Enantioselective cyanosilylation of Ketones by a Catalytic Double Activation Method Employing Chiral Lewis Acid and Achiral *N*-oxide Catalysts

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General. ¹H NMR spectra were recorded on a Varian Unity INOVA 400 (400 MHz) or on Bruker (300 MHz). Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (CDCl₃, δ = 7.26). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz), integration, and affiliation. ¹³C NMR data were collected on a Varian Unity INOVA 400 (100 MHz) or on Bruker (75 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard (CDCl₃, δ = 77.0). Elemental analyses were performed on Carlo-1160. Enantiomer ratios were determined by chiral GC analysis on Varian Chirasil DEX CB or by chiral HPLC analysis on Daicel Chiralcel OD/OJ in comparison with the authentic racemates. Optical rotation data were recorded on Perkin-Elmer Polarimeter-341. All ketones, TMSCN and substituted salicylal were purchased from Acros, Aldrich and Fluka, and used directly without further purification. Solvents were purified by usual method.

Preparation of ligands **1a-g** and **2**, and ligand screening

This kind of salen ligands was prepared according to the literature.^{1a} The *N*-oxide **2** was obtained by direct oxidization of *N,N*-dimethylaniline.² Satisfactory spectra data were obtained for these ligands. **1a-g** was evaluated by the addition of TMSCN to acetophenone as the model reaction. As shown in Table 1, both the electro-withdrawing and electro-donating substituents showed worse reactivity and enantioselectivity than 3,5-di-*tert*butyl one.

Table 1 Substituent effect of **1** on the addition of TMSCN to acetophenone.^a

entry	ligand	yield (%) ^b	<i>ee</i> (%) ^c
1	1a	95	67
2	1b	trace	46
3	1c	49	57
4	1d	32	33
5	1e	N.R.	--
6	1f	47	60
7	1g	16	20

^a conditions: **1**-Ti(OiPr)₄ (20 mol%), **2** (20 mol%), concentration of acetophenone 0.12 M in CH₂Cl₂, 0 °C, 84 h; ^b isolated yield; ^c determined by chiral GC analysis on Chiasil DEX CB. N.R. = no reaction.

(**1R,2R**)-*N,N'*-Bis(3,5-di-*tert*-butyl-salicylidene)-1,2-diphenyl-1,2-diamine (**1a**)

¹H NMR (400 MHz, CDCl₃): δ = 1.22 (d, 1.6 Hz, 18H; 2C(CH₃)₃), 1.42 (d, 2.0 Hz, 18H; 2C(CH₃)₃), 4.73 (d, 1.6 Hz, 2H; CH-CH), 6.98 (m, 2H; aromatic H), 7.16-7.20 (m, 10H; aromatic H), 7.31 (d, 2.4 Hz, 2H; aromatic H), 8.40 (d, 1.2 Hz, 2H; 2CH=N), 13.60 (d, 2.0 Hz, 2H; exchangeable with D₂O)ppm; ¹³C NMR (100 MHz, CDCl₃): δ = 29.4, 31.4, 34.0, 35.0, 80.1, 117.8, 126.3, 127.1, 127.4, 128.0, 128.2, 136.3, 139.8, 140.0, 157.9, 167.2; [α]_D²² = -33.3 ° (*c* = 0.6, CHCl₃) [lit. [α]_D²⁰ = +32.4 ° (*c* = 0.25, CHCl₃ for (1*S*,2*S*)-**1a**)].^{1a}

N,N-dimethylaniline *N*-oxide (**2**)²

¹H NMR (400 MHz, CDCl₃): δ = 3.66 (s, 6H; CH₃), 7.39-7.50 (m, 3H; aromatic H), 7.93 (d, 2H; 8 Hz, aromatic H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ = 62.7, 119.7, 129.1, 129.3, 153.6

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Asymmetric addition of TMSCN to ketones

Typical procedure: $\text{Ti}(\text{OiPr})_4$ (32 μL , 1 M in toluene, 0.032 mmol) was stirred with **1a** (20.8 mg, 0.032 mmol) in dry CH_2Cl_2 (1 mL) at 35 °C for 1 h under N_2 atmosphere. After the solvents were removed in vacuo, the resulted yellow powder was redissolved in CH_2Cl_2 (0.5 mL), cooled to – 20 °C, and followed by addition of acetophenone (1.6 mmol). Finally, to the reaction mixture, was added a solution of **2** (2.1 mg, 0.016 mmol) that was separately treated with TMSCN (3.2 mmol, 2 equiv.) in CH_2Cl_2 (1.0 mL) at 35 °C for 1 h. The reaction was performed at – 20 °C. At completion, the reaction mixture was concentrated and put on a silica gel column to give the pure product with ethyl ether/petroleum ether (1/200, V/V) as the eluent. The desired 2-trimethylsilyloxy-2-phenylpropanenitrile was obtained as colorless oil (280 mg, 75 %). The ee was determined by chiral GC analysis on Chirasil DEX CB to be 84 %.

2-trimethylsilyloxy-2-phenylpropanenitrile:

75 % yield, 84 % ee. ^1H NMR (300 MHz, CDCl_3): δ = 0.19 (s, 9H; $(\text{CH}_3)_3\text{Si}$), 1.87 (s, 3H; CH_3), 7.38-7.58 (m, 5H; aromatic H)ppm; $[\alpha]_D^{22}$ = –22.1° (c = 1.04, CHCl_3 , 84 % ee) [lit. $[\alpha]_D^{20}$ = +21.9° (c = 1.18, CHCl_3 , for *R* enantiomer with 93 % ee)];³ GC (CP-Chirasil DEX CB, 0.25 mm * 25 m, column temperature = 100 °C (isothermal), inject temperature = 200 °C, detector temperature = 250 °C, inlet pressure = 8 psi): t_r (major) = 24.5 min, t_r (minor) = 25.4 min. The chromatograms are illustrated below.

2-trimethylsilyloxy -2-(4'-methylphenyl)propanenitrile:

57 % yield, 73 % ee. ^1H NMR (400 MHz, CDCl_3): δ = 0.16 (s, 9H; $(\text{CH}_3)_3\text{Si}$), 1.84 (s, 3H; CH_3), 2.36 (s, 3H; ArCH_3), 7.21 (m, 2H; aromatic H), 7.43 (m, 2H; aromatic H)ppm; Anal. Calcd. for $\text{C}_{13}\text{H}_{19}\text{NOSi}$: C, 66.90; H, 8.21; N, 6.00; Found: C, 66.78; H, 8.03; N, 6.39; $[\alpha]_D^{22}$ = –19.3° (c = 0.57, CHCl_3 , 73 % ee) [lit. $[\alpha]_D^{20}$ = +21.3° (c = 1.28, CHCl_3 , 90 % ee)];³ GC (CP-Chirasil DEX CB, 0.25 mm * 25 m, column temperature = 105 °C (isothermal), inject temperature = 200 °C, detector temperature = 250 °C, inlet pressure = 8 psi): t_r (major) = 34.9 min, t_r (minor) = 36.0 min.

2-trimethylsilyloxy -2-(2'-fluorophenyl)propanenitrile

80 % yield, 76 % ee. ^1H NMR (400 MHz, CDCl_3): δ = 0.26 (s, 9H; $(\text{CH}_3)_3\text{Si}$), 1.94 (s, 3H; CH_3), 7.07-7.12 (m, 1H; aromatic H), 7.16-7.20 (m, 1H; aromatic H), 7.33-7.38 (m, 1H; aromatic H), 7.56-7.60 (m, 1H; aromatic H)ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 1.0, 30.8 (d, J = 3.0 Hz), 68.4, 116.5 (d, $^2J_{\text{CCF}}$ = 21.9 Hz), 120.6, 124.2 (d, $^4J_{\text{CCCCF}}$ = 3.6 Hz), 126.7 (d, J = 3.0 Hz), 130.6 (d, $^3J_{\text{CCCF}}$ = 8.5 Hz), 159.4 (d, $^1J_{\text{CF}}$ = 248.2 Hz); $[\alpha]_D^{22}$ = –12.7° (c = 1.18, CHCl_3 , 76 % ee); GC (CP-Chirasil DEX CB, 0.25 mm * 25 m, column temperature = 105 °C (isothermal), inject temperature = 200 °C, detector temperature = 250 °C, inlet pressure = 8 psi): t_r (major) = 20.8 min, t_r (minor) = 21.7 min. The chromatograms are illustrated below.

2-trimethylsilyloxy-2-(4'-fluorophenyl)propanenitrile

71 % yield, 83 % ee. ^1H NMR (400 MHz, CDCl_3): δ = 0.18 (s, 9H; $(\text{CH}_3)_3\text{Si}$), 1.84 (s, 3H; CH_3), 7.08 (m, 2H; aromatic H), 7.52 (m, 2H; aromatic H)ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 1.0, 33.5, 71.0, 115.6 (d, $^2J_{\text{CCF}}$ = 21.9 Hz), 121.4, 126.5 (d, $^3J_{\text{CCF}}$ = 8.5 Hz), 138.0, 162.2 (d, $^1J_{\text{CF}}$ = 246.4 Hz); $[\alpha]_D^{22}$ = -18.6° (c = 1.4, CHCl_3 , 83 % ee); GC (CP-Chirasil DEX CB, 0.25 mm * 25 m, column temperature = 115 $^\circ\text{C}$ (isothermal), inject temperature = 200 $^\circ\text{C}$, detector temperature = 250 $^\circ\text{C}$, inlet pressure = 8 psi): t_r (major) = 13.1 min, t_r (minor) = 13.9 min. The chromatograms are illustrated below.

2-trimethylsilyloxy-2-(4'-chlorophenyl)propanenitrile

58 % yield, 84 % ee. ^1H NMR (400 MHz, CDCl_3): δ = 0.19 (s, 9H; $(\text{CH}_3)_3\text{Si}$), 1.83 (s, 3H; CH_3), 7.38 (m, 2H; aromatic H), 7.48 (m, 2H; aromatic H)ppm; ^{13}C NMR (100 MHz, CDCl_3): δ = 1.0, 33.5, 71.0, 121.2, 126.1, 128.8, 134.6, 140.7; Anal. Calcd. for $\text{C}_{12}\text{ClH}_{16}\text{NOSi}$: C, 56.79; H, 6.35; N, 5.52; Found: C, 56.82; H, 6.41; N, 5.93; $[\alpha]_D^{22}$ = -19.5° (c = 0.89, CHCl_3 , 84 % ee) [lit. $[\alpha]_D^{20}$ = $+29.5^\circ$ (c = 1.04, CHCl_3 , 92 % ee)];³ GC (CP-Chirasil DEX CB, 0.25 mm * 25 m, column temperature = 125 $^\circ\text{C}$ (isothermal), inject temperature = 200 $^\circ\text{C}$, detector temperature = 250 $^\circ\text{C}$, inlet pressure = 8 psi): t_r (major) = 21.5 min, t_r (minor) = 22.7 min.

2-trimethylsilyloxy-2-(3'-chlorophenyl)propanenitrile:

93 % yield, 80 % ee. ^1H NMR (300 MHz, CDCl_3): δ = 0.22 (s, 9H; $(\text{CH}_3)_3\text{Si}$), 1.86 (s, 3H; CH_3), 7.34-7.55 (m, 4H; aromatic H)ppm; ^{13}C NMR (75 MHz, CDCl_3): δ = 1.0, 33.4, 70.9, 121.0, 122.7, 124.8, 128.8, 129.9, 134.6, 144.0; Anal. Calcd. for $\text{C}_{12}\text{ClH}_{16}\text{NOSi}$: C, 56.79; H, 6.35; N, 5.52; Found: C, 56.61; H, 6.39; N, 5.90; $[\alpha]_D^{22}$ = -19.7° (c = 0.76, CHCl_3 , 80 % ee); GC (CP-Chirasil DEX CB, 0.25 mm * 25 m, column temperature = 120 $^\circ\text{C}$ (isothermal), inject temperature = 200 $^\circ\text{C}$, detector temperature = 250 $^\circ\text{C}$, inlet pressure = 8 psi): t_r (major) = 16.7 min, t_r (minor) = 17.0 min. The chromatograms are illustrated below.

2-trimethylsilyloxy-2-(2'-naphthyl)propanenitrile:

50 % yield, 84 % ee. ^1H NMR (300 MHz, CDCl_3): δ = 0.22 (s, 9H; $(\text{CH}_3)_3\text{Si}$), 1.97 (s, 3H; CH_3), 7.54-7.66 (m, 3H; aromatic H), 7.90-7.93 (m, 3H; aromatic H), 8.07 (d, 1.8 Hz, 1H; aromatic H)ppm; $[\alpha]^{22}_{\text{D}} = -10.6^\circ$ ($c = 0.85$, CHCl_3 , 84 % ee) [lit $[\alpha]^{20}_{\text{D}} = +12.6^\circ$ ($c = 1.99$, CHCl_3 , 94 % ee)];⁴ HPLC (Chiralcel OJ, *i*PrOH/*n*-hexane, 1/99 V/V, 1.0 mL/min, 23 °C, UV 254 nm): t_{r} (minor) = 5.9 min, t_{r} (major) = 7.0 min.

1-trimethylsilyloxy-1,2,3,4-tetrahydronaphthane-1-carbonitrile:

37 % yield, 81 % ee. ^1H NMR (300 MHz, CDCl_3): δ = 0.24 (s, 9H; $(\text{CH}_3)_3\text{Si}$), 2.06 (m, 2H; CH_2), 2.23 (m, 1H; CH_2), 2.35 (m, 1H; CH_2), 2.85 (m, 2H; CH_2), 7.13 (m, 1H; aromatic H), 7.29 (m, 2H; aromatic H), 7.67 (m, 1H; aromatic H)ppm; ^{13}C NMR (75 MHz, CDCl_3): δ = 1.3, 18.6, 28.2, 37.6, 69.8, 122.0, 126.6, 127.9, 129.0, 129.2, 135.6, 136.0; Anal. Calcd. for $\text{C}_{14}\text{H}_{19}\text{NOSi}$: C, 68.52; H, 7.80; N, 5.71; Found: C, 68.30; H, 7.70; N, 6.11; $[\alpha]^{22}_{\text{D}} = -12.9^\circ$ ($c = 0.66$, CHCl_3 , 81 % ee); GC (CP-Chirasil DEX CB, 0.25 mm * 25 m, column temperature = 125 °C (isothermal), inject temperature = 200 °C, detector temperature = 250 °C, inlet pressure = 8 psi): t_{r} (minor) = 45.7 min, t_{r} (major) = 46.9 min. The chromatograms are illustrated below.

2-trimethylsilyloxy -2-methyl-4-phenylbutanenitrile:

85 % yield, 84 % ee. ^1H NMR (300 MHz, CDCl_3): δ = 0.29 (s, 9H; $(\text{CH}_3)_3\text{Si}$), 1.65 (s, 3H; CH_3), 2.02-2.08 (m, 2H; CH_2), 2.80-2.91 (m, 2H; CH_2), 7.22-7.33 (m, 5H; aromatic H)ppm; $[\alpha]^{22}_{\text{D}} = -10.6^\circ$ ($c = 1.08$, CHCl_3 , 84 % ee) [lit $[\alpha]^{20}_{\text{D}} = +9.7^\circ$ ($c = 1.78$, CHCl_3 , 80 % ee)];⁴ HPLC (Chiralcel OD, *i*PrOH/*n*-hexane, 1/99 V/V, 1.0 mL/min, 23 °C, 254 nm): t_{r} (major) = 5.2 min, t_{r} (minor) = 5.9 min.

2-trimethylsilyloxy -2-methyl-4-phenyl-3-butenenitrile:

79 % yield, 64 % ee. ^1H NMR (300 MHz, CDCl_3): δ = 0.27 (s, 9H; $(\text{CH}_3)_3\text{Si}$), 1.77 (s, 3H; CH_3), 6.15 (d, 15.9Hz, 1H; $\text{CH}=\text{C}$), 6.91 (d, 15.9Hz, 1H; $\text{CH}=\text{C}$), 7.33-7.45 (m, 5H; aromatic H)ppm; ^{13}C NMR (75 MHz, CDCl_3): δ = 1.3, 30.8, 69.9, 120.6, 126.8, 128.5, 128.7, 129.5, 130.9, 135.1; $[\alpha]^{22}_{\text{D}} = -48.0^\circ$ ($c = 0.5$, CHCl_3 , 64 % ee) [lit $[\alpha]^{20}_{\text{D}} = +62.3^\circ$ ($c = 1.76$, CHCl_3 , 95 % ee)];⁴ HPLC (Chiralcel OD, *i*PrOH/*n*-hexane, 1/99 V/V, 1.0 mL/min, 23 °C, 254 nm): t_{r} (major) = 5.0 min, t_{r} (minor) = 5.8 min.

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CFX-30 H1 CDCl3 2002-12-27
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CFX-28 H1 CDCl3 2002-12-15
Pulse Sequence: szpu1

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Pulse Sequence: szpu1

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 Pulse Sequence: zgpg1

HCSC, 1H NMR bdi probe

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PROCNO 1

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FILE: 13C NMR U01.P1006

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1-ESC IH NMR

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 D1: 2.00000000 sec

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F2 - Processing parameters
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1-tsc 13 NMR b01 probe

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 AQ 1.7980751 sec
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 d11 0.03000000 sec

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 F2P -11.185 ppm
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