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SYNTHESIS OF 7-SUBSTITUTED DERIVATIVES OF 5, 8-DIMETHYLISOQUINOLINE

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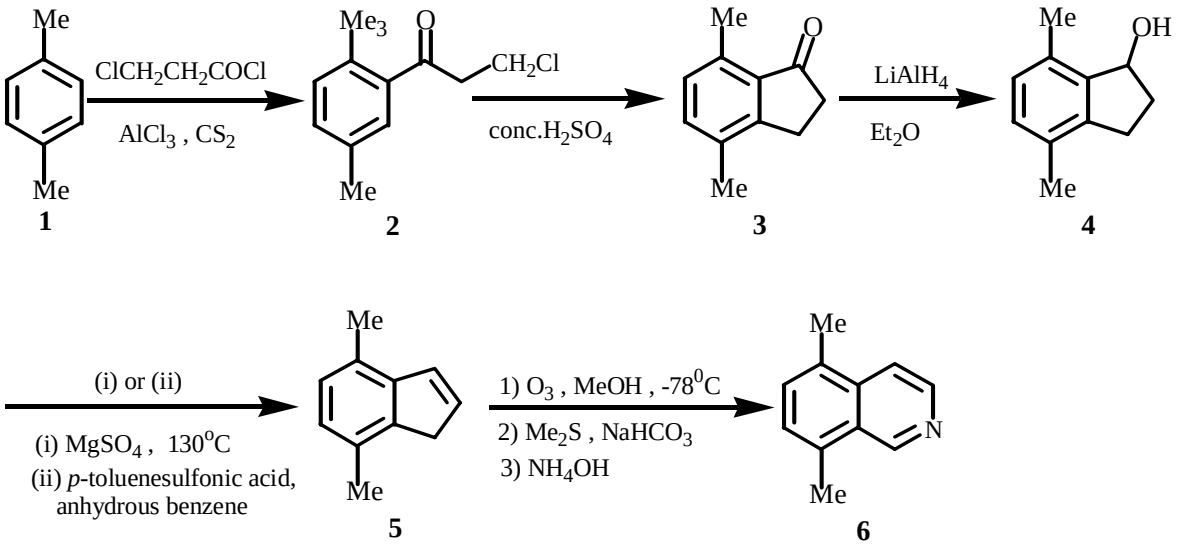
Abstract – 7-Bromo-5,8-dimethylisoquinoline was selectively synthesized by the bromination of 5,8-dimethylisoquinoline obtained in five steps from *p*-xylene as a starting material. Further, 7-bromo-5,8-dimethylisoquinoline gave 7-amino-5,8-dimethylisoquinoline by the reaction with ammonia and various 7-anilino-5,8-dimethylisoquinolines *via* a palladium-catalyzed coupling reaction with anilines.

Isoquinoline alkaloids and variously substituted isoquinolines have been reported to have special bioactives. For example, halide substituted isoquinoline has been reported to know an insecticide effect as to 6-bromoisoquinoline[1] and carbazole derivatives containing isoquinoline ring have been reported to show anticancer and antitumor effects as to ellipticine.[2] Therefore, such new isoquinoline derivatives also are expected to have biological activities.

Previously, synthesis of several isoquinoline derivatives has been investigated to obtain carbazole derivatives containing isoquinoline ring.[3] 5,8-Dimethylisoquinoline (**6**) is synthesized in high yield *via* five steps from *p*-xylene as shown in Scheme 1.[4] Therefore this compound is expected as good intermediate for new isoquinoline derivatives. Recently, 7-nitro- and 6-bromo-7-nitro-5,8-dimethyl-isoquinolines are obtained by similar steps.[5] In this work, substitution reactions of **6** was investigated and 7-bromo-5,8-dimethylisoquinoline (**7a**), which is expected as intermediate for new isoquinoline derivatives, was selectively synthesized by the bromination of 5,8-dimethylisoquinoline (**6**) (Scheme 2). Then the substitution reactions of these isoquinolines were investigated and the catalytic amination of bromoisoquinoline **7a** gave new anilinoisoquinoline derivatives (Scheme 3).

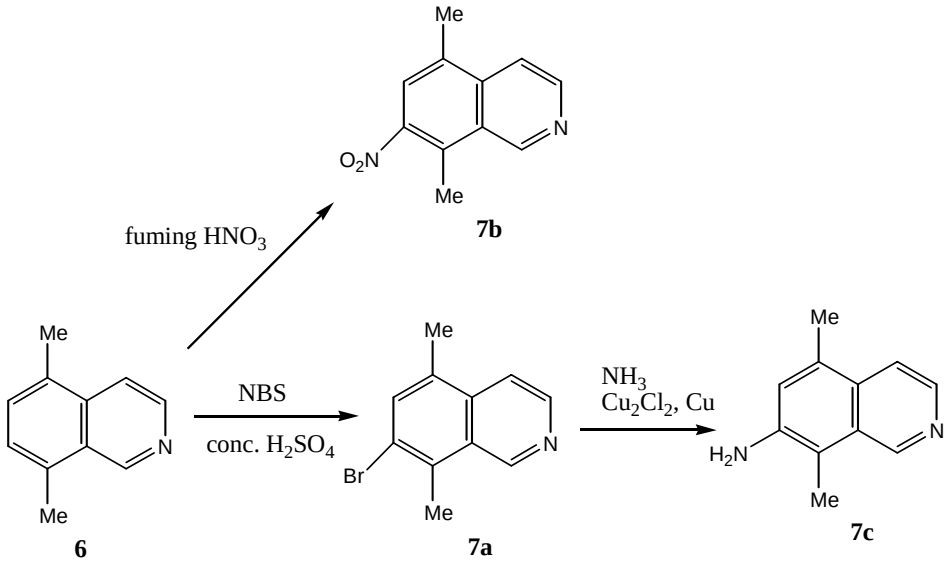
RESULTS AND DISCUSSION

≠ Dedicate to the memory of departed Professor Ivar Ugi.



Scheme 1

Bromination and nitration of 5,8-dimethylisoquinoline. 5,8-Dimethylisoquinoline (**6**) was synthesized *via* five steps from *p*-xylene as a starting material by improving each reported method.[4] Yields of each steps were 2',5'-dimethyl-3-chloropropiophenone (**2**): 76%, 4,7-dimethylindan-1-one (**3**): 98%, 4,7-dimethylindan-1-ol (**4**): 95%, 4,7-dimethylindene (**5**): 65%($MgSO_4$), 78% (*p*-toluenesulfonic acid), and 5,8-dimethylisoquinoline (**6**): 92%. On the preparation of indene **5**, high yield was obtained by dehydration with *p*-toluenesulfonic acid than with $MgSO_4$[6].



Scheme 2

The substitution reactions of **6** were investigated as in Scheme 2. Bromo substituted isoquinolines (**7a**) was synthesized in 77% yield according to the method in Scheme 2. Single crystal of **7a** was created by the crystallization from methylcyclohexane, and crystal data is shown in Table 1. The substituted position

of bromine was confirmed to be the 7-position of isoquinoline ring as shown by X-ray crystal structure analysis in Figure 1. It was shown that the bromination gave selectively 7-bromo compound. The nitration of **6** with fuming nitric acid gave only low yield(1%) of 7-nitro-5,8-dimethylisoquinoline (**7b**) because of many side reactions.

Table 1 Crystal data for **7a**

Formula		$C_{11}H_{10}NBr$
Temperature (K)		293(2)
Crystal size (mm^3)		0.50×0.30×0.10
Crystal system		Monoclinic
Space group		$P2_1/c$
Unit cell dimension	a (Å)	3.9984(4)
	b (Å)	29.463(3)
	c (Å)	8.0883(8)
	β (°)	90.845(2)
	Volume ($Å^3$)	952.73(17)
Z		4
Ref.used(|Fo|≧2σ(Fo))		1763
R factor		0.038
wR factor		0.091

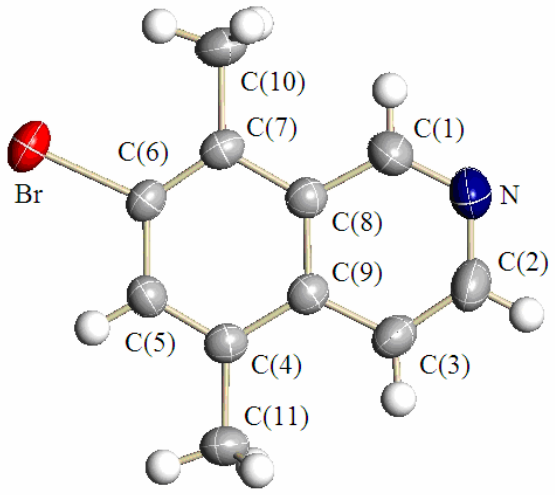
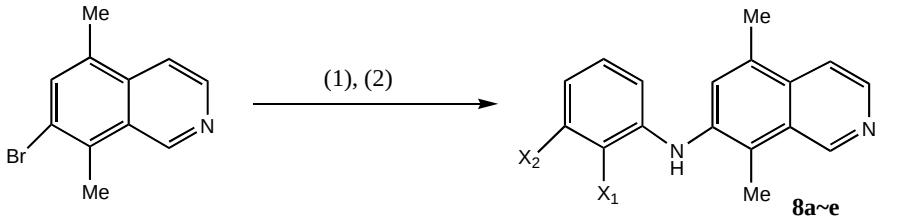


Figure 1 ORTEP drawing of **7a**

Amination and coupling reaction of 7-bromo-5,8-dimethylisoquinoline. The substitution reactions of **7a** were investigated as in Scheme 2 and Scheme 3. The catalytic amination of **7a** with ammonia in the presence of copper gave 7-amino-5,8-dimethylisoquinoline(**7c**) in 20% yield.

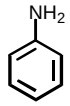
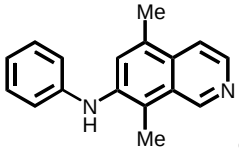
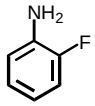
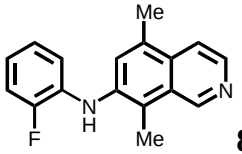
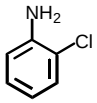
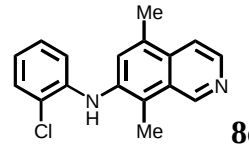
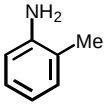
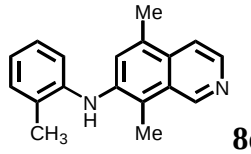
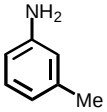
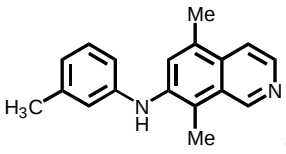
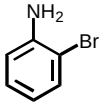
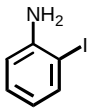
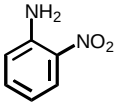


(1) : Ullmann reaction
(2) : $Pd(OAc)_2$/ DPEphos
NaO*t*-Bu, anilines

a : X_1=H, X_2=H **d** : X_1=Me, X_2=H
b : X_1=F, X_2=H **e** : X_1=H, X_2= Me
c : X_1=Cl, X_2=H

Scheme 3

Table 2 Palladium-catalyzed coupling reaction of bromoisoquinoline **7a** with anilines

Anilines	Products	Yield (%)	Mp (℃)
	 **8a**	80	152.2～153.5
	 **8b**	67	152.3～153.0
	 **8c**	52	165.8～166.3
	 **8d**	34	164.0～165.0
	 **8e**	52	146.9～147.3
	no reaction		
	no reaction		
	no reaction		

7-Anilino-5,8-dimethylisoquinoline (**8a**) was obtained in 40% yield by the Ullman reaction of **7a** with acetanilide. The coupling reaction was improved by palladium catalyzed metod[7] and various 7-anilino-5,8-dimethylisoquinolines **8a～e** were synthesized by the palladium-catalyzed coupling reaction of 7-bromo-5,8-dimethylisoquinoline (**7a**) with anilines. The yields in the palladium-catalyzed coupling reaction are shown in Table 2. Non-substituted aniline provided 7-anilino-5,8-dimethylisoquinolines(**8a**) in higher yield than Ullman reaction. Anilines having electron-donating substituent (X_1= CH_3) or poor electron-attracting substituents (X_1= F, Cl) on benzene ring reacted, but anilines having strong electron-attracting substituent (X_1= NO_2) did not react. Further, anilines having halogen substituents (X_1= Br, I) did not react. It can be considered that another reaction occurs mainly, since the electron-attracting of X_1= Br, I is poor than X_1=F, Cl. Moreover, on the reaction of methyl substituted anilines, *m*-position product was provided in higher yield than *o*-position. The high yield of non-substituted compound **8a** is considered that lower steric hindrance by the substituents has also influenced.

CONCLUSION

7-Bromo-5,8-dimethylisoquinoline was selectively synthesized in high yield by the bromination of 5,8-dimethylisoquinoline. Then various 7-anilino-5,8-dimethylisoquinolines were synthesized via a palladium-catalyzed coupling reaction of 7-bromo-5,8-dimethylisoquinolines with anilines.

EXPERIMENTAL

Instruments Melting points were determined by using a MRK MP-MG. IR spectra were recorded on a JASCO FT/IR-410 by using potassium bromide pellet or carbon tetrachloride solution. ^{1}H-NMR and ^{13}C-NMR spectra were acquired on a JEOL JNM-ECP500 at 500 and 125 MHz in deuteriochloroform or deuteriomethanol. ^{1}H-NMR coupling constants are given in Hz and all chemical shifts are relative to an internal standard of trtramethylsilane. Low-resolution electron impact mass spectra were obtained on a JEOL MS station. X-ray crystal structure analysis was determined by using a Bruker-AXS smart-APEX and analyzed on a SHELXS-97. Thin layer chromatography was performed on Merck Silica gel 60F_{254}.

Materials

5,8-Dimethylisoquinoline (**6**)[4] was prepared in five steps from *p*-xylene. All solvents and other reagents were purchased. 2',5'-Dimethyl-3-chloropropiophenone (**2**) was prepared from *p*-xylene and 4,7-dimethylindan-1-one (**3**) was prepared from **2**.[8] Then 4,7-dimethylindan-1-ol (**4**)[9] was prepared by the reduction of **3** with lithium aluminum hydride.

4,7-Dimethylindene (**5**) was prepared as follow**:** To a 500 mL, 4-necked flask equipped with a condenser and 200 ℃ thermometer was charged 4,7-dimethylindan-1-ol (**4**) (20.53 g, 0.126 mol) and anhydrous

benzene (400 mL). The solution was refluxed for 5 min. Further, *p*-toluenesulfonic acid was added and refluxed for 30 min. When the reaction was complete, 1 M aq.NaOH was added. The solution was extracted organic layer with benzene and washed by aq.sodium chloride. The organic layer was dried ($MgSO_4$) and evaporated under reduced pressure to give yellow liquid. The residual liquid was purified by distillation under reduced pressure to give the 4,7-dimethylindene (**5**) as a light yellow liquid (32.32 g, 78.0%), bp 61.0～62.0 ℃/1 mmHg (lit.,[6]: bp 63.0～65.0 ℃/1 mmHg). ^{1}H-NMR (500 MHz, $CDCl_3$): δ 6.91 (1H, d, CH arom. *J*=7.5 Hz), 6.89 (1H, d, CH, *J*=5.5 Hz), 6.82 (1H, d, CH arom. *J*=7.5 Hz), 6.45 (1H, d, CH, *J*=5.5 Hz), 3.18 (2H, d, CH_2, *J*=5.5 Hz), 2.32 (3H, s, CH_3), 2.23 (3H, s, CH_3), MS (*m/z*): 144 $[M]^+$

5,8-Dimethylisoquinoline (6)[4] was prepared as follows: To a 500 mL, 4-necked flask equipped with ozone debouchments and –100 ℃ thermometer was charged 4,7-dimethylindene (**5**) (11.45 g, 0.0793 mol) and MeOH (300 mL). The solution was stirred at –78 ℃ and flowed ozone at same temp. for 2 h. When the reaction was complete, the flask was light shielding by aluminum sheet and left to rt. And after, dimethyl sulfide (16.0 mL) and $NaHCO_3$ (16.0 g, 0.190 mol) were added and stirred at rt for 4 h. Further, 25% NH_4OH (150 mL) was added and stirred at rt for 17 h. When the reaction was complete, water (500 mL) was added and the solution was extracted organic layer with CH_2Cl_2. The organic layer was dried ($MgSO_4$), and evaporated under reduced pressure to give yellow liquid. The residual liquid was purified by distillation under reduced pressure to give 5,8-dimethylisoquinoline (**6**) as a yellow liquid (11.42 g, 92 %), bp107.5～110.2 ℃/ 2.5mmHg (107.2～109.5 ℃/2.5 mmHg).[4] ^{1}H-NMR (500 MHz, CD_3OD): δ 9.19 (1H, s, CH arom.), 8.37 (1H, d, CH arom. *J*=6.0 Hz), 7.68 (1H, d, CH arom. *J*=6.0 Hz), 7.30 (1H, d, CH arom. *J*=7.0 Hz), 7.17 (1H, d, CH arom. *J*=7.0 Hz), 2.59 (3H, s, CH_3), 2.49 (3H, s, CH_3); ^{13}C-NMR (125 MHz, CD_3OD): δ 149.8 (C-1), 142.3 (C-3), 136.7 (C-4a), 134.3 (C-8), 132.6 (C-6), 132.0(C-5), 129.0 (C-7), 128.6 (C-8a), 118.9 (C-4), 18.28, 18.18 (CH_3); MS (EI) (*m/z*): 157 $[M]^+$

Synthesis of 7-substituted 5,8-dimethyisoquinolines

7-Bromo-5, 8-dimethylisoquinoline (7a) To a 100 mL, 4-necked flask equipped with a condenser, 100 mL separtory funnel and 200 ℃ thermometer was charged 5,8-dimethylisoquinoline (**6**) (3.32 g, 0.0211 mol) and stirred in ice bath under the nitrogen atmosphere. 97% conc. H_2SO_4 (30 mL) was added portionwise over 30 min. After portionwise, the solution was added *N*-bromosuccinimide (4.55 g, 0.0255 mol) and stirred at 60 ℃ for 7 h. When the reaction was complete, the reacted solution was filled with 10 % aq.$NaNO_2$ (100 mL) and was neutralized with 45% aq.NaOH. The solution was filtered off under reduced pressure. The filtrate was extracted organic layer with $CHCl_3$. The organic layer was dried ($MgSO_4$). The filtered solid was refluxed in $CHCl_3$, filtered off under reduced pressure and dried

($MgSO_4$). The dried solution was evaporated under reduced pressure to give a brown solid. The residual solid was purified by distillation under reduced pressure to give a white solid. Further, the solid was recrystallised from mixed hexane and benzene to afford a white powder of 7-bromo-5,8-dimethylisoquinoline (**7a**) (3.87 g, 77 %), bp 162.2～164.4 ℃/4.0 mmHg, mp 93.2～94.8 ℃, ^{1}H-NMR (500 MHz, CD_3OD): δ 9.32 (1H, s, CH arom.), 8.48 (1H, d, CH arom. *J*=6.0 Hz), 7.80 (1H, d, CH arom. *J*=6.0 Hz), 7.64 (1H, s, CH arom.), 2.59(3H, s, CH_3), 2.49(3H, s, CH_3); ^{13}C-NMR (125MHz, CD_3OD): δ 150.2 (C-1), 143.0 (C-3), 136.4 (C-4a), 136.1 (C-6), 135.0 (C-8), 133.7 (C-5), 129.6 (C-8a), 124.6 (C-7), 119.2 (C-4), 18.0, 17.7 (CH_3); MS (EI) (*m/z*): 235 $[M]^+$, 237 $[M+2]^+$; HRMS Calcd for $C_{11}H_{10}BrN$: 234.9997, found M^+: 234.9995.

7-Nitro-5,8-dimethylisoquinoline (7b) To a 100 mL 4-necked flask equipped with a condenser, 100 mL separtory funnel and -100 ℃ thermometer was charged 5,8-dimethylisoquinoline (**6**) (3.00 g, 0.0191 mol) and 97% conc. H_2SO_4 (30 mL) was added portionwise over 30 min. After portionwise, the solution was stirred at -40 ℃ for 1.5 h. When the reaction was complete, the reacted solution was neutralized with 45% aq.NaOH The solution was filtered off under reduced pressure. The filtrate was extracted organic layer with $CHCl_3$. The organic layer was dried ($MgSO_4$). The filtered solid was refluxed in $CHCl_3$, filtered off under reduced pressure and dried ($MgSO_4$). The dried solution was evaporated under reduced pressure to give a brown solid. The crude product was purified by chromatography on silica gel using petroleum ether and toluene(1:1) as the eluant, affording the product as a yellow solid. The solid was recrystallised from methylcyclohexane to afford a yellow crystal of 7-nitro-5,8-dimethylisoquinoline (**7c**) (0.05 g, 0.8 %), mp 159.0～163.0 ℃ (161.0~162.0 ℃)[5], ^{1}H-NMR (500 MHz, CD_3OD): δ 9.56 (1H, s, CH arom.), 8.67 (1H, d, CH arom. *J*=10.0 Hz), 7.93 (1H, d, CH arom. *J*=10.0 Hz), 7.77 (1H, d, CH arom. *J*=10.0 Hz), 2.84 (3H, s, CH arom.), 2.63 (3H, s, CH_3); MS (EI) (*m/z*): 203 $[M]^+$

7-Amino-5,8-dimethylisoquinoline (7c) In a steel reaction vessel, (1.14 g, 5.0 mmol) of 7-bromo-5,8-dimethylisoquinoline (**7a**) are placed, 0.13 g copper, and 60 mL (1.0 mol) of 25 % ammonia containing 0.11 g of cuprous chloride. The steel reaction vessel is rocked and heated at 195 ℃ for 24 h. After cooling, the bomb is emptied and the two layers are separated; 2 mL of 40 % NaOH is added to the mixture, and the mixture was extracted with $CHCl_3$. The organic layer was washed with aq.NaCl, and dried ($MgSO_4$) and evaporated under reduced pressure to give yellow solid. The crude product was purified by chromatography on silica gel using $CHCl_3$ as eluent, affording the product as a yellow solid. The solid was recrystallised from methylcyclohexane to afford a yellow crystal of 7-amino-5,8-dimethylisoquinoline (**7c**) (1.07 g, 44 %) mp 210.0~212.0 ℃ (lit.,[5] 201.5~202.5 ℃), ^{1}H-NMR (500 MHz, CD_3OD): δ 9.23 (1H, s, CH arom.), 8.26 (1H, d, CH arom., *J*=6.0 Hz), 7.54 (1H, d, CH arom. *J*=6.0 Hz), 6.92 (1H, s, CH arom.), 3.69 (2H, bs, NH_2), 2.53 (3H, s, CH_3), 2.26 (3H, s, CH_3);

MS (EI) (*m/z*): 173 $[M]^+$.

Synthesis of anilino-5,8-dimethylisoquinolines

7-Anilino-5, 8-dimethylisoquinoline (8a)

(Ullmann reaction): To a 50 mL, 3-necked flask equipped with a condenser was charged 7-bromo-5,8-dimethylisoquinoline (**7a**) (3.63 g, 0.0154 mol), acetanilide (5.00 g, 0.037 mol), K_2CO_3 (3.00 g, 0.0217 mol) and copper powder (1.50 g, 0.0236 mol), which were crushed and mixed by mortar. Further, ten spatula of iodide was added. After heating to 100 ℃, the mixture was strongly stirred at 130 ℃ for 24 h. When the reaction was complete, the reacted mixture was melted by CH_2Cl_2 and filtered off under reduced pressure. The solution was extracted organic layer with CH_2Cl_2 and water. The organic layer was dried ($MgSO_4$), and evaporated under reduced pressure to give brown oil. Then, to a 200 mL, 2-necked flask equipped with a condenser and 200 ℃ thermometer was charged the oil and conc. hydrochloric acid. The solution was refluxed for 20 h. When the reaction was complete, the reacted solution was neutralized with 45% aq.NaOH and was filtered off under reduced pressure. The filtrate was extracted organic layer with CH_2Cl_2. The organic layer was dried ($MgSO_4$), and evaporated under reduced pressure to give brown oil. The crude product was purified by chromatography on silica gel using AcOEt as the eluant, affording the product as a yellow solid. The solid was recrystallised from methylcyclohexane to afford a yellow crystal of 7-anilino-5,8-dimethylisoquinoline (**8a**) (1.56 g, 40.9%)

(Palladium-catalyzed coupling reaction): To a 100 mL, 4-necked flask equipped with a condenser, 100 mL separtory funnel and 200 ℃ thermometer was charged palladium acetate (9.7 mg, 0.050 mmol) and bis[2-(diphenylphosphino)phenyl] ether (DPEphos) (0.035 g, 0.075 mmol)[7] under the nitrogen atmosphere. Anhydrous toluene (22 mL) was added portionwise over 5 min. After the addition, the solution was stirred for 5 min at rt under the nitrogen current. 7-bromo-5,8-dimethylisoquinoline (**7a**) (2.05 g, 8.68 mmol), aniline (0.90 g, 9.72 mmol) and solid NaO *t*-Bu (1.18 g, 0.012 mol) was added. Then, the flask was purged for 5 min. with nitrogen, and the mixture was stirred at 100 ℃ for 24 h. When the reaction was complete, the solution was extracted organic layer with ether and water. The organic layer was washed by aq.NaCl, dried ($MgSO_4$) and evaporated under reduced pressure to give yellow solid. The crude product was purified by chromatography on silica gel using hexane: AcOEt (8:2) as the eluant, affording the product as a yellow solid. The solid was recrystallised from methylcyclohexane to afford a yellow crystal of 7-anilino-5,8-dimethylisoquinoline (**8a**) (1.71 g, 80 %), mp 152.2～153.5 ℃, ^{1}H-NMR (500 MHz, CD_3OD): δ 9.32 (1H, s, CH arom.), 8.34 (1H, d, CH arom. *J*=5.5 Hz), 7.83 (1H, d, CH arom. *J*=5.5 Hz), 7.53 (1H, s, CH arom.), 7.20 (2H, t, CH arom. *J*=7.5 Hz), 6.89 (2H, d, CH arom. *J*=7.5 Hz), 6.83 (1H, t, CH arom. *J*=7.5 Hz), 2.59 (3H×2, s, CH_3); MS (FAB) (*m/z*): 249 $[M+1]^+$

Anal. Calcd for $C_{17}H_{16}N_2$: C, 82.22; H, 6.49 ; N, 11.28 . Found: C, 81.44 ; H, 6.42 ; N, 11.27.

7-(*o*-Fluoroanilino)-5,8-dimethylisoquinoline (8b) The same palladium-catalyzed coupling above p was used as for compound (**8a**), such that 67 % of **8b** was isolated. mp 152.3～153.0 ℃, ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.38 (1H, s, CH arom.), 8.46 (1H, d, CH arom. *J*=6.0 Hz), 7.64 (1H, d, CH arom. *J*=6.0 Hz), 7.38 (1H, s, CH arom.), 7.05 (1H, t, CH arom. *J*=7.5 Hz), 6.91 (1H, t, CH arom. *J*=7.5 Hz), 6.78～6.73 (2H, m, CH arom.), 5.66 (1H, s, NH), 2.56 (3H, s, CH_3), 2.54(3H, s, CH_3); MS (FAB) (*m/z*): 267 $[M+1]^+$

Anal. Calcd for $C_{17}H_{15}FN_2$: C, 76.67 ; H, 5.68 ; N, 10.52. Found: C, 76.43 ; H, 5.72 ; N, 10.60 .

7-(*o*-Chloroanilino)-5,8-dimethylisoquinoline (8c) The same palladium-catalyzed coupling above was used as for compound (**8a**), such that 52 % of **8c** was isolated. mp 165.8～166.3 ℃, ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.48 (1H, s, CH arom.), 8.54 (1H, d, CH arom. *J*=5.5 Hz), 7.76 (1H, d, CH arom. *J*=5.5 Hz), 7.47 (1H, s, CH arom.), 7.39 (1H, d, CH arom. *J*=8.5 Hz), 7.09 (1H, t, CH arom. *J*=8.5 Hz), 6.82 (1H, t, CH arom. *J*=8.5 Hz), 6.75 (1H, d, CH arom. *J*=8.5 Hz), 6.07 (1H, s, NH), 2.64 (3H×2, s, CH_3); MS (FAB) (*m/z*): 283 $[M+1]^+$

Anal. Calcd for $C_{17}H_{15}ClN_2$: C, 72.21; H, 5.35 ; N,9.91. Found: C, 72.04 ; H, 5.32 ; N, 9.59 .

7-(*o*-Methylanilino)-5,8-dimethylisoquinoline (8d) The same palladium-catalyzed coupling above was used as for compound (**8a**), such that 34% of **8e** was isolated. mp 164.0～165.0 ℃, ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.41 (1H, s, CH arom.), 8.36 (1H, d, CH arom. *J*=6.0 Hz), 7.72 (1H, d, CH arom. *J*=6.0 Hz), 7.26 (1H, s, CH arom.), 7.18 (1H, d, CH arom. *J*=7.5 Hz), 7.08 (1H, t, CH arom. *J*=7.5 Hz), 6.92 (1H, t, CH arom. *J*=7.5 Hz), 6.85 (1H, d, CH arom. *J*=7.5 Hz), 5.41 (1H, s, NH), 2.53 (3H, s, CH_3), 2.51 (3H, s, CH_3), 2.24(3H, s, CH_3); MS (FAB) (*m/z*): 263 $[M+1]^+$

Anal. Calcd for $C_{18}H_{18}N_2$: C, 82.41; H, 6.92 ; N, 10.68. Found: C, 82.11 ; H, 6.67 ; N, 10.58 .

7-(*m*-Methylanilino)-5,8-dimethylisoquinoline (8e) The same palladium-catalyzed coupling procedure above was used as for compound (**8a**), such that 52% of **8f** was isolated. mp 152.3～153.0 ℃, ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.43 (1H, s, CH arom.), 8.49 (1H, d, CH arom. *J*=6.0 Hz), 7.69 (1H, d, CH arom. *J*=6.0 Hz), 7.47 (1H, s, CH arom.), 7.15 (1H, t, CH arom. *J*=7.5 Hz), 6.75 (1H, d, CH arom. *J*=7.5 Hz), 6.72～6.71 (2H, m, CH arom.), 5.62 (1H, s, NH), 2.59 (3H×2, s, CH_3), 2.31 (3H, s, CH); MS (FAB) (*m/z*): 263 $[M+1]^+$

Anal. Calcd for $C_{18}H_{18}N_2$: C, 82.41; H, 6.92 ; N, 10.68. Found : C, 82.09 ; H, 6.59 ; N, 10.82.

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