Supporting Information

Regioselective Allylmetallation of Allenes with Tetraallylmanganate or Allylmagnesium Chloride under $MnCl_2$ Catalysis

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Instrumentation and Materials

^1H NMR (300 MHz) and ^{13}C NMR (75.3 MHz) spectra were taken on Varian GEMINI 300 and Mercury 300 spectrometers. ^1H NMR and ^{13}C NMR spectra were obtained in $CDCl_3$ with tetramethylsilane as an internal standard. IR spectra were recorded on SHIMADZU FTIR-8200PC spectrometer. Mass spectra were taken on a JEOL Mstation 700 spectrometer. Silica gel (Wakogel 200 mesh) was used for column chromatography. The analyses were carried out at the Elemental Analysis Center of Kyoto University. Tetrahydrofuran (THF) was freshly distilled from sodium benzophenone ketyl before use. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Anhydrous manganese(II) chloride purchased from Aldrich was dried at 160 ˚C for 2 h and stocked under argon.

Computational Method

Calculations were carried out using the Gaussian 98 program.[1] The structure was optimized

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[1] Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Zakrzewski, V. G.; Montgomery, Jr. J. A.; Stratmann, R. E.; Burant, J. C.; Dapprich, S.; Millam, J. M.; Daniels, A. D.; Kudin, K. N.; Strain, M. C.; Farkas, O.; Tomasi, J.; Barone, V.; Cossi, M.; Cammi, R.; Mennucci, B.; Pomelli, C.; Adamo, C.; Clifford, S.; Ochterski, J.; Petersson, G. A.; Ayala, P. Y.; Cui, Q.; Morokuma, K.; Malick, D. K.; Rabuck, A. D.; Raghavachari, K.; Foresman, J. B.; Cioslowski, J.; Ortiz, J. V.; Stefanov, B. B.; Liu, G.; Liashenko, A.; Piskorz, P.; Komaromi, I.; Gomperts, R.; Martin, R. L.; Fox, D. J.; Keith, T.; Al-Laham, M. A.; Peng, C. Y.; Nanayakkara, A.; Gonzalez, C.; Challacombe, M.; Gill, P. M. W.; Johnson, B.; Chen, W.; Wong, M. W.; Andres, J. L.; Gonzalez, C.; Head-Gordon, M.; Replogle, E. S.; Pople, J. A. *Gaussian 98*, Revision A.6; Gaussian, Inc.: Pittsburgh, PA, 1998.

with Becke's three-parameter hybrid exchange functional and the Lee–Yang–Parr correlation functional (B3LYP)[2] and the 6-31G* basis set.

Experimental Section

General Procedure for Stoichiometric Carbomanganation of Allenes

A solution of allylmagnesium chloride (2.4 mL, 1.0 M solution in THF, 2.4 mmol) was added to a solution of $MnCl_2$ (76 mg, 0.6 mmol) in THF at 0 °C under atmosphere. After stirring for 30 min, to the resulting clear brown solution was added cyclonona-1,2-diene (**1a**, 61 mg, 0.5 mmol) in THF and the mixture was stirred for 3 h at 25 °C. The mixture was then poured into 1 M HCl aq. and extracted with hexane (3 × 10 mL). The organic layers were dried over Na_2SO_4 and concentrated in vacuo. Purification by silica gel column chromatography (hexane) afforded (*Z*)-3-allylcyclononene (**2a**) in 69% yield.

(*Z*)-3-Allylcyclononene (2a): IR (neat) 3076, 2924, 2356, 1641, 1447, 991, 910, 736 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.15–1.80 (m, 10H), 2.00–2.24 (m, 4H), 2.50–2.65 (m, 1H), 4.94–4.99 (m, 2H) 5.15 (d, *J* = 9.9 Hz, 1H) 5.56 (dt, *J* = 9.9, 7.5 Hz, 1H), 5.74–5.85 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 24.61, 25.96, 26.07, 26.52, 26.79, 33.54, 36.66, 41.63, 115.21, 129.46, 134.89, 137.65. HRMS (*m*/*z*) Found: 164.1572. Calcd for $C_{12}H_{20}$: 164.1565.

3-Decylhexa-1,5-diene (2b): IR (neat) 3076, 2926, 2855, 1641, 1466, 993, 910 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 0.88 (t, *J* = 6.6 Hz, 3H) 1.18–1.40 (m, 18H), 2.00–2.18 (m, 3H), 4.91–5.03 (m, 4H), 5.52–5.69 (m, 1H) 5.71–5.83 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 14.07, 24.68, 27.06, 29.34, 29.63, 29.63, 29.66, 29.74, 31.93, 34.25, 39.48, 43.68, 114.01, 115.48, 137.19, 142.84. HRMS (*m*/*z*) Found: 222.2348. Calcd for $C_{16}H_{30}$: 222.2351.

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[2] (a) Becke, A. D. *Phys. Rev. A* **1988**, *38*, 3098–3100. (b) Lee, C.; Yang, W.; Parr, R. G. *Phys. Rev. B* **1988**, *37*, 785–789.

(*E*)-3-Allylcyclotridecene (2c): IR (neat) 2930, 2856, 1447, 972, 910 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.00–1.60 (m, 18H), 1.80–2.20 (m, 5H), 4.91–5.02 (m, 2H), 5.07–5.16 (m, 1H), 5.25–5.36 (m, 1H), 5.70–5.84 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 24.23, 24.88, 25.11, 25.84, 25.92, 26.01, 26.38, 27.82, 31.44, 34.13, 40.49, 42.73, 115.07, 130.84, 135.36, 137.58. Found: C, 86.91; H, 12.59%. Calcd for $C_{16}H_{28}$: C, 87.19; H, 12.81%.

4-Pentylundeca-1,5-diene (2d, 53/47 diastereomeric mixture): IR (neat) 2926, 2856, 1639, 1466, 1379, 993, 968, 910 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 0.84–0.90 (m, 6H), 1.00–1.50 (m, 14H), 1.90–2.20 (m, 4.53H), 2.30–2.45 (m, 0.47H), 4.90–5.20 (m, 3H), 5.30–5.45 (m, 1H), 5.70–5.82 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 14.05, 14.05, 22.52, 22.56, 22.66, 22.66, 26.81, 26.96, 27.68, 29.37, 29.55, 31.33, 31.60, 31.91, 31.97, 32.06, 32.53, 34.74, 35.26, 37.21, 40.10, 40.37, 40.37, 42.55, 115.21, 115.21, 129.84, 130.35, 134.19, 134.26, 137.51, 137.60. Found: C, 86.26; H, 13.74%. Calcd for $C_{16}H_{30}$: C, 86.40; H, 13.60%.

3-(3-*tert*-Butyldimethylsiloxypropyl)hexa-1,5-diene (2e): IR (neat) 3078, 2858, 1641, 1472, 1464, 1389, 1256, 1101, 993, 912, 887, 775 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 0.05 (s, 6H), 0.89 (s, 9H), 1.20–1.30 (m, 1H), 1.40–1.60 (m, 3H), 2.00–2.20 (m, 3H), 3.59 (t, J = 6.3 Hz, 2H), 4.93–5.04 (m, 4H), 5.52–5.64 (m, 1H), 5.69–5.83 (m, 1H); ^{13}C NMR ($CDCl_3$) δ –5.26, 18.36, 25.99, 30.37, 30.39, 39.50, 43.43, 63.28, 114.27, 115.63, 137.01, 142.57. Found: C, 70.24; H, 10.11%. Calcd for $C_{15}H_{30}OSi$: C, 70.33; H, 9.94%.

General Procedure for the Catalytic Allylmagesation and the Subsequent Reaction with Electrophiles

A solution of allylmagnesium chloride (1.50 mL, 1.0 M solution in THF, 1.5 mmol) and HMPA (1.0 mmol) was added to a solution of $MnCl_2$ (13 mg, 0.1 mmol) in THF at 0 °C under atmosphere. After stirring for 30 min, to the resulting clear brown solution was added 3-methyltrideca-1,2-diene (**1f**, 97 mg, 0.5 mmol) in THF and the reaction mixture was stirred for 15 h at 25 °C. The reaction mixture was then cooled to 0 °C. To the reaction mixture was added allyl bromide (0.19 mL, 1.75 mmol). The mixture was stirred for another 45 min. The mixture was then poured into 1 M HCl aq. and extracted with hexane (3 × 10 mL). The organic layers were dried over Na_2SO_4 and concentrated in vacuo. Purification by silica gel column chromatography (hexane) afforded 4-decyl-4-methyl-5-methyleneocta-1,7-diene (**7f**, 94 mg, 0.34 mmol) in 69% yield.

(*E*)-3-Allyl-2-iodocyclononene (5a): IR (neat) 2925, 2858, 1641, 1442, 1161, 991, 912 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.20–1.60 (m, 8H), 1.60–1.80 (m, 2H), 2.00–2.30 (m, 5H), 4.97–5.14 (m, 2H), 5.66–5.80 (m, 1H), 6.60 (dd, *J* = 9.3, 8.1 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 23.16, 26.19, 26.63, 28.20, 31.47, 32.72, 41.91, 42.07, 114.21, 115.88, 136.13, 141.96. HRMS (*m*/*z*) Found: 290.0536. Calcd for $C_{12}H_{19}I$: 290.0532.

(*E*)-2-Acetyl-3-allylcyclononene (6a): IR (neat) 2925, 2856, 1666, 1639, 1447, 1353, 1284, 910 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.20–1.80 (m, 9H), 1.80–2.00 (m, 1H), 2.18–2.50 (m, 4H), 2.28 (s, 3H), 2.90–3.05 (m, 1H), 4.86–4.96 (m, 2H), 5.59–5.73 (m, 1H), 6.72 (t, *J* = 8.7 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 26.32, 26.32, 27.01, 27.01, 27.75, 27.75, 32.16, 37.45, 38.93, 115.14, 137.83, 144.15, 144.52, 200.44. Found: C, 81.38; H, 10.59%. Calcd for $C_{14}H_{22}O$: C, 81.50; H, 10.75%.

3-Allyl-2-iodocyclotridecene (5c, *E*/*Z* = 18/82 diastereomeric mixture): IR (neat) 3076, 2928, 2856, 1639, 1445, 993, 912 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.10–1.60 (m, 17H), 1.55–1.70 (m, 1H) 1.74–1..84 (m, 1H) 1.92–2.02 (m, 1H) 2.10–2.15 (m, 2.82H), 2.36–2.50 (m, 0.18H), 4.94–5.10 (m, 2H), 5.60–5.80 (m, 1.82H) 6.24–6.29 (m, 0.18H); ^{13}C NMR ($CDCl_3$) δ23.73, 24.70, 24.70, 24.84, 25.05, 25.14, 25.14, 25.30, 25.30, 26.90, 27.20, 27.31, 27.87, 27.87, 28.79, 29.33, 30.07, 33.20, 34.86, 35.45, 40.19, 40.40, 41.74, 50.42, 115.84, 116.03, 117.83, 117.83, 136.10, 136.37, 136.96, 143.90. HRMS (*m*/*z*) Found: 346.1159. Calcd for $C_{16}H_{27}I$: 346.1158.

(*Z*)-2-Acetyl-3-allylcyclotridecene (6c): IR (neat) 2929, 2858, 1693, 1639, 1447, 1350, 993, 912 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.20–1.70 (m, 18H), 2.02–2.25 (m, 3H), 2.25–2.44 (m, 2H), 2.26 (s, 3H), 4.95–5.02 (m, 2H), 5.51 (d, *J* = 9.0, 6.0 Hz, 1H), 5.66–5.80 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 24.34, 24.78, 24.78, 25.04, 25.39, 26.46, 27.14, 28.29, 29.23, 31.28, 33.82, 40.83, 42.69, 116.00, 135.29, 136.86, 144.84, 205.11. Found: C, 82.10; H, 11.57%. Calcd for $C_{18}H_{30}O$: C, 82.38; H, 11.52%.

5-Iodo-4-pentylundeca-1,5-diene (5d, *E*/*Z* = 35/65 diastereomeric mixture): IR (neat) 2928, 2856, 1641, 1466, 1443, 1379, 1231, 993, 912 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 0.84–0.91 (m, 6H), 1.00–1.45 (m, 14H), 1.64–1.82 (m, 0.65H), 1.83–2.00 (m, 0.35H), 2.00–2.20 (m, 4H), 4.93–5.11 (m, 2H), 5.57–5.74 (m, 1H), 5.60 (t, *J* = 6.9 Hz, 0.65H), 6.30 (t, *J* = 7.2 Hz, 0.35H); ^{13}C NMR ($CDCl_3$) δ 14.09, 14.10, 14.15, 14.18, 22.55, 22.56, 22.63, 22.70, 26.64, 26.72, 28.22, 29.05, 31.35, 31.43, 31.64, 31.73, 31.92, 34.40, 35.23, 35.80, 39.88, 40.19, 43.17, 51.23, 113.56, 115.78, 115.98, 118.01, 135.30, 136.00, 136.23, 142.46. Found: C, 55.32; H, 8.23%. Calcd for $C_{16}H_{29}I$: C, 55.17; H, 8.39%.

(*E*)-(Non-1-en-4-yl)non-3-en-2-one (6d): IR (neat) 2925, 2858, 1672, 1460, 1253, 993, 910 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.84 (t, *J* = 6.9 Hz, 3H), 0.90 (d, *J* = 7.2 Hz, 3H), 1.00–1.75 (m, 14H), 2.15–2.45 (m, 4H), 2.25 (s, 3H), 2.55–2.70 (m, 1H), 4.84–4.96 (m, 2H), 5.56–5.71 (m, 1H), 6.58 (t, *J* = 7.2 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 14.08, 14.16, 22.58, 22.72, 27.06, 28.03, 28.80, 28.86, 31.76, 32.03, 33.24, 38.29, 38.97, 115.06, 137.92, 143.35, 147.17, 200.19. Found: C, 81.56; H, 12.29%. Calcd for $C_{18}H_{32}O$: C, 81.75; H, 12.20%.

3-Decyl-3-methylhexa-1,5-diene (2f): IR (neat) 3078, 2925, 2855, 1639, 1466, 1375, 995, 912 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.88 (t, *J* = 7.2 Hz, 3H), 0.94 (s, 3H), 1.10–1.40 (m, 18H), 2.03–2.10 (m, 2H), 4.84–5.02 (m, 4H), 5.67–5.82 (m, 2H); ^{13}C NMR ($CDCl_3$): δ 14.24, 22.81, 22.85, 24.11, 29.47, 29.76, 29.78, 29.81, 30.56, 32.04, 39.55, 40.67, 45.32, 111.38, 116.56, 135.31, 146.94. Found: C, 86.31; H, 13.42%. Calcd for $C_{17}H_{32}$: C, 86.36; H, 13.64%.

3-Decyl-2-iodo-3-methylhexa-1,5-diene (5f): IR (neat) 2926, 2855, 1605, 1466, 1443, 1088, 993, 914, 896 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.88 (t, *J* = 6.9 Hz, 3H), 1.10 (s, 3H), 1.15–1.60 (m, 17H), 2.02 (dd, *J* = 14.1, 8.4 Hz, 1H), 2.27 (dd, *J* = 14.1, 6.6 Hz, 1H), 5.02–5.10 (m, 2H), 5.60–5.75 (m, 2H), 5.89 (d, *J* = 2.1 Hz, 1H), 6.02 (d, *J* = 2.1 Hz, 1H); ^{13}C NMR ($CDCl_3$): δ 14.24, 21.54, 22.78, 23.85, 29.43, 29.64, 29.71, 29.75, 30.18, 32.00, 41.23, 45.58, 46.17, 117.37, 126.00, 126.59, 134.12. HRMS (*m*/*z*) Found: 362.1469. Calcd for $C_{17}H_{31}I$: 362.1471.

4-Decyl-4-methyl-3-methylenehept-6-en-2-one (6f): IR (neat) 2926, 2855, 1686, 1468, 1356, 1105, 912 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.87 (t, *J* = 6.9 Hz, 3H), 0.95–1.40 (m, 17H), 1.05 (s, 3H), 1.82 (dt, *J* = 12.3, 4.2 Hz, 1H), 2.10 (dd, *J* = 13.8, 7.5 Hz, 1H), 2.30 (s, 3H), 2.58 (dd, *J* = 13.8, 7.2 Hz, 1H), 4.89–4.96 (m, 2H), 5.52–5.67 (m, 1H), 5.61 (s, 1H), 5.93 (s, 1H); ^{13}C NMR ($CDCl_3$): δ 14.20, 22.75, 24.15, 24.58, 28.92, 29.40, 29.67, 29.69, 29.72, 30.26, 31.97, 39.50, 41.96, 44.32, 116.63, 124.30, 135.23, 154.37, 201.48. Found: C, 81.76; H, 12.08%. Calcd for $C_{19}H_{34}O$: C, 81.95; H, 12.31%.

4-Decyl-4-methyl-5-methyleneocta-1,7-diene (7f): IR (neat) 2928, 2855, 1639, 1468, 995, 910 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.88 (t, *J* = 6.9 Hz, 3H), 1.00 (s, 3H), 1.00–1.50 (m, 18H), 2.02 (dd, *J* = 13.8, 7.8 Hz, 1H), 2.22 (dd, *J* = 13.8, 6.6 Hz, 1H), 2.69–2.73 (m, 2H), 4.82–4.88 (m, 2H), 4.94–5.07 (m, 4H), 5.60–5.88 (m, 2H); ^{13}C NMR ($CDCl_3$): δ 14.23, 22.78, 23.26, 24.10, 29.43, 29.72, 29.72, 29.77, 30.41, 32.00, 35.79, 39.48, 42.28, 44.44, 110.96, 115.83, 116.32, 135.39, 137.34, 152.34 . HRMS (*m*/*z*) Found: 376.2829. Calcd for $C_{20}H_{36}$: 376.2817.

General Procedure for Diallylation for Allenes in the Presence of a Catalytic Amount of Manganese Salt

A solution of allylmagnesium chloride (2.50 mL, 1.0 M solution in THF, 2.5 mmol) was added to a solution of $MnCl_2$ (13 mg, 0.1 mmol) in THF at 0 °C under atmosphere. After stirring for 30 min, to the resulting clear brown solution was added cyclonona-1,2-diene (**1a**, 61 mg, 0.5 mmol) in THF and the mixture was stirred for 9 h at 25 °C. The resulting mixture was exposed to air and stirring was continued for another 40 h. The mixture was poured into 1 M HCl aq. and extracted with hexane (3 ×

10 mL). The organic layers were dried over Na_2SO_4 and concentrated in vacuo. Purification by silica gel column chromatography (hexane) afforded diallylated product (**7a**, 65 mg, 0.32 mmol) in 64% yield.

(*Z*)-2,3-Diallylcyclononene (7a): IR (neat) 2924, 2851, 1639, 1448, 993, 968, 910, 827 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.10–1.80 (m, 10H), 2.00–2.25 (m, 4H), 2.62 (d, *J* = 7.2 Hz, 2H), 2.79–2.90 (m, 1H), 4.90–5.10 (m, 4H), 5.41 (t, *J* = 8.1 Hz, 1H), 5.67–5.90 (m, 2H) ; ^{13}C NMR ($CDCl_3$) δ 23.94, 26.88, 26.94, 27.09, 27.60, 32.29, 35.94, 38.43, 39.17, 114.98, 115.64, 127.24, 137.60, 137.86, 139.70. HRMS (*m*/*z*) Found: 204.1872. Calcd for $C_{15}H_{24}$: 204.1878.

5-Allyl-4-pentylundeca-1,5-diene (7d, 64/36 diastereomeric mixture): IR (neat) 2926, 2856, 1639, 1466, 993, 910 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.84–0.90 (m, 6H), 1.00–1.50 (m, 14H), 1.90–2.20 (m, 4.64H), 2.55–2.63 (m, 1.64H), 2.70–2.74 (m, 0.72H), 4.89–5.05 (m, 4H), 5.14–5.22 (m, 1H), 5.63–5.85 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.20, 14.21, 22.70, 22.74, 27.07, 27.37, 27.67, 27.83, 29.69, 29.89, 31.64, 31.76, 32.06, 32.16, 32.97, 33.15, 33.57, 35.30, 38.44, 38.82, 40.16, 47.06, 114.62, 114.86, 114.87, 115.36, 127.42, 127.59, 137.14, 137.75, 137.94, 137.95, 138.04, 138.55. HRMS (*m*/*z*) Found: 262.2673. Calcd for $C_{19}H_{34}$: 262.2660.

General Procedure for the Reaction of Enallene with Tetraallylmanganate

Manganese(II) chloride (76 mg, 0.75 mmol) was sonicated in THF under argon atmosphere for 30 min. Allylmagnesium chloride (3.0 mL, 1.0 M solution in THF, 3.0 mmol) and HMPA (1.0 mmol) was added to a solution of $MnCl_2$ in THF at 0 °C. The mixture turned into a clear brown solution. After stirring for 30 min, to the resulting dark-red solution was added 5,5-di(methoxymethyl)hepta-1,2,6-triene (**8a**, 90 mg, 0.5 mmol) in THF and the mixture was stirred for 22 h at 0 °C. The mixture was then poured into 1 M HCl aq. and extracted with ethyl acetate (3 × 10 mL). The organic layers were dried over Na_2SO_4 and concentrated in vacuo. Purification by silica gel column chromatography (hexane) afforded 4-allyl-1,1-di(methoxymethyl)-2-methyl-3-methylenecyclopentane (**9**) in 46% yield.

4-Allyl-1,1-di(methoxymethyl)-2-methyl-3-methylenecyclopentane (9): IR (neat) 2873, 1650, 1460, 1198, 1113, 962, 912, 880 cm^{-1}; ^{1}H NMR (CDCl$_3$): δ 0.98 (d, *J* = 6.9 Hz, 3H), 1.20 (dd, *J* = 13.2, 9.6 Hz, 1H), 1.90 (dd, *J* = 13.2, 8.4 Hz, 1H), 2.00–2.12 (m, 1H), 2.30–2.50 (m, 2H), 2.52–2.68 (m, 1H), 3.06 (d, *J* = 9.3 Hz, 1H), 3.20 (d, *J* = 9.3 Hz, 1H), 3.27 (s, 3H), 3.32 (s, 2H), 3.34 (s, 3H), 4.79–4.87 (m, 2H), 4.95–5.07 (m, 2H), 5.73–5.86 (m, 1H); ^{13}C NMR (CDCl$_3$): δ 12.36, 35.84, 40.34, 40.74, 44.08, 48.31, 59.30, 59.37, 72.88, 76.50, 104.34, 115.53, 137.10, 160.24. Found: C, 74.90; H, 10.48%. Calcd for $C_{14}H_{24}O_2$: C, 74.95; H, 10.78%.