

Bu^tMe₂SiOTf-promoted cyanosilylation of six-membered cyclic nitronates with trialkylsilyl cyanides or *tert*-butyl isocyanide

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All reactions were performed in oven-dried (150 °C) glassware under argon. The following solvents and reagents were distilled from indicated drying agents: CH₂Cl₂, Et₃N (CaH₂); 2,6-lutidine (KOH). NMR spectra were recorded on a Bruker AM-300 or AC-200 instruments and referenced to residual solvent peak. Chemical shifts are reported in ppm (δ); multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet) and br (broad). Coupling constants, *J*, are reported in Hertz. The ratios of stereoisomers were derived from the relative integral intensities of the characteristic signals in the ¹H NMR spectra. Key NOESY correlations are shown on corresponding figures with arrows. IR-spectra were recorded on a Bruker VEKTOR 22 in the range 400–4000 cm⁻¹. Elemental analyses were performed by the Analytical Laboratory of N.D. Zelinsky Institute of Organic Chemistry. Melting points were determined on Kofler melting point apparatus and are uncorrected.

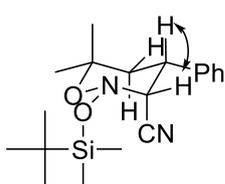
Analytical thin-layer chromatography was performed on Merck silica gel plates with QF-254 indicator. Visualization of TLC plates was accomplished with UV light and/or anisaldehyde/H₂SO₄. All solvents for chromatography and extractions were technical grade and distilled from the indicated drying agents: pentane, hexane, ethyl acetate (K₂CO₃). Column chromatography was performed using 230–400-mesh Merck silica gel.

Commercial reagents: TBSCN (Aldrich), TMSCN (Aldrich). The following reagents were prepared according to literature procedures: TBSOTf,^[1] Et₃SiOTf,^[1] *t*BuNC.^[2] Nitronates **1** syntheses were previously reported.^[3-4]

Spectral data and physical characteristics

rel*-(2*S*,3*R*,4*R*)-2-(*tert*-butyldimethylsilyloxy)-3-cyano-6,6-dimethyl-4-phenyl-[1,2]oxazinane **6a*

Yield 88%, mp = 112-114 °C (hexane); R_f = 0.72 (hexane/EtOAc, 1/1). For X-Ray data, see CCDC-973921.



^1H NMR (300.13 MHz, 300K, CDCl_3): δ = 0.23 and 0.25 [2s, 6H, $\text{Si}(\text{CH}_3)_2$], 0.96 [s, 9H, $\text{C}(\text{CH}_3)_3$], 1.41 and 1.47 [2s, $\text{C}(\text{CH}_3)_2$], 1.68 [dd, 1H, $^2J = 13.6$, $^3J = 3.7$, 1H, $\text{CH}_{\text{A-eq}}\text{H}_{\text{B}}$], 2.15 [t, $^2J \approx ^3J = 13.6$, 1H, $\text{CH}_{\text{A}}\text{H}_{\text{B-ax}}$], 3.54 [dt, 1H, $^3J = 13.6$, $^3J \approx ^3J = 3.7$, 1H, CHPh], 4.12 [d, $^3J = 4.0$, 1H, CHN], 7.30-7.42 [m, 5H, Ph].

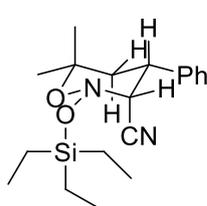
^{13}C NMR (75.47 MHz, 300K, CDCl_3): δ = -5.0 and -4.8 ($\text{Si}(\text{CH}_3)_2$), 18.0 ($\text{C}(\text{CH}_3)_3$), 23.5 ($\text{C}(\text{CH}_3)_3$), 26.1 and 29.4 ($\text{C}(\text{CH}_3)_2$), 36.9 (CHPh), 40.4 (CH_2), 67.1 (CHN), 76.7 ($\text{C}(\text{CH}_3)_2$), 114.6 ($\text{C}\equiv\text{N}$), 127.9 ($\text{CH}_{\text{p-Ph}}$), 128.2 and 129.1 (CH_{Ph}), 137.7 ($\text{CH}_{\text{i-Ph}}$).

^{29}Si NMR (59.63 MHz, 298K, CDCl_3): δ = 29.29.

$\text{C}_{19}\text{H}_{30}\text{N}_2\text{O}_2\text{Si}$ (346.54): calcd. C, 65.85; H, 8.73; N, 8.08; Si, 8.10; found C, 65.91; H, 8.96; N, 8.13; Si, 8.09.

rel-(2S,3R,4R)-3-cyano-2-triethylsilyloxy-6,6-dimethyl-4-phenyl-[1,2]oxazinane 6a[#]

Yield 55%, mp = 55-51°C (hexane); R_f = 0.77 (hexane/EtOAc, 1/1, labile on SiO_2).



^1H NMR (300.13 MHz, CDCl_3): δ = 0.74 [m, $^3J = 7.7$ Hz, 6H, $3\text{CH}_2\text{Et}$], 1.03 [t, $^3J = 7.7$ Hz, 9H, $3\text{CH}_3\text{Et}$], 1.41 and 1.47 [2s, 6H, $\text{C}(\text{CH}_3)_2$], 1.68 [dd, $^2J = 13.6$ Hz, $^3J = 3.7$, 1H, $\text{CH}_{\text{A-eq}}\text{H}_{\text{B}}$], 2.15 [t, $^2J \approx ^3J = 13.6$, 1H, $\text{CH}_{\text{A}}\text{H}_{\text{B-ax}}$], 3.54 [dt, 1H, $^3J = 13.3$, $^3J \approx ^3J = 3.7$, 1H, CHPh], 4.09 [d, $^3J = 4.0$, 1H, CHN], 7.28-7.41 [m, 5H, Ph].

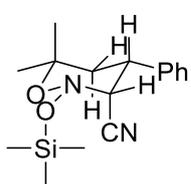
^{13}C NMR (75.47 MHz, CDCl_3): δ = 4.2 (3CH_2), 6.6 (3CH_3), 23.3 and 29.2 ($\text{C}(\text{CH}_3)_2$), 36.8 (CHPh), 40.4 (CH_2), 67.1 (CHN), 77.4 ($\text{C}(\text{CH}_3)_2$), 114.5 ($\text{C}\equiv\text{N}$), 127.8 and 128.9 ($\text{CH}_{\text{o-Ph}}$ and $\text{CH}_{\text{m-Ph}}$), 128.1 ($\text{CH}_{\text{p-Ph}}$), 137.6 ($\text{C}_{\text{i-Ph}}$).

^{29}Si NMR (59.63 MHz, 298K, CDCl_3): δ = 29.00.

$\text{C}_{19}\text{H}_{30}\text{N}_2\text{O}_2\text{Si}$ (346.54): calcd. C, 65.85; H, 8.73; N, 8.08; found C, 65.81; H, 8.90; N, 8.19.

rel-(2S,3R,4R)-3-cyano-2-trimethylsilyloxy-6,6-dimethyl-4-phenyl-[1,2]oxazinane 6a[#]

R_f = 0.68 (hexane/EtOAc, 1/1, unstable on SiO_2).

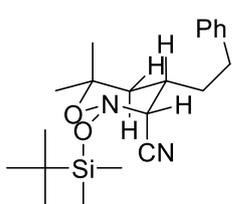


^1H NMR (300.13 MHz, CDCl_3): δ = 0.26 [s, 9H, $\text{Si}(\text{CH}_3)_3$], 1.41 and 1.48 [2s, 6H, $\text{C}(\text{CH}_3)_2$], 1.69 [dd, $^2J = 13.6$, $^3J = 3.7$, 1H, $\text{CH}_{\text{A-eq}}\text{H}_{\text{B}}$], 2.15 [t, $^2J \approx ^3J = 13.6$, 1H, $\text{CH}_{\text{A}}\text{H}_{\text{B-ax}}$], 3.55 [dt, 1H, $^3J = 13.6$, $^3J \approx ^3J = 3.7$, 1H, CHPh], 4.08 [d, $^3J = 4.0$, 1H, CHN], 7.29-7.43 [m, 5H, Ph].

^{13}C NMR (75.47 MHz, CDCl_3): δ = -0.7 ($\text{Si}(\text{CH}_3)_3$), 23.3 and 25.8 ($\text{C}(\text{CH}_3)_2$), 36.8 (CHPh), 42.5 (CH_2), 66.7 (CHN), 77.6 ($\text{C}(\text{CH}_3)_2$), 114.4 ($\text{C}\equiv\text{N}$), 127.6 and 129.1 ($\text{CH}_{\text{o-Ph}}$ and $\text{CH}_{\text{m-Ph}}$), 128.1 ($\text{CH}_{\text{p-Ph}}$), 137.6 ($\text{C}_{\text{i-Ph}}$).

rel-(2S,3R,4S)-2-(tert-butyltrimethylsilyloxy)-3-cyano-6,6-dimethyl-4-phenethyl-[1,2]oxazinane 6b

Yield 91%, mp = 34 °C (hexane), R_f = 0.82 (hexane/EtOAc, 1/1, unstable on SiO_2).



$^1\text{H NMR}$ (300.13 MHz, CDCl_3): δ = 0.19 [br s, 6H, $\text{Si}(\text{CH}_3)_2$], 0.95 [s, 9H, $\text{C}(\text{CH}_3)_3$], 1.30 and 1.33 [2s, $\text{C}(\text{CH}_3)_2$], 1.45 [t, $^2J \approx ^3J = 13.2$, 1H, $\text{CH}_A\text{H}_{B\text{-ax}}$], 1.54 [dd, 1H, $^2J = 13.2$, $^3J = 4.4$, 1H, $\text{CH}_{A\text{-eq}}\text{H}_B$], 1.76-1.88 [m, 2H, CH_2], 2.22-2.35 [m, 1H, CH], 2.70 [dd, $^3J = 8.1$, $^3J = 7.3$, CH_2Ph], 3.97 [d, $^3J = 3.7$, 1H, CHN], 7.17-7.36 [m, 5H, Ph].

$^{13}\text{C NMR}$ (75.47 MHz, CDCl_3): δ = -5.1 and -5.0 ($\text{Si}(\text{CH}_3)_2$), 17.9 ($\underline{\text{C}}(\text{CH}_3)_3$), 23.6 and 29.1 ($\text{C}(\underline{\text{C}}\text{H}_3)_2$), 26.0 ($\text{C}(\underline{\text{C}}\text{H}_3)_3$), 32.5 (CH_2), 34.0 (CH_2), 34.1 (CH), 38.3 (CH_2Ph), 65.1 (CHN), 77.5 (C), 114.6 ($\text{C}\equiv\text{N}$), 126.3, 128.3 and 128.6 (CH_{Ph}), 140.8 ($\text{C}_{i\text{-Ph}}$).

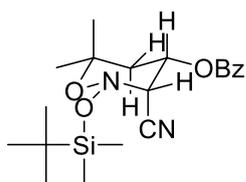
$^{29}\text{Si NMR}$ (59.63 MHz, 299K, CDCl_3): δ = 28.98.

$\text{C}_{21}\text{H}_{34}\text{N}_2\text{O}_2\text{Si}$ (374.59): calcd. C, 67.33; H, 9.15; N, 7.48; found C, 67.23; H, 9.35; N, 7.18;

4-benzoyloxy-2-(tert-butyldimethylsilyloxy)-3-cyano-6,6-dimethyl-[1,2]oxazinane 6c

Yield 86%, colorless solid, R_f 0.71 (hexane/EtOAc, 1/1), *dr* 1.0 : 4.8 : 0.2 : 2.1 (*cis*-6c/*trans*-6c/*cis**-6c/*trans**-6c)

rel-(2*S*,3*R*,4*R*)-isomer *cis*-6c (minor)



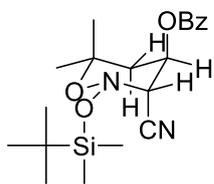
$^1\text{H NMR}$ (300.13 MHz, 300K, CDCl_3): δ = 0.21 [s, 6H, $\text{Si}(\text{CH}_3)_2$], 0.96 [s, 9H, $\text{C}(\text{CH}_3)_3$], 1.38 and 1.44 [2s, $\text{C}(\text{CH}_3)_2$], 1.86 [t, 1H, $^2J \approx ^3J = 12.0$, 1H, $\text{CH}_A\text{H}_{B\text{-ax}}$], 2.03 [dd, $^2J = 12.9$, $^3J = 5.6$, 1H, $\text{CH}_{A\text{-eq}}\text{H}_B$], 4.49 [d, $^3J = 4.9$, 1H, CHN], 5.51 [dt, $^3J = 11.3$, $^3J \approx 4.0$, 1H, CHOBz], 7.47 [t, $^3J \approx 7.6$, 2H, $\text{CH}_{m\text{-Ph}}$], 7.61 [td, $^3J \approx 7.6$, $^4J \approx 1.1$, 1H, $\text{CH}_{p\text{-Ph}}$], 8.08 [dd, $^3J = 7.9$, $^4J =$

1.1, 2H, $\text{CH}_{o\text{-Ph}}$].

$^{13}\text{C NMR}$ (75.47 MHz, 300K, CDCl_3): δ = -5.2 and -5.0 ($\text{Si}(\text{CH}_3)_2$), 17.9 ($\underline{\text{C}}(\text{CH}_3)_3$), 25.9 ($\text{C}(\underline{\text{C}}\text{H}_3)_3$), 24.1 and 29.1 ($\text{C}(\underline{\text{C}}\text{H}_3)_2$), 37.4 (CH_2), 63.7 and 66.3 (CHN and CHOBz), 76.3 ($\underline{\text{C}}(\text{CH}_3)_2$), 113.5 ($\text{C}\equiv\text{N}$), 128.6 and 129.7 (CH_{Ph}), 129.6 ($\text{C}_{i\text{-Ph}}$), 133.8 ($\text{CH}_{p\text{-Ph}}$), 165.3 (C=O).

rel-(2*S*,3*R*,4*S*)-isomer *trans*-6c (major). For X-Ray data see CCDC-973922.

mp = 86-88 °C (hexane).



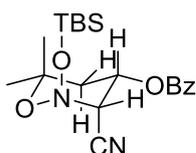
$^1\text{H NMR}$ (300.13 MHz, 300K, CDCl_3): δ = 0.18 and 0.21 [2s, 6H, $\text{Si}(\text{CH}_3)_2$], 0.90 [s, 9H, $\text{C}(\text{CH}_3)_3$], 1.36 and 1.62 [2s, $\text{C}(\text{CH}_3)_2$], 1.86 [dd, 1H, $^2J = 15.2$, $^3J = 1.4$, 1H, $\text{CH}_{A\text{-eq}}\text{H}_B$], 2.09 [dd, $^2J = 15.2$, $^3J = 4.4$, 1H, $\text{CH}_A\text{H}_{B\text{-ax}}$], 4.27 [d, $^3J = 2.8$, 1H, CHN], 5.55 [dt, $^3J = 4.4$, $^3J \approx ^3J = 2.3$, 1H, CHOBz], 7.46 [t, $^3J \approx 7.3$, 2H, $\text{CH}_{m\text{-Ph}}$], 7.62 [t, $^3J \approx 7.0$, 1H, $\text{CH}_{p\text{-Ph}}$], 8.03 [d, $^3J = 7.9$, 2H, $\text{CH}_{o\text{-Ph}}$].

Ph].

$^{13}\text{C NMR}$ (75.47 MHz, 300K, CDCl_3): δ = -5.3 and -5.0 ($\text{Si}(\text{CH}_3)_2$), 17.9 ($\underline{\text{C}}(\text{CH}_3)_3$), 25.8 ($\text{C}(\underline{\text{C}}\text{H}_3)_3$), 25.6 and 29.8 ($\text{C}(\underline{\text{C}}\text{H}_3)_2$), 35.7 (CH_2), 62.3 and 69.8 (CHN and CHOBz), 78.2 ($\underline{\text{C}}(\text{CH}_3)_2$), 113.8 ($\text{C}\equiv\text{N}$), 128.6 and 129.7 (CH_{Ph}), 129.1 ($\text{C}_{i\text{-Ph}}$), 133.7 ($\text{CH}_{p\text{-Ph}}$), 165.3 (C=O).

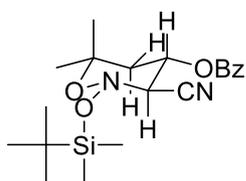
$^{29}\text{Si NMR}$ (59.63 MHz, 298K, CDCl_3): δ = 30.30.

rel-(2*R*,3*S*,4*R*)-isomer *cis**-6c (minor 3)



^1H NMR (300.13 MHz, 300K, CDCl_3): δ (selected signals) = 4.06 [d, $^3J = 2.9$, 1H, CHN].

rel-(2*S*,3*S*,4*R*)-isomer *trans**-**6c** (minor 2)



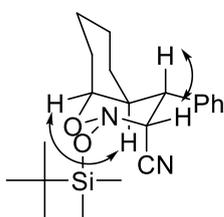
^1H NMR (300.13 MHz, 300K, CDCl_3): $\delta = 0.24$ and 0.28 [2s, 6H, $\text{Si}(\text{CH}_3)_2$], 0.98 [s, 9H, $\text{C}(\text{CH}_3)_3$], 1.30 and 1.47 [2s, $\text{C}(\text{CH}_3)_2$], 1.94 [t, 1H, $^2J \approx 11.9$, 1H, $\text{CH}_A\text{H}_{B\text{-ax}}$], 2.27 [dd, $^2J = 12.7$, $^3J = 5.2$, 1H, $\text{CH}_{A\text{-eq}}\text{H}_B$], 3.84 [d, $^3J = 10.6$, 1H, CHN], 5.53-5.56 [m, 1H, CHOBz], 7.43-7.52 [m, 2H, $\text{CH}_{m\text{-Ph}}$], 7.56-7.66 [m, 1H, $\text{CH}_{p\text{-Ph}}$], 8.01-8.11 [d, $^3J = 7.9$, 2H, $\text{CH}_{m\text{-Ph}}$].

^{13}C NMR (75.47 MHz, 300K, CDCl_3): $\delta = -4.8$ and -4.6 ($\text{Si}(\text{CH}_3)_2$), 17.9 ($\underline{\text{C}}(\text{CH}_3)_3$), 25.8 ($\text{C}(\underline{\text{C}}\text{H}_3)_3$), 23.9 and 29.0 ($\text{C}(\underline{\text{C}}\text{H}_3)_2$), 40.5 (CH_2), 64.9 and 68.5 (CHN and CHOBz), 76.4 ($\underline{\text{C}}(\text{CH}_3)_2$), 115.2 ($\text{C}\equiv\text{N}$), 128.5 and 129.8 (CH_{Ph}), 128.8 ($\text{C}_{i\text{-Ph}}$), 133.6 ($\text{CH}_{p\text{-Ph}}$), 165.3 ($\text{C}=\text{O}$).

$\text{C}_{20}\text{H}_{30}\text{N}_2\text{O}_4\text{Si}$ (390.55, for mixture of isomers): calcd. C, 61.51; H, 7.74; N, 7.17; found C, 61.24; H, 7.35; N, 7.20;

rel-(2*S*,3*S*,4*R*,4*aS*,8*aS*)-2-(*tert*-butyldimethylsilyl)-3-cyano-4-phenyl-octahydro-2*H*-benzo[*e*][1,2]oxazinane **6d**

Yield 84%, mp = 115-117 °C (hexane), R_f 0.76 (hexane/EtOAc, 1/1).



^1H NMR (300.13 MHz, 297K, CDCl_3): $\delta = 0.18$ and 0.19 [2s, 6H, $\text{Si}(\text{CH}_3)_2$], 0.91 [s, 9H, $\text{C}(\text{CH}_3)_3$], 1.02-1.60 [m, 5H, CH_2Cy], 1.76-1.95 [m, 2H, CH_2Cy], 2.25 [qd, $^2J \approx ^3J \approx ^3J = 12.3$, $^3J = 3.7$, 1H, CH_AH_B], 2.72 [dt, $^3J = 11.1$, $^3J \approx ^3J = 5.0$, 1H, CH_{Cy}], 3.56 [dd, $^3J = 12.4$, $^3J = 3.7$, 1H, $\text{CH}_{\text{ax}}\text{Ph}$], 4.04 [d, $^3J = 3.7$, 1H, CHCN], 4.21 [dt, $^3J = 12.1$, $^3J \approx ^3J = 4.8$, 1H, OCH], 7.26-7.41 [m, 5H, Ph].

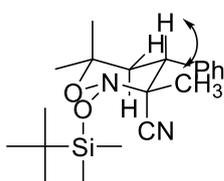
^{13}C NMR (75.47 MHz, 298K, CDCl_3): $\delta = -5.1$ and -4.9 ($\text{Si}(\text{CH}_3)_2$), 17.9 ($\text{C}(\text{CH}_3)_3$), 19.9, 25.1, 25.5 and 26.1 (CH_2Cy), 26.0 ($\text{C}(\text{CH}_3)_3$), 34.1 (CH_{Ph}), 42.2 (CH_{Cy}), 67.9 (CHN), 79.1 (OCH), 115.2 ($\text{C}\equiv\text{N}$), 128.2 ($\text{CH}_{p\text{-Ph}}$), 128.8 and 129.2 ($\text{CH}_{o\text{-Ph}}$ and $\text{CH}_{p\text{-Ph}}$), 136.5 ($\text{C}_{i\text{-Ph}}$).

^{29}Si NMR (59.63 MHz, 299K, CDCl_3): $\delta = 29.48$.

$\text{C}_{21}\text{H}_{32}\text{N}_2\text{O}_2\text{Si}$ (372.58): calcd. C, 67.70; H, 8.66; N, 7.52; Si, 7.54; found C, 67.81; H, 8.86; N, 7.48; Si, 7.44.

rel-(2*S*,3*R*,4*R*)-2-(*tert*-butyldimethylsilyloxy)-3-cyano-3,6,6-trimethyl-4-phenyl-[1,2]oxazinane **6e**

Yield 60% (2 days), mp = 88-90 °C (hexane); $R_f = 0.72$ (hexane/EtOAc, 1/1).



^1H NMR (300.13 MHz, 300K, CDCl_3): $\delta = 0.20$ and 0.25 [2s, 6H, $\text{Si}(\text{CH}_3)_2$], 0.96 [s, 9H, $\text{C}(\text{CH}_3)_3$], 1.29 and 1.36 [2s, $\text{C}(\text{CH}_3)_2$], 1.48 [s, 3H, CH_3], 1.62

[dd, 1H, $^2J = 13.6$, $^3J = 3.9$, 1H, $CH_{A-eq}H_B$], 2.22 [t, $^2J \approx ^3J = 13.6$, 1H, CH_AH_{B-ax}], 3.10 [dd, 1H, $^3J = 13.6$, $^3J = 3.9$, 1H, CHPh], 7.26-7.47 [m, 5H, Ph].

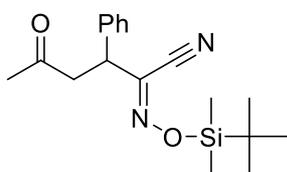
^{13}C NMR (75.47 MHz, 300K, $CDCl_3$): $\delta = -4.6$ and -4.3 ($Si(CH_3)_2$), 17.9 ($C(CH_3)_3$), 21.6 (CH_3), 25.9 ($C(CH_3)_3$), 23.3 and 29.3 ($C(CH_3)_2$), 40.4 (CH_2), 47.5 (CHPh), 70.8 (C-N), 76.8 ($C(CH_3)_2$), 117.9 ($C\equiv N$), 128.0 (CH_{p-Ph}), 128.7 and 128.9 (CH_{Ph}), 137.7 (CH_{i-Ph}).

^{29}Si NMR (59.63 MHz, 298K, $CDCl_3$): $\delta = 28.80$.

$C_{20}H_{33}N_2O_2Si$ (360.57): calcd. C, 66.62; H, 8.95; N, 7.77; found C, 66.92, H, 8.82, N, 7.48.

(Z)-N-(tert-butyldimethylsilyloxy)-4-oxo-2-phenylpentanimidoyl cyanide 8a

mp = 26 °C (hexane), R_f 0.75 (hexane/EtOAc, 1/1).



1H NMR (300.13 MHz, 297K, $CDCl_3$): $\delta = 0.23$ and 0.25 [2s, 6H, $Si(CH_3)_2$], 0.97 [s, 9H, $C(CH_3)_3$], 2.16 [s, 3H, CH_3], 2.83 [dd, $^2J = 17.6$, $^3J = 5.1$, 1H, CH_AH_B], 3.34 [dd, $^2J = 17.6$, $^3J = 9.2$, 1H, CH_AH_B], 4.34 [dd, $^3J = 9.2$, $^3J = 5.1$, 1H, CHPh], 7.23 [d, $^3J = 6.6$, 1H, CH_{o-Ph}], 7.31-7.41 [m, 3H, CH_{Ph}].

^{13}C NMR (75.47 MHz, 297K, $CDCl_3$): $\delta = -5.4$ and -5.3 ($Si(CH_3)_2$), 18.1 ($C(CH_3)_3$), 25.7 ($C(CH_3)_3$), 30.1 (CH_3), 43.5 and 46.2 (CHPh and CH_2), 110.1 ($C\equiv N$), 127.9 and 129.2 (CH_{Ph}), 128.1 (CH_{p-Ph}), 137.6 and 136.6 (CH_{i-Ph} and C=N), 204.5 (C=O).

^{29}Si NMR (59.63 MHz, 298K, $CDCl_3$): $\delta = 32.92$.

IR (neat): $\nu = 2231$ (w, $\nu_{C\equiv N}$), 1668 (w, $\nu_{C=O}$), 1603 (w, $\nu_{C=N}$).

$C_{18}H_{28}N_2O_2Si$ (330.50): calcd. C, 65.41; H, 7.93; N, 8.41; Si, 8.43; found: C, 65.55; H, 8.03; N, 8.45; Si, 8.42.

References

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