SYNTHESIS OF 2,4-DISUBSTITUTED ISOQUINOLIN-1(2H)-ONES BASED ON REACTIONS OF α-SUBSTITUTED 2-LITHIO-β-METHOXYSTYRENES WITH ISOCYANATES

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Abstract - An efficient method for the preparation of 2,4-disubstituted isoquinolin-1(2H)-ones is described. The reaction of α-substituted 2-lithio-β-methoxystyrenes, generated by treating α-substituted 2-bromo-β-methoxystyrenes with butyllithium, with isocyanates yields the corresponding α-substituted 2-(2-methoxyvinyl)benzamide derivatives, which in turn are transformed into 2,4-disubstituted isoquinolin-1(2H)-ones on treatment with a catalytic amount of concentrated hydriodic acid.

In a recent paper,¹ we demonstrated that α-substituted 2-lithio-β-methoxystyrenes, generated by a treatment of α-substituted 2-bromo-β-methoxystyrenes with butyllithium, reacted with various nitriles to give directly 1,4-disubstituted isoquinolines. As part of studies on the utility of these lithium products for organic synthesis, we have been interested in the reaction with isocyanates, which should give 2,4-disubstituted isoquinolin-1(2H)-ones. In this paper, we wish to report a convenient synthesis of 2,4-disubstituted isoquinolin-1(2H)-ones (3) from α-substituted 2-bromo-β-methoxystyrenes (1). We found that the reaction of α-substituted 2-lithio-β-methoxystyrenes with isocyanates gave only α-substituted 2-(2-methoxyvinyl)benzamide derivatives (2). However, these benzamides (2) could be converted into 2,4-disubstituted isoquinolin-1(2H)-ones (3) upon treatment with a catalytic amount of concentrated hydriodic acid. Recently, a number of efficient methods for the preparation of isoquinolin-1(2H)-one derivatives have been reported,² because of their biological activities.³ Moreover, some of them have been utilized as precursors for the synthesis of more complex and useful molecules.⁴ However, to date, there have been only a limited report on the synthesis of isoquinolin-1(2H)-one derivatives bearing substituents both at the 2- and 4-positions.⁵

Our two-step preparation of 2,4-disubstituted isoquinolin-1(2H)-ones (3) from α-substituted 2-bromo-β-methoxystyrenes (1) was conducted as illustrated in Scheme 1. The first step involves the reaction of...
α-substituted 2-lithio-β-methoxystyrenes, generated by the treatment of 1 with butyllithium in diethyl ether at 0 °C as described previously,\textsuperscript{1} with various isocyanates, affording the corresponding α-substituted 2-(2-methoxyvinyl)benzamide derivatives (2). As summarized in Table 1, the yields of 2 are generally fair to good, though those using t-butyl isocyanate are somewhat lower (Entries 7 and 9). These lower yields are considered to arise from the less reactivity of this isocyanate due to the bulkiness of the t-butyl group. Compounds (2) were obtained as mixtures of stereoisomers, and the ratios were determined by their \textsuperscript{1}H NMR spectra (see Experimental). However, the stereochemistry of each isomer could not be determined in every case.

\begin{table}
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\begin{tabular}{lllll}
\hline
Entry & 1 & R\textsuperscript{2} in RNCO & 2 (Yield/\textsuperscript{a}\textsuperscript{b}) & 3 (Yield/\textsuperscript{a}) \\
\hline
1 & 1a (R\textsuperscript{1} = H, R\textsuperscript{2} = Ph) & Ph & 2a (73) & 3a (67) \\
2 & 1a & o-Tol & 2b (74) & 3b (61) \\
3 & 1a & 4-CIC\textsubscript{6}H\textsubscript{4} & 2c (69) & 3c (75) \\
4 & 1b (R\textsuperscript{1} = H, R\textsuperscript{2} = 4-CIC\textsubscript{6}H\textsubscript{4}) & Ph & 2d (71) & 3d (66) \\
5 & 1b & 4-CIC\textsubscript{6}H\textsubscript{4} & 2e (62) & 3e (73) \\
6 & 1b & 3-MeOC\textsubscript{6}H\textsubscript{4} & 2f (73) & 3f (68) \\
7 & 1b & t-Bu & 2g (51) & 3g (64) \\
8 & 1c (R\textsuperscript{1} = H, R\textsuperscript{2} = Me) & 4-CIC\textsubscript{6}H\textsubscript{4} & 2h (65) & 3h (71) \\
9 & 1c & t-Bu & 2i (55) & 3i (69) \\
10 & 1d (R\textsuperscript{1} = OMe, R\textsuperscript{2} = Ph) & Ph & 2j (88) & 3j (63) \\
\hline
\end{tabular}
\caption{Preparation of Isoquinolin-1(2H)-ones (3) via 2-Vinylbenzamides (2)}
\end{table}

\textsuperscript{a}Isolated yields. \textsuperscript{b}Mixtures of stereoisomers.

Subsequent intramolecular replacement of the methoxy substituent in the methoxystyrene moiety with the amide nitrogen atom of 2 was easily achieved upon treatment of a catalytic amount of concentrated hydriodic acid in acetonitrile at 0 °C to room temperature to result in the formation of 3 in generally fair yields, independent of the substituents both at the 2- and 4-positions. These results are also summarized in Table 1.

In summary, we have shown that α-substituted 2-(2-methoxyvinyl)benzamide derivatives, which are readily prepared by reactions of α-substituted 2-lithio-β-methoxystyrenes with isocyanates, cyclize on treatment with concentrated hydriodic acid to afford 2,4-disubstituted isoquinoline-1(2H)-ones. Since these isoquinolinones derivatives are hard to obtain by conventional methods, the present new and simple method may find some value in organic synthesis. Further investigations on the development of methods
for the synthesis of heterocycles utilizing reactions of these lithium compounds with various electrophiles are now in progress in our laboratory, and results will be reported in near future.

EXPERIMENTAL

The melting points were determined on a Laboratory Devices MEL-TEMP II melting-point apparatus and are uncorrected. The IR spectra were recorded on a Shimadzu FTIR-8300 spectrometer. The $^1$H NMR spectra were determined using SiMe$_4$ as an internal reference with a JEOL ECP500 FT NMR spectrometer operating at 500 MHz. The $^{13}$C NMR spectra were determined in CDCl$_3$ using TMS as an internal reference with a JEOL ECP500 FT NMR spectrometer operating at 125 MHz. Low-resolution mass spectra (EI) were recorded on a JEOL AUTOMASS 20 spectrometer. Thin-layer chromatography (TLC) was carried out on Merck Kieselgel 60 PF$_254$. Column chromatography was performed using Merck Kieselgel 60 (0.063–0.200 mm). All of the solvents used were dried over appropriate drying agents and distilled under argon prior to use.

Starting Materials. 2-Bromostyrene derivatives (1a), (1b), (1c), and (1d) were prepared by appropriate reported procedures. All other chemicals used in this study were commercially available.

Typical Procedure for the Preparation of 2-(2-Methoxyvinyl)benzamides (2). 2-(2-Methoxy-1-phenylethenyl)-N-phenylbenzamide (2a). To a stirred solution of 1a (0.29 g, 1.0 mmol) in Et$_2$O (4 mL) at 0 °C was added dropwise n-BuLi (1.6M in hexane; 1.0 mmol). After 1 h, PhNCO (0.12 g, 1.0 mmol) was added and stirring was continued for an additional 30 min. The mixture was quenched with saturated aqueous NH$_4$Cl (15 mL) and extracted with Et$_2$O three times (10 mL each). The combined extracts were dried over anhydrous Na$_2$SO$_4$ and evaporated. The residue was purified by column chromatography on silica gel to afford 2a (0.24 g, 73%); a pale-yellow viscous oil; $R_f$ 0.40 (1:5 AcOEt–hexane); a mixture of stereoisomers (ca. 9:1); IR (neat) 3323, 1649, 1636, 1603 cm$^{-1}$; $^1$H NMR $\delta$ 3.71 (0.3H, s), 3.77 (2.7H, s), 6.40 (0.9H, s), 6.69 (0.1H, s), 7.04 (0.9H, tt, $J$ = 7.3, 1.4 Hz), 7.07 (0.1H, tt, $J$ = 7.3, 1.4 Hz), 7.13–7.29 (7H, m), 7.35 (0.9H, d, $J$ = 8.2 Hz), 7.36 (0.1H, d, $J$ = 8.2 Hz), 7.42–7.51 (4H, m), 7.67 (0.9H, br s), 7.85 (1H, dd, $J$ = 7.8, 1.8 Hz), 7.88 (0.1H, br s). Anal. Calcd for C$_{22}$H$_{19}$NO$_2$: C, 80.22; H, 5.81; N, 4.25. Found: C, 80.08; H, 5.85, N, 4.47.

2-(2-Methoxy-1-phenylethenyl)-N-(2-methylphenyl)benzamide (2b): a pale-yellow viscous oil; $R_f$ 0.14 (1:3 AcOEt–hexane); a mixture of stereoisomers (ca. 5:5); IR (neat) 3288, 1668, 1634 cm$^{-1}$; $^1$H NMR $\delta$ 1.93 (1.5H, s), 2.04 (1.5H, s), 3.70 (1.5H, s), 3.78 (1.5H, s), 6.42 (0.5H, s), 6.70 (0.5H, s), 6.99–7.05 (1H, m), 7.07–7.19 (4H, m), 7.22–7.26 (3H, m), 7.28 (0.5H, dd, $J$ = 7.8, 1.4 Hz), 7.34 (0.5H, d, $J$ = 7.3 Hz), 7.42–7.55 (2H, m), 7.57 (0.5H, br s), 7.66 (0.5H, d, $J$ = 7.8 Hz), 7.71 (0.5H, br s), 7.82 (0.5H, d, $J$ = 8.2 Hz), 7.87 (0.5H, dd, $J$ = 8.2, 1.8 Hz), 7.89 (0.5H, d, $J$ = 7.8 Hz). Anal. Calcd for C$_{25}$H$_{21}$NO$_2$: C, 80.44; H, 6.16; N, 4.08. Found: C, 80.31; H, 6.17; N, 3.99.

N-(4-Chlorophenyl)-2-(2-methoxy-1-phenylethenyl)benzamide (2c): a pale-yellow viscous oil; $R_f$ 0.14
(1:5 AcOEt–hexane); a mixture of stereoisomers (ca. 8:2); IR (neat) 3223, 1645, 1628 cm\(^{-1}\); \(^{1}\)H NMR \(\delta\) 3.70 (2.4H, s), 3.78 (0.6H, s), 6.39 (0.2H, s), 6.69 (0.8H, s), 7.08–7.51 (12H, m), 7.66 (0.2H, br s), 7.82 (1H, d, \(J = 7.3\) Hz), 7.88 (0.8H, br s). Anal. Calcd for \(C_{22}H_{18}ClNO_2\): C, 72.62; H, 4.99; N, 3.85. Found: C, 72.47; H, 5.27; N, 3.86.

2-[1-(4-Chlorophenyl)-2-methoxyethenyl]-N-phenylbenzamide (2d): a pale-yellow viscous oil; \(R_t\) 0.21 (1:4 AcOEt–hexane); a mixture of stereoisomers (ca. 7:3); IR (neat) 3288, 1652, 1636 cm\(^{-1}\); \(^{1}\)H NMR \(\delta\) 3.71 (0.9H, s), 3.79 (2.1H, s), 6.43 (0.7H, s), 6.65 (0.3H, s), 7.04–7.51 (12H, m), 7.59 (0.7H, br s), 7.79 (0.3H, br s), 7.82 (1H, d, \(J = 7.8\) Hz). Anal. Calcd for \(C_{22}H_{18}ClNO_2\): C, 72.62; H, 4.99; N, 3.85. Found: C, 72.49; H, 5.01, N, 3.71.

N-(4-Chlorophenyl)-2-[1-(4-chlorophenyl)-2-methoxyethenyl]benzamide (2e): a white solid; mp 140–160 °C; a mixture of stereoisomers (ca. 9:1); IR (neat) 3320, 1654, 1631, 1601 cm\(^{-1}\); \(^{1}\)H NMR \(\delta\) 3.71 (0.3H, s), 3.80 (2.7H, s), 6.42 (0.9H, s), 6.65 (0.1H, s), 7.14–7.22 (6H, m), 7.31–7.36 (3H, m), 7.44–7.51 (2H, m), 7.58 (0.9H, br s), 7.72 (0.1H, br s), 7.79 (1H, d, \(J = 7.3\) Hz). Anal. Calcd for \(C_{23}H_{20}ClNO_2\): C, 66.34; H, 4.30; N, 3.52. Found: C, 66.24; H, 4.30, N, 3.49.

2-[1-(4-Chlorophenyl)-2-methoxyethenyl]-N-(3-methoxyphenyl)benzamide (2f): a pale-yellow viscous oil; \(R_t\) 0.13 (1:4 AcOEt–hexane); a mixture of stereoisomers (ca. 5:5); IR (neat) 3300, 1659, 1634, 1605 cm\(^{-1}\); \(^{1}\)H NMR \(\delta\) 3.75 (1.5H, s), 3.78 (3H, s), 3.79 (1.5H, s), 6.18 (0.5H, s), 6.42 (0.5H, s), 6.59–7.50 (11H, m), 7.58 (0.5H, br s), 7.78–7.82 (1.5H, m). Anal. Calcd for \(C_{24}H_{20}ClNO_2\): C, 70.14; H, 5.12; N, 3.56. Found: C, 70.10; H, 5.26, N, 3.38.

2-[1-(4-Chlorophenyl)-2-methoxyethenyl]-N-(1,1-dimethylethyl)benzamide (2g): a pale-yellow oil; \(R_t\) 0.21 (1:3 AcOEt–hexane); a mixture of stereoisomers (ca. 7:3); IR (neat) 3400, 1653, 1634 cm\(^{-1}\); \(^{1}\)H NMR \(\delta\) 1.13 (6.3H, s), 1.23 (2.7H, s), 3.74 (0.9H, s), 3.83 (2.1H, s), 5.71 (0.7H, br s), 5.93 (0.3H, br s), 6.34 (0.7H, s), 6.71 (0.3H, s), 7.05 (0.6H, d, \(J = 8.7\) Hz), 7.18–7.25 (2.4H, m), 7.36–7.43 (4H, m), 7.67–7.71 (1H, m). Anal. Calcd for \(C_{20}H_{22}NO_2\): C, 65.51; H, 7.90; N, 4.77. Found: C, 65.48; H, 7.94, N, 4.76.

N-(4-Chlorophenyl)-2-(2-methoxy-1-methylethenyl)benzamide (2h): a pale-yellow oil; \(R_t\) 0.40 (1:30 THF–benzene); a mixture of stereoisomers (ca. 7:3); IR (neat) 3263, 3229, 1655, 1645 cm\(^{-1}\); \(^{1}\)H NMR \(\delta\) 1.88 (0.9H, d, \(J = 1.4\) Hz), 1.94 (2.1H, d, \(J = 0.9\) Hz), 3.59 (0.9H, s), 3.72 (2.1H, s), 6.12 (0.3H, q, \(J = 1.4\) Hz), 6.26 (0.7H, q, \(J = 0.9\) Hz), 7.23–7.55 (7H, m), 7.76 (0.3H, d, \(J = 7.3\) Hz), 7.81 (0.7H, dd, \(J = 7.3, 1.4\) Hz), 7.99 (0.7H, br s), 8.21 (0.3H, br s). Anal. Calcd for \(C_{17}H_{16}ClNO_2\): C, 67.66; H, 5.34; N, 4.64. Found: C, 67.41; H, 5.40; N, 4.59.

N-(1,1-Dimethylethyl)-2-(2-methoxy-1-methylethenyl)benzamide (2i): a pale-yellow solid; mp 65–70 °C; a mixture of stereoisomers (ca. 7:3); IR (KBr) 3267, 1666, 1626 cm\(^{-1}\); \(^{1}\)H NMR \(\delta\) 1.415 (2.7H, s), 1.420 (6.3H, s), 1.85 (0.9H, d, \(J = 1.4\) Hz), 1.94 (2.1H, d, \(J = 1.4\) Hz), 3.57 (0.9H, s), 3.69 (2.1H, s), 5.97 (0.7H, br s), 6.05 (0.3H, q, \(J = 1.4\) Hz), 6.14 (0.7H, q, \(J = 1.4\) Hz), 6.27 (0.3H, br s), 7.13–7.17 (1H, m), 7.28–7.39 (2H, m), 7.64 (0.7H, dd, \(J = 7.3, 1.4\) Hz), 7.67 (0.3H, dd, \(J = 7.8, 1.4\) Hz). Anal. Calcd for
C_{15}H_{21}NO₂: C, 72.84; H, 8.56; N, 5.66. Found: C, 72.59; H, 8.42; N, 5.53.

4-Methoxy-2-(2-methoxy-1-phenylethenyl)-N-phenylbenzamide (2j): a pale-yellow solid; mp 45–60 ºC; a mixture of stereoisomers (ca. 5:5); IR (KBr) 3223, 1649, 1636 cm⁻¹; ¹H NMR δ 3.71 (1.5H, s), 3.748 (1.5H, s), 3.754 (1.5H, s), 6.10 (0.5H, s), 6.48 (0.5H, s), 6.57 (0.5H, dd, J = 8.7, 2.7 Hz), 6.60 (0.5H, d, J = 2.7 Hz), 6.71 (0.5H, d, J = 2.7 Hz), 7.45 (2H, m). Anal. Calcd for C₃₁H₂₁NO: C, 84.86; H, 5.50; N, 4.50. Found: C, 84.67; H, 5.54; N, 4.46.

Typical Procedure for the Preparation of Isoquinolinone Derivatives (3). 2,4-Diphenylisoquinolin-1(2H)-one (3a).⁵ To a stirred solution of 2a (0.24 g, 0.72 mmol) in MeCN (7 mL) at 0 ºC was added a drop of concentrated HI. The mixture was allowed to warm to rt, and stirring was continued for 1.5 h before saturated aqueous NaHCO₃ (10 mL) was added. The organic materials were extracted with Et₂O (20 mL), and the extract was washed with brine and dried over anhydrous Na₂SO₄. Evaporation of the solvent gave a residue, which was purified by preparative TLC on silica gel to afford 3a (0.14 g, 67%); a white solid; mp 133–134 ºC (hexane–Et₂O) (lit.,⁵ mp 132–133 ºC). The IR and ¹H NMR data for this product were identical to those reported previously.⁵

2-(2-Methylphenyl)-4-phenylisoquinolin-1(2H)-one (3b): a white solid; mp 135–137 ºC (hexane–Et₂O); IR (KBr) 1662, 1624, 1603 cm⁻¹; ¹H NMR δ 2.24 (3H, s), 7.03 (1H, s), 7.29–7.37 (5H, m), 7.39–7.47 (4H, m), 7.56 (1H, ddd, J = 8.2, 6.9, 1.4 Hz), 7.63–7.68 (2H, m), 8.58 (1H, d, J = 7.8 Hz); ¹³C NMR δ 17.84, 119.57, 124.79, 126.39, 127.11, 127.57, 127.73, 128.64 (two overlapped C’s), 128.67, 128.86, 129.96 (two overlapped C’s), 131.11, 132.40, 135.42, 136.10, 136.58, 140.40, 161.20; MS m/z 311 (M⁺, 89), 294 (100). Anal. Calcd for C_{22}H_{17}NO: C, 84.86; H, 5.50; N, 4.50. Found: C, 84.67; H, 5.54; N, 4.46.

2-(4-Chlorophenyl)-4-phenylisoquinolin-1(2H)-one (3c): a white solid; mp 148–150 ºC (hexane–Et₂O) (lit.,² mp 148–149 ºC); IR (KBr) 1661, 1628, 1601 cm⁻¹; ¹H NMR δ 7.13 (1H, s), 7.41–7.50 (9H, m), 7.57 (1H, ddd, J = 8.2, 6.9, 1.4 Hz), 7.60 (1H, d, J = 8.2 Hz), 7.66 (1H, ddd, J = 8.2, 6.9, 1.4 Hz), 8.57 (1H, dd, J = 8.2, 1.4 Hz).

4-(4-Chlorophenyl)-2-phenylisoquinolin-1(2H)-one (3d): a white solid; mp 145–147 ºC (hexane–Et₂O); IR (KBr) 1661, 1626 cm⁻¹; ¹H NMR δ 7.17 (1H, s), 7.40 (2H, d, J = 8.2 Hz), 7.43–7.58 (9H, m), 7.66 (1H, td, J = 7.3, 1.4 Hz), 8.58 (1H, dd, J = 7.3, 1.4 Hz); ¹³C NMR δ 118.44, 124.46, 126.39, 126.83, 127.38, 128.22, 128.81, 128.91, 129.34, 131.24 (two overlapped C’s), 132.60, 133.82, 134.58, 136.15, 141.13, 161.49; MS m/z 331 (M⁺, 100). Anal. Calcd for C_{12}H_{11}ClNO: C, 76.36; H, 3.94; N, 4.24. Found: C, 76.17; H, 4.19; N, 4.08.

2,4-Bis(4-chlorophenyl)isoquinolin-1(2H)-one (3e): a white solid; mp 180–183 ºC (hexane–AcOEt); IR (KBr disk) 1657, 1622 cm⁻¹; ¹H NMR δ 7.11 (1H, s), 7.38 (2H, d, J = 8.7 Hz), 7.43 (2H, d, J = 8.7 Hz), 7.45 (2H, d, J = 8.7 Hz), 7.48 (2H, d, J = 8.7 Hz), 7.53 (1H, d, J = 8.2 Hz), 7.58 (1H, ddd, J = 8.2, 7.3, 1.4 Hz), 7.67 (1H, ddd, J = 8.2, 7.3, 1.4 Hz), 8.56 (1H, dd, J = 8.2, 1.4 Hz); MS m/z 365 (M⁺, 100). Anal.
Calcd for C$_2$H$_{13}$Cl$_2$NO: C, 68.87; H, 3.58; N, 3.82. Found: C, 68.93; H, 3.70; N, 3.73.

4-(4-Chlorophenyl)-2-(3-methoxyphenyl)isoquinolin-1(2H)-one (3f): a pale-yellow solid; mp 121–124 °C (Et$_2$O); IR (KBr) 1665, 1628, 1605 cm$^{-1}$; $^1$H NMR δ 3.85 (3H, s), 6.97 (1H, ddd, J = 8.2, 2.8, 0.9 Hz), 7.02–7.06 (2H, m), 7.16 (1H, s), 7.38–7.42 (3H, m), 7.45 (2H, d, J = 8.2 Hz), 7.54 (1H, d, J = 8.2 Hz), 7.57 (1H, ddd, J = 8.2, 7.3, 0.9 Hz), 7.66 (1H, ddd, J = 8.2, 7.3, 1.4 Hz), 8.58 (1H, dd, J = 8.2, 0.9 Hz); MS m/z 361 (M$^+$, 100). Anal. Calcd for C$_{22}$H$_{16}$ClNO: C, 73.03; H, 4.46; N, 3.87. Found: C, 73.07; H, 4.14; N, 3.84.

4-(4-Chlorophenyl)-2-(1,1-dimethylethyl)isoquinolin-1(2H)-one (3g): a white solid; mp 183–185 °C (hexane–Et$_2$O); IR (KBr) 1643, 1616 cm$^{-1}$; $^1$H NMR δ 1.76 (9H, s), 2.28 (3H, d, J = 8.7 Hz), 7.47 (2H, d, J = 8.7 Hz), 7.56 (1H, dd, J = 7.8, 7.3 Hz), 7.65 (1H, d, J = 7.8 Hz), 7.75 (1H, dd, J = 7.8, 7.3 Hz), 8.51 (1H, d, J = 7.8 Hz); MS m/z 269 (M$^+$, 100). Anal. Calcd for C$_{19}$H$_{16}$ClNO: C, 73.19; H, 5.82; N, 4.49. Found: C, 72.99; H, 5.86; N, 4.32.

2-(4-Chlorophenyl)-4-methylisoquinolin-1(2H)-one (3h): a white solid; mp 168–170 °C (hexane–Et$_2$O); IR (KBr) 1665, 1628, 1612 cm$^{-1}$; $^1$H NMR δ 2.32 (3H, s), 6.98 (1H, s), 7.39 (2H, d, J = 8.7 Hz), 7.47 (2H, d, J = 8.7 Hz), 7.56 (1H, dd, J = 7.8, 7.3 Hz), 7.65 (1H, d, J = 7.8 Hz), 7.75 (1H, dd, J = 7.8, 7.3 Hz), 8.51 (1H, d, J = 7.8 Hz); MS m/z 215 (M$^+$, 18), 159 (100). Anal. Calcd for C$_{14}$H$_{11}$NO: C, 78.10; H, 7.96; N, 6.51. Found: C, 78.16; H, 8.05; N, 6.29.

6-Methoxy-2,4-diphenylisoquinolin-1(2H)-one (3j): a white solid; mp 153–155 °C (hexane–Et$_2$O); IR (KBr disk) 1722, 1636, 1605 cm$^{-1}$; $^1$H NMR δ 3.81 (3H, s), 6.78 (1H, d, J = 2.3 Hz), 7.09 (1H, dd, J = 8.7, 2.3 Hz), 7.24 (1H, s), 7.39–7.41 (4H, m), 7.45–7.51 (6H, m), 8.33 (1H, d, J = 8.7 Hz); MS m/z (%) 327 (M$^+$, 13), 251 (94), 223 (100). Anal. Calcd for C$_{22}$H$_{17}$NO: C, 80.71; H, 5.23; N, 4.28. Found: C, 80.68; H, 5.41; N, 4.46.

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REFERENCES AND NOTES


