Supporting Information

Intramolecular Diels-Alder Reactions of Ester Tethered 1,7,9-Decatrienoates: Bis[chloro(methyl)aluminum]trifluoromethanesulfonamide as a Catalyst

Authored by

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Supporting Information Available. Experimental procedure for **2**, **3** and their characterization data and structure determination of **3b**, **3g**. This material is available free of charge via Internet at <http://pubs.acs.org>.

Experimental Section

Melting points were uncorrected. ^1H and ^{13}C NMR spectra were recorded on a 400 MHz spectrometer. In ^1H and ^{13}C NMR spectra, chemical shifts were expressed in δ (ppm) downfield from CHCl_3 (7.26 ppm) and CDCl_3 (77.0 ppm), respectively. Mass spectra were recorded by electron impact or chemical ionization. Column chromatography was performed on silica gel (75-150 μm). Medium-pressure liquid chromatography (MPLC) was performed on a 30 X 4 cm i. d. prepacked column (silica gel, 50 μm) with a UV or RI detector.

(*E*)-4-Methyl-3,5-hexadienyl (*E*)-4-methyl-2-pentenoate (2a) and (*E*)-3,5-hexadienyl 2-propenoate (2c). Compounds **2a** and **2c** were prepared according to Jung's¹ or Martin's² procedure. ^1H NMR spectra of these compounds were identical with those reported in the literature.

(*E*)-4,6-heptadien-2-yl 2-propenoate (2b). After a mixture of (*E*)-4,6-heptadienyl-2-ol (1.5 g, 13.4 mmol), acryloyl chloride (1.3 mL, 15 mmol) and triethylamine (2.4 mL, 17 mmol) in CH_2Cl_2 (30 mL) was stirred for 1 h at 0 $^\circ\text{C}$ and then for 2 h at room temperature, the reaction mixture was extracted with ether after addition of 5% HCl. The organic layer was washed with brine, dried over MgSO_4 and purified by silica gel column chromatography (hexane/AcOEt 5:1) to give **2b** (1.9 g, 85%). Colorless oil; IR (neat) vcm^{-1} ; 1724. ^1H NMR (400 MHz, CDCl_3) δ : 6.36 (1H, dd, $J = 17.4, 1.2$ Hz), 6.30 (1H, ddd, $J = 17.0, 10.2, 10.2$ Hz), 6.14-6.03 (1H, m), 6.08 (1H, dd, $J = 17.4, 10.4$ Hz), 5.78 (1H, dd, $J = 10.4, 1.2$ Hz), 5.62 (1H, ddd, $J = 15.2, 6.8, 6.8$ Hz), 5.10 (1H, d, $J = 17.0$ Hz), 5.06-4.97 (2H, m), 2.40 (1H, ddd, $J = 14.1, 6.8, 6.8$ Hz), 2.32 (1H, ddd, $J = 14.1, 6.8, 6.8$ Hz), 1.24 (3H, d, $J = 6.3$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ : 19.4, 38.9, 70.4, 115.9, 128.9, 129.2, 130.3, 133.9, 136.8, 165.6. EI-MS m/z : 166 (M^+). HRMS calcd for $\text{C}_{10}\text{H}_{14}\text{O}_2$: 166.0994 (M^+). Found: 166.1014.

By a similar procedure for the preparation of **2b**, the substrates **2d-2g** were prepared from the corresponding dienol and acid chloride.

(E)-4-Methyl-3,5-hexadienyl 2-propenoate (2d). colorless oil; IR (neat) vcm^{-1} ; 1733. ^1H NMR (400 MHz, CDCl_3) δ ; 6.39 (1H, dd, $J = 17.3, 1.5$ Hz), 6.37 (1H, dd, $J = 17.4, 10.8$ Hz), 6.11 (1H, dd, $J = 17.3, 10.4$ Hz), 5.81 (1H, dd, $J = 10.4, 1.5$ Hz), 5.48 (1H, t, $J = 7.1$ Hz), 5.13 (1H, d, $J = 17.4$ Hz), 4.98 (1H, d, $J = 10.8$ Hz), 4.19 (2H, t, $J = 7.1$ Hz), 2.52 (2H, dt, $J = 7.1, 7.1$ Hz), 1.77 (3H, s). ^{13}C NMR (100 MHz, CDCl_3) δ ; 11.8, 27.8, 63.7, 111.6, 127.2, 128.5, 130.6, 136.6, 141.0, 166.2. EI-MS m/z : 166 (M^+). Anal. Calcd for $\text{C}_{10}\text{H}_{14}\text{O}_2$: C, 72.26; H, 8.49. Found: C, 72.09; H, 8.33.

(3E,5E)-3,5-Octadienyl 2-propenoate (2e). colorless oil; IR (neat) vcm^{-1} ; 1727. ^1H NMR (400 MHz, CDCl_3) δ ; 6.40 (1H, dd, $J = 17.3, 1.5$ Hz), 6.12 (1H, dd, $J = 17.3, 10.4$ Hz), 6.11-5.97 (2H, m), 5.81 (1H, dd, $J = 10.4, 1.5$ Hz), 5.66 (1H, dt, $J = 14.7, 6.8$ Hz), 5.53 (1H, dt, $J = 14.3, 7.3$ Hz), 4.18 (2H, t, $J = 6.8$ Hz), 2.43 (2H, dt, $J = 6.8, 6.8$ Hz), 2.09 (2H, dq, $J = 7.3, 7.3$ Hz), 1.00 (3H, t, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ ; 13.5, 25.6, 31.9, 64.0, 126.4, 128.5, 128.9, 130.6, 133.1, 135.4, 166.2. EI-MS m/z : 180 (M^+). HRMS calcd for $\text{C}_{11}\text{H}_{16}\text{O}_2$: 180.1150 (M^+). Found: 180.1157.

(E)-3,5-Hexadienyl (E)-3-phenyl-2-propenoate (2f). colorless oil; IR (neat) vcm^{-1} ; 1713. ^1H NMR (400 MHz, CDCl_3) δ ; 7.69 (1H, d, $J = 16.0$ Hz), 7.55-7.50 (2H, m), 7.41-7.37 (3H, m), 6.44 (1H, d, $J = 16.0$ Hz), 6.33 (1H, ddd, $J = 16.8, 10.2, 10.2$ Hz), 6.17 (1H, dd, $J = 15.2, 10.2$ Hz), 5.72 (1H, dt, $J = 15.2, 6.8$ Hz), 5.15 (1H, d, $J = 16.8$ Hz), 5.03 (1H, d, $J = 10.2$ Hz), 4.26 (2H, t, $J = 6.8$ Hz), 2.50 (2H, dt, $J = 6.8, 6.8$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ ; 32.0, 63.7, 116.1, 118.1, 128.1, 128.9, 129.8, 130.3, 133.4, 134.4, 136.8, 144.8, 166.9. EI-MS m/z : 228 (M^+). HRMS calcd for $\text{C}_{15}\text{H}_{16}\text{O}_2$: 228.1150 (M^+). Found: 228.1162. Anal. Calcd for $\text{C}_{15}\text{H}_{16}\text{O}_2$: C, 78.92; H, 7.06. Found: C, 78.59; H, 7.04.

(E)-4,6-Heptadien-2-yl 2-methyl-2-propenoate (2g). colorless oil; IR (neat) vcm^{-1} ; 1717. ^1H NMR (400 MHz, CDCl_3) δ ; 6.29 (1H, ddd, $J = 16.9, 10.4, 10.4$ Hz), 6.09 (1H, dd, $J = 15.1, 10.4$ Hz), 6.06 (1H, br.s), 5.63 (1H, ddd, $J = 15.1, 6.7, 6.7$ Hz), 5.51 (1H, br.s), 5.11 (1H, d, $J = 16.9$ Hz), 5.04-4.95 (2H, m), 2.40 (1H, ddd, $J = 14.0, 6.7, 6.7$ Hz), 2.33 (1H, ddd, $J = 14.0, 6.7, 6.7$ Hz), 1.91 (3H, s), 1.25 (3H, d, $J = 6.7$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ ; 18.2, 19.4, 39.0, 70.4, 115.8, 125.0, 129.0, 133.9, 136.7, 136.8, 166.9. EI-MS m/z : 180 (M^+). HRMS calcd for $\text{C}_{11}\text{H}_{16}\text{O}_2$: 180.1150 (M^+). Found: 180.1124.

General procedure of the IMDA reaction catalyzed by $\text{TfN}[\text{Al}(\text{Me})\text{Cl}]_2$:

(4aR*,8R*,8aR*)-5-Methyl-8-(1-methylethyl)-3,4,4a,7,8,8a-hexahydro-isochromen-1-one (3a-endo) and (4aR*,8S*,8aS*)-5-Methyl-8-(1-methylethyl)-3,4,4a,7,8,8a-hexahydro-isochromen-1-one (3a-exo).

Under an argon atmosphere, to a solution of trifluoromethanesulfonamide (79 mg, 0.53 mmol) in 1,2-dichloroethane (4.5 mL) at 0 °C (with ice-bath) was added dimethylaluminum chloride (0.98 M hexane solution, 1.08 mL, 1.06 mmol). After the mixture was stirred for 30 min at rt, **2a** (100 mg, 0.48 mmol) in 1,2-dichloroethane (2.5 mL) was added, then the mixture was stirred at 80 °C for 3 h. To the reaction mixture was added 1N HCl and ethyl acetate. Organic layer was separated and washed with brine, dried over MgSO_4 , then concentrated under vacuum. The residue was chromatographed on silica gel (hexane/ Et_2O 10:1-6:1) to give **3a-exo** (4.5 mg, 4.5 %) and **3a-endo** (63.2 mg, 63 %), respectively. ^1H NMR spectra of these compounds were identical with those reported by Jung *et al.*¹

(3S*,4aS*,8aR*)-3-Methyl-3,4,4a,7,8,8a-hexahydro-isochromen-1-one (3b-endo).

colorless oil; IR (neat) vcm^{-1} ; 1734. ^1H NMR (400 MHz, CDCl_3) δ : 5.70 (1H, br.d, $J = 10.0$ Hz), 5.48 (1H, br.d, $J = 10.0$ Hz), 4.37 (1H, dqd, $J = 12.3, 6.2, 2.5$ Hz), 2.74-2.57 (2H, m), 2.20-2.09 (1H, m), 2.06-1.92 (1H, m), 1.96 (1H, ddd, $J = 14.2, 5.6, 2.5$ Hz), 1.90-1.75 (2H, m), 1.37-1.26 (1H, m), 1.30 (3H, d, $J = 6.2$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ : 21.3, 22.6, 23.3, 31.4, 35.3, 37.8, 75.2, 127.2, 128.1, 174.6. EI-MS m/z : 167 ($\text{M}^+ + 1$). HRMS calcd for $\text{C}_{10}\text{H}_{14}\text{O}_2$: 166.1007 (M^+). Found: 166.0994.

(4a*S,8a*R**)-3,4,4a,7,8,8a-Hexahydro-isochromen-1-one (3c-endo).** ^1H NMR spectra of this compound were identical with those reported by Martin *et al.*²

(4a*R,8a*R**)-5-Methyl-3,4,4a,7,8,8a-hexahydro-isochromen-1-one (3d-endo).** colorless solid; mp 43-44 °C. IR (KBr) vcm^{-1} ; 1728. ^1H NMR (400 MHz, CDCl_3) δ : 5.57 (1H, br.s), 4.35-4.21 (2H, m), 2.82-2.76 (1H, m), 2.50 (1H, br.s), 2.10-1.94 (4H, m), 1.88-1.76 (2H, m), 1.68 (3H, s). ^{13}C NMR (100 MHz, CDCl_3) δ : 21.1, 23.5, 23.5, 25.9, 36.2, 40.4, 68.0, 124.4, 132.8, 174.0. EI-MS m/z : 166 (M^+). HRMS calcd for $\text{C}_{10}\text{H}_{14}\text{O}_2$: 166.0994 (M^+). Found: 166.0977.

(4a*S,7*S**,8a*R**)-7-Ethyl-3,4,4a,7,8,8a-hexahydro-isochromen-1-one (3e-endo).** colorless oil; IR (neat) vcm^{-1} ; 1725. ^1H NMR (400 MHz, CDCl_3) δ : 5.69 (1H, br.d, $J = 10.0$ Hz), 5.58 (1H, ddd, $J = 10.0, 4.2, 2.3$ Hz), 4.41 (1H, ddd, $J = 11.2, 4.6, 2.7$ Hz), 4.26 (1H, ddd, $J = 11.2, 11.2, 3.3$ Hz), 2.85-2.77 (1H, m), 2.58-2.49 (1H, m), 2.17-2.07 (2H, m), 1.99-1.66 (2H, m), 1.46-1.25 (3H, m), 0.93 (3H, t, $J = 7.4$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ : 11.0, 27.2, 28.5, 29.6, 32.7, 37.2, 40.0, 69.1, 127.2, 133.3, 174.4. EI-MS m/z : 181 ($\text{M}^+ + 1$). HRMS calcd for $\text{C}_{11}\text{H}_{16}\text{O}_2$: 180.1150 (M^+). Found: 180.1153.

(4a*S,8*R**,8a*S**)-8-Phenyl-3,4,4a,7,8,8a-hexahydro-isochromen-1-one (3f-endo).** colorless solid; mp 77-78 °C. IR (KBr) vcm^{-1} ; 1731. ^1H NMR (400 MHz, CDCl_3) δ : 7.32-7.26 (4H, m), 7.25-7.18 (1H, m), 6.09 (1H, dddd, $J = 10.2, 4.8, 2.3, 2.3$ Hz), 5.63 (1H, dd, $J = 10.2, 1.8$ Hz), 4.27 (2H, dd, $J = 8.0, 4.1$ Hz), 3.90 (1H, dd, $J = 6.1, 3.9$ Hz), 2.74 (1H, dd, $J = 6.1, 3.2$ Hz), 2.61 (1H, dddd, $J = 19.1, 9.3, 3.9, 1.8$ Hz), 2.47-2.38 (1H, m), 2.32 (1H, br.d, $J = 19.1$ Hz), 2.01 (1H, dddd, $J = 14.0, 8.0, 8.0, 5.2$ Hz), 1.76 (1H, dddd, $J = 14.2, 4.1, 4.1, 4.1$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ : 25.6, 27.2, 28.2, 37.5, 45.8, 66.6, 126.5, 127.3, 128.4, 128.6, 129.8, 143.6, 172.6. EI-MS m/z : 228 (M^+). HRMS calcd for $\text{C}_{15}\text{H}_{16}\text{O}_2$: 228.1150 (M^+). Found: 228.1150. Anal. Calcd for $\text{C}_{15}\text{H}_{16}\text{O}_2$: C, 78.92; H, 7.06. Found: C, 78.87; H, 7.01.

(3*S,4a*S**,8a*R**)-3,8a-Dimethyl-3,4,4a,7,8,8a-hexahydro-isochromen-1-one (3g-endo-1).** colorless solid; mp 47-48 °C. IR (KBr) vcm^{-1} ; 1720. ^1H NMR (400 MHz, CDCl_3) δ : 5.75-5.68 (1H, m), 5.60-5.53 (1H, m), 4.42 (1H, dqd, $J = 12.5, 6.3, 2.6$ Hz), 2.24-1.91 (3H, m), 1.93 (1H, ddd, $J = 14.2, 3.1, 3.1$ Hz), 1.78-1.72 (1H, m), 1.46 (1H, ddd, $J = 14.2, 12.1, 12.1$ Hz), 1.35 (3H, d, $J = 6.3$ Hz), 1.30 (3H, s). ^{13}C NMR (100 MHz, CDCl_3) δ : 21.5, 22.0, 22.2, 28.5, 36.1, 39.5, 40.1, 76.2, 126.2, 127.5, 177.9. EI-MS m/z : 181 ($\text{M}^+ + 1$). HRMS calcd for $\text{C}_{11}\text{H}_{16}\text{O}_2$: 180.1150 (M^+). Found: 180.1165.

(3*S,4a*R**,8a*S**)-3,8a-Dimethyl-3,4,4a,7,8,8a-hexahydro-isochromen-1-one (3g-endo-2).** colorless oil; IR (neat) vcm^{-1} ; 1720. ^1H NMR (400 MHz, CDCl_3) δ : 5.90-5.82 (1H, m), 5.46 (1H, br.d, $J = 10.0$ Hz), 4.37 (1H, dqd, $J = 12.4, 6.3, 3.2$ Hz), 2.41-2.34 (1H, m), 2.27 (1H, br.d, $J = 13.0$ Hz), 2.11-1.92 (3H, m), 1.68 (1H, ddd, $J = 14.2, 3.2, 3.2$ Hz), 1.37 (1H, ddd, $J = 13.0, 8.6, 6.5$ Hz), 1.33 (3H, d, $J = 6.3$ Hz), 1.32 (3H, s). ^{13}C NMR (100 MHz, CDCl_3) δ : 21.8, 22.6, 27.6, 32.8, 33.0, 38.6, 40.9, 74.1, 128.4, 130.1, 176.7. EI-MS m/z : 181 ($\text{M}^+ + 1$). HRMS calcd for $\text{C}_{11}\text{H}_{16}\text{O}_2$: 180.1150 (M^+). Found: 180.1160.

For the structure determination of **3b-endo**, **3g-endo-1** and **3g-endo-2** following experiments were carried out (Scheme 4). Selected NOE experiment results are shown in Figure 1.

Scheme 4

Figure 1. NOE experiments

Hydrogenation of **3b-endo**.

Under a hydrogen atmosphere, a mixture of **3b-endo** (13 mg, 77.4 μmol) and $(\text{Ph}_3\text{P})_3\text{RhCl}$ (7.2 mg, 7.78 μmol) in benzene (2.0 mL) was stirred for 12 h at rt. The mixture was chromatographed on silica gel (hexane/ Et_2O 6:1) to give (3*S**,4*aR**,8*aR**)-3-methyloctahydro-isochromen-1-one **4a** (13.0 mg, quant.). ^1H NMR spectra of the product were identical with those reported by Fujiwara *et al.*³

Preparation of 3,8a-dimethyloctahydro-isochromen-1-one from 3-methyloctahydro-isochromen-1-one: (3*S**,4*aR**,8*aR**)-3,8a-Dimethyloctahydro-isochromen-1-one (**4c**) and (3*S**,4*aR**,8*aS**)-3,8a-dimethyloctahydro-isochromen-1-one (**4d**).

Under an argon atmosphere, to a solution of 1,1,1,3,3,3-hexamethyldisilazane (0.32 mL, 1.53 mmol) in THF (2.0 mL) at 0 $^\circ\text{C}$ was added *n*-BuLi (1.58 M hexane solution, 0.97 mL, 1.53 mmol). After the mixture was stirred for 30 min at rt, (3*S**,4*aR**,8*aR**)-3-methyloctahydro-isochromen-1-one **4a** (50 mg, 0.30 mmol) in THF (2.0 mL) was added at -78 $^\circ\text{C}$, then the mixture was stirred at -78 $^\circ\text{C}$ for 50 min and -40 $^\circ\text{C}$ for 10 min. To the reaction mixture was added iodomethane (0.2 mL, 3.21 mmol) and HMPA (0.5 mL) at -40 $^\circ\text{C}$. After the mixture was stirred for 2 h at -40~-20 $^\circ\text{C}$, to the reaction mixture was added 1N HCl and ethyl acetate. Organic layer was separated and washed with brine, dried over MgSO_4 , then concentrated under vacuum. The residue was chromatographed on silica gel (hexane/ Et_2O 6:1) to give a mixture of **4c** and **4d**, which was further separated by MPLC (hexane/ Et_2O 3:1, flow rate 7.0 mL/min) to give **4c** (10.9 mg, 20 %) and **4d** (18.3 mg, 34 %) in the order of elution.

4c. colorless solid; mp 28-30 $^\circ\text{C}$. IR (KBr) vcm^{-1} ; 1725. ^1H NMR (400 MHz, CDCl_3) δ ; 4.43 (1H, dqd, $J = 12.6, 6.3, 3.7$ Hz), 1.93-1.79 (3H, m), 1.78-1.63 (2H, m), 1.60-1.38 (6H, m), 1.36 (3H, s), 1.35 (3H, d, $J = 6.3$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ ; 21.2, 21.5, 22.3, 22.6, 28.1, 32.9, 33.3, 37.6, 41.6, 75.9, 177.9. EI-MS m/z : 183 ($\text{M}^+ + 1$). HRMS calcd for $\text{C}_{11}\text{H}_{18}\text{O}_2$: 182.1307 (M^+). Found: 182.1314. Anal. Calcd for $\text{C}_{11}\text{H}_{18}\text{O}_2$: C, 72.49; H, 9.95. Found: C, 72.61; H, 10.06.

4d. colorless solid; mp 42-44 $^\circ\text{C}$. IR (KBr) vcm^{-1} ; 1732. ^1H NMR (400 MHz, CDCl_3) δ ; 4.40 (1H, dqd, $J = 9.2, 6.2, 6.2$ Hz), 2.03-1.93 (1H, m), 1.86-1.50 (4H, m), 1.50-1.28 (4H, m), 1.30 (3H, d, $J = 6.2$ Hz), 1.28-1.14 (2H, m), 1.12 (3H, s). ^{13}C NMR (100 MHz, CDCl_3) δ ; 17.4, 21.1, 22.3, 25.6, 26.9, 33.9, 34.1, 39.2, 41.1, 77.6, 177.7. EI-MS m/z : 183 ($\text{M}^+ + 1$). Anal. Calcd for $\text{C}_{11}\text{H}_{18}\text{O}_2$: C, 72.49; H, 9.95. Found: C, 72.39; H, 9.96.

(3*S**,4*aS**,8*aS**)-3,8a-Dimethyloctahydro-isochromen-1-one (**4e**).

In a similar manner as above, methylation of (3*S**,4*aS**,8*aS**)-3-methyloctahydroisochromen-1-one **4b**³ (50 mg, 0.30 mmol) and purification by silica gel column (hexane/ Et₂O 6:1) gave **4e** (34 mg, 63 %).

4e. colorless oil; IR (neat) $\nu_{\text{cm}^{-1}}$; 1727. ¹H NMR (400 MHz, CDCl₃) δ ; 4.67 (1H, dqd, *J* = 12.1, 6.2, 4.7 Hz), 2.36 (1H, br.d, *J* = 13.5 Hz), 2.07 (1H, ddd, *J* = 14.3, 12.1, 3.8 Hz), 1.79-1.69 (1H, m), 1.69-1.52 (4H, m), 1.42-1.18 (3H, m), 1.36 (3H, d, *J* = 6.2 Hz), 1.28 (3H, s), 1.12-0.80 (1H, m). ¹³C NMR (100 MHz, CDCl₃) δ ; 22.6, 23.7, 25.7, 28.2, 29.4, 32.8, 36.2, 39.7, 42.8, 74.4, 176.5. EI-MS *m/z*: 183 (*M*⁺+1). HRMS calcd for C₁₁H₁₈O₂: 182.1307 (*M*⁺). Found: 182.1321. Anal. Calcd for C₁₁H₁₈O₂: C, 72.49; H, 9.95. Found: C, 72.56; H, 9.93.

Hydrogenation of **3g-endo-1**.

In a similar manner, hydrogenation of **3g-endo-1** (12.1 mg, 72.0 μ mol) and the subsequent purification by column chromatography on silica gel (hexane/ Et₂O 6:1) gave **4c** (12.0 mg, quant.).

Hydrogenation of **3g-endo-2**.

In a similar manner, hydrogenation of **3g-endo-2** (8.0 mg, 47.6 μ mol) and the subsequent purification by column chromatography on silica gel (hexane/ Et₂O 6:1) gave **4e** (8.1 mg, quant.).

References

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