Supporting Information

Nickel Catalyzed Grob Fragmentation via $\beta$-Carbon Elimination:
Efficient Entry to $\omega$-Dienyl Aldehydes

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Experimental Section

Reactions employed oven-dried glassware unless otherwise noted. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates with UV indicator (Merck, Silica gel 60F$_{254}$). Flash chromatography columns were packed with silica gel (Wakogel-C300) as a slurry in hexane. Gradient flash chromatography was conducted eluting with a continuous gradient from hexane to the indicated solvent. Proton and carbon NMR data were obtained with a JEOL-GX400 with tetramethylsilane as an internal standard. Chemical shift values were given in ppm downfield from the internal standard. Infrared spectra were recorded with a JASCO A-100 FT-IR spectrophotometer. High resolution mass spectra (HRMS) were measured with a JEOL JMS-DX303. Combustion analyses were performed by the Instrumental Analysis Center of Nagasaki University. Analysis agreed with the calculated values within $\pm$ 0.4%.
**Solvents and Reagents.** Tetrahydrofuran and ether were distilled from a blue solution of sodium benzophenone ketyl under N$_2$ immediately prior to use. Acetonitrile was distilled under nitrogen from calcium hydride. Ni(cod)$_2$ (Kanto Kagaku Kogyo, Co., Ltd.), triphenylphosphine, tri(n-butyl)phosphine, tri(c-hexyl)phosphine, 1,2-bis(diphenylphosphino)ethane, 1,3-bis(diphenylphosphino)propane, 1,4-bis(diphenylphosphino)butane, 1,1’-bis(diphenylphosphino)ferrocene (Aldrich Co., Ltd.) were purchased and used without further purification. Cyclic carbonates 1a - 1h were prepared according to the method reported previously from our laboratories.$^{1-3}$ One typical example is shown below.

**Preparation of Cyclic Carbonate (1d):** A 300 mL of three-necked round-bottomed flask, equipped with a dropping funnel, a rubber septum, and an air condenser at the top of which is attached a three-way stopcock fitted with a nitrogen balloon, is charged with freshly distilled THF (30 mL) and diisopropylamine (4.6 mL, 33 mmol) via syringe under nitrogen. Into the flask was added $n$-butyllithium (20 mL, 33 mmol; 1.6 M hexane solution) at -78 °C, and the mixture was stirred for 1 hour. To the reaction mixture was added cyclodecanone (4.6 g, 30 mmol) dissolved in THF (10 mL) via a dropping funnel at -78 °C, and the mixture was stirred at 0 °C for 1 h. A solution of acrolein (2.4 mL, 36 mmol) in dry THF (10 mL) was quickly added at -78 °C and the mixture was stirred for 1 minute. The reaction was quenched with 2M HCl at -78 °C and the mixture was extracted with ethyl acetate (2 × 30 mL). The combined extracts were washed with sat. NaHCO$_3$ then with sat. NaCl, and dried (MgSO$_4$). The solvent was removed *in vacuo*, and the residue was
subjected to column chromatography on silica gel (hexane/ethyl acetate = 8:1, v/v) to give an aldol product in 90% yield.

The aldol product (5.7 g, 27 mmol) dissolved in dry ether (10 mL) was added into a suspension of lithium aluminum hydride (1.5 g, 40 mmol) in ether (30 mL) at 0 °C. After stirring for 30 min at the same temperature, the excess lithium aluminum hydride was decomposed by adding aqueous THF (THF/water = 1:1, v/v) dropwise until gray slurry turned into white granules. After filtration with suction through a celite pad on a glass filter, the filtrate was washed with 15% aqueous NaOH then with sat. NaCl. The organic phase was dried (MgSO$_4$), and the solvent was removed in vacuo. The residue was subjected to column chromatography on silica gel (hexane/ethyl acetate = 8:1, v/v) to give a diol in 79% yield.

To a solution of the diol (4.23 g, 20 mmol) and triethylamine (24 mL, 170 mmol) in dichloromethane (30 mL) was added methyl chloroformate (11 mL, 140 mmol) at 0 °C. The solution was stirred at room temperature for 24 hours, and then water (20 mL) and 2M HCl (15 mL) were added at 0 °C. The mixture was extracted with dichloromethane (2 × 30 mL) and the combined organic layer was dried (MgSO$_4$) and concentrated in vacuo. The residue was subjected to column chromatography on silica gel (hexane/ethyl acetate = 8:1, v/v) to give cyclic carbonate 1d (2.57 g) in 54% yield.

(1S,7S,11S)-11-Vinyl-8,10-dioxatricyclo[5.4.0.0$^{2,6}$]-3-undecen-9-one (1a): Yields; aldol, 94 %; LAH reduction, 70 %; carbonation, 66 %; IR (neat) 3450 (m), 2980 (m), 2840 (m), 1760 (s), 1380 (m), 1200 (s), 1100 (s), 990 (m), 930 (m), 770 (m), 720 (s) cm$^{-1}$; $^1$H NMR
(400 MHz, CDCl$_3$) $\delta$ 2.47 (ddd, $J$ = 2.2, 4.0, 17.2 Hz, 1 H), 2.57 (dt, $J$ = 7.3, 1.8 Hz, 1 H), 2.69 (ddq, $J$ = 7.3, 17.2, 2.2 Hz, 1 H), 3.13 - 3.22 (m, 2 H), 4.52 (dd, $J$ = 4.0, 7.3 Hz, 1 H; coalescing to d, $J$ = 4.0 Hz, by irradiation at 2.57), 4.86 (ddt, $J$ = 6.2, 7.3, 1.1 Hz, 1 H; coalescing to d, $J$ = 6.2 Hz, by irradiation at 2.57), 5.37 (d, $J$ = 10.6 Hz, 1 H), 5.44 (dd, $J$ = 1.1, 16.9 Hz, 1 H), 5.77 (dq, $J$ = 5.5, 2.2 Hz, 1 H), 5.82 (dq, $J$ = 5.5, 2.2 Hz, 1 H), 5.89 (ddd, $J$ = 6.2, 10.9, 16.9 Hz, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 38.0, 43.1, 44.1, 44.2, 80.4, 81.5, 119.2, 132.6, 132.7, 150.0; HRMS calcd for C$_{11}$H$_{12}$O$_3$: 192.0786. Found m/z (relative intensity) 192.0782 (M$^+$, 3), 148 (8), 127 (81), 92 (100), 84 (99).

(1S,5R,6S)-5-Vinyl-benzo[8,9]-2,4-dioxabicyclo[4.3.0]nonan-3-one (1b): Yields; aldol, 85%; LAH reduction, 33%; carbonation, 68%; mp 86.0 – 87.0 °C (benzene-hexane); IR (KBr disk) 3400 (w), 2970 (w), 2900 (m), 1740 (s), 1590 (s), 1180 (m), 1030 (m), 900 (w), 720 (m) cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 2.97 (dd, $J$ = 7.7, 15.8 Hz, 1 H), 3.10 (dq, $J$ = 2.9, 7.7 Hz, 1 H), 3.18 (dd, $J$ = 7.7, 15.8 Hz, 1 H), 5.16 (d, $J$ = 7.7 Hz, 1 H), 5.52 (d, $J$ = 17.2 Hz, 1 H), 5.86 (ddd, $J$ = 1.5, 2.9, 5.1 Hz, 1 H), 5.87 (d, $J$ = 7.7 Hz, 1 H), 7.26 (d, $J$ = 7.7 Hz, 1 H), 7.28 (t, $J$ = 7.7 Hz, 1 H), 7.35 (dt, $J$ = 1.5, 7.7 Hz, 1 H), 7.50 (d, $J$ = 7.7 Hz, 1 H). Anal. calcd for C$_{13}$H$_{12}$O$_3$: C, 72.21; H, 5.59. Found: C, 71.96; H, 5.60.

(2R,6S,7S)-1,11,11-Trimethyl-6-vinyl-3,5-dioxatricyclo[6.2.1.0$^{2,7}$]undecan-4-one (1c):$^4$ Yields; aldol, 100%; LAH reduction, 100%; carbonation, 60%; mp 72.0–73.0 °C (hexane); IR (KBr disk) 3460 (m), 3020 (m), 2940 (s), 2910 (s), 2850 (m), 1760 (s), 1370 (s), 1220 (s), 1110 (s), 1070 (s), 990 (s), 930 (s) cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.92 (s, 3 H),
1.06 - 1.16 (m, 2 H), 1.07 (s, 3 H), 1.62 (m, 1 H), 1.76 (d, \(J = 4.0\) Hz, 1 H), 1.83 (m, 1 H), 2.15 (dd, \(J = 8.1, 11.4\) Hz, 1 H), 4.21 (d, \(J = 8.1\) Hz, 1 H), 4.83 (dd, \(J = 6.6, 11.4\) Hz, 1 H), 5.33 (dt, \(J = 10.3, 1.1\) Hz, 1 H), 5.40 (dt, \(J = 17.2, 1.1\) Hz, 1 H), 5.88 (ddd, \(J = 6.6, 10.3, 17.2\) Hz, 1 H). Anal. calcd for C\(_{14}\)H\(_{20}\)O\(_3\): C, 71.16; H, 8.53. Found: C, 71.17; H, 8.47.

14-Vinyl-11,13-dioxabicyclo[8.4.0]tetradecan-12-one (1d): a solid consisting of 4 isomers in 2:1:1:1 ratio: IR (KBr disk) 3740 (w), 2985 (m), 2910 (m), 2870 (m), 2360 (m), 1735 (s), 1170 (w), 1125 (w), 1065 (w), 935 (w), 770 (w), 670 (w), 635 (w) cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\), the major isomer) \(\delta\) 1.30 - 1.55 (m, 13 H), 1.81 - 1.88 (m, 2 H), 1.99 - 2.11 (m, 2 H), 4.49 (ddd, \(J = 3.2, 4.4, 8.3\) Hz, 1 H), 4.75 (t, \(J = 4.7\) Hz, 1 H), 5.39 (d, \(J = 10.2\) Hz, 1 H), 5.40 (d, \(J = 17.6\) Hz, 1 H), 5.85 (ddd, \(J = 4.7, 10.2, 17.6\) Hz, 1 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), the major isomer) \(\delta\) 22.6, 22.7, 23.2, 24.0 24.6, 24.9, 25.5, 27.7, 36.4, 78.5, 82.0, 118.7, 134.7, 148.5; HRMS calcd for C\(_{14}\)H\(_{22}\)O\(_3\): 238.1569. Found m/z (relative intensity) 238.1570 (M\(^+\), 2), 239 (100), 165 (62), 152 (38), 151 (81).

(1S,12S,16S)-16-Vinyl-13,15-dioxabicyclo[10.4.0]hexadecan-14-one (1e): Yields: aldol, 100%; LAH reduction, 97%; carbonation, 78%; mp 119.0–120.0 °C (benzene–hexane); IR (KBr disk) 3450 (m), 2950 (s), 2870 (s), 1740 (s), 1130 (s), 1070 (s), 930 (s) cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 1.30 - 1.55 (m, 18 H), 1.66 (m, 1 H), 1.81 - 1.91 (m, 2 H), 4.49 (ddd, \(J = 2.9, 6.2, 7.3\) Hz, 1 H), 4.78 (t, \(J = 5.1\) Hz, 1 H), 5.40 (d, \(J = 17.2\) Hz, 1 H), 5.41 (d, \(J = 10.6\) Hz, 1 H), 5.81 (ddd, \(J = 5.1, 10.6, 17.2\) Hz, 1 H). Anal. calcd for C\(_{16}\)H\(_{26}\)O\(_3\): C, 72.14; H, 9.84. Found: C, 72.35; H, 9.88.
5-Vinyl-benzo[9,10]-2,4-dioxabicyclo[4.4.0]decan-3-one (1f): a solid consisting of 4 isomers in a ratio of 4:3:2:1: Yields: aldol, 88%; LAH reduction, 91%; carbonation, 47%; IR (KBr disk) 3420 (m), 2910 (s), 2860 (m), 1750 (s), 1430 (s), 1350 (s), 1230 (s), 1180 (s), 1140 (s), 1060 (s), 990 (s), 940 (s), 760 (s) cm⁻¹; ¹H NMR (400 MHz, CDCl₃, the major isomer) δ 1.92 – 2.10 (m, 2 H), 2.38 (dddm, J = 5.5, 8.8, 11.4 Hz, 1 H), 2.90 - 2.95 (m, 2 H), 5.10 (br t, J = 5.5 Hz, 1 H), 5.32 (d, J = 10.6 Hz, 1 H), 5.46 (br d, J = 15.0 Hz, 1 H), 5.54 (br d, J = 8.8 Hz, 1 H), 5.96 (ddd, J = 5.5, 10.6, 15.0 Hz, 1 H), 7.16 (d, J = 8.1 Hz, 1 H), 7.25 (t, J = 8.1 Hz, 1 H), 7.26 (t, J = 8.1 Hz, 1 H), 7.57 (d, J = 8.1 Hz, 1 H). Anal. calcd for C₁₄H₁₄O₃: C, 73.02; H, 6.13. Found: C, 72.78; H, 5.99.

(1S,5R,6S)-5-(Isopropenyl)-benzo[8,9]-2,4-dioxabicyclo[4.3.0]nonan-3-one (1g): Yields; aldol, 70%; LAH reduction, 100%; carbonation, 19%; mp 99.0–100.0 °C (benzene–hexane); IR (KBr disk) 2925 (w), 2900 (w), 1740 (s), 1600 (w), 1110 (m), 1080 (m), 1040 (m), 920 (w), 900 (m), 760 (m) cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 1.85 (t, J = 1.5 Hz, 3 H), 2.87 (dd, J = 7.7, 16.9 Hz, 1 H), 3.11 (dd, J = 7.7, 16.9 Hz, 1 H), 3.21 (dq, J = 2.9, 7.7 Hz, 1 H), 5.02 (d, J = 1.5 Hz, 1 H), 5.11 (dd, J = 1.5, 2.9 Hz, 1 H), 5.29 (d, J = 1.5 Hz, 1 H), 5.88 (d, J = 7.7 Hz, 1 H), 7.25 (d, J = 7.7 Hz, 1 H), 7.28 (t, J = 7.7 Hz, 1 H), 7.35 (dt, J = 1.5, 7.7 Hz, 1 H), 7.50 (d, J = 7.7 Hz, 1 H). Anal. calcd for C₁₄H₁₄O₃: C, 72.96; H, 6.08. Found: C, 72.78; H, 6.15.

(1S,5R,6S)-5-[(1E)-Propenyl]-benzo[8,9]-2,4-dioxabicyclo[4.3.0]nonan-3-one (1h):
Yields: aldol, 70%; LAH reduction, 100%; carbonation 19%; mp 73.0–74.0 °C (ethyl acetate-hexane); IR (KBr disk) 3420 (m), 3000 (m), 2925 (m), 2850 (m), 1740 (s), 1610 (w), 1220 (s), 1080 (s), 950 (s), 910 (m), 890 (w), 750 (s) cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 1.78 (br d, \(J = 6.6 \text{ Hz}, 3\) H), 2.99 - 3.10 (m, 2 H), 3.24 (m, 1 H), 5.10 (dm, \(J = 7.3\) Hz, 1 H), 5.55 (dd, \(J = 7.3, 15.4\) Hz, 1 H), 7.26 (d, \(J = 7.3\) Hz, 1 H), 7.28 (t, \(J = 7.3\) Hz, 1 H), 7.34 (t, \(J = 7.3\) Hz, 1 H), 7.50 (br d, \(J = 7.3\) Hz, 1 H). Anal. calcd for C\(_{14}\)H\(_{14}\)O\(_3\): C, 72.96; H, 6.08. Found: C, 72.73; H, 6.12.

\((2R,6S,7S)-6-(\text{Isopropenyl})-1,11,11-\text{trimethyl}-3,5-\text{dioxatricyclo}[6.2.1.0^{2,7}]\text{undecan-4-one (1i)}\): Yields; aldol, 94%; LAH reduction, 95%; carbonation, 53%; mp = 96.0–97.0 °C (benzene-hexane); IR (KBr disk) 3450 (m), 3025 (m), 2970 (w), 2825 (w), 1740 (s), 1640 (w), 1190 (s), 1090 (s), 1070 (w), 1040 (s), 1000 (m), 940 (m), 880 (m), 780 (w), 760 (w), 720 (m) cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 0.91 (s, 3 H), 1.08 (s, 3 H), 1.10 - 1.19 (m, 2 H), 1.19 (s, 3 H), 1.62 (m, 1 H), 1.80 (m, 1 H), 1.83 (s, 3 H), 1.86 (m, 1 H), 2.24 (dd, \(J = 8.1, 11.4\) Hz, 1 H), 4.20 (d, \(J = 8.1\) Hz, 1 H), 4.83 (d, \(J = 11.4\) Hz, 1 H), 5.30 (d, \(J = 1.1\) Hz, 2 H). Anal. calcd for C\(_{15}\)H\(_{23}\)O\(_3\): C, 71.91; H, 8.79; Found: C, 72.02; H, 8.81.

\(8-\text{Phenyl-5-vinyl-2,4-dioxabicyclo}[4.4.0]\text{nonan-3-one (1j)}\): a solid containing 4 isomers in 8:2:2:1 ratio: Yields; aldol, 70%; LAH reduction, 100%; carbonation 19%; IR (KBr disk) 3460 (m), 3030 (m), 2940 (m), 2870 (w), 2360 (w), 1735 (s), 1600 (w), 1370 (s), 1200 (s), 1090 (s), 990 (m), 940 (m), 850 (w), 770 (m), 710 (m), 620 (w) cm\(^{-1}\); \(^1\)H NMR (400 MHz,
CDCl₃, the major isomer) δ 1.27 (q, J = 12.2 Hz, 1 H), 1.58 - 1.71 (m, 2 H), 1.81 (ddd, J = 2.9, 10.7, 12.2 Hz, 1 H), 2.00 (dq, J = 13.4, 2.3 Hz, 1 H), 2.07 (dq, J = 13.4, 2.3 Hz, 1 H), 2.30 (m, 1 H), 2.68 (tt, J = 3.4, 12.2 Hz, 1 H), 4.19 (dt, J = 4.6, 10.7 Hz, 1 H), 4.56 (dd, J = 7.4, 10.7 Hz, 1 H), 5.34 (d, J = 10.3 Hz, 1 H), 5.38 (d, J = 17.1 Hz, 1 H), 5.77 (ddd, J = 7.4, 10.3, 17.1 Hz, 1 H), 7.16 - 7.33 (m, 5 H); ¹³C NMR (100 MHz, CDCl₃, the major isomer) δ 31.1, 31.2, 33.3, 41.5, 42.6, 79.9, 85.0, 120.5, 126.5, 126.6, 128.5, 128.5, 132.8, 144.3, 148.4; HRMS calcd for C₁₆H₁₈O₃: 258.1256. Found m/z (relative intensity): 258.1255 (M⁺, 100), 196 (20), 185 (34), 170 (67).

7-Vinyl-4,6-dioxatricyclo[8.4.0.0³.₈]tetradecan-5-one (1k): a complex mixture of isomers, the ratio being not determined: Yields: aldol, 49%; LAH reduction, 100%; carbonation, 66%, one diastereomer was obtained after recrystallization from benzene-hexane; IR (KBr disk) 3460 (w), 2930 (s), 2860 (s), 1750 (s), 1380 (s), 1200 (s), 1100 (s), 990 (m), 940 (m), 760 (m) cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 1.19 - 2.10 (m, 15 H), 4.10 (dm, J = 10.6 Hz, 1 H), 4.46 (dd, J = 7.3, 10.6 Hz, 1 H), 5.34 (d, J = 10.6 Hz, 1 H), 5.38 (d, J = 16.9 Hz, 1 H), 5.75 (ddd, J = 7.3, 10.6, 16.9 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃) δ 20.5, 26.3, 26.6, 30.5, 31.1, 31.9, 34.0, 34.9, 36.4, 81.1, 85.5, 120.0, 133.1, 148.6; HRMS calcd for C₁₄H₂₀O₃: 237.1491. Found m/z (relative intensity) 237.1471 (M⁺, 3), 192 (9), 174 (19), 163 (39), 135(100), 121 (26), 107 (24).

12-Vinyl-9,11-dioxabicyclo[6.4.0]dodecan-10-one (1l): a solid consisting of 4 isomers in 4:2:1:1 ratio: Yields: aldol, 100%; LAH reduction, 80%; carbonation, 83%; IR (KBr disk)
2920 (s), 2840 (s), 1750 (s), 1210 (s), 1130 (s), 1090 (s), 990 (s), 930 (s), 760 (s) cm⁻¹; ¹H NMR (400 MHz, CDCl₃, the major isomer) δ 1.26 - 2.12 (m, 13 H), 4.30 (dm, J = 10.6 Hz, 1 H), 4.96 (dm, J = 4.4 Hz, 1 H), 5.38 (dt, J = 10.3, 1.1 Hz, 1 H), 5.49 (dt, J = 17.2, 1.1 Hz, 1 H), 5.87 (ddd, J = 4.4, 10.3, 17.2 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃, a mixture of 4 isomers) δ 18.9, 21.3, 21.4, 21.8, 22.4, 24.3, 24.4, 24.9, 25.0, 25.4, 25.9, 26.1, 26.3, 26.9, 27.3, 27.4, 28.7, 28.9, 29.0, 31.4, 31.6, 37.7, 37.8, 39.7, 78.8, 81.0, 81.5, 82.8, 83.6, 83.7, 84.5, 118.1, 118.4, 120.2, 121.1, 131.0, 132.5, 133.4, 134.5, 148.6, 148.8, 149.3; HRMS calcd for C₁₂H₁₈O₃: 211.1334. Found m/z (relative intensity) 211.1324 (M⁺, 55), 138 (65), 137 (100), 123 (81).

16-(Isopropenyl)-13,15-dioxabicyclo[10.4.0]hexadecan-14-one (1m): a mixture of 3 isomers in 2:2:1 ratio: Yields; aldol, 100%; LAH reduction, 70%; carbonation, 40%; a single diastereomer was obtained after recrystallization from benzene-hexane; mp 119.0–120.0 °C (benzene-hexane); IR (KBr disk) 3450 (m), 2910 (s), 2830 (s), 1760 (s), 1470 (m), 1380 (m), 1260 (m), 1200 (s), 1120 (s), 900 (m) cm⁻¹; ¹H NMR (400 MHz, CDCl₃, the major isomer) δ 1.36 - 1.55 (m, 18 H), 1.63 (dq, J = 13.9, 7.0 Hz, 1 H), 1.85 (dq, J = 13.9, 7.0 Hz, 1 H), 2.01 (dq, J = 3.3, 6.2 Hz, 1 H), 4.47 (dt, J = 3.3, 7.0 Hz, 1 H), 4.66 (d, J = 6.2 Hz, 1 H), 5.06 (s, 1 H), 5.12 (s, 1 H). Anal. calcd for C₁₇H₂₈O₃: C, 72.82; H, 10.06. Found: C, 72.76; H, 10.01.

**General Procedure for the Ni-Catalyzed Decarboxylative Ring-Opening Reaction of Cyclic Carbonates 1** (Table 1, run 4): In a N₂ purged flask, Ni(cod)₂ (14 mg, 0.05 mmol),
triphenylphosphine (52 mg, 0.2 mmol), and 1d (119 mg, 0.5 mmol) were dissolved in dry acetonitrile (2.5 mL). The mixture was stirred at room temperature for 24 h and diluted with EtOAc (30 mL). The mixture was washed with 2M HCl, sat. NaHCO₃, and sat. NaCl and then dried (K₂CO₃) and concentrated in vacuo. The residue was purified by column chromatography over silica gel (hexane/ethyl acetate = 64:1, v/v) to give dienyl aldehyde 2d (94 mg) in 97% yield.

2-(1,3-Butadienyl)-3-cyclopentenecarbaldehyde (2a): IR (neat) 3400 (m), 3040 (m), 2990 (m), 2920 (s), 2830 (s), 2740 (s), 1720 (s), 1650 (w), 1620 (w), 1390 (s), 1000 (s), 910 (m), 800 (m), 780 (m), 720 (s) cm⁻¹; ¹H NMR (400 MHz, CDCl₃, Z-2a) δ 2.44 (ddm, J = 10.3, 16.9 Hz, 1 H), 2.84 (ddm, J = 6.6, 16.9 Hz, 1 H), 3.24 (ddt, J = 2.2, 6.6, 10.3 Hz, 1 H), 4.23 (br t, J = 10.3 Hz, 1 H), 5.20 (tm, J = 10.3 Hz, 1 H), 5.21 (d, J = 10.3 Hz, 1 H), 5.27 (br d, J = 16.9 Hz, 1 H), 5.56 (dm, J = 5.5 Hz, 1 H), 5.82 (dm, J = 5.5 Hz, 1 H), 6.05 (t, J = 10.3 Hz, 1 H), 6.72 (dt, J = 16.9, 10.3 Hz, 1 H), 9.67 (d, J = 2.2 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃, Z-2a) δ 31.6, 50.4, 53.8, 116.8, 127.0, 131.3, 132.0, 132.5, 136.2, 202.9; ¹H NMR (400 MHz, CDCl₃, E-2a) δ 2.63 (dq, J = 8.8, 2.2 Hz, 1 H), 2.80 (dm, J = 8.8 Hz, 1 H), 3.24 (m, 1 H), 3.82 (tm, J = 10.3 Hz, 1 H), 5.04 (br d, J = 10.3 Hz, 1 H), 5.16 (br d, J = 17.0 Hz, 1 H), 5.30 (dm, J = 15.1 Hz, 1 H), 5.63 (dm, J = 5.6 Hz, 1 H), 5.73 (dm, J = 5.6 Hz, 1 H), 6.15 (dd, J = 10.3, 15.1 Hz, 1 H), 6.30 (dt, J = 17.0, 10.3 Hz, 1 H), 9.70 (d, J = 2.0 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃, E-2a) δ 32.3, 48.9, 56.5, 116.4, 129.5, 131.0, 131.9, 135.3, 136.4, 201.6; HRMS calcd for C₁₀H₁₂O: 148.0880. Found m/z (relative intensity) 148.0890 (M⁺, 56), 133 (27), 117 (100), 105 (50), 92 (51).
**o-(2,4-Pentadienyl)benzaldehyde (2b):** IR (neat) 2950 (w), 2800 (w), 2675 (m), 1700 (s), 1600 (m), 1180 (m), 980 (m), 875 (m), 725 (m) cm⁻¹; ¹H NMR (400 MHz, CDCl₃, E-2b) δ 3.85 (d, J = 6.6 Hz, 1 H), 4.98 (dd, J = 1.5, 10.3 Hz, 1 H), 5.07 (dd, J = 1.5, 16.5 Hz, 1 H), 5.89 (dt, J = 15.0, 6.6 Hz, 1 H), 6.01 (dd, J = 10.3, 15.0 Hz, 1 H), 6.30 (dt, J = 16.5, 10.3 Hz, 1 H), 7.29 (d, J = 7.7 Hz, 1 H), 7.40 (dt, J = 1.5, 7.7 Hz, 1 H), 7.53 (dt, J = 1.5, 7.7 Hz, 1 H), 7.84 (dd, J = 1.5, 7.7 Hz, 1 H), 10.24 (s, 1 H); ¹³C NMR (100 MHz, CDCl₃, E-2b) δ 35.3, 116.0, 126.8, 130.9, 131.9, 132.4, 132.5, 133.8, 136.5, 142.2, 192.1; ¹H NMR (400 MHz, CDCl₃, Z-2b) δ 4.00 (br d, J = 7.7 Hz, 1 H), 5.21 (d, J = 10.3 Hz, 1 H), 5.30 (dd, J = 1.5, 16.5 Hz, 1 H), 5.57 (dt, J = 10.3, 7.7 Hz, 1 H), 6.12 (t, J = 10.3 Hz, 1 H), 6.77 (dt, J = 16.5, 10.3 Hz, 1 H), 7.32 (d, J = 7.7 Hz, 1 H), 7.39 (dt, J = 1.5, 7.7 Hz, 1 H), 7.52 (dt, J = 1.5, 7.7 Hz, 1 H), 7.82 (dd, J = 1.5, 7.7 Hz, 1 H), 10.25 (s, 1 H); ¹³C NMR (100 MHz, CDCl₃, Z-2b) δ 30.8, 118.4, 126.7, 129.5, 130.1, 130.5, 131.6, 132.3, 133.7, 142.7, 192.3; HRMS calcd for C₁₂H₁₂O: 172.0888. Found m/z (relative intensity) 172.0871 (M⁺, 84), 157 (9), 154 (15), 131 (53), 128 (39), 118 (100).

**cis-1,2,2-Trimethyl-3-[(1E)-1,3-butadienyl]cyclopentanecarbaldehyde (2c):** IR (neat) 2940 (s), 2860 (m), 2800 (w), 1720 (s), 1650 (w), 1000 (s), 890 (s) cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 0.79 (s, 3 H), 0.96 (s, 3 H), 1.08 (s, 3 H), 1.37 (ddd, J = 5.5, 9.2, 13.9 Hz, 1 H), 1.65 (dddd, J = 5.5, 9.2, 11.7, 13.9 Hz, 1 H), 1.92 (ddt, J = 5.1, 13.9, 9.2 Hz, 1 H), 2.43 (ddd, J = 5.1, 11.7, 13.9 Hz, 1 H), 2.50 (q, J = 9.2 Hz, 1 H), 4.99 (dd, J = 1.5, 10.3 Hz, 1 H), 5.13 (dd, J = 1.5, 16.9 Hz, 1 H), 5.57 (dd, J = 9.2, 15.4 Hz, 1 H), 6.04 (dd, J = 10.3, 15.4 Hz, 1 H), 6.33 (dt, J = 16.9, 10.3 Hz, 1 H), 9.64 (s, 1 H). Anal. calcd for C₁₃H₂₀O: C,
Found: C, 81.39; H, 10.46.

**10,12-Tridecadienal (2d):** IR (neat) 3430 (w), 2930 (s), 2850 (s), 1720 (s), 1700 (m), 1640 (w), 1000 (w), 970 (w), 900 (w), 720 (w), 670 (w), 620 (w) cm⁻¹; ¹H NMR (400 MHz, CDCl₃, Z-2d) δ 1.24 - 1.42 (m, 10 H), 1.64 (m, 2 H), 2.18 (qm, J = 7.8 Hz, 2 H), 2.41 (dt, J = 2.0, 7.3 Hz, 2 H), 5.07 (dd, J = 2.0, 11.2 Hz, 1 H), 5.17 (dd, J = 2.0, 16.8 Hz, 1 H), 5.46 (dt, J = 10.5, 7.8 Hz, 1 H), 6.00 (dt, J = 2.0, 7.3 Hz, 2 H), 9.76 (t, J = 2.0 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃, Z-2d) δ 22.1, 29.1, 29.1, 29.1, 29.2, 29.3, 29.5, 43.9, 116.6, 129.0, 132.2, 137.2, 202.6; ¹H NMR (400 MHz, CDCl₃, E-2d) δ 1.24 - 1.42 (m, 10 H), 1.62 (quint, J = 7.6 Hz, 2 H), 2.07 (q, J = 7.6 Hz, 2 H), 2.41 (dt, J = 2.0, 7.6 Hz, 1 H), 4.94 (dm, J = 10.3 Hz, 1 H), 5.08 (dm, J = 16.7 Hz, 1 H), 5.69 (dt, J = 15.0, 7.6 Hz, 1 H), 6.04 (dd, J = 10.3, 15.0 Hz, 1 H), 6.30 (dt, J = 16.7, 10.3 Hz, 1 H), 9.76 (t, J = 2.0 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃, E-2d) δ 27.7, 29.1, 29.1, 29.1, 29.2, 29.3, 29.5, 32.5, 114.4, 130.8, 132.8, 135.3, 202.6; HRMS calcd for C₁₃H₂₂O: 194.1671. Found m/z (relative intensity): 194.1660 (M⁺, 100), 195 (20), 152 (13), 151 (20).

**12,14-Pentadecadienal (2e):** IR (neat) 2928 (s), 2855 (s), 2361 (m), 2341 (m), 1724 (s), 1679 (s), 1003 (w), 970 (w), 900 (w) cm⁻¹; ¹H NMR (400 MHz, CDCl₃, E-2e) δ 1.24 - 1.42 (m, 14 H), 1.62 (quint, J = 7.3 Hz, 2 H), 2.08 (q, J = 7.3 Hz, 2 H), 2.41 (dt, J = 1.8, 7.3 Hz, 2 H), 4.94 (d, J = 10.2 Hz, 1 H), 5.07 (d, J = 16.4 Hz, 1 H), 5.70 (dt, J = 14.8, 7.0 Hz, 1 H), 6.04 (dd, J = 14.8, 10.2 Hz, 1 H), 6.30 (dt, J = 16.4, 10.2 Hz, 1 H), 9.76 (t, J = 1.8 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃, E-2e) δ 27.7, 29.1, 29.3, 29.4, 29.4, 29.5, 29.6, 32.5, 116.6, 1
29.0, 132.2, 135.4, 202.6; $^1$H NMR (400 MHz, CDCl$_3$, Z-2e) $\delta$ 1.24 - 1.42 (m, 14 H), 1.62 (quint, $J = 7.3$ Hz, 2 H), 2.16 (q, $J = 7.3$ Hz, 2 H), 2.41 (dt, $J = 1.8$, 7.3 Hz, 2 H), 5.07 (d, $J = 10.6$ Hz, 1 H), 5.18 (dd, $J = 1.5$, 16.9 Hz, 1 H), 5.44 (dt, $J = 1.8$, 7.3 Hz, 1 H), 5.99 (t, $J = 10.6$ Hz, 1 H), 6.64 (dt, $J = 16.9$, 10.6 Hz, 1 H), 9.76 (t, $J = 1.8$ Hz); $^{13}$C NMR (100 MHz, CDCl$_3$, Z-2e) $\delta$ 22.1, 29.1, 29.3, 29.4, 29.4, 29.5, 29.6, 43.9, 114.4, 130.7, 132.9, 137.2, 202.5; HRMS calcd for C$_{15}$H$_{26}$O: 222.1984. Found m/z (relative intensity): 222.1985 (M$^+$, 100), 98 (16), 95 (40), 82 (45), 81 (67), 68 (92).

\textit{o-}(3,5-Hexadienyl)benzaldehyde (2f): IR (neat) 3020 (w), 2910 (w), 2850 (w), 2720 (m), 1700 (s), 1660 (w), 1600 (s), 1010 (s), 900 (m), 750 (s) cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$, E-2f) $\delta$ 2.39 (q, $J = 7.7$ Hz, 2 H), 3.13 (t, $J = 7.7$ Hz, 2 H), 4.98 (dd, $J = 1.1$, 10.3 Hz, 1 H), 5.09 (dd, $J = 1.1$, 16.9 Hz, 1 H), 5.75 (dt, $J = 15.0$, 7.7 Hz, 1 H), 6.08 (dd, $J = 10.3$, 15.0 Hz, 1 H), 6.30 (dt, $J = 16.9$, 10.3 Hz, 1 H), 7.26 (d, $J = 7.7$ Hz, 1 H), 7.39 (dt, $J = 1.1$, 7.7 Hz, 1 H), 7.50 (dt, $J = 1.1$, 7.7 Hz, 1 H), 7.82 (dd, $J = 1.1$, 7.7 Hz, 1 H), 10.25 (s, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$, E-2f) $\delta$ 32.3, 34.7, 115.4, 126.5, 130.9, 131.8, 132.2, 133.3, 133.6, 136.8, 144.2, 192.1; $^1$H NMR (400 MHz, CDCl$_3$, Z-2f) $\delta$ 2.52 (q, $J = 8.0$ Hz, 2 H), 3.30 (q, $J = 8.0$ Hz, 2 H), 5.07 (dd, $J = 1.2$, 8.5 Hz, 1 H), 5.17 (dd, $J = 1.2$, 16.8 Hz, 1 H), 5.50 (t, $J = 8.5$ Hz, 1 H), 6.02 (t, $J = 8.5$ Hz, 1 H), 6.54 (ddd, $J = 1.2$, 10.2, 16.8 Hz, 1 H), 7.29 (d, $J = 6.8$ Hz, 1 H), 7.43 (dt, $J = 2.9$, 6.8 Hz, 1 H), 7.45 (dt, $J = 2.9$, 6.8 Hz, 1 H), 7.67 (dd, $J = 2.9$, 6.8 Hz, 1 H), 10.25 (s, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$, Z-2f) $\delta$ 29.6, 32.4, 117.4, 128.5, 128.6, 130.2, 130.5, 130.6, 131.0, 132.2, 144.2, 192.1; HRMS calcd for C$_{13}$H$_{14}$O:
186.1045. Found m/z (relative intensity) 186.1048 (M$^+$, 49), 168 (12), 132 (31), 116 (100).

**o-[(2E)-4-Methyl-2,4-pentadienyl]benzaldehyde (2g):** IR (neat) 3040 (m), 3000 (m), 2920 (m), 2900 (m), 1700 (s), 1600 (s), 1180 (m), 1160 (w), 980 (s), 870 (m), 820 (w), 740 (m) cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) δ 1.81 (s, 3 H), 3.87 (d, $J = 6.6$ Hz, 2 H), 4.87 (br s, 1 H), 4.91 (br s, 1 H), 5.83 (dt, $J = 15.8$, 6.6 Hz, 1 H), 6.14 (d, $J = 15.8$ Hz, 1 H), 7.31 (d, $J = 7.7$ Hz, 1 H), 7.41 (dt, $J = 1.5$, 7.7 Hz, 1 H), 7.53 (dt, $J = 1.5$, 7.7 Hz, 1 H), 7.86 (dd, $J = 1.5$, 7.7 Hz, 1 H), 10.29 (s, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 18.0, 35.3, 126.6, 126.7, 128.3, 129.2, 130.8, 131.0, 131.4, 131.9, 133.8, 142.7, 192.1; HRMS calcd for C$_{13}$H$_{14}$O: 186.1045. Found m/z (relative intensity) 186.1054 (M$^+$, 93), 172 (13), 171 (100).

**o-[(2E,4E)-2,4-Hexadienyl]benzaldehyde (2h):** IR (neat) 3050 (m), 3000 (m), 2920 (w), 2825 (w), 1700 (s), 1600 (m), 1200 (m), 980 (m), 920 (w), 860 (w), 740 (m) cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) δ 1.72 (d, $J = 6.6$ Hz, 3 H), 3.81 (d, $J = 6.6$ Hz, 1 H), 5.60 (dq, $J = 14.3$, 6.6 Hz, 1 H), 5.73 (dt, $J = 14.3$, 6.6 Hz, 1 H), 5.97 (ddm, $J = 9.9$, 14.3 Hz, 1 H), 6.04 (ddm, $J = 9.9$, 14.3 Hz, 1 H), 7.29 (d, $J = 7.7$ Hz, 1 H), 7.39 (dt, $J = 1.5$, 7.7 Hz, 1 H), 7.51 (dt, $J = 1.5$, 7.7 Hz, 1 H), 7.83 (d, $J = 7.7$ Hz, 1 H), 10.26 (s, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 17.9, 35.2, 124.2, 126.6, 128.5, 129.1, 130.8, 131.0, 131.4, 131.9, 133.6, 142.7, 192.0. Anal. calcd for C$_{13}$H$_{14}$O: C, 83.83; H, 7.58. Found: C, 83.55; H, 7.65.

**cis-1,2,2-Trimethyl-3-[(1E)-3-methyl-1,3-butadienyl]cyclopentanecarbaldehyde (2i):** IR (neat) 3300 (w), 2900 (s), 2830 (m), 1700 (s), 1640 (w), 1600 (w), 1130 (w), 940 (m), 890
(w), 860 (m) cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.80 (s, 3 H), 0.97 (s, 3 H), 1.09 (s, 3 H), 1.38 (ddd, $J = 5.5$, 9.2, 13.9 Hz, 1 H), 1.65 (dddd, $J = 5.5$, 9.2, 11.7, 13.9 Hz, 1 H), 1.93 (ddt, $J = 4.7$, 13.9, 9.2 Hz, 1 H), 2.43 (ddd, $J = 4.7$, 11.7, 13.9 Hz, 1 H), 2.52 (q, $J = 9.2$ Hz, 1 H), 4.91 (s, 2 H), 5.50 (dd, $J = 9.2$, 15.4 Hz, 1 H), 6.13 (d, $J = 15.4$ Hz, 1 H), 9.65 (s, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 18.7, 18.8, 19.4, 22.5, 27.6, 30.5, 47.6, 51.8, 58.3, 114.9, 129.5, 134.4, 141.8, 206.0; HRMS calcd for C$_{14}$H$_{22}$O: 206.1680. Found m/z (relative intensity): 206.1680 (M$^+$, 84), 191 (11), 135 (7), 121 (15), 110 (33), 109 (84), 95 (100), 79 (56).

4-Phenyl-6,8-nonadienal (2j): IR (neat) 3025 (m), 2925 (m), 2825 (m), 2715 (w), 2360 (w), 1720 (s), 1650 (w), 1610 (w), 1005 (m), 905 (m), 765 (m), 700 (m) cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$, E-2j) $\delta$ 1.83 (m, 1 H), 2.07 (m, 1 H), 2.20 - 2.34 (dm, $J = 1.5$ Hz, 2 H), 2.35 - 2.47 (m, 2 H), 2.62 (ddt, $J = 4.6$, 11.7, 7.1 Hz, 1 H), 4.94 (d, $J = 10.4$ Hz, 1 H), 5.06 (d, $J = 16.6$ Hz, 1 H), 5.55 (dt, $J = 14.9$, 7.1 Hz, 1 H), 6.02 (dd, $J = 10.4$, 14.9 Hz, 1 H), 6.23 (dt, $J = 16.8$, 10.4 Hz, 1 H), 7.06 - 7.39 (m, 5 H), 9.64 (t, $J = 1.5$ Hz, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$, E-2j) $\delta$ 28.0, 40.0, 42.0, 45.4, 115.2, 126.4, 127.5, 128.4, 132.3, 132.6, 136.8, 143.6, 201.8; $^1$H NMR (400 MHz, CDCl$_3$, Z-2j) $\delta$ 1.83 (m, 1 H), 2.07 (m, 1 H), 2.20 - 2.34 (dm, $J = 1.5$ Hz, 2 H), 2.35 - 2.47 (m, 2 H), 2.62 (m, 1 H), 5.05 (d, $J = 10.0$ Hz, 1 H), 5.16 (d, $J = 16.6$ Hz, 1 H), 5.34 (q, $J = 7.9$ Hz, 1 H), 5.98 (dd, $J = 7.9$, 10.0 Hz, 1 H), 6.57 (dt, $J = 16.6$, 10.0 Hz, 1 H), 7.06 - 7.39 (m, 5 H), 9.64 (t, $J = 1.5$ Hz, 1 H); HRMS calcd for C$_{13}$H$_{18}$O: 214.1358. Found m/z (relative intensity): 214.1323 (M$^+$, 100), 215 (19), 197 (3), 196 (75), 170 (13).
1-(Formylmethyl)-2-(2',4'-pentadienyl)cyclohexane (2k): IR (neat) 2910 (s), 2840 (s), 2690 (m), 1725 (s), 1650 (w), 1000 (s), 950 (w), 900 (m), 860 (m) cm⁻¹; ¹H NMR (400 MHz, CDCl₃, E-2k) δ 1.24 - 1.72 (m, 10 H), 2.00 (t, J = 7.7 Hz, 2 H), 2.35 (dm, J = 2.2 Hz, 2 H), 4.97 (d, J = 10.3 Hz, 1 H), 5.08 (d, J = 16.9 Hz, 1 H), 5.63 (dt, J = 15.0, 7.7 Hz, 1 H), 6.03 (dd, J = 10.3, 15.0 Hz, 1 H), 6.29 (dt, J = 16.9, 10.3 Hz, 1 H), 9.70 (t, J = 2.2 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃, E-2k) δ 22.7, 23.7, 28.1, 29.4, 33.1, 39.3, 39.6, 43.9, 114.9, 130.2, 132.2, 136.9, 202.7; ¹H NMR (400 MHz, CDCl₃, Z-2k) δ 1.24 - 1.72 (m, 10 H), 2.00 (t, J = 7.7 Hz, 2 H), 2.35 (dm, J = 2.2 Hz, 2 H), 5.08 (d, J = 16.9 Hz, 1 H), 5.19 (d, J = 16.9 Hz, 1 H), 5.40 (dt, J = 10.3, 7.7 Hz, 1 H), 6.05 (t, J = 10.3 Hz, 1 H), 6.60 (dt, J = 16.9, 10.3 Hz, 1 H), 9.70 (t, J = 2.2 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃, Z-2k) δ 22.7, 23.7, 28.1, 29.4, 33.1, 39.3, 39.6, 43.9, 114.9, 130.2, 132.2, 136.9, 202.7; HRMS calcd for C₁₃H₂₀O: 192.1514. Found m/z (relative intensity): 192.1519 (M⁺, 48), 174 (9), 138 (99), 81 (100).

8,10-Undecadienal (2l): IR (neat) 3400 (m), 2920 (s), 2850 (s), 2720 (m), 1730 (s), 1650 (w), 1000 (m), 900 (s), 780 (m) cm⁻¹; ¹H NMR (400 MHz, CDCl₃, Z-2l) δ 1.31 - 1.45 (m, 5 H), 1.60 - 1.68 (m, 3 H), 2.18 (qm, J = 7.3 Hz, 2 H), 2.41 (dt, J = 1.8, 7.3 Hz, 2 H), 5.08 (d, J = 10.6 Hz, 1 H), 5.18 (br d, J = 16.9 Hz, 1 H), 5.43 (dd, J = 7.3, 10.6 Hz, 1 H), 5.99 (t, J = 10.6 Hz, 1 H), 6.62 (dt, J = 16.9, 10.6 Hz, 1 H), 9.76 (t, J = 1.8 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃, Z-2l) δ 22.0, 27.6, 28.8, 28.9, 29.0, 43.8, 116.6, 129.1, 132.2, 132.5, 202.4; ¹H NMR (400 MHz, CDCl₃, E-2l) δ 1.31 - 1.45 (m, 5 H), 1.60 - 1.68 (m, 3 H), 2.08 (qm, J
= 7.3 Hz, 2 H), 2.26 (t, J = 7.7 Hz, 2 H), 4.95 (d, J = 10.3 Hz, 1 H), 5.09 (d, J = 16.8 Hz, 1 H), 5.69 (dt, J = 7.0, 14.7 Hz, 1 H), 6.08 (dd, J = 10.3, 14.7 Hz, 1 H), 6.30 (dt, J = 16.8, 10.3 Hz, 1 H), 9.76 (t, J = 1.8 Hz, 1 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), \(E-2l\)) δ 27.7, 28.8, 28.9, 28.9, 29.0, 32.4, 116.6, 129.1, 132.2, 132.5, 195.0; HRMS calcd for C\(_{11}\)H\(_{18}\)O: 166.1358. Found m/z (relative intensity): 166.1356 (M\(^+\), 55), 137 (13), 123 (12), 111 (10), 98 (100), 93 (20), 83 (14), 67 (70).

\((12E)-14\)-Methyl-12,14-pentadecadienal (2m): IR (neat) 2920 (s), 2840 (s), 1730 (s), 1610 (m), 960 (s), 870 (m) cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) δ 1.25 - 1.43 (m, 14 H), 1.58 - 1.71 (m, 2 H), 1.83 (t, J = 1.1 Hz, 3 H), 2.09 (q, J = 7.0 Hz, 2 H), 4.86 (s, 2 H), 5.66 (dt, J = 3.3, 7.0 Hz, 1 H), 6.13 (d, J = 15.4 Hz, 1 H), 9.76 (t, J = 1.8 Hz, 1 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) δ 18.7, 22.1, 29.1, 29.2, 29.3, 29.4, 29.5, 29.6, 29.7, 32.7, 43.9, 113.9, 130.9, 132.6, 142.1, 202.6. Anal. calcd for C\(_{16}\)H\(_{28}\)O\(_3\): C, 81.29; H, 11.94. Found: C, 81.29; H, 11.95.

**Reference and Note**


[4] Crystallographic data of 1c has been deposited with the Cambridge Crystallographic Data
Center as supplementary publication number CCDC-601331. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, U.K. (fax: (+41)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).