Supporting Information

Mixed La-Li Heterobimetallic Complexes for Tertiary Nitroaldol Resolution

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General: Reactions were monitored by thin-layer chromatography carried out on 0.25 mm Merck silica gel plates (60F-254). Infrared (IR) spectra were recorded on a JASCO FT/IR 410 Fourier transform infrared spectrophotometer. NMR spectra were recorded on JEOL JNM-LA500 and JNM-ECX500 spectrometers, operating at 500 MHz for ^1H NMR and 125.65 MHz for ^{13}C NMR. For ^1H NMR, chemical shifts in CDCl_3 were reported downfield from TMS (= 0) or in the scale relative to CHCl_3 (7.24 ppm); chemical shifts in CD_3OD were reported in the scale relative to CH_3OH (3.31 ppm). For ^{13}C NMR, chemical shifts were reported in the scale relative to CHCl_3 (77.0 ppm for ^{13}C NMR) or to CH_3OH (49.0 ppm for ^{13}C NMR) as an internal reference. Optical rotations were measured on a JASCO P-1010 polarimeter. ESI mass spectra were measured on Waters-ZQ4000. FAB mass spectra were measured on a JEOL JMS-MS700V in positive ion mode. Column chromatography was performed with silica gel Merck 60 (230–400 mesh ASTM). The enantiomeric excess (ee) was determined by HPLC analysis. HPLC was performed on JASCO HPLC systems consisting of the following: pump, PU-2080; detector, UV-2075, measured at 230 nm or 254 nm; column, DAICEL CHIRALPAK AD-H, DAICEL CHIRALCEL OD-H; mobile phase, hexane–2-propanol; Reactions were carried out in dry solvents under an argon atmosphere, unless otherwise stated. Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl. $\text{La}(\text{O}-i\text{-Pr})_3$ was purchased from Kojundo Chemical Laboratory Co., LTD., 5-1-28, Chiyoda, Sakado-shi, Saitama 350-0214, Japan (Fax: +81-492-84-1351, sales@kojundo.co.jp). $\text{La}(\text{O}-i\text{-Pr})_3$ is also available from Aldrich (Cat. No. 665193). BuLi were purchased from Aldrich. Other reagents were purified by standard methods.

General Procedure for (*R*)-LLB **2a** and (*R*)-LLB* **2b**:

To a stirred solution of (*R*)-BINOL (859 mg, 3.00 mmol) in THF (8 mL) at 0 °C was added slowly a solution of La(O-*i*Pr)₃ (5 mL, 1.00 mmol, 0.20 M in THF). The solution was allowed to stir for 30 min at room temperature, and then solvent and *i*-PrOH was removed slowly under reduced pressure and dried for 1 h under vacuum. The residue was cooled at 0 °C, and THF (8 mL) was added. To the solution was added slowly BuLi (1.88 mL, 3.00 mmol, 1.60 M in hexane). After stirring for 1 h at room temperature, the solvent was removed slowly under reduced pressure and dried for 3 h under vacuum. The residue was cooled at 0 °C and THF (7.52 mL) was added. The mixture was stirred at room temperature for 1 h to afford (*R*)-LLB **2a** solution (0.133 M in THF).

(*R*)-LLB* **2b** was prepared in a similar procedure using (*R*)-biphenol **1b**. (*R*)-LLB* **2b** was stocked as 0.067 M solution in THF.

General Procedure for Kinetic Resolution of Racemic Tertiary Nitroaldols

To a test tube were added THF (0.2 mL), (*R*)-LLB **2a** (0.050 mL, 0.0067 mmol, 0.133 M in THF), and (*R*)-LLB* **2b** (0.0500 mL, 0.0033 mmol, 0.067 M in THF), and the mixture solution was stirred for 45 min at room temperature. The solution was cooled at –20 °C, and then (±)-**4a** (0.200 mL, 0.200 mmol, 1 M solution in THF) was added. After stirring for 23 h at –20 °C, the reaction was quenched by citric acid (0.5 mL, 0.25 M solution in THF). The resulting mixture was diluted with Et₂O and poured into water. The aqueous layer was extracted with Et₂O (x 3) and the combined organic layers were washed with brine, and then dried over Na₂SO₄. After removing solvent under reduced pressure, the residue was purified by flash column chromatography (neutral SiO₂, hexane/acetone = 20/1) to give (*R*)-**4a** (47% recovery) as pale yellow oil.

(*R*)-2-Cyclohexyl-1-nitro-2-propanol (**4a**):

pale yellow oil; IR (neat) ν 3543, 2926, 2855, 1384 cm⁻¹; ¹H NMR (CDCl₃) δ 4.52 (d, *J* = 12 Hz, 1H), 4.41 (d, *J* = 12 Hz, 1H), 2.81 (s, 1H), 1.87-1.79 (m, 4H), 1.70 (m, 1H), 1.44 (m, 1H), 1.25-0.97 (m, 5H), 1.23 (s, 3H); ¹³C NMR (CDCl₃) δ 83.3, 73.8, 46.4, 27.7, 26.7, 26.4, 26.2, 21.5; ESI-MS *m/z* 210 [M+Na]⁺; HRMS [FAB(+)] Calcd for C₉H₁₇N₃O₃⁺ [M+Cs]⁺: 320.0257; Found 320.0265; [α]_D²² –2.7 (*c* 2.4, CHCl₃); HPLC (DAICEL CHIRALPAK AD-H, 2-propanol/hexane 1/9, flow 1.0 mL/min, detection at 230 nm) *t*_R 8.3 min (minor) and 11.5 min (major). Absolute configuration of **4a** was determined after conversion into known compound following the scheme shown below. By comparing the sign of optical rotation with the reported data, the absolute configuration of **4a** was determined to be *R*. [Lit. (a) Blackwell, J. M.; Foster, K. L.; Beck, V. H.; Piers, W. E. *J. Org. Chem.* **1999**, *64*, 4887. (b) Matt, C.; Wagner, A.; Mioskowski, C. *J. Org. Chem.* **1997**,

62, 234. (c) Sharpless, K. B.; Amberg, W.; Beller, M.; Chen, H.; Hartung, J.; Kawanami, Y.; Lübben, D.; Manoury, E.; Ogino, Y.; Shibata, T.; Ukita, T. *J. Org. Chem.* **1991**, *56*, 4585.]

(R)-2,4-Dimethyl-1-nitro-2-pentanol (4b):

pale yellow oil; IR (neat) ν 3541, 2958, 2872, 1551, 1382 cm^{-1} ; ^1H NMR (CDCl_3) δ 4.45 (d, $J = 12$ Hz, 1H), 4.39 (d, $J = 12$ Hz, 1H), 2.81 (s, 1H), 1.88-1.81 (m, 1H), 1.52-1.43 (m, 2H), 1.32 (s, 3H), 1.00 (d, $J = 6.4$ Hz, 3H), 0.98 (d, $J = 6.7$ Hz, 3H); ^{13}C NMR (CDCl_3) δ 84.9, 72.1, 47.9, 24.7, 24.6, 24.3, 24.0; ESI-MS m/z 184 $[\text{M}+\text{Na}]^+$; HRMS [FAB(+)] Calcd for $\text{C}_7\text{H}_{15}\text{CsNO}_3^+ [\text{M}+\text{Cs}]^+$: 294.0101; Found 294.0097; $[\alpha]_{\text{D}}^{22} -2.3$ (c 1.4, CHCl_3); HPLC (DAICEL CHIRALPAK AD-H, 2-propanol/hexane 1/20, flow 1.0 mL/min, detection at 230 nm) t_{R} 10.7 min (minor) and 12.4 min (major). Absolute configuration was tentatively assigned as *R* based on absolute configurations of **4a**, **4e**, and **4f**.

(R)-3-Ethyl-2-methyl-1-nitro-2-pentanol (4c):

pale yellow oil; IR (neat) ν 3544, 2967, 2878, 1553, 1382 cm^{-1} ; ^1H NMR (CDCl_3) δ 4.54 (d, $J = 12$ Hz, 1H), 4.43 (d, $J = 12$ Hz, 1H), 2.84 (s, 1H), 1.67 (m, 1H), 1.54 (m, 1H), 1.27-1.20 (m, 3H), 1.24 (s, 3H), 1.00 (t, $J = 7.1$ Hz, 3H), 0.97 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (CDCl_3) δ 83.5, 75.0, 50.1, 23.2, 22.2, 21.8, 13.9, 13.4; ESI-MS m/z 198 $[\text{M}+\text{Na}]^+$; HRMS [FAB(+)] Calcd for $\text{C}_8\text{H}_{17}\text{CsNO}_3^+ [\text{M}+\text{Cs}]^+$: 308.0257; Found 308.0261; $[\alpha]_{\text{D}}^{22} +1.4$ (c 1.4, CHCl_3); HPLC (DAICEL CHIRALPAK AD-H, 2-propanol/hexane 1/20, flow 1.0 mL/min, detection at 230 nm) t_{R} 8.9 min (minor) and 10.7 min (major). Absolute configuration was tentatively assigned as *R* based on absolute configurations of **4a**, **4e**, and **4f**.

(R)-3-Cyclohexyl-2-methyl-1-nitro-2-propanol (4d):

pale yellow oil; IR (neat) ν 3541, 2923, 2851, 1550, 1382 cm^{-1} ; ^1H NMR (CDCl_3) δ 4.45 (d, $J = 12$ Hz, 1H), 4.39 (d, $J = 12$ Hz, 1H), 2.80 (s, 1H), 1.84-1.44 (m, 8H), 1.32 (s, 3H), 1.30-0.95 (m, 5H); ^{13}C NMR (CDCl_3) δ 84.9, 72.2, 46.8, 35.1, 34.9, 33.3, 26.3, 26.2, 26.1, 24.8; ESI-MS m/z 224 $[\text{M}+\text{Na}]^+$; HRMS [FAB(+)] Calcd for $\text{C}_{10}\text{H}_{19}\text{CsNO}_3^+ [\text{M}+\text{Cs}]^+$: 334.0414; Found 334.0410; $[\alpha]_{\text{D}}^{20} -1.9$ (c 1.3, CHCl_3); HPLC (DAICEL CHIRALPAK AD-H, 2-propanol/hexane 1/9, flow 1.0 mL/min, detection at 230 nm) t_{R} 8.3 min (minor) and 12.7 min (major). Absolute configuration was tentatively assigned as *R* based on absolute configurations of **4a**, **4e**, and **4f**.

(R)-2-Methyl-1-nitro-4-phenyl-2-butanol (4e):

colorless oil; IR (neat) ν 3269, 2922, 2849, 1548, 1384 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.31-7.18 (m, 5H), 4.50 (d, $J = 12$ Hz, 1H), 4.45 (d, $J = 12$ Hz, 1H), 2.92 (s, 1H), 2.77-2.74 (m, 2H), 1.90-1.86 (m, 2H), 1.39 (s, 3H); ^{13}C NMR (CDCl_3) δ 141.0, 128.6, 128.3, 126.2, 84.1, 71.5, 41.5, 29.8, 24.4; ESI-MS m/z 232 $[\text{M}+\text{Na}]^+$; HRMS $[\text{FAB}(+)]$ Calcd for $\text{C}_{11}\text{H}_{15}\text{NO}_3^+$ $[\text{M}+\text{Cs}]^+$: 342.0101; Found 342.0094; $[\alpha]_{\text{D}}^{20} -3.7$ (c 2.40, CHCl_3); HPLC (DAICEL CHIRALCEL OD-H, 2-propanol/hexane 1/4, flow 1.0 mL/min, detection at 254 nm) t_{R} 12.0 min (major) and 15.3 min (minor). Absolute configuration of **4e** was determined after conversion into known compound following the scheme shown below. By comparing the sign of optical rotation with the reported data, the absolute configuration of **4e** was determined to be *R*. [Lit. (a) Blackwell, J. M.; Foster, K. L.; Beck, V. H.; Piers, W. E. *J. Org. Chem.* **1999**, *64*, 4887. (b) Matt, C.; Wagner, A.; Mioskowski, C. *J. Org. Chem.* **1997**, *62*, 234. (c) Jiang, B.; Chen, Z.; Tang, X. *Org. Lett.* **2002**, *4*, 3451.]

(R)-1-Nitro-2-phenyl-2-propanol (4f):

($\pm$)-**4f** is a known compound (Bordwell, F. G. Garbisch, Jr. E. W. *J. Org. Chem.* **1962**, *27*, 2322.) colorless oil; $[\alpha]_{\text{D}}^{22} +6.7$ (c 3.6, CHCl_3); HPLC (DAICEL CHIRALCEL OD-H, 2-propanol/hexane 1/6, flow 1.0 mL/min, detection at 230 nm) t_{R} 13.0 min (major) and 15.3 min (minor). Absolute configuration of **4f** was determined after conversion into known compound following the scheme shown below. By comparing the sign of optical rotation with the reported data, the absolute configuration of **4f** was determined to be *R*. [Lit. (a) Jarvo, E. R.; Evans, C. A.; Copeland, G. T.; Miller, S. J. *J. Org. Chem.* **2001**, *66*, 5522.]

(R)-2-Ethyl-4-methyl-1-nitro-2-pentanol (4g):

colorless oil; IR (neat) ν 3550, 2958, 1555, 1381 cm^{-1} ; ^1H NMR (CDCl_3) δ 4.44 (d, $J = 12$ Hz, 1H), 4.41 (d, $J = 12$ Hz, 1H), 2.73 (s, 1H), 1.82-1.74 (m, 1H), 1.67-1.56 (m, 2H), 1.47 (dd, $J = 15, 5.5$ Hz, 1H), 1.38 (dd, $J = 15, 6.8$ Hz, 1H), 0.97-0.90 (m, 9H); ^{13}C NMR (CDCl_3) δ 82.9, 74.4, 44.7, 30.2, 24.6, 24.3, 23.6, 8.1; ESI-MS m/z 198 $[\text{M}+\text{Na}]^+$; $[\alpha]_{\text{D}}^{22} +1.0$ (c 2.05, CHCl_3); HPLC (DAICEL CHIRALPAK AD-H, 2-propanol/hexane 1/20, flow 1.0 mL/min, detection at 230 nm) t_{R} 9.5 min (minor) and 13.6 min (major). Absolute

configuration was tentatively assigned as *R* based on absolute configurations of **4a**, **4e**, and **4f**.

Transformation of *tert*-Nitroaldols:

(*R*)-*N*-(2-Cyclohexyl-2-hydroxy-propyl)-acetamide (**5a**):

A suspension of **4a** (21.4 mg, 0.114 mmol) and 10% Pd-C (6.1 mg) in MeOH (1 mL) was stirred under H₂ (1 atm) at room temperature for 12 h. Then, the reaction mixture was filtrated through Celite pad and washed with Et₂O. The filtrates were evaporated under reduced pressure to give a crude amine. To a solution of the crude amine and Et₃N (0.017 mL, 0.122 mmol) in CH₂Cl₂ was added acetic anhydride (0.013 mL, 0.138 mmol) at 0 °C and the reaction mixture was stirred at room temperature for 2 h. The reaction was quenched with 0.5 mL of *sat. aq.* NH₄Cl. The mixture was extrated with Et₂O (1 mL x 3), and combined organic layers were washed with brine (3 mL) and dried over Na₂SO₄. After concentration under reduced pressure, the residue was purified by silica gel column chromatography (CH₂Cl₂/MeOH = 90:10) to give **5a** (19.5 mg, 86%) as a colorless solid. Analytical data matched well with reported data in the literature [Lit. Jarvo, E. R.; Evans, C. A.; Copeland, G. T.; Miller, S. J. *J. Org. Chem.* **2001**, *66*, 5522.] colorless solid; ¹H NMR (CDCl₃) δ 5.85 (brs, 1H), 3.31 (dd, *J* = 14, 6.4 Hz, 1H), 3.21 (dd, *J* = 14, 5.2 Hz, 1H), 2.00 (s, 3H), 1.83-1.64 (m, 5H), 1.41-1.33 (m, 1H), 1.25-0.95 (m, 5H); ¹³C NMR (CDCl₃) δ 170.3, 74.7, 47.6, 46.5, 27.8, 26.7, 26.6, 26.6, 26.4, 23.2, 21.4; [α]_D²⁶ +2.4 (*c* 1.2, CHCl₃).

(*R*)-Triethyl-(1-methyl-1-nitromethyl-3-phenyl-propoxy)-silane (**6e**):

To a stirred solution of **4e** (51.7 mg, 0.247 mmol), and Et₃SiH (0.047 mL, 0.296 mmol) in CH₂Cl₂ (0.5 mL) was added B(C₆F₅)₃ (12.6 mg, 0.0247 mmol) at room temperature. After being stirred for 1.5 h at the same temperature, B(C₆F₅)₃ (12.6 mg) was added again. After 2.5 h, the reaction was quenched with 0.5 mL of H₂O. The mixture was extracted with Et₂O (1 mL x 3). The combined organic layers were washed with brine (3 mL), and dried over Na₂SO₄. After concentration under reduced pressure, the residue was purified by neutral silica gel column chromatography (neutral SiO₂, hexane/Et₂O = 95:5) to give **6e** (71.9 mg, 90%) as a colorless oil. IR (neat) ν 2956, 1552, 1145, 745 cm⁻¹; ¹H NMR (CDCl₃) δ 7.30-7.18 (m, 5H), 4.44 (d, *J* = 10 Hz, 1H), 4.41 (d, *J* = 10 Hz, 1H), 2.77-2.64 (m, 2H), 1.98-1.87 (m, 2H), 1.46 (s, 3H), 0.96 (t, *J* = 8.0 Hz, 9H), 0.63 (q, *J* = 8.0 Hz, 6H); ¹³C NMR (CDCl₃) δ 141.4, 128.5, 128.3, 126.0, 83.9, 74.2, 43.1, 30.4, 26.2, 7.0, 6.6; ESI-MS *m/z* 346 [M+Na]⁺; HRMS [FAB(+)] Calcd for C₁₇H₂₉CsNO₃Si⁺ [M+Cs]⁺: 456.0971; Found 456.0964; [α]_D²⁹ -6.5 (*c* 2.4, CHCl₃).

(*R*)-3-(1-Methyl-3-phenyl-1-triethylsilanyloxy-propyl)-5-phenyl-isoxazole (**7e**)

To a solution of **6e** (6.51 mg, 20.1 μmol) in dry benzene (0.5 mL) with one drop of Et₃N was

added phenyl acetylene (4.4 μ L, 40.2 μ mol) and phenyl isocyanate (10.9 μ L, 0.10 mmol) successively at room temperature. After stirring for 30 min, the mixture was refluxed for 40 h. Water was added to the mixture at rt, then one drop of 1 M HCl was added. The

product was extracted with diethyl ether (4 mL x 3), and combined organic layer was washed with brine (4 mL), then dried over Na_2SO_4 . After filtration, diethyl ether was evaporated under reduced pressure and the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 12:1) to give **7e** (6.87 mg, 84% yield) as a colorless oil. IR (neat) 2954, 1450 cm^{-1} ; ^1H NMR (CDCl_3) 7.81-7.79 (m, 2H), 7.49-7.43 (m, 3H), 7.26 (dd, J = 7.6, 7.7 Hz, 2H), 7.17-7.14 (m, 3H), 6.57 (s, 1H), 2.71 (ddd, J = 4.9, 12.5, 13.5 Hz, 1H), 2.61 (ddd, J = 5.2, 12.5, 12.5 Hz, 1H), 2.20 (ddd, J = 5.2, 12.5, 13.5 Hz, 1H), 2.09 (ddd, J = 4.9, 12.5, 12.5 Hz, 1H), 1.77 (s, 3H), 1.00-0.97 (m, 9H), 0.67-0.62 (m, 6H); ^{13}C NMR (CDCl_3) 171.1, 169.3, 142.2, 130.0, 128.9, 128.3, 127.7, 125.8, 125.7, 98.4, 73.7, 46.7, 30.5, 27.5, 7.1, 6.6; ESI-MS m/z 193 [$\text{M}+\text{Na}$] $^+$; HRMS [FAB(+)] Calcd for $\text{C}_{25}\text{H}_{33}\text{CsNO}_2\text{Si}^+$ [$\text{M}+\text{Cs}$] $^+$: 540.1335; Found 540.1334; $[\alpha]_{\text{D}}^{26} +1.1$ (c 3.2, CHCl_3).

(R)-2-Hydroxy-2-methyl-4-phenyl-butyric acid (**8e**)

To a stirred solution of **6e** (28.4 mg, 87.8 μ mol) in dimethyl sulfoxide (0.44 mL) and acetic acid (75.4 μ L, 1.32 mmol) was added sodium nitrite (60.6 mg, 0.878 mmol) at room temperature. The resulting

mixture was stirred at room temperature for 12 h and then at 40 $^{\circ}\text{C}$ for 8 h. The solution was cooled down to room temperature and quenched with 1 M HCl (1 mL). The mixture was extracted with diethyl ether (5 mL x 3). The combined organic layers were washed with 2 M NaOH (5 mL x 3). Then, the combined water layers were acidified with 4 M HCl. The acidic water layer was again extracted with diethyl ether (5 mL x 3), and combined organic layers were dried over Na_2SO_4 . After filtration of Na_2SO_4 , solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ = 6:1) to give **8e** (16.8 mg, 99% yield) as a colorless solid. IR (KBr) 3238, 2925, 1726 cm^{-1} ; ^1H NMR (CD_3OD) 7.21-7.18 (m, 2H), 7.13-7.09 (m, 3H), 2.82 (ddd, J = 3.4, 13.2, 13.2 Hz, 1H), 2.53 (ddd, J = 4.1, 12.6, 13.2 Hz, 1H), 2.10 (ddd, J = 3.4, 12.6, 13.2 Hz, 1H), 1.85 (ddd, J = 4.1, 13.2, 13.2 Hz, 1H), 1.45 (s, 3H); ^{13}C NMR (CD_3OD) 179.4, 143.3, 129.4, 129.3, 126.8, 75.4, 43.7, 31.3, 26.5; ESI-MS [anion mode] m/z 193 [$\text{M}-\text{H}$] $^-$; $[\alpha]_{\text{D}}^{31} +16.9$ (c 0.13, CHCl_3).

Synthesis of Racemic Tertiary Nitroaldols **2b** from α -nitroketone

For the synthesis of racemic **4a**, **4b**, **4d**, and **4e**, previously reported methods were applicable [(a) Seebach, D.; Lehr, F. *Angew. Chem., Int. Ed. Engl.* **1976**, *15*, 505. (b) Eyer, M.; Seebach, D. *J. Am. Chem. Soc.* **1985**, *107*, 3601. (c) Kisanga, P. B.; Verkade, J. G. *J. Org. Chem.* **1999**, *64*, 4298]. On the other hand, known methods for racemic tertiary nitroaldols were not suitable for the synthesis of **4f**. Therefore, we developed a new method using $B(OMe)_3$ and Grignard reagent (eq S-1). Optimizations of the reaction conditions are summarized in Table S-1. With either $MeMgBr$ or $MeLi$ alone, no reaction proceeded. In entry 3, addition reaction proceeded in the presence of HMPA and TMEDA at $-30\text{ }^{\circ}C$ to rt; however, retro-nitroaldol reaction proceeded under the reaction conditions, giving acetophenone instead of desired product **4f**. On the other hand, $MeMgBr$ in combination with $B(OMe)_3$ afforded selectively tertiary nitroaldol **4f** (quantitative conversion by NMR analysis) at $-20\text{ }^{\circ}C$ after 20 min (entry 4). Use of $MeLi$ instead of $MeMgBr$ gave worse results (entry 4 vs entry 5). Reaction proceeded cleanly at $-20\text{ }^{\circ}C$. Starting material α -nitro ketone **9f** is commercially available. The new method is also applicable to the synthesis of other racemic tertiary nitroaldols **4a-4e**. α -Nitro ketones **9** for the synthesis of **4a-4e** were synthesized from carboxylic acids by the reported method (Lit. Katritzky, A. R.; Abdel-Fattah, A. A. A.; Gromova, A. V.; Witek, R.; Steel, P. J. *J. Org. Chem.* **2005**, *70*, 9211). Detailed substrate scope and limitations will be reported in due course as a full article.

Table S-1. Optimization of Racemic Tertiary Nitroaldol Synthesis

entry	conditions	4f	3f
1	MeMgBr, THF	0 ^a	—
2	MeLi, THF	0 ^a	—
3	MeLi, HMPA, TMEDA, THF, −30 °C to rt	0 ^a	85 ^a
4	B(OMe) ₃ (1 equiv), MeMgBr (3 equiv), THF, −20 °C	quant ^a (89) ^b	—
5	B(OMe) ₃ (1 equiv), MeLi (3 equiv), THF, −20 °C	61 ^a	—

^a Conversion yield determined by NMR analysis of crude mixture.

^b Isolated yield after column chromatography.

General Procedure for the Synthesis of Racemic Tertiary Nitroaldols 4

To a stirred solution of benzoynitromethane **9f** (33.0 mg, 0.200 mmol) in THF (0.4 mL) at $-20\text{ }^{\circ}\text{C}$ was added B(OMe)_3 (0.022 mL, 0.200 mmol). After 5 min at $-20\text{ }^{\circ}\text{C}$, MeMgBr (0.625 mL, 0.600 mmol, 0.96 M in THF) was added. After stirring for 30 min at $-20\text{ }^{\circ}\text{C}$, the reaction was quenched with 5% *aq.* citric acid. The aqueous layer was extracted with Et_2O (x 3) and the combined organic layers were washed with brine, and then dried over Na_2SO_4 . After concentration under reduced pressure, the resulting residue was purified by flash column chromatography (neutral SiO_2 , hexane/ethyl acetate = 20/1) to give ($\pm$)-**4f** (32.3 mg, 89%) as a colorless oil.

To obtain information of structure of catalyst used in this manuscript, solutions of a mixture of LLB **2a**/LLB* **2b** = 2:1, LLB **2a**/LLB* **2b** = 1:2, LLB **2a**, and LLB* **2b** were analyzed by ESI-MS (Waters-ZQ4000). ESI-MS charts are summarized in Figure S-1, S-2, S-3, and S-4.

(1) A mixture of LLB **2a**/LLB* **2b** = 2:1

conditions: capillary 3.80 kV, cone 160.0 V, source temp 100 °C, desolvation temp 300 °C

The figure displays several chemical structures of lanthanide complexes. The top row shows four complexes: $\text{La-Li}_3\text{-(1a)}_3 = (R)\text{-2a}$, $\text{La-Li}_3\text{-(1a)}_2\text{(1b)}$ (proposed structure), $\text{La-Li}_3\text{-(1a)(1b)}_2$ (proposed structure), and $\text{La-Li}_3\text{-(1b)}_3 = (R)\text{-2b}$. The bottom row shows three fragments: 1b-Li_2 , La-Li-(1a)_2 (proposed structure), and La-Li-(1a)(1b) (proposed structure). The structures involve fluorenyl ligands (1a and 1b) coordinated to lanthanum (La) and lithium (Li) ions, with various substituents like methoxy (MeO) and methyl (Me) groups.

(2) A mixture of LLB **2a**/LLB* **2b** = 1:2

sample preparation: The LLB **2a** (5 μ L, 0.133 M in THF, 0.67 μ mol) and LLB* **2a** (20 μ L, 0.067M in THF, 1.33 μ mol) was diluted with dry THF (2 mL) and dry 2-propanol (0.5 mL).

conditions: capillary 4.00 kV, cone 160.0 V, source temp 100 $^{\circ}$ C, desolvation temp 300 $^{\circ}$ C

Figure S-2. ESI-MS of a mixture of LLB **2a**/LLB* **2b** = 1:2.

[postulated structures of observed peaks in Figure S-2]

(3) [(*R*)-LLB **2a**]

sample preparation: The LLB **2a** (10 μ L, 0.133 M in THF, 1.33 μ mol) was diluted with dry THF (2 mL) and dry 2-propanol (1 mL).

conditions: capillary 3.00 kV, cone 100.0 V, source temp 100 $^{\circ}$ C, desolvation temp 300 $^{\circ}$ C

Figure S-3. ESI-MS chart of LLB **2a**.

(4) [(*R*)-LLB* **2b**]

sample preparation: The LLB* **2a** (20 μ L, 0.067M in THF, 1.33 μ mol) was diluted with dry THF (2 mL) and dry 2-propanol (1 mL).

conditions: capillary 3.80 kV, cone 160.0 V, source temp 100 $^{\circ}$ C, desolvation temp 300 $^{\circ}$ C

Figure S-4. ESI-MS chart of LLB* **2b**.

[Discussion]

In Figure S-1, a mixture of LLB **2a**/LLB* **2b** = 2:1 was analyzed. Peaks corresponding to $[\text{La-Li}_3\text{-(1a)}_2\text{/(1b)} + \text{Li}]^+$ and $[\text{La-Li}_3\text{-(1a)}\text{/(1b)}_2 + \text{Li}]^+$ complexes were observed as major peaks in addition to minor peaks of LLB **2a** and LLB* **2b**. In Figure S-2, a mixture of LLB **2a**/LLB* **2b** = 1:2 was analyzed. Peaks corresponding to $[\text{La-Li}_3\text{-(1a)}\text{/(1b)}_2 + \text{Li}]^+$ and $[\text{LLB}^* \text{2b} + \text{Li}]^+$ complexes were observed as major peaks in addition to a minor peak of $[\text{La-Li}_3\text{-(1a)}_2\text{/(1b)} + \text{Li}]^+$. Only trace $[\text{LLB } \text{2a} + \text{Li}]^+$ peak was observed in Figure S-2. In the retro-nitroaldol reaction of **4a** (Table 1), selectivity factor: $s = 58.4$ with LLB **2a**/LLB* **2b** = 2:1 mixture (Table 1, entry 2), while $s = 5.5$ with LLB **2a**/LLB* **2b** = 1:2 mixture (Table 1, entry 3). Furthermore, a mixture of LLB **2a**/LLB* **2b** = 2:1 gave better results than LLB **2a** alone ($s = 23.8$, Table 1, entry 1). Therefore, we assume that the $\text{La-Li}_3\text{-(1a)}_2\text{/(1b)}$ complex, generated by ligand exchange between LLB **2a** and LLB* **2b**, would be the most enantioselective and active catalyst.

Table 1. Effects of LLB **2a**/LLB* **2b** Ratio on Kinetic Resolution of Tertiary Nitroaldol ($\pm$)-**4a**.

$(\pm)\text{-4a} \xrightarrow[\text{THF, } -20\text{ }^\circ\text{C}]{\begin{smallmatrix} (R)\text{-LLB } \textbf{2a} \text{ (x mol \%)} \\ (R)\text{-LLB}^* \textbf{2b} \text{ (y mol \%)} \end{smallmatrix}} (R)\text{-4a} + \textbf{3a}, \text{CH}_3\text{NO}_2$

entry	LLB (x mol %)	LLB* (y mol %)	time (h)	recov. of 4a ^a (%)	ee ^b (%)	<i>s</i>
1	5	0	24	48	86	23.8
2	3.33	1.67	23	50	90	58.4
3	1.67	3.33	24	51	52	5.5
4	0	5	48	58	14	1.7

^a Determined by ¹H NMR analysis using mesitylene as an internal standard. ^b Determined by chiral HPLC analysis.

DFILE F:\2005\11\051103-1-13C.als
COMNT cyclohexyl methyl 13C
DATIM Thu Nov 3 16:19:26 2005
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 120.00 KHz
OBFIN 7958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 102
ACQTM 0.967 sec
PD 2.033 sec
PW1 6.2 us
IRNUC 1H
CTEMP 27.5 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 30

27.7122
26.6593
26.3714
26.2069
21.4854

46.3596

83.2926
77.2550
77.0000
76.7532
73.7920

DFILE F:\2005\11\051123-1-13C.als
COMNT 1 13C
DATIM Wed Nov 23 17:48:21 2005
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 120.00 KHz
OBFIN 7958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 74
ACQTM 0.967 sec
PD 2.033 sec
PW1 6.2 us
IRNUC 1H
CTEMP 26.5 C
SIVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 31

24.7016
24.5947
24.2821
23.9942

47.8896

84.9459
77.2550
77.2056
77.0000
76.7532
72.1304

DFILE F:\2005\11\051116-1-13C.als
COMNT 1 13C
DATIM Wed Nov 16 17:48:00 2005
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 120.00 KHz
OBFIN 7958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 89
ACQTM 0.967 sec
PD 2.033 sec
PW1 6.2 us
IRNUC 1H
CTEMP 27.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 30

23.2457
22.2339
21.8227
13.9261
13.3585

50.0941

83.5229
77.2550
77.0000
76.7450
74.9601

DFILE F:\2005\11\051101-2-13C.als
COMNT 2 13C
DATIM Tue Nov 1 21:08:32 2005
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 120.00 KHz
OBFIN 7958.0 Hz
POINT 32768
FREQ 33898.3 Hz
SCANS 128
ACQTM 0.967 sec
PD 2.033 sec
PW1 6.2 us
IRNUC 1H
CTEMP 27.5 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 30

84.8801
77.2468
77.0000
76.7450
72.1880
46.8203
35.0659
34.9343
33.3385
26.3221
26.2563
26.0506
24.8003

81-5

DFILE F:\2005\06\050606-1-13C.als
COMNT 13C
DATIM Mon Jun 6 18:13:15 2005
OBNUC 13C
EXMOD bcm
OBFRQ 125.40 MHz
OBSET 140.00 KHz
OBFIN 3041.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 62
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.8 us
IRNUC 1H
CTEMP 23.4 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 29

141.0407
128.5721
128.2507
126.1739
84.1449
77.2555
77.0000
76.7445
71.5033
41.4896
29.7874
24.4060

F:\2005\08\050803-1-13C.als
1H Line

DFILE F:\2005\08\050803-1-13C.als
COMNT 1H Line
DATIM Wed Aug 3 19:22:17 2005
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 120.00 KHz
OBFIN 7958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 100
AQTM 0.967 sec
PD 2.033 sec
PW1 6.2 us
IRNUC 1H
CTEMP 27.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 31

61-19

Date : Tue Jul 4 19:36:17 2006

File Name : LoadingFID.hdata
 Comment : 1H Line
 Slice History :
 EXMODE : bcm

POINT : 32768 points
 SAMPO : 32768 points
 FREQU : 33898.3 Hz
 FILTR : 16950 Hz
 DELAY : 11.8 usec
 DEADT : 10.0 usec
 INTVL : 29.5 usec
 TIMES : 1000 times
 DUMMY : 1 times
 PD : 2.0333 sec
 ACQTM : 966.6560 msec
 PREDL : 0.01000 msec
 INIWT : 1000.0000 msec
 RESOL : 1.03 Hz
 PW1 : 6.25 usec
 13C :
 OBNUC : 125.65 MHz
 OBFRQ : 127958.00 Hz
 OBSET : 31
 RGAIN : 1H
 IRNUC : 500.00 MHz
 IRFRQ : 162160.00 Hz
 IRSET : 50.0 usec
 IRRPW : 0
 IRRNS :
 SCANS : 167 times
 SLVNT : CDCL3
 SPINNING : 14 Hz
 TEMP : 28.4 C

S-21

1H Line

170.287

77.255
77.000
76.753
74.656

47.593
46.467
27.778
26.684
26.635
26.569
26.413
23.237
21.436

200 150 100 50 0 ppm

Date : Fri May 5 16:48:49 2006

FileName : LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm

POINT 32768 points
SAMP0 32768 points
FREQ0 33898.3 Hz
FILTR 16950 Hz
DELAY 11.8 usec
DEADT 10.0 usec
INTVL 29.5 usec
TIMES 800 times
DUMMY 1 times
PD 2.0333 sec
ACQTM 966.6560 msec
PREDL 0.01000 msec
ININT 1000.0000 msec
RESOL 1.03 Hz
PW1 6.25 usec
OBNUC 13C
OBFRQ 125.65 MHz
OBSET 127958.00 Hz
RGAIN 30
IRNUC 1H
IRFRQ 500.00 MHz
IRSET 162160.00 Hz
IRRPW 50.0 usec
IRRS 0
SCANS 62 times
SLVNT CDCL3
SPINNING 12 Hz
TEMP 26.0 C

1H Line

Date : Sat Mar 18 18:03:56 2006

FileName : LoadingFID.nmdata
 Comment : 1H Line
 SliceHistory :
 EXMODE : bcm

POINT 32768 points
 SAMPO 32768 points
 FREQU 33898.3 Hz
 FILTR 16950 Hz
 DELAY 11.8 usec
 DEAT 10.0 usec
 INTVL 29.5 usec
 TIMES 60000 times
 DUMMY 1 times
 PD 2.0333 sec
 ACQTM 966.6560 msec
 PREDL 0.01000 msec
 INIWT 1000.0000 msec
 RESOL 1.03 Hz
 PW1 6.25 usec
 OBNUC 13C
 OBFRQ 125.65 MHz
 OBSET 127958.00 Hz
 RGAIN 30
 TRNUC 1H
 TRFRQ 500.00 MHz
 IRSET 162160.00 Hz
 IRRPW 50.0 usec
 IRRNS 0

SCANS 117 times

SLVNT COCL3
 SPINNING 11 Hz
 TEMP 25.9 C

SH-10-077 13C

Date : Mon Jun 26 21:31:11 2006

File Name : LoadingFID.nmdata
Comment : SH-10-077 13C
Slice History :
EXMODE : bcm

POINT 32768 points
SAMPO 32768 points
FREQU 33898.3 Hz
FILTR 16950 Hz
DELAY 11.8 usec
DEADT 10.0 usec
INTVL 29.5 usec
TIMES 512 times
DUMMY 1 times
PD 2.5000 sec
ACQTM 956.6560 msec
PREDL 0.01000 msec
ININT 1000.0000 msec
RESOL 1.03 Hz
PW1 6.25 usec
OBNUC 13C
OBFRQ 125.65 MHz
OBSET 127958.00 Hz
RGAIN 31
IRNUC 1H
IRFRQ 500.00 MHz
IRSET 162160.00 Hz
IRRPW 50.0 usec
IRRNS 0
SCANS 195 times
SLVNT CDCL3
SPINNING 11 Hz
TEMP 28.4 C

S-23

DFILE C:\Documents and Settings\NMRmanager\
COMNT SH-10-089_13C
DATIM Mon Jul 3 17:00:59 2006
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 120.00 KHz
OBFIN 7958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 192
ACQTM 0.957 sec
PD 3.000 sec
PW1 6.2 us
IRNUC 1H
CTEMP 28.2 c
SLVNT CD3OD
EXREF 49.00 ppm
BF 0.12 Hz
RGAIN 30

F:\2005\11\051103-1-1H.als
cyclohexyl methyl 1H

DFile F:\2005\11\051103-1-1H.als
COMNT Cyclohexyl methyl 1H
DATIM Thu Nov 3 16:13:11 2005
OBNUC 1H
EXMOD non

OBFRQ 500.00 MHz
OBSET 160.00 KHz
OBFIN 2160.0 Hz
POINT 32768
FREQU 10000.0 Hz
SCANS 8
ACQTM 3.277 sec
PD 3.723 sec
PWL 6.0 us
IRNUC 1H
CTEMP 27.0 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 20

S-25

F:\2005\11\051123-1-1H.als
1 1H

DEFILE F:\2005\11\051123-1-1H.als
COMNT 1 1H
DATIM Wed Nov 23 17:43:24 2005
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 160.00 KHz
OBFIN 2160.0 Hz
POINT 32768
FREQU 10000.0 Hz
SCANS 12
ACQTM 3.277 sec
PD 3.723 sec
PW1 6.0 us
IRNUC 1H
CTEMP 25.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 18

S-26

DFILE F:\2005\11\051116-1-1H.als
COMNT 1 1H
DATIM Wed Nov 16 17:42:22 2005
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 160.00 KHz
OBFIN 2160.0 Hz
POINT 32768
FREQU 10000.0 Hz
SCANS 8
ACQTM 3.277 sec
PD 3.723 sec
PW1 6.0 us
IRNUC 1H
CTEMP 26.9 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 21

S-27

S-28

DFILE 0.9559
 COMNT 0.9949
 DATIM 1.0089
 OBNUC 1H 1.0157
 EXMOD non 1.0273
 OBFRQ 500.00 MHz 1.0340
 OBSET 160.00 KHz 1.1206
 OBFIN 2160.0 Hz 1.1279
 POINT 32768 1.1346
 FREQU 10000.0 Hz 1.1456
 SCANS 8 1.1523
 ACQTM 3.277 sec 1.1590
 PD 3.723 sec 1.1731
 PW1 6.0 us 1.1767
 IRNUC 1H 1.1847
 CTEMP 26.9 c 1.2225
 SLVNT CDCL3 1.2298
 EXREF 7.26 ppm 1.2408
 BF 0.12 Hz 1.2481
 RGAIN 19 1.2555
 1.2622
 1.2731
 1.2805
 1.2878
 1.2982
 1.3055
 1.3195
 1.4428
 1.4525
 1.4617
 1.4757
 1.4818
 1.4910
 1.5569
 1.6179
 1.6423
 1.6722
 1.6984
 1.7057
 1.7448
 1.8015
 1.8272
 2.8016
 4.3770
 4.4008
 4.4405
 4.4643
 7.2600

F:\2005\06\050606-1.als
1H Line

DEFILE F:\2005\06\050606-1.als
COMNT 1H Line
DATIM Mon Jun 6 18:09:02 2005
OBNUC 1H
EXMOD non
OBFRQ 499.10 MHz
OBSET 120.00 KHz
OBFIN 8250.0 Hz
POINT 32768
FREQU 9980.0 Hz
SCANS 8
ACQTM 3.283 sec
PD 3.717 sec
PW1 5.8 us
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 17

7.3541
7.3510
7.3431
7.3394
7.3272
7.3242
7.2973
7.2644
7.2620
7.2492
7.2424
7.2400
7.2260
7.2247

4.5520
4.5282
4.5154
4.4946
4.4709

3.0207
2.9987
2.8096
2.7986
2.7925
2.7852
2.7748
1.9354
1.9256
1.9177
1.9116
1.9012
1.6926
1.4479
1.4272

5-29

D:\2005\08\050803-1.als
1H Line

D:\2005\08\050803-1.als
COMET 1H Line
DATIM Wed Aug 3 19:15:57 2005
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 160.00 KHz
OBFIN 2160.0 Hz
POINT 32768
FREQU 10000.0 Hz
SCANS 8
AQTM 3.277 sec
PD 3.723 sec
PWL 6.0 us
IRNUC 1H
CTEMP 26.8 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 19

S-30

F:\2006\02\060223-1-1H.als
iBu Et

7.2600

DFILE
CONNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

F:\2006\02\060223-1-1H.als
iBu Et
Thu Feb 23 17:13:25 2006
1H
non
500.00 MHz
160.00 KHz
2160.0 Hz
32768
10000.0 Hz
8
3.277 sec
3.723 sec
5.7 us
1H
31.5 c
CDCL3
7.26 ppm
0.12 Hz
20

4.4521
4.4502
2.7290
1.8400
1.8290
1.8272
1.8162
1.8137
1.8028
1.8003
1.7893
1.6704
1.6594
1.6551
1.6447
1.6398
1.6295
1.6246
1.6148
1.5203
1.5093
1.4910
1.4800
1.4300
1.4165
1.4007
1.3872
1.0004
0.9870
0.9717
0.9638
0.9485
0.9333

S-31

1H Line

Date : Fri May 5 16:45:04 2006
File Name : LoadingFID.nmdata
Comment : 1H Line
Slice History : non
EXMODE : non
POINT : 32768 points
SAMP : 32768 points
FREQ : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 57.1 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTH : 3276.7998 msec
PREDL : 0.01000 msec
ININT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 5.70 usec
OBNUC : 1H
OBFRG : 500.00 MHz
OBSET : 162150.00 Hz
RGAIN : 17
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 25.3 C

1H Line

Date : Sat Mar 18 18:07:28 2006
 FileName : LoadingFID.nmdata
 Comment : 1H Line
 SliceHistory :
 EXMODE : non

POINT 32768 points
 SAMPD 32768 points
 FREQD 10000.0 Hz
 FILTR 5000 Hz
 DELAY 40.0 usec
 DEADT 56.9 usec
 INTVL 100.0 usec
 TIMES 8 times
 DUMMY 1 times
 PD 2.0333 sec
 ACQTM 3276.7998 msec
 PREDL 0.01000 msec
 INIWT 1000.0000 msec
 RESOL 0.31 Hz
 PW1 6.25 usec
 OBNUC 1H
 OBFRO 500.00 MHz
 OBSET 162160.00 Hz
 RGAIN 14

SCANS 6 times
 SLVNT COCL3
 SPINNING 13 Hz
 TEMP 25.9 C

SH-10-077

Date : Mon Jun 26 21:19:01 2005

FileName : .LoadingFID.nmdata
Comment : SH-10-077

SliceHistory

EXMODE

7.8067
7.8036
7.7994
7.7933
7.7902
7.7878
7.4919
7.4882
7.4742
7.4711
7.4620
7.4595
7.4504
7.4473
7.4449
7.4333
7.2716
7.2600
7.2563
7.2411
7.1673
7.1612
7.1563
7.1514
7.1447
7.1422
6.5736
2.7070
2.6137
2.6039
2.2024
2.1920
2.1774
2.1200
2.1103
2.0950
2.0853
1.7729
1.5758
1.2646
1.0083
1.0016
0.9925
0.9858
0.9791
0.9699
0.9632
0.9697
0.6660
0.6544
0.6502
0.6392
0.6337
0.6233
0.6172

POINT 32768 points
SAMP0 32768 points
FREQU 10000.0 Hz
FILTR 5000 Hz
DELAY 40.0 usec
DEAOT 57.1 usec
INTVL 100.0 usec
TIMES 128 times
DUMMY 1 times
PD 3.7232 sec
ACQTM 3276.7998 msec
PREDL 0.01000 msec
INIWT 1000.0000 msec
RESOL 0.31 Hz
PW1 5.70 usec
OBNUC ¹H
OBFRQ 500.00 MHz
OBSE1 162160.00 Hz
RGAIN 18
SCANS 6 times
SLVNT CDCL3
SPINNING 11 Hz
TEMP 26.9 C

ppm

S-34

D:\2005\11\051129-1.als
1H Line

DFILE
COMNT 1H Line
DATIM Tue Nov 29 19:51:26 2005
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 160.00 KHz
OBFIN 2160.0 Hz
POINT 32768
FREQU 10000.0 Hz
SCANS 16
ACQTM 3.277 sec
PD 3.723 sec
PW1 6.0 us
IRNUC 1H
CTEMP 26.4 C
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 23