Synthesis of Some Novel Amino Phenols with Octahydro-1,1′-binaphthyl Backbone†

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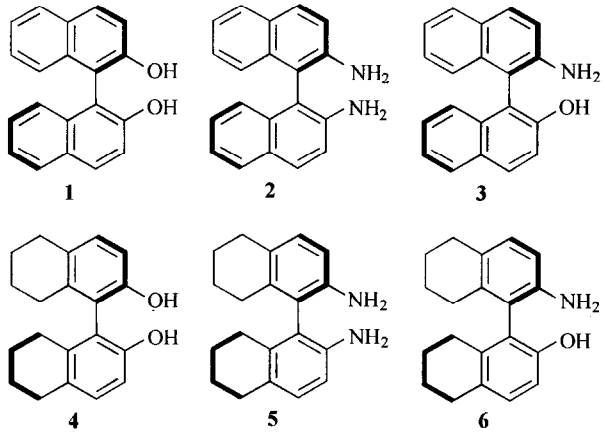
A series of new octahydro-1,1′-binaphthyl derivatives, namely (*R*)-(+)-2-(*N*, *N*-dialkylamino)-2′-hydroxy-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-binaphthyls (7,9), have been synthesized. Their asymmetric induction for enantioselective addition of Et_2Zn to benzaldehyde was examined and it was found that (*R*)-(+)-2-(*N*-cyclohexyl-*N*-methylamino)-2′-hydroxy-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-binaphthyl (9c) exhibited the best asymmetric induction among the ligands prepared, up to 55% *ee* of 1-phenylpropanol being obtained.

Keywords Biaryls, amino phenol, asymmetric catalysis, zinc, benzaldehyde

Recent research showed that the chiral catalysts derived from 5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-bi-2-naphthol (H_8-BINOL) (**4**) and 5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-bi-2-naphthylamine (H_8-BINAM) (**5**) exhibited higher efficiency and enantioselectivity for asymmetric reactions than those prepared from their parent ligands (*e.g.* **1** and **2**) did, due to the steric and electronic modulation in the binaphthyl backbone.[1,2] Enantiopure NOBIN (**3**) was a recently developed chiral inducer[3] and was found to be an excellent chiral ligand intermediate for asymmetric reactions.[4] Logically, its octahydrogenated form (H_8-NOBIN, **6**) should be an interesting compound for asymmetric induction as well. Based on a convenient procedure developed recently in our lab for the preparation of H_8-binaphthyls through the partial reduction of corresponding binaphthyls with commercially available Raney Ni-Al alloy in dilute aqueous alkaline solution, **6** could be obtained in good yield and excellent enantiomeric excess (>99%).[5] In the present work we will report our recent results on the synthesis of novel dialkyl H_8-NOBIN derivatives, (*R*)-(+)-2-(*N*, *N*-Dialkylamino)-2′-hydroxy-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-binaphthyls (**7** and **9**) and the primary screening for the application to asymmetric addition of diethylzinc to benzaldehyde.



Results and discussion

The methylation of **3** could be easily achieved through classical Eschweiler-Clarke reaction with CH_2O-

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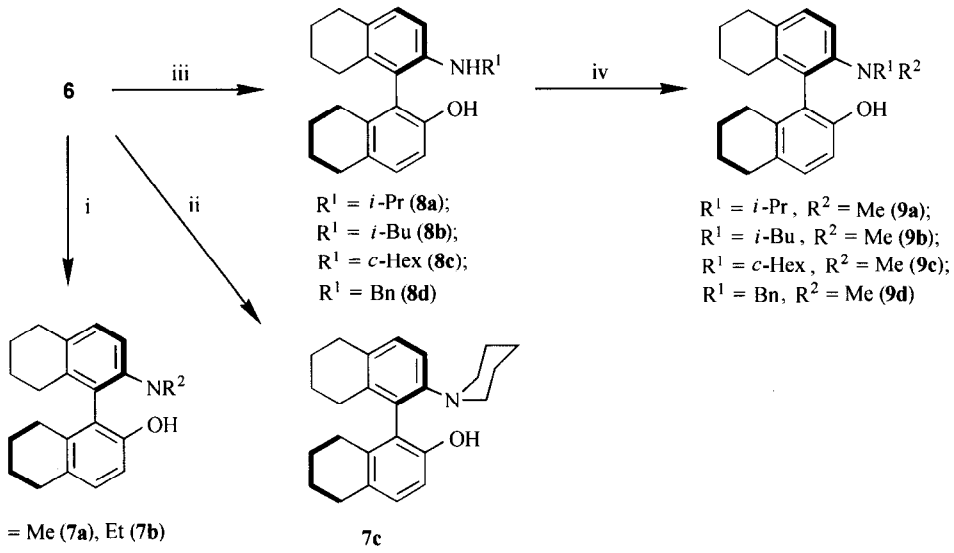
HCOOH.[3b] However under the same reaction condition, the methylation of corresponding octahydro derivative **6** proved to be hard, only 20% yield of expected product **7a** was obtained. Very recent reports showed that aldehyde-$NaBH_4$ or ketone-$NaBH_4$ and aldehyde-KBH_4 systems were effective for the alkylation of amine.[4e,6] Therefore, we applied this method to the synthesis of novel dialkyl H_8-NOBIN derivatives **7a—c**. As shown in Scheme 1, reductive alkylation of (*R*)-(+)-H_8-NOBIN (**6**) with formaldehyde, acetaldehyde and $NaBH_4$/H_2SO_4 in THF at room temperature resulted in the formation of corresponding *N*, *N*-dimethyl and diethyl-(*R*)-H_8-NOBIN **7a—b** in high yields (87% and 96%, respectively). In a similar manner, glutaric dialdehyde readily formed the corresponding piperidine derivative **7c** (86%). However, only monoalkylation occurred at nitrogen atom with a little bit more steric aldehydes. Accordingly, the alkylation with isobutyraldehyde and benzaldehyde resulted in the formation of **8b** and **8d**, respectively. In the case of ketones as the alkylation reagents, only monoalkylation was observed, which was consistent with the reaction of NOBIN. Thus, acetone and cyclohexanone furnished **8a** and **8c**, respectively. The monoalkylated H_8-NOBINs **8a-d** without purification were then submitted to further alkylation with CH_2O under the same condition to give **9a—d**, respectively in reasonable yields.

Scheme 1



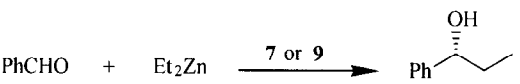
Reagents and conditions: i) HCHO or CH_3CHO, $NaBH_4$, H_2SO_4, THF, H_2O, r.t., 30 min, isolated yields for **7a**: 87%; **7b**: 96%; ii) $OHC(CH_2)_3CHO$, $NaBH_4$, H_2SO_4, THF, H_2O, r.t. 30 min, isolated yield for **7c**: 86%; iii) acetone, isobutyraldehyde, cyclohexanone or benzaldehyde, $NaBH_4$, H_2SO_4, THF, H_2O, r.t. 30 min; iv) HCHO, $NaBH_4$, H_2SO_4, THF, H_2O, r.t., 30 min, isolated yields for **9a**: 81%; **9b**: 75%; **9c**: 70%; **9d**: 35%.

With H_8-NOBIN derivatives (**7** and **9**) in hand, we then examined their asymmetric induction for enantioselective addition of diethylzinc to benzaldehyde. It was found that all these amino phenols (3 mol %) show low to medium asymmetric induction efficiency (18—55% *ee*) and the *R* ligand gives the product in *R* configuration exclusively (Table 1). The yields of the 1-phenylpropanol were from moderate to good (50—90%). A ligand with more steric bulk of the nitrogen substituent, *i.e.*, **9c**, exhibited the best asymmetric induction, up to 55% *ee* of product being obtained. It has been reported by Kocovsky[4e] that addition of a catalytic amount of *n*-BuLi (5 mol %) could significantly enhance the level of the catalytic efficiency and enantioselectivity in the same reaction with the derivatives of **3** as chiral ligands. However, in the present reaction system, we did not find such dramatic effect of *n*-BuLi on the reaction. For example, using **7a** as chiral inducer, 43% *ee* and 39% *ee* of the product were obtained with and without the presence of *n*-BuLi, respectively. The steric and

electronic modulation in the binaphthyl backbone by partial reduction of naphthyl rings in the present catalytic system was found to be unfavourable to the enantioselectivity of present reaction. The further transformation of **7** to the corresponding N, P and N, N or N, S ligands for the catalysis of other type of reactions is undergoing in the same laboratory.

In conclusion, we have demonstrated the synthesis of a series of new octahydro-1,1′-binaphthyl derivatives (*R*)-(+)-2-(*N*,*N*-Dialkylamino)-2′-hydroxy-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-binaphthyls (**7** and **9**). Their asymmetric induction for the enantioselective addition of Et_2Zn to benzaldehyde was primarily examined, of which **9c** exhibited the best asymmetric induction, up to 55% *ee* of product being obtained.

Table 1 Asymmetric induction of H_8-NOBIN derivatives **7** and **9**



Ligands	Yields (%)	*ee* (%)	Configuration
7a	80	43	*R*
7a[a]	54	39	*R*
7b	85	30	*R*
7c	85	18	*R*
9a	90	53	*R*
9b	78	29	*R*
9c	75	55	*R*
9c[a]	80	52	*R*
9d	50	34	*R*

[a] In the presence of 5 mol % BuLi.

Experimental

General consideration

Racemic NOBIN **3** was prepared by cross-coupling of 2-naphthol and 2-naphthylamine using $FeCl_3 \cdot 6H_2O$ as the oxidant in water.[3a] Its resolution and the reduction of NOBIN **3** to H_8-NOBIN **6** were carried out with the methods developed in the same lab recently.[3b,5] ^1H NMR and ^{13}C NMR spectra were taken on Bruker AM300 and Bruker DRX400 spectrometers at 25℃, respectively. Chemical shifts of ^1H NMR are expressed in ppm with tetramethylsilane as an internal standard ($\delta = 0$) in $CDCl_3$ and chemical shifts of ^{13}C NMR in ppm with residual signal of $CDCl_3$ as an internal standard ($\delta = 77$). Coupling constants are reported in hertz (Hz). IR spectra were recorded on a Bio Rad FTS-185 in KBr pellets. Mass spectra (EI, 70 eV) were taken on a HP5989A spectrometer. Melting points are uncorrected. $[\alpha]_D$ Values were measured on a PE 341 automatic polarimeter. The enantiomeric excesses were determined with HPLC-CD system.[7]

Preparation of dialkyl H_8-NOBIN *derivatives* **7** *and* **9**

(*R*)-(+)-2-(*N*, *N-Dimethylamino*)-2′-*hydroxy*-5,5′,6,6′,7,7′,8,8′-*octahydro*-1,1′-*binaphthyl* (**7a**)

To a solution of 40% aqueous formaldehyde (1.5 mL) and 20% aqueous H_2SO_4 (1.5 mL) in THF (5 mL) was simultaneously added a solution of (*R*)-(+)-H_8-NOBIN (**6**) (440 mg, 1.5 mmol, >99% *ee*) in THF (30 mL) and solid $NaBH_4$ (411 mg) slowly over a period of 30 min. The reaction mixture was stirred for additional 30 min and TLC check showed the disappearance of starting material. The reaction mixture was neutralized with 3% aqueous KOH (300 mL) and the resulting suspension was extracted with ethyl acetate (3 × 60 mL). The extract was washed with brine, dried over $MgSO_4$, and concentrated. The residue was submitted to column-chromatographic separation on silica gel with hexane/EtOAc (4:1) as eluent to give **7a** in 87% yield (420 mg) as amorphous solids: $[\alpha]_D^{15}$: +4.7 (*c* 0.5, THF). IR (KBr) ν: 3413, 2926, 2855, 1589, 1475, 1201, 815 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 7.11 (d, J = 8.2 Hz, 1H), 6.99—7.03 (m, 2H), 6.83 (d, J = 8.2 Hz, 1H), 4.24 (br, 1H), 2.73—2.77 (m, 4H), 2.55 (s, 6H), 2.14—2.24 (m, 4H), 1.63—1.74 (m, 8H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 150.23, 138.10, 136.25, 130.67, 130.00, 129.51, 129.16, 129.10, 118.86, 113.61, 112.60, 110.00, 46.87, 29.58, 29.24, 27.58, 27.40, 23.24, 23.21, 23.11, 22.88. *m/z* (%): 321 (M^+, 100). HRMS Calcd for $C_{22}H_{27}NO$: 321.2903. Found: 321.2703.

(*R*)-(+)-2-(*N*, *N-Diethylamino*)-2′-*hydroxy*-5,5′,6,6′,7,7′,8,8′-*octahydro*-1,1′-*binaphthyl* (**7b**)

Following the same procedure for the preparation of **7a** by using acetaldehyde instead of formaldehyde, **7b** was obtained in 96% yield: $[\alpha]_D^{15}$ + 20.8 (*c* 0.5, THF). IR (KBr pellet) ν: 3426, 2924, 2856, 1589,

1474, 1195, 813 cm^{-1}. ^1H NMR(300 MHz, $CDCl_3$) δ: 7.06(d, J = 8.2 Hz, 1H), 6.98(d, J = 8.2 Hz, 1H), 6.91(d, J = 8.2 Hz, 1H), 6.79(d, J = 8.2Hz, 1H), 2.74—2.90(m, 8H), 2.18—2.31(m, 4H), 1.54—1.75(m, 8H), 0.83(t, J = 7.0 Hz, 6H). ^{13}C NMR(100.6 MHz, $CDCl_3$) δ: 150.26, 137.75, 136.20, 129.80, 129.58, 129.32, 129.26, 129.10, 116.44, 116.40, 113.55, 113.51, 44.22, 44.15, 29.58, 29.28, 27.59, 27.25, 23.23, 23.16, 23.12, 22.86. m/z(%): 349(M^+, 40), 334(M^+ - CH_3, 100). HRMS Calcd for $C_{24}H_{31}NO$: 349.2406. Found: 349.2410.

(R)-(+)-2-(N-Piperidinyl)-2'-hydroxy-5,5',6,6',7,7',8,8'-octahydro-1,1'-bi-2-naphthyl (**7c**)

Following the same procedure for the preparation of **7a** by using 25% glutaric dialdehyde instead of formaldehyde, **7c** was obtained in 86% yield: mp 35—37℃. $[\alpha]_D^{15}$ +61.1(c 0.5, THF). IR (KBr pellet) ν: 3419, 2927, 2852, 1590, 1473, 1218, 811 cm^{-1}. ^1H NMR(300 MHz, $CDCl_3$) δ: 7.08(d, J = 8.2 Hz, 1H), 6.98(d, J = 8.2 Hz, 1H), 6.90(d, J = 8.2 Hz, 1H), 6.79 (d, J = 8.2 Hz, 1H), 2.72—2.76 (m, 8H), 2.17—2.33(m, 4H), 1.26—1.76(m, 8H), 0.88—0.95(m, 6H). ^{13}C NMR(100.6 MHz, $CDCl_3$) δ: 150.18, 137.54, 136.16, 132.61, 130.21, 129.64, 129.49, 128.99, 125.69, 125.53, 117.00, 113.66, 53.40, 30.36, 29.59, 29.40, 27.61, 27.31, 26.54, 24.22, 23.30, 23.29, 23.24, 22.94. m/z(%): 361(M^+, 100). HRMS Calcd for $C_{25}H_{31}NO$: 361.2406. Found: 361.2380.

(R)-(+)-2-(N-Isopropyl-N-methylamino)-2'-hydroxy-5,5',6,6',7,7',8,8'-octahydro-1,1'-bi-2-naphthyl (**9a**)

Following the same procedure for the preparation of **7a** by using acetone instead of formaldehyde, only monoalkylated H8-NOBIN (**8a**) was obtained. The crude product was submitted to methylation with formaldehyde following the same procedure, **9a** was obtained in 81% yield (two steps): $[\alpha]_D^{15}$ + 56(c 0.5, THF). IR (KBr pellet) ν: 3408, 2924, 2856, 1588, 1472, 1198, 814 cm^{-1}. ^1H NMR(300 MHz, $CDCl_3$) δ: 7.07(d, J = 8.2 Hz, 1H), 6.97(d, J = 8.2 Hz, 1H), 6.89(d, J = 8.2 Hz, 1H), 6.79(d, J = 8.2 Hz, 1H), 3.00—3.06(m, 1H), 2.72—2.79(m, 4H), 2.53(s, 3H), 2.21—2.31(m, 4H), 1.60—1.74(m, 8H), 0.77(d, J = 6.6 Hz, 3H), 0.73(d, J = 6.4 Hz, 3H), 0.88—0.95(m, 6H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 150.42, 136.27, 130.34, 129.98, 129.64, 129.40, 129.18, 128.40, 127.95, 118.18, 112.50, 109.78, 61.54, 44.50, 29.71, 29.61, 29.22, 27.56, 27.36, 23.19, 23.09, 22.82, 22.70. m/z(%): 349 (M^+, 18.8), 334 (M^+ - CH_3, 18.8). HRMS Calcd for $C_{24}H_{31}NO$: 349.2406. Found: 349.2415.

(R)-(+)-2-(N-Isobutyl-N-methylamino)-2'-hydroxy-5,5',6,6',7,7',8,8'-octahydro-1,1'-bi-2-naphthyl (**9b**)

Following the same procedure for the preparation of **9a** by using isobutyraldehyde instead of acetone, **9b** was obtained in 75% yield (two steps). IR (KBr) ν: 3414, 2926, 2856, 1591, 1475, 1280, 813 cm^{-1}; $[\alpha]_D^{15}$ + 3.0(c 1.0, THF). ^1H NMR(300 MHz, $CDCl_3$) δ: 7.09(d, J = 8.2 Hz, 1H), 6.97(d, 2H, J = 8.2 Hz, 2H), 6.76(d, J = 8.2 Hz, 1H), 2.71—2.79(m, 4H), 2.46(s, 3H), 2.10—2.43(m, 6H), 1.61—1.72(m, 8H), 0.94—0.99(m, 1H), 0.58(m, 6H). ^{13}C NMR(100.6 MHz, $CDCl_3$) δ: 150.71, 138.03, 136.02, 130.19, 130.10, 129.91, 129.68, 129.54, 117.74, 113.55, 112.51, 108.62, 65.23, 51.70, 29.63, 29.29, 27.57, 27.27, 26.91, 25.94, 23.13, 23.10, 22.77, 20.04. m/z(%): 363 (M^+, 16.9), 334 (M^+ - C_3H_7, 100). HRMS Calcd for $C_{25}H_{33}NO$: 363.2562. Found: 363.2526.

(R)-(+)-2-(N-Cyclohexyl-N-methylamino)-2'-hydroxy-5,5',6,6',7,7',8,8'-octahydro-1,1'-bi-2-naphthyl (**9c**)

Following the same procedure for the preparation of **9a** by using cyclohexanone instead of acetone, **9c** was obtained in 70% yield (two steps). $[\alpha]_D^{15}$ + 7.9(c 1.0, THF). IR (KBr pellet) ν: 3422, 2926, 2852, 1594, 1474, 1098, 812 cm^{-1}. ^1H NMR(300 MHz, $CDCl_3$) δ: 7.07(d, J = 8.2 Hz, 1H), 6.98(d, J = 8.2 Hz, 1H), 6.91(d, J = 8.2 Hz, 1H), 6.79(d, J = 8.2 Hz, 1H), 2.69—2.80(m, 4H), 2.60(s, 3H), 2.49—2.59(m, 1H), 2.17—2.26(m, 4H), 1.57—1.74(m, 8H), 1.15—1.26(m, 6H), 0.78—

0.98(m, 4H). ^{13}C NMR(100.6 MHz, $CDCl_3$) δ: 150.71, 138.34, 136.18, 130.35, 130.05, 129.80, 129.74, 129.69, 129.38, 114.57, 112.58, 109.92, 52.00, 33.53, 29.77, 29.72, 29.27, 27.83, 27.41, 25.89, 23.46, 23.28, 23.25, 23.17, 22.78. *m/z* (%): 389 (M^+, 16.9), 318 ($M^+ - C_5H_{11}$, 100). HRMS Calcd for $C_{27}H_{35}NO$: 389.2719. Found: 389.2740.

(R)-(+)-2-(N-Benzyl-N-methylamino)-2′-hydroxy-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-bi-2-naphthyl (**9d**)

Following the same procedure for the preparation of **7d** by using benzaldehyde instead of acetone, **7g** was obtained in 35% yield (two steps). $[\alpha]_D^{15}$ + 13.6(*c* 1.0, THF). IR (KBr) ν: 3417, 2925, 2853, 1591, 1474, 1157, 812 cm^{-1}. ^{1}H NMR(300 MHz, $CDCl_3$) δ: 7.13—7.18(m, 4H), 7.04(d, *J* = 8.2 Hz, 1H), 6.97(d, *J* = 8.2 Hz, 1H), 6.84—6.88 (m, 2H), 6.76(d, *J* = 8.2 Hz, 1H), 3.85(d, *J* = 13.8 Hz, 1H), 3.71(m, *J* = 13.8 Hz, 1H), 2.74—2.82(m, 4H), 2.42(s, 3H), 2.14—2.40(m, 4), 1.64—1.74 (m, 8H), 1.15—1.26(m, 6H), 0.78—0.98(m, 4H). ^{13}C NMR(100.6 MHz, $CDCl_3$) δ: 150.21, 137.78, 136.15, 130.52, 130.01, 129.85, 129.73, 129.28, 129.09, 128.50, 128.03, 126.87, 118.23, 113.09, 112.59, 108.23, 61.62, 40.50, 29.71, 29.46, 27.56, 27.35, 23.32, 23.24, 23.22, 22.97. *m/z* (%): 397 (M^+, 95.7), 306 ($M^+ - C_7H_7$, 100). HRMS Calcd for $C_{28}H_{31}NO$: 397.2406. Found: 397.2412.

General procedure for the evaluation of H_8-NOBIN *derivatives* **7** *and* **9** *for asymmetric addition of diethylzinc to benzaldehyde*

All reactions were carried under argon. Weighed amounts of H_8-NOBIN derivatives (0.0075 mmol) were introduced into 1 mL of polypropylene microtubes. Dried toluene (0.5 mL) and Et_2Zn (1 mol/L in hexane, 0.6 mL) were added with microsyringes. The microtubes were then set up in the reaction block to maintain the temperature at 0℃ for 30 min, and finally benzaldehyde (22 μL, 0.2 mmol) was introduced. After agitation for 24 h at 0℃, the tubes were opened. The reaction was quenched with aqueous NH_4Cl solution and extracted with ethyl acetate. The product mixtures were then submitted to HPLC-CD analysis as described previously.[7]

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