Supporting Information

Skeletal Reorganization of Enynes Catalyzed by InCl_3

Yuhei Miyanohana and Naoto Chatani*

*Department of Applied Chemistry, Faculty of Engineering, Osaka University,
Suita, Osaka 565-0871, Japan*

General Information ^1H NMR and ^{13}C NMR spectra were recorded on a JEOL JMN-270 spectrometer in CDCl_3 with tetramethylsilane as an internal standard. Data are reported as follows: chemical shift in ppm (δ), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and c = complex), coupling constant (Hz), integration, and interpretation. Infrared spectra (IR) were obtained on a Horiba FT-700 spectrometer. Mass spectra were obtained on a Shimadzu GCMS-QP 5000 instrument with ionization voltages of 70 eV. High resolution mass spectra (HRMS) were obtained on a JEOL JMS-DX303. Analytical GC was carried out on a Shimadzu GC-14B gas chromatography, equipped with a flame ionization detector. Melting points were determined on a Yamato melting point apparatus and are uncorrected. Column chromatography was performed with SiO_2 (Merck SilicaGel 60 (230-400 mesh)).

Materials Toluene and 1,2-dichloroethane was distilled over CaH_2 . InCl_3 was purchased from Aldrich Chemical Co. and was used without purification. All enynes without showing procedure were obtained from treatment of diethyl allylmaronate with NaOEt followed by the corresponding propargyl bromides.

Typical Procedure for InCl_3 Catalyzed Skeletal Reorganization. In a 8-mL screw vial was charged with InCl_3 (11.1 mg, 0.05 mmol), (2-pentynyl)-(2-propenyl)propanedionic acid diethyl ester (**4**) (133.2 mg, 0.5 mmol) and 1,2-dichloroethane (2.5 mL), and the resulting mixture was stirred at 80°C for 2 h. After cooling to room temperature, the reaction mixture was concentrated *in vacuo*. The residue was subjected by flash column chromatography on silica gel (hexane/ Et_2O = 10/1, R_f = 0.17) to afford 98.6 mg of 3-(2-butenyl)-3-cyclopentene-1,1-dicarboxylic acid diethyl ester (**5**) (74 %) as colorless liquid.

3-(2-Butenyl)-3-cyclopentene-1,1-dicarboxylic acid diethyl ester (5**).** Colorless oil; R_f 0.17 (hexane/ Et_2O = 10/1); ^1H NMR (CDCl_3) δ : 1.24 (t, J = 7.0 Hz, 6H), [1.61 ((Z)-**5**, d, J = 6.5 Hz), 1.65 ((E)-**5**, d, J = 4.6 Hz), 3H], [2.71 ((E)-**5**, brs), 2.78 ((E)-**5**, c), 2H], 2.90 (s, 2H), 2.98 (s, 2H), 4.19 (q, J = 7.0 Hz, 4H), 5.21 (s, 1H), 5.38-5.52 (c). ^{13}C NMR (CDCl_3) δ : 14.1, 17.8, [28.3 ((Z)-**5**), 33.9 ((E)-**5**)], 40.6, 42.9, 59.1, 61.3, 61.4, [120.6 ((Z)-**5**), 120.8 ((E)-**5**)], 126.7, 127.7, 140.9, 172.3. IR (neat): 2981 s, 2937 m, 1734 s, 1462 m, 1446 m, 1389 w, 1367 w, 1298 s, 1254 s, 1182 s, 1159 s, 1097 m, 1070 m, 1016 m, 968 m, 862 m, 810 w, 783 w, 754 w, 536 w. MS, m/z (relative intensity, %): 266 (M^+ , 12), 221 (10), 192 (29), 163 (25), 119 (100), 118 (28), 117 (18), 105 (17), 93 (17), 91 (64), 79 (45), 78 (19), 77 (42), 65 (32), 55 (55). Exact Mass Calcd for $\text{C}_{15}\text{H}_{22}\text{O}_4$: 266.1518. Found: 266.1513.

3-(2-Hexenyl)-3-cyclopentene-1,1-dicarboxylic acid diethyl ester (6).

Colorless oil; R_f 0.20 (hexane/Et₂O = 10/1); ¹H NMR (CDCl₃) δ : 0.88 (t, J = 6.9 Hz, 3H), 1.24 (t, J = 7.0 Hz, 6H), 1.32 (tq, J = 6.4 Hz, 6.9 Hz, 2H), 1.99 (dt, J = 6.4 Hz, 6.4 Hz, 2H), [2.70 ((*E*)-6, brs), 2.76 ((*Z*)-6, c), 2H], 2.90 (s, 2H), 2.97 (dd, J = 1.9 Hz, 1.9 Hz, 2H), 4.19 (q, J = 7.0 Hz, 4H), 5.21 (d, J = 1.9 Hz, 1H), 5.33-5.50 (c, 2H). ¹³C NMR (CDCl₃) δ : [13.6 ((*E*)-6), 13.8 ((*Z*)-6)], 14.0, [22.5 ((*E*)-6), 22.7 ((*Z*)-6)], [28.6, 29.2 ((*Z*)-6), 33.9, 34.6 ((*E*)-6)], 40.6, 42.9, 59.1, 61.4 [120.6 ((*Z*)-6), 120.8 ((*E*)-6)], [126.0 ((*Z*)-6), 126.7 ((*E*)-6)], [131.2 ((*Z*)-6), 132.2 ((*E*)-6)] 141.0, 172.3. IR (neat): 2960 s, 2931 s, 2871 m, 1734 s, 1658 w, 1464 m, 1446 m, 1389 m, 1367 m, 1298 s, 1254 s, 1180 s, 1159 s, 1097 m, 1070 s, 1016 m, 970 m, 862 m, 810 w, 781 w, 755 w, 459 w, 420 w. MS, m/z (relative intensity, %): 294 (M^+ , 9), 221 (17), 220 (50), 191 (32), 177 (14), 174 (14), 163 (14), 147 (82), 146 (18), 145 (16), 119 (17), 117 (38), 105 (48), 103 (17), 93 (20), 92 (18), 91 (93), 79 (54), 78 (16), 77 (45), 67 (22), 65 (29), 55 (100), 53 (22). Exact Mass Calcd for C₁₇H₂₆O₄: 294.1831. Found: 294.1842.

3-(2-Heptenyl)-3-cyclopentene-1,1-dicarboxylic acid diethyl ester

(7). Colorless oil; R_f 0.20 (hexane/Et₂O = 10/1); ¹H NMR (CDCl₃) δ : 0.89 (t, J = 6.6 Hz, 3H), 1.24 (t, J = 7.0 Hz, 6H), 1.30-1.32 (m, 4H), 2.00 (dt, J = 6.3 Hz, 6.3 Hz, 2H), [2.71 ((*E*)-7, brs), 2.77 ((*Z*)-7, c), 2H], 2.90 (s, 2H), 2.97 (s, 2H), 4.19 (q, J = 7.0 Hz, 4H), 5.21 (s, 1H), 5.33-5.50 (c, 2H). ¹³C NMR (CDCl₃) δ : 13.9, 14.0, 22.2, [26.8, 28.6 ((*Z*)-7), 31.6, 32.1 ((*E*)-7)], 40.6, 42.9, 59.1, 61.4 [120.5 ((*Z*)-7), 120.7 ((*E*)-7)], [125.7 ((*Z*)-7), 126.4 ((*E*)-7)], [131.4 ((*Z*)-7), 132.4 ((*E*)-5)] 141.0, 172.3. IR (neat): 2979 m, 2960 m, 2931 m, 1734 m, 1734 s, 1658 w, 1464 m, 1446 m, 1390 m, 1367 m, 1298 m, 1254 s, 1182 m, 1072 m, 1016 m, 970 m, 862 m, 814 w, 781 w, 494 w, 422 w. MS, m/z (relative intensity, %): 308 (M^+ , 8), 234 (31), 161 (45), 119 (21), 117 (35), 105 (40), 93 (18), 92 (15), 91 (65), 79 (45), 77 (30), 67 (20), 65 (19), 55 (100), 53 (13). Exact Mass Calcd for C₁₈H₂₈O₄: 308.1988. Found: 308.1991.

(5,5-Dimethyl-2-hexynyl)-(2-propenyl)propanedionic acid diethyl ester.

Colorless oil; bp 100 °C /0.2 mmHg; ¹H NMR (CDCl₃) δ : 0.95 (s, 9H), 1.24 (t, J = 7.0 Hz, 6H), 2.00 (t, J = 2.4 Hz, 2H), 2.78-2.82 (c, 4H), 4.19 (q, J = 7.0 Hz, 4H), 5.09-5.19 (c, 2H), 5.57-5.72 (m, 1H). ¹³C NMR (CDCl₃) δ : 14.1, 22.9, 28.9, 31.0, 33.8, 36.4, 57.0, 61.4, 75.7, 81.8, 119.4, 132.1, 170.0. IR (neat): 2958 m, 2904 w, 2871 w, 1738 s, 1558 w, 1541 w, 1475 w, 1367 w, 1288 m, 1215 m, 1192 m, 1136 w, 1097 w, 1068 w, 1039 w, 924 w, 862 w. MS, m/z (relative intensity, %): 308 (M^+ , 0.3), 235 (20), 221 (25), 178 (89), 163 (15), 149 (14), 108 (18), 105 (47), 91 (19), 57 (100). Exact Mass Calcd for C₁₈H₂₈O₄: 308.1988. Found: 308.1992.

3-(4,4-Dimethyl-2-pentenyl)-3-cyclopentene-1,1-dicarboxylic acid diethyl ester (8).

Colorless oil; R_f 0.19 (hexane/Et₂O = 12/1); ¹H NMR (CDCl₃) δ : 0.99 (s, 9H), 1.24 (t, J = 7.0 Hz, 6H), 2.70 (d, J = 6.5 Hz, 2H), 2.90 (s, 2H), 2.97 (s, 2H), 4.19 (q, J = 7.0 Hz, 4H), 5.20 (s, 1H), 5.30 (dt, J = 15.7 Hz, 6.5 Hz, 1H), 5.48 (d, J = 15.7 Hz, 1H). ¹³C NMR (CDCl₃) δ : 14.0, 29.7, 32.9, 33.9, 40.6, 42.9, 59.1, 61.4, 120.7, 121.1, 141.1, 143.5, 172.3. IR (neat): 2958 m, 2902 w, 2868 w, 1734 s, 1558 w, 1541 w, 1508 w, 1473 w, 1392 w, 1365 w, 1298 w, 1255 m, 1184 m, 1159 w, 1097 w, 1072 w, 1018 w, 973 w, 862 w. MS, m/z (relative intensity, %): 308 (M^+ , 58), 263 (21), 235 (38), 234 (100), 219 (29), 191 (45), 178 (82), 165 (29), 164

(50), 161 (48), 145 (35), 105 (38), 96 (23), 91 (32), 83 (57), 79 (23), 57 (40), 55 (42). Exact Mass Calcd for $C_{18}H_{28}O_4$: 308.1988. Found: 308.1993.

(4-Methyl-2-pentynyl)-(2-propenyl)propanedionic acid diethyl ester.

Colorless oil; bp 95 °C /0.7 mmHg; 1H NMR ($CDCl_3$) δ : 1.11 (d, $J = 7.0$ Hz, 6H), 1.25 (t, $J = 7.0$ Hz, 6H), 2.48 (m, 1H), 2.74 (d, $J = 1.9$ Hz, 2H), 2.78 (d, $J = 7.3$ Hz, 2H), 4.19 (q, $J = 7.0$ Hz, 4H), 5.08-5.19 (c, 2H), 5.56-5.72 (m, 1H). ^{13}C NMR ($CDCl_3$) δ : 14.1, 20.4, 23.2, 36.4, 57.1, 61.4, 73.6, 89.3, 119.4, 132.1, 170.0. IR (neat): 3080 w, 2978 s, 2935 m, 2908 m, 2873 m, 1738 s, 1641 w, 1466 m, 1442 m, 1387 m, 1367 m, 1325 m, 1288 s, 1250 m, 1215 s, 1194 s, 1146 m, 1097 m, 1068 m, 1057 m, 1046 m, 924 m, 858 m, 806 w, 764 w, 656 w, 582 w, 525 w. MS, m/z (relative intensity, %): 281 ($M^+ + 1$, 2), 280 (M^+ , 1), 135 (15), 133 (37), 132 (27), 131 (98), 205 (90), 177 (26), 159 (20), 119 (30), 117 (3), 105 (67), 93 (29), 91 (100), 83 (22), 80 (27), 79 (70), 77 (45), 69 (20), 67 (39), 65 (39), 55 (58), 53 (45), 51 (21). Exact Mass Calcd for $C_{16}H_{24}O_4$: 280.1675. Found: 281.1756 ($M+H$).

3-(3-Methyl-2-butenyl)-3-cyclopentene-1,1-dicarboxylic acid diethyl ester

(9). Colorless oil; R_f 0.17 (hexane/ $Et_2O = 10/1$); 1H NMR ($CDCl_3$) δ : 1.24 (t, $J = 7.0$ Hz, 6H), 1.50-1.62 (c, 6H), 2.71 (s, 2H), 2.83 (s, 2H), 2.99 (m, 2H), 4.19 (q, $J = 7.0$ Hz, 4H), 5.23-5.26 (c, 2H). ^{13}C NMR ($CDCl_3$) δ : 13.4, 14.0, 15.3, 40.4, 41.2, 42.5, 59.2, 61.4, 120.4, 122.0, 133.0, 140.3, 172.3. IR (neat): 3051 w, 2981 s, 2933 s, 2912 s, 2868 m, 1734 s, 1658 w, 1464 m, 1446 m, 1387 m, 1367 m, 1298 s, 1252 s, 1182 s, 1157 s, 1095 s, 1070 s, 1016 m, 862 m, 837 w, 806 w, 773 w, 721 w, 578 w, 455 w. MS, m/z (relative intensity, %): 280 (M^+ , 5), 206 (19), 177 (15), 160 (14), 133 (100), 132 (27), 117 (17), 105 (31), 93 (20), 91 (38), 79 (28), 77 (28), 69 (25), 65 (16), 55 (24), 53 (17). Exact Mass Calcd for $C_{16}H_{24}O_4$: 280.1675. Found: 280.1674.

4,4-Bis(acetoxymethyl)-1-undecene-6-yne. To a suspension of $LiAlH_4$ (2.28 g, 60 mmol) in Et_2O (100 mL) was added dropwise a solution of (2-heptynyl)-(2-propenyl)propanedionic acid diethyl ester (15.4 g, 50 mmol) in Et_2O (6 mL) at 0 °C and the resulting mixture was stirred at 0 °C for 20 min and then stirred at room temperature overnight.

Water (2.3 mL), 15% NaOH aq (2.3 mL), and water (6.9 mL) were added dropwise to the mixture. The resulting mixture was filtered and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (hexane/ $EtOAc = 2/1$, $R_f = 0.17$) to afford 2-(2-octynyl)-2-(2-pentenyl)propane-1,3-diol (44 %, 4.88 g, 21.8 mmol) as colorless oil.

A solution of acetic anhydride (2.8 mL, 30 mmol) and 2-(2-octynyl)-2-(2-pentenyl)propane-1,3-diol (2.24 g, 10 mmol) in pyridine (4.0 mL, 50 mmol) was stirred at room temperature overnight. The resulting mixture was quenched with water (20 mL) and extracted with hexane (20 mL). The organic layer was washed with cold 5% aqueous HCl solution (20 mL x 2), water (20 mL) and brine (20 mL), dried with $MgSO_4$, concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (hexane/ $Et_2O = 8/1$) to provide 4,4-bis(acetoxymethyl)-1-undecene-6-yne as colorless oil.¹

Colorless oil; bp 140 °C /1.0 mmHg; 1H NMR ($CDCl_3$) δ : 0.89 (t, $J = 7.0$ Hz, 3H), 1.27-1.33 (c, 4H), 1.46-1.51 (m, 2H), 2.06 (s, 6H), 2.14 (t, $J = 7.0$ Hz, 2H), 2.20-2.22 (c, 4H), 4.00 (s, 4H), 5.09 (d, $J = 4.3$ Hz, 1H), 5.14 (s, 1H), 5.70-5.83 (m, 1H). ^{13}C NMR ($CDCl_3$) δ : 14.0, 18.7, 20.8, 22.2, 22.3, 28.6, 31.0, 40.1, 65.6, 74.6, 83.5, 119.2, 132.3, 170.8. IR (neat): 3080 w, 2958 m, 2933 m, 2860 w, 1745 s, 1641 w, 1469 m, 1436 m, 1383 m, 1365 m, 1234 s, 1041 m, 922 w, 640 w, 603 w, 422 w. MS,

m/z (relative intensity, %): 309 ($M^+ + 1$, 1), 248 (1), 207 (3), 187 (7), 159 (11), 145 (15), 132 (50), 131 (46), 119 (20), 118 (22), 116 (100), 105 (42), 104 (26), 95 (19), 91 (67), 79 (45), 78 (19), 77 (20), 67 (35), 54 (72). Exact Mass Calcd for $C_{18}H_{28}O_4$: 308.1988. Found: 309.2068 ($M+H$).

1,1-Bis(acetoxymethyl)-3-(2-heptenyl)-3-cyclopentene (10).

Colorless oil; R_f 0.10 (hexane/Et₂O = 7/1); ¹H NMR (CDCl₃) δ : 0.89 (t, J = 6.8 Hz, 3H), 1.10-1.15 (c, 4H), 1.77-1.81 (c, 2H), 2.06 (s, 6H), 2.15 (s, 2H), 2.21 (s, 2H), [2.69 ((*E*)-**9**, brs), 2.72 ((*Z*)-**9**, c), 2H], 4.02 (s, 4H), 5.21 (s, 1H), 5.23-5.30 (c, 2H). ¹³C NMR (CDCl₃) δ : 13.9, 14.0, 20.9, 22.1, [26.8, 29.0 ((*Z*)-**9**), 31.6, 32.1 ((*E*)-**9**)], 38.7, 41.3, 44.9, 67.2, [121.2 ((*Z*)-**9**), 121.3 ((*E*)-**9**)], [126.0 ((*Z*)-**9**), 126.7 ((*E*)-**9**)], [131.3((*Z*)-**9**), 132.2 ((*E*)-**9**)] 141.5, 171.2. IR (neat): 2956 s, 2927 s, 2871 m, 2854 s, 1749 s, 1741 s, 1655 w, 1466 m, 1441 m, 1379 s, 1363 s, 1240 s, 1038 s, 970 m, 847 w, 812 w, 781 w, 735 w, 640 w, 604 w, 447 w. MS, m/z (relative intensity, %): 308 (M^+ , 0.1), 248 (3), 188 (13), 131 (26), 117 (25), 105 (19), 104 (30), 91 (100), 81 (16), 79 (18), 67 (15), 55 (100). Exact Mass Calcd for $C_{18}H_{28}O_4$: 308.1988. Found: 308.1991.

4,4-(Bisphenylsulfonyl)-1-nonene-6-yne. To a solution of bis-(phenylsulfonyl)methane (4.74 g, 16 mmol) in 25 mL DMF was added portionwise NaH (60 % oil suspension, 0.64 g, 16 mmol) over 5 min. The solution was stirred at room temperature for 5 min and was added dropwise 1-bromo-2-pentyne (2.21 g, 15 mmol) to the resulting mixture. After stirred at 70 °C for 15 h, the mixture was quenched with saturated aqueous NH₄Cl solution (30 mL), extracted with dichloromethane (50 mL), washed with brine (30 mL), dried with MgSO₄, and concentrated in *vacuo*. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 4/1, R_f = 0.09) to afford 1,1-(bisphenyl sulfonyl)-3-hexyne (42 %, 2.27 g, 6.3 mmol) as a white powder.

To a solution of 1,1-(bisphenylsulfonyl)-3-hexyne (2.27 g, 6.3 mmol) and allylmethylcarbonate (0.82 g, 7.0 mmol) in THF (15 mL) was added tetrakis(triphenylphosphine)palladium (0.15 g, 0.13 mmol). The resulting mixture was stirred at 55 °C for 12 h, concentrated in *vacuo*, and purified by flash column chromatography on silica gel (hexane/EtOAc = 4/1, R_f = 0.17) to provide 4,4-(bisphenylsulfonyl)-1-nonene-6-yne (51 %, 1.27 g, 3.2 mmol) as a white powder.²

White solid; mp 97 °C; ¹H NMR (CDCl₃) δ : 1.01 (t, J = 7.6 Hz, 3H), 2.01 (q, J = 7.6 Hz, 2H), 3.08 (d, J = 6.6 Hz, 2H), 3.17 (s, 2H), 5.28 (d, J = 11.9 Hz, 2H), 6.00-6.15 (m, 1H), 7.58 (t, J = 7.7 Hz, 4H), 7.71 (t, J = 7.7 Hz, 2H), 8.10 (d, J = 7.7 Hz, 4H). ¹³C NMR (CDCl₃) δ : 12.4, 13.5, 33.5, 70.9, 87.8, 89.2, 120.6, 128.5, 129.9, 131.5, 134.6, 136.9. IR (KBr): 3059 w, 2978 m, 2937 w, 2912 w, 1639 w, 1583 m, 1477 w, 1446 s, 1335 s, 1308 s, 1250 m, 1153 s, 1144 s, 1078 s, 993 w, 935 m, 845 m, 756 s, 737 s, 727 s, 698 s, 687 s, 671 m, 617 m, 576 s, 546 m, 538 s, 451 m, 418 w. MS, m/z (relative intensity, %): 403 ($M^+ + 1$, 0.1), 261 (4), 167 (2), 143 (6), 141 (8), 125 (40), 119 (26), 91 (58), 78 (19), 77 (100), 65 (17), 51 (43). Exact Mass Calcd for $C_{21}H_{22}O_4S_2$: 402.0960. Found: 402.0967.

1,1-(Bisphenylsulfonyl)-3-(2-butenyl)-3-cyclopenten (11).

Colorless oil; R_f 0.17 (hexane/EtOAc = 4/1); ¹H NMR (CDCl₃) δ : [1.47 ((*Z*)-**11**, d, J = 6.3 Hz), 1.61 ((*E*)-**11**, d, J = 5.9 Hz), 3H], 2.44 (d, J = 5.9 Hz, 2H), 3.21 (s, 2H), 3.32 (s, 2H), 4.94 (s, 1H), 4.95-5.06 (m, 1H), [5.25-5.38 ((*E*)-**11**, m, 1H), 5.42-5.51 ((*Z*)-**11**, m, 1H)], 7.55 (t, J = 7.7 Hz, 4H), 7.69 (t, J = 7.3 Hz, 2H), 7.99 (d, J = 7.6 Hz, 2H). ¹³C NMR (CDCl₃) δ : [12.6 ((*Z*)-**11**), 17.8 ((*E*)-**11**)], [27.7 ((*Z*)-**11**), 33.5 ((*E*)-

11], 38.8, 40.7, 91.4, 120.5, 126.4, 127.5, 128.7, 130.8, 136.9, 140.8. IR (KBr): 3062 w, 2969 w, 2927 w, 1581 w, 1477 w, 1444 s, 1325 s, 1309 s, 1157 s, 1144 s, 1076 s, 993 m, 970 m, 779 m, 756 m, 727 m, 717 m, 688 s, 650 m, 607 m, 575 s, 536 s, 486 w, 451 w, 418. MS, m/z (relative intensity, %): 403 ($M^+ + 1$, 0.3), 261 (29), 159 (26), 135 (17), 125 (44), 119 (80), 118 (33), 117 (59), 91 (79), 79 (25), 78 (27), 77 (100), 65 (21), 55 (34), 51 (53). Exact Mass Calcd for $C_{21}H_{22}O_4S_2$: 402.0960. Found: 402.0953.

(6-Acetoxy-2-hexynyl)-(2-propenyl)propanedionic acid diethyl ester

White solid; mp 165 °C; R_f 0.19 (hexane/EtOAc = 7/1); 1H NMR ($CDCl_3$) δ : 1.24 (t, J = 7.0 Hz, 6H), 1.78 (tt, J = 6.3 Hz, 6.9 Hz, 2H), 2.23 (t, J = 6.9 Hz, 2H), 2.74-2.78 (c, 4H), 4.12 (t, J = 6.3 Hz, 2H), 4.19 (q, J = 7.0 Hz, 4H), 5.09-5.18 (c, 2H), 5.55-5.67 (m, 1H). ^{13}C NMR ($CDCl_3$) δ : 14.1, 15.4, 20.9, 22.8, 27.9, 36.4, 56.9, 61.5, 63.1, 75.2, 81.9, 119.5, 132.0, 170.0, 171.0. IR (neat): 3080 w, 2981 s, 2937 m, 2875 w, 2852 w, 1738 s, 1641 w, 1468 m, 1439 m, 1390 m, 1367 s, 1325 m, 1288 s, 1246 s, 1217 s, 1192 s, 1136 m, 1095 m, 1043 s, 926 m, 858 m, 658 w, 607 w, 582 w, 418 w. MS, m/z (relative intensity, %): 338 (M^+ , 0.04), 251 (18), 219 (49), 205 (57), 204 (42), 178 (21), 177 (41), 173 (26), 163 (32), 159 (35), 153 (50), 149 (22), 132 (34), 131 (100), 117 (21), 108 (20), 105 (29), 97 (23), 79 (24), 77 (26). Exact Mass Calcd for $C_{18}H_{26}O_6$: 338.1729. Found: 338.1721.

3-(5-acetoxy-2-pentenyl)-3-cyclopentene-1,1-dicarboxylic acid diethyl ester (12) Colorless oil; R_f 0.19 (hexane/EtOAc = 7/1); 1H NMR ($CDCl_3$) δ : 1.24 (t, J = 7.1 Hz, 6H), 2.04 (s, 3H), 2.33 (dt, J = 5.7 Hz, 6.8 Hz, 2H), 2.73-2.80 (c, 2H), 2.89 (s, 2H), 2.97 (s, 2H), 4.06 (t, J = 6.8 Hz, 2H), 4.18 (q, J = 7.1 Hz, 4H), 5.21 (s, 1H), 5.37-5.57 (c, 2H). ^{13}C NMR ($CDCl_3$) δ : 14.0, 20.1, [26.8, 28.6 ((Z)-**11**), 31.6, 32.1 ((E)-**11**)], 40.6, 42.9, 59.1, 61.4 [120.5 ((Z)-**11**), 120.7 ((E)-**11**)], [125.7 ((Z)-**11**), 126.4 ((E)-**11**)], [131.4 ((Z)-**11**), 132.4 ((E)-**11**)] 141.0, 172.3. IR (neat): 2981 m, 2937 w, 2904 w, 1734 s, 1464 w, 1444 w, 1387 w, 1365 m, 1296 m, 1250 s, 1182 m, 1159 m, 1097 w, 1070 m, 972 w, 862 w, 814 w, 781 w, 606 w. MS, m/z (relative intensity, %): 338 (M^+ , 0.4), 293 (2), 278 (8), 219 (27), 218 (31), 204 (41), 159 (23), 158 (43), 131 (100), 130 (33), 117 (41), 115 (20), 105 (28), 91 (78), 84 (20), 79 (31), 77 (29), 67 (35), 65 (24), 55 (23). Exact Mass Calcd for $C_{18}H_{26}O_6$: 338.1729. Found: 338.1727.

(2-Pentynyl)-(3- ^{13}C -2-propenyl)propanedionic acid diethyl ester (12) was prepared from 3,3-bis(ethoxycarbonyl)propanal, $^{13}CH_3PPh_3I$ and 1-bromo-2-pentyne as reported by Oi.³ 1H NMR ($CDCl_3$) δ : 1.09 (t, J = 7.4 Hz, 3H), 1.25 (t, J = 6.9 Hz, 6H), 2.09-2.17 (m, 2H), 2.74-2.81 (c, 4H), 4.20 (q, J = 6.9 Hz, 4H), 5.11 (dd, J = 10.3 Hz, $J_{CH} = 157.4$ Hz, 1H), 5.15 (dd, J = 16.9 Hz, $J_{CH} = 154.4$ Hz, 1H), 5.57-5.72 (m, 1H). ^{13}C NMR ($CDCl_3$) δ : 12.3, 14.1, 22.9, 36.4, 57.0, 61.4, 73.7, 84.9, 119.4 (strong), 132.1, 170.0.

3-(2- ^{13}C -2-Butenyl)-3-cyclopentene-1,1-dicarboxylic acid diethyl ester (14). Colorless oil; R_f 0.17 (hexane/Et₂O = 10/1); 1H NMR ($CDCl_3$) δ : 1.24 (t, J = 7.0 Hz, 6H), [1.61 ((Z)-**14**, d, J = 6.5 Hz), 1.65 ((E)-**14**, d, J = 4.6 Hz), 3H], [2.71 ((E)-**14**, brs), 2.78 ((E)-**14**, c), 2H], 2.90 (s, 2H), 2.98 (s, 2H), 4.19 (q, J = 7.0 Hz, 4H), 5.21 (s, 1H), 5.38-5.52 (c, 1H). ^{13}C NMR ($CDCl_3$) δ : 14.1, 17.9, [28.4 ((Z)-**14**), 34.0 ((E)-**14**)] (strong), 40.6, 43.0, 59.2, 61.5, 120.7, 126.6, 127.6, 140.8, 172.1.

(2-Pentynyl)-(3,3-d₂-2-propenyl)propanedionic acid diethyl ester (15) was prepared from 3,3-bis(ethoxycarbonyl)propanal, CD₃PPh₃I and 1-bromo-2-pentyne in the same procedure of **13**.³ ¹H NMR (CDCl₃) δ: 1.09 (t, *J* = 7.4 Hz, 3H), 1.25 (t, *J* = 6.9 Hz, 6H), 2.09-2.17 (m, 2H), 2.74-2.81 (c, 4H), 4.20 (q, *J* = 6.9 Hz, 4H), 5.07-5.18 (c, 1H), 5.57-5.72 (c, 1H).

3-(2,2-d₂-2-Butenyl)-3-cyclopentene-1,1-dicarboxylic acid diethyl ester (16). Colorless oil; *R_f* 0.17 (hexane/Et₂O = 10/1); ¹H NMR (CDCl₃) δ: 1.24 (t, *J* = 7.0 Hz, 6H), [1.61 ((*Z*)-**16**, d, *J* = 6.5 Hz), 1.65 ((*E*)-**16**, d, *J* = 4.6 Hz), 3H], [2.71 ((*E*)-**16**, brs), 2.78 ((*E*)-**16**, c), 1H], 2.90 (s, 2H), 2.98 (s, 2H), 4.19 (q, *J* = 7.0 Hz, 4H), 5.21 (s, 1H), 5.38-5.52 (c, 1H).

(1-Methyl-2-octynyl)-(2-propenyl)propanedionic acid diethyl ester (17). Colorless oil; bp 115 °C/0.4 mmHg; ¹H NMR (CDCl₃) δ: 0.89 (t, *J* = 6.9 Hz, 3H), 1.23-1.33 (c, 11H), 1.44-1.49 (m, 2H), 2.13 (dt, *J* = 1.9 Hz, 6.9 Hz, 2H), 2.79 (d, *J* = 5.1 Hz, 2H), 3.14 (tq, *J* = 1.9 Hz, 7.0 Hz, 1H), 4.14-4.25 (c, 4H), 5.04-5.15 (c, 2H), 5.73-5.89 (m, 1H). ¹³C NMR (CDCl₃) δ: 14.0, 14.1, 18.0, 18.6, 22.2, 28.6, 30.6, 30.9, 38.5, 61.0, 61.1, 80.0, 83.2, 118.5, 133.3, 169.6, 170.0. IR (neat): 3080 w, 2981 s, 2958 s, 2935 s, 2860 m, 1734 s, 1641 m, 1462 m, 1367 m, 1335 m, 1302 m, 1275 m, 1227 s, 1173 m, 1134 m, 1097 m, 1038 m, 920 m, 860 m, 775 w, 663 w, 575 w, 457 w, 418 w. MS, *m/z* (relative intensity, %): 323 (M⁺+1, 11), 322 (M⁺, 0.5), 281 (4), 192 (36), 177 (21), 175 (27), 173 (24), 153 (38), 137 (27), 133 (33), 127 (20), 125 (26), 121 (24), 119 (84), 109 (22), 108 (27), 107 (34), 105 (51), 93 (36), 91 (49), 81 (68), 79 (64), 77 (40), 69 (26), 67 (48), 66 (23), 65 (32), 57 (27), 55 (100), 53 (52). Exact Mass Calcd for C₁₉H₃₀O₄: 322.2144. Found: 323.2216(M+H).

2-Methyl-3-(2-heptenyl)-3-cyclopentene-1,1-dicarboxylic acid diethyl ester (18) Colorless oil; *R_f* 0.26 (hexane/Et₂O = 10/1); ¹H NMR (CDCl₃) δ: 0.88 (t, *J* = 6.8 Hz, 3H), 0.96 (d, *J* = 7.9 Hz, 3H), 1.20-1.27 (c, 6H), 1.31-1.35 (m, 4H), 2.00 (dt, *J* = 6.2 Hz, 6.2 Hz, 2H), 2.64 (d, *J* = 15.6 Hz, 1H), [2.73 ((*E*)-**18**, d, *J* = 2.0 Hz), 2.78 ((*Z*)-**18**, d, *J* = 3.4 Hz), 2H], 3.19 (d, *J* = 15.6 Hz, 1H), 3.38 (q, *J* = 6.8 Hz, 1H), 4.08-4.24 (c, 4H), 5.17 (s, 1H), 5.37-5.51 (c, 2H). ¹³C NMR (CDCl₃) δ: 13.9, 14.0, 14.1, 22.2, [26.8, 27.0 ((*Z*)-**18**), 32.2, 32.4 ((*E*)-**18**)], [31.6 ((*Z*)-**18**), 31.7 ((*E*)-**18**)], [38.4 ((*Z*)-**18**), 38.5 ((*E*)-**18**)], [46.2 ((*Z*)-**18**), 46.3 ((*E*)-**18**)], 61.1, 61.2, 64.2, [119.5 ((*Z*)-**18**), 119.6 ((*E*)-**18**)], [126.0 ((*Z*)-**18**), 126.5 ((*E*)-**18**)], [131.3 ((*Z*)-**18**), 132.3 ((*E*)-**18**)], [145.9 ((*Z*)-**18**), 146.2 ((*E*)-**18**)], 170.6, 172.4. IR (neat): 2960 m, 2931 m, 2873 m, 1734 s, 1462 m, 1389 w, 1367 w, 1298 w, 1252 s, 1176 m, 1153 m, 1097 m, 1078 m, 1057 m, 1036 w, 968 w, 860 w, 795 w, 768 w, 696 w. MS, *m/z* (relative intensity, %): 322 (M⁺, 7), 249 (17), 248 (26), 191 (17), 175 (50), 152 (26), 133 (18), 131 (25), 119 (30), 117 (30), 107 (16), 105 (57), 93 (29), 91 (71), 81 (19), 79 (53), 78 (16), 77 (52), 67 (18), 55 (100), 53 (16). Exact Mass Calcd for C₁₉H₃₀O₄: 322.2144. Found: 322.2149.

(4,4-d₂-2-Pentynyl)-(2-propenyl)propanedionic acid diethyl ester (20) was prepared from (2-propynyl)-(2-propenyl)propanedionic acid diethyl ester (**1**) and 1,1-d₂-bromoethane as reported by Fu⁴.; ¹H NMR (CDCl₃) δ: 1.08 (s, 3H), 1.25 (t, *J* = 6.9 Hz, 6H), 2.74 (s, 2H), 2.78 (d, *J* = 7.6 Hz, 2H), 4.19 (q, *J* = 6.9 Hz, 4H), 5.09-5.19 (c, 2H), 5.57-5.72 (m, 1H).

3-(2,3-d₂-2-Butenyl)-3-cyclopentene-1,1-dicarboxylic acid diethyl ester

(21). Colorless oil; R_f 0.17 (hexane/Et₂O = 10/1); ¹H NMR (CDCl₃) δ : 1.24 (t, J = 7.0 Hz, 6H), [1.60 ((Z)-**20**, s), 1.65 ((E)-**20**, s), 3H], [2.70 ((E)-**20**, s), 2.77 ((E)-**20**, s), 2H], 2.90 (s, 2H), 2.97 (s, 2H), 4.18 (q, J = 7.0 Hz, 4H), 5.21 (s, 1H).

References

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EXPCM
NON
IRNUC
1H
IRSET
270.05 MHz
IRFIN
112.00 KHz
IRRPW
5800.0 Hz
IRATN
50 us
511
M:\MYR\MYMR-441 HPLC2 F2.als
TH5AT
SF
64.60 KHz
LKSET
59.0 Hz
LKFIN
200
LKLEV
23
LGAIN
117
LKPHS
630
LKSIG
16 Hz
CSPED
FILDG
FILDG

C:\WINNMR98\COMMON_DEFAULTS

14

(1 : 4)

MENUF BCM
 OFNUC 13C
 OFR 67.80 MHz
 OBSET 135.00 KHz
 OFIN 5200.0 Hz
 PW1 3.9 us
 DEAT 30.8 us
 PREDL 0.2000 ms
 IWT 1.0 sec
 POINT 32768
 SPO 32768
 TIMES 10000
 DUMMY 1
 18315.0 Hz
 FREQU 9150 Hz
 FLT 21.9 us
 DELAY 1.789 sec
 ACQTM 1.211 sec
 PD 16.00
 ADBIT 26
 RGAIN 1.20 Hz
 BF 0.00
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 BCM
 EXMOD Elvel, complete decoupling Set_IRR
 EXPCM 1H
 IRNUC 270.05 MHz
 IFR 112.00 KHz
 IRFIN 5800.0 Hz
 IRPW 50 us
 IRATN 511
 DFILE C:\WINNMR98\COMMON_DEFAULTS
 SF TH5AT04100
 LKSET 64.60 KHz
 LKFIN 180
 LKLEV 20
 LGAIN 124
 LKPS 538
 LKSIG 20 Hz
 CSFED
 FILDC
 FILDF

M:\YMR\YMR-423 F3-6.als

MENUF NON
 OFR 1H
 OBSET 270.05 MHz
 OFIN 112.00 KHz
 PW1 5900.0 Hz
 DEADT 5.3 us
 PREDL 108.5 us
 IWT 0.2000 ms
 POINT 1.0 sec
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 5402.4 Hz
 FLT 2700 Hz
 DELAY 74.1 us
 ACQTM 3.033 sec
 PD 3.967 sec
 ADBIT 16.00
 RGAIN 20
 BF 1.20 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON Single coupled PW1 ACQTM_1
 IFIR 1H
 IRSET 270.05 MHz
 IRFIN 112.00 KHz
 IRRPW 5900.0 Hz
 IRATN 50 us
 DFILF 511
 SF THSAT
 M:\YMR\YMR-423 F3-6.als
 THSAT 64.60 KHz
 LKSET 59.0 Hz
 LKFIN 200
 LKLEV 20
 LGAIN 117
 LKPHS 624
 LKSIG 23 Hz
 CSPED
 FILDC
 FILDG

16 (50%D)

M:\Ymp\YMP-147-2 F2 pure als

17

MENUC
OBRUC
OFR
ORSET
ORFIN
PW1
DEADT
PREDL
IWT
POINT
SPO
TIMES
DUMMY
FREQ
FLT
DELAY
ACQTM
PD
ADBIT
RGAIN
BF
T1
T2
T3
T4
EXMOD
EXPCM
IRNUC
IRSET
IRFIN
IRRPW
IRATN
DFILE
SF
LKSET
LKFIN
LKLEV
LGAIN
LKPHS
LKSIG
CSPED
FILDC
FILDF

NON
1H
270.05 MHz
112.00 KHz
5800.0 Hz
5.3 us
108.5 us
0.2000 ms
1.0 sec
16384
16384
8
1
5402.4 Hz
2700 Hz
74.1 us
3.033 sec
3.967 sec
16.00
21
1.20 Hz
0.00
0.00
90.00
100.00

NON
Single coupled PW1_ACQTM_1
1H
270.05 MHz
112.00 KHz
5800.0 Hz
50 us
511
M:\Ymp\YMP-147-2 F2 pure als
TH5AT

C:\WINNMR8\TEMPDATA\Auto2BCM_E1_FT.als
YMR-336 F2-3

18

BCM
 13C
 87.80 MHz
 135.00 KHz
 5200.0 Hz
 3.9 us
 30.8 us
 0.2000 ms
 1.0 sec
 32768
 32768
 3000
 1
 18348.8 Hz
 9160 Hz
 21.8 us
 1.786 sec
 1.211 sec
 16.00
 27
 1.20 Hz
 0.00
 0.00
 90.00
 100.00
 BCM
 EXMOD
 EXP-PC
 IRNUC
 1H
 270.05 MHz
 112.00 KHz
 5800.0 Hz
 50 us
 511
 C:\WINNMR8\TEMPDATA\Auto2
 THSATO5022YMR-336 F2-3
 64.60 KHz
 59.0 Hz
 200
 22
 117
 518
 14 Hz
 FIDF

M:\YMR\YMR-426 F4-7.als

MENUF
 OBRUC
 OFR 270.05 MHz
 OBSSET 112.00 KHz
 OBSIN 5800.0 Hz
 PW1 5.3 us
 DEADT 108.5 us
 PREDL 0.2000 ms
 IWT 1.0 sec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 5402.4 Hz
 FLT 2700 Hz
 DELAY 74.1 us
 ACQTM 3.033 sec
 PD 3.967 sec
 ADBIT 16.00
 RGAIN 14
 BF 1.20 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD
 EXPCM
 IRNUC
 IFR 270.05 MHz
 IRSET 112.00 KHz
 IRFIN 5800.0 Hz
 IRBPW 50 us
 IRATN 511
 SF
 DFIL
 THSAT
 LKSET 64.60 KHz
 LKFIN 59.0 Hz
 LKLEV 200
 LGAIN 22
 LKPHS 117
 LKSIG 6200
 CSPED 17 Hz
 FILDC
 FILDF

21 (94%D)

M:\YMR\YMR-344 F5-6 13C.als

23

MENUF	BCM	67.80 MHz
OBNUC	13C	135.00 KHz
OFR		5200.0 Hz
OBSET		3.9 us
OBFIN		30.8 us
PW1		0.2000 ms
DEADT		1.0 sec
PREDL		327.68
IWT		327.68
POINT		10000
SFO		1
TIMES		18315.0 Hz
DUMMY		9150 Hz
FREQU		21.9 us
FLT		1.789 sec
DELAY		1.211 sec
ACQTM		16.00
PD		25
ADBIT		0.12 Hz
RGAIN		0.00
BF		0.00
T1		0.00
T2		90.00
T3		100.00
T4		BCM
EXMOD		Bilevel complete decoupling Set_IRR
EXPCM		1H
IRNUC		270.05 MHz
IRSET		112.00 KHz
IRFIN		5800.0 Hz
IRRPW		50.0 us
IRATN		511
DEILE		M:\YMR\YMR-344 F5-6 13C.als
SF		TH5AT05022
LKSET		64.60 KHz
LKFIN		59.0 Hz
LKLEV		200
LGAIN		23
LKPHS		117
LKSIG		491
CSPED		16 Hz
FILDC		
FILDF