New Bis(imino)pyridine Complexes of Iron(II) and Iron(III) and their Catalytic Activity in the Mukaiyama Aldol Reaction

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1. Procedures and Spectroscopic Data of the Catalysis

Products in Tables 3 and 4

**General.** Chemicals were treated as follows: diethyl ether, distilled from Na/benzophenone; CH$_2$Cl$_2$, distilled from CaH$_2$. CHCl$_3$, silica, AgSbF$_6$, and 1-(t-butyl-dimethylsiloxy)-1-methoxyethene (7, all Aldrich) and other materials were used as received. The silyl enol ethers (cyclopent-1-en-1-yloxy)trimethylsilane (5)$^1$ and di-t-butyl(methyl)((1-phenylvinyl)oxy)silane (6)$^2$ were prepared according to the literature. All reactions were carried out under nitrogen employing standard Schlenk techniques, and the workup was carried out in the air.

NMR spectra were obtained at room temperature on a Bruker Avance 300 MHz or a Varian Unity Plus 300 MHz instrument at room temperature and referenced to a residual solvent signal; all assignments are tentative. Exact masses were obtained on a JEOL MStation [JMS-700] Mass Spectrometer. IR spectra were recorded on a Thermo Nicolet 360 FT-IR spectrometer.

**General procedure for the catalysis experiment** (Table 3, entry 1)

The complex [FeCl$_2$8] (0.010 g, 0.018 mmol) was dissolved in CH$_2$Cl$_2$ (0.5 mL) and in another flask AgSbF$_6$ (0.012 g, 0.036 mmol) was dissolved in CH$_2$Cl$_2$ (0.5 mL). The solution of AgSbF$_6$ was added to the solution of the metal complex and stirred at room temperature for 1 h. In a third flask, 4-ethylbenzaldehyde (0.049 g, 0.362 mmol) and the silane 5 (0.085 g, 0.543 mmol) was mixed with CH$_2$Cl$_2$ (1 mL) and stirred at room temperature. The solution of the activated catalyst was then filtered through celite directly into the substrate, and the color of the solution turned to reddish. The reaction mixture was stirred overnight after which the reaction mixture was filtered through silica gel pad (3.5 cm). The silica gel was washed with CH$_2$Cl$_2$ (about 2 mL) and the filtrate was evaporated. The product was obtained as yellow oil (0.093 g, 0.319 mmol) in 88% yield as a mixture of diastereomers (62:38) as determined by $^1$H-NMR.
spectroscopy. The syn / anti ratios were determined based on the \$^3J_{HH}\$ coupling constants for the $H$-$C$-$OSiR_3$ proton ($syn \sim 1$ Hz, $anti \sim 4.5$ Hz).

$^1$H-NMR (300 MHz, CDCl$_3$): \(\delta = 7.23-7.09\) (m, 11H, Ar+Ar’), 5.30 (d, \(1J_{HH} = 1.7\) Hz, 1.6H), 5.17 (d, \(1J_{HH} = 4.5\) Hz, 1H), 2.68-2.59 (m, 6 H, $CH_2CH_3+CHCO$, $CH_2'CH_3$), 2.29-1.99 (m, 17H, $CH_2+CH_2'+CH'CO$), 1.27-1.20 (m, 8H, $CH_3+CH_3'$), 0.08 (s, 9H, OSi$CH_3$), 0.05 (s, 15H, OSi$CH_3$).

$^{13}$C{\(1\)H}-NMR (75.41 MHz, CDCl$_3$): \(\delta = 220.0\) (C=O), 219.2 (C=O), 143.2, 142.9, 141.3, 139.5, 127.7, 127.5, 126.7, 125.6, 73.5 (CHO$Si(CH_3)_3$), 72.6 (C'HOSi(CH$_3)_3$), 57.5 (CHCO), 57.4 (C'HCO), 39.8, 39.5, 28.7, 24.8, 22.4, 21.1, 20.7, 15.7, 15.6, 0.26.

MS-EI: \textit{m/z} 216 ([M–Si(CH$_3$)$_3$]$^+$, 35%), 200 ([M–OSi(CH$_3$)$_3$]$^+$, 60%), 135 ([M–Si(CH$_3$)$_3$–C$_5$H$_7$O, 100%]$^+$, 100%).

Table 3 entry 2

The aldol reaction of 2-methylbenzaldehyde (0.043 g, 0.360 mmol) was carried out with the silane 5 (0.084 g, 0.540 mmol) and the activated catalyst [FeCl$_2$8] (0.010 g, 0.0181 mmol catalyst with 0.013 g, 0.0362 mmol AgSbF$_6$) overnight. The product was obtained as colorless oil (0.060 g, 0.216 mmol, 60%) as a mixture of diastereomers (50:50) as determined by $^1$H-NMR spectroscopy. The syn / anti ratios were determined based on the $^3J_{HH}$ coupling constants for the $H$-$C$-$OSiR_3$ proton ($syn < 1$ Hz, $anti \sim 4.5$ Hz).
$^1$H-NMR (300 MHz, CDCl$_3$): δ = 7.43-7.41 (m, 1 H, Ar), 7.41-7.40 (m, 1 H, Ar'), 7.27-7.07 (m, 6 H, Ar+Ar'), 5.50 (s, 1 H, CHOSi(CH$_3$)$_3$), 5.12 (d, $J_{HH} = 4.4$ Hz, 1 H, CH'$OSi(CH$_3$)$_3$), 2.55-2.49 (m, 1 H, CHCO), 2.31 s, (6 H, CH$_3$+CH$_3$'), 2.26-1.57 (m, 13 H, CH$_2$ + CH$_2$' + CHCO + CHCO'), –0.02 (s, 9 H, 3 SiCH$_3$), –0.003 (s, 9 H, 3 SiCH$_3$').

$^{13}$C{$^1$H}-NMR (75.41 MHz, CDCl$_3$): δ = 219.9 (C=O), 218.2 (C=O'), 142.0, 141.0, 133.9, 133.0, 130.4, 130.2, 127.9, 127.2, 126.9, 126.4, 125.9, 125.8, 71.6 (CHCO), 69.2 (C’HCO), 55.5 (CHOSi(CH$_3$)$_3$), 54.6 (C’HOSi(CH$_3$)$_3$), 39.7, 39.2, 27.4, 22.4, 21.1, 20.8, 19.7, 19.0, 0.2 (SiCH$_3$), 0.09 (SiCH$_3$').

Table 3 entry 3

The aldol reaction of 4-methylbenzaldehyde (0.043 g, 0.360 mmol) was carried out with the silane 5 (0.084 g, 0.540 mmol) with the activated catalyst [FeCl$_2$8] (0.010 g, 0.0181 mmol catalyst with 0.013 g, 0.0362 mmol AgSbF$_6$) overnight. The product was obtained as colorless oil (0.059 g, 0.327 mmol, 91%) as a mixture of diastereomers (62:38) as determined by $^1$H-NMR spectroscopy. The syn / anti ratios were determined based on the $^3$J$_{HH}$ coupling constants for the H-C-O$\text{SiR}_3$ proton (syn < 1 Hz, anti ~4.5 Hz).^3,4

$^1$H-NMR (300 MHz, CDCl$_3$): δ = 7.27-7.07 (m, 7 H, Ar+Ar'), 5.30 (s, 1 H, CHOSiCH$_3$), 5.17 (d, $J_{HH} = 4.4$ Hz, 0.7 H, CH'$OSiCH$_3$), 2.34 (s, 3 H, CH$_3$), 2.32 (s, 2 H, OCH$_3$'), 2.26-1.57 (m, 12 H, CH$_2$+CH$_2$'+CH+CH'), 0.06 (6 H, SiCH$_3$), 0.01 (9 H, SiCH$_3$').
Table 3, Entry 4

The aldol reaction of 4-nitrobenzaldehyde (0.052 g, 0.341 mmol) was carried out with the silane 5 (0.064 g, 0.409 mmol) with the activated catalyst [8Fe(μCl)$_3$FeCl$_3$] (0.012 g, 0.017 mmol catalyst with 0.012 g, 0.034 mmol AgSbF$_6$) for 3.5 h. The product was obtained as colorless oil (0.090 g, 0.293 mmol, 86%) as a mixture of diastereomers (50:50) as determined by $^1$H-NMR spectroscopy. The syn / anti ratios were determined based on the $^3$J$_{HH}$ coupling constants for the H-C-OSiR$_3$ proton (syn: ~ 2 Hz, anti ~4.5 Hz). This was the only product where the yield could not accurately be reproduced and a yield range from 10 to 90 % was observed.

$^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ = 8.20-8.09 (m, 4 H, Ar+Ar'), 7.49-7.44 (m, 4 H, Ar+Ar'), 5.38 (d, $J_{HH} = 2.1$ Hz, 1 H, CHO$_2$(CH$_3$)$_3$), 5.27 (d, $J_{HH} = 4.0$ Hz, 1 H, CH′OSi(CH$_3$)$_3$), 2.65-1.50 (m, 14 H, CH$_2$+CH$_2$′+CH+CH′), 0.07 (s, 9 H, SiCH$_3$), 0.02 (s, 9 H, SiCH$_3$).
Table 3, Entry 5

The aldol reaction of 4-methoxybenzaldehyde (0.023 g, 0.170 mmol) was carried out with the silane 5 (0.040 g, 0.255 mmol) with the activated catalyst [8Fe(μCl)3FeCl3] (0.004 g, 0.009 mmol catalyst with 0.006 g, 0.0289 mmol AgSbF6) for 3.5 h. The product was obtained as colorless oil (0.031 g, 0.105 mmol, 62%) as a mixture diastereomers (71:29) as determined by 1H-NMR spectroscopy. The syn/anti ratios were determined based on the 3JHH coupling constants for the H-C-OSiR3 proton (syn: ~0 Hz, anti ~4.2 Hz).3,4

1H-NMR (300 MHz, CDCl3): δ = 7.28-7.17 (m, 3 H, Ar+Ar’), 6.87-6.84 (m, 3 H, Ar+Ar’), 5.27 (1 s, H, CHOSi(CH3)3), 5.16 (d, JHH = 4.2 Hz, 0.4H, CH’OSi(CH3)3), 3.82-3.80 (4 H, m, OCH3 + OCH3’), 2.53-1.60 (10 H, m, CH2 + CH2’ + CHCO + CH’CO), 0.05 (3H, s, SiCH3), 0.01 (9H, s, SiCH3’).

13C{1H}-NMR (75.41 MHz, CDCl3): δ = 220.0 (s, C=O), 219.4 (s, C=O’), 158.9, 158.7, 136.2, 134.5, 127.9, 126.8, 113.6, 113.4, 73.2, 72.3, 57.5, 57.4, 55.4, 55.3, 39.8, 39.4, 24.7, 22.4, 21.0, 20.7, 0.24 (s, SiCH3), 0.16 (s, SiCH3’).

HRMS-EI: m/z [M]+ calcd for C16H24O3Si: 292.1495; found, 292.1497.

IR (neat): 1737 (C=O) cm⁻¹.

Table 4 Entry 1

The aldol reaction of 4-methylbenzaldehyde (0.020 g, 0.170 mmol) was carried out with tert-butyldimethyl(1-phenylvinylxyloxy)silane (6, 0.060 g, 0.255 mmol) with the activated catalyst
The product was obtained as colorless oil (0.045 g, 0.128 mmol, 75%).

\[ \text{[8Fe(μCl)_3FeCl}_3] \] (0.006 g, 0.0085 mmol catalyst with 0.006 g, 0.017 mmol AgSbF_6) for 3.5 h.

The aldol reaction of 4-methoxybenzaldehyde (0.023 g, 0.170 mmol) was carried out with tert-butyldimethyl(1-phenylvinroyloxy)silane (6, 0.060 g, 0.255 mmol) with the activated catalyst [8Fe(μCl)_3FeCl}_3] (0.006 g, 0.0085 mmol catalyst with 0.006 g, 0.017 mmol AgSbF_6) for 3.5 h.

The product was obtained as colorless oil (0.027 g, 0.0737 mmol, 43%).

\[ \text{δ = 7.99-7.96 (m, 2 H, Ar), 7.56-7.53 (m, 1 H, Ar), 7.48-7.43 (m, 2 H, Ar), 7.36-7.33 (m, 2 H, Ar), 6.89-6.85 (m, 2 H, Ar), 5.31 (dd, } J_{HH} = 8.7 \text{ Hz, } J_{HH} = 3.8 \text{ Hz, 1 H, CH}\text{H'O}CO) \]
4.0 Hz, 1 H, CHOSi′Bu(CH₃)₂), 3.81 (s, 3 H, CH₃), 3.56 (dd, \(^2J_{HH} = 15.1\) Hz, \(^3J_{HH} = 8.7\) Hz, 1 H, CHH’CO), 2.94 (dd, \(^2J_{HH} = 15.1\) Hz, \(^3J_{HH} = 4.0\) Hz, 1 H, CHH’CO), 0.75 (s, 9 H, C(CH₃)₃), –0.09 (s, 3 H, SiCH₃), –0.18 (s, 3 H, SiCH₃’).

\(^{13}\)C\{\(^1\)H\}-NMR (75.41 MHz, CDCl₃): \(\delta = 199.1\) (C=O), 159.1, 137.9, 137.3, 133.2, 128.73, 128.69, 127.3, 113.8, 72.2, 55.5, 50.1, 25.9, 18.3, –4.5 (SiCH₃), –5.0 (SiCH₃’).

IR (neat): 1684 (C=O) cm\(^{-1}\).

Table 4 entry 3

The aldol reaction of 4-ethylbenzaldehyde (0.023 g, 0.170 mmol) was carried out with tert-butyldimethyl(1-phenylvinylxylo)silane (6, 0.060 g, 0.255 mmol) with the activated catalyst [\(8\)Fe(μCl)₃FeCl₃] (0.006 g, 0.0085 mmol catalyst with 0.006 g, 0.017 mmol AgSbF₆) for 3.5 h. The product was obtained as colorless oil (0.045 g, 0.123 mmol, 73%).³

\(^1\)H-NMR (300 MHz, CDCl₃): \(\delta = 8.00\) - 7.94 (m, 2 H, Ar), 7.58-7.55 (m, 1 H, Ar), 7.54-7.43 (m, 2 H, Ar), 7.37-7.34 (m, 2 H, Ar), 7.19-7.17 (m, 2 H, Ar), 5.36 (dd, \(^2J_{HH} = 8.9\) Hz, \(^3J_{HH} = 3.8\) Hz, 1 H, CHOSi′Bu₂CH₃), 3.60 (dd, \(^2J_{HH} = 15.1\) Hz, \(^3J_{HH} = 8.9\) Hz, 1 H, CHH’CO), 2.95 (d, \(^2J_{HH} = 15.1\) Hz, \(^3J_{HH} = 3.8\) Hz, 1 H, CHH’CO), 2.67 (q, \(^2J_{HH} = 7.6\) Hz, 2 H, CH₂CH₃), 1.25 (t, \(^2J_{HH} = 6.6\) Hz, 3 H, CH₂CH₃), 0.77 (s, 9 H, C(CCH₃)₃), –0.07 (s, 3 H, SiCH₃), –0.16 (s, 3 H, SiCH₃).

\(^{13}\)C\{\(^1\)H\}-NMR (75.41 MHz, CDCl₃): \(\delta = 199.0\) (C=O), 143.5, 142.3, 137.9, 133.2, 128.73, 128.67, 127.9, 126.0, 72.4, 50.1, 28.7, 25.9, 18.3, 15.7, –4.5 (s, SiCH₃), –5.1 (s, SiCH₃’).
IR (neat): 1685 (C=O) cm\(^{-1}\).

Table 4 entry 4\(^6\)

The aldol reaction of 4-methylbenzaldehyde (0.043 g, 0.360 mmol) was carried out with tert-butylidimethyl(1-methoxyvinloxy)silane (7, 0.102 g, 0.540 mmol) with the activated catalyst [FeCl\(_2\)\(^8\)] (0.010 g, 0.0085 mmol catalyst with 0.006 g, 0.017 mmol AgSbF\(_6\)) after 3.5 h. The product was obtained as colorless oil (quantitative yield containing 5% impurity).\(^3\)

\(^1\)H-NMR (300 MHz, CDCl\(_3\)): \(\delta = 7.25\) (d, \(2J_{HH} = 8.0\) Hz, 2 H, Ar), 7.14 (d, 6.84, \(2J_{HH} = 8.0\) Hz, 2 H, Ar), 5.14 (dd, \(2J_{HH} = 9.4\) Hz, \(3J_{HH} = 4.0\) Hz, 1 H, CHOSi\(^t\)Bu(CH\(_3\))\(_2\)), 3.69 (s, 3 H, OCH\(_3\)), 2.74 (dd, \(2J_{HH} = 14\) Hz, \(3J_{HH} = 9.4\) Hz, 1 H, CHH\(^t\)), 2.55 (dd, \(2J_{HH} = 15.0\) Hz, \(3J_{HH} = 4.0\) Hz, 1 H, CHH\(^t\)), 2.35 (s, 3 H, COOCH\(_3\)), 0.88 (s, 9 H, C(CH\(_3\))\(_3\)), 0.03 (s, 3 H, SiCH\(_3\)), –0.15 (s, 3 H, SiCH\(_3\)).

\(^13\)C\(^{1}\)H\(^{-}\)-NMR (75.41 MHz, CDCl\(_3\)): \(\delta = 171.8, 141.3, 137.3, 129.2, 125.9, 72.3, 51.7, 46.6, 25.9, 21.4, 18.3, –4.4\) (s, SiCH\(_3\)), –5.1 (s, SiCH\(_3\)).

Table 4 entry 56

The aldol reaction of 4-methoxybenzaldehyde (0.049 g, 0.362 mmol) was carried out with tert-butylidimethyl(1-methoxyvinloxy)silane (7, 0.102 g, 0.543 mmol) with the activated catalyst [FeCl\(_2\)\(^8\)] (0.010 g, 0.0085 mmol catalyst with 0.006 g, 0.017 mmol AgSbF\(_6\)) after 3.5 h. The product was obtained as colorless oil (quantitative yield containing 5% impurity).\(^3\)
$^{1}$H-NMR (300 MHz, CDCl$_3$): $\delta = 7.26$-$7.23$ (d, $^2J_{HH} = 8.6$ Hz, 2 H, Ar), $6.85$-$6.82$ (d, $^2J_{HH} = 8.6$ Hz, 2 H, Ar), $5.09$ (dd, $^2J_{HH} = 9.3$ Hz, $^3J_{HH} = 4.1$ Hz, 1 H, CHOSi$'$Bu$_2$CH$_3$), $3.78$ (s, 3 H, OCH$_3$), $3.66$ (s, 3 H, COOCH$_3$), $2.76$-$2.48$ (m, 2 H, CH$_2$), $0.83$ (s, 9 H, C(CH$_3$)$_3$), $0.00$ (s, 3 H, SiCH$_3$), $-0.19$ (s, 3 H, SiCH$_3$).

$^{13}$C{$^1$H}-NMR (75.41 MHz, CDCl$_3$): $\delta = 171.9$, $159.2$, $136.4$, $127.2$, $113.8$, $72.0$, $55.4$, $51.7$, $45.6$, $25.9$, $18.2$, $-4.4$ (s, SiCH$_3$), $-5.1$ (s, SiCH$_3$').

Table 4 entry 6$^7$

The aldol reaction of 4-ethylbenzaldehyde (0.049 g, 0.362 mmol) was carried out with tert-butyl(1-methoxyvinyl)oxy)dimethylsilane (7, 0.102 g, 0.543 mmol) with the activated catalyst [FeCl$_2$8] (0.010 g, 0.0181 mmol catalyst with 0.012 g, 0.036 mmol AgSbF$_6$) overnight. The product was obtained as colorless oil (quantitative yield containing 5% impurity).$^3$

$^{1}$H-NMR (300 MHz, CDCl$_3$): $\delta = 7.28$-$7.25$ (m, 2 H, Ar), $7.17$-$7.14$ (m, 2 H, Ar), $5.14$ (dd, $^2J_{HH} = 9.5$ Hz, $^3J_{HH} = 3.9$ Hz, 1 H, CHOSi$'$Bu$_2$CH$_3$), $3.69$ (s, 3 H, OCH$_3$), $2.78$-$2.52$ (m, 4 H, CH$_2$CO+CH$_2$CH$_3$), $1.24$ (t, $^2J_{HH} = 6.2$ Hz, 3 H, CH$_2$CH$_3$), $0.87$ (s, 9 H, C(CH$_3$)$_3$), $0.03$ (s, 3 H, SiCH$_3$), $-0.02$ (s, 3 H, SiCH$_3$).

$^{13}$C{$^1$H}-NMR (75.41 MHz, CDCl$_3$): $\delta = 171.9$, $143.6$, $141.5$, $130.5$, $128.1$, $127.9$, $125.9$, $72.3$, $51.7$, $46.6$, $28.7$, $25.9$, $18.3$, $15.7$, $-4.4$ (s, SiCH$_3$), $-5.1$ (s, SiCH$_3$').
The aldol reaction of 2-methylbenzaldehyde (0.043 g, 0.360 mmol) was carried out with tert-butyldimethyl(1-methoxyvinylxylo)silane (7, 0.102 g, 0.540 mmol) with the activated catalyst [FeCl2\[8\]] (0.010 g, 0.0181 mmol catalyst with 0.012 g, 0.036 mmol AgSbF6) overnight. The product was obtained as colorless oil (quantitative yield).3

\[\text{\(^1\)H-NMR (300 MHz, CDCl}_3\): } \delta = 7.48-7.47 \text{ (m, 1 H, Ar), 7.21-7.10} \text{ (m, 3 H, Ar), 5.39} \text{ (dd,} \quad \text{2J}\_\text{HH} = 9.8 \text{ Hz, } \text{3J}\_\text{HH} = 3.1 \text{ Hz, 1 H, CHOSi'Bu(CH}_3\text{)}_2\text{), 3.72} \text{ (s, 3 H, OCH}_3\text{), 2.72-2.49} \text{ (m, 2 H,} \quad \text{CH}_2\text{), 2.37} \text{ (s, 3 H, ArCH}_3\text{), 0.87} \text{ (s, 9 H, C(CH}_3\text{)}_3\text{), 0.025} \text{ s, (s, 3 H, SiCH}_3\text{), } \text{–0.2} \text{ (s, 3 H,} \quad \text{SiCH}_3\text{).}

\[\text{\(^{13}\)C\{\(^1\)H\} -NMR (75.41 MHz, CDCl}_3\): } \delta = 171.9, 142.3, 133.5, 130.4, 127.4, 126.39, 126.35, 69.3, 51.8, 45.1, 26.0, 19.2, 18.3, -4.6 \text{ (s, SiCH}_3\text{), } -5.2 \text{ (s, SiCH}_3’\text{).}

MS-EI: \text{m/z} 309 ([M]^+, 1\%), 293 ([M–Me]^+, 15\%), 251 ([M–C}_4\text{H}_9]^+, 100\%).

HRMS-EI: \text{m/z} [M–((CH}_3\text{)}_3]^+ \text{ calcd for C}_{13}\text{H}_{19}\text{O}_3\text{Si: 251.1103; found, 251.1069. m/z} [M–CH}_3–((CH}_3\text{)}_3]^+ \text{ calcd for C}_{12}\text{H}_{16}\text{O}_3\text{Si: 235.0790; found, 235.1485.}
2. UV-vis spectra of the complexes $[\text{FeCl}_2\text{8}]$ (top) and $[\text{8Fe(μCl)}_3\text{FeCl}_3]$ before and after chloride abstraction.
3. References


3 Some of the isolated products contained small (up to 5 mol%) silyl impurities, as assessed by \(^1\)H NMR. All \(^{13}\)C\{\(^1\)H\}-NMR spectra showed baseline purity.


4. $^1$H and $^{13}$C$^1$H NMR spectra of the catalysis products in Tables 3 and 4

Table 3, entry 1.
Table 3, entry 2
Table 3, entry 3
Table 3, entry 4
Table 3, entry 5.
Table 4, entry 1
### Table 4, entry 2

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### Structure

![TBDMSO and Ph](image)

**TBDMSO**

**O**

**O**

**Ph**
Table 4, entry 3
Table 4, entry 4
Table 4, entry 5
Table 4, entry 6
Table 4, entry 7

![NMR spectrum image]
5. Attempted reaction of benzaldehyde and trimethylsiloxycyclopentene (5), $^1$H NMR $^{13}$C{$^1$H}
6. Attempted reaction of furfural and trimethylsiloxy cyclopentene (5), $^1$H NMR
7. Attempted reaction of heptanal and trimethylsiloxy cyclopentene (5), $^1$H NMR
8. Decomposition products of trimethylsiloxy-cyclopentene (5), $^1$H and $^{13}$C{${}^1$H} 

![Chemical structure](image)

The sample could not be dried by high vacuum due to the volatility of the substrate and consequently contains some CH$_2$Cl$_2$ solvent.
8. Decomposition products of trimethylsiloxy cyclopentene (5), GC-MS
8. Decomposition products of 6, GC-MS

\[
\begin{align*}
&\text{Ph} \quad \text{OSiMe}_2\text{Bu} \\
&\quad \quad \quad \quad [8\text{Fe}(\mu\text{Cl})_2\text{FeCl}_3] \\
&\quad \quad \quad \quad + 2 \text{AgSbF}_6
\end{align*}
\]
8. Decomposition products of 7, GC-MS

\[ \text{OMe} \quad \text{OSiMe}_2^{Bu} \quad [8\text{Fe(μCl)}_3\text{FeCl}_3] \quad + 2 \text{AgSbF}_6 \]
9. ESI MS of [8ClFe(μO)FeCl₃]; no molecular ion was observed
9. ESI-MS of $[8\text{Fe}(\mu\text{Cl})_3\text{FeCl}_3]$ after chloride abstraction; no molecular ion was observed