Supporting Information

Initiator-Dependent Chemoselective Addition of THF Radical to Aldehyde and Aldimine and Its Application to a Three-Component Reaction

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General

¹H NMR (500 MHz) and ¹³C NMR (125 MHz) were measured in CDCl₃. Chemical shift values were expressed in ppm downfield from internal tetramethylsilane. IR spectroscopy of oil and solid samples were measured as neat liquid films and KBr pellets, respectively. The wave-numbers of maximum absorption peaks of IR spectroscopy were presented in cm⁻¹.

***N*-(4-Formylbenzylidene)-4-toluenesulfonamide (5):** prepared from 4-toluenesulfonamide and terephthalaldehyde (3 equiv) according to the reported procedure (Love, B. E.; Raje, P. S.; Williams, T. C. II *Synlett* **1994**, 493–494.) Recrystallization from ethyl acetate gave colorless needles of mp >300 °C. ¹H NMR: 2.46 (s, 3H), 7.38 (d, *J* = 8.3 Hz, 2H), 7.91 (d, *J* = 8.3 Hz, 2H), 7.99 (d, *J* = 8.3 Hz, 2H), 8.10 (d, *J* = 8.3 Hz, 2H), 9.09 (s, 1H). ¹³C NMR: 21.7, 128.3, 129.9, 130.0, 131.6, 134.4, 137.0, 140.1, 145.1, 168.5, 191.2. IR: 1700, 1600, 1570, 1320, 1160, 1090. EIMS (*m/z*): 287 (M⁺), 155 (Ts), 91 (tolyl). Anal. Calcd for C₁₅H₁₃NO₃S: C, 62.70; H, 4.56; N, 4.87. Found: C, 62.41; H, 4.66; N, 4.86.

Typical procedure for the THF radical addition: to a solution of an acceptor (1 mmol) in THF (22mL, 0.27 mol) was added the indicated amount of a radical initiator (1.0 M solution in hexane) at room temperature. The reaction mixture was stirred at the same temperature with continuous air bubbling (flow rate; 0.5 mL/h). After the indicated period, saturated aqueous NaHCO₃ was added to the mixture, and the organic was separated. The aqueous layer was extracted with ethyl acetate three times, and the combined organic layers were washed with brine, dried over Na₂SO₄, and then concentrated. The resulting crude mixture was purified by silica gel column chromatography.

***N*-(4-Formylphenyl)(oxolan-2-yl)methyl]-4-toluenesulfonamide (6):** the diastereomer ratio was 58/42 (less polar/more polar isomer). The diastereomers were separated by column chromatography (hexane/EtOAc = 3/1).

The less polar isomer (**6-1**) was recrystallized from EtOAc to give colorless prisms of mp 132.0–133.0 °C. ¹H-NMR: 1.68–1.92 (m, 4H), 2.32 (s, 3H), 3.70–3.82 (m, 2H), 3.94–3.98 (m, 1H), 4.28 (dd, *J* = 5.5, 5.5 Hz, 1H), 5.61–5.68 (m, 1H), 7.08 (d, *J* = 7.9 Hz, 2H), 7.29 (d, *J* = 7.9 Hz, 2H), 7.50 (d, *J* = 8.2 Hz, 2H), 7.67 (d, *J* = 8.2 Hz, 2H), 9.94 (s, 1H). ¹³C NMR: 21.3, 25.5, 28.4, 60.7, 68.7, 81.3, 127.0, 128.1, 129.2, 129.6, 135.6, 137.1, 143.2, 145.5, 191.7. IR: 3140, 1700, 1600, 1330, 1160, 1090, 1060. EIMS (*m/z*): 289 (M⁺ + 1 – C₄H₈O), 155 (Ts), 91 (tolyl), 71 (C₄H₈O). Anal. Calcd for C₁₉H₂₁NO₄S: C, 63.49; H, 5.89; N, 3.90. Found: C, 63.33; H, 5.90; N, 3.83.

The more polar isomer (**6-2**) was recrystallized from EtOAc to give colorless prisms of mp 138.0–139.0 °C. ¹H-NMR: 1.46–1.54 (m, 2H), 1.68–1.87 (m, 2H), 2.32 (s, 3H), 3.63–3.70 (m, 2H), 4.10–4.16 (m, 1H), 4.21 (dd, *J* = 4.6, 7.3 Hz, 1H), 5.52 (d, *J* = 7.3 Hz, 1H), 7.07 (d, *J* = 7.4 Hz, 2H), 7.27 (d, *J* = 7.4 Hz, 2H), 7.48 (d, *J* = 7.9 Hz, 2H), 7.66 (d, *J* = 7.9 Hz, 2H), 9.94 (s, 1H). ¹³C NMR: 21.4, 25.4, 27.6, 60.4, 68.8, 81.0, 127.0, 128.8, 129.3, 129.3, 135.6, 137.1, 143.1, 144.2, 191.7. IR: 3260, 1690, 1610, 1330, 1160, 1090, 1070. EIMS (*m/z*): 289 (*M*⁺ + 1 – C₄H₈O), 155 (Ts), 91 (tolyl), 71 (C₄H₈O). Anal. Calcd for C₁₉H₂₁NO₄S: C, 63.49; H, 5.89; N, 3.90. Found: C, 63.52; H, 5.89; N, 3.90.

***N*-(1-{4-[Hydroxy(oxolan-2-yl)methyl]phenyl}propyl)-4-toluenesulfonamide (7):** the ratio of four diastereomers was 7:7:43:43. After column chromatography (hexane/EtOAc = 1/1), the first two diastereomers were obtained as a less polar mixture and the last two as a more polar mixture.

The less polar mixture (**7-1**) as a pale yellow oil. ¹H NMR: 0.77 (t, *J* = 7.3 Hz, 3H), 1.48–1.56 (m, 1H), 1.65–1.93 (m, 5H), 2.36 (s, 3H), 2.44 (br s, 1H), 3.79–3.83 (m, 1H), 3.90–3.94 (m, 1H), 3.98–4.03 (m, 1H), 4.17–4.21 (m, 1H), 4.86 (d, *J* = 3.4 Hz, 1H), 4.96 (br s, 1H), 6.99–7.01 (m, 2H), 7.13 (d, *J* = 7.3 Hz, 2H), 7.17 (d, *J* = 7.9 Hz, 2H), 7.56 (d, *J* = 7.9 Hz, 2H). ¹³C NMR: 10.3, 21.4, 24.6, 26.0, 30.5, 59.4, 59.5, 69.0, 73.6, 73.6, 83.0, 126.0, 126.4, 127.0, 129.3, 137.8, 139.7, 139.9, 143.0. IR: 3450, 3280, 1600, 1320, 1160, 1100, 1050. EIMS (*m/z*): 388 (*M*⁺ – 1), 360 (*M*⁺ – Et), 318 (*M*⁺ – C₄H₈O), 242, 212, 155 (Ts), 148, 91 (tolyl), 71 (C₄H₈O). HRMS–FAB (*m/z*): calcd for C₂₁H₂₇NO₄S, 389.1661; found, 389.1667.

The more polar mixture (**7-2**) as a pale yellow oil. ¹H NMR: 0.76 (t, *J* = 7.3 Hz, 3H), 1.50–2.00 (m, 6H), 2.35 (s, 3H), 2.99 (br s, 1H), 3.8–4.0 (m, 3H), 4.19 (ddd, *J* = 7.0, 7.0, 7.0 Hz, 1H), 4.36 (d, *J* = 7.0 Hz, 1H), 5.1–5.3 (m, 1H), 7.00 (d, *J* = 7.0 Hz, 2H), 7.12 (d, *J* = 7.9 Hz, 2H), 7.16 (d, *J* = 7.9 Hz, 2H), 7.55 (d, *J* = 7.0 Hz, 2H). ¹³C NMR: 10.3, 21.3, 26.0, 27.9, 30.5, 59.4, 68.4, 76.5, 83.3, 126.6, 126.6, 127.0, 127.0, 129.3, 137.8, 137.8, 139.8, 140.5, 140.6, 142.9. IR: 3470, 3280, 1600, 1450, 1320, 1160, 1090, 1050. EIMS (*m/z*): 388 (*M*⁺ – 1), 360 (*M*⁺ – Et), 318 (*M*⁺ – C₄H₈O), 242, 212, 155 (Ts), 148, 91 (tolyl), 71 (C₄H₈O). HRMS–FAB (*m/z*): calcd for C₂₁H₂₇NO₄S, 389.1661; found, 389.1651.

***N*-({4-[Hydroxy(oxolan-2-yl)methyl]phenyl}(oxolan-2-yl)methyl)-4-toluenesulfonamide (8):** the ratio of four diastereomers was 7:7:43:43. After column chromatography (hexane/EtOAc = 1/2), the first two diastereomers were obtained as a less polar mixture and the last two as a more polar mixture.

The less polar mixture (**8-1**) as a colorless oil. ¹H NMR: 1.53–1.95 (m, 8H), 2.33 (s, 3H), 2.30–2.37 (m, 1H), 3.71–4.03 (m, 6H), 4.16–4.19 (m, 1H), 4.83–4.87 (m, 1H), 5.38 (d, *J* = 5.2 Hz), 7.06–7.14 (m, 6H), 7.46–7.48 (m, 2H). ¹³C NMR: 21.4, 24.6, 25.6, 26.1, 28.5, 60.9, 61.0, 68.7, 69.1, 73.6, 81.7, 81.8, 82.96, 83.00, 125.9, 127.2, 127.41, 127.45, 129.09, 129.12, 137.6, 137.7, 139.8, 139.9, 142.8. IR: 3400, 3290, 1600, 1330, 1160, 1100, 1060. EIMS (*m/z*): 360 (*M*⁺ – C₄H₈O), 343 (*M*⁺ – OH – C₄H₈O), 276 (*M*⁺ – Ts), 242, 206, 190, 155 (Ts), 148, 91 (tolyl), 71 (C₄H₈O). HRMS–FAB (*m/z*): [*M* – H]⁺ calcd for C₂₃H₂₈NO₅S, 430.1688; found, 430.1695.

The more polar mixture (**8-2**) as a colorless oil. ¹H NMR: 1.53–1.95 (m, 8H), 2.33 (s, 3H), 2.96 (br s, 1H), 3.70–3.94 (m, 6H), 4.16–4.19 (m, 1H), 4.35 (d, *J* = 7.7 Hz, 1H), 5.47 (d, *J* = 4.9 Hz), 7.01–7.15 (m, 6H), 7.46–7.48 (m, 2H). ¹³C NMR: 21.4, 25.5, 26.0, 27.9, 28.5, 60.87, 60.91, 68.4, 68.59, 68.61, 76.6, 81.6, 81.7, 83.3, 126.8, 127.09, 127.10, 127.46, 127.53, 129.0, 129.1, 137.47, 137.49, 138.1, 138.2, 140.0, 142.7, 142.8. IR: 3300, 1650, 1600, 1380, 1160, 1100, 1060. EIMS (*m/z*): 414 (*M*⁺ – OH), 360 (*M*⁺ – C₄H₈O), 343 (*M*⁺ – OH – C₄H₈O), 276 (*M*⁺ – Ts), 242, 206, 190, 155 (Ts), 148, 91 (tolyl), 71 (C₄H₈O). HRMS–FAB (*m/z*): [*M* – H]⁺ calcd for C₂₃H₂₈NO₅S, 430.1688; found, 430.1683.

General procedure for the three-component reaction (Table 2). *N*-(4-Methoxyphenyl)- α -phenyloxolane-2-methylamine (**3a**): to a solution of 1 mmol of benzaldehyde (**2a**) and 1 mmol of *p*-anisidine (**9a**) in 22 mL of THF was added 12 mmol of dimethylzinc (1.0 M solution in hexane), and the mixture was stirred at room temperature for 22 h with continuous air bubbling (flow rate; 0.5 mL/h). To the reaction mixture was added saturated aqueous NaHCO₃ (25 mL), and then the mixture was extracted with EtOAc three times. The combined organic layers were washed with brine and dried over Na₂SO₄. Concentration and purification by silica gel column chromatography (hexane/acetone = 19/1) gave **3a** with the diastereomer ratio of 51/49 (less polar/more polar isomer) in 74% yield.

TLC analysis and NMR spectra showed that **3a** was identical to the authentic sample (Yamada, K.; Fujihara, H.; Yamamoto, Y.; Miwa, Y.; Taga, T.; Tomioka, K.; *Org. Lett.* **2002**, *4*, 3509–3511).

α ,*N*-Diphenyloxolane-2-methylamine (3b): the flow rate of air bubbling was 10 mL/h. The diastereomer ratio was 53/47 (less polar/more polar isomer). Two diastereomers were separated by column chromatography (hexane/EtOAc 9/1).

The less polar isomer (**3b-1**) as a pale yellow oil. ¹H NMR: 1.75–1.95 (m, 4H), 3.77–3.81 (m, 1H), 3.89–3.93 (m, 1H), 4.03 (ddd, *J* = 6.4, 6.4, 6.4 Hz, 1H), 4.21 (d, *J* = 6.4 Hz, 1H), 4.76 (br s, 1H), 6.52 (d, *J* = 7.7 Hz, 2H), 6.61 (t, *J* = 7.3 Hz, 1H), 7.05 (dd, *J* = 7.3, 7.7 Hz, 2H), 7.21–7.24 (m, 1H), 7.31 (dd, *J* = 7.2, 8.0 Hz, 2H), 7.41 (d, *J* = 8.0 Hz, 2H). ¹³C NMR: 25.7, 28.8, 62.0, 83.0, 113.8, 117.4, 127.1, 127.3, 128.6, 128.9, 141.6, 147.8. IR: 3390, 1600, 1500, 1060. EIMS (*m/z*): 253 (M⁺), 182 (M⁺ – C₄H₈O). HRMS–EI (*m/z*): calcd for C₁₇H₁₉NO, 253.1467; found, 253.1477.

The more polar isomer (**3b-2**) as a pale yellow oil. ¹H NMR: 1.54–1.61 (m, 1H), 1.73–1.80 (m, 3H), 3.73–3.82 (m, 2H), 4.23–4.27 (m, 1H), 4.43 (d, *J* = 4.3 Hz, 1H), 4.56 (br s, 1H), 6.52 (d, *J* = 8.0 Hz, 2H), 6.62 (t, *J* = 7.3 Hz, 1H), 7.06 (dd, *J* = 7.3, 8.0 Hz, 2H), 7.23 (t, *J* = 7.3 Hz, 1H), 7.30 (dd, *J* = 7.3, 7.3 Hz), 7.38 (d, *J* = 7.3 Hz, 2H). ¹³C NMR: 25.5, 27.2, 60.8, 68.8, 82.0, 113.9, 117.4, 127.2, 127.7, 128.3, 128.9, 140.0, 147.4. IR: 3390, 1600, 1500, 1060. EIMS (*m/z*): 253 (M⁺), 182 (M⁺ – C₄H₈O). HRMS–EI (*m/z*): calcd for C₁₇H₁₉NO, 253.1467; found, 253.1461.

α -Phenyl-*N*-(4-trifluoromethylphenyl)oxolane-2-methylamine (3c): the diastereomer ratio was 54/46 (less polar/more polar isomer). The diastereomers were separated by column chromatography (hexane/EtOAc = 3/1).

The less polar isomer (**3c-1**) as a pale yellow oil. ¹H NMR: 1.80–1.99 (m, 4H), 3.80–3.84 (m, 1H), 3.92–3.97 (m, 1H), 4.02–4.06 (m, 1H), 4.23–4.25 (m, 1H), 5.08 (br s, 1H), 6.53 (d, *J* = 8.6 Hz, 2H), 7.26–7.39 (m, 7H). ¹³C NMR: 25.7, 28.9, 61.8, 68.7, 82.9, 113.1, 119.0 (q, ²*J*_{CF} = 32.4 Hz), 125.1 (q, ¹*J*_{CF} = 270.4 Hz), 126.5 (q, ³*J*_{CF} = 3.6 Hz), 127.1, 127.8, 128.9, 140.9, 150.4. IR: 1620, 1530, 1330, 1110, 1060. EIMS (*m/z*): 321 (M⁺), 250 (M⁺ – C₄H₈O), 71(C₄H₈O). HRMS–EI (*m/z*): calcd for C₁₈H₁₈F₃NO, 321.1340; found, 321.1331.

The more polar isomer (**3c-2**) as a pale yellow oil. ¹H NMR: 1.48–1.54 (m, 1H), 1.66–1.84 (m, 3H), 3.73–3.79 (m, 2H), 4.28 (ddd, *J* = 4.2, 6.9, 6.9 Hz, 1H), 4.44 (m, 1H), 4.97 (br s, 1H), 6.52 (d, *J* = 8.6 Hz, 2H), 7.24–7.36 (m, 7H). ¹³C NMR: 25.4, 27.2, 60.4, 68.8, 81.6, 112.9, 118.9 (q, ²*J*_{CF} = 31.9 Hz), 125.0 (q, ¹*J*_{CF} = 268.6 Hz), 126.4 (q, ³*J*_{CF} = 4.1 Hz), 127.6, 127.8, 128.4, 139.0, 149.9. IR: 1620, 1530, 1060. EIMS (*m/z*): 321 (M⁺), 250 (M⁺ – C₄H₈O), 71(C₄H₈O). HRMS–EI (*m/z*): calcd for C₁₈H₁₈F₃NO, 321.1340; found, 321.1344.

α -(4-Chlorophenyl)-*N*-(4-methoxyphenyl)oxolane-2-methylamine (3d): the diastereomer ratio was 50/50 (less polar/more polar isomer). The diastereomers were separated by column chromatography (hexane/EtOAc = 5/1).

The less polar isomer (**3d-1**) as a pale yellow oil. ^1H NMR: 1.75–1.95 (m, 4H), 3.68 (s, 3H), 3.80 (ddd, $J = 6.7, 7.5, 7.5$ Hz, 1H), 3.90 (ddd, $J = 6.7, 6.7, 8.3$ Hz, 1H), 3.97 (ddd, $J = 6.7, 6.7, 6.7$ Hz, 1H), 4.12 (d, $J = 6.7$ Hz, 1H), 4.51 (br s, 1H), 6.45 (d, $J = 8.9$ Hz, 2H), 6.66 (d, $J = 8.9$ Hz, 2H), 7.28 (d, $J = 8.6$ Hz, 2H), 7.35 (d, $J = 8.6$ Hz, 2H). ^{13}C NMR: 25.7, 28.7, 55.7, 62.4, 68.6, 82.8, 114.6, 115.1, 128.6, 128.7, 132.9, 140.4, 141.6, 152.2. IR: 3380, 1510, 1240, 1060, 1040. EIMS (m/z): 317 (M^+), 246 ($\text{M}^+ - \text{C}_4\text{H}_8\text{O}$), 71 ($\text{C}_4\text{H}_8\text{O}$). HRMS–EI (m/z): calcd for $\text{C}_{18}\text{H}_{20}\text{ClNO}_2$, 317.1183; found, 317.1194.

The more polar isomer (**3d-2**) as a pale yellow oil. ^1H NMR: 1.55–1.85 (m, 4H), 3.68 (s, 3H), 3.70–3.85 (m, 2H), 4.15–4.25 (m, 1H), 4.30 (br s, 1H), 4.34 (d, $J = 4.3$ Hz, 1H), 6.45 (d, $J = 8.9$ Hz, 2H), 6.66 (d, $J = 8.9$ Hz, 2H), 7.26 (d, $J = 8.6$ Hz, 2H), 7.31 (d, $J = 8.6$ Hz, 2H). ^{13}C NMR: 25.5, 27.2, 55.6, 61.0, 68.8, 81.9, 114.6, 115.2, 128.5, 129.1, 132.9, 138.9, 141.2, 152.2. IR: 3380, 1510, 1240, 1060, 1040. EIMS (m/z): 317 (M^+), 246 ($\text{M}^+ - \text{C}_4\text{H}_8\text{O}$), 71 ($\text{C}_4\text{H}_8\text{O}$). HRMS–EI (m/z): calcd for $\text{C}_{18}\text{H}_{20}\text{ClNO}_2$, 317.1183; found, 317.1183.

α ,*N*-Bis(4-methoxyphenyl)oxolane-2-methylamine (3e): the diastereomer ratio was 50/50 (less polar/more polar isomer). The diastereomers were separated by column chromatography (hexane/EtOAc = 4/1).

The less polar isomer (**3e-1**) as a pale yellow oil. ^1H NMR: 1.69–1.92 (m, 4H), 3.65 (s, 3H), 3.75 (s, 3H), 3.75–3.81 (m, 1H), 3.88 (ddd, $J = 7.1, 7.3, 7.3$ Hz, 1H), 3.96 (ddd, $J = 6.7, 7.0, 7.3$ Hz, 1H), 4.06 (d, $J = 6.7$ Hz, 1H), 4.50 (br s, 1H), 6.47 (d, $J = 8.9$ Hz, 2H), 6.64 (d, $J = 8.9$ Hz, 2H), 6.84 (d, $J = 8.6$ Hz, 2H), 7.31 (d, $J = 8.6$ Hz, 2H). ^{13}C NMR: 25.7, 28.7, 55.2, 55.7, 62.6, 68.4, 83.1, 113.9, 114.6, 115.1, 128.2, 133.7, 142.1, 152.0, 158.8. IR: 3380, 1610, 1510, 1240, 1060, 1030. EIMS (m/z): 313 (M^+), 242 ($\text{M}^+ - \text{C}_4\text{H}_8\text{O}$), 71 ($\text{C}_4\text{H}_8\text{O}$). HRMS–EI (m/z): calcd for $\text{C}_{19}\text{H}_{23}\text{NO}_3$, 313.1678; found, 313.1677.

The more polar isomer (**3e-2**) as a pale yellow oil. ^1H NMR: 1.53–1.66 (m, 1H), 1.70–1.80 (m, 3H), 3.67 (s, 3H), 3.72–3.81 (m, 3H), 3.76 (s, 3H), 4.19 (ddd, $J = 4.3, 7.0, 7.0$ Hz, 1H), 4.25 (br s, 1H), 4.32 (d, $J = 4.3$ Hz, 1H), 6.48 (d, $J = 8.9$ Hz, 2H), 6.65 (d, $J = 8.9$ Hz, 2H), 6.83 (d, $J = 8.6$ Hz, 2H), 7.27 (d, $J = 8.6$ Hz, 2H). ^{13}C NMR: 25.5, 27.1, 55.1, 55.6, 60.9, 68.7, 82.3, 113.7, 114.6, 115.2, 128.7, 132.2, 141.6, 152.0, 158.7. IR: 3380, 1610, 1510, 1240, 1060, 1040. EIMS (m/z): 313 (M^+), 242 ($\text{M}^+ - \text{C}_4\text{H}_8\text{O}$), 71 ($\text{C}_4\text{H}_8\text{O}$). HRMS–EI (m/z): calcd for $\text{C}_{19}\text{H}_{23}\text{NO}_3$, 313.1678; found, 313.1670.

***N*-(4-Methoxyphenyl)- α -(2-phenylethyl)oxolane-2-methylamine (3f):** the diastereomer ratio was 40/60 (less polar/more polar isomer). TLC analysis and NMR spectra showed that **3f** was identical to the authentic sample (Yamada, K.; Fujihara, H.; Yamamoto, Y.; Miwa, Y.; Taga, T.; Tomioka, K.; *Org. Lett.* **2002**, *4*, 3509–3511).

α -Cyclohexyl-*N*-(4-methoxyphenyl)oxolane-2-methylamine (3g): the diastereomer ratio was 34/66 (less polar/more polar isomer). The diastereomers were separated by column chromatography (hexane/EtOAc = 8/1).

The less polar isomer (**3g-1**) as a pale yellow oil. ^1H NMR: 0.96–1.26 (m, 5H), 1.54–1.87 (m, 10H), 3.06 (dd, $J = 1.3, 6.4$ Hz, 1H), 3.67–3.78 (m, 2H), 3.72 (s, 3H), 3.83–3.89 (m, 1H), 4.08–4.13 (m,

1H), 6.51 (d, $J = 9.2$ Hz, 2H), 6.73 (d, $J = 9.2$ Hz, 2H). ^{13}C NMR: 26.0, 26.4, 26.4, 26.5, 29.2, 30.2, 30.3, 42.2, 55.8, 60.9, 68.9, 78.9, 113.3, 115.0, 143.9, 151.0. IR: 3400, 1510, 1230, 1040. EIMS (m/z): 289 (M^+), 218 ($\text{M}^+ - \text{C}_4\text{H}_8\text{O}$), 71 ($\text{C}_4\text{H}_8\text{O}$). HRMS–EI (m/z): calcd for $\text{C}_{18}\text{H}_{27}\text{NO}_2$, 289.2042; found, 289.2052.

The more polar isomer (**3g-2**) as a pale yellow oil. ^1H NMR: 1.03–1.30 (m, 5H), 1.60–1.96 (m, 10H), 3.17 (br s, 1H), 3.18 (dd, $J = 4.0, 7.3$ Hz, 1H), 3.70–3.77 (m, 1H), 3.73 (s, 3H), 3.84 (ddd, $J = 6.7, 7.0, 8.0$ Hz, 1H), 3.89 (ddd, $J = 7.0, 7.0, 7.3$ Hz, 1H), 6.59 (d, $J = 8.9$ Hz, 2H), 6.73 (d, $J = 8.9$ Hz, 2H). ^{13}C NMR: 25.8, 26.3, 26.5, 26.5, 27.2, 28.9, 31.0, 40.5, 55.8, 62.8, 68.1, 80.1, 114.1, 114.8, 143.5, 151.5. IR: 3400, 1510, 1240, 1060, 1040. EIMS (m/z): 289 (M^+), 218 ($\text{M}^+ - \text{C}_4\text{H}_8\text{O}$), 71 ($\text{C}_4\text{H}_8\text{O}$). HRMS–EI (m/z): calcd for $\text{C}_{18}\text{H}_{27}\text{NO}_2$, 289.2042; found, 289.2038.

6-1 less polar

6-1 less polar

6-2 more polar

6-2 more polar

7-1 less polar

7-2 more polar

7-2 more polar

8-1 less polar

8-1 less polar

8-2 more polar

8-2 more polar

3b-1 less polar

3b-1 less polar

3b-2 more polar

3b-2 more polar

3c-1 less polar

3c-1 less polar

3c-2 more polar

3c-2 more polar

3d-2 more polar

3d-2 more polar

3e-1 less polar

3e-1 less polar

3e-2 more polar

3e-2 more polar

3g-1 less polar

3g-1 less polar

3g-2 more polar

3g-2 more polar