Supporting Information

Highly Enantio- and Diastereoselective Brassard Type Hetero-Diels-Alder Approach to 5-Methyl-containing α , β -unsaturated δ -lactones

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Table of contents for supporting information:

List of figures.....	S1
General Remarks for experimental	S3
Table 1. Initial study of Lewis acid effects	S3
Synthesis and characterization of 4a-m	S4-S13
References	S13
NMR spectra	S14-S38

List of figures

Figure 1. CD spectra of 4a	S 4
Figure 2. CD spectra of 4b	S 5
Figure 3. CD spectra of 4c	S 6
Figure 4. CD spectra of 4d	S 6
Figure 5. CD spectra of 4e	S 7
Figure 6. CD spectra of 4f	S 8

Figure 7. CD spectra of 4g	S 9
Figure 8. CD spectra of 4h	S 10
Figure 9. CD spectra of 4i	S 10
Figure 10. CD spectra of 4j	S 11
Figure 11. 300M ^1H NMR of compound 4a	S14
Figure 12. 75M ^{13}C NMR of compound 4a	S15
Figure 13. 300M ^1H NMR of compound 4b	S16
Figure 14. 75M ^{13}C NMR of compound 4b	S17
Figure 15. 300M ^1H NMR of compound 4c	S18
Figure 16. 75M ^{13}C NMR of compound 4c	S19
Figure 17. 300M ^1H NMR of compound 4d	S20
Figure 18. 75M ^{13}C NMR of compound 4d	S21
Figure 19. 300M ^1H NMR of compound 4e	S22
Figure 20. 75M ^{13}C NMR of compound 4e	S23
Figure 21. 300M ^1H NMR of compound 4f	S24
Figure 22. 75M ^{13}C NMR of compound 4f	S25
Figure 23. 300M ^1H NMR of compound 4g	S26
Figure 24. 75M ^{13}C NMR of compound 4g	S27
Figure 25. 300M ^1H NMR of compound 4h	S28
Figure 26. 75M ^{13}C NMR of compound 4h	S29
Figure 27. 300M ^1H NMR of compound 4i	S30
Figure 28. 75M ^{13}C NMR of compound 4i	S31
Figure 29. 300M ^1H NMR of compound 4j	S32
Figure 30. 75M ^{13}C NMR of compound 4j	S33
Figure 31. 300M ^1H NMR of compound 4k	S34
Figure 32. 75M ^{13}C NMR of compound 4k	S35

Figure 33. 300M ¹H NMR of compound **4l** S36

Figure 34. 75M ¹³C NMR of compound **4l** S37

Figure 35. 300M ¹H NMR of compound **4m** S38

General Remarks. Unless otherwise noted, all non-aqueous reactions were carried out under an atmosphere of dry nitrogen in dried glassware. Melting points (m.p.) were measured on electrothermal digital melting point apparatus and were uncorrected. Enantiomeric excesses (*ee*) and the ratio of *anti/syn* were determined by HPLC or GC using corresponding commercial chiral column as stated, the experimental procedures at 23 °C with UV detector at 254 nm. Optical rotations were reported as follows: $[\alpha]^{20}_D$ (c g/100 mL, solvent).

Brassard type diene **2b** was prepared from methyl 3-methoxypent-2-enoate according to a literature procedures.¹ Ligands were prepared according to literature procedures.² Liquid aldehydes were distilled in vacuo prior to use, and solid aldehydes were used directly without further purification. Racemic samples of **4a-m** were prepared with anhydrous ZnCl₂ as the catalyst.

Table 1. Preliminary trials of Lewis acid effects on the HDA reaction of benzaldehyde with Brassard type diene **1b**^a

Entry	Metal	Yield (%) ^b	<i>Anti/syn</i> ^c	<i>Ee</i> (%) ^c
2	Al(OC ₃ H ₇) ₃	7	76:24	0
3	Sc(OTf) ₃	20	55:45	0

4	Zn(OTf) ₂	24	76:24	0
5	Yb(OTf) ₃	15	27:73	2
6	Sn(OTf) ₂	15	77:23	0
7	Cu(OTf) ₂	13	67:33	53
7	Ce(OiPr) ₃	N.D.	-	-
8	La(OiPr) ₃	N.D.	-	-

^a All reactions were performed with benzaldehyde (0.25 mmol) and diene **1b** (0.3 mmol) in 1.0 ml CH₂Cl₂ at 0 °C for 72 h. Catalysts were composed of a 1.1:1 molar ratio of **2e** to metal, catalyst loading was 10%. ^b Isolated yield. ^c Diastereoselectivities and enantioselectivities were determined by HPLC, with a chiralpak AD-H column.

HPLC graphic of **4a**:

Figure1. CD spectra of **4a** (c = 0.5mg/ml, in methanol)

(5R, 6S)-6-(o-Tolyl)-4-methoxy-5-methyl-5,6-dihydropyran-2-one (4b): The *ee* and the ratio of *anti/syn* were determined by HPLC using a Chiralpak AD-H column (90/10 hexane/isopropanol; flow rate 1.0 mL/min; *t*_{major} = 11.5 min, *t*_{minor} = 10.9 min). Pale yellow liquid; [α]_D²⁰ = -50.0 (c = 0.290, CH₂Cl₂, 94% *ee*; 99:1 *anti/syn*). ¹H NMR (300MHz, CDCl₃): δ 7.36-7.18 (m, 4H), 5.30 (d, *J* = 9.7 Hz,

1H), 5.23(s, 1H), 3.77 (s, 3H), 3.03-2.93 (m, 1H), 2.38 (s, 3H), 1.06 (d, $J = 7.0$ Hz, 3H). ^{13}C NMR (75MHz, CDCl_3): δ 175.4, 166.6, 136.2, 135.4, 130.7, 128.5, 126.9, 126.2, 89.9, 80.0, 56.2, 37.6, 19.5, 12.5. HRMS: $(\text{M}+\text{H})^+$, calcd 233.1172, found 233.1173

Figure2. CD spectra of **4b** ($c = 0.5\text{mg/ml}$, in methanol)

(5R, 6S)-6-(p-Tolyl)-4-methoxy-5-methyl-5, 6-dihydropyran-2-one(4c): The *ee* and the ratio of *anti/syn* were determined by HPLC using a Chiralcel AD-H column (90/10 hexane/isopropanol; flow rate 1.0 mL/min; $t_{\text{major}} = 17.3$ min, $t_{\text{minor}} = 12.3$ min). White solid; m.p. 107-108°C; $[\alpha]_{\text{D}}^{20} = -87.3$ ($c = 0.110$, CH_2Cl_2 , 96% *ee*; 98:2 *anti/syn*). ^1H NMR (300MHz, CDCl_3): δ 7.23-7.16 (m, 4H), 5.19 (s, 1H), 4.98 (d, $J = 9.3$ Hz, 1H), 3.75 (s, 3H), 2.91-2.86 (m, 1H), 2.35 (s, 3H), 1.06 (d, $J = 7.0$ Hz, 3H). ^{13}C NMR (75MHz, CDCl_3): δ 175.3, 166.6, 138.6, 134.6, 129.2, 127.21, 89.9, 83.5, 56.2, 38.0, 21.2, 12.9. HRMS: $(\text{M}+\text{H})^+$, calcd 233.1172, found 233.1169.

Figure3. CD spectra of **4c** (c = 0.5mg/ml, in methanol)

(5R, 6S)-6-(4-Fluorophenyl)-4-methoxy-5-methyl-5, 6-dihydropyran-2-one (4d): The *ee* and the ratio of *anti/syn* were determined by HPLC using a Chiralpak AD-H column (90/10 hexane/isopropanol; flow rate 1.0 mL/min; $t_{\text{major}} = 18.7$ min, $t_{\text{minor}} = 13.6$ min). White solid; m.p. 78-79 °C; $[\alpha]_{\text{D}}^{20} = -66.2$ (c = 0.388, CH₂Cl₂, 97% *ee*; 97:3 *anti/syn*; 60% yield). ¹H NMR δ (300MHz, CDCl₃): 7.37-7.04 (m, 4H), 5.20 (d, *J* = 1.1 Hz, 1H), 4.98 (d, *J* = 9.6 Hz, 1H), 3.76 (s, 3H), 2.91-2.81 (m, 1H), 1.05 (d, *J* = 7.0 Hz, 3H). ¹³C NMR δ (75MHz, CDCl₃): 175.1, 166.3, 164.5, 161.2, 133.4, 129.1, 129.0, 115.7, 115.4, 89.9, 82.9, 56.3, 38.1, 12.5. HRMS: (M+H)⁺, calcd 237.0921, found 237.0923.

Figure4. CD spectra of **4d** (c = 0.5mg/ml, in methanol)

(5*R*, 6*S*)-6-(4-Chlorophenyl)-4-methoxy-5-methyl-5, 6-dihydropyran-2-one (4e): The *ee* and the ratio of *anti/syn* were determined by HPLC using a Chiralpak AD-H column (90/10 hexane/isopropanol; flow rate 1.0 mL/min; $t_{\text{major}} = 19.1$ min, $t_{\text{minor}} = 14.4$ min). White solid; m.p. 88-89 °C; $[\alpha]_{\text{D}}^{20} = -85.7$ ($c = 0.120$, CH_2Cl_2 , >94% *ee*; >95:5 *anti/syn*). ^1H NMR (300MHz, CDCl_3): δ 7.37-7.29 (m, 4H), 5.20 (s, 1H), 4.99 (d, $J = 9.3$ Hz, 1H), 3.76 (s, 3H), 2.89-2.80 (m, 1H), 1.07 (d, $J = 7.0$ Hz, 3H). ^{13}C NMR (75MHz, CDCl_3): δ 175.0, 166.1, 136.1, 134.6, 128.7, 128.5, 89.9, 82.8, 56.3, 38.1, 18.8. HRMS: $(\text{M}+\text{H})^+$, calcd 253.0626, found 253.0627.

Figure 5. CD spectra of **4e** ($c = 0.5\text{mg/ml}$, in methanol)

(5*R*, 6*S*)-6-(4-Bromophenyl)-4-methoxy-5-methyl-5, 6-dihydropyran-2-one (4f): The *ee* and the ratio of *anti/syn* were determined by HPLC using a Chiralpak AD-H column (90/10 hexane/isopropanol; flow rate 1.0 mL/min; $t_{\text{major}} = 21.2$ min, $t_{\text{minor}} = 15.5$ min). Pale yellow solid; m.p. 85-86 °C; $[\alpha]_{\text{D}}^{20} = -72.8$ ($c = 0.268$, CH_2Cl_2 , >91 % *ee*; >94:6 *anti/syn*). ^1H NMR (300MHz, CDCl_3): δ 7.54-7.22 (m, 4H), 5.19 (d, $J = 0.9$, 1H), 4.98 (d, $J = 9.4$ Hz, 1H), 3.76 (s, 3H), 2.89-2.79 (m, 1H), 1.07 (d, $J = 7.0$ Hz, 3H). ^{13}C NMR (75MHz, CDCl_3): δ 175.0, 136.7, 131.7, 128.8, 127.3, 122.8, 89.9, 82.8, 56.3, 38.0, 12.8. HRMS: $(\text{M}+\text{H})^+$, calcd 297.0121, found 297.0124.

Figure 1. Molecular structure of (5*S*,6*R*)-(-)-**4f** in the crystal.

Figure6. CD spectra of **4f** (*c* = 0.5mg/ml, in methanol)

(5*R*, 6*S*)-6-(3-Nitrophenyl)-4-methoxy-5-methyl-5, 6-dihydropyran-2-one (4g): The *ee* and the ratio of *anti/syn* were determined by HPLC using a Chiralpak AD-H column (90/10 hexane/isopropanol; flow rate 1.0 mL/min; $t_{\text{major}} = 37.5$ min, $t_{\text{minor}} = 25.4$ min). Pale yellow solid; m.p. 107-108°C; ^1H NMR (300MHz, CDCl_3): δ 8.25-8.23 (m, 2H), 7.75-7.73 (m, 1H), 7.63-7.56 (m, 1H), 5.23 (s, 1H), 5.13 (d, $J = 9.4$ Hz, 1H), 3.78 (s, 3H), 2.97-2.89 (m, 1H), 1.12 (d, $J = 7.0$ Hz, 3H). ^{13}C NMR (75MHz, CDCl_3): δ 174.7, 165.5, 148.2, 139.7, 133.3, 129.7, 123.8, 122.2, 89.9, 82.2, 56.5, 38.1, 12.7. HRMS: $(\text{M}+\text{H})^+$, calcd 264.0866, found 264.0868.

Figure7. CD spectra of **4g** ($c = 0.5\text{mg/ml}$, in methanol)

(5R, 6S)-6-(4-Nitrophenyl)-4-methoxy-5-methyl-5,6-dihydropyran-2-one (4h): The *ee* and the ratio of *anti/syn* were determined by HPLC using a Chiralpak AD-H column (90/10 hexane/isopropanol; flow rate 1.0 mL/min; $t_{\text{major}} = 54.9$ min, $t_{\text{minor}} = 38.0$ min). White solid; m.p. 140-141°C; ^1H NMR (300MHz, CDCl_3): δ 8.28-8.22 (m, 2H), 7.60-7.54 (m, 2H), 5.21 (d, $J = 0.9$ Hz, 1H), 5.14 (d, $J = 8.9$ Hz, 1H), 3.77 (s, 3H), 2.90-2.84 (m, 1H), 1.13 (d, $J = 7.0$ Hz, 3H). ^{13}C NMR (75MHz, CDCl_3): δ 174.7, 165.5, 148.1, 144.7, 128.0, 123.8, 89.9, 82.2, 56.5, 38.1, 13.0. HRMS: $(\text{M}+\text{Na})^+$, calcd 286.0686, found 286.0689.

Figure8. CD spectra of **4h** ($c = 0.5\text{mg/ml}$, in methanol)

(5R, 6S)-6-(Naphthalen-1-yl)-4-methoxy-5-methyl-5,6-dihydropyran-2-one (4i): The *ee* and the ratio of *anti/syn* were determined by HPLC using a Chiralpak AD-H column (90/10 hexane/isopropanol; flow rate 1.0 mL/min; $t_{\text{major}} = 16.3$ min, $t_{\text{minor}} = 18.4$ min). White solid; m.p. 89-90 °C; $[\alpha]_{\text{D}}^{20} = -55.5$ ($c = 0.550$, CH_2Cl_2 , 92% *ee*; 98:2 *anti/syn*). ^1H NMR (300MHz, CDCl_3): δ 8.07-7.84 (m, 3H), 7.54-7.43 (m, 4H), 5.81 (d, $J = 8.4$ Hz, 1H), 5.27 (s, 1H), 3.76 (d, $J = 3.5$, 3H), 3.25-3.16 (m, 1H), 1.16 (d, $J = 7.0$ Hz, 3H). ^{13}C NMR (75MHz, CDCl_3): δ 175.5, 166.5, 133.9, 132.9, 130.8, 129.4, 129.0, 126.5, 125.7, 125.4, 124.9, 123.2, 89.8, 80.9, 56.2, 37.6, 14.0. HRMS: $(\text{M}+\text{H})^+$, calcd 269.1172, found 269.1174

Figure9. CD spectra of **4i** ($c = 0.5\text{mg/ml}$, in methanol)

(5*R*, 6*S*)-6-(Naphthalen-2-yl)-4-methoxy-5-methyl-5, 6-dihydropyran-2-one (4j): The *ee* and the ratio of *anti/syn* were determined by HPLC using a Chiralpak AD-H column (90/10 hexane/isopropanol; flow rate 1.0 mL/min; $t_{\text{major}} = 26.8$ min, $t_{\text{minor}} = 18.6$ min). White solid; m.p. 129-130 °C; $[\alpha]_{\text{D}}^{20} = -79.5$ ($c = 0.176$, CH_2Cl_2 , >97% *ee*; >97:3 *anti/syn*). ^1H NMR (300MHz, CDCl_3): δ 7.89-7.81 (m, 4H), 7.54-7.47 (m, 3H), 5.24 (d, $J = 1.0$, 1H), 5.20 (d, $J = 9.1$ Hz, 1H), 3.77 (s, 3H), 3.06-2.96 (m, 1H), 1.12 (d, $J = 7.0$ Hz, 3H). ^{13}C NMR (75MHz, CDCl_3): δ 175.2, 166.4, 134.9, 133.4, 132.9, 128.5, 128.1, 127.7, 126.8, 126.5, 124.3, 90.0, 83.7, 56.3, 38.1, 13.0. HRMS: $(\text{M}+\text{H})^+$, calcd 269.1172, found 269.1172

Figure 10. CD spectra of **4j** ($c = 0.5\text{mg/ml}$, in methanol)

6-(Furan-2-yl)-4-methoxy-5-methyl-5, 6-dihydropyran-2-one (4k): The *ee* and the ratio of *anti/syn* were determined by HPLC using a Chiralpak OJ column (85/15 hexane/isopropanol; flow rate 1.0 mL/min; $t_{\text{major}} = 19.2$ min, $t_{\text{minor}} = 27.6$ min). Brown liquid; $[\alpha]_{\text{D}}^{20} = +26.8$ ($c = 0.142$, CH_2Cl_2 , 99% *ee*; 98:2 *anti/syn*;). ^1H NMR (300MHz, CDCl_3): δ 7.40 (m, 1H), 6.39-6.34 (m, 2H), 5.16 (s, 1H), 5.09 (d, $J = 8.0$ Hz, 1H), 3.76 (s, 3H), 3.14-3.04 (m, 1H), 1.19 (d, $J = 0.6$ Hz, 3H). ^{13}C NMR (75MHz, CDCl_3): δ 175.4, 165.9, 150.1, 142.9, 110.4, 109.6, 89.6, 76.4, 56.3, 35.3, 13.6. HRMS: $(\text{M}+\text{H})^+$, calcd 209.0808, found 209.0807.

(E)-4-Methoxy-5-methyl-6-(prop-1-enyl)-5, 6-dihydropyran-2-one (4l): The *ee* and the ratio of *anti/syn* were determined by GC using a Chiralsil DEX CB column (160 °C; $t_{\text{minor}} = 15.2$ min, $t_{\text{major}} = 15.5$ min). Yellow oil; ^1H NMR δ (300MHz, CDCl_3): 5.84-5.77 (m, 1H), 5.56-5.47 (m, 1H), 5.08 (s, 1H), 4.42 (t, $J = 15.2$ Hz, 1H), 3.70 (s, 3H), 2.53-2.43 (m, 1H), 1.72-1.69 (m, 3H), 1.14-1.08 (m, 3H). ^{13}C NMR (75MHz, CDCl_3): δ 175.3, 166.3, 131.8, 127.5, 89.6, 82.3, 56.0, 36.8, 17.6, 13.5. HRMS: $(\text{M}+\text{H})^+$, calcd 183.1016, found 183.1012

4-Methoxy-5, 6-dihydro-6-propyl-pyran-2-one (4m): The *ee* and the ratio of *anti/syn* were determined by GC using a Chiralsil DEX CB column (130 °C; $t_{\text{minor}} = 41.5$ min, $t_{\text{major}} = 43.3$ min). Yellow oil; ^1H NMR (300MHz, CDCl_3): δ 5.09 (s, 1H), 4.36-4.07 (m, 1H), 3.73 (s, 3H), 2.46-2.44 (m, 1H), 1.18 (d, $J = 7.0$ Hz, 3H), 0.98-0.91 (m, 7H)

References:

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- (2) (a) Jiang, Y.-Z.; Zhou, X.-G.; Hu, W.-H.; Wu, L.-J.; Mi, A.-Q. *Tetrahedron: Asymmetry* **1995**, 6, 405-408. (b) Cogan, D. A.; Liu, G.-C.; Kim, K.; Backes, B. J.; Ellman, J. A. *J. Am. Chem. Soc.* **1998**, 120, 8011-8019. (c) Dossetter, A. G.; Jamison, T. F.; Jacobsen, E. N. *Angew. Chem., Int. Ed.* **1999**, 38, 2398-2400.

Current Data Parameters
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PROCNO 1

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DE 6.00 usec
TE 300.0 K
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P4DR-358

PAUR-366

1409.470

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14.00-3.00

PAUR-372

PADR-377

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PROCNO 1

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200R 359

Current Data Parameters
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EXPNO 2
PROCNO 1

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***** CHANNEL f2 *****
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SF02 300.1312005 MHz

F2 - Processing parameters
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WDW EM
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1D NMR plot parameters
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FAUR-443