Supporting Information

Title

Palladium-catalyzed Reaction of Acylzirconocene Chloride and Stereoselective Formation of Bicyclo[3.3.0]compounds

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Preparation of a solution of acylzirconocene chloride complex **1** in THF

Under Ar atmosphere, to a suspension of $\text{Cp}_2\text{Zr(H)Cl}$ (1 mmol, 1.0 equiv) in CH_2Cl_2 (3 mL) was added alkene or alkyne (2.0 mmol) and the mixture was stirred at ambient temperature for 0.5 h. The reaction mixture was further stirred under CO atmosphere (1 atm) for 2 h at ambient temperature. The clear solution was concentrated to dryness *in vacuo* by using vacuum pump (10^{-3} mmHg) to give acylzirconocene chloride powder. After the powder was further subjected to vacuum for 2 h, a solution of **1** in THF was prepared by the addition of anhydrous THF (6 mL) to the powder, and the solution was directly used as a 1 mmol solution for the next step.

Typical procedure for the formation of **4** (entry 6, Table 1)

To a solution of n-nonanoylzirconocene chloride (1 mmol) (**1a**) in THF (6 mL) was added successively a solution of (*E*)-1-phenyl-4-(2-propenyloxy)-2-buten-1-one (101 mg, 0.5 mmol) in ether (12 mL), 1M solution of Me_2Zn in hexane (0.5 mmol) and $\text{Pd}(\text{OAc})_2$ (0.05 mmol) at 0 °C, and the mixture was stirred at ambient temperature for 3 h. The reaction mixture darkened and became turbid. After being quenched with aq. NaHCO_3 under ice cooling, the mixture was extracted with ethyl acetate and dried over MgSO_4 . The filtered solution was concentrated to dryness *in vacuo*, providing a crude product. Purification by silica gel column chromatography (hexanes/ethyl acetate = 10 : 1) afforded the product (143 mg, 83% yield). Yield was calculated from (*E*)-1-phenyl-4-(2-propenyloxy)-2-buten-1-one (101 mg, 0.5 mmol).

[(3*aS**,4*R**,5*R**,6*aS**)-5-hydroxy-5-octylhexahydro-1H-cyclopenta[c]furan-4-yl](phenyl)methanone (entry 6, Table 1).

Colorless crystals; mp 62 °C; ^1H NMR (400 MHz, CDCl_3): δ = 0.81 (t, J = 7.0 Hz, 3H), 1.12-1.37 (m, 12H), 1.41-1.54 (m, 3H), 2.23 (dd, J = 13.0, 7.9 Hz, 1H), 3.03-3.13 (m, 2H), 3.39 (dd, J = 9.7, 5.2 Hz, 1H), 3.56 (dd, J = 15.7, 6.7 Hz, 2H), 3.73 (d, J = 9.7 Hz, 2H), 4.48 (s, 1H), 7.47-7.98 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3): δ = 14.0, 22.6, 24.9, 29.1, 29.4, 30.0, 31.8, 39.9, 43.1, 46.1, 50.7, 56.8, 72.0, 74.2, 87.2, 128.5, 128.7, 134.1, 137.7, 206.6; IR (neat): ν = 3400, 1670 cm^{-1} ; EIMS m/z 344 (M^+); Anal. Calcd for $\text{C}_{22}\text{H}_{32}\text{O}_3$: C, 76.70; H, 9.36. Found: C, 76.75; H, 9.28.

[(1*R**,2*R**,3*aS**,6*aS**)-2hydroxy-2-octyloctahydro-1- pentalenyl](phenyl)methanone (entry 1, Table 1)

Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ = 0.84 (t, J = 7.1 Hz, 3H), 1.12 -1.37 (m, 14H), 1.38-1.53 (m, 4H), 1.58-1.72 (m, 3H), 2.15 (dd, J = 13.0, 8.0 Hz, 1H), 2.82-2.96 (m, 2H), 3.32 (d, J = 9.3 Hz, 1H), 4.46 (bd, J = 1.8 Hz, 1H), 7.46-7.64 (m, 3H), 7.91-7.94 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3): δ = 14.1, 22.6, 25.0, 25.4, 29.1, 29.4, 30.1, 31.8, 31.9, 32.8, 40.4, 42.0, 46.8, 49.8, 58.0, 86.8, 128.4, 128.8, 133.7, 138.2, 207.7; IR (neat): ν = 3460, 1653 cm^{-1} ; EIMS m/z 343 ($\text{M}^+ + 1$); HRMS Calcd for $\text{C}_{23}\text{H}_{34}\text{O}_2$: 342.25724, Found: 342.2559.

[(1*R**,2*R**,3*aS**,6*aS**)-2hydroxy-5,5-dimethyl-2-octyloctahydro-1-pentalenyl](phenyl)methanone (**4a**) (entry 2, Table 1)

Colorless crystals; mp 32 °C; ^1H NMR (400 MHz, CDCl_3): δ = 0.84 (t, J = 7.3 Hz, 3H), 0.88 (s, 3H), 1.07 (s, 3H), 1.13-1.40 (m, 14H), 1.44-1.63 (m, 4H), 1.76 (ddd, J = 12.4, 8.3, 2.0 Hz, 1H),

2.16 (dd, $J = 12.5, 8.2$ Hz, 1H), 2.87-3.14 (m, 2H), 3.41 (d, $J = 8.2$ Hz, 1H), 4.82 (bd, $J = 1.8$ Hz, 1H), 7.47-7.93 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3): $\delta = 14.4, 23.0, 25.3, 27.2, 29.5, 29.8, 30.5, 32.2, 41.0, 42.9, 45.0, 47.3, 48.3, 48.8, 50.3, 60.1, 89.4, 128.7, 129.1, 134.0, 138.3, 208.0$; IR (neat): $\nu = 3458, 1654\text{ cm}^{-1}$; EIMS m/z 370 (M^+); HRMS Calcd for $\text{C}_{25}\text{H}_{38}\text{O}_2$: 370.28578, Found: 370.28718.

[(1*R,2*R**,3*aS**,6*aS**)-2-hydroxy-5,5-dimethyl-2-(4-phenylbutyl)octahydro-1-pentalenyl](phenyl)methanone (4b) (entry 3, Table 1)**

Colorless crystals; mp 81.0 - 82.0 °C, ^1H NMR (400 MHz, CDCl_3) δ 0.88 (s, 3H), 1.07 (s, 3H), 1.17 (dd, $J = 12.2, 9.8$ Hz, 1H), 1.25 - 1.46 (m, 4H), 1.48 - 1.60 (m, 5H), 1.75 (ddd, $J = 12.2, 7.9, 1.8$ Hz, 1H), 2.18 (dd, $J = 13.0, 7.9$ Hz, 1H), 2.49 - 2.54 (m, 2H), 2.90 - 3.10 (m, 2H), 3.40 (d, $J = 9.5$ Hz, 1H), 4.89 (d, $J = 1.8$ Hz, 1H), 7.07 - 7.23 (m, 5H), 7.48 - 7.92 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 24.7, 26.8, 29.1, 32.0, 35.8, 40.5, 42.5, 44.6, 46.9, 47.9, 48.4, 49.9, 59.7, 88.9, 125.5, 128.2, 128.3, 128.4, 128.7, 133.7, 137.9, 142.6, 207.6; IR (neat) ν 3453, 1652 cm^{-1} ; EIMS m/z 391 ($\text{M}^+ + 1$); HRMS Calcd for $\text{C}_{27}\text{H}_{32}\text{O}$ ($\text{M}^+ - \text{H}_2\text{O}$): 372.24305, Found: 372.24532.

((1*R,2*R**,3*aS**,6*aS**)-2-hydroxy-2-octyl-5,5-bis{[(phenylmethyl)oxy]methyl}octahydro-1-pentalenyl)(phenyl)methanone (entry 4, Table 1)**

^1H NMR (400 MHz, CDCl_3) δ 0.79 (t, $J = 7.0$ Hz, 3H), 1.06 - 1.45 (m, 17H), 1.72 (dd, $J = 13.0, 7.7$ Hz, 1H), 1.92 (dd, $J = 13.0, 7.9$ Hz, 1H), 2.09 (dd, $J = 13.0, 7.7$ Hz, 1H), 2.79 - 2.96 (m, 2H), 3.21 (d, $J = 4.8$ Hz, 2H), 3.38 - 3.42 (m, 3H), 4.37 (s, 2H), 4.46 (s, 2H), 4.70 (bd, $J = 1.5$ Hz, 1H), 7.15 - 7.87 (m, 15H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 14.1, 22.6, 24.9, 29.1, 29.4, 30.1, 31.8, 37.8, 39.2, 40.7, 41.9, 47.0, 49.4, 53.3, 59.3, 72.5, 73.2, 73.3, 74.7, 76.7, 77.0, 77.3, 88.7, 127.3, 127.4, 128.2, 128.3, 128.4, 128.7, 133.6, 137.9, 138.6, 138.9, 207.4; IR (neat) ν 3453, 1651 cm^{-1} ; EIMS m/z 583 ($\text{M}^+ + 1$); Anal. Calcd for $\text{C}_{39}\text{H}_{50}\text{O}_4$: C, 80.37; H, 8.65. Found: C, 80.19; H, 8.65.

1-((1*R,2*R**,3*aS**,6*aS**)-2-hydroxy-2-octyl-5,5-bis{[(phenylmethyl)oxy]methyl}-octahydro-1-pentalenyl)-1-ethanone (entry 5, Table 1)**

^1H NMR (400 MHz, CDCl_3) δ 0.88 (t, $J = 7.1$ Hz, 3H), 1.20 - 1.62 (m, 17H), 1.92 - 2.10 (m, 3H), 2.15 (s, 3H), 2.50 (d, $J = 9.6$ Hz, 1H), 2.70 - 2.91 (m, 2H), 3.33 (s, 2H), 3.46 (d, $J = 5.4$ Hz, 2H), 4.07 (bd, $J = 1.8$ Hz, 1H), 4.48 (s, 2H), 4.52 (s, 2H), 7.23 - 7.38 (m, 10H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 14.1, 22.7, 24.8, 29.3, 29.5, 30.2, 31.9, 32.1, 39.0, 39.4, 40.4, 53.4, 66.0, 72.3, 73.2, 73.3, 74.7, 87.5, 127.4, 127.5, 128.3, 138.7, 138.8, 215.0; IR (neat) ν 3472, 1691 cm^{-1} ; EIMS m/z 502 ($\text{M}^+ - \text{H}_2\text{O}$); HRMS Calcd for $\text{C}_{34}\text{H}_{48}\text{O}_4$: 520.35526, Found: 520.35413.

{(3*aS,4*R**,5*S**,6*aS**)-5-hydroxy-5-[(*E*)-1-octenyl]hexahydro-1*H*-cyclopenta[*c*]furan-4-yl}(phenyl)methanone (entry 7, Table 1)**

Colorless crystals; mp 83-85 °C, ^1H NMR (400 MHz, CDCl_3) δ 0.79 (t, $J = 7.2$ Hz, 3H), 1.01 - 1.19 (m, 8H), 1.62 (dd, $J = 13.0, 6.7$ Hz, 1H), 1.78 - 1.92 (m, 2H), 2.19 (dd, $J = 13.3, 8.0$ Hz, 1H), 3.08 - 3.18 (m, 2H), 3.40 (dd, $J = 9.6, 5.4$ Hz, 1H), 3.58 (dd, $J = 9.0, 6.2$ Hz, 1H), 3.69 (d, $J = 9.0$ Hz, 1H), 3.72 - 3.78 (m, 2H), 4.50 (d, $J = 1.5$ Hz, 1H), 5.45 (d, $J = 15.4$ Hz, 1H), 5.64 (dt, $J = 15.4, 6.8$ Hz, 1H), 7.45 - 7.93 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 14.0, 22.4, 28.5, 28.9, 31.5, 31.9, 43.4, 47.7, 50.0, 57.7, 72.0, 74.0, 85.9, 128.6, 128.8, 130.4, 132.3, 133.9, 137.6, 205.6; IR (neat) ν 3381, 1668 cm^{-1} ; EIMS m/z 342 (M^+); Anal. Calcd for $\text{C}_{22}\text{H}_{30}\text{O}_3$: C, 77.16; H, 8.83. Found: C, 77.15; H, 8.94.

[(3*aS,4*R**,5*S**,6*aS**)-5-((*E*)-4-[(1,1-dimethylethyl)(diphenyl)silyl]oxy)-1-butenyl]-5-hydroxyhexahydro-1*H*-cyclopenta[*c*]furan-4-yl](phenyl)methanone (entry 8, Table 1)**

Colorless crystals; mp 112.0 - 115.0 °C, ^1H NMR (400 MHz, CDCl_3) δ 0.98 (s, 9H), 1.60 (ddd, $J = 13.1, 6.2, 1.8$ Hz, 1H), 2.12 - 2.20 (m, 4H), 3.07 - 3.18 (m, 2H), 3.41 (dd, $J = 9.7, 5.3$ Hz, 1H), 3.45 (dd, $J = 7.6, 3.2$ Hz, 1H), 3.49 (dd, $J = 7.6, 3.2$ Hz, 1H), 3.58 (dd, $J = 9.7, 5.3$ Hz, 1H), 3.75

(dd, $J = 8.9, 7.8$ Hz, 2H), 4.49 (s, 1H), 5.51 (d, $J = 15.4$ Hz, 1H), 5.67 (dt, $J = 15.4, 6.9$ Hz, 1H), 7.32 - 7.89 (m, 15H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 19.1, 26.8, 35.4, 43.4, 47.7, 50.0, 57.6, 63.4, 72.1, 74.1, 85.9, 126.6, 127.6, 128.6, 128.8, 129.5, 133.9, 134.4, 135.5, 137.6, 205.5; IR (neat) ν 3455, 1655 cm^{-1} ; EIMS m/z 483 ($\text{M}^+ - t\text{Bu}$); HRMS Calcd for $\text{C}_{30}\text{H}_{31}\text{O}_4\text{Si}$ ($\text{M}^+ - t\text{Bu}$) : 483.198418, Found: 483.199163.

1,1-dimethylethyl(3a*S,4*R**,5*R**,6a*S**)-5-hydroxy-5-octyl-4-(phenylcarbonyl)hexahydrocyclopenta[*c*]pyrrole-2(1*H*)-carboxylate (entry 9, Table 1)**

^1H NMR (400 MHz, CDCl_3) δ 0.84 (t, $J = 7.1$ Hz, 3H), 1.13 - 1.53 (m, 24H), 2.18 - 2.27 (m, 1H), 3.00 - 3.47 (m, 6H), 3.51 (d, $J = 9.2$ Hz, 1H), 4.50 (bs, 1H), 7.48 - 7.96 (m, 5H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 14.1, 22.6, 24.9, 28.5, 29.1, 29.4, 30.0, 31.8, 40.5, 41.7, 46.0, 49.9, 52.0, 77.2, 79.5, 86.8, 128.5, 129.0, 134.1, 137.7, 154.9, 206.1; IR (neat) ν 3460, 1697 cm^{-1} ; EIMS m/z 444 ($\text{M}^+ + 1$); Anal. Calcd for $\text{C}_{27}\text{H}_{41}\text{NO}_4$: C, 73.10; H, 9.32; N, 3.16. Found: C, 73.00; H, 9.39; N, 3.27.

[(1*R,2*R**,6a*R**)-2-hydroxy-5,5-dimethyl-2-octyl-1,2,4,5,6,6a-hexahydro-1-pentalenyl](phenyl)methanone (entry 10, Table 1)**

^1H NMR (400 MHz, CDCl_3) δ 0.85 (t, $J = 7.1$ Hz, 3H), 1.06 (s, 3H), 1.09 (s, 3H), 1.10 - 1.40 (m, 13H), 1.60 - 1.68 (m, 2H), 2.05 (d, $J = 17.0$ Hz, 1H), 2.14 (d, $J = 17.0$ Hz, 1H), 3.57 - 3.65 (m, 2H), 4.16 (s, 1H), 5.36 (s, 1H), 7.46 - 7.62 (m, 3H), 7.90 - 7.94 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 14.1, 22.6, 24.9, 29.1, 29.4, 30.0, 30.1, 30.7, 31.8, 39.9, 41.2, 58.5, 92.1, 124.0, 128.3, 128.6, 133.4, 138.1, 156.6, 204.1; IR (neat) ν 3447, 1666 cm^{-1} ; EIMS m/z 368 (M^+); Anal. Calcd for $\text{C}_{25}\text{H}_{36}\text{O}_2$: C, 81.47; H, 9.85. Found: C, 81.21; H, 9.80.

[(2*R,3a*S**)-2-hydroxy-5,5-dimethyl-2-octyl-2,3,3a,4,5,6-hexahydro-1-pentalenyl](phenyl)methanone (entry 1, Table 2)**

^1H NMR (400 MHz, CDCl_3) δ 0.85 (t, $J = 7.0$ Hz, 3H), 0.92 (s, 3H), 1.05 (s, 3H), 1.16 (dd, $J = 12.0, 11.0$ Hz, 1H), 1.20 - 1.44 (m, 12H), 1.65 (dd, $J = 18.5, 1.7$ Hz, 1H), 1.72 - 1.85 (m, 4H), 1.89 (d, $J = 18.5$ Hz, 1H), 2.41 (dd, $J = 12.7, 7.6$ Hz, 1H), 3.18 (quint, $J = 9.0$ Hz, 1H), 5.11 (s, 1H), 7.42 - 7.66 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 14.1, 22.6, 24.9, 29.2, 29.5, 30.1, 30.3, 30.4, 31.8, 41.0, 42.2, 48.4, 91.9, 128.4, 128.7, 132.3, 133.6, 139.7, 171.4, 197.3; IR (neat) ν 3460, 1622 cm^{-1} ; EIMS m/z 368 (M^+); HRMS Calcd for $\text{C}_{25}\text{H}_{34}\text{O}$ ($\text{M}^+ - \text{H}_2\text{O}$): 349.25314, Found: 349.25311.

[(5*R,6a*S**)-5-hydroxy-5-octyl-3,5,6,6a-tetrahydro-1*H*-cyclopenta[*c*]furan-4-yl](phenyl)methanone (entry 2, Table 2)**

^1H NMR (400 MHz, CDCl_3) δ 0.86 (t, $J = 7.1$ Hz, 3H), 1.18 - 1.50 (m, 12H), 1.79 - 1.92 (m, 3H), 2.40 (dd, $J = 12.8, 7.3$ Hz, 1H), 3.27 (dd, $J = 10.4, 7.9$ Hz, 1H), 3.37 (quint, $J = 8.1$ Hz, 1H), 3.73 (dd, $J = 16.0, 1.3$ Hz, 1H), 4.07 (dd, $J = 16.0, 1.3$ Hz, 1H), 4.17 (dd, $J = 7.9, 7.9$ Hz, 1H), 5.02 (s, 1H), 7.44 - 7.64 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 14.1, 22.6, 24.8, 29.2, 29.5, 30.0, 31.8, 40.9, 42.2, 49.1, 65.7, 128.7, 132.9, 133.8, 139.4, 165.9, 195.9; IR (neat) ν 3460, 1625 cm^{-1} ; EIMS m/z 343 ($\text{M}^+ + 1$); Anal. Calcd for $\text{C}_{22}\text{H}_{30}\text{O}_3$: C, 77.16; H, 8.83. Found: C, 76.82; H, 8.70.

[(2*R,3a*S**)-2-hydroxy-2-octyl-5,5-bis{[(phenylmethyl)oxy]methyl}-2,3,3a,4,5,6-hexahydro-1-pentalenyl](phenyl)methanone (entry 3, Table 2)**

^1H NMR (500 MHz, CDCl_3) δ 0.86 (t, $J = 7.1$ Hz, 3H), 1.14 (dd, $J = 12.8, 10.6$ Hz, 1H), 1.18 - 1.40 (m, 12H), 1.71 - 1.85 (m, 4H), 2.02 - 2.09 (m, 2H), 2.14 (dd, $J = 12.7, 7.7$ Hz, 1H), 3.10 - 3.21 (m, 3H), 3.38 (s, 2H), 4.36 (d, $J = 5.8$ Hz, 2H), 5.03 (s, 1H), 7.10 - 7.68 (m, 15H); ^{13}C NMR (125.7 MHz, CDCl_3) δ 14.1, 22.6, 24.9, 29.2, 29.5, 30.1, 31.8, 34.8, 37.9, 41.0, 45.9, 47.8, 50.9, 73.1, 73.2, 74.2, 74.6, 92.2, 127.3, 127.4, 127.4, 127.5, 128.2, 128.3, 128.4, 128.7, 132.5, 133.6, 138.3, 138.6, 139.6, 169.6, 197.1; IR (neat) ν 3289, 1622 cm^{-1} ; EIMS m/z 563 ($\text{M}^+ - \text{H}_2\text{O}$); HRMS Calcd for $\text{C}_{39}\text{H}_{48}\text{O}_4$: 580.807720, Found: 580.807821.

1-((2*R,3*aS**)-2-hydroxy-2-octyl-5,5-bis{[(phenylmethyl)oxy]methyl}-2,3,3*a*,4,5,6-hexahydro-1-pentalenyl)-1-ethanone (entry 4, Table 2)**

¹H NMR (400 MHz, CDCl₃) δ 0.86 (t, *J* = 7.1 Hz, 3H), 1.13 - 1.45 (m, 9H), 1.51 - 1.64 (m, 4H), 1.69 - 1.78 (m, 1H), 2.03 (dd, *J* = 12.6, 8.4 Hz, 1H), 2.25 (s, 3H), 2.29 (dd, *J* = 12.6, 8.4 Hz, 1H), 2.47 (d, *J* = 19.0 Hz, 1H), 2.60 (d, *J* = 19.0 Hz, 1H), 3.01 (quint, *J* = 9.0 Hz, 1H), 3.36 (d, *J* = 8.7 Hz, 1H), 3.43 (d, *J* = 8.7 Hz, 1H), 3.48 (s, 4H), 4.13 (s, 1H), 4.48 - 4.68 (m, 4H), 7.26 - 7.40 (m, 10H); ¹³C NMR (100.6 MHz, CDCl₃) δ 14.1, 22.7, 24.6, 29.3, 29.6, 30.1, 30.3, 31.9, 34.9, 37.3, 39.5, 44.0, 48.0, 51.8, 73.3, 73.4, 73.6, 74.7, 90.6, 127.4, 127.5, 127.6, 127.7, 128.4, 138.5, 169.3, 198.4; IR (neat) ν 3289, 1622 cm⁻¹; EIMS *m/z* 518 (M⁺); HRMS Calcd for C₃₄H₄₆O₄: 518.73684, Found: 518.73429.

Intramolecular Aldol reaction

1-[3-hydroxy-3-methyl-2-(phenylcarbonyl)cyclopentyl]-1-nonanone (7*a* and *b*) (Scheme 5)

A reaction of 1-phenyl-2-hepten-1,6-dione (101.1 mg, 0.5 mmol) and **1a** (1.0 mmol) was carried out as described in typical reaction procedure to give a mixture of **7a,b** (112 mg, 65%). The mixture was separated by silica gel column chromatography (hexanes/ethyl acetate = 10 : 1) to give 1*S**-isomer as a first effluent and 1*R**-isomer as a second effluent.

1-[(1*S,2*R**,3*S**)-3-hydroxy-3-methyl-2-(phenylcarbonyl)cyclopentyl]-1-nonanone**

¹H NMR (400 MHz, CDCl₃) δ 0.86 (t, *J* = 7.2 Hz, 3H), 1.10 - 1.32 (m, 10H), 1.29 (s, 3H), 1.39 - 1.50 (m, 2H), 1.68 - 1.82 (m, 2H), 1.90 - 1.99 (m, 1H), 2.24 (dt, *J* = 16.9, 7.6 Hz, 1H), 2.34 - 2.47 (m, 2H), 3.59 - 3.67 (m, 1H), 3.80 (s, 1H), 4.16 (d, *J* = 9.7 Hz, 1H), 7.25 - 7.59 (m, 3H), 7.98 - 8.01 (m, 2H); ¹³C NMR (100.6 MHz, CDCl₃) δ 14.1, 22.6, 23.5, 26.6, 27.2, 29.0, 29.1, 29.3, 31.8, 40.7, 42.5, 128.7, 128.8, 133.8, 137.7, 205.1, 211.9; IR (neat) ν 3465, 1706, 1671 cm⁻¹; EIMS *m/z* 345 (M⁺ + 1); HRMS Calcd for C₂₂H₃₂O₃: 344.23514, Found: 344.23217.

1-[(1*R,2*R**,3*S**)-3-hydroxy-3-methyl-2-(phenylcarbonyl)cyclopentyl]-1-nonanone**

¹H NMR (400 MHz, CDCl₃) δ 0.86 (t, *J* = 7.2 Hz, 3H), 1.13 (s, 3H), 1.17 - 1.30 (m, 10H), 1.50 - 1.59 (m, 2H), 1.86 - 1.95 (m, 3H), 2.12 - 2.20 (m, 1H), 2.41 (dt, *J* = 17.0, 7.2 Hz, 1H), 2.47 (s, 1H), 2.52 (dt, *J* = 17.0, 7.2 Hz, 1H), 3.62 - 3.69 (m, 1H), 4.25 (d, *J* = 6.7 Hz, 1H), 7.45 - 7.60 (m, 3H), 8.05 - 8.08 (m, 2H); ¹³C NMR (100.6 MHz, CDCl₃) δ 14.1, 22.6, 23.6, 25.2, 26.9, 29.1, 29.2, 29.3, 31.8, 42.0, 42.1, 128.7, 129.0, 133.4, 137.7, 200.9, 213.0; IR (neat) ν 3483, 1705, 1671 cm⁻¹; EIMS *m/z* 344 (M⁺); HRMS Calcd for C₂₂H₃₂O₃: 344.23514, Found: 344.23560.