

Synthesis and crystal structure of enantiomeric *N*-(dimethylfluorosilylmethyl) and *N*-(dimethylchlorosilylmethyl)-[*N*-(*S*)-1-phenylethyl] acetamides¹

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Abstract

Synthesis of the first optically active compounds containing asymmetric carbon and pentacoordinated silicon has been carried out. The enantiomeric (O–Si)-chelate *N*-(dimethylhalogenosilylmethyl) acetamides, MeC(O)N(CH(Ph)Me)CH₂SiMe₂X (X = F, Cl), obtained are the model compounds in studies of the stereodynamic processes at the trigonal-bipyramidal silicon atom and asymmetric induction. An X-ray structural investigation confirmed a 3 + 2 pentacoordination of the Si atom in the title compounds and an axial arrangement of the oxygen and halogen atoms. The geometrical parameters of hypervalent moieties are in good agreement with relations established earlier. © 1997 Elsevier Science S.A.

Keywords: Silicon; Pentacoordinated organosilicon compounds; Intramolecular coordination; Optically active compounds; X-ray study

1. Introduction

Among the hypervalent silicon compounds [1], Si-substituted *N*-(dimethylsilylmethyl) amides and lactams, wherein in the case of sufficiently electronegative Si-substituents the Si atom is pentacoordinated due to the intramolecular O → Si coordinative bond, have been studied rather well [2]. Simple general methods for their preparation have been developed [3–8], from which a wide range of such compounds containing various Si-substituents can be obtained. Some of their structural features have been investigated in detail: the effects of the Si-substituent nature, the size of the lactam cycle and heteroatoms present on the spatial structure of the hypervalent SiC₃OX moiety (see the review in Ref. [2] and references cited therein, and also Refs. [9,10]).

However, few known compounds containing this

moiety, or the related SiC₂OX₂ and SiCOX₃ units, have chiral labels, which would allow the study of the stereodynamics of such units. Among these compounds are [α -(*N*-pyrrolidonyl-2-ethyl)trifluorosilane [11], 4-phenyl-*N*-(dimethylsilylmethyl)-2-pyrrolidone chloride and trifluoroacetate [12], *N*-(methyl- α -naphthylchlorosilylmethyl)hexahydro-2-azepinone [13], and *N*-methyl-*N*-(methylphenylchlorosilylmethyl) acetamide [14].

No data are available on pentacoordinated silicon compounds containing an additional configurationally stable stereogenic centre, which would be useful for studying the possibility of transferring the asymmetric induction through the trigonal-bipyramidal silicon atom.

In this work, we present our recent results on the synthesis and structure of the first optically active compounds containing asymmetric carbon and pentacoordinated silicon, i.e. enantiomers *N*-(dimethylfluorosilylmethyl)-*N*-[(*S*)-1-phenylethyl] acetamide (**I**) and *N*-(dimethylchlorosilylmethyl)-*N*-[(*S*)-1-phenylethyl] acetamide (**II**).

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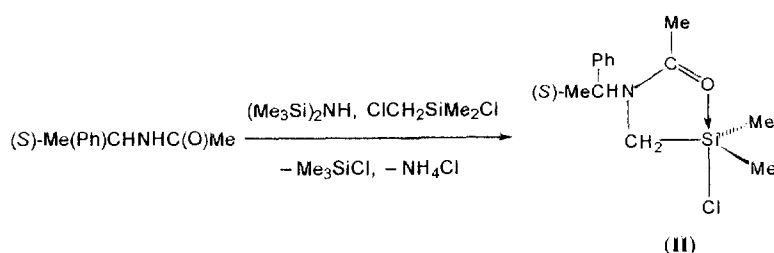
¹ Dedicated to the memory of Yu.T. Struchkov, whose life came to an end before this work was finished.

2. Results and discussion

2.1. Synthesis and structure

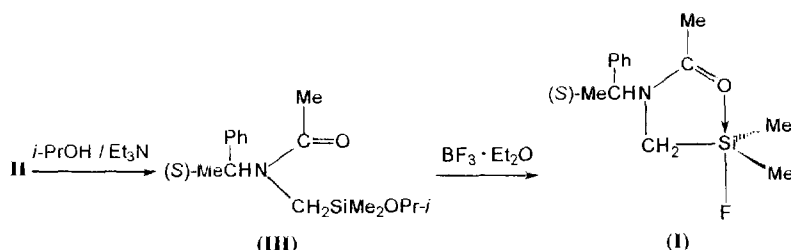
For the *N*-dimethylchlorosilylmethylation of amides and lactams, their reaction with dimethylchloromethylchlorosilane in the presence of triethylamine (amination) or interaction of $\text{ClCH}_2\text{SiMe}_2\text{Cl}$ with their *N*-tri-

methylsilyl derivatives (*trans*-silylation) is usually applied [3,4]. We showed recently that a one-bulb method using amide or lactam, hexamethyldisilazane and dimethylchloromethylchlorosilane gives the best results [6]. This method was used in the present work for synthesis of chloride (**II**). *N*-Acetyl-[(*S*)-1-phenylethyl] amine served as the starting compound with a carbon chiral centre [15].



The synthesis of fluoride **I** was carried out using chloride **II** as the initial compound, through appropriate isopropoxide (**III**) by the method described earlier for

producing *N*-(dimethylfluorosilylmethyl)hexahydro-2-azepinone [8].



Similar to the other *N*-(dimethylfluorosilylmethyl) and *N*-(dimethylchlorosilylmethyl) amides and lactams already described [3,4,6–8], in halogenides **I** and **II** the silicon atom is pentacoordinated due to the intramolecular $\text{O} \rightarrow \text{Si}$ interaction. This is evident from the presence of two characteristic absorption bands in the $1500\text{--}1700\text{ cm}^{-1}$ region of the IR spectra which correspond to the strongly coupled stretching vibrations ($\text{C}=\text{O}$) and ($\text{C}=\text{N}$) in the amide fragments, and also from the high-field shift in the ^{29}Si NMR spectra ($\delta - 19.3$ ppm for fluoride **I**, $\delta - 38.1$ ppm for chloride **II**, 20°C , CDCl_3), compared with model compounds of the tetracoordinate silicon [2].

The protons of the pro-chiral groups NCH_2 and SiMe_2 are diastereotopic by virtue of the chirality of the substituent at the nitrogen atom. As a result, at room temperature, protons of the NCH_2 group appear in the ^1H NMR spectra as the AB pattern ($^2J_{\text{HH}} \sim 16$ Hz) and protons of the SiMe_2 group appear as an equal doublet.

Table 1
Atomic coordinates ($\times 10^4$) in structure **I**

Atom	x	y	z
Si(1)	8373.4(6)	356.6(6)	2967.8(6)
F(1)	8493(2)	-701(2)	1971(2)
O(1)	7980(2)	1755(2)	4168(2)
N(1)	6052(2)	1482(2)	3423(2)
C(1)	6657(3)	668(2)	2616(2)
C(2)	6819(2)	2014(2)	4180(2)
C(21)	6342(3)	2889(3)	5043(3)
C(3)	4673(2)	1705(2)	3315(2)
C(30)	4382(3)	2325(3)	2173(3)
C(31)	3952(2)	562(2)	3514(2)
C(32)	4082(2)	-26(2)	4566(2)
C(33)	3387(3)	-1032(2)	4801(2)
C(34)	2526(3)	-1462(3)	3989(3)
C(35)	2407(3)	-903(2)	2935(3)
C(36)	3112(2)	106(2)	2698(2)
C(4)	9615(3)	1321(3)	2358(2)
C(5)	8746(4)	-546(3)	4271(3)

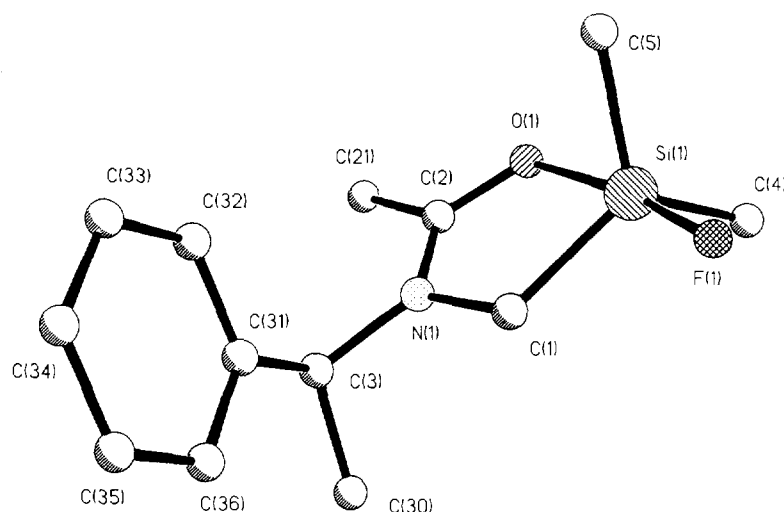


Fig. 1. General view of molecule **I** in the crystal. Hydrogen atoms are omitted.

Similar to *N*-(dimethylalkoxysilylmethyl) lactams [5], the intramolecular interaction $O \rightarrow Si$ in isopropoxide **III** is practically absent. Its IR spectrum in the $1500\text{--}1700\text{ cm}^{-1}$ region reveals only one absorption band at 1640 cm^{-1} (NCO), and no significant high-field shift in the ^{29}Si NMR spectrum is observed ($\delta \sim 5\text{--}6\text{ ppm}$, 20°C , CDCl_3). By comparison, the ^{29}Si shift of the model compound $(\text{CH}_2\text{Cl})\text{Si}(\text{CH}_3)_2\text{OCH}_3$ with the tetracoordinate Si is 12.9 ppm [16].

2.2. X-ray study

As follows from a comparison of the parameters of the elementary cells and the coordinates of the respective atoms, the compounds **I** and **II** are isostructural. Atomic coordinates in these basis molecules (Tables 1 and 2), if small divergences of the geometry of molecules and cells are taken into account, are con-

nected by the transformation $(1-x, -y, 0.5+z)$. The conformation of the molecule (Fig. 1) is rather rigid. Its form is determined by the configuration of the asymmetric atom C(3), which has the type (*S*) in both compounds according to X-ray data. The bond length and the valent angles in chemically identical parts of molecules **I** and **II** actually coincide (Tables 3–6).

Essentially, the structures of the hypervalent fragments differ owing to the different properties of the F- and Cl-substituents at the Si atom. The less mobile electronic system of the SiC_3F unit forms a weaker hypervalent bond with the acetamide fragment than with the SiC_3Cl unit, as demonstrated by the markedly greater $O \rightarrow Si$ bond length, and by the greater deviation Δ of the Si atom from the equatorial plane: 0.20 \AA in fluoride **I** vs. 0.05 \AA in chloride **II**.

These parameters fit well the $d(\Delta)$ curves which were obtained by us for pentacoordinated silicon monochlorides [10]. As for their F-analogues, only one similar structure is reported, i.e. *N*-(dimethylfluorosilylmethyl)-2-pyrrolidone [17], in which four crystallographically independent molecules have the $O \rightarrow Si$ bond lengths $2.32\text{--}2.46\text{ \AA}$, Si-F $1.63\text{--}1.67\text{ \AA}$ and the Δ parameters $0.24\text{--}0.31\text{ \AA}$. Comparing the latter with the appropriate values in structure **I**, it is possible to con-

Table 2
Atomic coordinates ($\times 10^4$) in structure **II**

Atom	x	y	z
Si(1)	1756.6(5)	-513.5(5)	8124.7(5)
Cl(1)	1576.0(6)	1067.1(5)	6893.2(6)
O(1)	2172(1)	-1911(1)	9060(1)
N(1)	4064(2)	-155.3(2)	8372(2)
C(1)	3445(2)	-638(2)	7689(2)
C(2)	3317(2)	-2177(2)	9040(2)
C(21)	3775(3)	-3192(2)	9740(2)
C(3)	5408(2)	-1756(2)	8223(2)
C(30)	5648(2)	-2239(3)	7021(3)
C(31)	6110(2)	-624(2)	8518(2)
C(32)	5866(2)	-45(2)	9547(2)
C(33)	6528(2)	964(2)	9850(2)
C(34)	7461(2)	1393(2)	9138(2)
C(35)	7710(2)	826(2)	8116(2)
C(36)	7042(2)	-177(2)	7811(2)
C(4)	507(2)	-1339(2)	7377(2)
C(5)	1353(3)	364(3)	9416(2)

Table 3
Bond lengths d in structure **I**

Bond	$d/\text{\AA}$	Bond	$d/\text{\AA}$
Si(1)-F(1)	1.668(2)	C(2)-C(21)	1.494(4)
Si(1)-O(1)	2.149(2)	C(3)-C(30)	1.526(4)
Si(1)-C(1)	1.891(3)	C(3)-C(31)	1.522(3)
Si(1)-C(4)	1.848(3)	C(31)-C(32)	1.392(3)
Si(1)-C(5)	1.863(3)	C(31)-C(36)	1.394(3)
O(1)-C(2)	1.260(3)	C(32)-C(33)	1.385(4)
N(1)-C(1)	1.460(3)	C(33)-C(34)	1.394(4)
N(1)-C(2)	1.336(3)	C(34)-C(35)	1.378(4)
N(1)-C(3)	1.482(3)	C(35)-C(36)	1.393(4)

Table 4
Bond lengths d in structure II

Bond	$d/\text{\AA}$	Bond	$d/\text{\AA}$	Bond	$d/\text{\AA}$
Si(1)–Cl(1)	2.306(1)	N(1)–C(1)	1.469(3)	C(31)–C(32)	1.397(3)
Si(1)–O(1)	1.975(2)	N(1)–C(2)	1.326(3)	C(31)–C(36)	1.39(3)
Si(1)–C(1)	1.894(2)	N(1)–C(3)	1.476(3)	C(32)–C(33)	1.391(4)
Si(1)–Cl(4)	1.858(3)	C(3)–C(30)	1.533(4)	C(34)–C(35)	1.385(4)
Si(1)–C(5)	1.861(3)	C(2)–C(21)	1.495(3)	C(33)–C(34)	1.393(4)
O(1)–C(2)	1.269(3)	C(3)–C(31)	1.526(3)	C(35)–C(36)	1.391(3)

clude that for the hypervalent O → Si–F bond the $d(\Delta)$ dependences are similar to those described in Ref.[10].

3. Experimental details

3.1. General

The X-ray diffraction experiment for compounds I and II was carried out with a Siemens P3/PC diffractometer (λ Mo K α , graphite monochromator, θ – 2θ scan, $2\theta_{\max} = 56^\circ$ (I) and 60° (II)). Structures were solved by direct methods and refined by full-matrix least squares in anisotropic approximation for nonhydrogen atoms. H atoms located by difference synthesis were refined isotropically.

Crystals I (C₁₃H₂₀NOFSi) are orthorhombic, at -130°C $a = 10.558(2)$, $b = 11.355(3)$, $c = 11.543(4)$ Å, $V = 1384(1)$ Å³, $d_{\text{calc}} = 1.216$ g cm⁻³, $\mu = 0.17$ mm⁻¹, $Z = 4$, space group $P2_12_12_1$. The final discrepancy factors were $R = 0.037$, $R_w = 0.044$ for 1624 reflections with $I > 2\sigma(I)$; for the inverted structure $R = 0.038$, $R_w = 0.038$.

Crystals II (C₁₃H₂₀NOClSi) are orthorhombic, at -120°C $a = 10.771(4)$, $b = 11.313(4)$, $c = 11.717(5)$ Å, $V = 1427.8(10)$ Å³, $d_{\text{calc}} = 1.255$ g cm⁻³, $\mu = 0.34$ mm⁻¹, $Z = 4$, space group $P2_12_12_1$. The final discrepancy factors were $R = 0.031$, $R_w = 0.032$ for 2123

reflections with $I > 3\sigma(I)$; for the inverted structure $R = 0.032$, $R_w = 0.033$.

According to the Hamilton test [18], the determined absolute structures are true with a probability exceeding 0.99. Atom coordinates in structures I and II are listed in Tables 1 and 2. All calculations were carried out with an IBM PC/AT computer using the SHELXTL PLUS programs [19]. Tables of hydrogen atom coordinates and anisotropic displacement parameters have been deposited at the Cambridge Crystallographic Data Centre.

IR spectra in 5% CHCl₃ solutions or thin films were measured with a Specord IR-75 instrument in KBr cells.

The specific rotation for compounds in solutions were determined with an EPO-1A VNIKIPRODMASH polarimeter to the accuracy of 0.01° .

The ¹H and ²⁹Si spectra were recorded on a Varian XL-400 spectrometer, at 400.1 MHz and 79.5 MHz respectively, in a pulsing mode with the subsequent Fourier transformation. The chemical shifts were measured using tetramethylsilane as internal reference for 0.5 M solutions.

Initial *N*-acetyl-(*S*)-(1-phenylethyl)amine was prepared in 65% yield by the reaction of (*S*)- α -phenylethylamine with acetic anhydride, m.p. 105 – 106°C (benzene–pentane, 10:1), $[\alpha]_D -124.6^\circ$ (c 2.3, CH₂Cl₂), -153.0° (c 2.0, CH₃OH) [15]. Its optical purity estimated from the specific rotation is about 100%.

Table 5
Bond angles ω in structure I

Angle	ω/deg	Angle	ω/deg
F(1)–Si(1)–O(1)	172.7(1)	O(1)–C(2)–N(1)	118.5(2)
F(1)–Si(1)–C(1)	93.4(1)	O(1)–C(2)–C(21)	119.3(2)
O(1)–Si(1)–C(1)	79.3(1)	N(1)–C(2)–C(21)	122.1(2)
F(1)–Si(1)–C(4)	96.3(1)	N(1)–C(3)–C(30)	110.4(2)
O(1)–Si(1)–C(4)	85.8(1)	N(1)–C(3)–C(31)	109.5(2)
C(1)–Si(1)–C(4)	119.2(1)	C(30)–C(3)–C(31)	115.1(2)
F(1)–Si(1)–C(5)	98.3(1)	C(3)–C(31)–C(32)	119.4(2)
O(1)–Si(1)–C(5)	85.8(1)	C(3)–C(31)–C(36)	122.2(2)
C(1)–Si(1)–C(5)	118.6(1)	C(32)–C(31)–C(36)	118.3(2)
C(4)–Si(1)–C(5)	118.9(1)	C(31)–C(32)–C(33)	121.0(2)
Si(1)–O(1)–C(2)	111.5(2)	C(32)–C(33)–C(34)	120.2(2)
C(1)–N(1)–C(2)	116.0(2)	C(33)–C(34)–C(35)	119.4(3)
C(1)–N(1)–C(3)	118.9(2)	C(34)–C(35)–C(36)	120.3(3)
C(2)–N(1)–C(3)	125.0(2)	C(31)–C(36)–C(35)	120.8(2)
Si(1)–C(1)–N(1)	113.6(2)		

Table 6
Bond angles ω in structure II

Angle	ω/deg	Angle	ω/deg
Cl(1)–Si(1)–O(1)	170.8(1)	O(1)–C(2)–N(1)	118.4(2)
Cl(1)–Si(1)–C(1)	88.3(1)	O(1)–C(2)–C(21)	119.5(2)
O(1)–Si(1)–C(1)	82.7(1)	N(1)–C(2)–C(21)	122.1(2)
Cl(1)–Si(1)–C(5)	94.3(1)	N(1)–C(3)–C(30)	109.2(2)
O(1)–Si(1)–C(5)	91.6(1)	N(1)–C(3)–C(31)	109.2(2)
C(1)–Si(1)–C(5)	118.9(1)	C(30)–C(3)–C(31)	115.0(2)
Cl(1)–Si(1)–C(4)	91.9(1)	C(3)–C(31)–C(32)	119.7(2)
O(1)–Si(1)–C(4)	91.3(1)	C(3)–C(31)–C(36)	121.7(2)
C(1)–Si(1)–C(4)	122.1(1)	C(32)–C(31)–C(36)	118.5(2)
C(4)–Si(1)–C(5)	118.9(1)	C(31)–C(32)–C(33)	120.6(2)
Si(1)–O(1)–C(2)	113.5(1)	C(32)–C(33)–C(34)	120.2(2)
C(1)–N(1)–C(2)	114.9(2)	C(33)–C(34)–C(35)	119.7(2)
C(1)–N(1)–C(3)	119.4(2)	C(34)–C(35)–C(36)	120.0(2)
C(2)–N(1)–C(3)	125.6(2)	C(31)–C(36)–C(35)	121.0(2)
Si(1)–C(1)–N(1)	110.0(2)		

3.2. (O-Si)-chelate *N*-(dimethylchlorosilylmethyl)-*N*-[(*S*)-1-phenylethyl] acetamide (**II**)

To a mixture of *N*-acetyl-(*S*)-(1-phenylethyl)amine (8.16 g, 0.05 M) and hexamethyldisilazane (3.22 g, 0.02 M) in 70 ml absolute benzene 7.15 g (0.05 M) of dimethylchloromethylchlorosilane was added with stirring. The reaction mixture was refluxed for 2 h. After cooling, the resulting precipitate was filtered. On evaporation of the benzene solution the residue was crystallized. The yield of **II** was 11.0 g (82%). M.p. 141–145 °C (benzene–hexane, 1:1), $[\alpha]_D^{20} + 48.8^\circ$, (*c* 2.1, CH₂Cl₂). IR (ν , cm⁻¹, CHCl₃): 1513, 1578 (NCO). ¹H NMR (δ , ppm, CDCl₃): 0.57 (s, 6H, SiMe₂), 1.68 (d, 3H, *C–CH₃, ³J_{HH} 6.9 Hz), 2.26 (t, 3H, CH₃CO, ⁵J_{HH} 1.0 Hz), 2.47 d.q., 2.75 d.q. (2H, NCH₂, ²J_{HH} 16.2 Hz, ⁵J_{HH} 0.9 Hz), 5.05 (q, 1H, *C–CH, ³J_{HH} 6.9 Hz), 7.4–7.52 (m, 5H, C₆H₅). Anal. Found: C, 57.70; H, 7.45; Si, 11.09. C₁₃H₂₀ClNOSi. Calc.: C, 57.85; H, 7.46; Si, 10.40%.

3.3. *N*-(Dimethylisopropoxisilylmethyl)-*N*-[(*S*)-1-phenylethyl] acetamide (**III**)

The mixture of isopropanol (4.8 g, 0.08 M) and triethylamine (8.08 g, 0.08 M) was added dropwise to the solution of chloride **II** (10.8 g, 0.04 M) in 40 ml absolute benzene. The reaction mixture was refluxed for 1 h. Next day the precipitated triethylamine hydrochloride was filtered. After the solvent evaporation and subsequent fractionation of the residue, 7.2 g (62%) of isopropoxide **III** was obtained, b.p. 150–153 °C (3 Torr), n_D^{20} 1.4990, $[\alpha]_D^{20} - 43.8^\circ$, (*c* 2.1, CH₂Cl₂). IR (ν , cm⁻¹, thin film): (NCO). ¹H NMR (δ , ppm, CDCl₃): 0.09 s, 0.20 s (6H, SiMe₂), 1.61 (d, 3H, *C–CH₃, ³J_{HH} 6.9 Hz), 2.23 (s, 3H, CH₃CO), 2.32 (s, 2H, NCH₂), 5.09 (q, 1H, *C–CH, ³J_{HH} 6.9 Hz), 0.99 d, 1.03 d (3H, (CH₃)₂C, ³J_{HH} 6.0 Hz), 3.74 (septet, 1H, CH(CH₃)₂, ³J_{HH} 6.0 Hz), 6.89–7.20 (m, 5H, C₆H₅). Anal. Found: C, 65.40; H, 9.16; Si, 10.09. C₁₆H₂₇NO₂Si. Calc.: C, 65.47; H, 9.27; Si, 9.57%.

3.4. (O-Si)-chelate *N*-(dimethylfluorosilylmethyl)-*N*-[(*S*)-1-phenylethyl] acetamide (**I**)

Isopropoxide **III** (3.6 g, 0.012 M) and boron trifluoride diethyl etherate (0.677 g, 0.0047 M) were placed in a distilling flask and heated for 5–10 min. After removal of the low-boiling fraction (50–80 °C, 0.5 ml) the crystals formed were filtered. The yield of fluoride **I** was 2.2 g (71%). M.p. 96–98 °C (octane), $[\alpha]_D^{20} - 11.0^\circ$, (*c* 1.9, CH₂Cl₂). IR (ν , cm⁻¹, CH₂Cl₂): 1505, 1598 (NCO). ¹H NMR (δ , ppm, CDCl₃): 0.20 s, 0.27 s (6H, SiMe₂), 1.64 (d, 3H, *C–CH₃, ³J_{HH} 6.9 Hz), 2.22 (s, 3H, CH₃CO), 2.02 d, 2.32 d (2H, NCH₂, ²J_{HH} 15.5 Hz), 5.05 q (1H, *C–CH, ³J_{HH} 6.9 Hz), 7.2–7.4 (m, 5H,

C₆H₅). Anal. Found: C, 61.76; H, 8.02; N, 5.18. C₁₃H₂₀FNOSi. Calc.: C, 61.60; H, 7.95; N, 5.52%.

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