Nitrile-Promoted Rh-Catalyzed Intermolecular Hydroacylation of Olefins with Salicylaldehyde

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Supporting Information

Synthetic Scheme of Substrates 8 and 9	S2
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Synthetic Scheme of Substrates **8** and **9**

Experimental Section

General. ^1H NMR spectra were determined at 270 MHz or 400 MHz unless otherwise noted. Infrared spectra were recorded on a JASCO FT/IR-7000 spectrometer. EIMS spectra were taken on a Hitachi M-2000 mass spectrometer. The melting points were measured with Yanagimoto micro-melting point apparatus and are uncorrected.

Undec-10-enenitrile (8). A mixture of dec-9-en-1-ol (1.70 g, 10.9 mmol), Et_3N (2.30 mL, 16.4 mmol), and *p*-toluenesulfonyl chloride (2.12 g, 10.9 mmol) in CH_2Cl_2 (20 mL) was stirred overnight at room temperature. Then, the solution was diluted with water, extracted with CHCl_3 , washed with saturated aqueous NH_4Cl , and dried over MgSO_4 . After removal of the solvent, the residue was purified by column chromatography on silica gel (30% EtOAc in hexane) to give tosylate (3.05 g, 90%) as a colorless oil: IR (neat) 2928, 2860, 1640, 1601 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.79 (br d, $J = 8.8$ Hz, 2H), 7.34 (br d, $J = 8.8$ Hz, 2H), 5.80 (m, 1H), 4.92—5.00 (m, 2H), 4.02 (t, $J = 6.8$ Hz, 2H), 2.45 (s, 3H), 2.02 (quartet, $J = 6.8$ Hz, 2H), 1.63 (quintet, $J = 6.8$ Hz, 2H), 1.23—1.36 (m, 10H). A mixture of the tosylate (3.05 g, 9.82 mmol) and KCN (730 mg, 10.8 mmol) in DMSO (30 mL) was stirred overnight at room temperature. Then, the solution was diluted with water, extracted with ether, washed with 5% aqueous NaHCO_3 , and dried over MgSO_4 . After removal of the solvent, the residue was purified by column chromatography on silica gel (20% EtOAc in hexane) to give **8** (1.09 g, 67%) as a colorless oil: IR (neat) 2928, 2860, 2250, 1642 cm^{-1} ; ^1H NMR (CDCl_3) δ 5.76—5.86 (m, 1H), 4.92—5.02 (m, 2H), 2.33 (t, $J = 7.3$ Hz, 2H), 2.04 (quartet, $J = 7.3$ Hz, 2H), 1.65 (quintet, $J = 7.3$ Hz, 2H), 1.31—1.46 (m, 10H); EI-HRMS calcd for $\text{C}_{11}\text{H}_{19}\text{N}$ (M^+) 165.1517, found 165.1500.^{S1}

Dodec-11-enenitrile (9). Compound **9** was prepared from undec-10-en-1-ol in a manner similar to that describe for the preparation of **8**. A colorless oil: IR (neat) 2930, 2860, 2250, 1640 cm^{-1} ; ^1H NMR (CDCl_3) δ 5.81 (m, 1H), 4.92—5.01 (m, 2H), 2.33 (t, $J = 6.8$ Hz, 2H), 2.04 (quartet, $J = 6.8$ Hz, 2H), 1.66 (quintet, $J = 6.8$ Hz, 2H), 1.29—1.46 (m, 12H); EI-HRMS calcd for $\text{C}_{12}\text{H}_{21}\text{N}$ (M^+) 179.1674, found 179.1690.^{S1}

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and Allyl Cyanide (4). RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and allyl cyanide **4** at room temperature afforded **10a** and **10b** in 98% yield as a mixture of isomers in the ratio of 5 to 1. Each compound **10a** and **10b** could be separated by preparative TLC. **5-(2-Hydroxyphenyl)-5-oxopentanenitrile (10a).** Colorless crystals: mp 49.0—50.0 °C (recryst. from CDCl₃-hexane); IR (KBr) 3438 (br), 2958, 2908, 2250, 1644, 1613 cm⁻¹; ¹H NMR (CDCl₃) δ 12.07 (s, 1H), 7.76 (br d, *J* = 8 Hz, 1H), 7.49 (br t, *J* = 8 Hz, 1H), 7.00 (br d, *J* = 8 Hz, 1H), 6.92 (br t, *J* = 8 Hz, 1H), 3.22 (t, *J* = 7.0 Hz, 2H), 2.54 (t, *J* = 7.0 Hz, 2H), 2.13 (quintet, *J* = 7.0 Hz, 2H); EI-HRMS calcd for C₁₁H₁₁NO₂ (M⁺) 189.0790, found 189.0812. Anal. Calcd for C₁₁H₁₁NO₂: C, 69.83; H, 5.86; N, 7.40; Found: C, 69.96; H, 5.97; N, 7.26. **4-(2-Hydroxyphenyl)-3-methyl-4-oxobutyronitrile (10b).** A colorless oil: IR (neat) 3024, 2982, 2252, 1640, 1615 cm⁻¹; ¹H NMR (CDCl₃) δ 12.00 (s, 1H), 7.71 (br d, *J* = 8 Hz, 1H), 7.52 (br t, *J* = 8 Hz, 1H), 7.03 (br d, *J* = 8 Hz, 1H), 6.95 (br t, *J* = 8 Hz, 1H), 3.86 (sextet, *J* = 7.3 Hz, 1H), 2.75 (dd, *J* = 6.9, 16.9 Hz, 1H), 2.64 (dd, *J* = 7.7, 16.9 Hz, 1H), 1.47 (d, *J* = 6.9 Hz, 3H); EI-HRMS calcd for C₁₁H₁₁NO₂ (M⁺) 189.0790, found 189.0760.

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and 4-Pentenitrile (5). RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and 4-pentenitrile **5** at room temperature afforded **11** in 94% yield as a mixture of isomers in the ratio of >20 (*n*) to 1 (*i*). Colorless crystals: mp 83.0—84.0 °C (recryst. from CDCl₃-hexane); IR (KBr) 3448 (br), 2946, 2878, 2242, 1642, 1615 cm⁻¹; ¹H NMR (CDCl₃) δ 12.23 (s, 1H), 7.74 (br d, *J* = 8 Hz, 1H), 7.47 (br t, *J* = 8 Hz, 1H), 6.98 (br d, *J* = 8 Hz, 1H), 6.91 (br t, *J* = 8 Hz, 1H), 3.07 (t, *J* = 6.8 Hz, 2H), 2.42 (t, *J* = 6.8 Hz, 2H), 1.91 (quintet, *J* = 6.8 Hz, 2H), 1.77 (quintet, *J* = 6.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 205.2, 162.4, 136.4, 129.7, 119.3, 119.1, 118.9, 118.5, 37.0, 24.9, 23.0, 17.1; EI-HRMS calcd for C₁₂H₁₃NO₂ (M⁺) 203.0946, found 203.0935. Anal. Calcd for C₁₂H₁₃NO₂: C, 70.92; H, 6.45; N, 6.89; Found: C, 70.99; H, 6.57; N, 6.77.

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and 5-Hexenenitrile (6). RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and 5-hexenenitrile **6** at room temperature afforded **12** in 83% yield as a mixture of isomers in the ratio of

>20 (*n*) to 1 (*i*). Colorless crystals: mp 74.0—75.0 °C (recryst. from CDCl₃-hexane); IR (KBr) 3452 (br), 2950, 2878, 2248, 1644, 1615 cm⁻¹; ¹H NMR (CDCl₃) δ 12.29 (s, 1H), 7.75 (br d, *J* = 8 Hz, 1H), 7.47 (br t, *J* = 8 Hz, 1H), 6.99 (br d, *J* = 8 Hz, 1H), 6.90 (br t, *J* = 8 Hz, 1H), 3.03 (t, *J* = 6.8 Hz, 2H), 2.38 (t, *J* = 6.8 Hz, 2H), 1.70—1.83 (m, 4H), 1.53—1.60 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 205.9, 162.2, 136.1, 129.7, 119.4, 119.0, 118.8, 118.3, 37.5, 28.0, 25.0, 23.0, 16.8; EI-HRMS calcd for C₁₃H₁₅NO₂ (M⁺) 217.1103, found 217.1077. Anal. Calcd for C₁₃H₁₅NO₂: C, 71.87; H, 6.96; N, 6.45; Found: C, 71.81; H, 7.06; N, 6.34.

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and 6-Heptenenitrile (7). RhCl(PPh₃)₃-Catalyzed hydroacylation between salicylaldehyde **1** and 6-heptenenitrile **7** at room temperature afforded **13** in 99% yield as a mixture of isomers in the ratio of >20 (*n*) to 1 (*i*). Colorless crystals: mp 60.0—61.0 °C (recryst. from CDCl₃-hexane); IR (KBr) 3452 (br), 2944, 2868, 2252, 1636, 1618 cm⁻¹; ¹H NMR (CDCl₃) δ 12.29 (s, 1H), 7.76 (br d, *J* = 8 Hz, 1H), 7.47 (br t, *J* = 8 Hz, 1H), 6.98 (br d, *J* = 8 Hz, 1H), 6.90 (br t, *J* = 8 Hz, 1H), 3.01 (t, *J* = 7.3 Hz, 2H), 2.36 (t, *J* = 7.3 Hz, 2H), 1.78 (quintet, *J* = 7.3 Hz, 2H), 1.69 (quintet, *J* = 7.3 Hz, 2H), 1.42—1.56 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 206.3, 162.3, 136.2, 129.8, 119.6, 119.1, 118.8, 118.4, 37.8, 28.3, 28.2, 25.0, 23.8, 16.9; EI-HRMS calcd for C₁₄H₁₇NO₂ (M⁺) 231.1259, found 231.1245. Anal. Calcd for C₁₄H₁₇NO₂: C, 72.70; H, 7.41; N, 6.06; Found: C, 72.82; H, 7.54; N, 5.96.

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (11) and Undec-10-enenitrile (8). RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and undec-10-enenitrile **8** at room temperature afforded **14** in 70% yield as a mixture of isomers in the ratio of >20 (*n*) to 1 (*i*). Colorless crystals: mp 74.0—75.0 °C (recryst. from CDCl₃-hexane); IR (KBr) 3428 (br), 2922, 2854, 2244, 1638, 1611 cm⁻¹; ¹H NMR (CDCl₃) δ 12.40 (s, 1H), 7.76 (br d, *J* = 8 Hz, 1H), 7.45 (br t, *J* = 8 Hz, 1H), 6.97 (br d, *J* = 8 Hz, 1H), 6.89 (br t, *J* = 8 Hz, 1H), 2.98 (t, *J* = 7.3 Hz, 2H), 2.32 (t, *J* = 7.3 Hz, 2H), 1.73 (quintet, *J* = 7.3 Hz, 2H), 1.64 (quintet, *J* = 7.3 Hz, 2H), 1.30—1.45 (m, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 206.9, 162.5, 136.2, 130.0, 119.8, 119.3, 118.8, 118.5, 38.3, 29.3, 29.24, 29.20, 29.18, 28.7, 28.6, 25.3, 24.4, 17.1; EI-

HRMS calcd for $C_{18}H_{25}NO_2$ (M^+) 287.1885, found 287.1903. Anal. Calcd for $C_{18}H_{25}NO_2$: C, 75.22; H, 8.77; N, 4.87; Found: C, 75.40; H, 8.67; N, 4.86.

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and Dodec-11-enenitrile (9). RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and dodec-11-enenitrile **9** at room temperature afforded **15** in 80% yield as a mixture of isomers in the ratio of >20 (*n*) to 1 (*i*). Colorless crystals: mp 73.0—74.0 °C (recryst. from CHCl₃-hexane); IR (KBr) 3484 (br), 2924, 2854, 2248, 1640, 1618 cm⁻¹; ¹H NMR (CDCl₃) δ 12.39 (s, 1H), 7.76 (br d, *J* = 8 Hz, 1H), 7.46 (br t, *J* = 8 Hz, 1H), 6.98 (br d, *J* = 8 Hz, 1H), 6.89 (br t, *J* = 8 Hz, 1H), 2.98 (t, *J* = 7.3 Hz, 2H), 2.33 (t, *J* = 7.3 Hz, 2H), 1.74 (quintet, *J* = 7.3 Hz, 2H), 1.65 (quintet, *J* = 7.3 Hz, 2H), 1.29—1.46 (m, 14H); ¹³C NMR (100 MHz, CDCl₃) δ 206.8, 162.3, 136.0, 129.9, 119.7, 119.2, 118.7, 118.3, 38.1, 29.22, 29.20 (x2), 29.1 (x2), 28.6, 28.5, 25.2, 24.3, 16.9; EI-HRMS calcd for $C_{19}H_{27}NO_2$ (M^+) 301.2042, found 301.2043. Anal. Calcd for $C_{19}H_{27}NO_2$: C, 75.71; H, 9.03; N, 4.65; Found: C, 75.88; H, 9.10; N, 4.59.

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and 2-Methyl-1,5-hexadiene (16).^{3a,b} RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and 2-methyl-1,5-hexadiene **16** at room temperature afforded **25a** and **25b** in 95% yield as a mixture of isomers in the ratio of 1 (*normal*) to 10 (*iso*). A colorless oil: IR (neat) 3075, 1640, 1610, 1580 cm⁻¹; ¹H NMR (CDCl₃) δ 12.57 (s, 0.91H), 12.39 (s, 0.09H), 7.79 (br d, *J* = 8 Hz, 1H), 7.47 (br t, *J* = 8 Hz, 1H), 6.99 (br d, *J* = 8 Hz, 1H), 6.90 (br t, *J* = 8 Hz, 1H), 4.72 (br s, 1H), 4.65 (br s, 1H), 3.53 (m, 0.91H), 3.00 (t, *J* = 7.3 Hz, 0.18H), 1.95—2.20 (m, 3.09H), 1.71 (s, 3H), 1.55—1.75 (m, 1.18H), 1.24 (d, *J* = 6.9 Hz, 2.73H).

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and 1,5-heptadiene (17).^{3a,b} RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and 1,5-heptadiene **17** at room temperature afforded **26a,b,c** in 82% yield as a mixture of isomers in the ratio of 10 : 1 : 1. A colorless oil: IR (neat) 2938, 1634, 1582 cm⁻¹; **26a**: ¹H NMR (CDCl₃) δ 12.39 (s, 1H), 7.76 (br d, *J* = 8 Hz, 1H), 7.46 (br t, *J* = 8 Hz, 1H), 6.98 (br d, *J* = 8 Hz, 1H), 6.89 (br

t, $J = 8$ Hz, 1H), 5.30—5.55 (m, 2H), 2.95—3.02 (m, 2H), 2.00—2.15 (m, 2H), 1.15—1.80 (m, 7H). Each compound **26a,b,c** could be separated by preparative TLC.^{3a,b}

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and 1,7-Octadiene (18). RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and 1,7-octadiene **18** at room temperature afforded **27** in 73% yield as a mixture of isomers in the ratio of >20 (*n*) to 1 (*i*). A colorless oil; IR (neat) 3080, 2932, 1642, 1615 cm⁻¹; ¹H NMR (CDCl₃) δ 12.39 (s, 1H), 7.76 (br d, $J = 8.1$ Hz, 1H), 7.45 (br t, $J = 8.3$ Hz, 1H), 6.98 (br d, $J = 8.3$ Hz, 1H), 6.89 (br t, $J = 8.1$ Hz, 1H), 5.81 (m, 1H), 4.92—5.02 (m, 2H), 2.98 (t, $J = 7.3$ Hz, 2H), 2.05 (m, 2H), 1.75 (quintet, $J = 6.8$ Hz, 2H), 1.35—1.44 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 206.7, 162.4, 138.8, 136.0, 129.9, 119.2, 118.7, 118.4, 114.2, 38.1, 33.6, 29.0, 28.8, 28.6, 24.3; EI-HRMS calcd for C₁₅H₂₀O₂ (M⁺) 232.1463, found 232.1466.

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and 2,2-Diallylmalonic Acid Diethyl Ester (19). RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and 2,2-diallylmalonic acid diethyl ester **19** at room temperature afforded **28** in 80% yield as a colorless oil.; IR (neat) 3082, 2984, 1734, 1642, 1615 cm⁻¹; ¹H NMR (CDCl₃) δ 12.30 (s, 1H), 7.72 (br d, $J = 8$ Hz, 1H), 7.46 (br t, $J = 8$ Hz, 1H), 6.98 (br d, $J = 8$ Hz, 1H), 6.89 (br t, $J = 8$ Hz, 1H), 5.67 (m, 1H), 5.09—5.14 (m, 2H), 4.19 (q, $J = 7.1$ Hz, 4H), 3.01 (t, $J = 7.1$ Hz, 2H), 2.70 (d, $J = 7.6$ Hz, 2H), 1.93—1.98 (m, 2H), 1.61—1.72 (m, 2H), 1.25 (t, $J = 7.1$ Hz, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 205.5, 170.9 (x2), 162.3, 136.1, 132.2, 129.7, 119.1, 118.9, 118.7, 118.4, 61.1 (x2), 57.2, 38.0, 36.8, 31.6, 18.5, 14.0 (x2); EI-HRMS calcd for C₂₀H₂₆O₆ (M⁺) 362.1729, found 362.1729.

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and 1-Hexene (20).^{3a,b} RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and 1-hexene **20** at room temperature afforded **29** in 86% yield (ratio: >20 : 1) as a colorless oil: IR (neat) 2925, 1635 cm⁻¹; ¹H NMR (CDCl₃) δ 12.40 (s, 1H), 7.76 (br d, $J = 8$ Hz, 1H), 7.47 (br t, $J = 8$ Hz, 1H), 6.99 (br d, $J = 8$ Hz, 1H), 6.89 (br t, $J = 8$ Hz, 1H), 2.99 (t, $J = 7.3$ Hz, 2H), 1.75 (m, 2H), 1.25—1.50 (m, 6H), 0.90 (t, $J = 6.9$ Hz, 3H).

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and 3,3-Dimethyl-1-butene (22). RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and 3,3-dimethyl-1-butene **22** at room temperature afforded **31** in 82% yield as a colorless oil.; IR (neat) 2960, 2870, 1642, 1615 cm⁻¹; ¹H NMR (CDCl₃) δ 12.39 (s, 1H), 7.77 (br d, *J* = 8 Hz, 1H), 7.46 (br t, *J* = 8 Hz, 1H), 6.98 (br d, *J* = 8 Hz, 1H), 6.90 (br t, *J* = 8 Hz, 1H), 2.93—2.97 (m, 2H), 1.63—1.67 (m, 2H), 0.97 (s, 9H); ¹³C NMR (68 MHz, CDCl₃) δ 207.5, 162.5, 136.1, 129.9, 119.2, 118.8, 118.5, 38.3, 34.1, 30.2, 29.1 (x3); EI-HRMS calcd for C₁₃H₁₈O₂ (M⁺) 206.1307, found 206.1299.

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and *N*-Allyl-4-methylbenzenesulfonamide (23). RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and *N*-allyl-4-methylbenzenesulfonamide **23** at room temperature afforded **32** in 53% yield as colorless crystals: mp 137.0—138.0 °C (recryst. from CHCl₃-hexane); IR (KBr) 3336, 2976, 2922, 1638, 1611 cm⁻¹; ¹H NMR (CDCl₃) δ 12.12 (s, 1H), 7.73 (br d, *J* = 8 Hz, 2H), 7.69 (br d, *J* = 8 Hz, 1H), 7.47 (br t, *J* = 8 Hz, 1H), 7.28 (br d, *J* = 8 Hz, 2H), 6.98 (br d, *J* = 8 Hz, 1H), 6.89 (br t, *J* = 8 Hz, 1H), 4.50 (t, *J* = 6.3 Hz, 1H), 3.05—3.10 (m, 4H), 2.40 (s, 3H), 1.93 (quintet, *J* = 6.8 Hz, 2H); EI-HRMS calcd for C₁₇H₁₉NO₄S (M⁺) 333.1035, found 333.1013. Anal. Calcd for C₁₇H₁₉NO₄S: C, 61.24; H, 5.74; N, 4.20; Found: C, 61.28; H, 5.79; N, 4.14.

RhCl(PPh₃)₃-Catalyzed Intermolecular Hydroacylation between Salicylaldehyde (1) and 1-Hexyne (24). RhCl(PPh₃)₃-catalyzed hydroacylation between salicylaldehyde **1** and 1-hexyne **24** at room temperature afforded **33a** and **33b** in 91% yield as a mixture of isomers in the ratio of 1.1 to 1. **1-2-(Hydroxyphenyl)hept-2-en-1-one (33a).** A colorless oil: IR (neat) 3022, 2962, 1651, 1622, 1591 cm⁻¹; ¹H NMR (CDCl₃) δ 12.73 (s, 1H), 7.81 (br d, *J* = 8 Hz, 1H), 7.47 (br t, *J* = 8 Hz, 1H), 7.21 (dt, *J* = 15.1, 7.1 Hz, 1H), 6.98—7.05 (m, 2H), 6.90 (br t, *J* = 8 Hz, 1H), 2.36 (m, 2H), 1.53 (quintet, *J* = 7.3 Hz, 2H), 1.44 (sextet, *J* = 7.3 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H); EI-HRMS calcd for C₁₃H₁₆O₂ (M⁺) 204.1150, found 204.1163. **2-Butyl-1-(2-hydroxyphenyl)propenone (33b).** A colorless oil: IR (neat) 3026, 2962, 1624, 1603 cm⁻¹; ¹H NMR (CDCl₃) δ 11.99 (s, 1H), 7.75 (br d, *J* = 8 Hz, 1H), 7.48 (br t, *J* = 8 Hz, 1H), 7.01 (br d, *J* = 8

Hz, 1H), 6.87 (br t, $J = 8$ Hz, 1H), 5.65 (br s, 1H), 5.38 (s, 1H), 2.47 (t, $J = 7.3$ Hz, 2H), 1.44—1.49 (m, 2H), 1.38 (quintet, $J = 7.3$ Hz, 2H), 0.91 (t, $J = 7.3$ Hz, 3H); EI-HRMS calcd for $C_{13}H_{16}O_2$ (M^+) 204.1150, found 204.1173.

S1 Camps, F. Eta l., *Synth. Comm.* **1988**, *18*, 445.

C:\WINNR98\DATA\Yakka\Imai\0,%,',',%,G,...\allyl cyanide-Hydroacylation.als
 Allyl cyanide Hydroacylation

CC(C#N)C(=O)c1ccccc1C(=O)O

10b

C:\WINMR98\DATA\Yakka\Imai\□,%,',',%,&,...\4-Pentenitrile Hydroacylation.als
4-Pentenitrile Hydroacylation

N#CCCCC(=O)c1ccccc1O

13C NMR spectrum of compound 10. The x-axis represents chemical shift in PPM, ranging from 0 to 200. The spectrum shows three stacked traces (top, middle, bottom) with corresponding peak assignments.

Chemical Shift (PPM)	Assignment
205.190	Carbonyl (C=O)
162.385	Aromatic/Quinone
136.423	Aromatic/Quinone
129.669	Aromatic/Quinone
119.289	Aromatic/Quinone
119.106	Aromatic/Quinone
118.940	Aromatic/Quinone
118.536	Aromatic/Quinone
77.316	Solvent (CDCl3)
77.200	Solvent (CDCl3)
77.000	Solvent (CDCl3)
76.680	Solvent (CDCl3)
36.994	Aliphatic
24.855	Aliphatic
22.971	Aliphatic
17.091	Aliphatic

N#CCCCC(=O)c1ccccc1O

C:\WINNMR98\DATA\Yakka\Imai\□,%,',',%,@,...\6-Heptenenitrile recolumn.als
6-Heptenenitrile recolumn

13a

\\A1400\data\Yakka\main\13C\13C1BOM_E1_FT.als
MI-53 13C

DFILE
COMMT
DATIM
ORNUC
EXMOD
OFRQ
OBSF
OBSF
POINT
FREQU
SCANS
ACQTM
PD
PW1
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9-Decenenitrile hydroacylation

14a

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MI-96 13C

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MI-199

28a

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MI-199 13C

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\\A1400\data\yakka\Imai\1-Octene Hydroacylation.als
1-Octene Hydroacylation

FILE \\A1400\data\yakka\Imai\1-
 COUNT 1-Octene Hydroacylation
 DATE Thu Jan 15 16:13:58 2004
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQ 8000.00 Hz
 SCANS 64
 ACQTM 4.0960 sec
 PD 2.9040 sec
 PW1 6.00 usec
 IRNUC 1H
 CTMP 25.0 C
 SLVT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 15

C:\WINNMR98\DATA\Yakka\Imai\additive nitrile\MI-172.als
MI-172

C:\WINNMR8\DATA\YAKKAI\met1\1721BOM_EL_FT_als
 172

DT FILE
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 RGAIN

31

C:\WINMR98\DATA\Yakka\Imai\additive nitrile\N-Ts allylamine hydroacylation CDCl3.als
 N-Ts allylamine hydroacylation CDCl3

32a

C:\WINNMR98\DATA\Yakka\Imai\additive nitrile\MI-154-B.als
MI-154-B

33a

C:\WINNR98\DATA\Yakka\Imai\additive nitrile\MI-154-A Fr11-12.als
MI-154-A Fr 11-12

33b