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SYNTHESIS OF HETEROARYLQUINOLINES BY THE PHOTOLYSIS OF 2-, 3-, AND 4-IODOQUINOLINES[1]

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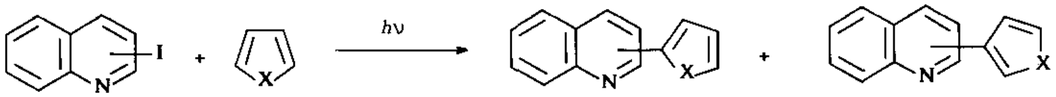
Abstract —— Photoreaction of 2-, 3-, and 4-iodoquinolines with five membered heterocycles (i.e. 1-methylpyrrole, pyrrole, furan and thiophene) afforded the corresponding n-(2-heteroaryl)quinolines (n=2, 3, 4) in appreciable yields.

Although numerous reports[2] appeared for the syntheses of quinoline derivatives, general methodology for introducing an aryl or heteroaryl group to the quinoline ring is lacking except preparation of arylquinolines by palladium-catalyzed cross-coupling between dialkylquinolylboranes and aryl bromides.[3] This method, however, is inapplicable without heteroaryl halides available, and limited to the synthesis of 2-, and 3-arylquinolines. Previously we have reported that photoreaction of 2- and 4-iodopyridines with five-membered heterocycles yields the corresponding n-(2-heteroaryl)pyridines (n = 2, 4) in appreciable yields.[4] In the present paper, we describe the application of this reaction to synthesis of heteroarylquinolines.

Irradiation of 2-iodoquinoline (n-IQ; n=2) in five-membered heterocycles (1-methylpyrrole, **1a**; pyrrole, **1b**; furan, **1c**) with a low-pressure mercury lamp (LP. Hg lamp)(254 nm) afforded the corresponding n-(m-heteroaryl)quinolines (n-HarQ)(**2a**$_2$, **2b**$_2$, **2c**$_2$; n=2, m=2) in good yield as each single isomer (Scheme 1).



n-IQ 1(a-d) n-HarQ n(a_2-d_2) n-HarQ n(a_3-d_3)

n = 2, 3, 4: a, X =NCH_3; b, X =NH; c, X = O; d, X = S.

Scheme 1

Similar photolysis of 3- and 4-iodoquinolines (3-IQ , 4-IQ) with **1a-c** afforded 3-, and 4-HarQ (**3a**$_2$-c_2, **4a**$_2$-c_2; m=2) in satisfactory yields. Although, the reactions with thiophene (**1d**) were sluggish to produce n-(m-thienyl)quinolines (**2d**$_2$; n=2, m=2: **3d**$_2$ and **3d**$_3$; n=3, m=2 and 3 : **4d**$_2$; n=4, m=2) in low yields (13%, 10% and 1.4%, 4%), irradiation with a high-pressure mercury lamp (HP. Hg lamp) using a Pyrex filter (>300 nm) appreciably improved the yields of the products (35% for **2d**$_2$, 71% and 9% for **3d**$_2$ and **3d**$_3$, 48% for **4d**$_2$).

These results are summarized in Table I. Structures of these products were determined on the basis of their mass spectra (*ms*), *proton-nuclear magnetic resonance* (^{1}H-*nmr*) *spectra* and elemental analysis and those of known compounds were confirmed by comparison of their physical properties.

Table I. Photolysis of 2-, 3-, and 4-Iodoquinolines (n-IQ, n=2,3,4) in Heteroaryl Compounds (**1**)

n-IQ/ n	1	Yield of HarQ(%)	Yield of quinoline(**5**)(%)	Recovery of IQ (%)
2	a	86	~0	-0
2	b	82	-0	-0
2	c	76	-0	5
2	d	13 (35)$^{a)}$	32 (52)$^{a)}$	43 (0)$^{a)}$
3	a	91	-0	1
3	b	85	-0	1
3	c	78	-0	12
3	d	10 $^{b)}$(71)$^{a,c)}$	4 (10)$^{)a)}$	66 (7)$^{a)}$
4	a	93	-0	1
4	b	90	-0	3
4	c	75	-0	14
4	d	4 (48)$^{a)}$	7 (22)$^{a)}$	63 (23)$^{a)}$

a) Yields given in parentheses were *obtained by irradiation with a HP. Hg lamp for 10 h.*
b) 3-Isomer (**3d**$_3$) was produced in 1.4% yield. c) Yield of **3d**$_3$ was 9 %.

From a synthetic view point, it would be desirable to carry out the reaction in an appropriate solvent. As an preliminary experiment, photoreaction of *n-IQ* (n=2,3,4) *with* **1a** *was carried out with a LP. Hg lamp in* various solvents, i. e., hexane, dichloromethane (CH_2Cl_2), t-butyl alcohol (tBuOH), and acetonitrile (MeCN).

Table II. Photoreaction of 2-Iodoquinoline (2-IQ) and 1-Methylpyrrole (**1a**) in Various Solvents$^{a)}$

Solvent	Yield (%)$^{a)}$ of **2a**$_2$	**2a**$_3$	**5**	Recovery of 2-IQ (%)
Hexane	26 (37)	3 (5)	7 (26)	44 (0)
$CH_2Cl_2$$^{b)}$	12 (27)	6 (18)	9 (9)	0 (3)
tBuOH	39 (37)	6 (10)	0 (0)	2 (0)
MeCN	18 (5)	7 (5)	3 (0)	6 (7)

a) ( ), HP. Hg lamp (10 h). b) 2-Chloro-*quinoline was obtained as a major product* (65%).

For 2-IQ, the reactions in solution afforded 2-(1-methylpyrrol-m-yl)quinoline (**2a**$_2$,m=2) and the 3-isomer **2a**$_3$ (m=3) in suppressed yields. Among the solvents examined, hexane seemed most preferable (Table II).

By contrast, reactions of 3- and 4-IQ proceeded smoothly in tBuOH to afford **3a**$_2$, and **4a**$_2$, respectively, with high regioselectivity, whereas the reaction in hexane effected hydrogen abstraction to furnish quinoline (**5**) significantly (Tables III and IV). Irradiation with a HP. Hg lamp seemed less effective on the yields of the coupling products, except for the reaction of 2-IQ in hexane or dichloromethane.

Table III. Photoreaction of 3-Iodoquinoline (3-IQ) and 1-Methylpyrrole (**1a**) in Various Solvents

	Yield (%)a) of		Recovery
Solvent	**3a$_2$**	**5**	of 3-IQ(%)
Hexane	34 (30)	50 (39)	7 (7)
CH_2Cl_2	53 (44)	11 (37)	11 (10)
tBuOH	81 (66)	6 (6)	7 (15)
MeCN	72 (37)	14 (13)	0 (19)

a) ( ), HP. Hg lamp.

Table IV. Photoreaction of 4-Iodoquinoline(4-IQ) and 1-Methylpyrrole (**1a**) in Various Solvents

	Yield (%) of		Recovery
Solvent	**4a$_2$**	**5**	of 4-IQ(%)
Hexane	25	51	8
CH_2Cl_2	33	6	28
tBuOH	72	5	12
MeCN	73	17	0

We then examined the photoreactions of n-IQ (n=2, 3, 4) and **1b-d** with a LP. Hg lamp in hexane (for 2-IQ) and tBuOH (for 3- and 4-IQ). The results were shown in Table V. The reaction of 2-IQ in hexane afforded the corresponding HarQ in moderate yields. On the other hand, 3- and 4-IQ reacted smoothly with **1a-c** and **1a-b** in tBuOH to give **3a**-c and **4a-b** in satisfactory yields. However, hydrogen abstraction took place preferentially with **1d** to furnish **5** as a major product and the formation of **3d** and **4d** was significantly reduced. Similar results were obtained with a HP. Hg lamp. The fact that hexane was an effective solvent for the photoreaction of 2-IQ can be understood by taking into account the important participation of a cationic intermediate, corresponding to the 2-pyridyl cation generated through the photolysis of 2-iodopyridine, on which we have reported recently.[1] The finding that 2-chloroquinoline was produced preferentially in the photolysis of 2-IQ and **1a** in dichloromethane (Table II) may also support the involvement of the 2-quinolyl cation in the reaction pathway.[1, 5]

Table V. Photoreaction of 2-, 3-, and 4-Iodoquinolines (2-, 3-, and 4-IQ) with Heteroaryl Compounds (**1**) in t-Butyl Alcohol

n-IQ/ n	**1**	Solvent	Yield of HarQ (%)	Yield of **5** (%)	Recovery of IQ (%)
2	ba)	Hexane	36 [51]c)	12	30
2	c	"	30 [52]	10	42
2	d	"	21 [43]	9	51
3	b	tBuOH	70 [78]	6	10
3	c	"	63 [72]	14	12
3	db)	"	23 [37]	27	38
4	b	"	76 [88]	4	14
4	c	"	26 [35]	12	26
4	d	"	14 [24]	25	41

a) **2b$_3$** was obtained in 2 % yield. b) **3d$_3$**, 4 %. c) [ ], yields based on IQ consumed.

Because of a simple procedure and moderate yields, the present method may provide a facile synthesis of heteroarylquinolines.

EXPERIMENTAL

All melting points are uncorrected. ^{1}H-Nmr spectra were measured with a JEOL FX 90Q (90 MHz) and a JNM-GX 270 (270 MHz) spectrometer in $CDCl_3$, and chemical shifts were given on the δ (ppm) scales with tetramethylsilane as an internal standard. Abbreviations are used as follows: s, singlet; d, doublet; t, triplet; m, multiplet. Ms were determined on a Shimadzu LKB-9000 mass spectrometer. Glc was performed on a Shimadzu GC 7A gas chromatograph equipped with a hydrogen flame-ionization detector using nitrogen as a carrier gas (30 ml/min). Glass columns (1.5 m, 3 mmϕ) of 3% OV-17 on Shimalite W (80-100 mesh) were used. Column chromatography was performed on a pre-packed silica gel column (Lobar column, Lichroprep Si-60, Merck). Thin layer chromatography (tlc) and preparative layer chromatography (plc) were carried out on Kieselgel 60 F_{254} and Kieselgel 60 PF_{254} (Merck) plates, respectively. Uv-irradiation was carried out with a 60 W low-pressure mercury lamp (Eiko-sha) in a quartz tube argon atmosphere using a merry-go-round apparatus (Eiko-sha) at room temperature, unless otherwise stated. Irradiation of the region at >300 nm was conducted with a 500 W high-pressure mercury lamp (Eiko-sha) in a Pyrex tube. The yields of the products given in Tables were determined by glc.

Materials

2-Iodoquinoline (Eastman Kodak Co.) and five-membered heterocylic compounds, 1-methylpyrrole, pyrrole, furan, and thiophene, are commercially available (Wako Pure Chemical Industries Ltd., Japan). 3-Iodoquinoline^6 and 4-iodoquinoline^7 were prepared according to the reported procedure.

General Procedure

A solution of iodoquinoline (n-IQ, n=2, 3, 4)(0.05 mmol) and **1** (1-methylpyrrole, pyrrole, furan, thiophene) (0.1 ml) in a solvent (or in **1** neat) (5 ml) was irradiated for 10 h. The reaction mixture was neutralized with 30 % aqueous K_2CO_3, and dried over anhydrous Na_2SO_4, followed by glc analysis with an internal standard of diphenyl. The residual reaction mixture was concentrated and submitted to column chromatography or plc with a solvent system described below and the yields are listed in Tables I-V.

2-(1-Methylpyrrol-2-yl)quinoline (**2a**$_2$) and 2-(1-Methylpyrrol-3-yl)quinoline (**2a**$_3$)

Plc (hexane-$CH_3CO_2C_2H_5$ = 5 : 1). **2a**$_2$: Colorless plates (from hexane), mp 55-56 °C (lit. 58-60 °C).^{8} Ms m/z(relative intensity, %): 208(M^+, 54), 207(100). Nmr: 4.20 (3H, s, N-CH_3), 6.21 [1H, dd, J (Hz)=3.6, 2.7, 4'-H], 6.77 (2H, 3'-H, 5'-H), 7.3-8.2 (6H, m, quinolyl H). **2a**$_3$: Columns (from benzene-hexane), mp 161-162°C. Anal. Calcd for $C_{14}H_{12}N_2$: C, 80.74; H, 5.81; N, 13.45. Found: C, 80.83; H, 5.84; N, 13.27. Ms m/z (%): 208 (M^+, 54), 207(22). Nmr: 3.75 (3H, s, N-CH_3), 6.69 (1H, dd, J=2.6, 2.2, 5' or 4'-H), 6.81 (1H, dd, J=2.6, 1.8, 4' or 5'-H), 7.43 (1H, t, J=7.5, quinolyl H), 7.61 (1H, broad s, 2'-H), 7.6-7.8 (3H, quinolyl H), 8.09 (1H, d, J = 8.8, quinolyl H), 8.14 (1H, d, J=8.8, quinolyl H).

2-(2-Pyrrolyl)quinoline (**2b**$_2$) and 2-(3-pyrrolyl)quinoline (**2b**$_3$)

Plc (hexane-ethyl acetate = 5:1). **2b**$_2$: Colorless plates (from hexane), mp 130.5-131 °C (lit. 133-135°C).^{8} Ms m/z(%): 194(M^+, 100), 193(14). Nmr: 6.23 (1H, dd, J=3.6, 2.6, 4'-H), 6.80 (2H, 3'-H, 5'-H), 7.1-8.1 (6H, m, quinolyl H). **2b**$_3$: Prisms (from benzene), mp 194-196°C. Anal. Calcd for $C_{13}H_{10}N_2$: C, 80.38; H, 5.19; N, 14.42. Found: C, 80.42; H, 5.27; N, 14.25. Ms m/z (%): 194 (M^+, 100), 193 (26.5). Nmr: 6.90 (2H, m, 4'-H, 5'-H), 7.43 (1H, t, J=7.4, quinolyl H), 7.60 (1H, broad s, 2'-H), 7.6-7.8 (3H, quinolyl H), 8.0-8.2 (2H, m, quinolyl H).

2-(2-Furyl)quinoline (**2c$_2$**)

Column chromatography (benzene-hexane = 5:1). Colorless needles (from hexane), mp 90-91°C. Anal. Calcd for $C_{13}H_9N_2O$: C, 79.98; H, 4.65; N, 7.71. Found: C, 79.90; H, 4.67; N, 7.47. Ms m/z(%): 195(M^+, 100). Nmr: 6.59 (1H, dd, J=3.5, 1.8, 4'-H), 7.23 (1H, dd, J=3.5, 0.9, 5'-H), 7.73(1H, dd, J=1.8, 0.9, 5'-H), 7.3-8.3 (6H, m, quinolyl H).

2-(2-Thienyl)quinoline (**2d$_2$**)

Column chromatography (benzene-hexane = 2:1). Colorless needles (from hexane), mp 132-133°C (lit. 130°C).[9] Ms m/z(%): 211(M^+, 100). Nmr: 7.06 (1H, dd, J=5.4, 4.0, 4'-H), 7.39 (1H, dd, J=4.0, 1.5, 3'-H), 7.69 (1H, dd, J=5.4, 1.5, 5'-H), 7.5-8.2 (6H, m, quinolyl H).

3-(1-Methylpyrrol-2-yl)quinoline (**3a$_2$**)

Column chromatography (CH_2Cl_2). Colorless oil. Picrate: prisms (from ethanol), mp 214-216°C(sealed tube). Anal. Calcd for $C_{20}H_{15}N_5O_7$: C, 54.92; H, 3.46; N, 16.01. Found: C, 54.82; H, 3.48; N, 15.89. Ms m/z(%): 208(M^+, 100), 207 (41). Nmr: 3.75 (3H, s, N-CH_3), 6.28 (1H, dd, J=3.7, 2.6, 4'-H), 6.42 (1H, J=3.7, 1.8, 3'-H), 6.82 (1H, dd, J=2.6, 1.8, 5'-H), 7.57 (1H, ddd, J=8.1, 7.0, 1.1, 6-H), 7.71 (1H, ddd, J=8.4, 7.0, 1.5, 7-H), 7.83 (1H, dd, J=8.1, 1.5, 5-H), 8.11 (1H, d, J=2.2, 4-H), 8.12 (1H, d, J=8.4, 8-H), 9.02 (1H, d, J=2.2, 2-H).

3-(2-Pyrrolyl)quinoline (**3b$_2$**)

Column chromatography (CH_2Cl_2-acetone = 20:1). Colorless needles (from benzene-hexane), mp 174-175 °C. Anal. Calcd for $C_{13}H_{10}N_2$: C, 80.38; H, 5.19; N, 14.42. Found: C, 80.35; H, 5.17; N, 14.26. Ms m/z(%): 194 (M^+, 100), 193(20), 167(16), 166(10). Nmr(10% D_2O in acetone-d_6): 6.28 (1H, dd, J=3.3, 2.7, 4'-H), 6.82 (1H, dd, J=3.3, 1.7, 3'-H), 7.02 (1H, dd, J=2.7, 1.7, 5'-H), 7.55 (1H, ddd, J=8.2, 6.7, 1.1, 6-H), 7.65 (1H, ddd, J =8.2, 6.7, 1.1, 7-H), 7.86 (1H, dd, J=8.2, 1.1, 5-H), 8.00 (1H, d, J=8.2, 8-H), 8.39 (1H, d, J=2.2, 4-H), 9.27 (1H, d, J=2.2, 2-H).

3-(2-Furyl)quinoline (**3c$_2$**)

Column chromatography (CH_2Cl_2). Colorless prisms (from hexane), mp 75-76°C. Anal. Calcd for $C_{13}H_9N_2O$: C, 79.98; H, 4.65; N, 7.71. Found: C, 79.84; H, 4.54; N, 7.09. Ms m/z(%): 195(M^+, 100), 167(31), 166(36). Nmr: 6.55 (1H, dd, J=3.3, 1.8, 4'-H), 6.86 (1H, dd, J=3.3, 0.7, 3'-H), 7.54 (1H, ddd, J=8.1, 7.0, 1.1, 6-H), 7.57 (1H, s like, 5'-H), 7.67 (1H, ddd, J=8.4, 7.0, 1.5, 7-H), 7.83 (1H, dd, J=8.1, 1.5, 5-H), 8.08 (1H, d, J=8.4, 8-H), 8.34 (1H, d, J=2.2, 4-H), 9.21 (1H, d, J=2.2, 2-H).

3-(2-Thienyl)quinoline (**3d$_2$**)[10] and 3-(3-Thienyl)quinoline (**3d$_3$**)

Plc (hexane-acetone = 5:1). **3d$_2$**: Colorless plates (from hexane), mp 68-69°C. Exact Ms m/z Calcd for $C_{13}H_9NS$ (M^+): 211.0455. Found: 211.0426. Ms m/z(%): 211(M^+, 100), 210 (20). Nmr: 7.16 (1H, dd, J=5.1, 3.7, 4'-H), 7.39 (1H, dd, J=5.1, 1.1, 5'-H), 7.49 (1H, dd, J=3.7, 1.1, 3'-H), 7.55 (1H, ddd, J=8.1, 7.0, 1.1, 6-H), 7.68 (1H, ddd, J=8.1, 7.0, 1.5, 7-H), 7.83 (1H, dd, J=8.1, 1.5, 5-H), 8.10 (1H, d, J=8.1, 8-H), 8.26 (1H, d, J=2.2, 4-H), 9.20 (1H, d, J=2.2, 2-H). **3d$_3$** was identical with the authentic sample prepared by the reported method by comparison of their chromatographic behavior (glc, tlc) and ms.[3]

4-(1-Methylpyrrol-2-yl)quinoline (**4a$_2$**)

Column Chromatography (hexane-ether = 1 : 1). Picrate: prisms (from acetone), mp 199-201°C. Anal. Calcd for

$C_{20}H_{15}N_5O_7$: C, 54.92; H, 3.46; N, 16.01. Found: C, 55.08; H, 3.42; N, 15.92. Ms m/z (%): 208(M^+, 100), 207 (61), 206(24). Nmr: 3.45 (3H, s, N-CH_3), 6.34 (2H, dd, J=2.6, 1.8, 3'-H, 4'-H), 6.85 (1H, dd, J=2.6, 1.8, 5'-H), 7.30 (1H, d, J=4.4, 3-H), 7.49 (1H, ddd, J=8.0, 7.1, 1.8, 6-H), 7.70 (1H, ddd, J=8.4, 7.1, 1.8, 7-H), 7.88 (1H, d, J=8.0, 5-H), 8.15 (1H, d, J=8.4, 8-H), 8.92 (1H, d, J=4.4, 2-H).

4-(2-pyrrolyl)quinoline (**4b$_2$**)

Column chromatography (CH_2Cl_2-ethyl acetate = 5:1). Colorless plates (from benzene), mp 159.5-161°C . Anal. Calcd for $C_{13}H_{10}N_2$: *C, 80.38; H, 5.19; N, 14.42. Found: C, 80.48; H, 5.14; N, 14.31. Ms m/z(%): 194(M*$^+$, 100), 193(63), 167(20), 166(20). Nmr: 6.44 (1H, dd, J=3.6, 2.7, 4'-H), 6.71 (1H, dd, 3.6, 1.6, 3'-H), 7.06 (1H, dd, J=2.7, 1.6, 5'-H), 7.32 (1H, d, J=4.4, 3-H), 7.52 (1H, ddd, J=7.9, 7.0, 1.8, 6-H), 7.69 (1H, ddd, J=7.9, 7.0, 1.8, 7-H), 8.09 (1H, dd, J=7.9, 1.8, 8-H), 8.38 (1H, dd, J=7.9, 1.8, 5-H), 8.43 (1H, d, J=4.4, 2-H).

4-(2-furyl)quinoline (**4c$_2$**)

Column chromatography (hexane-ether-triethylamine = 20:2:1). Picrate: yellow needles (from ethanol). mp 214-215 °C. Ms m/z(%): 195(M^+, 91), 166(100). Nmr: 6.62 (1H, dd, J=3.7, 1.8, 4'-H), 6.97 (1H, dd, J=3.7, 0.7, 3'-H), 7.58 (1H, ddd, J=8.4, 7.0, 1.5, 6-H), 7.67 (1H, dd, J=1.8, 0.7, 5'-H), 7.72 (1H, ddd, J=8.4, 7.0, 1.1, 7-H), 7.61 (1H, d, J=4.4, 3-H), 8.15 (1H, dd, J=8.4, 1.1, 8-H), 8.49 (1H, dd, J=8.4, 1.1, 5-H), 8.91 (1H, d, J=4.4, 2-H).

4-(2-thienyl)quinoline (**4d$_2$**)

Plc (pre-treated with acetic acid) (hexane-ether = 1:1). Picrate: yellow needles (from ethanol), mp 212-213°C (lit. 204-206°C).[11] Ms m/z(%): 211 (M^+, 100), 210(75), 166(28). Nmr: 7.23 (1H, dd, J=5.1, 3.7, 4'-H), 7.39 (1H, dd, J=3.7, 1.3, 3'-H), 7.45 (1H, d, J=4.4, 3-H), 7.53 (1H, dd, J=5.1, 1.3, 5'-H), 7.57 (1H, ddd, J=8.4, 7.0, 1.1, 6-H), 7.75 (1H, ddd, J=8.4, 7.0, 1.5, 7-H), 8.17(1H, dd, J=8.4, 1.1, 8-H), 8.30 (1H, dd, J=8.4, 1.1, 5-H), 8.90 (1H, d, J=4.4, 2-H).

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