Electronic Supplementary Information

Tandem carbon-carbon bond insertion and intramolecular aldol reaction of benzyne with aroylacetones: Novel formation of 4,4'-disubstituted 1,1'-binaphthols

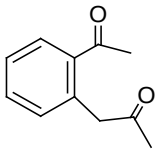
Kentaro Okuma*, Ryoichi Itoyama, Ayumi Sou, Noriyoshi Nagahora, and Kosei Shioji
Department of Chemistry, Fukuoka University, Jonan-ku, Fukuoka 814-0180, Japan

General: All chemicals were obtained from commercial suppliers and were used without further purification. Analytical TLC was carried out on precoated plates (Merck silica gel 60, F254) and flash column chromatography was performed with silica (Merck, 70-230 mesh). NMR spectra (^{1}H at 400 MHz; ^{13}C at 100 MHz) were recorded in $CDCl_3$, and chemical shifts are expressed in ppm relative to internal TMS for ^{1}H- and ^{13}C-NMR. Melting points were uncorrected.

Experimental

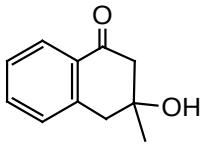
Reaction of triflate 1 with acetylacetone 2a at rt

To a solution of acetylacetone (45 mg, 0.45 mmol) and CsF (228 mg, 1.5 mmol) in acetonitrile (7 mL) was added triflate **1** (149 mg, 0.50 mmol). After being stirred for 15 h at rt, the reaction mixture was evaporated to give pale yellow oil, which was chromatographed over silica gel by elution with hexane dichloromethane (1:1) to afford a mixture of 2-benzoylmethylacetopheone and 2-acetylmethylbenzophenone. The mixture was subjected to gel permeation chromatography to give pure 1-(2-acetylphenyl)propane-2-one **3a** (41 mg, 0.23 mmol) and **4a** (9 mg, 0.05 mmol).



3a

Compound **3a**: colorless oil[1]: ^{1}H NMR ($CDCl_3$) δ = 2.29 (s, 3H, Me), 2.58 (s, 3H, Me), 4.02 (s, 2H, CH_2), 7.18 (d, 1H, *J* = 7.6 Hz, Ar), 7.38 (t, 1H, *J* = 7.6 Hz, Ar), 7.45 (t, 1H, *J* = 7.6 Hz, Ar), 7.97 (d, 1H, *J* = 7.6 Hz, Ar).

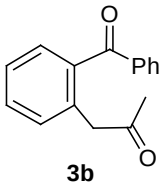


4a

Compound **4a**: Colorless oil: ^{1}H NMR ($CDCl_3$) δ = 1.44 (s, 3H, Me), 2.76 (d, 1H, *J* = 16.4 Hz, CHH), 2.83 (d, 1H, *J* = 16.4 Hz, CHH), 3.11 (d, 1H, *J* = 16.4 Hz, CHH), 3.15 (d, 1H, *J* = 16.4 Hz, CHH), 7.27 (d, 1H, *J* = 7.6 Hz, Ar), 7.34 (t, 1H, *J* = 7.6 Hz, Ar), 7.52 (t, 1H, *J* = 7.6 Hz, Ar), 8.04 (d, 1H, *J* = 7.6 Hz, Ar). ^{13}C NMR ($CDCl_3$) δ = 29.43 (Me), 43.73 (CH_2), 52.70 (CH_2), 72.10 (q-C), 127.20, 127.24,129.75, 131.81, 134.28,141.06 (Ar), 197.29 (C=O). HRMS (EI): m/z: Calcd for $C_{11}H_{12}O_2$;176.0837(M^+). Found: 176.0839 (M^+)

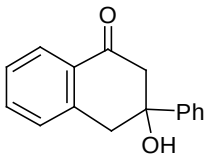
Reaction of triflate 1a with benzoylacetone 2b at rt

To a solution of benzoylacetone (73 mg, 0.45 mmol) and CsF (228 mg, 1.5 mmol) in acetonitrile (7 mL) was added triflate **1** (149 mg, 0.50 mmol). After being stirred for 20 h at rt, the reaction mixture was evaporated to give pale yellow oil, which was chromatographed over silica gel by elution with hexane dichloromethane (1:1) to afford a mixture of **3a** and 3-hydroxyl-3-phenyltetralone **4b**. The mixture was subjected to gel permeation chromatography to give pure **3b** (64 mg, 60%) and **4b** (16 mg, 15%).



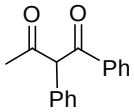
3b

Compound **3b**: colorless oil.[2] ^{1}H NMR ($CDCl_3$) δ = 2.19 (s, 3H, Me), 3.98 (s, 2H, CH_2), 7.28 (d, 1H, *J* = 8.0 Hz, Ar), 7.33 (t, 1H, *J* = 8.0 Hz, Ar), 7.41-7.51 (m, 4H, Ar), 7.58 (t, 1H, *J* = 8.0 Hz, Ar), 7.80 (d, 2H, *J* = 8.0 Hz, Ar). ^{13}C NMR ($CDCl_3$) δ = 30.10 (Me), 48.37 (CH_2), 126.59, 128.54, 130.55, 130.61, 131.31, 132.23, 133.12, 134.81, 138.09 (Ar), 198.46 (C=O), 205.76 (C=O).



4b

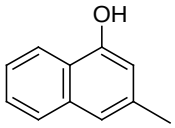
Compound **4b**: colorless oil; ^{1}H NMR ($CDCl_3$) δ = 3.04 (d, 1H, *J* = 16.0 Hz, C<u>H</u>H), 3.25 (d, 1H, *J* = 16.0 Hz, CH<u>H</u>), 3.31 (d, 1H, *J* = 16.0 Hz, CHH), 3.59 (d, 1H, *J* = 16.0 Hz, CHH), 7.28 (d, 1H, *J* = 8.0 Hz, Ar), 7.31-7.35 (m, 2H, Ar), 7.40 (t, 2H, *J* = 8.0 Hz, Ar), 7.51-7.56 (m, 3H, Ar), 8.06 (d, 1H, *J* = 8.0 Hz, Ar). ^{13}C NMR ($CDCl_3$) δ = 43.85 (Me), 51.78 (CH_2), 95.43 (q-C), 124.90, 127.14, 127.29, 128.11, 128.96, 129.66, 132.02, 134.38, 140.61, 145.82 (Ar), 196.98 (C=O). HRMS (EI): *m/z* Cacd for $C_{16}H_{14}O_2$ 238.0993 (M^+). Found 238.0990 (M^+).



1,2-diphenylbutane-1,3-dione mp 82-84 °C (lit.[3] mp 84-86 °C).

Reaction of triflate 1 with aceylacetone 2a in refluxing acetonitrile

To a solution of acetylacetone (45 mg, 0.45 mmol) and CsF (228 mg, 1.5 mmol) in acetonitrile (7 mL) was added triflate (149 mg, 0.50 mmol). After refluxing for 5 h, the reaction mixture was evaporated to give pale yellow oil, which was chromatographed over silica gel by elution with hexane dichloromethane (1:1) to afford 3-methyl-1-naphthol **5a**. (57 mg, 0.36 mmol)

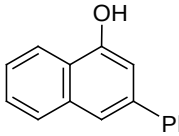


5a

Compound **5a**: colorless crystals; mp 154-155 °C (lit.[4] mp 155-156 °C).

Reaction of triflate 1a with benzoylacetone 2b in refluxing acetonitrile

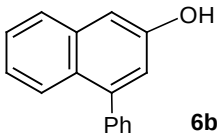
To a solution of benzoylacetone (73 mg, 0.45 mmol) and CsF (228 mg, 1.5 mmol) in acetonitrile (7 mL) was added triflate (149 mg, 0.50 mmol). After refluxing for 5 h at rt, the reaction mixture was evaporated to give pale yellow oil, which was chromatographed over silica gel by elution with hexane dichloromethane (1:1) to afford a mixture of 3-phenyl-1-naphthol **5b** and 4-phenyl-2-naphthol **6b**. The mixture was subjected to gel permeation chromatography to give pure 1-naphthol **5b** (15 mg, 0.07 mmol) and 2-naphthol **6b** (57 mg, 0.26 mmol).



5b

3-phenyl-1-naphthol **5b**: colorless needles, mp 96-97 °C (lit.[5] mp 97-98 °C). ^{1}H NMR

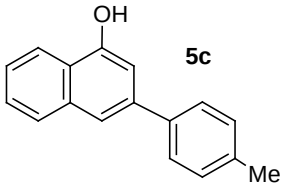
(CD_3CN) δ= 7.18 (s, 1H, Ar), 7.38(t, 1H, *J* =15.1 , Ar), 7.45-7.53 (m, 5H, Ar), 7.67 (s, 1H, Ar), 7.74 (d, 2H, *J* = 7.6, Ar), 7.88 (d, 1H, *J* = 7.7, Ar), 8.18 (d, 1H, *J* = 8.56, Ar). ^{13}C NMR ($CDCl_3$) δ = 108.76, 119.10, 121.77, 123.85, 125.69, 127.23, 127.60, 127.76, 128.36, 129.13, 135.25, 139.16, 141.13, 151.93 (Ar). MS(EI):calcd for $C_{16}H_{12}O$ *m/z*= 220 (M^+), found *m/z*= 220 (M^+).



6b

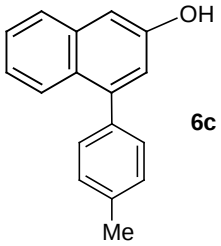
4-phenyl-2-naphthol **6b**: red oil.[6] ^1H NMR (CD_3CN) δ = 7.23 (s, 1H, Ar), 7.26 (t, 1H, *J* = 15.3, Ar), 7.41-7.48 (m, 3H, Ar), 7.50 (d, 2H, *J* = 4.1, Ph), 7.53-7.54 (m, 1H, Ar), 7.71 (d, 1H, *J* = 8.0, Ar), 7.77 (d, 1H, *J* = 8.0, Ar). ^{13}C NMR ($CDCl_3$) δ = 109.89, 119.14, 124.29, 126.48, 126.95, 127.34, 127.77, 127.87, 128.68, 130.28, 135.59, 140.37, 142.91, 152.76 (Ar).

Other reactions were carried out in a similar manner.



5c

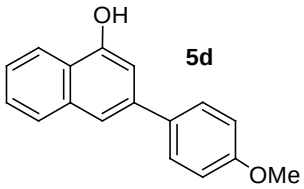
3-(4'-tolyl)-1-naphthol **5c:** colorless needles; mp 149-150 ^oC. ^1H NMR ($CDCl_3$) δ = 2.44 (s, 3H, CH_3), 5.46 (s, 1H, OH), 7.06(s, 1H, Ar), 7.25 (d, 2H, *J* = 7.3, Ar), 7.48 (m, 2H, Ar), 7.55 (d, 2H, *J* = 7.5, Ar), 7.62 (s, 1H, Ar), 7.82 (d, 1H, *J* = 8.0, Ar), 8.15 (d, 1H, *J* = 8.0, Ar). ^{13}C NMR ($CDCl_3$) δ = 21.38 (Me), 108.58, 118.66, 121.66, 123.63, 125.41, 127.09, 127.36, 128.20, 129.80, 135.22, 137.54, 138.21, 139.05, 151.87 (Ar). HRMS(EI): calcd for $C_{17}H_{15}O$ *m/z* 234.1044 (M^+). Found *m/z*= 234.1039 (M^+).



6c

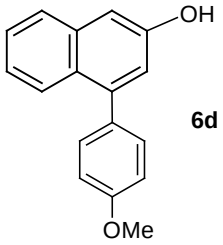
4-(4'-tolyl)-2-naphthol **6c:** brown oil. ^1H NMR ($CDCl_3$) δ = 2.45 (s, 3H, CH_3), 5.14 (s, 1H, OH),

7.05(s, 1H, Ar), 7.15 (s, 1H, Ar), 7.24 (t, *J* = 7.2 , Ar), 7.28 (d, 2H, *J* = 7.3, Ar), 7.35 (d, 2H, *J* = 7.3, Ar), 7.41 (t, 1H, *J* = 7.5, Ar), 7.71 (d, 1H, *J* = 7.8, Ar) , 7.80 (d, 1H, *J* = 7.8, Ar). HRMS(EI): calcd for $C_{17}H_{15}O$ *m/z*= 234.1044. Found *m/z*= 234.1048 (M^+).



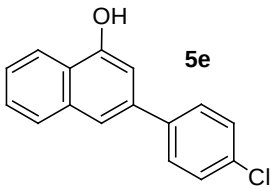
5d

3-(4'-methoxyphenyl)-1-naphthol **5d**: brown oil. ^{1}H NMR ($CDCl_3$) δ = 3.86 (s, 3H, CH_3), 7.04 (d, 2H, *J* = 8.8, Ar), 7.06 (s, 1H, Ar), 7.43-7.51(m, 2H, Ar), 7.58-7.62 (m, 1H, Ar), 7.82 (d, 1H, *J* = 7.9, Ar) , 7.15 (d, 1H, *J* = 7.9, Ar). HRMS(EI): Calcd for $C_{17}H_{14}O_2$; *m/z* = 250.0993 (M^+). Found *m/z*= 250.0986 (M^+).



6d

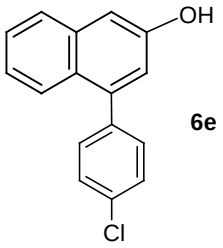
4-(4'-methoxyphenyl)-2-naphthol **6d**: purple oil. ^{1}H NMR ($CDCl_3$) δ = 3.89 (s, 3H, CH_3), 7.00 (d, 2H, *J* = 8.6, Ar), 7.04 (s, 1H, Ar), 7.15(s, 1H, Ar), 7.25 (t, 1H, *J* = 13.8, Ar), 7.38-7.43 (m, 3H, Ar), 7.71 (d, 1H, *J* = 7.4, Ar), 7.80 (d, 1H, *J* = 7.4, Ar). ^{13}C NMR ($CDCl_3$) δ = 55.59 (OMe), 109.08, 113.95, 118.26, 123.87, 126.35, 126.64, 127.00, 127.73, 131.20, 132.68, 135.43, 142.35, 152.92, 159.32 (Ar). MS(EI): Calcd for $C_{17}H_{14}O_2$; *m/z* =250.0993 (M^+). Found m/z = 250.0986 (M^+).



5e

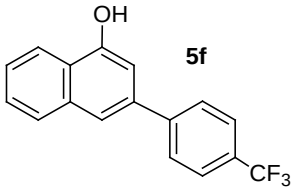
3-(4'-chlorophenyl)-1-naphthol **5e**: colorless needles; mp 160-161 °C. ^{1}H NMR ($CDCl_3$) δ = 5.55 (s, 1H, OH), 7.03 (d, 1H, *J* = 2.6, Ar), 7.42 (d, 2H, *J* = 7.5, Ar), 7.47-7.54 (m, 2H, Ar), 7.58 (m, 3H, Ar), 7.84 (d, 1H, *J* = 7.9, Ar), 8.16 (d, 1H, *J* = 7.9, Ar). ^{13}C NMR ($CDCl_3$) δ = 108.26,

118.92, 121.92, 123.88, 125.74, 129.31, 128.26, 128.94, 129.20, 133.76, 135.13, 137.85, 139.58, 152.11 (Ar). HRMS(EI): calcd for $C_{16}H_{11}ClO$ *m/z* 254.0498 (M^+). Found *m/z*= 254.0505 (M^+).



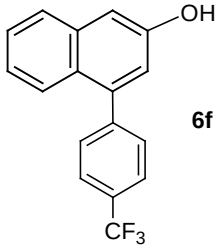
6e

4-(4'-chlorophenyl)-2-naphthol **6e**: pale purple oil; ^{1}H NMR ($CDCl_3$) δ = 5.08(s, 1H, OH), 7.03 (s, 1H, Ar), 7.18 (s, 1H, Ar), 7.27 (t, 1H, *J* = 7.3, Ar), 7.36-7.42 (m, 2H, Ar), 7.44-7.48 (m, 3H, Ar), 7.71-7.74 (d.d, 2H, *J* = 8.1, Ar). ^{13}C NMR ($CDCl_3$) δ = 109.76, 118.98, 124.19, 125.94, 126.85, 127.13, 128.75, 131.42, 132.62, 133.80, 135.41, 138.73, 141.30, 152.88. HRMS (EI): calcd for $C_{17}H_{14}ClO_2$ *m/z* = 254.0498 (M^+). Found; *m/z* = 254.0490 (M^+).



5f

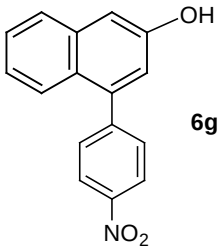
3-(4'-trifluromethylphenyl)-1-naphthol **5f**: colorless crystals; mp 107-108 ^{o}C. ^{1}H NMR ($CDCl_3$) δ = 5.69 (s, 1H, OH), 7.56-7.51 (m, 2H, Ar), 7.06 (s, 1H, Ar), 7.64 (s, 1H,Ar), 7.69 (d, 2H, Ar), 7.75 (d, 2H, *J* =8.0, Ar), 7.87(d, 1H, *J* =8.0 , Ar), 8.19 (d, 1H, *J* =12.0, Ar). ^{13}C NMR ($CDCl_3$) δ = 107.99, 119.23, 121.55, 123.97, 124.30 (CF_3, J_{C-F} = 270 Hz), 125.71, 125.80, 127.19, 127.52, 128.15, 129.69 (Ar, J_{C-F} = 32 Hz), 130.30, 134.85, 137.34, 144.44, 152.06 (Ar). HRMS(EI): Calcd for $C_{17}H_{11}F_3O$ *m/z* = 288.0762 (M^+). Found *m/z*= 288.0762 (M^+).



6f

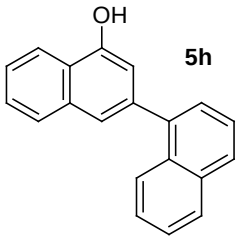
4-(4'-trifluoromethylphenyl)-2-naphthol **6f**: colorless crystals; mp 111-112 ^{o}C. ^{1}H NMR

($CDCl_3$) δ= 7.05 (s, 1H, Ar), 7.20 (s, 1H, Ar), 7.28 (t, 1H, *J* =8.0, Ar), 7.44 (t, 1H, *J* =8.0 , Ar), 7.58 (d, 2H, *J* =8.0, Ar), 7.69(d, 1H, *J* = 8.0, Ar), 7.75 -7.73 (m, 3H, Ar). ^{13}C NMR ($CDCl_3$) δ = 110.13, 119.03, 124.42, 124.49 (CF_3, J_{C-F} = 269 Hz), 125.34, 125.80, 125.84, 127.00, 127.19, 129.95 (Ar, J_{C-F} = 32 Hz), 130.47, 135.41, 141.10, 143.99, 152.79 (Ar). HRMS(EI): Calcd for $C_{17}H_{11}F_3O$ *m/z*= 288.0762. Found *m/z*= 288.0780.



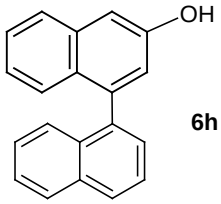
6g

4-(4'-nitrophenyl)-2-naphthol **6g**: Yellow crystals; mp 193-194^{o}C. ^{1}H NMR ($CDCl_3$) δ= 5.38(s, 1H, OH), 7.08 (s, 1H, Ar), 7.24 (s, 1H, Ar), 7.31 (t, 1H, *J* = 12.0, Ar), 7.47 (t, 1H, *J* = 12.0, Ar), 7.65 (d, 2H, *J* = 7.8, Ar), 7.76 (d, 1H, *J* =8.6 , Ar) , 8.34 (d, 2H, *J* = 7.8, Ar). ^{13}CNMR ($CDCl_3$) δ = 110.75, 119.24, 123.97, 124.79, 125.54, 126.94, 127.26, 127.41, 131.16, 135.55, 140.17, 147.30, 147.65, 152.95 (Ar). MS(ESI); Calcd for $C_{16}H_{11}NO_3$; m/z = 264.05 (M^+). Found; m/z = 265.02 (M^+).



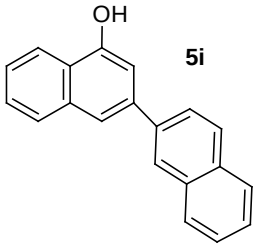
5h

3-(1'-naphthyl)-1-naphthol **5h**: colorless oily crystals, ^{1}H NMR ($CDCl_3$) δ = 5.43 (s, 1H, OH), 6.94 (s, 1H, Ar), 7.49 (d, 1H, *J* =8.0 , Ar), 7.54-7.42 (m, 6H, Ar), 7.96-7.83 (m, 1H, Ar), 8.24 (d, 1H, Ar). ^{13}C NMR ($CDCl_3$) δ = 111.54, 121.87, 121.97, 123.79, 125.66, 125.70, 126.11, 126.38, 127.19, 127.32, 128.05, 128.14, 128.58, 131.92, 134.05, 134.93, 138.77, 140.20, 151.27 (Ar). MS(EI): Calcd for $C_{20}H_{14}O$; *m/z* = 270.1044 (M^+). Found; 270.1047 (M^+).



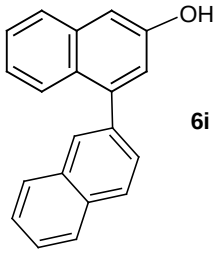
6h

4-(1'-naphhtyl)-2-naphthol **6h**: deep red oil. ^{1}H NMR ($CDCl_3$) δ = 5.14 (s, 1H, OH), 7.12-7.10 (m, 2H, Ar), 7.29-7.23 (m, 3H, Ar), 7.47-7.37 (m, 4H, Ar), 7.55 (t, 1H, *J* =8.0, Ar), 7.74(d, 1H, *J* =8.0, Ar), 7.93 (m 2H, *J* =8.0, Ar). HRMS(EI): Calcd for $C_{20}H_{14}O$ *m/z*= 270.1044. Found *m/z*= 270.1055 (M^+).



5i

3-(2'-naphthyl)-1-naphthol **5i**: orange needles, mp 107-108 ^{o}C. ^{1}H NMR ($CDCl_3$) δ = 5.71 (s, 1H, OH), 7.16 (s, 1H, Ar), 7.53-7.45 (m, 4H, Ar), 7.74 (s, 1H, Ar), 7.79 (d, 1H, *J* =8.0, Ar), 7.90-7.83 (m, 4H, Ar), 8.08(s, 1H, Ar), 8.19 (d, 1H, *J* =8.0, Ar). ^{13}C NMR ($CDCl_3$) δ = 108.81, 119.37, 121.81, 123.92, 125.71, 125.89, 126.26, 126.31, 126.64, 127.25, 127.96, 128.37, 128.52, 128.74, 132.98, 133.93, 135.28, 138.43, 138.99, 152.05. HRMS(EI): Calcd for $C_{20}H_{14}O$ *m/z*= 270.1044. Found *m/z*= 270.1050 (M^+).



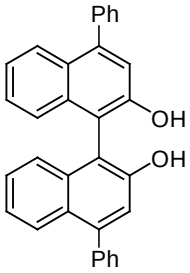
6i

4-(2'-naphthyl)-2-naphthol **6i**: red oil. ^{1}H NMR ($CDCl_3$) δ = 5.39 (s, 1H, Ar), 7.18 (s, 1H, Ar), 7.21 (s, 1H, Ar), 7.54 (t, 1H, *J* =8.0, Ar), 7.63-7.61 (m, 2H, Ar), 7.76 (d, 1H, Ar), 7.84 (d, 1H, *J* =8.0, Ar), 7.92 (d, 1H, *J* =8.0, Ar), 7.96-7.94 (m 2H, Ar). ^{13}C NMR ($CDCl_3$) δ = 108.80, 119.36, 121.81, 123.92, 125.71, 125.89, 126.26, 126.31, 126.64, 127.25, 127.95, 128.36, 128.52, 128.74, 132.97, 133.93, 135.27, 138.43, 138.99, 152.04 (Ar). HRMS(EI): Calcd for $C_{20}H_{14}O$ *m/z*=

270.1044. Found *m/z*= 270.1043 (M^+).

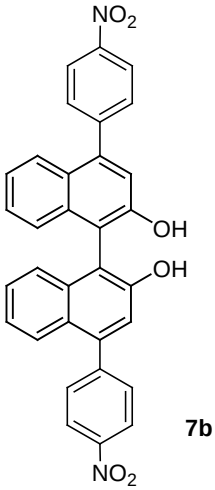
Oxidative coupling of 2-naphthol 6b.

To a suspension of V_2O_5 (9 mg, 0.05 mmol),and conc HCl (3 mL) in 1,2-dichloroethane (3 mL) was added 4-phenyl-2-naphthol **6b** (110 mg, 0.50 mmol) in one portion. After being stirred for 2 days at 70 ^oC, the reaction mixture was washed with water (10 mL x 2), dried over magnesium sulfate, filtered, and evaporated to give yellow solid, which was chromatographed over silica gel by elution with dichloromethane to afford pale yellow crystals of 4,4'-diphenyl-2,2'-binaphthol **7a** (72 mg, 0.16 mmol).



7a

Compound **7a**: yellow crystals mp 145-146 ^oC; ^1H NMR ($CDCl_3$) δ = 5.16 (br, 2H, OH), 7.28-7.32 (m, 6H, Ar), 7.36 (s, 2H, Ar), 7.48-7.54 (m, 2H, Ar), 7.59-7.61 (m, 4H, Ar), 7.53-7.63 (m, 4H, Ar), 7.93 (d, 2H, *J* = 8.8 Hz, Ar). ^{13}C NMR ($CDCl_3$), δ = 111.76, 119.13, 124.04, 125.04, 125.14, 126.27, 127.54, 128.23, 131.11, 134.16, 141.70, 146.80, 147.81 (Ar). Anal. Calcd for $C_{32}H_{22}O_2$ + 2EtOH; C, 81.48; H, 6.46. Found, 81.34; H, 6.65.



7b

Compound **7b**: yellow crystals. mp 235-236 ^{o}C; ^{1}H NMR ($CDCl_3$) δ = 5.19 (s, 2H, OH), 7.32-7.36 (m, 2H, Ar), 7.38-7.43 (m, 6H, Ar), 7.80-7.84 (m, 6H, Ar), 8.44-8.46 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$), δ = 111.71, 119.12, 124.04, 125.03, 125.15, 126.28, 127.55, 128.24, 131.11, 134.14, 141.73, 146.79, 147.82, 152.28 (Ar). Anal. Calcd for $C_{32}H_{20}N_2O_6$ + 2 H_2O; C, 68.08; H, 4.28; N, 4.96. Found: C, 68.00; H, 4.41, N, 4.94.

References

1) H. Yoshida, M. Watanabe, J. Ohshita, A. Kunai, *Chem. Commun*., **2005**, 3292-3294.
2) R. G. Sutherland, R. L. Chowdhury, A. Piorko, C. C. Lee, *Chem. Commun.*, **1985**, 1296-1297.
3) M. E. Lloris, R. A. Abramovitch, J. Marquet, M. Moreno-Mañas, *Tetrahedron Lett.*, **1992**, 48, 6909-6916.
4) H. R. Khavasi, N. Safari, *J. Mol. Cat. A*, **2004**, 220, 127-132.
5) R. Redic, G. B. Schuster, *J. Photochem. Photobio. A*, **2006**, 179, 66-74.
6) T. Jin, Y. Yamamoto, *Org. Lett*., **2007**, 9, 5259-5262.

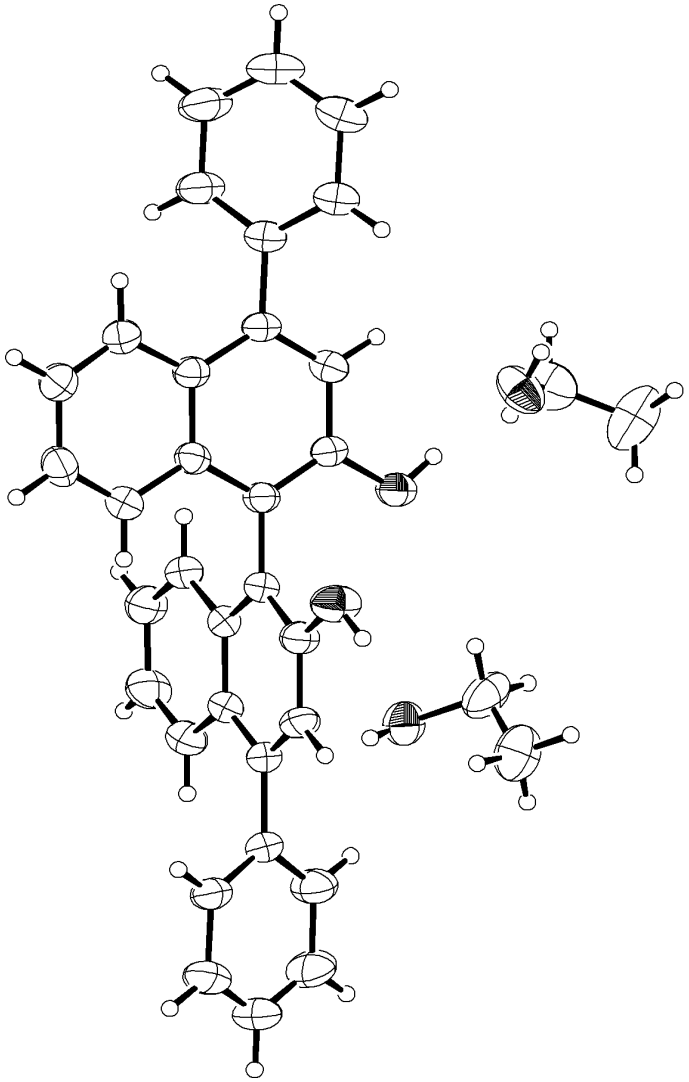


Figure S1. ORTEP drawing of 4,4'-diphenyl-1,1'-binaphthol **7a**.

Table 1. Crystal data and structure refinement for 4,4'-diphenyl-1,1'-binaphthol **7a**

Identification code	4,4'-diphenyl-1,1'-binaphthol	
Empirical formula	C36 H34 O4	
Formula weight	530.63	
Temperature	293(2) K	
Wavelength	0.71075 Å	
Crystal system	monoclinic	
Space group	$P2_1/a$	
Unit cell dimensions	a = 8.067(3) Å	α= 90°.
	b = 25.723(8) Å	β= 103.213(6)°.
	c = 14.556(5) Å	γ = 90°.
Volume	2940.5(16) $Å^3$	
Z	4	
Density (calculated)	1.199 Mg/m^3	
Absorption coefficient	0.077 mm^{-1}	
F(000)	1128	
Crystal size	0.50 x 0.20 x 0.15 mm^3	
Theta range for data collection	3.04 to 25.50°.	
Index ranges	-7<=h<=9, -30<=k<=31, -17<=l<=17	
Reflections collected	19698	
Independent reflections	5453 [R(int) = 0.0501]	
Completeness to theta = 25.50°	99.7 %	
Max. and min. transmission	0.9886 and 0.9626	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5453 / 0 / 395	
Goodness-of-fit on F^2	1.011	
Final R indices [I>2sigma(I)]	R1 = 0.0629, wR2 = 0.1538	
R indices (all data)	R1 = 0.1296, wR2 = 0.1888	

Largest diff. peak and hole 0.355 and -0.187 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for 111128. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	5539(3)	1182(1)	4420(2)	53(1)
C(2)	6238(3)	709(1)	4746(2)	56(1)
C(3)	6740(3)	605(1)	5721(2)	58(1)
C(4)	6584(3)	970(1)	6379(2)	54(1)
C(5)	5484(4)	1841(1)	6711(2)	67(1)
C(6)	4648(4)	2291(1)	6400(2)	78(1)
C(7)	4119(4)	2387(1)	5430(2)	78(1)
C(8)	4433(4)	2033(1)	4797(2)	66(1)
C(9)	5274(3)	1556(1)	5089(2)	53(1)
C(10)	5820(3)	1457(1)	6080(2)	53(1)
O(1)	6454(3)	345(1)	4097(1)	77(1)
C(11)	5063(4)	1289(1)	3383(2)	53(1)
C(12)	3486(4)	1155(1)	2854(2)	61(1)
C(13)	3019(4)	1257(1)	1873(2)	64(1)
C(14)	4129(4)	1483(1)	1412(2)	58(1)
C(15)	7025(4)	1859(1)	1503(2)	73(1)
C(16)	8591(4)	1997(1)	2018(2)	81(1)
C(17)	9034(4)	1908(1)	2993(2)	75(1)
C(18)	7905(4)	1671(1)	3428(2)	63(1)
C(19)	6246(3)	1525(1)	2920(2)	53(1)
C(20)	5795(4)	1620(1)	1927(2)	57(1)
O(2)	2370(3)	926(1)	3309(1)	85(1)
C(21)	7209(4)	832(1)	7399(2)	60(1)
C(22)	6694(4)	377(1)	7750(2)	72(1)
C(23)	7297(5)	240(2)	8697(2)	91(1)
C(24)	8421(5)	559(2)	9282(2)	99(1)

C(25)	8963(5)	1007(2)	8949(2)	99(1)
C(26)	8372(4)	1141(1)	8012(2)	81(1)
C(27)	3595(4)	1594(1)	379(2)	64(1)
C(28)	4222(4)	1324(1)	-282(2)	81(1)
C(29)	3740(5)	1444(2)	-1228(2)	95(1)
C(30)	2637(5)	1842(2)	-1528(2)	94(1)
C(31)	1994(5)	2117(1)	-896(2)	99(1)
C(32)	2443(5)	1989(1)	64(2)	81(1)
C(33)	8955(6)	-1215(2)	4131(3)	141(2)
C(34)	9188(5)	-752(2)	4692(3)	106(1)
O(3)	7743(3)	-539(1)	4876(2)	95(1)
C(35)	-2889(14)	125(6)	1653(8)	111(4)
C(36)	-1376(15)	215(7)	2260(9)	108(4)
O(4)	-632(6)	719(2)	2027(5)	87(3)
O(5)	-766(11)	489(5)	2760(10)	150(5)
C(37)	-1960(40)	-96(8)	1870(20)	227(13)
C(38)	-1510(70)	383(10)	1872(19)	370(30)

Table 3. Bond lengths [Å] and angles [°] for 111128.

C(1)-C(2)	1.378(3)	C(16)-C(17)	1.401(4)
C(1)-C(9)	1.420(3)	C(17)-C(18)	1.366(4)
C(1)-C(11)	1.496(3)	C(18)-C(19)	1.424(4)
C(2)-O(1)	1.369(3)	C(19)-C(20)	1.429(3)
C(2)-C(3)	1.410(3)	C(21)-C(22)	1.378(4)
C(3)-C(4)	1.367(3)	C(21)-C(26)	1.388(4)
C(4)-C(10)	1.421(4)	C(22)-C(23)	1.398(4)
C(4)-C(21)	1.497(3)	C(23)-C(24)	1.367(5)
C(5)-C(6)	1.363(4)	C(24)-C(25)	1.359(5)
C(5)-C(10)	1.416(4)	C(25)-C(26)	1.381(4)
C(6)-C(7)	1.399(4)	C(27)-C(28)	1.374(4)
C(7)-C(8)	1.360(4)	C(27)-C(32)	1.382(4)
C(8)-C(9)	1.419(4)	C(28)-C(29)	1.377(4)
C(9)-C(10)	1.432(3)	C(29)-C(30)	1.362(5)
C(11)-C(12)	1.372(4)	C(30)-C(31)	1.354(5)
C(11)-C(19)	1.423(4)	C(31)-C(32)	1.401(4)
C(12)-O(2)	1.366(3)	C(33)-C(34)	1.433(5)
C(12)-C(13)	1.416(4)	C(34)-O(3)	1.369(4)
C(13)-C(14)	1.365(4)	C(35)-C(36)	1.354(17)
C(14)-C(20)	1.425(4)	C(36)-O(4)	1.499(17)
C(14)-C(27)	1.494(4)	O(5)-C(38)	1.32(3)
C(15)-C(16)	1.360(4)	C(37)-C(38)	1.28(4)
C(15)-C(20)	1.423(4)		

C(2)-C(1)-C(9)	118.4(2)	C(3)-C(4)-C(10)	119.6(2)
C(2)-C(1)-C(11)	120.1(2)	C(3)-C(4)-C(21)	117.9(2)
C(9)-C(1)-C(11)	121.5(2)	C(10)-C(4)-C(21)	122.5(2)
O(1)-C(2)-C(1)	118.1(2)	C(6)-C(5)-C(10)	122.0(3)
O(1)-C(2)-C(3)	120.9(2)	C(5)-C(6)-C(7)	119.9(3)
C(1)-C(2)-C(3)	121.0(2)	C(8)-C(7)-C(6)	120.3(3)
C(4)-C(3)-C(2)	121.6(2)	C(7)-C(8)-C(9)	121.7(3)

C(8)-C(9)-C(1)	121.2(2)	C(25)-C(26)-C(21)	121.4(3)
C(8)-C(9)-C(10)	118.1(2)	C(28)-C(27)-C(32)	117.8(3)
C(1)-C(9)-C(10)	120.7(2)	C(28)-C(27)-C(14)	122.7(3)
C(5)-C(10)-C(4)	123.4(2)	C(32)-C(27)-C(14)	119.5(3)
C(5)-C(10)-C(9)	118.0(2)	C(27)-C(28)-C(29)	121.5(3)
C(4)-C(10)-C(9)	118.5(2)	C(30)-C(29)-C(28)	120.2(3)
C(12)-C(11)-C(19)	118.5(2)	C(31)-C(30)-C(29)	119.9(3)
C(12)-C(11)-C(1)	120.8(2)	C(30)-C(31)-C(32)	120.2(3)
C(19)-C(11)-C(1)	120.7(2)	C(27)-C(32)-C(31)	120.3(3)
O(2)-C(12)-C(11)	117.7(2)	O(3)-C(34)-C(33)	115.9(4)
O(2)-C(12)-C(13)	121.0(3)	C(35)-C(36)-O(4)	110.4(15)
C(11)-C(12)-C(13)	121.3(3)	C(37)-C(38)-O(5)	105(3)
C(14)-C(13)-C(12)	121.4(3)		
C(13)-C(14)-C(20)	119.5(2)		
C(13)-C(14)-C(27)	120.4(3)		
C(20)-C(14)-C(27)	120.1(3)		
C(16)-C(15)-C(20)	121.7(3)		
C(15)-C(16)-C(17)	120.3(3)		
C(18)-C(17)-C(16)	120.0(3)		
C(17)-C(18)-C(19)	121.7(3)		
C(11)-C(19)-C(18)	121.3(2)		
C(11)-C(19)-C(20)	120.6(3)		
C(18)-C(19)-C(20)	118.1(2)		
C(15)-C(20)-C(14)	123.1(3)		
C(15)-C(20)-C(19)	118.1(3)		
C(14)-C(20)-C(19)	118.7(2)		
C(22)-C(21)-C(26)	117.7(3)		
C(22)-C(21)-C(4)	120.4(3)		
C(26)-C(21)-C(4)	121.8(3)		
C(21)-C(22)-C(23)	120.9(3)		
C(24)-C(23)-C(22)	119.6(3)		
C(25)-C(24)-C(23)	120.6(3)		
C(24)-C(25)-C(26)	119.8(4)		

Symmetry transformations used to generate equivalent atoms: