The Enantioselective Aldol Reaction of Trimethoxysilyl Enol Ether Catalyzed by Lithium Binaphtholate

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SUPPLEMENTARY MATERIAL

General Experimental

Melting points were measured using a Büchi 535 melting point apparatus and were not corrected. Optical rotations were obtained on a JASCO DIP-370 digital polarimeter. Infrared spectra were recorded on a JASCO FT/IR-5300. ¹H NMR and ¹³C NMR spectra were recorded on a JEOL EX-270 (¹H, 270 MHz; ¹³C, 68 MHz) spectrometer in deuteriochloroform. Chemical shift values are expressed in ppm relative to internal tetramethylsilane. Coupling constants (*J*) are reported in Hertz (Hz). Abbreviations are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. Mass spectra were obtained by electron impact with ionization voltage of 70 eV on JEOL JMS-DX303. HPLC was performed on a JASCO PU-1580 with a JASCO UV-1575 (λ = 254 nm) and chiral separations were performed using Daicel chiralpak or chiralcel columns (ϕ 0.46 x 25 cm). HPLC peaks of *syn* / *anti* isomers were assigned by the comparison with that of authentic samples prepared with racemic BINOL as a catalyst. Relative configurations of new compounds were assigned on the basis of the splitting pattern of the hydroxyl bearing methine.¹

3,3'-Disubstituted binaphthols were prepared by the literature methods.² Trimethoxysilyl enol ethers **2a**, **b**, **f** were prepared by the literature methods.³

Preparation of 3,3'-Bis(trifluoromethyl)-1,1'-binaphthalene-2,2'-diol.^{2a}

To a double-necked 20 mL round-bottomed flask equipped with a magnetic stirring bar, septum, and dry-ice condenser containing activated cadmium (675 mg, 6 mmol) was added DMF (2.5 mL) under argon atmosphere. Dibromodifluoromethane (0.28 mL, 3 mmol) was added to the mixture at 0 °C and the whole was immediately warmed to room temperature. After stirring for 2 h, the suspension was filtered with glass filter under argon pressure and the precipitate was washed

with DMF (0.5 mL). To the resulting brown solution was added HMPA (3.0 mL) followed by cuprous bromide (215 mg, 1.5 mmol) in one portion at 0 °C. After stirring for a few minutes, 3,3'-diiodo-2,2'-bis(methoxymethoxy)-1,1'-binaphthyl (62.6 mg, 0.1 mmol) was added in one portion and then the reaction mixture was heated to 70 °C for 6 h. Benzene (10 mL) and H₂O (5 mL) was added to the reaction mixture and the mixture was stirred vigorously at room temperature for 12 h. After removal of the precipitate by celite filtration, the organic layer was washed with H₂O and brine, dried over Na₂SO₄, and evaporated. The brown residue was purified with silica gel column chromatography (hexane/benzene, 1/1). Dichloromethane (2 mL), methanol (2 mL), and conc. HCl (0.3 mL) were added to the resulting yellow oil and the mixture was stirred overnight at room temperature. Then the organic layer was washed with sat. NaHCO₃ and brine, dried over Na₂SO₄, and evaporated. Purification with silica gel column chromatography (hexane/AcOEt, 20/1) afforded 3,3'-bis(trifluoromethyl)-2,2'-dihydroxy-1,1'-binaphthyl (34.0 mg, 81% yield) as pale yellow prisms. ¹H NMR δ 5.35 (br, 2H, OH), 7.10 (d, 2H, J = 7.3 Hz, Ar-H), 7.44 (m, 4H, Ar-H), 7.86 (d, 2H, J = 7.3 Hz, Ar-H), 8.36 (s, 2H, Ar-H)

IR (KBr) 3549, 3063, 1630, 1332, 1207, 1155 cm⁻¹

[α]_D²³ +657 (c 1.01, THF)

mp 235-237 °C.

LR-FABMS 422 (M⁺), 154, 136. HR-FABMS calcd. 422.0742, found. 422.0752.

Anal. calcd for C, 62.57; H, 2.86 found C, 62.18; H, 2.96.

Preparation of 1-Trimethoxysilyloxy-3*H*-indene (2c).

To a solution of diisopropylamine (2.9 g, 28.8 mmol, 1.15 eq) in THF (50 mL) was added *n*-butyllithium in hexane (1.6 M, 17.2 mL, 27.5 mmol, 1.1 eq) at -78 °C. After stirring for 15 min, a solution of 1-indanone (3.3 g, 25 mmol) in THF (5 mL) was added dropwise to the pale yellow mixture via cannula over 30-min period. After additional stirring for 15 min, chlorotrimethoxysilane (4.31 g, 27.5 mmol, 1.1 eq) was added dropwise. After being stirred for 30 min, the reaction mixture was allowed to warm to room temperature. The mixture was evaporated and the resulting white precipitate was removed by celite filtration and washed with hexane. Then ice-cooled sat. NaHCO₃ (30 mL) was added to the filtrate and the mixture was stirred vigorously. After removal of cloudy

material by celite filtration, the organic layer was dried over Na₂SO₄ and evaporated to give the crude product as brown oil. Distillation *in vacuo* (125 °C, 4 mmHg) gave the silyl enol ether (3.9 g, 62% yield) as a colorless oil.

¹H NMR δ 3.27 (d, 2H, *J* = 2.5, benzyl-CH₂), 3.65 (s, 9H, SiOCH₃), 5.67 (t, 1H, *J* = 2.5, olefin-*H*), 7.1-7.5 (m, 4H, Ar-*H*).

¹³C NMR δ 33.8, 51.5, 107.3, 118.0, 123.8, 125.2, 126.0, 140.5, 142.6, 151.6.

IR (neat) 2948, 2847, 1605, 1578, 1366, 1181, 1082, 901, 837 cm⁻¹.

LR-EIMS 252 (M⁺), 236, 121, 91. HR-EIMS calcd for C₁₂H₁₆O₄Si 252.0818, found 252.0806.

1-Trimethoxysilyloxy-1-phenylethylene (2d).

bp 122-124 °C / 14 mmHg.

¹H NMR δ 3.62 (s, 9H, SiOCH₃), 4.72 (d, 1H, *J* = 2.2, olefin-*H*), 5.00 (d, 1H, *J* = 2.2, olefin-*H*), 7.34-7.28 (m, 3H, Ar-*H*), 7.65-7.60 (m, 2H, Ar-*H*).

¹³C NMR δ 51.6, 92.0, 125.1, 128.2, 128.4, 136.3, 153.8.

IR (neat) 2945, 2840, 1692, 1621, 1573, 1492, 1317, 1201, 1096, 1037, 828, 775, 743, 715 cm⁻¹.

LR-EIMS 241 ((M+H)⁺), 120, 105, 77. HR-EIMS calcd for C₁₁H₁₆O₄Si 241.0896, found 241.0892.

(Z)-1-Trimethoxysilyloxy-1-phenylpropene (2e).

bp 116 °C / 11 mmHg.

¹H NMR δ 1.80 (d, 3H, *J* = 6.5, C=CH-CH₃), 3.49 (s, 9H, SiOCH₃), 5.37 (q, 1H, *J* = 6.5, olefin-*H*), 7.32-7.18 (m, 3H, Ar-*H*), 7.51 (d, 2H, Ar-*H*).

¹³C NMR δ 11.0, 51.4, 105.8, 125.0, 127.4, 128.2, 137.9, 148.0.

IR (neat) 3057, 2947, 2847, 2361, 1948, 1690, 1659, 1599, 1495, 1447, 1381, 1323, 1267, 1196, 1100, 1032 cm⁻¹.

LR-EIMS 254 (M⁺), 225, 121, 91. HR-EIMS calcd for C₁₂H₁₇O₄Si 254.0974, found 254.0966.

General Procedure of Enantioselective Aldol Reaction: Preparation of 2,3-Dihydro-2-(hydroxycyclohexylmethyl)-1*H*-inden-1-one (3i).

To a stirred solution of (*R*)-3,3'-dibromo-1,1'-binaphthalene-2,2'-diol (21.0 mg, 0.047 mmol, 0.1 eq) in THF (3 mL) was added H₂O in THF (2.8 M, 0.25 mL, 0.70 mmol, 1.5 eq) and *n*-butyllithium in hexane (0.16 M, 0.6 mL, 0.094 mmol, 0.2 eq) at -23 °C, and the mixture was stirred

for a few minutes. To this yellow mixture, a solution of cyclohexanecarbaldehyde in THF (1.0 M, 0.5 mL, 0.47 mmol) and 1-trimethoxysilyloxy-3*H*-indene (0.17 mL, 0.70 mmol, 1.5 eq) was added. The mixture was stirred for 0.5 h at the same temperature and the reaction was quenched with KF/KH₂PO₄ aq (15% KF, 10% KH₂PO₄ solution, 2 mL). The mixture was diluted with AcOEt (10 mL) and washed with brine for 3 times. The organic layer was dried over Na₂SO₄, and then evaporated. The crude product was purified by silica gel column chromatography (CH₂Cl₂/hexane 8/1, then CH₂Cl₂/AcOEt 160/1→10/1) to give *syn* / *anti* mixture (104.7 mg, 93% yield) as white needles.

¹H NMR δ 0.9-1.4 (m, 8H), 1.5-1.9 (m, 12H), 2.0-2.1 (m, 2H), 2.8-2.9 (m, 2H), 3.0-3.3 (m, 3H), 3.66 (d, 1H, *J* = 9.2 Hz, *HC*-OH *anti*), 4.10 (d, 1H, *J* = 7.3 Hz, *HC*-OH *syn*), 4.2-4.3 (m, 1H), 7.3-7.8 (m, 8H).

¹³C NMR δ 25.6, 25.8, 26.1, 26.2, 26.4, 26.6, 26.7, 29.3, 29.5, 29.8, 30.1, 41.5, 41.9, 49.3, 50.8, 74.9, 76.4, 123.5, 123.8, 126.4, 126.5, 127.0, 127.5, 134.7, 135.2, 136.5, 137.2, 153.8, 154.9, 209.0, 211.0 (*syn/anti* mixture).

[α]_D²⁵ +46.8 (*c* 1.17, CHCl₃) (*syn* : *anti* = 1.4 : 1)

IR (KBr) 3409, 2924, 2847, 1678, 1605, 1466, 1296, 1084, 1024, 960.

LR-EIMS 244 (M⁺), 161, 132. HR-EIMS calcd for C₁₆H₂₀O₂ 244.1463, found 244.1455.

HPLC (Daicel chiralpak AD-H, Hex/IPA = 19/1, 1.0 mL/min): *t*_R 15.4 (*anti*-minor), 19.4 (*anti*-major), 22.4 (*syn*-major), 28.4 min (*syn*-minor).

2-(Hydroxyphenylmethyl)cyclohexanone (3a).³

¹H NMR δ 4.79 (d, 1H, *J* = 8.9 Hz, *HC*-OH *anti*), 5.40 (d, 1H, *J* = 2.7 Hz, *HC*-OH *syn*).

[α]_D²⁵ +78.4 (*c* 0.99, CHCl₃) (*syn* : *anti* = 3.1 : 1)

HPLC (Daicel chiralcel OD-H, Hex/IPA = 19/1, 1.0 mL/min): *t*_R 9.4 (*syn*-minor, 2*S*, 1'*S*), 10.6 (*syn*-major, 2*R*, 1'*R*), 12.5 (*anti*-major, 2*S*, 1'*R*), 18.5 min (*anti*-minor, 2*R*, 1'*S*).

2-(Hydroxyphenylmethyl)cyclopentanone (3b).³

¹H NMR δ 4.69 (d, 1H, *J* = 9.5 Hz, *HC*-OH *anti*), 5.29 (t, 1H, *J* = 4.0 Hz, *HC*-OH *syn*).

[α]_D²⁵ +118 (*c* 1.18, CHCl₃) (*syn* : *anti* = 2.9 : 1)

HPLC (Daicel chiralcel OD-H, Hex/IPA = 9/1, 1.0 mL/min): t_R 7.9 (*syn*-minor), 9.5 (*syn*-major), 11.5 (*anti*-minor), 13.7 min (*anti*-major).

2,3-Dihydro-2-(hydroxyphenylmethyl)-1*H*-inden-1-one (3c).⁴

¹H NMR δ 3.85 (m, 1H, HC-OH *anti*), 4.34 (m, 1H, HC-OH *syn*).

¹³C NMR δ 27, 55, 72, 124, 126, 127, 129, 135, 137, 143, 155, 207 (*syn*); 30, 53, 76, 124, 126, 127, 128, 135, 136, 141, 154, 210(*anti*).

$[\alpha]_D^{25} +108$ (*c* 1.01, CHCl₃) (*syn* : *anti* = 2.9 : 1)

IR (KBr) 3553, 3449, 3055, 2910, 1703, 1605, 1462, 1449, 1329, 1294, 1280, 1095, 765.

LR-EIMS 238 (M⁺), 219, 132. HR-EIMS calcd for C₁₆H₁₄O₂ 238.0994, found 238.0990.

HPLC (Daicel chiralpak AS-H, Hex/IPA = 9/1, 1.0 mL/min): t_R 15.4 (*anti*-minor), 16.9 (*anti*-major), 22.1 (*syn*-minor), 34.2 min (*syn*-major).

3-Hydroxy-1,3-diphenyl-1-propanone (3d).⁵

¹H NMR δ 3.38 (d, 2H, *J*=6.1, CH₂), 7.6-7.3 (m, 8H, Ar-*H*), 5.36 (t, 1H, *J*=6.1, HC-OH), 8.0-7.9 (m, 2H, Ar-*H*).

$[\alpha]_D^{25} +54.6$ (*c* 1.50, CHCl₃)

HPLC (Daicel chiralcel OB-H, Hex/IPA = 9/1, 1.0 mL/min) t_R 17.8 min (*R*), 25.8 min (*S*).

3-Hydroxy-2-methyl-1,3-diphenyl-1-propanone (3e).¹

¹H NMR δ 4.95 (m, 1H, HC-OH *anti*), 5.23 (s, 1H, HC-OH *syn*).

$[\alpha]_D^{25} +16.3$ (*c* 1.13, CHCl₃) (*syn* : *anti* = 1.9 : 1)

HPLC (Daicel chiralcel OD-H, Hex/IPA = 19/1, 1.0 mL/min): t_R 10.5 (*syn*-major), 12.3 (*syn*-minor), 15.2 (*anti*-minor), 16.9 min (*anti*-major).

2,3-Dihydro-2-[hydroxy-(4-methoxyphenyl)methyl]-1*H*-inden-1-one (3f).

¹H NMR δ 2.6-2.7 (m, 1H), 2.8-3.0 (m, 4H), 3.2-3.3 (m, 1H), 3.77 (s, 6H, OCH₃), 4.78 (d, 1H, *J* = 19.1 Hz, HC-OH *anti*), 5.51 (s, 1H, HC-OH *syn*), 6.8-6.9 (m, 4H), 7.3-7.8 (m, 12H).

¹³C NMR δ 26.7, 29.8, 53.1, 54.7, 55.1, 71.4, 75.0, 76.6, 113.6, 113.8, 123.7, 124.0, 126.4, 126.5,

126.6, 127.1, 127.5, 128.1, 133.4, 134.8, 135.3, 136.2, 136.9, 153.9, 154.8, 158.6, 159.3, 207.5, 209.6 (*syn/anti* mixture).

$[\alpha]_D^{25} +143$ (*c* 1.23, CHCl₃) (*syn* : *anti* = 7.0 : 1)

IR (KBr) 3493, 1692, 1609, 1512, 1298, 1256, 1092, 1028, 853.

LR-EIMS 268 (M⁺), 132. HR-EIMS calcd for C₁₇H₁₆O₃ 268.1099, found 268.1102.

HPLC (Daicel chiralpak AS-H, Hex/IPA = 3/1, 1.0 mL/min): *t*_R 10.2 (*anti*-minor), 13.7 (*anti*-major), 17.6 (*syn*-major), 30.2 min (*syn*-minor).

2,3-Dihydro-2-(1-hydroxy-3-phenyl-2-propenyl)-1*H*-inden-1-one (3g).

¹H NMR δ 2.8-3.0 (m, 2H), 3.1-3.3 (m, 4H), 4.52 (t, 1H, *J* = 6.5 Hz, HC-OH *anti*), 5.02 (m, 1H, HC-OH *syn*), 6.2-6.3 (m, 2H), 6.7-6.8 (m, 2H), 7.2-7.8 (m, 18H).

¹³C NMR δ 27.3, 29.6, 52.0, 52.6, 71.6, 74.0, 123.8, 124.1, 126.4, 126.5, 126.6, 127.3, 127.6, 127.7, 127.8, 128.5, 128.7, 129.4, 131.0, 132.2, 135.0, 135.4, 136.3, 136.3, 136.4, 137.0, 154.0, 154.8, 207.8, 209.0 (*syn/anti* mixture).

$[\alpha]_D^{25} +82.2$ (*c* 0.96, CHCl₃) (*syn* : *anti* = 3.4 : 1)

IR (KBr) 3478, 1698, 1605, 1464, 1329, 1296, 1205, 963, 765, 705.

LR-EIMS 264 (M⁺), 246, 132. HR-EIMS calcd for C₁₈H₁₆O₂ 264.1150, found 264.1158.

HPLC (Daicel chiralpak AS-H, Hex/IPA = 9/1, 1.0 mL/min): *t*_R 22.1 (*anti*-major), 26.8 (*anti*-minor), 31.8 (*syn*-minor), 37.2 min (*syn*-major).

2,3-Dihydro-2-(1-hydroxy-3-phenylpropyl)-1*H*-inden-1-one (3h).

¹H NMR δ 1.7-2.0 (m, 4H), 2.4-2.9 (m, 8H), 3.1-3.3 (m, 2H), 3.84 (m, 1H, HC-OH *anti*), 4.38 (m, 1H, HC-OH *syn*), 7.1-7.7 (m, 18H).

¹³C NMR δ 27.2, 29.6, 29.7, 31.3, 32.4, 36.6, 37.3, 51.9, 53.1, 70.2, 71.7, 123.6, 123.9, 125.8, 125.9, 126.4, 126.5, 127.2, 127.6, 128.3, 128.4, 128.5, 128.7, 134.9, 135.3, 136.4, 137.0, 141.7, 142.0, 153.7, 154.7, 208.4, 209.9 (*syn/anti* mixture).

$[\alpha]_D^{25} +39.6$ (*c* 0.93, CHCl₃) (*syn* : *anti* = 2.6 : 1)

IR (KBr) 3486, 1698, 1605, 1464, 1329, 1296, 1209, 1039, 770.

LR-EIMS 266 (M⁺), 248, 161, 132. HR-EIMS calcd for C₁₈H₁₈O₂ 266.1306, found 266.1299.

HPLC (Daicel chiralpak AS-H, Hex/IPA = 9/1, 1.0 mL/min) t_R 15.4 (*anti*-minor), 16.9 (*anti*-major), 22.1 (*syn*-minor), 34.2 min (*syn*-major).

2-(1-Hydroxy-3-phenylpropyl)cyclohexanone (3j).¹

¹H NMR δ 3.73 (m, 1H, HC-OH *syn*), 4.10 (m, 1H, HC-OH *anti*).

$[\alpha]_D^{25} +11.9$ (c 0.77, CHCl₃) (*syn* : *anti* = 1.2 : 1)

HPLC (Daicel chiralcel OJ-H, Hex/IPA = 40/1, 1.0 mL/min): t_R 16.1 (*syn*-major), 20.5 (*syn*-minor), 23.2 (*anti*-major), 24.8 min (*anti*-minor).

References

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C-R&A CHROMATOPAC CH=1 Report No.=4 DATA=1:CHRM1.C01 04/02/27 22:08:44

**** CALCULATION REPORT ****

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	5	9.614	174548	8967			7.4835	
	6	10.697	1595099	66391	V		68.3874	
	7	12.752	422375	15469			18.1087	
	8	19.095	140423	2945			6.0204	
TOTAL			2332446	93772			100

3b

C-RSA CHROMATOPAC CH=1 Report No.=3 DATA=1:@CHRM1.C01 03/10/16 15:17:00

**** CALCULATION REPORT ****

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	5	7.855	389667	23894	V		10.3936	
	6	9.487	2407941	113460			64.227	
	7	11.548	447828	18960			11.9449	
	8	13.717	503671	15046			13.4344	
TOTAL			3749107	171359			100

3c

C-R8A CHROMATOPAC CH=1 Report No.=9 DATA=1:@CHRM1.C05 03/10/14 16:34:44

**** CALCULATION REPORT ****

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1	13	19.842	515339	16179			6.708	
	15	23.402	1467740	36937			19.105	
	16	27.768	5206017	91841			67.7647	
	17	31.817	493393	6232	V		6.4223	
TOTAL			7682487	151190			100

3d

C-R8A CHROMATOPAC CH=1 Report No.=13 DATA=1:@CHRM1.C03 03/10/16 17:53:26

**** CALCULATION REPORT ****

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	3	20.254	13980988	279587	E		87.5519	
	4	30.804	1987818	29138	E		12.4481	
TOTAL			15968806	308725			100

C-R8A CHROMATOPAC CH=1 Report No.=4 DATA=1:@CHRM1.C01 04/04/14 17:42:36

**** CALCULATION REPORT ****

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	11	10.467	24703862	1001869	SVE		39.0451	
	13	12.328	16721897	622553	V		26.4294	
	14	15.156	6786804	208572	V		10.7267	
	15	16.917	15057448	395130	V		23.7987	
TOTAL			63270008	2228124			100

CH. 1 C.S 1.25 ATT 9 OFFS 0 00/00/00 01:29

FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC
2	10.19	331111	0.316	BB
3	13.71	1273287	12.157	BB
4	17.62	8671899	82.795	BB
5	30.22	495650	4.732	BB
TOTAL		10473947	100.000

C-R8A CHROMATOPAC CH=1 Report No.=3 DATA=1:@CHRM1.C01 03/12/09 21:25:50

**** CALCULATION REPORT ****

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	15	22.069	1801301	46590	SV		12.559	
	17	26.762	1488642	30462			10.3791	
	18	31.781	1512782	13258			10.5474	
	19	37.179	9539932	116321	V		66.5144	
TOTAL			14342656	206631			100

C-RSA CHROMATOPAC CH=1 Report No.=9 DATA=1:@CHRM1.C04 03/12/03 16:23:30

**** CALCULATION REPORT ****

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	10	15.394	1199427	48586			7.346	
	11	16.926	3323069	112016	SV		20.3525	
	15	22.051	492997	8453	V		3.0194	
	18	34.221	11312108	100488			69.2821	
TOTAL			16327600	269543			100

C-RSA CHROMATOPAC CH=1 Report No.=18 DATA=1:CHRM1.C05 04/03/27 00:41:00

** CALCULATION REPORT **

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1	1	15.402	197304	10611			3.5295	
	2	19.388	2185351	89898			39.0931	
	3	22.416	3160493	104236			56.5372	
	4	28.406	46964	1298			0.8401	
TOTAL			5590112	206043			100

3j

C-RSA CHROMATOPAC CH=1 Report No.=3 DATA=1:@CHRM1.C01 04/04/12 17:24:44

**** CALCULATION REPORT ****

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	10	12.429	1089909	33072			30.1455	
	11	15.309	471952	12773			13.0536	
	12	16.573	1962360	35705	V		54.2764	
	13	18.674	91277	2093	V		2.5246	
TOTAL			3615497	83643			100	