Asymmetric Ring Opening of Meso-Epoxides with TMSCN Catalyzed by (pybox)Lanthanide Complexes

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Supporting Information

General: Optical rotations were measured on a Jasco DIP 370 digital polarimeter with a sodium (λ= 489 nm) lamp, and are reported as follows: $[\alpha]^{T}_{\lambda}$, (*c* g/100 mL, solvent). Infrared spectra were recorded on a Mattson Galaxy Series FTIR 3000 spectrometer. ^{1}H NMR spectra were recorded on Bruker AM-500 (500 MHz) or AM-400 (400 MHz) spectrometers. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (deuterochloroform: δ 7.26 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), and coupling constants (Hz). ^{13}C NMR spectra were recorded on Bruker AM-500 (125 MHz) or AM-400 (100MHz) spectrometers with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (deuterochloroform: δ 77.0 ppm). Mass spectra were obtained on a JEOL AX-5-5 or SX-102 high resolution magnetic sector mass spectrometer by the Harvard Mass Spectrometry Laboratory.

Analytical and preparative thin layer chromatography were performed on EM Reagent 0.25 or 0.50 mm silica gel 60-F plates. Flash chromatography was performed using EM silica gel 60 (230-400 mesh). Solvents for extraction and chromatography were HPLC grade.

When necessary, solvents and reagents were dried prior to use. Tetrahydrofuran (THF) and benzene were distilled from sodium or potassium metal/benzophenone ketyl. Dichloromethane were distilled from calcium hydride. Chloroform was dried over activated 3 Å molecular sieves prior to use. All other commercially obtained reagents were used as received. The pybox ligand 2,6-bis[4'-(*S*)-isopropyloxazolin-2'-yl]pyridine **2a** was purchased from Aldrich and was used as received.

Determination of Enantiomeric Purity. Enantiomeric excesses (ee's) were determined by capillary GC analysis using a Hewlett Packard 5890 Series II Gas Chromatograph with H_2 as a carrier gas. The following GC columns were employed: Cyclodex-B (30m x 0.25mm id x 0.25µm film; J&W Scientific) set at a column head pressure of 13 psi; and Chiraldex γ-TA (20m x 0.25mm id x 0.125µm film; Advanced Separation Technologies, Inc.) set at a column head pressure of 7 psi.

General Experimental for the Asymmetric Ring Opening of Meso-epoxides with TMSCN. (1*S*, 2*R*)-2-Trimethylsilyloxy-cyclohexane-1-carbonitrile (1). A 25 mL

flask equipped with a stir bar was charged with $YbCl_3$[1] (28 mg, 0.10 mmol) and 2,6-bis[4'-(*S*)-phenyloxazolin-2'-yl]pyridine **2c** (45 mg, 0.12 mmol).[2] The flask was sealed with a septum and charged with 6 mL THF. The mixture was allowed to stir for 45 min at rt at which time 2 mL $CHCl_3$ was added to generate a clear solution containing a fine white suspension of uncomplexed $YbCl_3$. The solution was stirred for an additional 15 min at rt and then filtered into a 25 mL flask through an oven-dried pipette fitted with 0.5 cm cotton. The filtrate was concentrated by rotary evaporation and the residue dried at 0.5 Torr for 10 min to yield an opaque white solid. The flask was fitted with a rubber septum and the catalyst thus obtained was dissolved in 1 mL $CHCl_3$,[3] treated with TMSCN (160 μL, 1.20 mmol), and cooled to –45 °C. Cyclohexene oxide (100 μL, 1.00 mmol) was added and the reaction was allowed to stir 4 days. The mixture was diluted with 20 mL CH_2Cl_2 and filtered through a 2 cm SiO_2 plug. The SiO_2 was rinsed with 100 mL CH_2Cl_2 and the filtrates were combined and concentrated *in vacuo* to yield (1*S*, 2*R*)-2-trimethylsilyloxy-cyclohexane-1-carbonitrile (172 mg, 0.87 mmol, 87%). The product was determined to be in 91% ee by chiral GC analysis (Cyclodex-B, 100 °C, isothermal, t_R(major) = 21.94 min, t_R(minor) = 22.81 min). $[\alpha]^{27}_D$ -38.5° (*c* 4.52, CH_2Cl_2); IR (thin film) 2945, 2864, 2243, 1450, 1252, 1135, 1107, 925, 842; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.64-3.70 (m, 1 H), 2.38-2.44 (m, 1 H), 2.08-2.11 (m, 1 H), 1.90-

(1) Both anhydrous $YbCl_3$ and the corresponding hexahydrate afforded catalysts that displayed similar enantioselectivity in the ARO reactions. However, the reactivity of catalyst prepared from anhydrous $YbCl_3$ was significantly greater.

(2) The stoichiometry of the catalyst-ligand complex is apparently 1:1, and the use of a slight excess of ligand (1.2 equiv. relative to $YbCl_3$) afforded best results. Use of larger excess of ligand led to similar enantioselectivities in the ARO but suppressed rates.

(3) Reactions were carried out using $CHCl_3$ that had been pre-dried by storage over oven dried 3Å molecular sieves. It was found that over the period of several days, $CHCl_3$ stored in this manner became contaminated with HCl, and this had a significant deleterious effect on the ARO reaction. As a result, freshly dried chloroform (stored <2 days over sieves) was employed.

2.07 (m, 1 H), 1.55-1.75 (m, 3 H), 1.25-1.33 (m, 3 H), 0.17 (s, 9 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 121.6, 71.1, 37.7, 34.7, 28.2, 23.9, 23.3, 0.18; HRMS calculated for $C_{10}H_{23}N_2OSi$ $(M+NH_4)^+$: 215.1580; found: 215.1578.

Determination of Absolute Configuration. (1*S*, 2*R*)-2-Cyanocyclohexanol. A 25 mL flask equipped with a stir bar was charged with (1*R*, 2*S*)-2-trimethylsilyloxy-cyclohexane-1-carbonitrile **1** (110 mg, 0.56 mmol, 80% ee), 2 mL MeOH, and 50 μL TFA at rt. The reaction was allowed to stir for 1 h and concentrated by rotary evaporator. The crude residue obtained was dissolved in 50 mL CH_2Cl_2 and filtered through a SiO_2 plug. The filtrate was concentrated *in vacuo* to yield (1*S*, 2*R*)-2-cyanocyclohexanol (46 mg, 0.37 mmol, 66%). $[\alpha]^{26}_D$ +34.1° (*c* 0.255, CH_2Cl_2), lit.[4] $[\alpha]^{26}_D$ +52.0° (*c* 2.5, CH_2Cl_2); IR (thin film) 3460, 2950, 2253, 1450, 1125, 765; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.72-3.75 (m, 1 H), 2.38-2.44 (m, 1 H), 2.04-2.18 (m, 3 H), 1.19-1.79 (m, 4 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 123.5, 70.5, 37.5, 33.8, 28.2, 23.9, 23.4; HRMS calculated for $C_7H_{15}N_2O$ $(M+NH_4)^+$: 143.1184; found: 143.1189.

(1*R*, 2*S*)-2-Trimethylsilyloxy-cyclopentane-1-carbonitrile. The catalyst was prepared by the general experimental procedure using 40 mg (0.12 mmol) 2,6-bis[4'-(*S*)-(*tert*-butyl)oxazolin-2'-yl]pyridine **2b** and 28 mg (0.10 mmol) $YbCl_3$. Using 120 mg (1.20 mmol) TMSCN, 84 mg (1.00 mmol) cyclopentene oxide, in 1 mL $CHCl_3$ at –10 °C for 7 days yielded the product (1*R*, 2*S*)-**7** (152mg, 0.83 mmol, 83%) in 92% ee by chiral GC analysis (Cyclodex-B, 90 °C, isothermal, t_R(major) = 19.31 min, t_R(minor) = 18.40 min).

(4) Forró, E.; Lundell, K.; Fülöp, F.; Kanerva, L. T. *Tetrahedron: Asymmetry* **1997**, *8*, 3095-3099.

$[\alpha]^{26}_{D}$ +72.0º (*c* 1.58, CH_2Cl_2); IR (thin film) 2960, 2240, 1453, 1378, 1255, 1146, 1023, 842, 752; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.35 (dd, 1 H, *J* = 5.6 and 11.7 Hz), 2.60-2.64 (m, 1 H), 2.13-2.17 (m, 1 H), 1.56-1.98 (m, 5 H), 0.15 (s, 9 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 122.0, 77.3, 37.6, 34.7, 28.4, 22.1, -0.1; HRMS calculated for $C_9H_{17}NOSi$ $(M)^+$: 183.1079; found: 183.1078. The absolute configuration was assigned by analogy to compound **1**.

(2*R*, 3*S*)-2-Methyl-3-trimethylsilyloxybutyronitrile. The catalyst was prepared by the general experimental procedure using 45 mg (0.12 mmol) 2,6-bis[4'-(*R*)-phenyloxazolin-2'-yl]pyridine **2c** and 28 mg (0.10 mmol) $YbCl_3$. Using 120 mg (1.20 mmol) TMSCN, 72 mg (1.0 mmol) *cis*-2,3-epoxybutane, in 1 mL $CHCl_3$ at –40 °C for 7 days yielded the product (136 mg, 0.80 mmol, 80%) in 90% ee as determined by chiral GC analysis (γ-TA, 50 °C, isothermal, t_R(major) = 29.79 min, t_R(minor) = 27.77 min). $[\alpha]^{24}_{D}$ +5.9º (*c* 3.8, CH_2Cl_2); IR (KBr) 2984, 2244, 1379, 1253, 1068, 954, 885, 761; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.82-3.88 (m, 1 H), 2.56-2.63 (m, 1H), 1.26 (dd, 6 H, *J* = 7.1 and 9.7 Hz), 0.14 (s, 9 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 121.3, 68.8, 34.4, 21.4, 14.1, 0.1; HRMS calculated for $C_8H_{21}N_2OSi$ $(M+NH_4)^+$: 189.1423; found: 189.1423. The absolute configuration was assigned by analogy to compound **1**.

(1*R*, 3*S*, 4*R*)-4-Cyano-3-trimethylsilyloxy-ethyl-1-cyclopentanecarboxylate. The catalyst was prepared by the general experimental procedure using 40 mg (0.12 mmol) 2,6-bis[4'-(*S*)-(*tert*-butyl)oxazolin-2'-yl]pyridine **2b** and 28 mg (0.10 mmol) $YbCl_3$. Using 120 mg (1.20 mmol) TMSCN, 156 mg (1.00 mmol) *trans*-ethyl-3,4-epoxy-ethyl-

1-cyclopentanecarboxylate,[5] in 1 mL $CHCl_3$ at 0 °C for 7 days yielded the product (219 mg, 0.86 mmol, 86%) after flash chromatography over SiO_2 using 9:1 hexanes:EtOAc as the eluent (TLC R_f = 0.45, 4:1 hexanes:EtOAc, SiO_2). $[\alpha]^{27}_D$ +30° (*c* 1.9, CH_2Cl_2); IR (KBr) 2959, 2242, 1735, 1375, 1254, 1185, 1094, 845, 756; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.45 (dd, 1 H, *J* = 5.5 and 11.4 Hz), 4.14 (dd, 2 H, *J* = 7.1 and 14.3 Hz), 3.00-3.08 (m, 1 H), 2.67-2.73 (m, 1 H), 2.41-2.49 (m, 1 H), 2.08-2.23 (m, 2 H), 1.85-1.93 (m, 1 H), 4.45 (t, 3 H, *J* = 8.7 Hz), 0.16 (s, 9 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 174.1, 120.8, 76.2, 61.0, 40.8, 38.0, 37.6, 31.4, 14.2, -0.14; HRMS calculated for $C_{12}H_{25}N_2O_3Si$ $(M+NH_4)^+$: 273.1634; found: 273.1630. The product was determined to be in 83% ee by chiral GC analysis of the TFA ester prepared by treatment of product with TFA and TFAA (G-TA, 115 °C, isothermal, t_R(major) = 22.95 min, t_R(minor) = 20.36 min). The absolute configuration was assigned by analogy to compound **1**.

(3*R*, 4*R*)-4-Cyano-3-trimethylsilyloxy-1-(2,2,2-trifluoroacetyl)pyrrolidine. The catalyst was prepared by the general experimental procedure using 40 mg (0.12 mmol) 2,6-bis[4'-(*S*)-(*tert*-butyl)oxazolin-2'-yl]pyridine **2b** and 28 mg (0.10 mmol) $YbCl_3$. Using 120 mg (1.20 mmol) TMSCN, 181 mg (1.00 mmol) 3,4-epoxy-1-(2,2,2-trifluoroacetyl)pyrrolidine,[6] in 1 mL $CHCl_3$ at -10 °C for 7 days yielded the product (201 mg, 0.72 mmol, 72%) after flash chromatography over SiO_2 using 7:3 hexanes:EtOAc as the eluent (TLC R_f = 0.68, 7:3 hexanes:EtOAc, SiO_2). The ring-opened product was determined to be in 87% ee by chiral GC analysis (Cyclodex-B, 115 °C, isothermal,

(5) Prepared according to the published procedure: Martínez, L. E.; Nugent, W. A.; Jacobsen, E. N. *J. Org. Chem.* **1996**, *61*, 7963.

t_R(major) = 56.43 min, t_R(minor) = 57.74 min). $[\alpha]^{27}_D$ +1.2° (*c* 1.1, CH_2Cl_2); IR (KBr) 2966, 2251, 1682, 1471, 1372, 1257, 1203, 1151, 843, 755; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.61-4.68 (m, 1 H), 3.91-4.08 (m, 3 H), 3.58-3.65 (m, 1 H), 3.04-3.15 (m, 1 H), 0.22 (s, 9 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.5, 117.5, 116 (q, J_{CF} = 296 Hz), 73.4, 71.1, 54.0, 53.0, 48.0, 47.2 (q, J_{CF} = 79.3 Hz), 37.7, 35.1 (q, J_{CF} = 264 Hz), -0.1; HRMS calculated for $C_{10}H_{19}N_3O_2Si$ $(M+NH_4)^+$: 298.1199; found: 298.1201. The absolute configuration was assigned by analogy to compound **1**.

Kinetic Experiments. Catalyst was generated according to the general procedure. The flask containing the catalyst residue was fitted with a rubber septum and the catalyst residue was dissolved in 1.2 mL $CHCl_3$ and sequentially treated with TMSCN (670 μL, 5.00 mmol) and 50 μL dodecane (internal standard) at rt. The reaction was initiated by the addition of cyclohexene oxide (100 μL, 1.00 mmol). The reaction progress was monitored by the removal of 10 μL aliquots from the reaction, filtration through a SiO_2 plug with CH_2Cl_2 as the eluent, and GC analysis (HP-5, 50 °C, 4 min, 15 °C / min).

[Yb] (M)	k_{obs} (sec^{-1})
0.00566	0.000052
0.0108	0.00018
0.0275	0.0011
0.0334	0.0017
0.0459	0.0029

General Procedure for the Synthesis of Pybox Ligands. Method A.[7] **2,6-Bis[4'-(*S*)-(*tert*-butyl)oxazolin-2'-yl]pyridine (2b).** To a solution of (*S*)-*tert*-leucinol (1.1g, 9.4

(6) Prepared according to the published procedure: Martínez, L. E.; Leighton, J. L.; Carsten, D. H.; Jacobsen, E. N. *J. Am. Chem. Soc.* **1995**, *117*, 5897.

(7)Davies, I. W.; Gerena, L.; Lu, N.; Larsen, R. D.; Reider, P. J. *J. Org. Chem.* **1996**, *61*, 9629.

mmol) in 16 mL of *i*-PrOAc and 3.8 mL $KHCO_3$ (1.5 M) at 65 °C was added pyridine dicarbonyldichloride (0.96 g, 4.7 mmol) portion-wise over 10 min. The mixture was heated to 80 °C and allowed to proceed at that temperature for 2 h. The reaction was allowed to cool and then partitioned with 100 mL $CHCl_3$ in a separatory funnel. The layers were separated and the aqueous layer was extracted 2 X 100 mL $CHCl_3$. The organic extracts were dried over Na_2SO_4, filtered through Celite® and concentrated *in vacuo* to yield an opaque white foam. The crude material obtained was suspended in 15 mL $BF_3 \cdot OEt_2$ and heated to 125 °C for 2 h. The solution was allowed to cool to rt and carefully poured into a cooled (0 °C) Erlenmeyer containing 100 mL CH_2Cl_2 and 100 mL 2 N NaOH. The mixture was placed in a separatory funnel, mixed well, and the layers were separated. The aqueous layer was extracted 3 X 100 mL CH_2Cl_2. The organic extracts were diluted with 200 mL EtOAc and treated with activated carbon. The mixture was filtered through SiO_2 and concentrated *in vacuo* to yield an opaque white solid. The solid was recrystallized from 9:1 hexanes:EtOAc to yield 2,6-bis[4'-(*S*)-(*tert*-butyl)oxazolin-2'-yl]pyridine (1.16 g, 3.52 mmol, 75%). $[\alpha]^{27}_D$ -118° (*c* 0.50, CH_2Cl_2); IR (KBr) 2959, 2899, 2858, 1643, 1569, 1477, 1377, 1363, 1278, 1108, 1075, 1061, 956, 931; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.26 (d, 2 H, *J* = 8.1 Hz), 7.86 (t, 1 H, *J* = 7.7 Hz), 4.49 (t, 2 H, *J* = 8.8 Hz), 4.33 (t, 2 H, *J* = 8.8 Hz), 4.11 (dd, 2 H, *J* = 8.8 and 10.6 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 147.0, 137.2, 126.0, 76.6, 69.8, 34.4, 26.3; HRMS calculated for $C_{19}H_{28}N_3O_2$ $(M+H)^+$: 330.2181; found: 330.2167.

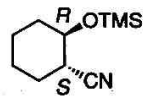
Procedure for the Synthesis of Pybox Ligand (2c).[8] **2,6-Bis[4'-(*S*)-phenyloxazolin-2'-yl]pyridine.** To a solution of (*S*)-phenylglycinol (2.2 g, 16.2 mmol) in 33 mL of *i*-PrOAc and 8 mL $KHCO_3$ (1.5 M) at 65 °C was added pyridine dicarbonyldichloride (1.65 g, 8.1 mmol) portion-wise over 15 min. The mixture was heated to 80 °C and allowed to proceed at that temperature for 2 h. The reaction was allowed to cool and then partitioned with 150 mL $CHCl_3$ in a separatory funnel. The layers were separated and the aqueous layer was extracted 2 X 100 mL $CHCl_3$. The organic extracts were dried over Na_2SO_4, filtered through Celite® and concentrated *in vacuo* to yield an opaque white solid. To a solution of the crude solid (3.24 g), DMAP (100 mg, 0.8 mmol), TEA (5.0 mL, 35 mmol) in 32 mL CH_2Cl_2 was added a solution of *p*-TsCl (3.05 g, 16 mmol) in 8 mL CH_2Cl_2 via cannula at rt over 20 min. The reaction was allowed to stir at rt 40 h. The reaction was poured into a cooled (0 °C) Erlenmeyer flask containing 200 mL CH_2Cl_2 and 200 mL 2 N NaOH. The mixture was placed in a separatory funnel, mixed well, and the layers were separated. The aqueous layer was extracted 3 X 200 mL CH_2Cl_2. The organic extracts were diluted with 300 mL EtOAc and treated with activated carbon. The mixture was filtered through SiO_2 and concentrated *in vacuo* to yield an opaque white solid. The crude solid was recrystallized from 9:1 hexanes:EtOAc and the solid obtained was collected by vacuum filtration to yield 2,6-bis[4'-(*S*)-phenyloxazolin-2'-yl]pyridine (2.13g, 5.75 mmol, 72%). $[\alpha]^{27}_{D}$ -182° (*c* 0.91, CH_2Cl_2); IR (KBr) 3150, 1738, 1650, 1644, 1568, 1453, 1354, 1264, 1240, 1164, 982, 700; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.34 (d, 2 H, *J* = 7.8 Hz), 7.91 (t, 1 H, *J* = 7.8 Hz),

(8) Modification of a similar procedure used for 2,2-Bis[1-[4-(*S*)-*tert*-butyl-1,3-oxazolinyl]]propane: Evans, D. A.; Peterson, G. S.; Johnson, J. S.; Barnes, D. M.; Campos, K. R.; Woerpel, K. A. *J. Org. Chem.* **1998**, *63*, 4541.

7.29-7.36 (m, 10 H), 5.59 (dd, 2 H, *J* = 8.7 and 10.2 Hz), 4.92 (dd, 2 H, *J* = 8.7 and 10.3 Hz), 4.42 (t, 2 H, *J* = 8.7 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 163.3, 146.6, 141.6, 137.3, 128.7, 127.7, 126.7, 126.2, 75.4, 70.2; HRMS calculated for $C_{23}H_{19}N_3O_2$ $(M+H)^+$: 370.1555; found: 370.1570.

2,6-Bis[4'-(*S*)-benzyloxazolin-2'-yl]pyridine (2d). Method A. $[\alpha]^{23}_D$ -38° (*c* 0.67, $CHCl_3$); IR (KBr) 2931, 1634, 1497, 1453, 1383, 1109, 1065, 960, 735, 699; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.22 (d, 2 H, *J* = 7.8 Hz), 7.90 (t, 1 H, *J* = 7.8 Hz), 7.21-7.33 (m, 10 H), 4.61-4.69 (m, 2 H), 4.46 (t, 2 H, *J* = 8.7 Hz), 4.26 (t, 2 H, *J* = 8.7 Hz), 3.27 (dd, 2 H, *J* = 5.2 and 13.8 Hz), 2.74 (dd, 2 H, *J* = 9.1 and 13.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.6, 146.7, 137.6, 137.3, 129.1, 128.5, 126.5, 125.7, 72.5, 68.0, 41.6; HRMS calculated for $C_{25}H_{24}N_3O_2$ $(M+H)^+$: 398.1868; found: 398.1885.

[3a*S*-[2(3'a*R,8'a*S**),3aa,8aa]]-2,2'-(2,6-Pyridinediyl)bis-[3a,8a-dihydro-8*H*-indeno[1,2-*d*]oxazole] (2e).** Method A. $[\alpha]^{23}_D$ -493° (*c* 1.0, $CHCl_3$); IR (KBr) 2985, 1638, 1572, 1462, 1381, 1110, 829; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.06 (d, 2 H, *J* = 8.2 Hz), 7.73 (t, 1 H, *J* = 8.2 Hz), 7.49-7.53 (m, 2 H), 7.18-7.24 (m, 6 H), 5.74 (d, 2 H, *J* = 8.0 Hz), 5.52-5.63 (m, 2 H), 3.44 (d, 2 H, *J* = 4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.7, 146.9, 141.4, 139.8, 136.9, 128.5, 127.3, 125.8, 125.6, 125.2, 84.2, 76.9, 39.6; HRMS calculated for $C_{25}H_{19}N_3O_2$ $(M+H)^+$: 394.1556; found: 394.1566.



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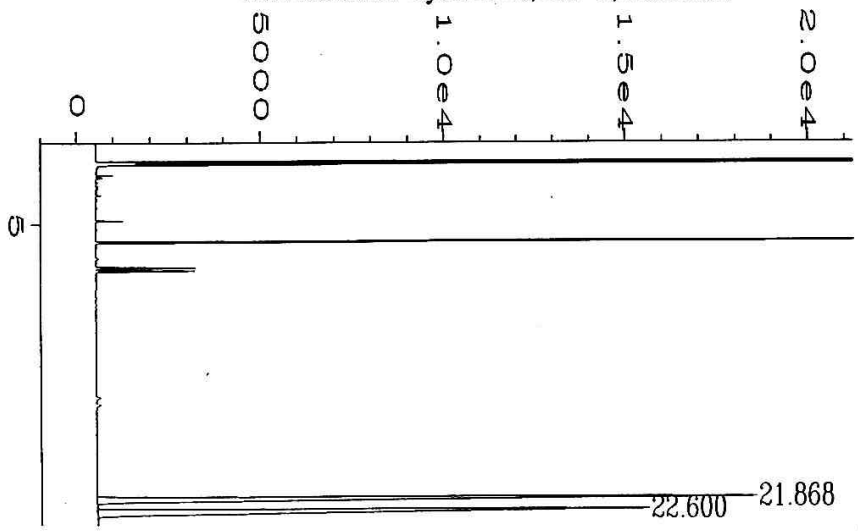

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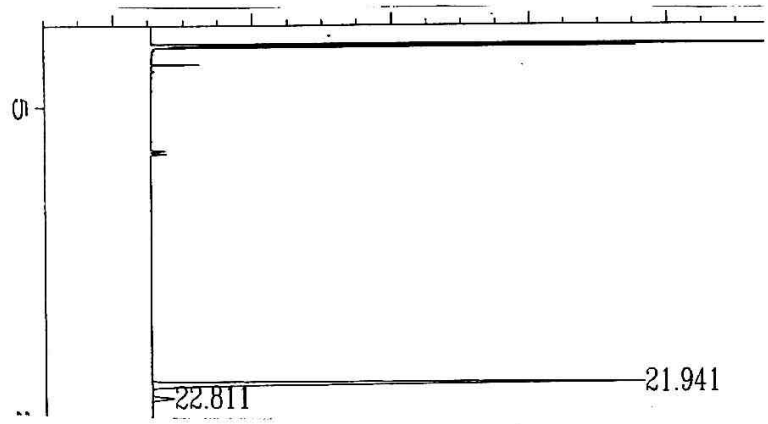

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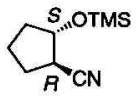

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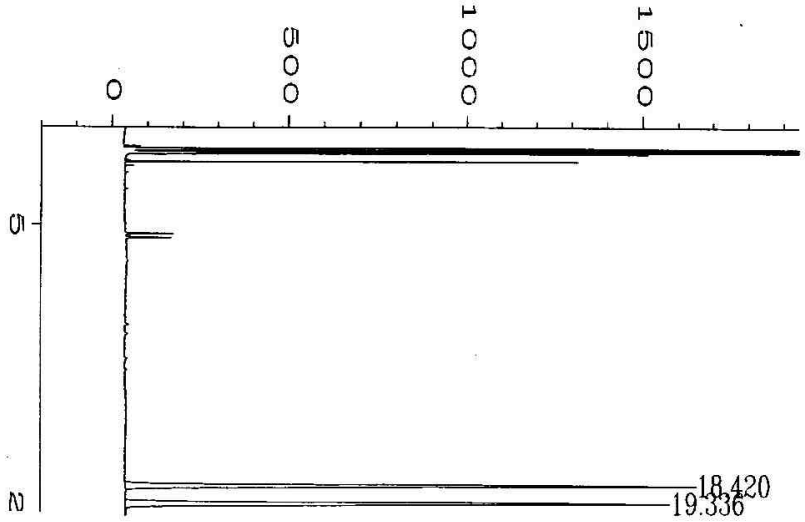

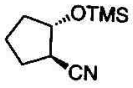

racemic

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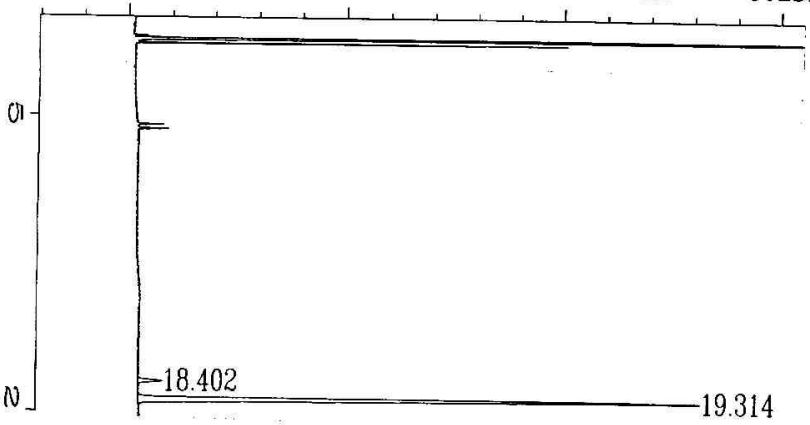
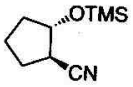
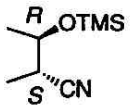




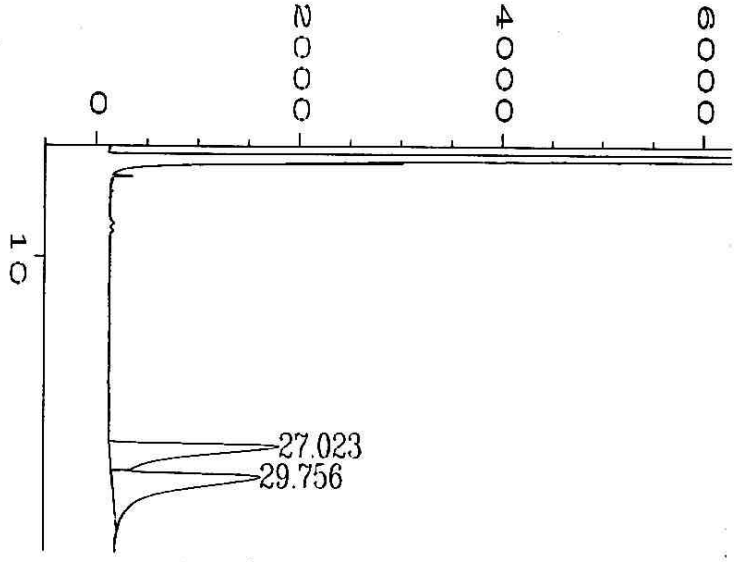
product

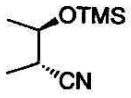
Sig. 1 in C:\PEAK\SES\SIGNAL.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	18.402	459	55	BB	0.126	3.8187
2	19.314	11551	1297	BB	0.138	96.1813



GC conditions: G-TA, 50 °C, isothermal

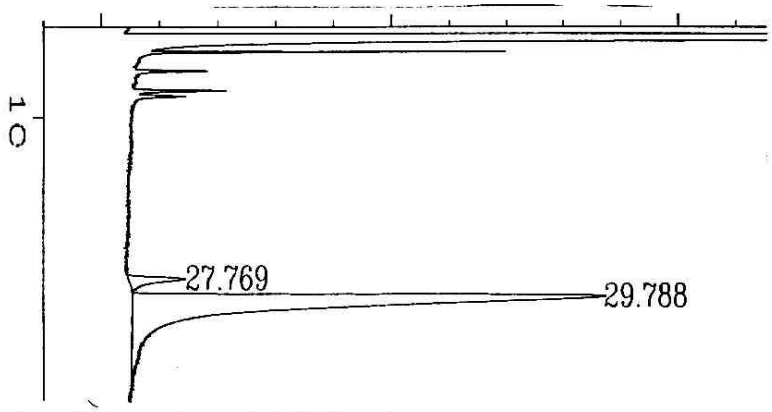


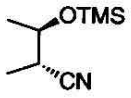


racemic

Sig. 1 in C:\PEAK\SES\SIGNAL.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	27.023	122822	1663	BV	1.053	46.1948
2	29.756	143057	1453	VB	1.358	53.8052

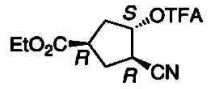




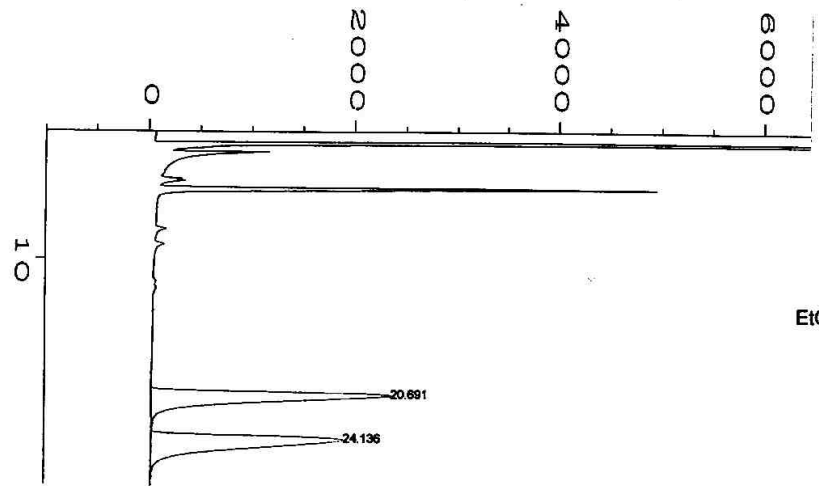
product

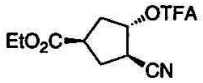
Sig. 1 in C:\PEAK\SES\SIGNAL.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	27.769	8185	193	BB	0.862	4.8063
2	29.788	162108	1628	BBA	1.462	95.1937



GC conditions: G-TA, 115 °C, isothermal

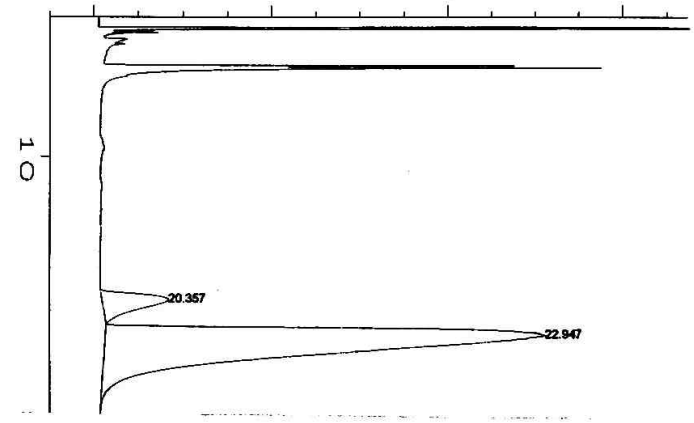


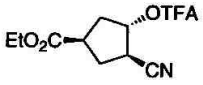


racemic

Sig. 1 in C:\PEAK\SES\SIGNAL.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	20.691	119492	2326	BB	0.782	49.9965
2	24.136	119509	1864	BB	0.993	50.0035

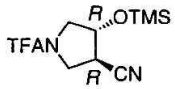




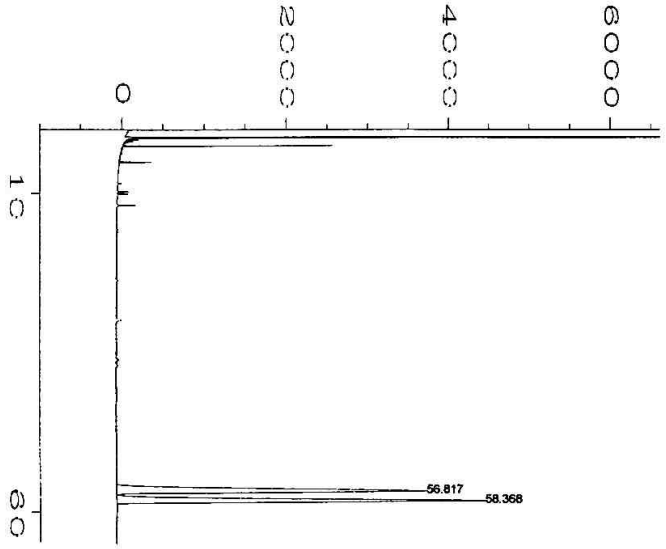
product

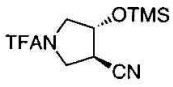
Sig. 1 in C:\PEAK\SES\SIGNAL.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	20.357	53588	742	BB	0.905	8.2236
2	22.947	598052	4978	BB	1.455	91.7764



GC conditions: Cyclodex-B, 115 °C, isothermal

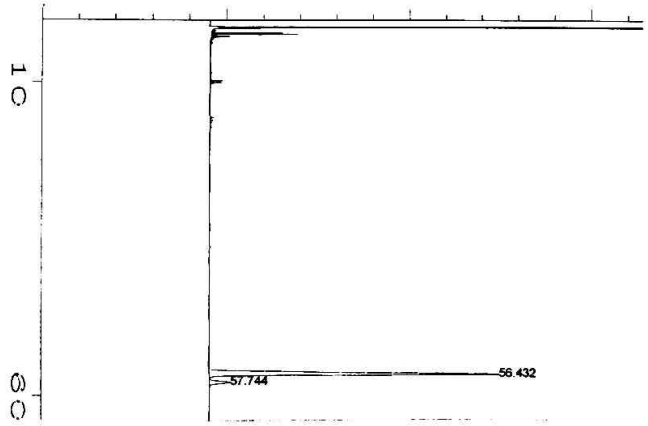


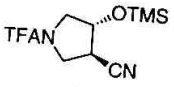


racemic

Sig. 1 in C:\PEAK\SES\F3RAC.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	56.817	151528	3778	BV	0.607	49.9338
2	58.368	151930	4505	VB	0.502	50.0662





product

Sig. 1 in C:\PEAK\SES\SIGNAL.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	56.432	43650	1588	BV	0.416	93.5560
2	57.744	3007	114	PB	0.412	6.4440