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A SHORT ACCESS TO CHIRAL NON-RACEMIC OXA- AND AZAHETEROCYCLES BY CROSS-METATHESIS AND PD-CATALYZED CYCLIZATION SEQUENCE

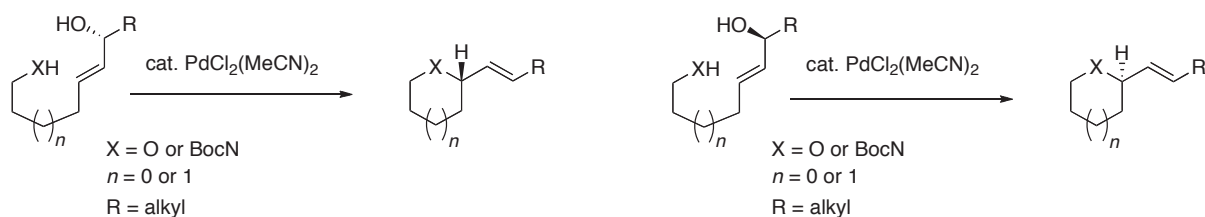
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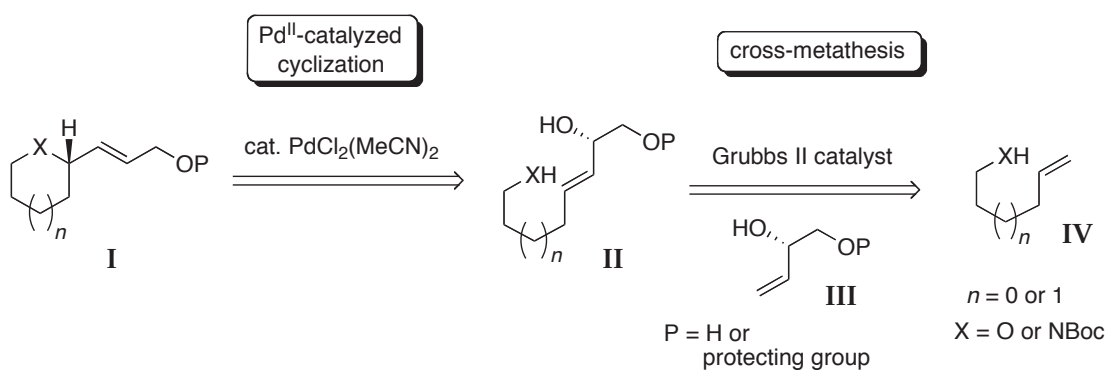
Abstract – A concise synthesis of chiral non-racemic 2-(3-benzoyloxyprop-1-enyl)tetrahydrofuran (**5a**), tetrahydropyran (**5b**), and piperidine (**8**) is described. Cross-metathesis of optically pure (*S*)-1-*O*-benzoyl-3-butene-1,2-diol (**2**) with protected 4-pentenol, 5-hexenol, and 5-hexenylamine gave the corresponding allyl alcohols (**3a**), (**3b**), and (**7**) in one step, respectively. PdCl₂(MeCN)₂ catalyzed cyclization of **4a**, **4b**, and **7** afforded **5a**, **5b**, and **8** in excellent yields with high enantiomeric purity.

Pd^{II}-catalyzed reactions are valuable in stereoselective organic synthesis.¹ We have recently reported that the Pd^{II}-catalyzed reaction of chiral non-racemic ζ-, ε-hydroxy, and ζ-*N*-Boc-amino allyl alcohol occurs to give substituted tetrahydrofurans, tetrahydropyrans and piperidines with high stereoselectivity through the 1,3-chirality transfer process.² The *syn* oxy- and azapalladations occur predominantly in intra- and intermolecular reactions,^{3,2d} and we have achieved the stereocontrolled synthesis of natural products, such as (-)-aspergillide B,^{4a} (-)-diospongine B,^{4b} (-)-laulimalide,^{4c} and (+)-coniine,^{2d} using this reaction.



Scheme 1

However, there were a few drawback using this synthesis. First, the substituent R group has been limited to alkyl groups so far. Second, chiral secondary allyl alcohol has to be prepared for every substrate. Therefore, flexible syntheses for various chiral non-racemic allyl alcohols are highly desired for the synthesis of chiral heterocycles.⁵ For this reason, we designed a new synthetic approach for the preparation of chiral non-racemic heterocyclic compound **I**, as shown in Scheme 2. A cross-metathesis of terminal alkene **IV** that has heteroatom functionality at γ - or δ -position, with chiral non-racemic but-3-en-1,2-diol **III**, would provide chiral non-racemic allyl alcohol **II** in one step. This allyl alcohol could be transformed quite easily with $\text{PdCl}_2(\text{MeCN})_2$ catalyst into **I** via an intramolecular $\text{S}_{\text{N}}2'$ reaction. The resulting heterocyclic compound **I** possesses a protected allyl alcohol unit, which is able to transform into other functional groups to extend its carbon chain.

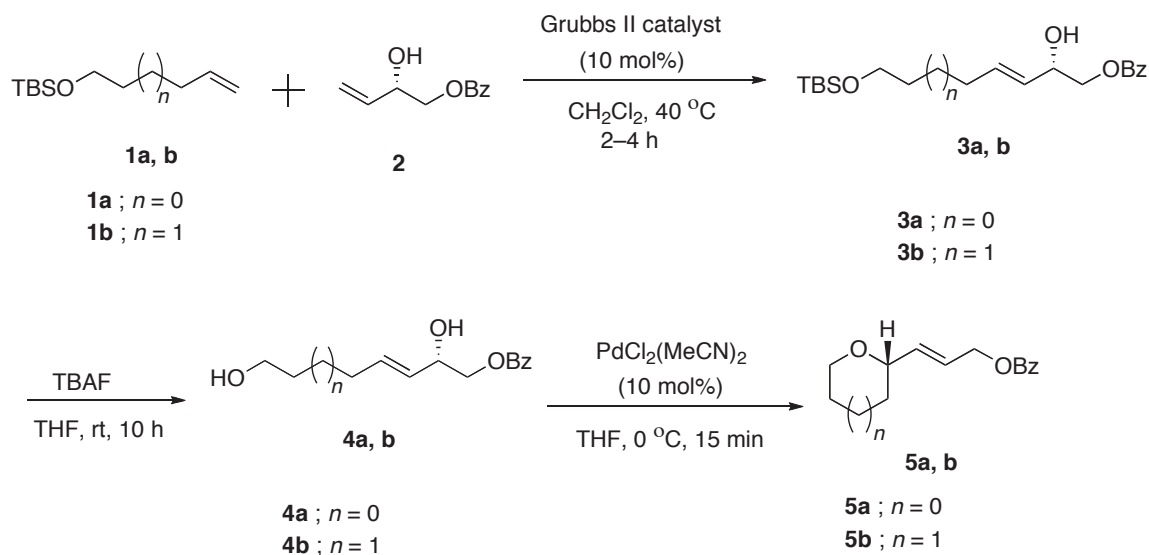


Scheme 2 A synthetic plan of chiral non-racemic heterocycles

In this note, we report a short and convenient synthetic route for the 2-(3-benzoyloxyprop-1-enyl) substituted chiral non-racemic tetrahydrofuran (**5a**), tetrahydropyran (**5b**), and piperidine (**8**) by cross-metathesis and consecutive Pd^{II} -catalyzed cyclization reaction.

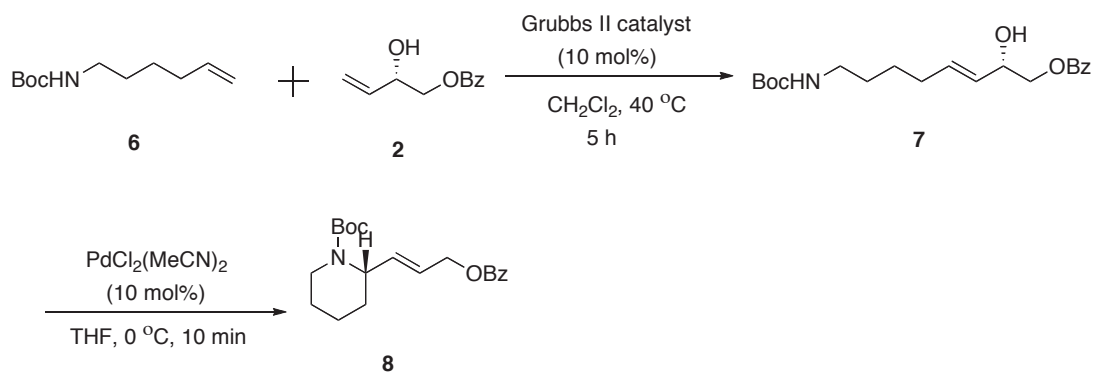
The synthesis of oxa-heterocycles is shown in Scheme 3. A mixture of alkene **1a** and optically pure allyl alcohol (**2**) (>98% ee)⁶ was heated in CH_2Cl_2 at 40 °C in the presence of 10 mol% of Grubbs II catalyst⁷ to give **3a** in 60% yield along with two alkenes derived from the homo-metathesis reactions of each **1a** and **2**. Similarly, the reaction of **1b** with **2** gave **3b** in 61% yield. Deprotection of the TBS group of **3a** and **3b** with TBAF in THF at rt for 10 h afforded the precursors for the cyclization, **4a** and **4b**, in 91% and 84% yields, respectively. The cyclization of **4a** and **4b** were conducted in the presence of 10 mol% $\text{PdCl}_2(\text{MeCN})_2$ at 0 °C for 15 min in THF. Compound **5a** was obtained in 87% yield from **4a**. The enantiomeric ratio was determined to be 97.5:2.5 by chiral HPLC analysis, while cyclization of **4b** afforded **5b** in 92% yield with a 99:1 ratio of enantiomers. We have also examined a cross-metathesis

reaction of **1** with (*S*)-3-butene-1,2-diol, though the chemical yield of the cross-metathesis product was unsatisfactory. The stereochemistry of the products were assumed to have an (*S*)-configuration based on the previous results that we have reported in this series.²⁻⁴ In fact, ozonolysis and Kraus oxidation of **5a** afforded (–)-tetrahydrofuran-2-carboxylic acid, of which the chiral center was identified to be *S*.⁸



Scheme 3 Synthesis of **5a** and **5b**

The synthesis of **8** is performed by the same reaction sequence described for **5** using *N*-Boc protected 5-hexenylamine (**6**)⁹ as a partner of cross-metathesis instead of **1**. The cross-metathesis of **6** and **2** was carried out in CH_2Cl_2 at 40 °C in the presence of 10 mol% of Grubbs II catalyst for 5 h to give **7** in 56% yield. Then, the precursor **7** was subjected to a Pd^{II}-catalyzed cyclization in THF at rt for 10 min to give piperidine (**8**) in 97% yield. Although the chemical yield was excellent, the enantiomeric ratio was found to be slightly lower (93:7) than that of **5**. This trend is consistent with the previous results,^{2d} in which the reaction of an *N*-protected nitrogen nucleophiles was less stereoselective than that of a hydroxy nucleophiles.



Scheme 4

We have demonstrated a short synthetic method for the optically pure oxa- and azaheterocycles by cross-metathesis and Pd^{II}-catalyzed cyclization reactions. An allyl alcohol unit of the resulting heterocycles can be functionalized for the further carbon extension reaction. The formation of (*R*)-enantiomers of **5** and **8** would be expected, if an (*R*)-enantiomer of **5** is used for the metathesis reaction. Thus, this method would be useful for the synthesis of natural products containing chiral THF, THP and piperidine rings in the molecules.

EXPERIMENTAL

General. Column chromatography was performed on E. Merck silica gel (230–400 mesh). The plate used for TLC is E. Merck precoated silica gel 60 F₂₅₄ (0.25–mm thick). Optical rotations were measured on a JASCO P–2200 polarimeter. Infrared (IR) spectra were recorded on a JASCO FT/IR–410 spectrometer. NMR spectra were recorded on a JEOL–AL300 (300 MHz for ¹H NMR and 75 MHz for ¹³C NMR) in CDCl₃, and chemical shifts are reported relative to TMS as internal standard or solvent (CDCl₃, 7.26 ppm). Low-resolution and high-resolution mass spectra (Exact FAB–MS) were obtained with a JEOL JMS–SX 102. Non-aqueous reactions were carried out in flame-dried glassware under an Ar atmosphere. THF were dried over sodium benzophenone ketyl. CH₂Cl₂ was dried over P₄O₁₀. These solvents were distilled freshly before use.

Cross-metathesis reaction; Synthesis of 3a and 3b. A mixture of (*S*)-2-hydroxybut-3-enyl benzoate (**2**) (100 mg, 0.52 mmol) and terminal alkene **1a** or **1b** (0.78 mmol) in CH₂Cl₂ (10 mL) was heated at 40 °C for 2–4 h in the presence of Grubbs II catalyst (44 mg, 0.052 mmol). Solvent was removed and the residue was purified by flash chromatography on silica gel eluted with 25% EtOAc in hexane to give **3a** in 60% yield or **3b** in 61% yield. (**2S,3E**)-7-(*tert*-Butyldimethylsilyloxy)-2-hydroxyhept-3-enyl benzoate (**3a**); Colorless oil; [α]_D²⁰ +3.8 (*c* 0.8, CHCl₃); *R*_f = 0.27 (20% EtOAc in hexane); ¹H NMR (300 MHz, CDCl₃) δ 8.07–8.03 (m, 2H), 7.59–7.53 (m, 1H), 7.46–7.41 (m, 2H), 5.83 (dtd, *J* = 15.4, 6.7, 1.1 Hz, 1H), 5.58 (dtd, *J* = 15.4, 6.4, 1.2 Hz, 1H), 4.47 (m, 1H), 4.36 (dd, *J* = 11.3, 3.6 Hz, 1H), 4.27 (dd, *J* = 11.3, 7.3 Hz, 1H), 3.60 (t, *J* = 6.2 Hz, 2H), 2.5–2.2 (br, 1H), 2.12 (q, *J* = 6.9 Hz, 2H), 1.64–1.55 (m, 2H), 0.89 (s, 9H), 0.04 (s, 6H); ¹³C NMR (75 MHz, CDCl₃) δ 166.6, 134.1, 133.1, 129.9, 129.7, 128.4, 128.0, 70.9, 68.6, 62.3, 32.0, 28.6, 25.9, 18.3, –5.30, –5.32; IR (film, cm^{–1}) 3434, 2929, 1723, 1602, 1452, 1274, 1177, 1100, 970, 836, 776, 711; MS (CI) *m/z* 365 (M⁺+1); HRMS calcd for C₂₀H₃₃O₄Si (M⁺+1) 365.2148; Found: *m/z* 365.2150. (**2S,3E**)-8-(*tert*-Butyldimethylsilyloxy)-2-hydroxy-oct-3-enyl benzoate (**3b**); Colorless oil; [α]_D²⁰ +5.7 (*c* 1.01, CHCl₃); *R*_f = 0.53 (20% EtOAc in hexane); ¹H NMR (300 MHz, CDCl₃) δ 8.07–8.04 (m, 2H), 7.60–7.54 (m, 1H), 7.47–7.41 (m, 2H), 5.82 (dtd, *J* = 15.4, 6.6, 0.7 Hz, 1H), 5.56 (dtd, *J* = 15.4, 6.6, 1.4 Hz, 1H), 4.48 (m, 1H), 4.37 (dd, *J* = 11.3, 3.6 Hz, 1H), 4.27 (dd, *J* = 11.3, 7.3 Hz,

1H), 3.59 (t, $J = 5.8$ Hz, 2H), 2.17 (d, $J = 3.8$ Hz, 1H), 2.08 (q, $J = 6.6$ Hz, 2H), 1.56–1.37 (m, 4H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.6, 134.5, 133.1, 129.9, 129.7, 128.4, 127.9, 71.0, 68.6, 62.9, 32.2, 32.0, 25.9, 25.2, 18.4, –5.28, –5.3; IR (film, cm^{-1}) 3431, 2930, 2857, 1723, 1602, 1452, 1386, 1274, 1177, 1101, 1026, 971, 835, 776, 771; MS (CI) m/z 379 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{21}\text{H}_{35}\text{O}_4\text{Si}$ ($\text{M}^+ + 1$) 379.2304; Found: m/z 379.2313.

Preparation of 4a and 4b. To a solution of **3a** or **3b** (0.12 mmol) in THF (1 mL) was added TBAF (182 μL , 0.18 mmol, 1 M in THF) and the mixture was stirred for 10–12 h at rt. The mixture was diluted with EtOAc and washed with water. The organic layer was dried over MgSO_4 and evaporated. The residue was purified on flash silica gel column chromatography eluted with 80% EtOAc in hexane to give **4a** in 91% yield or **4b** in 84% yield. **(2S,3E)-2,7-Dihydroxyhept-3-enyl benzoate (4a)**; Colorless oil; $[\alpha]_{\text{D}}^{20}$ –1.6 (c 0.63, CHCl_3); $R_f = 0.23$ (60% EtOAc in hexane); ^1H NMR (300 MHz, CDCl_3) δ 8.06–8.03 (m, 2H), 7.60–7.53 (m, 1H), 7.46–7.40 (m, 2H), 5.82 (dtd, $J = 15.4, 6.8, 1.1$ Hz, 1H), 5.59 (ddt, $J = 15.4, 6.4, 1.2$ Hz, 1H), 4.46 (m, 1H), 4.35 (dd, $J = 11.2, 3.8$ Hz, 1H), 4.27 (dd, $J = 11.2, 7.3$ Hz, 1H), 3.62 (t, $J = 6.4$ Hz, 2H), 2.22 (br s, 2H), 2.15 (q, $J = 6.9$ Hz, 2H), 1.64 (quin, $J = 6.6$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.7, 133.6, 133.1, 129.8, 129.6, 128.4, 128.3, 70.8, 68.5, 62.1, 31.7, 28.5; IR (film, cm^{-1}) 3389, 2938, 1716, 1601, 1451, 1277, 1119, 971, 712; MS (CI) m/z 251 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{14}\text{H}_{19}\text{O}_4$ ($\text{M}^+ + 1$) 251.1283; Found: m/z 251.1277. **(2S,3E)-2,8-Dihydroxyoct-3-enyl benzoate (4b)**; Colorless oil; $[\alpha]_{\text{D}}^{20}$ –9.9 (c 0.55, CHCl_3); $R_f = 0.1$ (40% EtOAc in hexane); ^1H NMR (300 MHz, CDCl_3) δ 8.07–8.03 (m, 2H), 7.6–7.54 (m, 1H), 7.47–7.41 (m, 2H), 5.81 (dtd, $J = 15.4, 6.7, 1.1$ Hz, 1H), 5.57 (ddt, $J = 15.5, 6.6, 1.1$ Hz, 1H), 4.46 (m, 1H), 4.36 (dd, $J = 11.3, 3.6$ Hz, 1H), 4.27 (dd, $J = 11.3, 7.3$ Hz, 1H), 3.61 (t, $J = 6.4$ Hz, 2H), 2.1 (q, $J = 6.6$ Hz, 2H), 1.75–1.40 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.6, 134.1, 133.1, 129.8, 129.6, 128.4, 128.2, 70.9, 68.5, 62.6, 32.0, 31.9, 25.0; IR (film, cm^{-1}) 3392, 2935, 1714, 1602, 1452, 1275, 1116, 1070, 971, 755, 713; MS (CI) m/z 265 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{15}\text{H}_{21}\text{O}_4$ ($\text{M}^+ + 1$) 265.1440; Found: m/z 265.1437.

Pd-Catalyzed cyclization of 4a and 4b. A mixture of **4a** or **4b** (0.1 mmol) and $\text{PdCl}_2(\text{MeCN})_2$ (2.6 mg, 0.01 mmol) in THF (3 mL) was stirred at 0 °C for 15 min. Then, the mixture was diluted with hexane (2 mL) and purified directly by flash column chromatography on silica gel eluted with 10% EtOAc in hexane to give **5a** in 87% yield or **5b** in 92% yield. **(S,E)-2-(3-Benzoyloxyprop-1-enyl)-tetrahydrofuran (5a)** Colorless oil; $[\alpha]_{\text{D}}^{20}$ –5.4 (c 1.1, CHCl_3); $R_f = 0.43$ (10% EtOAc in hexane); ^1H NMR (300 MHz, CDCl_3) δ 8.07–8.04 (m, 2H), 7.58–7.52 (m, 1H), 7.46–7.41 (m, 2H), 5.96–5.82 (m, 2H), 4.82 (d, $J = 4.4$ Hz, 2H), 4.38–4.32 (m, 1H), 3.95–3.88 (m, 1H), 3.83–3.76 (m, 1H), 2.13–2.03 (m, 1H), 1.98–1.86 (m, 2H), 1.70–1.59 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.3, 135.3, 133.0, 130.1, 129.6,

128.3, 124.9, 78.7, 68.1, 64.7, 32.0, 25.8; IR (film, cm^{-1}) 2972, 1720, 1601, 1451, 1271, 1112; MS (EI) m/z 232 (M^+), 110 (base), 105; HRMS calcd for $\text{C}_{14}\text{H}_{16}\text{O}_3$ (M^+) 232.1099; Found: m/z 232.1102. The enantiomeric ratio was determined to be 97.5:2.5 by chiral HPLC analysis using the following conditions; column, Chiralcel OD-H; detector, 254 nm; solvent, 2-propanol/hexane (1/99); flow rate, 0.8 mL/min. Retention time; $t_r=15.6$ min (major isomer) and $t_r=16.5$ min (minor isomer). **(S,E)-2-(3-Benzoyloxyprop-1-enyl)tetrahydro-2H-pyran (5b)** Colorless oil; $[\alpha]_{\text{D}}^{20} -4.7$ (c 0.97, CHCl_3); $R_f = 0.4$ (10% EtOAc in hexane); ^1H NMR (300 MHz, CDCl_3) δ 8.07–8.03 (m, 2H), 7.58–7.52 (m, 1H), 7.46–7.40 (m, 2H), 5.96–5.82 (m, 2H), 4.82 (dd, $J = 4.4, 0.9$ Hz, 2H), 4.06–4.0 (m, 1H), 3.88–3.82 (m, 1H), 3.49 (td, $J = 11.0, 2.5$ Hz, 1H), 1.89–1.82 (m, 1H), 1.71–1.34 (m, 5H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.3, 135.5, 132.9, 130.1, 129.6, 128.3, 124.2, 77.1, 68.4, 64.9, 31.8, 25.8, 23.3; IR (film, cm^{-1}) 2864, 1717, 1601, 1452, 1268, 1084, 971, 711; MS (EI) m/z 246 (M^+), 124 (base), 105; HRMS calcd for $\text{C}_{15}\text{H}_{18}\text{O}_3$ (M^+) 246.1256; Found: m/z 246.1258. The enantiomeric ratio was determined to be 99:1 by chiral HPLC analysis using the following conditions; column, Chiralcel OF; detector, 254 nm; solvent, 2-propanol/hexane (1/99); flow rate, 1 mL/min. Retention time; $t_r=27.1$ min (minor isomer) and $t_r=35.4$ min (major isomer).

(2S,3E)-8-(tert-Butoxycarbonylamino)-2-hydroxyoct-3-enyl benzoate (7). The compound was obtained in 56% yield by the same manner described for the synthesis of **3**. Colorless oil; $[\alpha]_{\text{D}}^{20} +8.1$ (c 0.66, CHCl_3); $R_f = 0.28$ (30% EtOAc in hexane); ^1H NMR (300 MHz, CDCl_3) δ 8.06–8.03 (m, 2H), 7.59–7.53 (m, 1H), 7.46–7.41 (m, 2H), 5.78 (dtd, $J = 15.4, 6.6, 0.9$ Hz, 1H), 5.56 (ddt, $J = 15.4, 6.4, 1.2$ Hz, 1H), 4.52 (br s, 1H), 4.46 (m, 1H), 4.36 (dd, $J = 11.2, 3.6$ Hz, 1H), 4.28 (dd, $J = 11.3, 7.1$ Hz, 1H), 3.07 (q, $J = 6.2$ Hz, 2H), 2.50 (br s, 1H), 2.07 (q, $J = 6.4$ Hz, 2H), 1.56–1.33 (m, 13H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.6, 155.9, 133.8, 133.1, 129.8, 129.6, 128.4, 128.3, 79.1, 70.9, 68.5, 40.3, 31.8, 29.3, 28.4, 25.9; IR (film, cm^{-1}) 3389, 2928, 1695, 1452, 1276, 756, 711; MS (CI) m/z 364 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{20}\text{H}_{30}\text{NO}_5$ ($\text{M}^+ + 1$) 364.2124; Found: m/z 264.2129.

(S,E)-N-tert-Butoxycarbonyl-2-(3-benzoyloxyprop-1-enyl)piperidine (8). The compound was obtained in 97% yield by the same manner described for the synthesis of **5**. Colorless oil; $[\alpha]_{\text{D}}^{20} -18.4$ (c 0.84, CHCl_3); $R_f = 0.44$ (20% EtOAc in hexane); ^1H NMR (300 MHz, CDCl_3) δ 8.06–8.03 (m, 2H), 7.59–7.53 (m, 1H), 7.46–7.41 (m, 2H), 5.79 (dd, $J = 15.7, 4.0$ Hz, 1H), 5.73 (dtd, $J = 15.7, 5.6, 1.1$ Hz, 1H), 4.83–4.81 (m, 3H), 3.95 (d, $J = 13.3$ Hz, 1H), 2.83 (td, $J = 13.0, 2.5$ Hz, 1H), 1.74–1.38 (m, 15H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.3, 155.3, 133.8, 133.0, 130.2, 129.6, 128.3, 125.2, 79.5, 65.1, 51.5, 39.8, 29.0, 28.4, 25.4, 19.5; IR (film, cm^{-1}) 2937, 1722, 1692, 1452, 1409, 1271, 1163, 1114, 1025, 973, 869, 713; MS (EI) m/z 345 (M^+), 289, 272, 167 (base); HRMS calcd for $\text{C}_{20}\text{H}_{27}\text{NO}_4$ (M^+) 345.1940; Found: m/z

345.1935. The enantiomeric ratio was determined to be 93:7 by chiral HPLC analysis using the following conditions; column, Chiralcel AS-H; detector, 254 nm; solvent, 2-propanol/hexane (1/99); flow rate, 1 mL/min. Retention time; $t_r=7.7$ min (major isomer) and $t_r=8.4$ min (minor isomer).

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