Supporting Information

Secondary Benzylation Using Benzyl Alcohols Catalyzed by Lanthanoid, Scandium, and Hafnium Triflate

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Table of Contents

General Procedure	S1	Product for Scheme 3.	S21
Selected Reaction Procedure for Table 1.	S2	Reaction Procedure for Table 7.	S22
Product for Table 1.	S2	Products for Table 7.	S22
Syntheses of Substrates for Table 2.	S3	Syntheses of substrate for Scheme 4.	S24
Selected Reaction Procedure for Table 2.	S6	Reaction Procedure for Scheme 4.	S25
Products for Table 2.	S6	Products for Scheme 4.	S25
Syntheses of Substrates for Table 3.	S8	Substrate for Table 8.	S26
Selected Reaction Procedure for Table 3.	S10	Reaction Procedure for Table 8.	S27
Products for Table 3.	S10	Products for Table 8.	S27
Selected Reaction Procedure for Table 4.	S11	Selected Reaction Procedure for Table 9.	S29
Product for Table 4.	S11	Products for Table 9.	S29
Synthesis of Substrate for Table 5.	S12	Reaction Procedure for Table 10.	S31
Reaction Procedure for Table 5.	S12	Product for Table 10.	S31
Products for Table 5.	S12	By-product of the reaction using 1.	S32
Synthesis of Substrates for Table 6.	S17	GC-Analysis.	S33
Reaction Procedure for Table 6.	S19	References.	S34
Products for Table 6.	S19	¹ H-, ¹³ C-MMR of 5g , 38 , 39 , 40	S36
Reaction Procedure for Scheme 3.	S21		

General Information

Warning: Nitromethane is hazardous because of the possibility of explosion. Melting points are uncorrected. Preparative TLC was performed on Wakogel B5-F. GC analysis was performed with Rascot OV-1 (0.25 mm I.D. × 30 m, df = 0.4 μm) column with flame ionization detection.

Selected Reaction Procedure for Table 1.

Entry 2. Under argon atmosphere, to a solution of **1** (46.29 mg, 272.4 μmol) and **2a** (55.09 mg, 272.4 μmol) in anhydrous CH₂Cl₂ (1.09 mL) was added BF₃·Et₂O (34.5 μL, 272.4 μmol) over 3 min at 0 °C. The resulting yellow solution was stirred for 1 h. Cold water was added to the reaction mixture,

and allowed to warm to room temperature. The mixture was poured into a separatory funnel and washed twice with H₂O and once with brine. The organic layer was dried over MgSO₄, filtered, and the filtrate was concentrated to give an yellow oil. The oil was purified by preparative TLC (silica gel, cyclohexane/CH₂Cl₂ = 1/1) to afford an yellow solid **3** (103.4 mg, 96%). The solid was recrystallized from EtOH for analyses to give an yellow prisms.

Entry 6. To a mixture of La(OTf)₃ (1.22 mg, 2.08 μ mol) and **2** (4.209 g, 20.82 mmol) in CH₃NO₂ (20.82 mL), **1** (3.585 g, 20.82 mmol) was added at room temperature. And the whole was heated at refluxing condition for 24 h. After the solvent was evaporated under reduced pressure, the residue was dissolved in cyclohexane, and washed with H₂O. The cyclohexane layer was concentrated and the residue was purified by column chromatography (silica gel, cyclohexane/CH₂Cl₂ = 2/1 to 1/1) to afford **3** (6.901 g, 93%).

Entry 8. To a mixture of Yb(OTf)₃ (2.02 mg, 3.26 μ mol) and **2a** (65.8 mg, 326 μ mol) in CH₃NO₂ (1.30 mL), **1** (56.1 mg, 326 μ mol) was added at room temperature. The whole mixture was heated at 100 °C for 20 min. Solvent was evaporated under reduced pressure, and the residue was purified by preparative TLC (silica gel, cyclohexane/CH₂Cl₂ = 1/1) to afford **3** (116.0 mg, 96%).

Product for Table 1.

Methyl 3-hydroxy-4-(1-(2-naphthyl))ethyl-2-naphthoate (3). Yellow needle (EtOH): mp 116–117 °C; TLC R_f = 0.62 (cyclohexane/CH₂Cl₂ = 1/1), 0.54 (AcOEt/hexane = 5/1); ¹H NMR (400 MHz, CDCl₃, δ): 10.93 (s, OH, 1H), 8.46 (s, naphthalene H-1, 1H), 7.86–7.13 (m, Ar H, 5H), 7.64 (d, J = 8.6 Hz, Ar H, 1H), 7.45–7.36 (m, Ar H, 2H), 7.31–7.19 (m, Ar H, 3H), 5.49 (q, J = 7.2 Hz, -CHCH₃, 1H), 4.01 (s, CO₂CH₃, 3H), 1.96 (d, J = 7.2 Hz, -CHCH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): 170.52 (C=O), 153.79 (naphthalene C-3), (142.98, 135.85, 133.39, 131.64, 127.44, 125.58 (naphthalene C-2, 4a, 8a and side chain naphthalene C-2, 4a, 8a)), 131.58 (naphthalene C-1), (130.22, 128.52, 127.61, 127.43, 127.35, 126.53, 125.58, 124.92, 124.34, 124.05, 123.09 (naphthalene C-5, 6, 7, 8 and side chain naphthalene C-1, 3, 4, 5, 6, 7, 8), 113.70 (naphthalene C-4), 52.69 (CO₂CH₃), 34.50 (-CHCH₃), 17.79 (-CHCH₃); IR (KBr) $\tilde{\nu}_{\max}$: (cm⁻¹): 3150 (w), 1670 (m), 1440 (m), 1200 (m), 800 (m), 750 (m); EIMS (70 eV) m/z (relative intensity): 356 (M⁺, 100), 341 (M – CH₃, 18), 324 (M – CH₃OH, 41), 309 (33), 296 (29), 281 (45), 252 (22); Anal. Calcd for C₂₄H₂₀O₃: C, 80.88; H, 5.66. Found: C, 81.08; H, 5.71. The ee value of **3** was determined by HPLC analysis using a chiral column (DAICEL CHIRALCEL OD, hexane/2-propanol = 30/1, 0.3 mL/min, 26.7 min, 29.1 min).

Syntheses of Substrates for Table 2.

4-*tert*-Butylldiemthylsilyloxyacetophenone.¹ Under argon atmosphere, to a solution of 4-hydroxyacetophenone (2.993 g, 21.99 mmol) and imidazole (4.501 g, 66.11 mmol) in anhydrous dimethylformamide (22.03 mL) was added chloro *tert*-butylldiemthylsilane (4.983 g, 33.06 mmol). The resulting solution was stirred for 24 h at room temperature. The reaction mixture was diluted with hexane/Et₂O = 1/1 (200 mL), and organic layer was washed three times with H₂O (100 mL) and once with brine (100 mL), dried over MgSO₄, filtered, and the filtrate was concentrated to give a color less liquid (5.93g, quant.), which crystallized on standing and was used for the next reaction without further purification. Colorless solid: TLC R_f = 0.43 (ethyl acetate/hexane = 4/1); ¹H NMR (300 MHz, CDCl₃, δ): 7.86 (d, *J* = 6.9 Hz, Ar H-2, 6, 2H), 6.84 (d, *J* = 6.9 Hz, Ar H-3, 5, 2H), 2.53 (s, -COCH₃, 3H), 0.97 (s, -SiC(CH₃)₃, 9H), 0.22, (s, -Si(CH₃)₂, 6H); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 2950 (m), 1680 (m), 1600 (m), 1510 (m), 1360 (m), 1270 (s), 1170 (m), 910 (m), 840 (m), 790 (m); EIMS (70 eV) *m/z* (relative intensity): 250 (M⁺, 27), 193 (100), 151 (17).

1-(4-*tert*-Butylldiemthylsilyloxy)phenylethanol (4b). To a solution of 4-*tert*-butylldiemthylsilyloxyacetophenone (3.006 g, 12.00 mmol) in EtOH (12.00 mL) at 0 °C was added NaBH₄ (227.1 mg, 6.00 mmol). The mixture was allowed to warm to room temperature and stirred for 30 min. The whole was concentrated, and the residue was partitioned with H₂O and Et₂O, extracted three times with Et₂O (50 mL). The organic layer was washed once with brine (100 mL), dried over MgSO₄, filtered, and the filtrate was concentrated. The residue was purified by bulb-to-bulb distillation (150 °C/0.7 Torr) to give a colorless oil (2.57g, 85%): TLC R_f = 0.15 (AcOEt/hexane = 4/1); ¹H NMR (300 MHz, CDCl₃, δ): 7.22 (d, *J* = 8.2 Hz, Ar H-2, 6, 2H), 6.80 (d, *J* = 8.2 Hz, Ar H-3, 5, 2H), 4.82 (qd, *J* = 6.3, 2.1 Hz, -CHCH₃ 1H), 1.76 (br, OH, 1H), 1.47 (d, *J* = 6.3, -CHCH₃, 3H), 0.98 (s, -SiC(CH₃)₃, 9H), 0.21 (0.22, (s, -Si(CH₃)₂, 6H); ¹³C NMR (100 MHz, CDCl₃, δ): 158.82 (ph C-4), 138.36 (ph C-1), 126.43 (ph C-2), 119.86 (ph C-3), 70.07 (C-1), 25.81 (-SiC(CH₃)₃), 25.11 (C-2), -4.21 (-Si(CH₃)₂); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 3320 (br), 2960 (m), 2930 (m), 2860 (m), 1610 (m), 1520 (s), 1270 (s), 1180 (m), 1100 (m), 1020 (m), 920 (s) 850 (s), 790 (s); EIMS (70 eV) *m/z* (relative intensity): 252 (M⁺, 2), 234 (M - H₂O, 40), 177 (M - H₂O, *tert*-butyl, 100), 151 (10); HRMS-EI (*m/z*): M⁺ calcd for C₁₄H₂₄OSi, 252.2546; found, 252.1550; Anal. Calcd for C₁₄H₂₄OSi: C, 66.61; H, 9.58. Found: C, 66.34; H, 9.71.

4-Acetylphenyl acetate.² To a solution of 4-hydroxyacetophenone (5.007 g, 36.77 mmol), NEt_3 (7.647 mL, 55.16 mmol), 4-dimethylaminopyridine (90.9 mg, 774.0 μmol) in CH_2Cl_2 (200 mL) was added Ac_2O (5.214 mL, 55.16 mmol) at 0 °C, and the whole was stirred for 1.5 h at room temperature. The reaction mixture was poured into water (200 mL), extracted twice with CH_2Cl_2 (100 mL). The organic layer was washed once with saturated aq NH_4 (100 mL), dried over MgSO_4 , filtered, and the filtrate was concentrated to give a colorless liquid (5.850 g, 98%). An analytical sample was obtained by bulb-to-bulb distillation. Colorless liquid; TLC R_f = 0.56 (AcOEt /hexane = 1/1); ^1H NMR (300 MHz, CDCl_3 , δ): 7.99 (d, J = 8.7 Hz, phenyl H-3, 5, 2H), 7.19 (d, J = 8.7 Hz, phenyl H-2, 6, 2H), 2.60 (s, CH_3COPh , 3H), 2.33 (s, CH_3COO , 3H).

4-(1-Hydroxyethyl)phenyl acetate (4c).³ To a solution of 4-acetylphenyl acetate (8.750 g, 53.96 mmol) in 2-propanol (108.0 mL) was added NaBH_4 (2.045 g, 54.04 mmol) at 0 °C. The mixture was stirred for 2.5 h. Excess NaBH_4 was decomposed by addition of acetone (3 mL). The whole was concentrated, and the residue was partitioned with H_2O and AcOEt , extracted three times with AcOEt (100 mL). The organic layer was washed once with brine (100 mL), dried over MgSO_4 , filtered, and the filtrate was concentrated. The residue was purified by column chromatography (silica gel, hexane/ AcOEt = 10/1 to 2/1) to give a colorless oil (8.011 g, 91%): TLC R_f = 0.46 (AcOEt /hexane = 1/1); ^1H NMR (300 MHz, CDCl_3 , δ): 7.36 (d, J = 8.1 Hz, phenyl H-2, 6, 2H), 7.04 (d, J = 8.4 Hz, phenyl H-3, 5, 2H), 4.87 (q, J = 6.3 Hz, $-\text{CHCH}_3$ 1H), 2.29 (s, CH_3CO , 3H), 1.47 (d, J = 6.3 Hz, $-\text{CHCH}_3$ 3H); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3430 (br), 2960 (m), 1750 (s), 1600 (m), 1600 (m), 1370 (m), 1220 (s), 1170 (m), 1090 (m), 1020 (m), 910 (m), 850 (m); EIMS (70 eV) m/z (relative intensity): 180 (M^+ , 15), 138 (47), 123 (100), 95 (22).

4-(Benzyloxy)acetophenone.⁴ Benzyl bromide (4.805 g, 40.40 mmol) was added to a mixture of 4-hydroxyacetophenone (4.999 g, 36.72 mmol), K_2CO_3 (7.619 g, 55.13 mmol) and KI (1.217 g, 7.329 mmol) in acetone (36.72 mL) at room temperature. The whole was stirred for 17 hr and concentrated. The residue was partitioned with H_2O (100 mL) and AcOEt , extracted twice with AcOEt (100 mL). The combined organic layer was washed once with brine (100 mL), dried over MgSO_4 , filtered, and the filtrate was concentrated to give a yellow solid (8.87g, quant.), which was used for the next reaction

without further purification. An analytical sample was obtained by preparative TLC (silica gel, hexane/AcOEt = 1/1). TLC R_f = 0.33 (AcOEt/hexane = 4/1); ^1H NMR (300 MHz, CDCl_3 , δ): 7.93 (d, J = 9.0 Hz, acetophenone H-2, 6, 2H), 7.45–7.31 (m, C_6H_5 , 5H), 7.01 (d, J = 9.0 Hz, acetophenone H-3, 5, 2H), 5.13 (s, CH_2 2H), 2.55 (s, CH_3 , 3H); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 1675 (C=O, s), 1600 (s), 1360 (m), 1305 (m), 1260 (s), 1180 (s), 1020 (s), 835 (s), 765 (s), 720 (s); EIMS (70 eV) m/z (relative intensity): 226 (M^+ , 29), 91 (100).

1-(4-Benzyloxyphenyl)ethanol (4d).⁵ To a solution of 4-(Benzyloxy)acetophenone (6.540 g, 28.91 mmol) in EtOH (21 mL) and tetrahydrofuran (20 mL) was added NaBH_4 (1.093 g, 28.90 mmol) at room temperature. The mixture was stirred for 20 h. Excess NaBH_4 was decomposed by addition of acetone (3 mL). The whole was concentrated, and the residue was partitioned with H_2O and Et_2O , extracted twice with Et_2O (100 mL). The combined organic layer was washed once with brine (100 mL), dried over MgSO_4 , filtered, and the filtrate was concentrated to give a colorless solid (5.975 g, 91%). The solid was recrystallized from hexane to afford colorless prism (5.802 g, 88%): mp 77–78 °C (lit⁶ 79–80); TLC R_f = 0.52 (AcOEt/hexane = 1/1); ^1H NMR (300 MHz, CDCl_3 , δ): 7.45–7.31 (m, C_6H_5 , phenyl H-2, 6 7H), 6.95 (d, J = 8.7 Hz, phenyl H-3,5, 2H), 5.06 (s, CH_2 , 2H), 4.85 (qd, J = 6.3, 3.0 Hz, $-\text{CHCH}_3$ 1H), 1.76 (br, OH, 1H), 1.48 (d, J = 6.3 Hz, $-\text{CHCH}_3$ 3H); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3310 (OH, br), 1620 (m), 1510 (s), 1385 (m), 1250 (ms), 1190 (m), 1020 (s), 830 (s), 740 (m), 700 (m); EIMS (70 eV) m/z (relative intensity): 228 (M^+ , 34), 91 (100).

1-(4-Hydroxyphenyl)ethanol (4e).⁷ To a solution of 4-(1-hydroxyethyl)phenyl acetate (6.564 g, 40.23 mmol) in EtOH (40.23 mL) was added 4 mol/L aq KOH (11.06 mL, 44.25 mmol) at room temperature. The mixture was stirred for 22 h. Excess NaBH_4 was decomposed by addition of acetone (3 mL). The whole was concentrated, and the residue was partitioned with saturated aq NH_4Cl and AcOEt, and extracted twice with AcOEt (100 mL). The combined organic layer was washed once with brine (100 mL), dried over MgSO_4 , filtered, and the filtrate was concentrated. The residue was recrystallized from toluene to give colorless fine needles (3.566 g, 71%): mp 134–138 °C; TLC R_f = 0.27 (AcOEt/hexane = 1/1); ^1H NMR (300 MHz, CDCl_3 , δ): 7.27 (d, J = 8.3 Hz, phenyl H-2, 6, 2H), 6.82 (d, J = 8.8 Hz, phenyl H-3, 5, 2H), 4.86 (q, J = 6.4 Hz, $-\text{CHCH}_3$ 1H), 4.82 (s, OH, 1H), 1.72 (s, OH, 1H), 1.53 (d, J = 6.4 Hz, $-\text{CHCH}_3$ 3H); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3400 (OH, s), 3100 (br), 1600 (m), 1510 (m), 1460 (m), 1240 (s), 1080 (s), 1010 (m), 900 (m), 840 (s); EIMS (70 eV) m/z (relative intensity):

138 (M^+ , 37), 123 (106), 95 (42).

Selected Reaction Procedure for Table 2.

Entry 4. To a mixture of $\text{La}(\text{OTf})_3$ (1.20 mg, 2.05 μmol) and **2a** (41.4 mg, 205 μmol) in CH_3NO_2 (819 μL), was added **4d** (46.7 mg, 205 μmol) at room temperature. The whole mixture was stirred at 50 °C for 14 h. The solvent was evaporated under reduced pressure, and the residue was purified by preparative TLC (silica gel, $\text{CH}_2\text{Cl}_2/\text{Et}_2\text{O}/\text{toluene} = 15/1/51$) to afford **5d** as a yellow solid (81.6 mg, 97%).

Products for Table 2.

Methyl 3-hydroxy-4-(1-phenylethyl)-2-naphthoate (5a). Yellow prism (EtOH): mp 113–114 °C; TLC $R_f = 0.51$ (cyclohexane/ $\text{CH}_2\text{Cl}_2 = 1/1$); ^1H NMR (300 MHz, CDCl_3 , δ): 10.87 (s, OH, 1H), 8.44 (s, naphthalene H-1, 1H), 7.38–7.73 (m, Ar H, 7H), 5.33 (q, $J = 7.2$ Hz, $-\text{CHCH}_3$ 1H), 4.01 (s, CO_2CH_3 , 3H), 1.85 (d, $J = 7.2$ Hz, $-\text{CHCH}_3$ 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 170.47, (C=O), 153.73 (naphthalene C-3), 145.20 (phenyl C-4), 135.77 (naphthalene C-4a), 131.43 (naphthalene C-1), 130.19 (phenyl C-8), 128.42 (naphthalene C-6), 127.91 (phenyl C-3, 5), 127.40 (naphthalene C-8a), 126.90 (phenyl C-2, 6), 125.82 (naphthalene C-2), 125.20 (phenyl C-4), 124.04 (naphthalene C-5), 123.03 (naphthalene C-7), 113.68 (naphthalene C-4), 52.63 (CO_2CH_3), 34.30 ($-\text{CHCH}_3$), 17.73 ($-\text{CHCH}_3$); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3180 (br), 1690 (C=O, s), 1625 (m), 1440 (s), 1306 (s), 1205 (s), 1070 (m), 805 (m), 750 (m); EIMS (70 eV) m/z (relative intensity): 306 (M^+ , 100), 291 ($M - \text{CH}_3$, 19), 274 ($M - \text{CH}_3\text{OH}$, 76), 259 (35), 246 (48), 231 (39), 202 (39); Anal. Calcd for $\text{C}_{20}\text{H}_{18}\text{O}_3$: C, 78.41; H, 5.92. Found: C, 78.16; H, 5.99.

Methyl 4-(1-(4-tert-butyl dimethylsilyloxy)phenyl)ethyl-3-hydroxy-2-naphthoate (5b). Yellow viscous oil: TLC $R_f = 0.85$ ($\text{AcOEt}/\text{CH}_2\text{Cl}_2/\text{hexane} = 1/1/2$); ^1H NMR (400 MHz, CDCl_3 , δ): 10.87 (s, OH, 1H), 8.43 (s, naphthalene H-1, 1H), 7.76 (d, $J = 8.8$ Hz, naphthalene H-8, 1H), 7.74 (d, $J = 9.0$ Hz, naphthalene H-5, 1H), 7.30 (t, $J = 7.6$ Hz, naphthalene H-6, 1H), 7.22 (t, $J = 7.4$ Hz, naphthalene H-7, 1H), 7.14 (d, $J = 8.8$ Hz, phenyl H-2, 6, 2H), 6.72 (d, $J = 8.4$ Hz, phenyl H-3, 5, 2H), 5.29 (q, $J = 6.8$ Hz, $-\text{CHCH}_3$, 1H), 4.00 (s, CO_2CH_3 , 3H), 1.81 (d, $J = 7.2$ Hz, $-\text{CHCH}_3$, 3H), 0.95 (d, $J = 0.8$ Hz, $-\text{Si}(\text{CH}_3)_3$, 9H), 0.17 (d, $J = 2.4$ Hz, $-\text{Si}(\text{CH}_3)_2$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 170.52,

(C=O) 153.62 (naphthalene C-3), 153.03 (phenyl C-4), 137.94 (phenyl C-1), 135.76 (naphthalene C-4a), 131.34 (naphthalene C-1), 130.14, (naphthalene C-1), 128.21 (naphthalene C-6), 127.75 (phenyl C-2, 6), 127.42 (naphthalene C-8a), 126.10 (naphthalene C-2), 124.29 (naphthalene C-5), 122.99 (naphthalene C-7), 119.48 (phenyl C-3, 5), 113.60 (naphthalene C-4), 52.64 (CO₂CH₃), 33.50 (-CHCH₃), 25.82 (-CHCH₃), 18.32, 18.04 (-SiC(CH₃)₃), -4.22 (-Si(CH₃)₂); IR (neat) $\tilde{\nu}_{\max}$: (cm⁻¹): 3190 (br), 2950 (m), 2850 (m), 1680 (C=O, s), 1510 (m), 1440 (m), 1330 (m), 1250 (s), 1210 (m), 920 (m), 840 (m), 800 (m); EIMS (70 eV) *m/z* (relative intensity): 436 (M⁺, 100), 421 (M - CH₃, 40), 379 (M - C₄H₉, 40), 229 (27), 197 (69); HRMS-EI (*m/z*): M⁺ calcd for C₂₆H₃₂O₄Si, 436.2070; found, 436.2069; Anal. Calcd for C₂₆H₃₂O₄Si: C, 71.52; H, 7.39. Found: C, 71.84; H, 7.30.

Methyl 4-(1-(4-acetoxyphenyl)ethyl)-3-hydroxy-2-naphthoate (5c). Yellow prism (EtOH): mp 123–124 °C; TLC *R_f* = 0.30 (hexane/Et₂O = 2/1); ¹H NMR (300 MHz, CDCl₃, δ): 10.86 (s, OH, 1H), 8.44 (s, naphthalene H-1, 1H), 7.81–7.73 (m, naphthalene H-5, 8, 2H), 7.40–7.22 (m, Ar H, 4H), 6.96 (d, *J* = 9.0 Hz, phenyl H-3, 5, 2H), 5.30 (q, *J* = 7.2 Hz, -CHCH₃, 1H), 4.01 (s, CO₂CH₃, 3H), 2.24 (s, COCH₃, 3H), 1.84 (d, *J* = 7.2 Hz, -CHCH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): 170.41 (C=O, acetyl), 169.25 (C=O, naphthalene), 153.70 (naphthalene C-1), 148.18 (naphthalene C-4), 142.68 (phenyl C-1), 135.63 (naphthalene C-4a), 131.51 (naphthalene C-1), 130.22 (naphthalene C-8), 128.55 (naphthalene C-6), 127.83 (phenyl C-3, 5), 127.34 (naphthalene C), 125.36 (naphthalene C), 123.86 (naphthalene C-5), 123.08 (naphthalene C-7), 120.80 (phenyl C-2, 6), 113.66 (naphthalene C-4), 52.65 (CO₂CH₃), 33.94 (-CHCH₃), 21.21 (-CHCH₃), 17.82 (CH₃C=O); IR (KBr) $\tilde{\nu}_{\max}$: (cm⁻¹): 3160 (m), 2960 (m), 1760 (acetyl, C=O, s), 1680 (ester, C=O, s), 1630 (m), 1510 (m), 1440 (s), 1370 (m), 1330 (m), 1310 (m), 1210 (s), 1200 (s), 1105 (m), 920 (m), 800 (m); EIMS (70 eV) *m/z* (relative intensity): 364 (100), 322 (57), 307 (48), 290 (35), 275 (43), 262 (80), 180 (20); HRMS-EI (*m/z*): M⁺ calcd for C₂₂H₂₀O₅, 364.1311; found, 364.1316; Anal. Calcd for C₂₂H₂₀O₅: C, 72.51; H, 5.53. Found: C, 72.50; H, 5.64.

Methyl 4-(1-(4-benzyloxyphenyl)ethyl)-3-hydroxy-2-naphthoate (5d). Yellow prism (EtOH): mp 96–100 °C; TLC *R_f* = 0.74 (CH₂Cl₂/Et₂O/toluene = 15/1/51); ¹H NMR (400 MHz, CDCl₃, δ): 10.85 (s, OH, 1H), 8.44 (s, naphthalene H-1, 1H), 7.82–7.75 (m, Ar H, 1H), 7.44–7.13 (m, Ar H, 10H), 6.86 (d, *J* = 8.8 Hz, benzyloxyphenyl H-3, 5, 2H), 5.27 (q, *J* = 7.2 Hz, -CHCH₃, 1H), 5.00 (s, CH₂, 2H), 4.02 (s, CO₂CH₃, 3H), 1.83 (d, *J* = 7.2 Hz, -CHCH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): 170.57 (C=O), 156.51 (phenyl C-1), 153.76 (naphthalene C-3), 137.66 (Bn, Ar C-1), 137.15 (phenyl C-1), 135.82

(naphthalene C-4a), 131.41 (naphthalene C-1), 130.24 (naphthalene C-8), 128.44 (naphthalene C-6), 128.37 (phenyl C-2, 6), 127.88 (Bn, Ar C-3, 5), 127.69 (Bn, Ar C-4), 127.42 (naphthalene C-8a), 127.35 (Bn, Ar C-2, 6), 125.97 (naphthalene C-2), 124.09 (naphthalene C-5), 123.05 (naphthalene C-7), 114.37 (phenyl C-3, 5), 113.67 (naphthalene C-4), 69.94 (CH₂), 52.55 (CO₂CH₃), 33.52 (-CHCH₃), 17.88 (-CHCH₃); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 1680 (C=O, s), 1515 (m), 1460 (m), 1330 (m), 1310 (m), 1240 (m), 1220 (m), 810 (m), 750 (m); EIMS (70 eV) m/z (relative intensity): 412 (M⁺, 100), 397 (M - CH₃, 33), 352 (24), 289 (32), 91 (84); HRMS-EI (m/z): M⁺ calcd for C₂₇H₂₄O₄, 412.1675; found, 412.1677; Anal. Calcd for C₂₇H₂₄O₄: C, 78.62; H, 5.86. Found: C, 78.39; H, 5.91.

Methyl 3-hydroxy-4-(1-(4-hydroxyphenyl)ethyl)-2-naphthoate (5e). Yellow prism (cyclohexane): mp 142–144 °C; TLC R_f = 0.66 (hexane/AcOEt = 1/1); ¹H NMR (400 MHz, CDCl₃, δ): 10.86 (s, naphthol, OH, 1H), 8.43 (s, naphthalene H-1, 1H), 7.79 (d, J = 8.4 Hz, naphthalene H-5, 8, 2H), 7.36 (t, J = 7.4 Hz, naphthalene H-6, 1H), 7.26 (t, J = 7.4 Hz, naphthalene H-7, 1H), 7.16 (d, J = 8.0 Hz, phenyl H-2, 6, 2H), 6.72 (d, J = 8.8 Hz, phenyl H-3, 5, 2H), 5.26 (q, J = 7.2 Hz, -CHCH₃, 1H), 4.61 (s, phenol, OH, 1H), 4.03 (s, CO₂CH₃, 3H), 1.82 (d, J = 7.2 Hz, -CHCH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): 170.52 (C=O), 153.54 (phenyl C-4), 152.95 (naphthalene C-3), 137.38 (phenyl C-1), 135.72 (naphthalene C-4a), 131.39 (naphthalene C-1), 130.17 (naphthalene C-6), 128.34 (naphthalene C-8), 127.97 (phenyl C-2, 6), 127.37 (naphthalene C-8a), 125.93 (naphthalene C-2), 124.05 (naphthalene C-5), 123.02 (naphthalene C-7), 114.84 (phenyl C-3, 5), 113.59 (naphthalene C-4), 52.66 (CO₂CH₃), 33.52 (-CHCH₃), 17.95 (-CHCH₃); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 3450 (s), 3200 (m), 2950 (m), 1670 (C=O, s), 1510 (s), 1440 (s), 1330 (s), 1220 (s), 840 (m), 800 (m), 750 (m); EIMS (70 eV) m/z (relative intensity): 322 (M⁺, 100), 307 (M - CH₃, 43), 290 (M - CH₃OH, 31), 275 (50), 262 (68); Anal. Calcd for C₂₀H₁₈O₄: C, 74.52; H, 5.63. Found: C, 74.35; H, 5.65.

Syntheses of Substrates for Table 3.

Benzyl 3-hydroxy-2-naphthoate (2b).⁸ Thionyl chloride (17.0 mL, 233.3 mmol) was added dropwise to a mixture of 3-hydroxy-2-naphthoic acid (14.63 g, 77.80 mmol) and DMF (0.30 mL) in benzene (150 mL) under anhydrous condition (CaCl₂ tube), and the mixture was stirred at 40 °C for 3 h. The mixture became clear, and was concentrated under reduced pressure to give an acid chloride as an orange solid, and the solid was dissolved in THF (150 mL). Benzyl alcohol (25.23 g, 233.3 mmol) was added dropwise to a suspension of NaH (9.33 g, 60% in mineral oil, 233.3 mmol) in THF at room

temperature. After being stirred for 15 min, the mixture was cooled to $-78\text{ }^{\circ}\text{C}$. To this mixture, the THF solution of the acid chloride was added dropwise over 4 h, and the mixture was allowed to room temperature, stirred for 24 h. The mixture was poured into a H_2O (400 mL) solution of KH_2PO_4 (21.18 g, 155.6 mmol), and the whole was extracted twice with Et_2O (200 mL). The combined organic layer was successively washed with saturated aq NaHCO_3 (200 mL $\times$ 3) and brine (200 mL), and the extract was dried over MgSO_4 , filtered, and the filtrate was evaporated to afford an yellow oil. The oil was purified by column chromatography (hexane/ Et_2O = 200/1) to afford **2b** as yellow solid (12.37 g, 57%), which was recrystallized from hexane to give an yellow prism: mp $84\text{--}85\text{ }^{\circ}\text{C}$; TLC R_f = 0.38 (hexane/ Et_2O = 5/1); ^1H NMR (270 MHz, CDCl_3 , δ): 10.43 (s, OH, 1H), 8.50 (s, naphthalene H-1, 1H), 7.77 (d, J = 8.3 Hz, naphthalene H-8, 1H), 7.66 (d, J = 8.6 Hz, naphthalene H-5, 1H), 7.51–7.36 (m, naphthalene H-6, phenyl, 6H), 7.30 (s, naphthalene H-4, 1H), 7.32–7.27 (m, naphthalene H-7, 1H), 5.44 (s, $-\text{CH}_2-$, 2H); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3420 (br), 3350 (m), 1670 (C=O, s), 1510 (m), 1310 (m), 1280 (m), 1200 (m), 1140 (m), 1070 (m), 780 (m), 740 (m); EIMS (70 eV) m/z : 278 (M^+), 133, 91.

tert-Butyl 3-hydroxy-2-naphthoate (2c).⁸ Thionyl chloride (17.0 mL, 233.3 mmol) was added dropwise to a mixture of 3-hydroxy-2-naphthoic acid (14.63 g, 77.80 mmol) and DMF (0.30 mL) in benzene (150 mL) under anhydrous condition (CaCl_2 tube), and the mixture was stirred at $40\text{ }^{\circ}\text{C}$ for 3 h. The mixture became clear, and was concentrated under reduced pressure to give an acid chloride as an orange solid, and the solid was dissolved in THF (150 mL). This solution was added dropwise to a mixture of KOt-Bu (26.18 g, 233.3 mmol) and 18-crown-6 (100 mg) in anhydrous Et_2O (200 mL) at $-78\text{ }^{\circ}\text{C}$ over 5h. The resulting mixture was allowed to warm to room temperature with stirring for 48 h. The mixture was poured into a H_2O (500 mL) solution of KH_2PO_4 (21.18 g, 155.6 mmol), and the whole was extracted twice with Et_2O (200 mL). The combined organic layer was successively washed with saturated aq NaHCO_3 (200 mL $\times$ 5) and brine (200 mL), and the extract was dried over MgSO_4 , filtered, and the filtrate was evaporated to afford an yellow oil. The oil was purified column chromatography (hexane/ Et_2O = 300/1) to afford **2c** as an yellow oil (14.42 g, 76%), which crystallized on standing. The solid was recrystallized from pentane to give pale yellow prism: mp $67.5\text{--}68.5\text{ }^{\circ}\text{C}$; TLC R_f = 0.84 (hexane/ Et_2O = 20/1); ^1H NMR (270 MHz, CDCl_3 , δ): 10.71 (s, OH, 1H), 8.39 (s, naphthalene H-1, 1H), 7.79 (d, J = 8.3 Hz, naphthalene H-8, 1H), 7.67 (d, J = 8.3 Hz, naphthalene H-5, 1H), 7.47 (td, J = 6.9, 1.3 Hz, naphthalene H-6, 1H), 7.32–7.29 (m, naphthalene H-7, 1H), 7.28 (s, naphthalene H-4, 1H), 1.67 (s, $-\text{C}(\text{CH}_3)_3$, 9H); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3210 (m), 2975 (m), 1670 (s), 1630 (m), 1510 (m), 1460 (m), 1370 (m), 1330 (m), 1290 (m), 1230 (m), 1160 (s), 1140 (m), 1070 (s), 870 (m), 790 (s), 740(m); EIMS (70 eV) m/z : 244 (M^+), 188, 160, 142, 114.

Selected Reaction Procedure for Table 3.

Entry 5. To a mixture of $\text{La}(\text{OTf})_3$ (2.05 mg, 3.50 μmol) and **2b** (97.3 mg, 350 μmol) in CH_3NO_2 (1.40 mL), **4a** (42.7 mg, 350 μmol) was added at room temperature. The whole mixture was stirred at 100 °C for 2.5 h. The solvent was evaporated under reduced pressure, and the residue was purified by preparative TLC (silica gel, cyclohexane/ CH_2Cl_2 = 1/1) to afford **5f** as a yellow amorphous solid (131.9 mg, 99%).

Products for Table 3.

Benzyl 3-hydroxy-4-(1-phenyl)ethyl-2-naphthoate (5f). Yellow amorphous solid: mp 113–114 °C; TLC R_f = 0.55 (cyclohexane/ CH_2Cl_2 = 1/1); ^1H NMR (400 MHz, CDCl_3 , δ): 10.88 (s, OH, 1H), 8.46 (s, naphthalene H-1, 1H), 7.77–7.71 (m, naphthalene H-5, 8, 2H), 7.48–7.09 (m, Ar H, 12H), 5.42 (s, CH_2 , 2H), 5.33 (q, J = 7.2 Hz, $-\text{CHCH}_3$, 1H), 1.84 (d, J = 7.6 Hz, $-\text{CHCH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 169.87 (C=O), 153.81 (naphthalene C-3), 145.17 (phenyl C-1), 135.81 (ester, phenyl C-1), 135.07 (naphthalene C-4a), 131.47 (naphthalene C-1), 130.22 (naphthalene C-1), 128.58, 128.58, 128.47, 128.29, 127.91, 126.89 (phenyl C-2, 6 and 3, 5 and 4, ester, phenyl C-2, 6 and 3, 5 and 4), 127.37 (naphthalene C-8a), 125.86 (naphthalene C-2), 125.20 (naphthalene C-6), 124.01 (naphthalene C-5), 123.02 (naphthalene C-7), 113.65 (naphthalene C-4), 67.35 (CH_2), 34.28 ($-\text{CHCH}_3$), 17.71 ($-\text{CHCH}_3$); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 1670 (C=O, s), 1440 (m), 1320 (m), 1300 (m), 1210 (m), 1165 (m), 750 (m), 700 (m); EIMS (70 eV) m/z (relative intensity): 382 (M^+ , 96), 291 ($\text{M} - \text{Bn}$, 10), 274 ($\text{M} - \text{BnO}$, 38), 91 (Bn^+ , 100); HRMS-EI (m/z): M^+ calcd for $\text{C}_{26}\text{H}_{22}\text{O}_3$, 382.1569; found, 328.1574; Anal. Calcd for $\text{C}_{26}\text{H}_{22}\text{O}_3$: C, 81.65; H, 5.80. Found: C, 81.84; H, 5.79.

tert-Butyl 3-hydroxy-4-(1-phenylethyl)-2-naphthoate (5g). Yellow prism (EtOH): mp 93–95 °C; TLC R_f = 0.71 (cyclohexane/ CH_2Cl_2 = 1/1); ^1H NMR (400 MHz, CDCl_3 , δ): 11.19 (s, OH, 1H), 8.35 (s, naphthalene H-1, 1H), 7.78 (d, J = 8.4 Hz, naphthalene H-8, 1H), 7.75 (d, J = 9.9 Hz, naphthalene H-5, 1H), 7.36–7.10 (m, Ar H, 7H), 5.33 (q, J = 7.5 Hz, $-\text{CHCH}_3$, 1H), 1.85 (d, J = 7.5 Hz, $-\text{CHCH}_3$, 3H), 1.07 (s, $-\text{C}(\text{CH}_3)_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 169.71 (C=O), 154.16 (naphthalene C-3), 145.34 (phenyl C-1), 135.49 (naphthalene C-4a), 131.29 (naphthalene C-1), 130.10 (naphthalene C-8), 128.10 (naphthalene C-6), 127.88 (phenyl C-3, 5), 127.31 (naphthalene C-8a), 126.93 (phenyl C-2, 6), 125.60 (naphthalene C-2), 125.13 (phenyl C-4), 123.97 (naphthalene C-7),

122.81 (naphthalene C-5), 83.27 ($-\underline{\text{C}}(\text{CH}_3)_3$), 34.21 ($-\underline{\text{CHCH}}_3$), 28.35 ($-\text{C}(\underline{\text{CH}}_3)_3$), 17.71 ($-\text{CH}\underline{\text{CH}}_3$); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2960 (m), 1670 (C=O, s), 1370 (m), 1330 (s), 1300 (m), 1150 (s), 810 (m), 750 (m), 700 (m); EIMS (70 eV) m/z (relative intensity): 348 (M^+ , 22), 292 ($\text{M} - \text{CH}_2=\text{C}(\text{CH}_3)_2$, 100), 274 ($\text{M} - \text{C}_4\text{H}_9\text{OH}$, 90), 259 (17), 246 ($\text{M} - \text{C}_4\text{H}_9\text{OH}$, $-\text{C}=\text{O}$, 38), 231 (202); HRMS-EI (m/z): M^+ calcd for $\text{C}_{23}\text{H}_{24}\text{O}_3$, 348.1725; found, 348.1724.

3-Hydroxy-4-(1-phenyl)ethyl-2-naphthoic acid (5h). Yellow fine needle (AcOEt): mp 232–234 °C; TLC R_f = 0.43 ($\text{CHCl}_3/\text{MeOH}$ = 10/1); ^1H NMR (300 MHz, CDCl_3 , δ): 10.53 (br, OH, 1H), 8.56 (s, naphthalene H-1, 1H), 7.86–7.77 (m, naphthalene H-5, 8, 2H), 7.42–7.12 (m, Ar H, 7H), 5.34 (q, J = 6.9 Hz, $-\underline{\text{CHCH}}_3$, 1H), 1.87 (d, J = 7.5 Hz, $-\text{CH}\underline{\text{CH}}_3$, 3H); ^{13}C NMR (100 MHz, acetone- d_6 , δ): 172.82 (C=O), 155.13 (naphthalene C-3), 146.18 (phenyl C-1), 136.67 (naphthalene C-4a), 132.85 (naphthalene C-1), 131.26 (naphthalene C-8), 129.31 (naphthalene C-6), 128.75 (phenyl C-3, 5), 128.49 (naphthalene C-8a), 127.74 (phenyl C-2, 6), 126.32 (naphthalene C-2), 126.05 (phenyl C-4), 124.81 (naphthalene C-7), 123.95 (naphthalene C-3), 114.51 (naphthalene C-4), 34.96 ($-\underline{\text{CHCH}}_3$), 18.06 ($-\text{CH}\underline{\text{CH}}_3$); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3250 (w), 1680 (C=O, s), 1460 (m), 1310 (m), 1210 (m), 1180 (w), 1100 (w), 900 (w), 820 (w), 750 (w); EIMS (70 eV) m/z (relative intensity): 292 (M^+ , 100), 274 ($\text{M} - \text{H}_2\text{O}$, 76), 259 (31), 246 (48), 231 (38), 202 (39); HRMS-EI (m/z): M^+ calcd for $\text{C}_{19}\text{H}_{16}\text{O}_3$, 292.1099; found, 292.1098; Anal. Calcd for $\text{C}_{19}\text{H}_{16}\text{O}_3$: C, 78.06; H, 5.52. Found: C, 78.07; H, 5.55.

Selected Reaction Procedure for Table 4.

Entry 7. To a mixture of $\text{Sc}(\text{OTf})_3$ (1.13 mg, 2.30 μmol) and **2a** (46.6 mg, 230 μmol) in CH_3NO_2 (918 μL), **6** (34.0 mg, 230 μmol) was added at 20 °C. The whole mixture was stirred at 20 °C for 24 h. The solvent was evaporated under reduced pressure, and the residue was purified by preparative TLC (silica gel, cyclohexane/ CH_2Cl_2 = 1/2) to afford **7** as an yellow solid (72.5 mg, 95%).

Product for Table 4.

Methyl 3-hydroxy-4-(1,2,3,4-tetrahydro-1-naphthyl)-2-naphthoate (7). Yellow prism (EtOH): mp 129–130 °C; TLC R_f = 0.68 (cyclohexane/ CH_2Cl_2 = 1/2); ^1H NMR (400 MHz, CDCl_3 , δ): (10.97, 10.51 (br, OH, 1H)), 8.45 (s, naphthalene H-1, 1H), (8.13, 7.77, 7.57, 7.19 (br, naphthalene H-5', 6', 7', 8', 4H), 7.15 (d, J = 7.6 Hz, naphthalene H-8, 1H), 6.79 (t, J = 7.5 Hz, naphthalene H-8, 1H),

6.87 (t, $J = 7.8$ Hz, naphthalene H-7, 1H), 6.66 (d, $J = 7.8$ Hz, naphthalene H-5, 1H), 5.36, 4.97 (br, CH, 1H), 4.03 (s, CO_2CH_3 , 3H), 3.16–2.86 (m, ArCH_2 -, 2H), 2.59–1.83 (m, $-\text{CH}_2\text{CH}_2$ -, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 170.54 (C=O), 154.01 (naphthalene C-3), 140.24 (Ar C), 136.43 (Ar C), 135.29 (Ar C), 131.46 (Ar CH), 130.22 (Ar CH), 128.79 (Ar CH), 128.02 (Ar CH), 127.86 (Ar CH), 125.86 (Ar CH), 125.36 (Ar CH), 125.03 (Ar CH), 122.95 (Ar CH), 52.69 (CO_2CH_3), 36.20 (naphthalene C-1'), 30.40, 29.99 (naphthalene C-2', 4'), 24.12 (naphthalene C-3'), Three quaternary carbon signal were not detected because of the broad characters.; IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3190 (br), 2920 (m), 1670 (C=O, s), 1440 (s), 1320 (s), 1290 (s), 1210 (s), 1160 (m), 1080 (m), 960 (m), 800 (m), 750 (s); EIMS (70 eV) m/z (relative intensity): 332 (M^+ , 100), 300 ($\text{M} - \text{CH}_3\text{OH}$, 62), 272 ($\text{M} - \text{CH}_3\text{OH}$, $-\text{C}=\text{O}$, 60), 253 (15), 228 (24), 215 (14); Anal. Calcd for $\text{C}_{22}\text{H}_{20}\text{O}_3$: C, 79.50; H, 6.06. Found: C, 79.32; H, 6.09.

Synthesis of Substrate for Table 5.

3,4-Dihydro-1-naphthyl acetate (12). In a 500 mL flask, *p*-toluenesulfonic acid hydrate (1.303 g, 6.853 mmol) was azeotropically dehydrated using toluene (20 mL $\times$ 2). Isopropenyl acetate (205.8 g, 226.4 mmol) and 1-tetralone (10.02 g, 68.52 mmol) were added to the toluene solution, and the whole was heated at reflux for 42 h. The mixture was cooled and poured into saturated aq NaHCO_3 (300 mL), extracted three times with hexane (200 mL). The combined organic layer was washed with brine (200 mL), dried over MgSO_4 , filtered. The filtrate was concentrated under reduced pressure to give a pale brown oil. The oil was purified by bulb-to-bulb distillation (171–180 $^\circ\text{C}$ /0.69 Torr) to afford colorless oil (12.652 g, 98%), which crystallized on standing. Colorless prism: mp 44–53 $^\circ\text{C}$; TLC R_f = 0.54 (hexane/ CH_2Cl_2 = 1/2); ^1H NMR (300 MHz, CDCl_3 , δ): 7.19–7.07 (m, Ar H, 4H), 5.70 (t, $J = 4.7$ Hz, naphthalene H-2, 1H), 2.87 (t, $J = 7.2$ Hz, ArCH_2CH_2 -, 2H), 2.44 (td, $J = 8.1, 4.6$ Hz, ArCH_2CH_2 -, 2H), 2.30 (s, COCH_3 , 3H); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2930 (m), 1740 (C=O, s), 1660 (m), 1370 (m), 1210 (s), 1130 (m), 1080 (s), 1040 (m), 770 (s), 740 (s); EIMS (70 eV) m/z (relative intensity): 188 (M^+ , 20), 146 (100), 131 (27), 115 (13).

Reaction Procedure for Table 5.

The reactions were carried out as the same procedure as entry 8 in Table 1.

Products for Table 5.

2-(1-(2-Methoxyphenyl)ethyl)naphthalene (14a). Colorless viscous oil: TLC R_f = 0.57

(cyclohexane/CH₂Cl₂ = 2/1); ¹H NMR (300 MHz, CDCl₃, δ): 7.90–7.68 (m, naphthalene H-1, 4, 5, 8, 4H), 7.43–7.36 (m, naphthalene H-6, 7, 2H), 7.34 (dd, *J* = 8.4, 1.8 Hz, naphthalene H-3, 1H), 7.22–7.13 (m, phenyl H-4, 6, 2H), 6.93–6.84 (m, phenyl H-3, 5, 2H), 4.73 (q, *J* = 7.3 Hz, -CHCH₃, 1H), 3.78 (s, OCH₃, 3H), 1.67 (d, *J* = 7.2 Hz, -CHCH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): 156.48 (phenyl C-2), 143.52 (naphthalene C-2), 134.43 (Ar C), 133.18 (Ar C), 131.67 (Ar C), 127.54 (Ar CH), 127.36 (Ar CH), 127.21 (Ar CH), 127.16 (Ar CH), 126.89 (Ar CH), 126.79 (Ar CH), 125.36 (Ar CH), 124.70 (Ar CH), 120.22 (phenyl C-5), 55.31 (OCH₃), 37.40 (-CHCH₃), 20.73 (-CHCH₃); IR (CHCl₃) $\tilde{\nu}_{\max}$: (cm⁻¹): 2950 (m), 1600 (m), 1490 (s), 1460 (m), 1290 (m), 1240 (s), 1030 (m), 860 (m), 820 (m); EIMS (70 eV) *m/z* (relative intensity): 262 (M⁺, 92), 247 (M – CH₃, 74), 231 (17), 215 (16), 202 (11), 141 (100); HRMS-EI (*m/z*): M⁺ calcd for C₁₉H₁₈O, 262.1358; found, 262.1352.

2-(1-(4-Methoxyphenyl)ethyl)naphthalene (14b). Colorless viscous oil: TLC *R_f* = 0.42 (cyclohexane/CH₂Cl₂ = 2/1); ¹H NMR (400 MHz, CDCl₃, δ): 7.79–7.76 (m, naphthalene H-5, 8, 2H), 7.72 (d, *J* = 8.5 Hz, naphthalene H-4, 1H), 7.67 (s, naphthalene H-1, 1H), 7.44 (td, *J* = 7.8, 1.7 Hz, naphthalene H-7, 1H), 7.40 (td, *J* = 6.8, 1.7 Hz, naphthalene H-6, 1H), 7.29 (dd, *J* = 8.5, 1.7 Hz, naphthalene H-3, 1H), 7.16 (d, *J* = 9.0 Hz, phenyl H-2, 6, 2H), 6.82 (d, *J* = 8.5 Hz, phenyl H-3, 5, 2H), 4.26 (q, *J* = 7.1 Hz, -CHCH₃, 1H), 3.76 (s, OCH₃, 3H), 1.70 (d, *J* = 7.1 Hz, -CHCH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): 157.68 (phenyl C-4), 143.98 (Ar C), 138.19 (Ar C), 133.38 (Ar C), 131.92 (Ar C), 128.48 (phenyl C-2, 6), 127.75 (Ar CH), 127.55 (Ar CH), 127.40 (Ar CH), 126.64 (Ar CH), 125.75 (Ar CH), 125.15 (Ar CH), 125.06 (Ar CH), 113.66 (phenyl C-3, 5), 55.24 (OCH₃), 44.09 (-CHCH₃), 22.07 (-CHCH₃); IR (neat) $\tilde{\nu}_{\max}$: (cm⁻¹): 2960 (m), 1610 (m), 1520 (s), 1470 (m), 1300 (m), 1260 (m), 1250 (s), 1180 (s), 1040 (s), 860 (m), 840 (s), 740 (s); EIMS (70 eV) *m/z* (relative intensity): 262 (M⁺, 49), 247 (M – CH₃, 100), 215 (14); HRMS-EI (*m/z*): M⁺ calcd for C₁₉H₁₈O, 262.1358; found, 262.1357; Anal. Calcd for C₁₉H₁₈O: C, 86.99; H, 6.92. Found: C, 87.35; H, 7.05.

2-(1-(4-Methoxy-3-(1-(2-naphthyl)ethyl)phenyl)ethyl)naphthalene, diastereo mixture (14c). Colorless viscous oil: TLC *R_f* = 0.51 (cyclohexane/CH₂Cl₂ = 2/1); ¹H NMR (400 MHz, CDCl₃, δ): 7.78–7.60 (m, naphthalene H, 8H), 7.46–7.37 (m, naphthalene H, 4H), 7.32–7.27 (m, naphthalene H, 2H), (7.12 (d, *J* = 9.2 Hz) and 7.11 (d, *J* = 8.8 Hz), phenyl H-6, 1H), 7.05–7.01 (m, phenyl H-2, 1H), (6.77 (d, *J* = 8.4 Hz) and 6.76 (d, *J* = 8.4 Hz), phenyl H-5, 1H), (4.69 (q, *J* = 7.2 Hz) and 4.68 (d, *J* = 7.2 Hz), *ortho* position to OMe, 1H), (4.23 (q, *J* = 7.2 Hz) and 4.21 (d, *J* = 7.2 Hz), *para* position to OMe,

1H), (3.74, 3.74, 3.72 and 3.72 (s, OCH₃, 3H)), 1.67–1.61 (m, -CHCH₃, 7H); ¹³C NMR (100 MHz, CDCl₃, δ): 155.08 (phenyl C-4), 144.12 (Ar C), 144.07 (Ar C), 143.75 (Ar C), 143.73 (Ar C), 137.91 (Ar C), 137.88 (Ar C), 134.29 (Ar C), 134.15 (Ar C), 133.35 (Ar C), 131.87 (Ar C), 131.82 (Ar CH), 127.65 (Ar CH), 127.56 (Ar CH), 127.34 (Ar CH), 126.99 (Ar CH), 126.93 (Ar CH), 126.67 (Ar CH), 125.86 (Ar CH), 125.66 (Ar CH), 125.50 (Ar CH), 125.47 (Ar CH), 125.20 (Ar CH), 125.18 (Ar CH), 125.08 (Ar CH), 125.03 (Ar CH), 124.88 (Ar CH), 110.45 (phenyl C-5), 55.54 (OCH₃), 44.32 and 44.20 (-CHCH₃, *para* position to -OCH₃), 37.83 and 37.72 (-CHCH₃, *ortho* position to -OCH₃), 22.26, 22.12, 21.08 and 20.90 (-CHCH₃); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 2960 (s), 1630 (m), 1600 (m), 1500 (m), 1470 (m), 1450 (m), 1380 (m), 1290 (m), 1250 (s), 1130 (m), 1040 (s), 960 (m), 900 (m), 860 (m), 820 (s), 750 (s); EIMS (70 eV) *m/z* (relative intensity): 416 (M⁺, 100), 401 (M – CH₃, 92), 261 (16), 155 (24), 141 (38); HRMS-EI (*m/z*): M⁺ calcd for C₃₁H₂₈O, 416.2140; found, 416.2147; Anal. Calcd for C₃₁H₂₈O: C, 89.38; H, 6.78. Found: C, 89.65; H, 6.69.

(1-(2-Naphthyl)ethyl)-2-methoxynaphthalene, regioisomer, high R_f value (**15a**). Colorless viscous oil: TLC R_f = 0.92 (cyclohexane/CH₂Cl₂ = 1/1); ¹H NMR (400 MHz, CDCl₃, δ): 7.92–7.70 (m, Ar H, 6H), 7.62 (d, *J* = 8.5 Hz, Ar H, 1H), 7.45–7.32 (m, Ar H, 2H), 7.30–7.20 (m, Ar H, 4H), 5.41 (q, *J* = 7.1 Hz, -CHCH₃, 1H), 3.44 (s, OCH₃, 3H), 1.92 (d, *J* = 7.3 Hz, -CHCH₃, 3H); EIMS (70 eV) *m/z* (relative intensity): 312 (M⁺, 100), 297 (M – CH₃, 42), 281 (17), 265 (16), 156 (13), 141 (65), 126 (11); HRMS-EI (*m/z*): M⁺ calcd for C₂₃H₂₀O, 312.1514; found, 312.1521.

(1-(2-Naphthyl)ethyl)-2-methoxynaphthalene, regioisomer, low R_f value (**15b**). Colorless viscous oil: TLC R_f = 0.86 (cyclohexane/CH₂Cl₂ = 1/1); ¹H NMR (400 MHz, CDCl₃, δ): 7.80–7.75 (m, Ar H, 2H), 7.73–7.61 (m, Ar H, 2H), 7.47–7.35 (m, Ar H, 2H), 7.33 (dd, *J* = 8.5, 1.7 Hz, Ar H, 1H), 7.29 (dd, *J* = 8.3, 1.7 Hz, Ar H, 1H), 7.14–7.07 (m, Ar H, 2H), 4.43 (q, *J* = 7.3 Hz, -CHCH₃, 1H), 3.89 (s, OCH₃, 3H), 1.80 (d, *J* = 7.1 Hz, -CHCH₃, 3H); EIMS (70 eV) *m/z* (relative intensity): 312 (M⁺, 100), 297 (M – CH₃, 81), 265 (15), 253 (10), 156 (15), 141 (21), 126 (11); HRMS-EI (*m/z*): M⁺ calcd for C₂₃H₂₀O, 312.1514; found, 312.1521.

Bis(1-(2-naphthyl)ethyl)-2-methoxynaphthalene, regioisomer, diastereomixture (**15c**) Colorless viscous oil: TLC R_f = 0.80 (cyclohexane/ CH_2Cl_2 = 1/1); ^1H NMR (400 MHz, CDCl_3 , δ): 7.85–7.55 (m, Ar H, 11H), 7.47–7.23 (m, Ar H, 7H), 7.17–7.05 (m, Ar H, 1H), 5.36 (q, J = 7.1 Hz, $-\text{CHCH}_3$, 1H), (4.36 (q, J = 7.1 Hz, $-\text{CHCH}_3$) 4.34 (q, J = 6.8 Hz, $-\text{CHCH}_3$) 1H), (1.89 (d, J = 7.3 Hz, $-\text{CHCH}_3$), 1.88 (d, J = 7.1 Hz, $-\text{CHCH}_3$) 3H), (1.74 (d, J = 7.1 Hz, $-\text{CHCH}_3$), 1.75 (d, J = 7.2 Hz, $-\text{CHCH}_3$) 3H); IR (CHCl_3) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2960 (m), 1610 (m), 1600 (s), 1500 (s), 1470 (m), 1380 (m), 1300 (m), 1270 (s), 1250 (s), 1080 (s), 950 (m), 900 (m), 860 (m), 820 (s); EIMS (70 eV) m/z (relative intensity): 466 (M^+ , 100), 451 ($\text{M} - \text{CH}_3$, 23), 169 (11), 155 (20), 141 (19); HRMS-EI (m/z): M^+ calcd for $\text{C}_{35}\text{H}_{30}\text{O}$, 466.2297; found, 466.2293.

2-Methyl-5-(1-(2-naphthyl)ethyl)furan (16). Colorless oil: TLC R_f = 0.61 (hexane/ AcOEt = 5/1); ^1H NMR (400 MHz, CDCl_3 , δ): 7.80–7.74 (m, naphthalene H-4, 5, 8, 3H), 7.65 (s, naphthalene H-1, 1H), 7.45–7.37 (m, naphthalene H-7, 7, 2H), 7.35 (d, J = 8.8 Hz, naphthalene H-3, 1H), 5.95–5.93 (m, furan H-3, 1H), 5.88–5.86 (m, furan H-4, 1H), 4.22 (q, J = 7.2 Hz, $-\text{CHCH}_3$, 1H), 2.21 (s, furan CH_3 , 3H), 1.63 (d, J = 7.6 Hz, $-\text{CHCH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 156.86 (furan C-5), 150.68 (furan C-2), 141.74 (naphthalene C-2), 133.42 (naphthalene C-8a), 132.20 (naphthalene C-4a), (127.86, 127.56, 127.42 (naphthalene C-4, 5, 8)), (125.89, 125.73, 125.42, 125.23 (naphthalene C-1, 3, 6, 7)), 105.9 (furan C-3), 105.63 (furan C-4), 39.49 ($-\text{CHCH}_3$), 20.65 ($-\text{CHCH}_3$), 13.70 (furan CH_3); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2960 (m), 1700 (m), 1600 (m), 1550 (m), 1440 (m), 1370 (m), 1210 (m), 1020 (m), 940 (m), 860 (m), 820 (m), 780 (m), 740 (s); EIMS (70 eV) m/z (relative intensity): 236 (M^+ , 41), 221 ($\text{M} - \text{CH}_3$, 100), 178 (10); HRMS-EI (m/z): M^+ calcd for $\text{C}_{17}\text{H}_{16}\text{O}$, 236.1201; found, 236.1204.

4-(2-Naphthyl)-1-pentene (17).⁹ Colorless oil: TLC R_f = 0.54 (hexane/ AcOEt = 15/1); ^1H NMR (400 MHz, CDCl_3 , δ): 7.80–7.77 (m, naphthalene H-4, 5, 8, 3H), 7.61 (s, naphthalene H-1, 1H), 7.44 (td, J = 6.8, 1.5 Hz, naphthalene H-7, 1H), 7.40 (td, J = 6.8, 1.5 Hz, naphthalene H-6, 1H), 7.35 (dd, J = 8.5, 1.7 Hz, naphthalene H-3, 1H), 5.79–5.68 (m, $-\text{CH}=\text{CH}_2$, 1H), 5.04–4.98 (m, $-\text{CH}=\text{CH}_2$, *trans*, 1H), 4.96–4.93 (m, $-\text{CH}=\text{CH}_2$, *cis*, 1H), 2.96 (qt, J = 6.8, 6.8 Hz, $-\text{CHCH}_3$, 1H), 2.51–2.44 (m, with quintet character, CHCHH , 1H), 2.40–2.33 (m, with quintet character, CHCHH , 1H), 1.33 (d, J = 7.1 Hz,

-CHCH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): 144.29 (naphthalene C-2), 136.92 (-CH=CH₂), 133.45 (naphthalene C-8a), 132.05 (naphthalene C-4a), (127.73, 127.43, 127.43 (naphthalene C-4, 5, 8)), 125.73 (naphthalene C-3), 125.68 (naphthalene C-7), 124.98 (naphthalene C-6), 124.94 (naphthalene C-1), 115.87 (-CH=CH₂), 42.63 (-CHCH₂=), 40.00 (-CHCH₃), 21.64 (-CHCH₃); IR (neat) $\tilde{\nu}_{\max}$: (cm⁻¹): 3060 (w), 2960 (w), 2920 (w), 1645 (m), 1605 (w), 1505 (w), 1460 (w), 1440 (w), 1380 (w), 1280 (w), 1140 (w), 1000 (m), 920 (m), 860 (m), 820 (m), 755 (s); EIMS (70 eV) *m/z* (relative intensity): 196 (M⁺, 17), 155 (M - CH₂CH=CH₂, 100); HRMS-EI (*m/z*): M⁺ calcd for C₁₅H₁₆, 196.1252; found, 196.1249; Anal. Calcd for C₁₅H₁₆: C, 91.78; H, 8.22. Found: C, 92.12; H, 8.34.

2-(1-(2-Naphthyl)ethyl)3,4-dihydro-1(2H)-naphthalenone, diastereomixture (18). Colorless viscous oil: TLC *R_f* = 0.33 (cyclohexane/CH₂Cl₂ = 1/1); ¹H NMR (400 MHz, CDCl₃, δ): 8.10–8.03 (m, Ar H, 2H), 7.84–7.76 (m, Ar H, 6H), 7.72–7.67 (m, Ar H, 2H), 7.49–7.37 (m, Ar H, 8H), 7.34–7.26 (m, Ar H, 2H), 7.21–7.16 (m, Ar H, 2H), 4.10 (qd, *J* = 7.0, 3.7 Hz, -CHCH₃, 1H), 3.58 (qd, *J* = 7.6, 7.6 Hz, -CHCH₃, 1H), 3.02 (ddd, *J* = 17.1, 8.1, 5.0 Hz, -CHC=O, 1H), 2.90 (ddd, *J* = 12.3, 4.1, 4.1 Hz, -CHC=O, 1H), 2.86–2.73 (m, Ar CH₂ x 2, 4H), (2.14–1.92 (m, 7H), 1.60–1.55 (m, , 1H) Ar CH₂CH₂-, 2H x 2), 1.43 (d, *J* = 6.7 Hz, -CHCH₃, 3H), 1.36 (d, *J* = 7.0 Hz, -CHCH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): for a major isomer 198.28 (C=O), 143.28 (tetralone C-4a), 141.81 (naphthalene C-2), 54.60 (-CHC=O), 38.02 (-CHCH₃), 29.20 (ArCH₂CH₂-), 25.80 (ArCH₂CH₂-), 20.77 (-CHCH₃), for a minor isomer 199.43 (C=O), 143.79 (tetralone C-4a), 142.55 (naphthalene C-2), 53.59 (-CHC=O), 37.15 (-CHCH₃), 27.11 (ArCH₂CH₂-), 23.11 (ArCH₂CH₂-), 14.17 (-CHCH₃), for the major and minor isomers (133.29, 133.26 (tetralone C-8a)), (132.98, 132.98 (naphthalene C-6)), (132.78, 132.42 (naphthalene C-8a)), 132.12, 131.97 (naphthalene C-4a)), (128.49, 128.44 (tetralone C-5)), (127.88, 127.70 (tetralone C-8)), 127.46 (Ar CH), 127.43 (Ar CH), 127.38 (Ar CH), 127.38 (Ar CH), 127.38 (Ar CH), 127.34 (Ar CH), 126.82 (Ar CH), 126.41 (Ar CH), 126.39 (Ar CH), 126.21 (Ar CH), 125.81 (Ar CH), 125.78 (Ar CH), 125.78 (Ar CH), 125.53 (Ar CH), 125.23 (Ar CH), 125.17 (Ar CH); IR (neat) $\tilde{\nu}_{\max}$: (cm⁻¹): 2940 (m), 1685 (m), 1670 (s), 1600 (s), 1450 (m), 1280 (s), 1220 (s), 910 (m), 860 (m), 810 (m), 750 (s); EIMS (70 eV) *m/z* (relative intensity): 300 (36), 155 (100), 146 (14); HRMS-EI (*m/z*): M⁺ calcd for C₂₂H₂₀O, 300.1514; found, 300.1519.

1-Methyl-3-phenyl-2,3-dihydro-1H-cyclopenta[b]naphthalene, diastereomixture (19). Colorless oil: TLC *R_f* = 0.56 (hexane/AcOEt = 20/1); ¹H NMR (400 MHz, CDCl₃, δ): for a major isomer

7.86–7.76 (m, Ar H, 2H), 7.42 (d, $J = 8.3$ Hz, Ar H, 2H), 7.36–7.11 (m, Ar H, 7H), 7.03–7.01 (m with doublet character, Ar H, 1H), 4.73 (dd, $J = 8.1, 8.1$ Hz, $-\underline{\text{CH}}\text{Ph}$, 1H), 3.36 (qt, $J = 7.3, 7.3$ Hz, $-\underline{\text{CH}}\text{CH}_3$, 1H), 3.00 (ddd, $J = 12.8, 8.5, 8.5$ Hz, $-\underline{\text{CHH}}-$, 1H), 1.72 (ddd, $J = 12.9, 7.3, 7.3$ Hz, $-\underline{\text{CHH}}-$, 1H), 1.40 (d, $J = 7.1$ Hz, $-\underline{\text{CHCH}}_3$, 3H), for a minor isomer 7.86–7.76 (m, Ar H, 2H), 7.51 (d, $J = 8.3$ Hz, Ar H, 1H), 7.44 (d, $J = 8.3$ Hz, Ar H, 1H), 7.36–7.11 (m, Ar H, 7H), 4.85 (dd, $J = 6.3, 2.7$ Hz, $-\underline{\text{CH}}\text{Ph}$, 1H), 3.54 (qt, $J = 7.6, 7.6$ Hz, $-\underline{\text{CH}}\text{CH}_3$, 1H), 2.49 (ddd, $J = 12.7, 7.3, 2.9$ Hz, $-\underline{\text{CHH}}-$, 1H), 2.29 (ddd, $J = 12.7, 8.8, 8.8$ Hz, $-\underline{\text{CHH}}-$, 1H), 1.36 (d, $J = 6.8$ Hz, $-\underline{\text{CHCH}}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): for the major isomer 147.16 (Ar C), 145.65 (Ar C), 139.29 (Ar C), 133.20 (Ar C), for minor isomer 146.64 (Ar C), 146.14 (Ar C), 140.03 (Ar C), 132.99 (Ar C), 130.14 (Ar C), for the major and minor isomers 128.39 (Ar CH), 128.31 (Ar CH), 128.26 (Ar CH), 128.02 (Ar CH), 127.94 (Ar CH), 127.70 (Ar CH), 127.28 (Ar CH), 125.83 (Ar CH), 125.48 (Ar CH), 124.88 (Ar CH), for the major isomer 121.92 (Ar CH), 50.32 ($-\underline{\text{CH}}\text{Ph}$), 45.96 ($\underline{\text{CH}}_2$), 39.48 ($-\underline{\text{CHCH}}_3$), 21.32 ($-\underline{\text{CHCH}}_3$), for the minor isomer 121.81 (Ar CH), 48.86 ($-\underline{\text{CH}}\text{Ph}$), 45.70 ($\underline{\text{CH}}_2$), 38.46 ($-\underline{\text{CHCH}}_3$), 20.23 ($-\underline{\text{CHCH}}_3$); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2950 (m), 2850 (m), 1600 (m), 1450 (m), 1390 (m), 820 (m), 750 (m), 700 (m); EIMS (70 eV) m/z (relative intensity): 258 (M^+ , 100), 243 ($\text{M} - \text{CH}_3$, 58), 228 (11), 181 (12), 165 (26), 115 (10), 91 (11); HRMS-EI (m/z): M^+ calcd for $\text{C}_{20}\text{H}_{18}$, 258.1409; found, 258.1404.

Synthesis of Substrates for Table 6.

1-(6-Methoxy-2-naphthyl)ethanol (20). To a solution of 6-methoxy-2-acenaphthone (3.00 g, 14.18 mmol) in EtOH (50 mL) was added NaBH_4 (0.68 g, 17.98 mmol) at room temperature. The whole was heated at reflux for 30 min, then cooled, and concentrated at reduced pressure. The residue was dissolved in AcOEt (50 mL) and H_2O (50 mL), and extracted three times with AcOEt (50 mL). The combined organic layer was washed once with brine (100 mL), dried over MgSO_4 , filtered, and the filtrate was concentrated to afford a colorless solid. The solid was recrystallized from cyclohexane to give colorless fine needle (2.84 g, 94%): TLC $R_f = 0.15$ (hexane/AcOEt = 4/1); ^1H NMR (300 MHz, CDCl_3 , δ): 7.74–7.70 (m, naphthalene H-1, 4, 8, 3H), 7.48 (dd, $J = 9.8, 1.5$ Hz, naphthalene H-3, 1H), 7.17–7.12 (m, naphthalene H-5, 7, 2H), 5.03 (dd, $J = 6.3, 1.5$ Hz, $-\underline{\text{CH}}-$, 1H), 3.92 (s, OCH_3 , 3H), 1.92 (br, OH, 1H), 1.57 (d, $J = 6.3$ Hz, $-\underline{\text{CHCH}}_3$, 3H); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3350 (br), 1610 (m), 1260 (m), 1170 (m), 1030 (m), 860 (m); EIMS (70 eV) m/z (relative intensity): 202 (M^+ , 88), 187 ($\text{M} - \text{CH}_3$, 100), 184 ($\text{M} - \text{H}_2\text{O}$, 32), 159 (48), 144 (38), 115 (22).

1-(2-Methoxy-1-naphthyl)ethanol (21). Under argon atmosphere, a solution of 2-methoxy-1-naphthol (500.0 mg, 2.68 mmol) in Et₂O (8 mL) was added to a solution of methyl magnesium bromide (3.0 mol/L in Et₂O, 0.89 mL, 2.68 mmol) at 0 °C. After stirred for 1.5 hr, saturated aq NH₄Cl was added, and extracted with Et₂O (50 mL x 3). The combined organic layer was washed with brine (100 mL), and dried over MgSO₄, filtered, and the filtrate was evaporated to afford colorless solid (516.5 mg, 95%). The solid was recrystallized from hexanes to give colorless prism: mp 82–82 °C; TLC R_f = 0.27 (hexane/AcOEt = 3/1); ¹H NMR (300 MHz, CDCl₃, δ): 8.09 (d, *J* = 8.7 Hz, naphthalene H-8, 1H), 7.82–7.76 (m, naphthalene H-4, 5, 2H), 7.48 (ddd, *J* = 8.4, 6.6, 1.5 Hz, naphthalene H-7, 1H), 7.35 (ddd, *J* = 8.1, 6.6, 0.9 Hz, naphthalene H-6, 1H), 7.29 (d, *J* = 9.0 Hz, naphthalene H-3, 1H), 5.74 (qd, *J* = 9.3, 6.6 Hz, -CHCH₃, 1H), 4.02 (s, OCH₃, 3H), 3.96 (d, *J* = 9.6 Hz, OH, 1H), 1.67 (d, *J* = 6.6 Hz, -CHCH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): 153.99 (naphthalene C-2), 131.08 (naphthalene C-8a), 129.21 (naphthalene C-4a), 128.87 (naphthalene C-5), 128.42 (naphthalene C-4), 126.39 (naphthalene C-7), 125.49 (naphthalene C-1), 123.43 (naphthalene C-8), 122.70 (naphthalene C-6), 113.26 (naphthalene C-3), 65.98 (-CHCH₃), 56.37 (OCH₃), 23.87 (-CHCH₃); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 3300 (br), 1600 (m), 1250 (s), 1080 (m), 820 (m); EIMS (70 eV) *m/z* (relative intensity): 202 (M⁺, 47), 187 (M – CH₃, 100), 169 (15), 153 (12), 144 (23), 127 (11), 115 (15); HRMS-EI (*m/z*): M⁺ calcd for C₁₃H₁₄O₂, 202.0994; found, 202.0995; Anal. Calcd for C₁₃H₁₄O₂: C, 77.20; H, 6.98. Found: C, 77.49; H, 7.05.

1-Methyl-1,2,3,4-tetrahydro-1-naphthol (24).¹⁰ Under argon atmosphere, a solution of 1-tetralone (7.00 g, 47.88 mmol) in Et₂O (20 mL) was added to a solution of methyl magnesium bromide (3.0 mol/L in Et₂O, 19.15 mL, 57.46 mmol) at 0 °C over 30 min. The mixture became a suspension. Et₂O was added (15 mL) was added, and the whole was stirred for 1.5 hr. The mixture was poured into saturated aq NH₄Cl, and extracted with Et₂O (100 mL x 3). The combined organic layer was washed with brine (100 mL), dried over MgSO₄, filtered, concentrated, and purified by column chromatography (silica gel, hexane/AcOEt = 10/1 to 4/1) to afford colorless solid (6.72 g, 87%). The solid was recrystallized from hexane to give colorless plate: mp 86–88 °C; TLC R_f = 0.19 (hexane/AcOEt = 10/1); ¹H NMR (300 MHz, CDCl₃, δ): 7.58 (dd, *J* = 7.5, 1.5 Hz, Ar H, 1H), 7.24–7.13 (m, Ar H, 2H), 7.09–7.04 (m, Ar H, 1H), 2.88–2.70 (m, ArCH₂-, 2H), 1.97–1.76 (m, ArCH₂ CH₂CH₂-, 4H), 1.56 (s, CCH₃, 3H); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 3310 (br), 2940 (s), 1440 (m), 1360 (m), 1180 (m), 1150 (m), 1100 (m), 990 (m), 930 (m), 760 (m), 730 (m); EIMS (70 eV) *m/z* (relative intensity): 162 (M⁺, 5), 147 (M – CH₃, 100), 129 (19), 119 (15), 91 (21).

1-(2-Thienyl)ethanol (25).¹¹ To a solution of 2-acetylthiophene (5.02 g, 39.76 mmol) in EtOH (40 mL) was added NaBH₄ (1.51 g, 39.76 mmol) at room temperature. The whole was stirred for 30 min, then concentrated at reduced pressure. The residue was dissolved in AcOEt (50 mL) and H₂O (50 mL), and extracted three times with AcOEt (50 mL). The combined organic layer was washed once with brine (100 mL), dried over MgSO₄, filtered, and the filtrate was concentrated to afford colorless oil (4.87 g, 96%). The residue was purified by bulb-to-bulb distillation (105 °C/0.55 Torr) to give a colorless oil (4.72g, 93%): TLC R_f = 0.50 (AcOEt/hexane = 1/1); ¹H NMR (300 MHz, CDCl₃, δ): 7.23 (dd, *J* = 4.5, 1.5 Hz, thiophene H-5, 1H), 6.98–6.93 (m, thiophene H-3, 4, 2H), 5.12 (dd, *J* = 6.3, 4.2 Hz, -CHCH₃, 1H), 2.09 (d, *J* = 4.2 Hz, OH, 1H), 1.60 (d, *J* = 6.3 Hz, -CHCH₃, 3H); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 3350 (br), 2960 (m), 1460 (m), 1380 (m), 1090 (m), 860 (m); EIMS (70 eV) *m/z* (relative intensity): 128 (M⁺, 66), 113 (100), 97 (85).

Reaction Procedure for Table 6.

The reactions were carried out as the same procedure as entry 8 in Table 1.

Products for Table 6.

Methyl 3-hydroxy-4-(1-(6-methoxy-2-naphthyl)ethyl)-2-naphthoate (26). Yellow prism (EtOH): mp 158–158.5 °C; TLC R_f = 0.33 (hexane/AcOEt = 4/1); ¹H NMR (300 MHz, CDCl₃, δ): 10.90 (s, OH, 1H), 8.46 (s, naphthalene H-1, 1H), 7.82–7.77 (m, Ar H, 3H), 7.68 (d, *J* = 9.0 Hz, Ar H, 1H), 7.55 (d, *J* = 8.7 Hz, Ar H, 1H), 7.30–7.19 (m, Ar H, 3H), 7.10 (dd, *J* = 9.0, 2.4 Hz, side chain naphthalene H-7, 1H), 7.06 (d, *J* = 2.4 Hz, side chain naphthalene H-5, 1H), 5.46 (q, *J* = 7.2 Hz, -CHCH₃, 1H), 4.03 (s, CO₂CH₃, 3H), 3.88 (s, OCH₃, 3H), 1.94 (d, *J* = 7.2 Hz, -CHCH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): 170.53 (C=O), 156.95 (side chain, naphthalene C-1), 153.77 (naphthalene C-3), 140.64 (Ar C), 135.88 (naphthalene C-4a), 132.58 (Ar C), 131.50 (Ar CH), 130.19 (Ar CH), 129.08 (Ar CH), 128.87 (Ar CH), 128.44 (Ar CH), 127.44 (naphthalene C-8a), 126.97 (Ar CH), 126.36 (Ar CH), 125.77 (naphthalene C-1), 124.24 (Ar CH), 124.11 (Ar CH), 123.05 (Ar CH), 118.31 (side chain, naphthalene C-7), 113.69 (naphthalene C-4), 105.58 (side chain, naphthalene C-5), 55.31 (OCH₃), 52.67 (CO₂CH₃), 34.27 (-CHCH₃), 17.84 (-CHCH₃); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 3450 (m), 3170 (m), 1680 (C=O, s), 1610 (m), 1440 (s), 1220 (s), 1210 (s), 800 (m); EIMS (70 eV) *m/z* (relative intensity): 380 (M⁺, 100), 371 (M – CH₃, 27), 339 (M – CH₃, – CH₃OH, 39), 326 (44), 311 (18), 177 (20), 155 (16); Anal. Calcd for C₂₅H₂₂O₄: C, 77.70; H, 5.74. Found: C, 77.42; H, 5.90.

Methyl 3-hydroxy-4-(1-(2-methoxy-1-naphthyl)ethyl)-2-naphthoate (27). Yellow prism (EtOH): mp 178.5–179.5 °C; TLC R_f = 0.36 (hexane/AcOEt = 5/1); ^1H NMR (300 MHz, CDCl_3 , δ): 11.22 (s, OH, 1H), 8.40 (s, naphthalene H-1, 1H), 8.31 (d, J = 8.4 Hz, naphthalene H-8, 1H), 8.18 (d, J = 8.2 Hz, naphthalene H-5, 1H), 7.72–7.66 (m, side chain naphthalene H-4, 5, 8, 3H), 7.35–7.13 (m, side chain naphthalene H-3, 6, 7, naphthalene H-6, 7, 3H), 5.85 (q, J = 7.2 Hz, $-\text{CHCH}_3$, 1H), 4.04 (s, CO_2CH_3 , 3H), 3.97 (s, OCH_3 , 3H), 2.05 (d, J = 6.9 Hz, $-\text{CHCH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 170.83 (C=O), 154.71 (side chain, naphthalene C-2), 153.63 (naphthalene C-3), 136.34 (naphthalene C-4a), 133.20 (side chain, naphthalene C-8a), 131.51 (naphthalene C-1), ((129.94, 128.31, 128.31 (side chain, naphthalene C-4, 5, 8)), 129.60 (side chain, naphthalene C-4a), (128.48, 126.07, 123.03, 122.89 (naphthalene C-6, 7, side chain, naphthalene C-6, 7)), 127.56 (naphthalene C-8a), 127.12 (naphthalene C-2), 125.86 (naphthalene C-4), 124.00 (naphthalene C-8), 123.96 (naphthalene C-5), 114.91 (side chain, naphthalene C-3), 113.62 (side chain, naphthalene C-1), 56.87 (OCH_3), 52.63 (CO_2CH_3), 33.13 ($-\text{CHCH}_3$), 21.15 ($-\text{CHCH}_3$); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3060 (m), 2960 (m), 1680 (C=O, s), 1450 (s), 1340 (s), 1300 (s), 1260 (s), 1210 (s), 1170 (m), 1080 (m), 810 (s); EIMS (70 eV) m/z (relative intensity): 386 (M^+ , 41), 339 ($\text{M} - \text{CH}_3$, $-\text{CH}_3\text{OH}$, 43), 228 (13), 168 (34), 158 (100), 115 (16); Anal. Calcd for $\text{C}_{25}\text{H}_{22}\text{O}_4$: C, 77.70; H, 5.74. Found: C, 77.62; H, 5.78.

Methyl 4-(diphenyl)methyl-3-hydroxy-2-naphthoate (28). Yellow prism (EtOH): mp 149–150 °C; TLC R_f = 0.68 (cyclohexane/ CH_2Cl_2 = 1/1); ^1H NMR (300 MHz, CDCl_3 , δ): 10.96 (s, OH, 1H), 8.50 (s, naphthalene H-1, 1H), 7.82–7.76 (m, naphthalene H-5, 8, 2H), 7.33–7.15 (m, naphthalene H-6, 7 and $\text{C}_6\text{H}_5 \times 2$, 12H), 6.67 (s, CH, 1H), 4.01 (s, CO_2CH_3 , 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 170.49 (C=O), 154.42 (naphthalene C-3), 142.72 (phenyl C-1), 136.55 (naphthalene C-4a), 132.25 (naphthalene C-1), 130.20 (naphthalene C-8), 129.09 (phenyl C-3, 5), 128.53 (naphthalene C-6), 128.06 (phenyl C-2, 6), 127.49 (naphthalene C-8a), 125.90 (phenyl C-4), 125.04 (naphthalene C-5), 123.36 (naphthalene C-2), 123.18 (naphthalene C-7), 113.82 (naphthalene C-4), 52.66 (CO_2CH_3), 46.86 ($-\text{CHPh}_2$); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 1680 (C=O, s), 1495 (m), 1440 (s), 1330 (s), 1300 (m), 1210 (s), 1160 (m), 1080 (m), 960 (m), 800 (m), 740 (s), 705 (s); EIMS (70 eV) m/z (relative intensity): 368 (M^+ , 91), 336 ($\text{M} - \text{CH}_3\text{OH}$, 65), 308 ($\text{M} - \text{CO}_2\text{CH}_3$, 100), 231 (20), 202 (17); HRMS-EI (m/z): M^+ calcd for $\text{C}_{25}\text{H}_{20}\text{O}_3$, 368.1412; found, 368.1417; Anal. Calcd for $\text{C}_{25}\text{H}_{20}\text{O}_3$: C, 81.50; H, 5.47. Found: C, 81.56; H,

5.47.

Methyl 3-hydroxy-4-(4-hydroxybenzyl)-2-naphthoate (29). Yellow needle (toluene or EtOH (–20 °C)): mp 165–166.5 °C (EtOH); TLC R_f = 0.66 (hexane/AcOEt = 1/1); ^1H NMR (300 MHz, CDCl_3 , δ): 10.80 (s, naphthol, OH, 1H), 8.46 (s, naphthalene H-1, 1H), 7.89 (dd, J = 8.4, 0.6 Hz, naphthalene H-8, 1H), 7.81 (dt, J = 7.8, 0.9 Hz, naphthalene H-5, 1H), 7.49 (ddd, J = 8.4, 6.9, 1.5 Hz, naphthalene H-6, 1H), 7.31 (ddd, J = 7.8, 6.6, 0.9 Hz, naphthalene H-7, 1H), 7.10 (d, J = 8.4 Hz, phenyl H-2, 6, 2H), 6.68 (d, J = 8.4 Hz, phenyl H-3, 5, 2H), 4.62 (br, phenol, OH, 1H), 4.41 (s, CH_2 , 2H), 4.03 (s, CO_2CH_3 , 3H); ^{13}C NMR (100 MHz, acetone- d_6 , δ): 171.14 (C=O), 155.94 (phenyl C-4), 154.15 (naphthalene C-3), 137.04 (phenyl C-1), 132.09 (naphthalene C-4a), 131.68 (naphthalene C-1), 130.71 (naphthalene C-8), 129.80 (phenyl C-2, 6), 129.76 (naphthalene C-6), 127.83 (naphthalene C-8a), (124.18, 121.04 (naphthalene C-5, 7)), 121.85 (naphthalene C-2), 115.65 (phenyl C-3, 5), 114.37 (naphthalene C-4), 53.14 (CO_2CH_3), 29.81 (CH_2); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3400 (br), 1670 (C=O, s), 1500 (s), 1440 (s), 1300 (m), 1220 (m), 1090 (m), 970 (m), 800 (m), 780 (m); EIMS (70 eV) m/z (relative intensity): 308 (M^+ , $\text{M} - \text{CH}_3\text{OH}$, 75), 276 ($\text{M} - \text{CH}_3\text{OH}$, – CO, 33), 248 (100), 231 (12), 202 (10), 189 (11); Anal. Calcd for $\text{C}_{19}\text{H}_{16}\text{O}_4$: C, 74.01; H, 5.23. Found: C, 74.23; H, 5.33.

Reaction Procedure for Scheme 3.

The reactions were carried out as the same procedure as entry 8 in Table 1.

Product for Scheme 3.

2-(1-(6-Methoxy-2-naphthyl)ethyl)furan (30). Colorless needle (EtOH): mp 78–79 °C; TLC R_f = 0.52 (hexane/AcOEt = 4/1); ^1H NMR (400 MHz, CDCl_3 , δ): 7.69–7.65 (m with doublet character, naphthalene H-4, 8, 2H), 7.58 (d, J = 1.5 Hz, naphthalene H-1, 1H), 7.33–7.29 (m, naphthalene H-3, furan H-5, 2H), 7.13–7.19 (m, naphthalene H-5, 7, 2H), 6.30 (dd, J = 3.2, 2.0 Hz, furan H-4, 1H), 6.07 (ddd, J = 3.2, 0.8, 0.8 Hz, furan H-3, 1H), 4.25 (q, J = 7.2 Hz, $-\text{CHCH}_3$, 1H), 3.90 (s, OCH_3 , 3H), 1.66 (d, J = 7.2 Hz, $-\text{CHCH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 158.90 (furan C-2), 157.23 (naphthalene C-6), 141.15 (furan C-5), 139.13 (naphthalene C-2), 133.30 (naphthalene C-4a), (129.67, 126.84, 126.28, 125.30 (naphthalene C-1, 3, 4, 8), 128.91 (naphthalene C-8a), 118.60 (naphthalene C-7), 109.87 (furan C-4), 105.62 (naphthalene C-5), 104.90 (furan C-3), 55.34 (OCH_3), 39.29 ($-\text{CHCH}_3$), 20.58 ($-\text{CHCH}_3$); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 1605 (s), 1390 (m), 1270 (s), 1220 (m), 1180 (m), 1030 (s), 1005 (s),

930 (m), 860 (s), 820 (m), 750 (m); EIMS (70 eV) m/z (relative intensity): 252 (M^+ , 46), 237 ($M - CH_3$, 100), 194 (13), 165 (25); Anal. Calcd for $C_{17}H_{16}O_2$: C, 80.93; H, 6.39. Found: C, 80.80; H, 6.42.

2,5-Bis(1-(6-methoxy-2-naphthyl)ethyl)furan diastereomixture (31). Colorless needle (EtOH): mp 110–113 °C; TLC R_f = 0.67 (hexane/AcOEt = 4/1); ^1H NMR (300 MHz, $CDCl_3$, δ): 7.65–7.55 (m, naphthalene H, 4H), 7.53–7.49 (m, naphthalene H, 2H), 7.30 (ddd, J = 7.8, 5.7, 1.8 Hz, naphthalene H, 2H), 7.11–7.06 (m, naphthalene H, 2H), (5.98, 5.92 (s x 2, furan H-3, 4, 2H)), 4.23–4.13 (m with two quartet character, $-CHCH_3$, 2H), 3.90 (s, OCH_3 , 3H), 1.62 (d, J = 7.2 Hz, $-CHCH_3$, 3H), 1.59 (d, J = 7.2 Hz, $-CHCH_3$, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, δ): (157.63, 157.57 (furan C-2)), 157.15 (naphthalene C-6), (139.55, 139.34 (naphthalene C-2)), 133.18 (naphthalene C-4a), (129.08 and 129.06, 126.66 and 126.63, 126.49 and 126.44, 125.27 (naphthalene C-1, 3, 4, 8)), 128.87 (naphthalene C-8a), 118.46 (naphthalene C-7), 105.58 (naphthalene C-5), (105.38 and 105.25 (furan C-3)), 55.34 (OCH_3), (39.30 and 39.23 ($-CHCH_3$)), (20.76 and 20.61 ($-CHCH_3$)); IR (KBr) $\tilde{\nu}_{max}$: (cm^{-1}): 3470 (m), 2960 (m), 1600 (s), 1385 (m), 1190 (m), 1270 (m), 1220 (s), 1230 (m), 1030 (s), 860 (s), 820 (m), 800 (m); EIMS (70 eV) m/z (relative intensity): 436 (M^+ , 71), 421 ($M - CH_3$, 100), 251 (11), 203 (11), 185 (12); Anal. Calcd for $C_{30}H_{28}O_3$: C, 82.54; H, 6.46. Found: C, 82.24; H, 6.50.

Reaction Procedure for Table 7.

The reactions were carried out as same procedure as entry 8 in Table1.

Products for Table 7.

2-Methyl-5-(1,2,3,4-tetrahydro-1-naphthyl)furan (32) Colorless liquid: bulb to bulb dist. 150 °C/0.51 Torr; TLC R_f = 0.73 (hexane/AcOEt = 4/1); ^1H NMR (400 MHz, $CDCl_3$, δ): 7.17–7.12 (m, naphthalene H-5, 6, 7, 8, 4H), 5.81 (d, J = 2.8 Hz, furan H, 1H), 5.66 (d, J = 2.8 Hz, furan H, 1H), 4.11 (q, J = 5.6 Hz, $-CHCH_3$, 1H), 2.88–2.72 (m, $ArCH_2-$, 2H), 2.24 (s, furan CH_3 , 3H), (2.16–2.07, 2.05–1.95, 1.90–1.79, 1.79–1.69 (m, $ArCH_2CH_2CH_2-$, 4H)); ^{13}C NMR (100 MHz, $CDCl_3$, δ): (157.13, 150.29 (furan C-2, 5)), (137.03, 136.67 (naphthalene C-4a, 8a)), (129.60, 128.94 (naphthalene C-5, 8)), (126.05, 125.34 (naphthalene C-6, 7)), (107.21, 105.64 (furan C-2, 5)), 38.77 (CH), (29.53, 28.98 (naphthalene C-2,4)), 20.57 (naphthalene C-3), 13.72 (CH_3); IR (neat) $\tilde{\nu}_{max}$: (cm^{-1}): 2920 (s), 2860 (m),

1570 (m), 1490 (m), 1450 (s), 1220 (s), 1020 (s), 790 (s), 750 (s); EIMS (70 eV) m/z (relative intensity): 212 (M^+ , 60), 197 (6), 184 (14), 169 (34), 141 (24), 130 (100); HRMS-EI (m/z): M^+ calcd for $C_{15}H_{16}O$, 212.1201; found, 212.1197.

2-(1-(6-Methoxy-2-naphthyl)ethyl)-5-methylfuran (33). Colorless prism (EtOH): mp 70–71 °C; TLC R_f = 0.57 (hexane/AcOEt = 4/1); ^1H NMR (300 MHz, $CDCl_3$, δ): 7.70–7.64 (m, naphthalene H-4, 8, 2H), 7.58 (d, J = 1.2 Hz, naphthalene H-1, 1H), 7.32 (d, J = 8.4, 1.8 Hz, naphthalene H-3, 1H), 7.13–7.08 (m, naphthalene H-5, 7, 2H), (5.93–5.91, 5.89–5.85 (m, furan H-3, 4, 2H)), 4.19 (q, J = 7.2 Hz, $-CHCH_3$, 1H), 3.90 (s, OCH_3 , 3H), 2.22 (d, J = 0.3 Hz, furan CH_3 , 3H), 1.63 (d, J = 7.2 Hz, $-CHCH_3$, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, δ): (157.13, 157.08 (naphthalene C-1, furan C-2)), 150.58 (furan C-5), 139.46 (naphthalene C-2), 133.21 (naphthalene C-4a), (129.02, 126.73, 126.37, 125.25 (naphthalene C-1, 3, 4, 8)), 128.88 (naphthalene C-8a), 118.51 (naphthalene C-7), (105.67, 105.57, 105.51 (furan C-3, 4, naphthalene C-5)), 55.29 (OCH_3), 39.29 ($-CHCH_3$), 20.70 ($-CHCH_3$), 13.69 (furan CH_3); IR (KBr) $\tilde{\nu}_{max}$: (cm^{-1}): 2960 (m), 1600 (s), 1480 (m), 1390 (m), 1265 (m), 1230 (s), 1160 (m), 1030 (s), 900 (m), 860 (m), 820 (m), 790 (s); EIMS (70 eV) m/z (relative intensity): 266 (M^+ , 49), 251 ($M - CH_3$, 100), 208 (10); HRMS-EI (m/z): M^+ calcd for $C_{18}H_{18}O_2$, 266.1307; found, 266.1306; Anal. Calcd for $C_{18}H_{18}O_2$: C, 81.17; H, 6.81. Found: C, 81.09; H, 6.59.

2-(1-(2-Methoxy-1-naphthyl)ethyl)-5-methylfuran (34). Colorless solid: mp 32–38 °C; TLC R_f = 0.57 (hexane/AcOEt = 4/1); ^1H NMR (300 MHz, $CDCl_3$, δ): 7.97 (d, J = 8.5 Hz, naphthalene H-8, 1H), 7.77–7.73 (m, naphthalene H-4, 5, 2H), 7.34–7.24 (m, naphthalene H-3, 6, 7, 2H), 5.93 (s, furan H, 1H), 5.87 (s, furan H, 1H), 5.19 (q, J = 7.1 Hz, $-CHCH_3$, 1H), 3.88 (s, OCH_3 , 3H), 2.16 (s, furan CH_3 , 3H), 1.73 (d, J = 7.3 Hz, $-CHCH_3$, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, δ): 157.31 (naphthalene C-2), 154.52 (furan C-2), 149.81 (furan C-5), 132.57 (naphthalene C-8a), 129.77 (naphthalene C-4a), (128.46, 128.44 (naphthalene C-4, 5)), 125.62 (naphthalene C-7), 125.08 (naphthalene C-1), 124.07 (naphthalene C-8), 123.00 (naphthalene C-6), 114.50 (naphthalene C-3), (105.76, 104.89 (furan C-3, 4)), 57.27 (OCH_3), 30.27 ($-CHCH_3$), 17.91 ($-CHCH_3$), 13.74 (furan CH_3); IR (neat) $\tilde{\nu}_{max}$: (cm^{-1}): 2975 (m), 2940 (m), 1620 (m), 1600 (m), 1560 (m), 1510 (m), 1460 (m), 1380 (m), 1300 (m), 1260 (m), 1250 (s), 1220 (m), 1090 (m), 1030 (m), 820 (m), 790 (m), 760 (m); EIMS (70 eV) m/z (relative intensity): 266 (M^+ , 88), 251 ($M - CH_3$, 100), 235 (9), 223 (11), 209 (9), 95 (30); HRMS-EI (m/z): M^+ calcd for $C_{18}H_{18}O_2$, 266.1307; found, 266.1310; Anal. Calcd for $C_{18}H_{18}O_2$: C, 81.17; H, 6.81. Found: C, 81.12; H, 6.85.

2-Methyl-5-(1-methyl-1,2,3,4-tetrahydro-1-naphthyl)furan (35). Colorless oil: bulb-to-bulb dist. 160 °C /0.35 Torr; TLC R_f = 0.77 (hexane/AcOEt = 4/1); ^1H NMR (400 MHz, CDCl_3 , δ): 7.22–7.88 (m, naphthalene H-5, 6, 7, 8, 4H), 5.79 (m, furan H-3, 1H), 5.69 (d, J = 2.9 Hz, furan H-4, 1H), 2.88–2.74 (m, ArCH_2 -, 2H), 2.34–2.24, 1.88–1.71 (m, 1H and m, 3H, $\text{ArCH}_2\text{CH}_2\text{CH}_2$ -), 2.22 (s, furan CH_3 , 3H), 1.50 (s, CCH_3 , 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 160.68 (furan C-5), 150.27 (furan C-2), 147.71 (naphthalene C-8a), 136.46 (naphthalene C-4a), 128.92 (naphthalene C-5), 128.14 (naphthalene C-8), 125.79 (naphthalene C-5), 125.50 (naphthalene C-6), (106.40, 105.34 (furan C-3, 4)), 39.90 (naphthalene C-1), 37.02 (naphthalene C-2), 30.29 (naphthalene C-4), 28.73 ($-\text{CCH}_3$), 19.78 (naphthalene C-3), 13.78 (furan CH_3); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2930 (s), 1560 (m), 1490 (s), 1450 (s), 1230 (s), 1030 (s), 790 (s), 770 (s), 750 (s); EIMS (70 eV) m/z (relative intensity): 226 (M^+ , 49), 211 ($\text{M} - \text{CH}_3$, 100), 144 (72), 129 (41); HRMS-EI (m/z): M^+ calcd for $\text{C}_{16}\text{H}_{18}\text{O}$, 226.1358; found, 226.1360.

2-Methyl-5-(1-(2-thenyl)ethyl)furan (36). Colorless oil: TLC R_f = 0.50 (hexane/ CHCl_3 = 7/2); ^1H NMR (400 MHz, CDCl_3 , δ): 7.15 (dd, J = 5.2, 1.2 Hz, thiophene H-5, 1H), 6.92 (dd, J = 4.8, 3.6 Hz, thiophene H-4, 1H), 6.85 (ddd, J = 3.6, 1.2, 1.2 Hz, thiophene H-3, 1H), 5.93 (d, J = 3.2 Hz, furan H-4 1H), 5.86 (dd, J = 2.8, 0.8 Hz, furan H-3 1H), 4.35 (q, J = 7.2 Hz, $-\text{CHCH}_3$, 1H), 2.25 (d, J = 0.8 Hz, furan CH_3 , 3H), 1.66 (d, J = 7.2 Hz, $-\text{CHCH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 156.18 (furan C-5), 150.67 (furan C-2), 147.93 (thiophene C-2), 126.36 (thiophene C-4), 123.55 (thiophene C-3), 123.25 (thiophene C-5), 105.75 (furan C-3), 105.28 (furan C-4), 34.70 ($-\text{CHCH}_3$), 21.62 ($-\text{CHCH}_3$), 13.71 (furan CH_3); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2980 (m), 1560 (m), 1460 (m), 1440 (m), 1220 (s), 1020 (s), 860 (m), 790 (s), 700 (s); EIMS (70 eV) m/z (relative intensity): 192 (M^+ , 40), 177 ($\text{M} - \text{CH}_3$, 100), 149 (6), 134 (6); HRMS-EI (m/z): M^+ calcd for $\text{C}_{11}\text{H}_{12}\text{OS}$, 192.0609; found, 192.0615.

Syntheses of substrate for Scheme 4.

1-(2-Furyl)ethanol (37).¹² To a solution of 2-acetylfuran (5.00 g, 45.42 mmol) in EtOH (45 mL) was added NaBH_4 (1.71 g, 45.42 mmol) at room temperature. The whole was stirred for 30 min, then concentrated at reduced pressure. The residue was dissolved in AcOEt (50 mL) and H_2O (50 mL), and extracted three times with AcOEt (50 mL). The organic layer was washed once with brine (100 mL), dried over MgSO_4 , filtered, and the filtrate was concentrated to afford colorless liquid, which was purified by bulb-to-bulb distillation (50 °C/0.8 Torr) to give a colorless liquid (4.15 g, 81%): TLC R_f =

0.85 (AcOEt/hexane = 1/1); ^1H NMR (300 MHz, CDCl_3 , δ): 7.36 (dd, $J = 1.8, 0.9$ Hz, furan H-5 1H), 6.32 (dd, $J = 3.3, 1.8$ Hz, furan H-4, 1H), 6.22 (dd, $J = 3.3, 0.9$ Hz, furan H-3, 1H), 4.86 (q, $J = 6.3$ Hz, $-\text{CHCH}_3$, 1H), 2.23 (br, OH, 1H), 1.53 (d, $J = 6.6$ Hz, $-\text{CHCH}_3$, 3H); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3460 (br), 2970 (m), 1500 (w), 1370 (w), 1330 (w), 1230 (w), 1150 (m), 1080 (m), 1010 (m), 930 (m), 880 (m), 810 (m), 750 (m); EIMS (70 eV) m/z (relative intensity): 112 (M^+ , 45), 95 (100); HRMS-EI (m/z): M^+ calcd for $\text{C}_{11}\text{H}_{12}\text{O}_2$, 176.0837; found, 176.0838.

Reaction Procedure for Scheme 4.

The reactions were carried out as the same procedure as entry 8 in Table 1.

Products for Scheme 4.

2-(1-(2-Furyl)ethyl)-5-methylfuran (38).¹³ Colorless liquid: TLC $R_f = 0.59$ (hexane/ $\text{CHCl}_3 = 4/1$); ^1H NMR (400 MHz, CDCl_3 , δ): 7.32 (dd, $J = 1.6, 0.8$ Hz, side chain furan H-5, 1H), 6.29 (dd, $J = 3.2, 2.0$ Hz, side chain furan H-4, 1H), 6.04 (ddd, $J = 3.2, 0.8, 0.8$ Hz, side chain furan H-3, 1H), 5.98 (d, $J = 2.8$ Hz, furan H-4, 1H), 5.87 (d, $J = 2.8, 1.2$ Hz, furan H-4, 1H), 4.15 (q, $J = 7.2$ Hz, $-\text{CHCH}_3$, 1H), 2.25 (d, $J = 0.8$ Hz, furan CH_3 , 3H), 1.57 (d, $J = 7.2$ Hz, $-\text{CHCH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 156.64 (side chain, furan C-2), 154.47 (furan C-2), 150.61 (furan C-5), 141.01 (side chain, furan C-5), 109.95 (side chain, furan C-4), (105.82, 105.43, 104.69 (furan C-3, 4 and side chain, furan C-3), 33.21 ($-\text{CHCH}_3$), 18.20 ($-\text{CHCH}_3$), 13.70 (furan CH_3); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2970 (m), 1570 (m), 1510 (m), 1460 (m), 1220 (m), 1160 (m), 13030 (m), 1020 (m), 840 (m), 820 (m), 790 (m), 740 (s); EIMS (70 eV) m/z (relative intensity): 176 (M^+ , 37), 161 ($\text{M} - \text{CH}_3$, 100); HRMS-EI (m/z): M^+ calcd for $\text{C}_{11}\text{H}_{12}\text{O}_2$, 176.0837; found, 176.0838.

2-(1-(5-(1-(2-furyl)ethyl)-2-furyl)ethyl)-5-methylfuran, diastereomixture (39). Colorless oil: TLC $R_f = 0.44$ (hexane/ $\text{CHCl}_3 = 4/1$); ^1H NMR (400 MHz, CDCl_3 , δ): 7.32 (dd, $J = 2.0, 0.8$ Hz, furan H-5'', 1H), 6.29 (dd, $J = 3.2, 2.0$ Hz, furan H-4'', 1H), 6.01 (dd, $J = 3.2, 0.4$ Hz, furan H-3'', 1H), (5.94–5.91 (m, 2H), 5.88–5.84 (m, 2 H), furan H-3, 4, 3', 4'), 4.17 (q, $J = 7.2$ Hz, $-\text{CHCH}_3$, 1H), 4.11 (q, $J = 7.2$ Hz, $-\text{CHCH}_3$, 1H), 2.25 (s, furan CH_3 , 3H), 1.57 (d, $J = 7.2$ Hz, $-\text{CHCH}_3$, 3H), 1.54 (d, $J = 7.2$ Hz, $-\text{CHCH}_3$,

3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 156.76 (furan C-2'' minor), 156.66 (furan C-2''), 155.47 (furan C-2), 154.97 (furan C-5'), 154.78 (furan C-2'), 154.59 (furan C-2' minor), 150.69 (furan C-5 minor), 150.53 (furan C-5), 141.10 (furan C-5'' minor), 141.04 (furan C-5''), 110.01 (furan C-4''), 105.87 (furan C-4 minor), 105.82 (furan C-4), 105.49 (furan C-3 minor), 105.43 (furan C-3), 105.34 (furan C-4'), 105.24 (furan C-3'), 104.83 (furan C-3''), 104.73 (furan C-3'' minor), 33.20 ($-\underline{\text{CHCH}}_3 \times 2$), 33.15 ($-\underline{\text{CHCH}}_3$, minor), (18.24, 18.18, 18.14, 18.07 ($-(\underline{\text{CHCH}}_3 \times 2)$), 13.57 (furan CH_3); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2960 (m), 1560 (m), 1450 (m), 1220 (m), 1005 (m), 790 (s), 740 (m); EIMS (70 eV) m/z (relative intensity): 270 (M^+ , 54), 255 ($\text{M} - \text{CH}_3$, 100), 240 (16), 175 (10); HRMS-EI (m/z): M^+ calcd for $\text{C}_{17}\text{H}_{18}\text{O}_3$, 270.1256; found, 270.1254.

2-(1-(5-(1-(5-(1-(2-furyl)ethyl)-2-furyl)ethyl)-2-furyl)ethyl)-5-methylfuran, diastereomixture (**40**). Colorless oil: TLC R_f = 0.33 (hexane/ CHCl_3 = 4/1); ^1H NMR (400 MHz, CDCl_3 , δ): 7.32 (dd, J = 2.0, 0.8 Hz, furan H-5'''), 6.29 (dd, J = 3.6, 2.0 Hz, furan H-4'', 1H), 6.01 (d, J = 3.2 Hz, furan H-3'', 1H), 5.94–5.84 (m, furan H-3, 4, 3'', 4'', 3''', 4''', 6H), 4.20–4.07 (m, $-\underline{\text{CHCH}}_3$, 3H), 2.25 (s, furan CH_3 , 3H), 1.57–1.52 (m, $-\underline{\text{CHCH}}_3$, 9H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 156.66 (furan C-2), (155.48 (minor), 155.35, 155.35, 155.16, 154.97, 154.83, 154.78 (minor), (furan C-2, 2', 5', 2'', 5'')), 150.54 (furan C-5), 141.06 (furan C-5'''), 110.03 (furan C-4'''), 105.82, 105.44, 105.33, 105.33, 105.29, 105.24 (minor), 105.21 (furan C-3, 4, 3', 4', 3'', 4''), 104.83 (furan C-3'''), 33.21 ($-\underline{\text{CHCH}}_3$), 33.21 ($-\underline{\text{CHCH}}_3$), 33.21 ($-\underline{\text{CHCH}}_3$), (18.27, 18.24, 18.17, 18.14, 18.12, 18.12, 18.12 ($-\underline{\text{CHCH}}_3 \times 2$), 13.60 (furan CH_3); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2980 (m), 1560 (m), 1540 (m), 1510 (w), 1460 (m), 1380 (w), 1295 (w), 1220 (m), 1180 (w), 1060 (w), 1020 (m), 960 (w), 920 (w), 790 (s), 720 (m); EIMS (70 eV) m/z (relative intensity): 364 (M^+ , 80), 349 ($\text{M} - \text{CH}_3$, 100), 319 (10), 296 (8), 282 (11), 269 (15), 175 (13), 167 (16), 95 (12); HRMS-EI (m/z): M^+ calcd for $\text{C}_{23}\text{H}_{24}\text{O}_4$, 364.1675; found, 364.1677.

Substrate for Table 8.

N-Propyl-*p*-toluenesulfonamide (44).¹⁴ Colorless oil: ^1H NMR (400 MHz, CDCl_3 , δ): 7.76 (d, J = 8.4 Hz, phenyl H-2, 6, 2H), 7.30 (d, J = 7.8 Hz, phenyl H-3, 5, 2H), 4.77 (br, NH, 1H), 2.89 (q, J = 6.8

Hz, -NHCH₂CH₂CH₃, 2H), 2.43 (s, PhCH₃, 3H), 1.48 (qt, $J = 7.2$, 7.2 Hz, -NHCH₂CH₂CH₃, 3H), 0.86 (t, $J = 7.5$ Hz, -NHCH₂CH₂CH₃, 3H); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 3270 (m), 2960 (m), 1600 (m), 1460 (m), 1420 (m), 1320 (s), 1160 (s), 1095 (m), 820 (m) EIMS (70 eV) m/z (relative intensity): 213 (M⁺, 19), 184 (83), 155 (100), 91 (66).

Reaction Procedure for Table 8.

The reactions were carried out as same procedure as entry 8 in Table 1.

Products for Table 8.

***N*-1-(2-Naphthyl)ethyl-*p*-toluenesulfonamide (45).**¹⁵ Colorless prism: mp 148–149 °C; $R_f = 0.69$ (hexane/AcOEt = 2/3); ¹H NMR (400 MHz, CDCl₃, δ): 7.74–7.70 (m, naphthalene H 1H), 7.65–7.61 (m, naphthalene H 2H), 7.57 (d, $J = 8.3$ Hz, phenyl H-2, 6, 2H), 7.46 (s, naphthalene H-1, 1H), 7.44–7.39 (m, naphthalene H 2H), 7.21 (dd, $J = 8.5$, 1.7 Hz, naphthalene H-3, 1H), 6.98 (d, $J = 7.8$ Hz, phenyl H-3, 5, 2H), 5.42 (br, NH, 1H), 4.62 (qd, $J = 6.8$, 6.8 Hz, -CHCH₃, 1H), 2.20 (s, tolueneCH₃, 3H), 1.48 (d, $J = 7.1$ Hz, -CHCH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): 142.80 (phenyl C-4), 138.95 (phenyl C-1), 137.44 (naphthalene C-2), 132.89 (naphthalene C-8a), 132.48 (naphthalene C-4a), 129.06 (phenyl C-2, 6), (128.21, 127.66, 127.30, 125.71 (naphthalene C-4, 5, 6, 7, 8)), 126.89 (phenyl C-3, 5), 124.90 (naphthalene C-1), 123.96 (naphthalene C-3), 53.87 (-CHCH₃), 23.52 (-CHCH₃), 21.37 (ArCH₃); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 3420 (s), 1600 (w), 1430 (m), 1320 (SO₂, s), 1310 (m), 1300 (m), 1150 (SO₂, s), 1090 (s), 970 (m), 940 (m), 810 (m), 750 (s), 650 (s); EIMS (70 eV) m/z (relative intensity): 325 (M⁺, 63), 310 (M⁺, 83), 169 (M – CH₃C₆H₄SO₂H, 100), 155 (55), 127 (21), 91 (60); Anal. Calcd for C₁₉H₁₉NO₂S: C, 70.12; H, 5.88; N, 4.30. Found: C, 70.08; H, 5.89; N, 4.25.

***N*-(1-Phenyl)ethyl-*p*-toluenesulfonamide (46).**¹⁶ Colorless needle: mp 82–83 °C; $R_f = 0.51$ (hexane/AcOEt = 1/4); ¹H NMR (300 MHz, CDCl₃, δ): 7.62 (d, $J = 8.4$ Hz, toluene H-3, 5, 2H), 7.58–6.90 (m, phenyl, toluene H-2, 6, 7H), 5.13 (br, NH, 1H), 4.45 (qd, $J = 6.9$, 6.9 Hz, -CHCH₃, 1H), 2.37 (s, tolueneCH₃, 3H), 1.41 (d, $J = 6.9$ Hz, -CHCH₃, 3H); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 3240 (s), 2960 (m), 1600 (m), 1490 (m), 1450 (m), 1430 (m), 1320 (s), 1300 (m), 1290 (m), 1160 (s), 1120 (m), 1080 (m), 1020 (m), 960 (m), 820 (s), 770 (m), 670 (m); EIMS (70 eV) m/z (relative intensity): 275 (M⁺, 2), 260 (M – CH₃, 100), 155 (52), 120 (53), 91 (64).

***N*-(1-Phenyl)ethyl-*N*-propyl-*p*-toluenesulfonamide (47).** Colorless needle: mp 76–78 °C; R_f = 0.49 (hexane/AcOEt = 1/1); ^1H NMR (400 MHz, CDCl_3 , δ): 7.74 (d, J = 8.4 Hz, toluene H-3, 5, 2H), 7.30–7.21 (m, Ar H, 7H), 5.18 (q, J = 7.2 Hz, $-\text{CHCH}_3$, 1H), 3.01–2.90 (m, $-\text{NHCH}_2\text{CH}_2\text{CH}_3$, 2H), 2.42 (s, PhCH_3 , 3H), 1.39–1.37, 1.26–1.14 (m, $-\text{NHCH}_2\text{CH}_2\text{CH}_3$, 2H), 0.64 (t, J = 7.3 Hz, $-\text{NHCH}_2\text{CH}_2\text{CH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 142.68 (toluene C-4), 140.18 (phenyl C-1), 138.27 (toluene C-1), (129.39, 128.05, 127.32, 127.32, 126.89 (phenyl C-2, 6 and 3, 5, toluene C-2, 6 and 3, 5), 55.24 ($-\text{CHCH}_3$), 45.94 ($-\text{NCH}_2\text{CH}_2\text{CH}_3$), 24.06 ($-\text{NCH}_2\text{CH}_2\text{CH}_3$), 21.55 (Ar CH_3), 16.82 ($-\text{CHCH}_3$), 11.37 ($-\text{NCH}_2\text{CH}_2\text{CH}_3$); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2970 (m), 1605 (m), 1500 (m), 1460 (m), 1390 (m), 1340 (s), 1300 (m), 1310 (m), 1170 (s), 1150 (s), 1100 (m), 1060 (m), 955 (m), 980 (m), 830 (m), 740 (s), 700 (m), 660 (m); EIMS (70 eV) m/z (relative intensity): 317 (M^+ , 6), 302 ($\text{M} - \text{CH}_3$, 21), 288 (30), 184 (18), 155 (14), 105 (100), 91 (18); HRMS-EI (m/z): M^+ calcd for $\text{C}_{18}\text{H}_{23}\text{NO}_2\text{S}$, 317.1499; found, 317.1477.

***N*-1-(2-Naphthyl)ethylmethanesulfonamide (48).**¹⁷ Colorless prism: mp 137–138 °C; R_f = 0.074 (hexane/AcOEt = 1/1); ^1H NMR (300 MHz, CDCl_3 , δ): 7.93–7.12 (m, naphthalene H-1, 4, 5, 8, 4H), 7.53–7.45 (m, naphthalene H-3, 6, 7, 3H), 5.06 (d, J = 6.6 Hz, NH, 1H), 4.82 (qd, J = 7.0, 6.6 Hz, $-\text{CHCH}_3$, 1H), 2.60 (s, $-\text{SO}_2\text{CH}_3$, 3H), 1.02 (d, J = 7.0 Hz, $-\text{CHCH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 139.65 (naphthalene C-2), 133.23 (naphthalene C-8a), 132.93 (naphthalene C-4a), (129.02, 127.93, 127.69, 125.24, 126.55, 126.28, 123.81 (naphthalene C-1, 2, 3, 4, 5, 6, 7, 8)), 53.88 ($-\text{CHCH}_3$), 41.83 ($-\text{SO}_2\text{CH}_3$), 23.96 ($-\text{CHCH}_3$); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3280 (s), 1440 (m), 1330 (s), 1320 (s), 1300 (m), 1160 (s), 1150 (s), 1140 (m), 1120 (m), 1090 (m), 1020 (m), 990 (m), 840 (m), 770 (s); EIMS (70 eV) m/z (relative intensity): 249 (M^+ , 70), 234 ($\text{M} - \text{CH}_3$, 88), 169 (100), 155 (85), 127 (30); Anal. Calcd for $\text{C}_{13}\text{H}_{13}\text{NO}_2\text{S}$: C, 62.62; H, 6.06; N, 5.62. Found: C, 62.73; H, 6.06; N, 5.62.

***N*-(1-Phenyl)ethylmethanesulfonamide (49).**¹⁸ Colorless prism: R_f = 0.48 (hexane/AcOEt = 2/1); ^1H NMR (400 MHz, CDCl_3 , δ): 7.40–7.28 (m, Ar H, 5H), 4.80 (d, J = 6.8 Hz, NH, 1H), 4.66 (qd, J = 6.8, 6.8 Hz, $-\text{CHCH}_3$, 1H), 2.62 (s, $-\text{SO}_2\text{CH}_3$, 3H), 1.55 (d, J = 6.8 Hz, $-\text{CHCH}_3$, 3H); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3370 (s), 1460 (m), 1430 (m), 1320 (s), 1300 (m), 1150 (s), 1110 (m), 1090 (m), 1030 (m), 990 (m), 770 (s), 710 (s); EIMS (70 eV) m/z (relative intensity): 199 (M^+ , 2), 184 ($\text{M} - \text{CH}_3$, 100), 120 (50),

106 (53), 77 (14).

N-1-(2-Naphthyl)ethylbenzamide (50).¹⁹ Colorless fine prism: mp 147–148 °C; TLC R_f = 0.094 (hexane/AcOEt = 4/1); ^1H NMR (400 MHz, CDCl_3 , δ): 7.85–7.77 (m, Ar H, 6H), 7.52–7.60 (m, Ar H, 6H), 6.43 (d, J = 7.3 Hz, NH, 1H), 5.51 (qd, J = 7.3, 7.3 Hz, $-\text{CHCH}_3$, 1H), 1.70 (d, J = 7.1 Hz, $-\text{CHCH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): 166.16 (C=O), 140.10 (naphthalene C-2), 134.26, 133.04, 132.46 (naphthalene C-4a, 8a, phenyl C-1), 131.15 (phenyl C-4), (128.27, 128.59, 127.30, 125.94, 125.62, 124.33 (naphthalene C-1, 3, 4, 5, 6, 7, 8)), 128.24 (phenyl C-3, 5), 126.01 (phenyl C-2, 6), 49.17 ($-\text{CHCH}_3$), 21.58 ($-\text{CHCH}_3$); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3320 (s), 3050 (m), 2970 (w), 2930 (w), 1650 (s), 1640 (s), 1610 (m), 1580 (m), 1530 (s), 1500 (m), 1340 (s), 1320 (m), 1280 (m), 830 (s), 760 (s), 700 (s); EIMS (70 eV) m/z (relative intensity): 275 (M^+ , 51), 260 ($\text{M} - \text{CH}_3$, 15), 170 (41), 154 (26), 127 (10), 105 (100), 77 (30); HRMS-EI (m/z): M^+ calcd for $\text{C}_{19}\text{H}_{17}\text{NO}$, 275.1310; found, 275.1314; Anal. Calcd for $\text{C}_{19}\text{H}_{17}\text{NO}$: C, 82.88; H, 6.22; N, 5.09. Found: C, 82.94; H, 6.22; N, 5.10.

N-(1-Phenyl)ethylbenzamide (51).²⁰ Colorless needle: mp 119–120 °C; TLC R_f = 0.34 (hexane/AcOEt = 1/1); ^1H NMR (300 MHz, CDCl_3 , δ): 7.76 (d, J = 7.2 Hz, PhCO, H-2, 6, 2H), 7.51–7.23 (m, Ar H, 8H), 6.39 (br, NH, 1H), 5.33 (qd, J = 7.2, 7.2 Hz, $-\text{CHCH}_3$, 1H), 1.60 (d, J = 6.9 Hz, $-\text{CHCH}_3$, 3H); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3320 (m), 1620 (s), 1520 (s), 1320 (m), 760 (s), 700 (s); EIMS (70 eV) m/z (relative intensity): 225 (M^+ , 79), 210 (11), 105 (100), 77 (128).

Selected Reaction Procedure for Table 9.

Entry 1. To a suspension of $\text{La}(\text{OTf})_3$ (2.64 mg, 4.50 μmol) in CH_3NO_2 (1.80 mL), **1** (77.6 mg, 450 μmol) was added at room temperature. The whole mixture was stirred at 100 °C for 1 h. The solvent was evaporated under reduced pressure, and the residue was purified by preparative TLC (silica gel, AcOEt/hexane = 20/1) to afford **53** (59.6 mg, 81%).

Entry 4. The reactions were carried out as the same procedure as entry 8 in Table 1.

Products for Table 9.

Bis 1-(2-naphthyl)ethyl ether, diastereomixture (53).²¹ Colorless viscous oil: TLC R_f = 0.62

(cyclohexane/dichloromethane = 1/1), 0.65 (AcOEt/hexane = 5/1); ^1H NMR (400 MHz, CDCl_3 , δ): for the major isomer (*dl*) 7.89–7.83 (m, Ar H, 6H), 7.65 (s, naphthalene H-1, 2H), 7.52–7.41 (m, Ar H, 6H), 4.45 (q, $J = 6.6$ Hz, $-\text{CHCH}_3$, 1H), 1.48 (d, $J = 6.6$ Hz, $-\text{CHCH}_3$, 3H) for the minor isomer (*meso*) 7.89–7.73 (m, Ar H, 8H), 7.52–7.41 (m, Ar H, 6H), 4.74 (q, $J = 6.3$ Hz, $-\text{CHCH}_3$, 1H), 1.58 (d, $J = 6.3$ Hz, $-\text{CHCH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): for the major isomer 141.32 (naphthalene C-2), 133.19 (naphthalene C-8a), 132.93 (naphthalene C-4a), (128.29, 127.70, 127.60, 125.95, 125.59, 125.12, 124.19 (naphthalene C-1, 3, 4, 5, 6, 7, 8)), 74.84 ($-\text{CHCH}_3$), 24.72 ($-\text{CHCH}_3$) for the minor isomer 141.47 (naphthalene C-2), 133.12 (naphthalene C-8a), 132.73 (naphthalene C-4a), (127.91, 127.72, 127.47, 125.76, 125.44, 124.75, 124.43 (naphthalene C-1, 3, 4, 5, 6, 7, 8)), 74.66 ($-\text{CHCH}_3$), 23.10 ($-\text{CHCH}_3$); IR (film) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2960 (m), 1600 (m), 1440 (m), 1370 (m), 1310 (m), 1090 (s), 860 (m), 820 (s), 750 (s); EIMS (70 eV) m/z (relative intensity): 326 (M^+ , 1), 172 (3), 154 (100), 128 (7), 115 (3), 76 (15); Anal. Calcd for $\text{C}_{24}\text{H}_{22}\text{O}$: C, 88.31; H, 6.79. Found: C, 88.23; H, 6.80; GC analysis. The analytical conditions were as follows; injector temperature: 260 $^{\circ}\text{C}$, detector temperature: 260 $^{\circ}\text{C}$, column temperature control: initial temperature (100 $^{\circ}\text{C}$, 2 min), 10 $^{\circ}\text{C}$ /min, final temperature (260 $^{\circ}\text{C}$, 4 min). The retention times were as follows; 24.82 min (*meso*-**53**, minor), 20.36 min. (*dl*-**53**, major)

Bis (1-phenyl)ethyl ether, diastereomixture (**54**).²² Colorless oil: TLC $R_f = 0.72$ (AcOEt/hexane = 1/1); for the major isomer (*dl*) ^1H NMR (300 MHz, CDCl_3 , δ): 7.37–7.19 (m, Ar H, 10H), 4.24 (q, $J = 6.6$ Hz, $-\text{CHCH}_3$, 1H), 1.66 (d, $J = 6.6$ Hz, $-\text{CHCH}_3$, 3H) for the minor isomer (*meso*) 7.37–7.19 (m, Ar H, 10H), 4.53 (q, $J = 6.3$ Hz, $-\text{CHCH}_3$, 1H), 1.46 (d, $J = 6.3$ Hz, $-\text{CHCH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): for the major isomer 143.99 (phenyl C-1), 128.29 (phenyl C-3, 5), 127.22 (phenyl C-4), 126.17 (phenyl C-2, 6), 74.65 ($-\text{CHCH}_3$), 24.80 ($-\text{CHCH}_3$) for the minor isomer 144.07 (phenyl C-1), 128.07 (phenyl C-3, 5), 126.97 (phenyl C-2, 6), 126.08 (phenyl C-4), 74.44 ($-\text{CHCH}_3$), 23.13 ($-\text{CHCH}_3$); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2960 (m), 1490 (m), 1450 (m), 1370 (m), 1210 (m), 1090 (s), 1030 (m), 950 (m), 770 (s), 700 (s); EIMS (70 eV) m/z (relative intensity): 226 (M^+ , 0.5), 211 (22), 121 (65), 105 (100); GC analysis. The analytical conditions were as follows; injector temperature: 260 $^{\circ}\text{C}$, detector temperature: 260 $^{\circ}\text{C}$, column temperature control: initial temperature (150 $^{\circ}\text{C}$, 2 min), 10 $^{\circ}\text{C}$ /min, final temperature (260 $^{\circ}\text{C}$, 7 min). The retention times were as follows; 7.62 min (*dl*-**54**), 7.76 min. (*meso*-**54**)

Bis (1, 2, 3, 4-tetrahydro-1-naphthyl) ether, diastereomixture (**55**).²³ Colorless prism: mp 40–49 $^{\circ}\text{C}$; TLC $R_f = 0.58$ (AcOEt/hexane = 10/1); ^1H NMR (400 MHz, CDCl_3 , δ): for the major isomer (7.39–7.36, 7.30–7.27, 7.17–7.04 (m, Ar H, 8H)), 4.71–4.65 (m, ArCH-, 2H), 2.88–2.80, 2.77–2.67 (m, ArCH₂-,

2H), 2.21–1.97, 1.87–1.72 (m, ArCHCH₂CH₂-, 8H); ¹³C NMR (100 MHz, CDCl₃, δ): for the major isomer (137.35, 137.14 (naphthalene C-4a, 8a)), (129.38, 128.63, 127.05, 127.70 (naphthalene C-5, 6, 7, 8)), 72.77 (naphthalene C-1, CH), 29.44 (naphthalene C-2, CH₂), 28.59 (naphthalene C-4, CH₂), 19.09 (naphthalene C-3, CH₂) for the minor isomer (137.47, 137.39 (naphthalene C-4a, 8a)), (128.87, 128.74, 127.15, 125.45 (naphthalene C-5, 6, 7, 8)), 73.83 (naphthalene C-1, CH), 29.87, 29.44 (naphthalene C-2, CH₂), 29.14 (naphthalene C-4, CH₂), 19.15 (naphthalene C-3, CH₂); IR (KBr) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 2930 (m), 1490 (m), 1450 (m), 1440 (m), 1340 (m), 1310 (m), 1070 (s), 1010 (m), 1005 (m), 990 (m), 770 (m), 740 (s); EIMS (70 eV) *m/z* (relative intensity): 147 (M – 1,2,3,4-tetrahydro-1-naphthyl, 57), 131 (100); GC analysis. The analytical conditions were as follows; injector temperature: 260 °C, detector temperature: 260 °C, column temperature control: initial temperature (150 °C, 1 min), 10°C /min, final temperature (260 °C, 7 min). The retention times were as follows; 14.11 min (**55**, minor), 14.64 min. (**55**, major)

2-(1-Ethoxyethyl)naphthalene (56).²⁴ Colorless liquid: TLC *R_f* = 0.33 (AcOEt/hexane = 20/1); ¹H NMR (300 MHz, CDCl₃, δ): 7.85–7.80 (m, naphthalene H-4, 5, 8, 3H), 7.73 (s, naphthalene H-1, 1H), 7.50–7.42 (m, naphthalene H-3, 6, 7, 3H), 4.57 (q, *J* = 6.5 Hz, -CHCH₃, 1H), 3.39 (q, *J* = 7.0 Hz, -CH₂CH₃, 1H), 1.52 (d, *J* = 6.4 Hz, -CHCH₃, 3H), 1.20 (d, *J* = 7.0 Hz, -CH₂CH₃, 3H); ¹³C NMR (100 MHz, CDCl₃, δ): 141.48 (naphthalene C-2), (133.15, 132.84 (naphthalene C-4a, 8a), (128.13, 127.66, 127.54, 125.87, 125.50, 124.79, 124.06 (naphthalene C-1, 3, 4, 5, 6, 7, 8)), 77.85 (-CHCH₃), 64.01 (-OCH₂CH₃), 24.30 (-CHCH₃), 15.57 (-OCH₂CH₃); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 2970 (m), 2860 (m), 1600 (m), 1450 (m), 1380 (m), 1180 (m), 1130 (m), 1100 (s), 860 (m), 820 (m), 760 (m); EIMS (70 eV) *m/z* (relative intensity): 200 (M⁺, 56), 185 (M – CH₃, 100), 157 (56), 141 (12), 129 (64); HRMS-EI (*m/z*): M⁺ calcd for C₁₄H₁₆O, 200.1201; found, 200.1199.

Reaction Procedure for Table 10.

The reactions were carried out as the same procedure as entry 8 in Table 1.

Product for Table 10.

1-(2-Naphthyl)ethyl benzoate (59).²⁵ Colorless needle: TLC *R_f* = 0.32 (AcOEt/hexane = 20/1); ¹H NMR (300 MHz, CDCl₃, δ): 8.13–8.08 (m, phenyl H-2, 6, 2H), 7.89–7.79 (m, naphthalene H-1, 4, 5, 8, 4H), 7.59–7.41 (m, naphthalene H-3, 7, 6, 3H), 6.30 (q, *J* = 6.6 Hz, -CHCH₃, 1H), 1.76 (d, *J* = 6.6 Hz, -CHCH₃, 3H); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm⁻¹): 1725 (C=O, s), 1460 (m), 1270 (s), 1120 (s), 1080 (s), 720 (s);

EIMS (70 eV) m/z (relative intensity): 276 (M^+ , 66), 171 (9), 154 (88), 105 (100).

1-(2-Naphthyl)ethyl acetate (60).²⁶ Colorless oil: TLC R_f = 0.19 (AcOEt/hexane = 20/1); ^1H NMR (400 MHz, CDCl_3 , δ): 7.84–7.80 (m, naphthalene H-1, 4, 5, 8, 4H), 7.50–7.44 (m, naphthalene H-3, 5, 7, 3H), 6.05 (q, J = 6.6 Hz, $-\text{CHCH}_3$, 1H), 2.10 (s, COCH_3 , 3H), 1.62 (d, J = 6.8 Hz, $-\text{CHCH}_3$, 3H); IR (neat) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 2980 (m), 1740 (C=O, s), 1510 (m), 1370 (m), 1240 (s), 1180 (m), 1130 (m), 1070 (m), 1020 (m), 950 (m), 870 (m), 820 (m), 760 (m); EIMS (70 eV) m/z (relative intensity): 214 (M^+ , 15), 172 (28), 154 (100), 128 (9), 76 (14).

By-product of the reaction using 1.

1-Methyl-3-(2-naphthyl)-2,3-dihydro-1H-cyclopenta[b]naphthalene, diastereomixture (62). Colorless viscous oil: TLC R_f = 0.43 (AcOEt/hexane = 20/1); ^1H NMR (400 MHz, CDCl_3 , δ): for the major isomer 7.87–7.64 (m, Ar H, 6H), 7.56–7.20 (m, Ar H, 6H), 7.11 (t, J = 8.3 Hz, Ar H, 1H), 4.93 (dd, J = 8.1, 8.1 Hz, 2-naphthyl CH -, 1H), 3.42 (qt, J = 7.3, 7.3 Hz, $-\text{CHCH}_3$, 1H), 3.07 (ddd, J = 12.9, 8.5, 8.5 Hz, $-\text{CHCHHCH}$ -, 1H), 1.81 (ddd, J = 12.9, 7.6, 7.6 Hz, $-\text{CHCHHCH}$ -, 1H), 1.44 (d, J = 7.1 Hz, $-\text{CHCH}_3$, 3H) for the minor isomer 7.87–7.64 (m, Ar H, 6H), 7.56–7.20 (m, Ar H, 7H), 5.04 (dd, J = 8.8, 6.1 Hz, 2-naphthyl CH -, 1H), 3.61 (qt, J = 7.3, 7.3 Hz, $-\text{CHCH}_3$, 1H), 2.48 (ddd, J = 12.9, 10.3, 2.9 Hz, $-\text{CHCHHCH}$ -, 1H), 2.37 (ddd, J = 12.9, 8.5, 8.5 Hz, $-\text{CHCHHCH}$ -, 1H), 1.40 (d, J = 6.8 Hz, $-\text{CHCH}_3$, 3H); ^{13}C NMR (100 MHz, CDCl_3 , δ): for the major isomer (146.77, 144.61, 139.11, 133.51, 133.21, 132.05, 130.38 (cyclopenta[b]naphthalene C-3a, 4a, 9a, naphthalene C-2, 4a, 8a)), for the minor isomer (146.33, 143.23, 139.83, 133.41, 133.04, 132.03, 130.22 (cyclopenta[b]naphthalene C-3a, 4a, 9a, naphthalene C-2, 4a, 8a)), for the major and minor isomer 128.29 (Ar CH), 128.26 (Ar CH), 128.14 (Ar CH), 128.06 (Ar CH), 127.51 (Ar CH), 127.41 (Ar CH), 126.33 (Ar CH), 126.21 (Ar CH), 125.86 (Ar CH), 125.76 (Ar CH), 125.69 (Ar CH), 125.60 (Ar CH), 125.30 (Ar CH), 125.10 (Ar CH), 125.04 (Ar CH), 124.86 (Ar CH), 124.67 (Ar CH), 124.64 (Ar CH), 124.52 (Ar CH), for major isomer 121.96 (Ar CH), 50.52 (2-naphthyl CH -, 45.90 (CH_2), 39.53 ($-\text{CHCH}_3$), 21.32 ($-\text{CHCH}_3$), for the minor isomer 121.88 (Ar CH), 49.11 (2-naphthyl CH -, 45.57 (CH_2), 38.60 ($-\text{CHCH}_3$), 20.37 ($-\text{CHCH}_3$); IR (CHCl_3) $\tilde{\nu}_{\text{max}}$: (cm^{-1}): 3040 (s), 2930 (s), 2850 (s), 1630 (s), 1600 (s), 1510 (s), 1450 (s), 1370 (s), 1330 (m),

1270 (m), 1160 (m), 1130 (m), 1020 (m), 950 (m), 860 (s), 820 (s), 750 (s); EIMS (70 eV) m/z (relative intensity): 308 (100), 293 (26), 278 (10), 180 (43), 165 (35), 141 (18), 133; HRMS-EI (m/z): M^+ calcd for $C_{24}H_{20}$, 308.1565; found, 308.1560; Anal. Calcd for $C_{24}H_{20}$: C, 93.46; H, 6.54. Found: C, 93.08; H, 6.59.

GC-Analysis. An internal standard method was used for the quantitative analysis of the substrates and the products. Nitrocyclohexane was used as internal standard because of the miscibility for nitromethane. Three standard solutions were prepared with nitrocyclohexane, **4a**, **2a**, **54**, and **5a**: Sample A, nitrocyclohexane (50 mg), **4a** (50 mg), **2a** (50 mg), **54** (50 mg), and **5a** (50 mg); Sample B, nitrocyclohexane (50 mg), **4a** (100 mg), **2a** (100 mg), **54** (100 mg), and **5a** (100 mg); Sample C, nitrocyclohexane (50 mg), **4a** (150 mg), **2a** (150 mg), **54** (150 mg), and **5a** (150 mg). The analytical conditions were as follows; injector temperature: 260 °C, detector temperature: 260 °C, column temperature control: initial temperature (150 °C, 2 min), 10°C /min, final temperature (260 °C, 7 min). The retention times were as follows; **4a** (2.68 min), nitrocyclohexane (2.89 min), **54** (7.62 (*dl*), 7.76 (*meso*) min), **2a** (9.66 min), and **5a** (18.66 min). The reactions were carried out as the same procedure as entry 8 in Table 1 with 2–5 mmol scale. The analytical samples were taken (50–150 μ L) from the reaction mixture and filtered through a short pad of silica gel with AcOEt (1–4 mL).

Full details of the reaction of **2a** with **4a** (Figure 1) including Figure 2.

Sc(OTf)₃-catalyzed reaction.

Hf(OTf)₄-catalyzed reaction

TfOH (3 mol%)-catalyzed reaction.

TfOH (1 mol%)-catalyzed reaction

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