

Communication

## Hypervalent Iodine(III) Sulfonate Mediated Synthesis of Quinoxalines in Liquid PEG-400

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PEG-400[poly(ethylene glycol-400)] is used as a “green” recyclable solvent in the one-pot synthesis of quinoxalines by reaction with aryl ketones, hypervalent Iodine(III) Sulfonate, and *o*-phenylenediamines. Significant rate enhancements and improved yields have been observed.

**Keywords:** Hypervalent iodine; Quinoxalines; Poly(ethylene glycol).

### INTRODUCTION

Nitrogen-containing heteroaromatic and heterocyclic compounds are indispensable structural units for both the chemist and the biochemist. Quinoxalines constitute the basis of many insecticides, fungicides, herbicides and anthelmintics, as well as being important in human health and as receptor antagonists.<sup>1,2</sup> A number of methods have been developed for the synthesis of substituted quinoxalines involving condensation of 1,2-diamines with  $\alpha$ -diketones,<sup>3</sup> 1,4-addition of 1,2-diamines to diazenylbutenes,<sup>4</sup> oxidation-trapping of  $\alpha$ -hydroxy ketones with 1,2-diamine,<sup>5</sup> cyclization-oxidation of phenacyl bromides and *o*-phenylenediamines through solid-phase synthesis<sup>6</sup> and oxidative coupling of epoxides with ene-1,2-diamines.<sup>7</sup> Nevertheless, most of these methods suffer from unsatisfactory yields, difficult experimental procedures, expensive and detrimental metal precursors and harsh reaction conditions. Therefore, the development of improved methods for the synthesis of quinoxaline derivatives has acquired relevance to current research.

Poly(ethylene glycol) (PEG),<sup>8</sup> a biologically acceptable polymer used extensively in drug delivery and in bioconjugates as tools for diagnostics has been used as a solvent medium support for various transformations.<sup>9</sup> In recent times ionic liquids have been in the forefront of research, and several publications and reviews have already appeared.<sup>10</sup> Even though ionic liquids offer some advantages, the tedious preparation of ionic liquids (and raw ma-

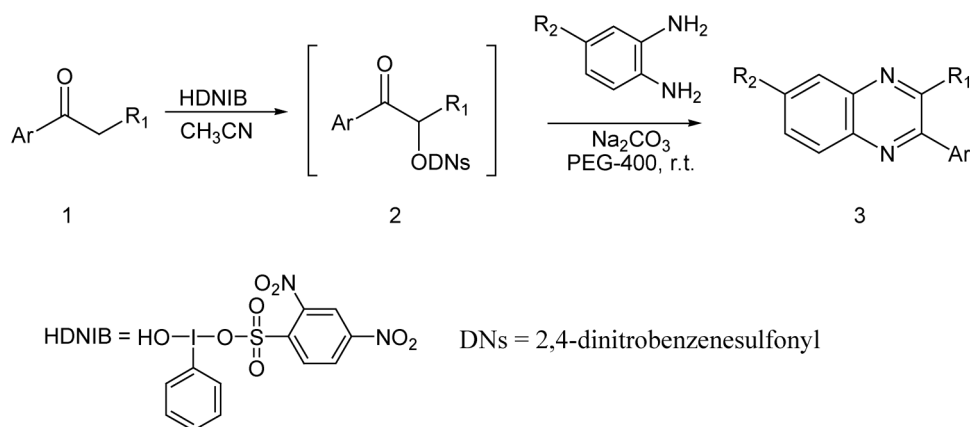
terials for ionic liquids) and their environmental safety is still debated. Compared with PEG, however, toxicity and environmental burden of ionic liquids are for the most part unknown. Furthermore, the cost of ionic liquids is often more expensive than that of PEG.<sup>11</sup> To date some of the more important reactions have been carried out and investigated in PEG, for example, Heck reaction,<sup>12</sup> Lindlar catalytic hydrogenation,<sup>13</sup> asymmetric dihydroxylation,<sup>14</sup> Baylis-Hillman reaction,<sup>15</sup> Biginelli reaction,<sup>16</sup> Suzuki-Miyaura reaction, Stille cross-coupling reaction,<sup>17</sup> Wacker reaction,<sup>18</sup> and asymmetric aldol reaction,<sup>19</sup> etc.

Recently, hypervalent iodine(III) reagents have been used extensively in organic synthesis due to their low toxicity, ready availability and easy handling.<sup>20</sup> In continuation of our efforts to develop greener organic reaction procedures, we now report [hydroxyl(2,4-dinitrobenzenesulfonyloxy)iido]benzene (HDNIB) mediated facile and efficient synthesis of quinoxalines. The required HDNIB was prepared in satisfactory yields from the reaction 2,4-dinitrobenzenesulfonic acid with phenyliodine(III) diacetate (PIDA).<sup>21</sup> Treatment of aromatic ketones with HDNIB in acetonitrile produced the  $\alpha$ -(2,4-dinitrobenzene)sulfonyloxy ketone intermediates (**2**). Subsequent cyclocondensation by *o*-phenylenediamines in PEG-400 gave the corresponding quinoxalines (**3**) in good yields (Scheme I).

### RESULTS AND DISCUSSION

The 2,4-dinitrobenzenesulfonyloxy group located at

Scheme I



the  $\alpha$  position to a carbonyl group represents an increasingly important entity in both mechanistic and synthetic organic chemistry. One reason for this importance is that 2,4-dinitrobenzenesulfonyloxy group is a good leaving group, and this accounts for the considerable synthetic utility associated with this group in functionalization of carbonyl compounds.

As shown in Scheme I, our experiments involving a one-pot procedure for the preparation of quinoxalines (**3**) by cyclocondensation of *o*-phenylenediamines with  $\alpha$ -sulfonyloxy aryl ketone (**2**) in PEG-400 at room temperature were successful. PEG-400 acts as a solvent medium and an activator in the cyclocondensation reaction. The results are summarized in the Table 1. An array of aryl ketones having electron-donating and -withdrawing substituents on the aromatic ring attached to carbonyl carbon reacted with *o*-phenylenediamine to afford 2-aryl substituted quinoxalines (**3b–3f**) in good yields. The reaction proceeds likewise with aryl ketones having heteroaryl group attached to carbonyl carbon to give the corresponding 2-heteroaryl substituted quinoxalines (**3j–3l**) in 89%, 87% and 90% yields, respec-

Table 1. Synthesis of quinoxalines **3a–m**

| Entry | Ar                                 | R <sub>1</sub>                | R <sub>2</sub> | Product   | Yield (%) |
|-------|------------------------------------|-------------------------------|----------------|-----------|-----------|
| 1     | C <sub>6</sub> H <sub>5</sub>      | H                             | H              | <b>3a</b> | 91        |
| 2     | 4-MeC <sub>6</sub> H <sub>4</sub>  | H                             | H              | <b>3b</b> | 87        |
| 3     | 4-MeOC <sub>6</sub> H <sub>4</sub> | H                             | H              | <b>3c</b> | 85        |
| 4     | 4-FC <sub>6</sub> H <sub>4</sub>   | H                             | H              | <b>3d</b> | 88        |
| 5     | 4-ClC <sub>6</sub> H <sub>4</sub>  | H                             | H              | <b>3e</b> | 84        |
| 6     | 4-BrC <sub>6</sub> H <sub>4</sub>  | H                             | H              | <b>3f</b> | 86        |
| 7     | C <sub>6</sub> H <sub>5</sub>      | Me                            | H              | <b>3g</b> | 87        |
| 8     | C <sub>6</sub> H <sub>5</sub>      | C <sub>6</sub> H <sub>5</sub> | H              | <b>3h</b> | 90        |
| 9     | C <sub>6</sub> H <sub>5</sub>      | C <sub>6</sub> H <sub>5</sub> | Me             | <b>3i</b> | 92        |
| 10    | 2-Pyridyl                          | H                             | H              | <b>3j</b> | 89        |
| 11    | 2-Furyl                            | H                             | H              | <b>3k</b> | 87        |
| 12    | 2-Thienyl                          | H                             | H              | <b>3l</b> | 90        |
| 13    | 2-Naphthyl                         | H                             | H              | <b>3m</b> | 86        |

tively. When the reaction was conducted in the classical molecular solvent, such as acetonitrile, the preparation of 2-phenylquinoxaline (**3a**) needs refluxing for 6 h; however, in PEG-400, the reaction took place at room temperature for 30 min and gave a higher yield (Table 2).

The PEG-400 can be typically recovered by extract-

Table 2. Effect of solvent on the cyclocondensation of  $\alpha$ -sulfonyloxy aryl ketone (**2a–c**) with *o*-phenylenediamine to form **3a–c**

| Entry | Solvent | Reaction temperature (°C) | Reaction time (h) | Quinoxaline <b>3</b> | Yield (%) |
|-------|---------|---------------------------|-------------------|----------------------|-----------|
| 1     | MeCN    | 80                        | 6                 | <b>3a</b>            | 80        |
| 2     | PEG-400 | 25                        | 0.5               | <b>3a</b>            | 91        |
| 3     | MeCN    | 80                        | 6                 | <b>3b</b>            | 78        |
| 4     | PEG-400 | 25                        | 0.5               | <b>3b</b>            | 87        |
| 5     | MeCN    | 80                        | 6                 | <b>3c</b>            | 77        |
| 6     | PEG-400 | 25                        | 0.5               | <b>3c</b>            | 85        |

ing out the product first and the recovered solvent can be reused without loss activity.

In conclusion, we have described a novel and efficient method for the synthesis of quinoxalines using PEG-400 as reaction medium. The important features of this procedure are enhanced reaction rate, mild reaction condition, high yields and green aspects such as avoiding hazardous organic solvents, toxic catalysts and waste, ease of recovery and reuse of this novel reaction medium.

## EXPERIMENTAL SECTION

All melting points are uncorrected. The IR spectra were recorded on a Shimadzu IR-27 G spectrophotometer.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were recorded on a Varian Unity Plus 400 MHz. Chemical shifts ( $\delta$ ) were measured in ppm with respect to TMS. MS were obtained on a JEOL JMS D-300 instrument.

### General procedure for the synthesis of quinoxalines (3)

To a solution of aryl ketone (**1**) (1.0 mmol) in acetonitrile (20 mL), was added HDNIB (1.2 mmol) and refluxed for 1 h. After removal of acetonitrile, Then PEG (2 g), *o*-phenylenediamine (1.0 mmol) and sodium carbonate (0.6 mmol) were added to the reaction mixture and was stirred at room temperature for 0.5 h to complete the reaction. Subsequently, the reaction mixture was extracted with  $\text{Et}_2\text{O}$  ( $3 \times 5$  mL). The remaining PEG suspension was filtered and reused for further runs. The combined ethereal solution was evaporated under reduced pressure. The resulting residue was chromatographed on silica gel using ethyl acetate as eluent to give **3**.

#### 2-Phenylquinoxaline (3a)

mp 74–75 °C (Lit.,<sup>22</sup> 75–76 °C). IR (KBr)  $\nu$ : 3119, 1614, 1560, 1076  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 7.55–7.58 (m, 3H), 7.74–7.82 (m, 2H), 8.11–8.22 (m, 4H), 9.34 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 127.5, 128.5, 129.1, 129.5, 129.6, 130.1, 130.2, 136.8, 141.5, 142.3, 143.3, 151.8; EI-MS  $m/z$  (relative intensity) 206 ( $\text{M}^+$ ), 179, 152.

#### 2-(4-Methylphenyl)quinoxaline (3b)

mp 92–93 °C (Lit.,<sup>22</sup> 90–91 °C). IR (KBr)  $\nu$ : 3053, 952, 748  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 2.46 (s, 3H), 7.38 (dd,  $J$  = 0.8, 8.8 Hz, 2H), 7.71–7.80 (m, 2H), 8.09–8.16 (m, 4H), 9.32 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 21.4, 127.4, 129.0, 129.3, 129.5, 129.8, 130.2, 133.9, 140.5, 141.4, 142.3, 143.3, 151.8; EI-MS  $m/z$  (relative intensity) 220 ( $\text{M}^+$ ), 193, 192.

#### 2-(4-Methoxyphenyl)quinoxaline (3c)

mp 97–98 °C (Lit.,<sup>22</sup> 99–100 °C). IR (KBr)  $\nu$ : 3057, 2360, 954, 759  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 3.90 (s, 3H), 7.07 (dd,  $J$  = 2.0, 6.8 Hz, 2H), 7.68–7.77 (m, 2H), 8.09 (t,  $J$  = 8.0 Hz, 2H), 8.15–8.18 (m, 2H), 9.28 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 55.3, 114.5, 128.9, 129.0, 129.1, 129.3, 130.1, 141.1, 142.2, 143.0, 151.4, 161.4; EI-MS  $m/z$  (relative intensity) 236 ( $\text{M}^+$ ), 221, 209, 193, 166.

#### 2-(4-Fluorophenyl)quinoxaline (3d)

mp 119–120 °C (Lit.,<sup>22</sup> 120–121 °C). IR (KBr)  $\nu$ : 3048, 2361, 956, 755  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 7.22–7.28 (m, 2H), 7.72–7.81 (m, 2H), 8.11–8.14 (m, 2H), 8.18–8.22 (m, 2H), 9.29 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 116.3, 129.1, 129.4, 129.5, 130.3, 132.8, 132.9, 141.4, 142.1, 142.8, 150.7, 165.4; EI-MS  $m/z$  (relative intensity) 225, 224 ( $\text{M}^+$ ), 197, 196.

#### 2-(4-Chlorophenyl)quinoxaline (3e)

mp 134 °C. IR (KBr)  $\nu$ : 3056, 2360, 955, 760  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 7.52–7.55 (m, 2H), 7.74–7.82 (m, 2H), 8.11–8.17 (m, 4H), 9.30 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 128.7, 129.1, 129.3, 129.5, 129.7, 130.4, 135.1, 136.5, 141.6, 142.1, 142.8, 150.5; EI-MS  $m/z$  (relative intensity) 242, 240 ( $\text{M}^+$ ), 213, 205, 178, 151; Anal. Calcd for  $\text{C}_{14}\text{H}_9\text{ClN}_2$ : C, 69.86; H, 3.77; N, 11.64. Found: C, 69.75; H, 3.68; N, 11.78.

#### 2-(4-Bromophenyl)quinoxaline (3f)

mp 128 °C. IR (KBr)  $\nu$ : 3057, 2359, 955, 760  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 7.68–7.71 (m, 2H), 7.74–7.82 (m, 2H), 8.07–8.15 (m, 4H), 9.29 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 124.9, 128.9, 129.1, 129.5, 129.7, 130.4, 132.3, 135.6, 141.6, 142.7, 150.6; EI-MS  $m/z$  (relative intensity) 287, 286, 285 ( $\text{M}^+$ ), 284, 205, 178, 151; Anal. Calcd for  $\text{C}_{14}\text{H}_9\text{BrN}_2$ : C, 58.97; H, 3.18; N, 9.82. Found: C, 59.23; H, 3.26; N, 9.74.

#### 2-Methyl-3-phenyl-quinoxaline (3g)

mp 55 °C (Lit.,<sup>23</sup> 56 °C). IR (KBr)  $\nu$ : 3058, 2359, 1558, 1342, 816, 764  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 2.78 (s, 3H), 7.46–7.54 (m, 3H), 7.63–7.75 (m, 4H), 8.04–8.12 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 24.3, 128.2, 128.4, 128.8, 128.9, 129.1, 129.6, 138.9, 140.8, 141.1, 152.4, 154.8; EI-MS  $m/z$  (relative intensity) 220 ( $\text{M}^+$ ), 219, 179, 151.

#### 2,3-Diphenylquinoxaline (3h)

mp 123–124 °C (Lit.,<sup>24</sup> 126–127 °C). IR (KBr)  $\nu$ : 3054, 2922, 1539, 1344, 768, 729  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 7.32–7.40 (m, 6H), 7.52–7.54 (m, 4H), 7.75 (m, dd,  $J$  =

2.4, 9.2 Hz, 2H), 8.20 (dd,  $J = 2.4, 8.8$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 128.2, 128.7, 129.1, 129.7, 129.9, 139.0, 141.1, 153.4; EI-MS  $m/z$  (relative intensity) 282 ( $\text{M}^+$ ), 178, 152, 140, 77.

#### 6-Methyl-2,3-diphenylquinoxaline (3i)

mp 118–119 °C (Lit.,<sup>24</sup> 116–117 °C). IR (KBr)  $\nu$ : 3055, 2940, 1617, 1199, 1020, 699  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 2.60 (s, 3H), 7.30–7.33 (m, 6H), 7.49–7.52 (m, 4H), 7.58 (dd,  $J = 2.4, 11.2$  Hz, 1H), 8.06 (d,  $J = 11.2$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 21.8, 127.9, 128.1, 128.5, 128.6, 129.7, 129.7, 132.2, 139.1, 140.3, 142.2, 152.4, 153.2; EI-MS  $m/z$  (relative intensity) 296 ( $\text{M}^+$ ), 192, 165, 89.

#### 2-(2-Pyridyl)quinoxaline (3j)

mp 110–111 °C (Lit.,<sup>25</sup> 113–114 °C). IR (KBr)  $\nu$ : 3048, 2923, 1589, 1401, 805, 770  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 7.38–7.41 (m, 1H), 7.74–7.81 (m, 2H), 7.86–7.91 (m, 1H), 8.13–8.17 (m, 2H), 8.57–8.60 (m, 1H), 8.77–8.78 (m, 1H), 9.96 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 121.9, 124.5, 139.2, 129.6, 129.9, 130.0, 137.0, 141.7, 142.4, 144.0, 149.3, 150.0, 154.4; EI-MS  $m/z$  (relative intensity) 207 ( $\text{M}^+$ ), 179, 105, 79.

#### 2-(2-Furanyl)quinoxaline (3k)

mp 99–100 °C (Lit.,<sup>22</sup> 97–98 °C). IR (KBr)  $\nu$ : 3117, 2364, 961, 755  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 6.64 (dd,  $J = 2.0, 3.6$  Hz, 1H), 7.33 (d,  $J = 3.2$  Hz, 1H), 7.69–7.78 (m, 3H), 8.09 (ddd,  $J = 1.6, 8.0, 12.8$  Hz, 2H), 9.25 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 111.7, 112.4, 126.9, 130.4, 130.8, 141.2, 142.0, 143.8, 144.2, 145.0, 151.5; EI-MS  $m/z$  (relative intensity) 196 ( $\text{M}^+$ ), 169, 168, 141, 140, 114.

#### 2-(2-Thienyl)quinoxaline (3l)

mp 118–119 °C (Lit.,<sup>25</sup> 120–121 °C). IR (KBr)  $\nu$ : 3052, 2360, 927, 756  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 7.20 (ddd,  $J = 0.4, 4.0, 4.8$  Hz, 1H), 7.54 (dd,  $J = 0.4, 4.4$  Hz, 1H), 7.67–7.76 (m, 2H), 7.85 (d,  $J = 3.6$  Hz, 1H), 8.05–8.07 (m, 2H), 9.23 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 126.9, 128.4, 129.0, 129.1, 129.7, 130.3, 141.3, 142.0, 142.1, 142.2, 147.3; EI-MS  $m/z$  (relative intensity) 212 ( $\text{M}^+$ ), 211, 185, 141.

#### 2-(2-Naphthyl)quinoxaline (3m)

mp 141–142 °C (Lit.,<sup>22</sup> 140–142 °C). IR (KBr)  $\nu$ : 3054, 2355, 1541, 1358, 825, 743  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 7.55–7.59 (m, 2H), 7.74–7.83 (m, 2H), 7.91–7.93 (m, 1H), 8.01–8.04 (m, 2H), 8.15 (dd,  $J = 1.2, 8.0$  Hz, 1H), 8.20 (d,  $J = 8.0$  Hz, 1H), 8.37 (d,  $J = 0.8, 8.4$  Hz, 1H), 8.66 (s, 1H), 9.49 (s, 1);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$ : 124.4, 126.6, 127.2, 127.4, 127.7, 128.8, 129.0, 129.1, 129.5,

129.5, 130.3, 133.3, 134.0, 134.0, 141.5, 142.3, 143.4, 151.6; EI-MS  $m/z$  (relative intensity) 256 ( $\text{M}^+$ ), 202, 153, 126.

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