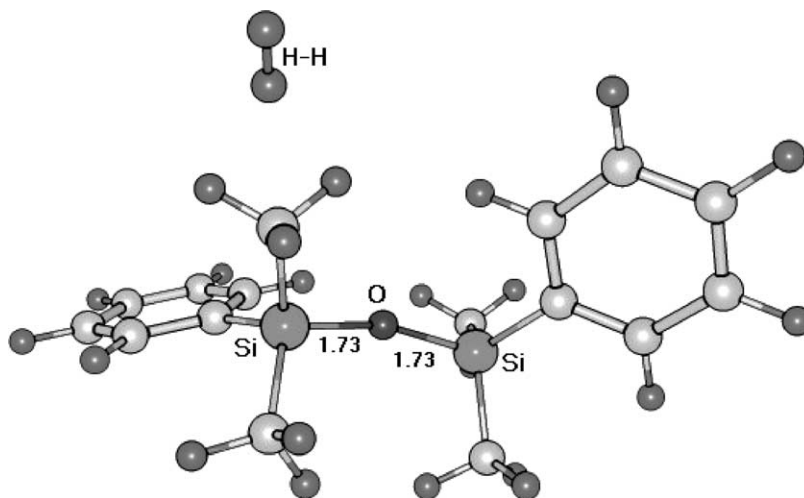


Fig. 14. The hydrogen molecule and siloxane formation.

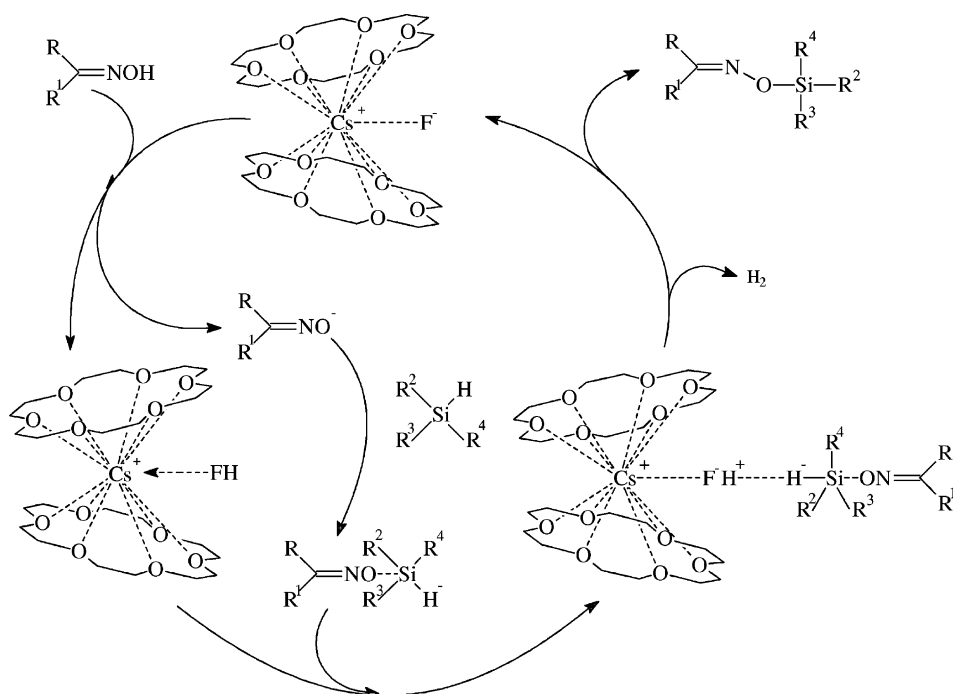


Fig. 15. Fluoride ion mediated silylation of ketoximes by hydrosilanes.

producing hydrogen molecule. The following siloxane ($R^2R^3R^4SiOSi R^2R^3R^4$) formation occurred (Fig. 14). The heat of reaction is equal to -285.8 kcal/mol.

4. Conclusions

It has been shown that silylation of aliphatic, aromatic and heteroaromatic oximes with hydrosilanes

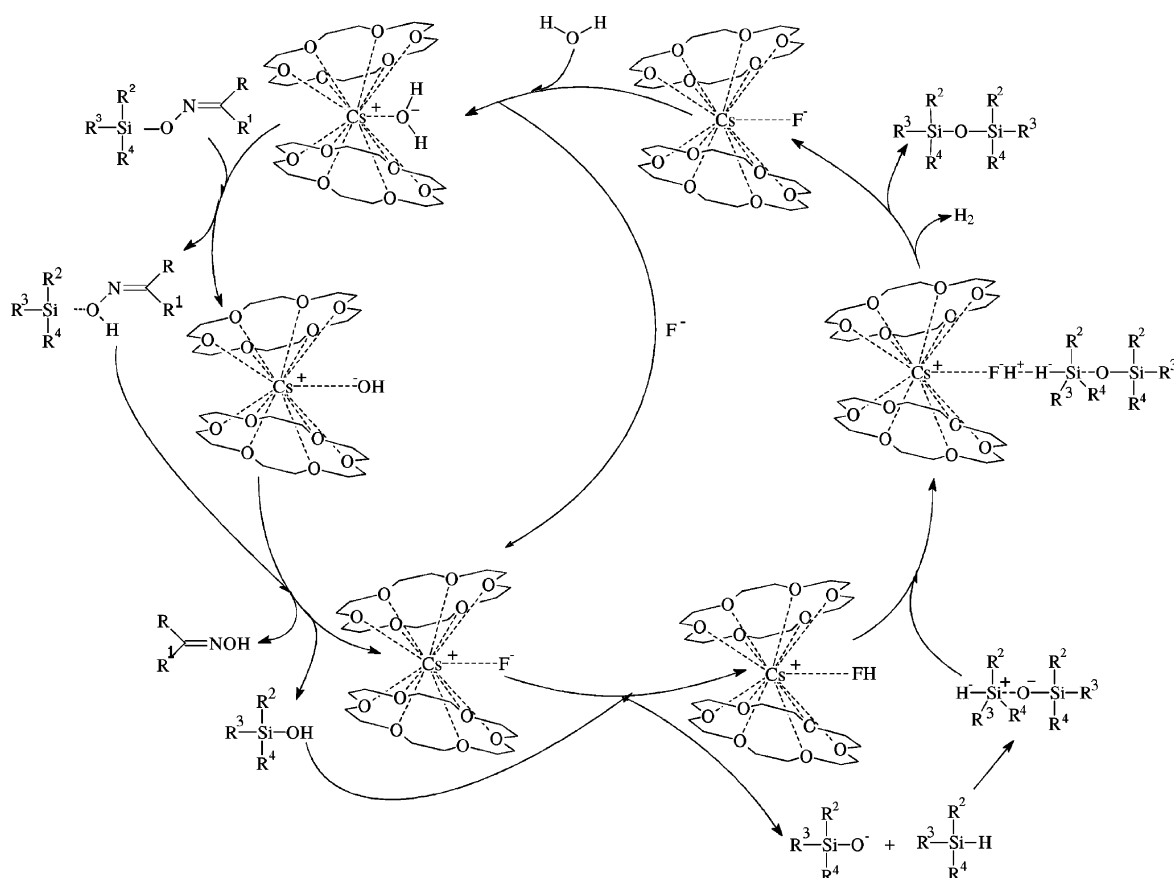


Fig. 16. Formation of symmetric siloxanes from oxime silyl ethers and hydrosilanes.

readily proceeds in the presence of catalytic amount of cesium fluoride and 18-crown-6.

The quantum chemical calculations allow to propose the mechanism of silylation of oximes shown in Fig. 15 and formation of symmetric siloxanes from oxime silyl ethers and hydrosilanes shown in Fig. 16.

Supporting information available: Multi-structure XYZ format files and/or video clips files, containing data of the change of system geometry and energy during the reaction of synthesis of oxime silyl ethers. This material is available from the authors on request.

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