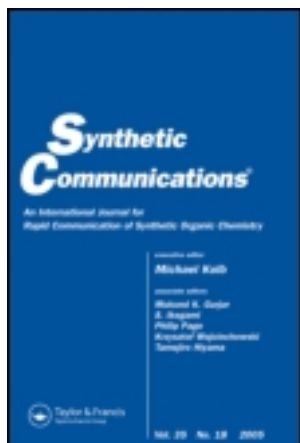


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### CONDENSATION OF O-PHENYLENEDIAMINE WITH CINNAMIC ACIDS

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## CONDENSATION OF *o*-PHENYLENEDIAMINE WITH CINNAMIC ACIDS

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### ABSTRACT

Condensation of *o*-phenylenediamine sulfate with cinnamic acids in refluxing ethylene glycol yielded the corresponding 2-styrylbenzimidazoles in excellent yields.

### INTRODUCTION

In an earlier communication,<sup>1</sup> we have reported the preparation of 2-styrylbenzimidazoles by condensation of 2-methyl/ethylbenzimidazoles with aromatic aldehydes. Literature survey revealed that *o*-phenylenediamine condenses with aliphatic acids in aq. HCl (Phillips conditions)<sup>2</sup> and

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with aromatic acids in PPA<sup>3</sup> or PPE<sup>4</sup> at 180–200°C, and it appears that there is no standard method for the condensation of unsaturated acids with *o*-phenylenediamine. Hence, it was considered worthwhile to develop a simple and efficient method for the condensation of *o*-phenylenediamine with unsaturated acids, which forms the subject matter of this paper.

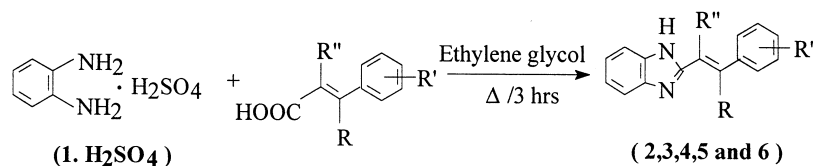
### RESULTS AND DISCUSSION

Attempted condensation of *o*-phenylenediamine (**1**) with cinnamic acid in refluxing methanol containing a trace of sulfuric acid or in refluxing benzene with a trace of *p*-toluenesulphonic acid, and removal of water by the Dean–Stark apparatus led to the recovery of starting materials. However, condensation of **1** with cinnamic acid in PPA at 200°C, yielded 2-styrylbenzimidazole (**2a**) in 30% yield.

Nagarajan has reported the synthesis of **2a** in 57% yield by condensation of *o*-phenylenediamine dihydrochloride with cinnamic acid at 200°C, for 2 h.<sup>5</sup> Treatment of **1** with cinnamic acid under Phillips' conditions led to the recovery of starting materials.

In the present work, condensation of *o*-phenylenediamine sulfate (**1.H<sub>2</sub>SO<sub>4</sub>**) with cinnamic acid in refluxing ethylene glycol for 3 h followed by simple processing yielded **2a** in 95% yield. This condensation of **1.H<sub>2</sub>SO<sub>4</sub>**, with cinnamic acid has been extended to fourteen more cinnamic acids to obtain the corresponding 2-styrylbenzimidazoles in excellent yields. The results are shown in the Table. The authenticity of **2a–2f** and **3a–3f** was confirmed by comparison of mp, mmp, tlc and IR spectra with those of the products obtained by condensation of 2-methyl/ethylbenzimidazoles with aromatic aldehydes as reported earlier.<sup>1</sup> The structures **4,5** and **6** are assigned based on their spectral and analytical data.

This appears to be a general method and may be applied for the preparation of other benzimidazole derivatives with unsaturated moiety in 2-position, which are difficult to obtain by other known methods. Thus, compounds **5** and **6**, which cannot be prepared by the condensation of



**Table.** Condensation of 1.H<sub>2</sub>SO<sub>4</sub> with Cinnamic Acids to Obtain 2,3,4,5 and 6

Cinnamic acid used	Product obtained			Yield (%)	Rexn. solvent	M.P. (°C)	
	R	R'	R''				
Cinnamic acid	<b>2a</b>	H	H	H	95	Aq. CH <sub>3</sub> OH	203–05 (Lit <sup>11</sup> m.p. = 201–02)
<i>p</i> -Methylcinnamic acid	<b>2b</b>	H	4-CH <sub>3</sub>	H	92	Aq. CH <sub>3</sub> OH	214–16
<i>o</i> -Chlorocinnamic acid	<b>2c</b>	H	2-Cl	H	99	Aq. CH <sub>3</sub> OH	176–78
<i>p</i> -Chlorocinnamic acid	<b>2d</b>	H	4-Cl	H	96	Aq. CH <sub>3</sub> OH	221–23 (Lit <sup>12</sup> m.p. = 223–224)
<i>m</i> -Nitrocinnamic acid	<b>2e</b>	H	3-NO <sub>2</sub>	H	97	EtOH + Hexane	219–20
<i>p</i> -Nitrocinnamic acid	<b>2f</b>	H	4-NO <sub>2</sub>	H	96	AcOH	260–62
$\alpha$ -Methylcinnamic acid	<b>3a</b>	H	H	CH <sub>3</sub>	91	CH <sub>3</sub> OH	242–44 (Lit <sup>13</sup> m.p. = 245)
<i>p</i> -Methyl- $\alpha$ -methylcinnamic acid	<b>3b</b>	H	4-CH <sub>3</sub>	CH <sub>3</sub>	94	EtOAc	178–80
<i>o</i> -Chloro- $\alpha$ -methylcinnamic acid	<b>3c</b>	H	2-Cl	CH <sub>3</sub>	95	Aq. CH <sub>3</sub> OH	264–65
<i>p</i> -Chloro- $\alpha$ -methylcinnamic acid	<b>3d</b>	H	4-Cl	CH <sub>3</sub>	97	Aq. CH <sub>3</sub> OH	223–25
<i>m</i> -Nitro- $\alpha$ -methylcinnamic acid	<b>3e</b>	H	3-NO <sub>2</sub>	CH <sub>3</sub>	93	Aq. CH <sub>3</sub> OH	231–33
<i>p</i> -Nitro- $\alpha$ -methylcinnamic acid	<b>3f</b>	H	4-NO <sub>2</sub>	CH <sub>3</sub>	98	Aq. CH <sub>3</sub> OH	294–95
$\alpha$ -Phenylcinnamic acid	<b>4</b>	H	H	C <sub>6</sub> H <sub>5</sub>	92	Aq. CH <sub>3</sub> OH	281–83
$\beta$ -Methylcinnamic acid	<b>5</b>	CH <sub>3</sub>	H	H	90	Aq. CH <sub>3</sub> OH	195–96
$\beta$ -Phenylcinnamic acid	<b>6</b>	C <sub>6</sub> H <sub>5</sub>	H	H	94	EtOAc + Hexane	102–04

2-methylbenzimidazole with acetophenone and benzophenone respectively can be prepared by this method.

## EXPERIMENTAL

All melting points are uncorrected and were obtained using open capillary tubes in sulfuric acid bath. TLC analysis was carried out on glass plates coated with silica gel-G, and spots were visualized using iodine or a UV Lamp. IR spectra were recorded using Perkin–Elmer Model 46 instrument in KBr discs. <sup>1</sup>H-NMR and <sup>13</sup>C-NMR spectra were recorded on a Bruker instrument operating at 250 MHz and 62.9 MHz



respectively. Mass spectra were recorded on a CEC-21-110 mass spectrometer under electron impact conditions.

### Preparation of 1.H<sub>2</sub>SO<sub>4</sub>

To a solution of **1** (5.4 gms, 50 mM) in methanol (50 ml), conc. H<sub>2</sub>SO<sub>4</sub> (4 ml) was added, drop wise under ice-cold conditions over a period of 30 min. After the completion of addition, the separated product was filtered, washed with methanol and dried. Yield = 7.21 gms (70%) (Anal. Calcd for C<sub>6</sub>H<sub>10</sub>N<sub>2</sub>O<sub>4</sub>S: %C, 30.762; %H, 4.301; %N, 13.561; Found: %C, 30.760; %H, 4.299; %N, 13.560).

### Condensation of 1.H<sub>2</sub>SO<sub>4</sub> with Cinnamic Acids<sup>6-10</sup> to Obtain 2,3,4,5 and 6

A mixture of 1.H<sub>2</sub>SO<sub>4</sub> (2.06 g, 10 mM) and substituted cinnamic acid (10 mM) in ethylene glycol (10 ml) was refluxed for 3 h. Then, the reaction mixture was cooled to RT and poured into water (100 ml). The pH of the solution was adjusted to >7 with NaHCO<sub>3</sub>. The product was filtered, washed, dried and recrystallized to obtain **2,3,4,5** and **6**.

### Spectral Data of Compounds 2a-6

**2a** <sup>1</sup>H-NMR (CDCl<sub>3</sub>/TMS): δ 7.2-7.8 (complex m, 11H, five phenyl protons, four aryl protons and two vinylic protons, <sup>3</sup>J<sub>H-H</sub> for vinylic system = 16.5 Hz), 12.04 (1H, NH); <sup>13</sup>C-NMR (CDCl<sub>3</sub>/TMS): δ 114.93, 117.10, 122.61, 126.93, 128.76, 128.82, 135.36, 135.79, 139.23, 151.65 (for all carbons of 2-styrylbenzimidazole); MS: *m/z* (%I): 221 (6, M + 1), 220 (40, M<sup>+</sup>), 219 (100, M - 1), 218 (12, M - 2), 110 (8), 109 (12), 108 (4), 92 (4), 91 (6), 77 (3), 51 (2) etc. (Anal. Calcd for C<sub>15</sub>H<sub>12</sub>N<sub>2</sub>: %C, 81.791; %H, 5.490; %N, 12.717; Found: %C, 81.789; %H, 5.488; %N, 12.71).

**2b** <sup>1</sup>H-NMR (CDCl<sub>3</sub>/TMS): δ 2.31 (s, 3H, CH<sub>3</sub>), 7.12-7.66 (complex m, 10H, four *p*-methyl phenyl protons, four aryl protons and two vinylic protons, <sup>3</sup>J<sub>H-H</sub> = 16.63 Hz). <sup>13</sup>C-NMR (CDCl<sub>3</sub>/TMS): δ 20.97 (CH<sub>3</sub>), 116.68, 122.02, 126.99, 129.60, 133.02, 134.32, 138.53, 151.12 (six *p*-methyl-phenyl carbons, one imidazole quaternary carbon and two vinylic carbons); MS: *m/z* (%I): 235 (6.6, M + 1), 234 (41.7, M<sup>+</sup>), 233 (100, M - 1), 232 (10.2), 116 (12), 103 (3.7), 92 (1.2), 91 (1.9), 90 (1.2), 78 (2.7), 77 (2.3), 65 (2.3) etc.



(Anal. Calcd for C<sub>16</sub>H<sub>14</sub>N<sub>2</sub>: %C, 82.020; %H, 6.022; %N, 11.952; Found: %C, 82.019; %H, 6.020; %N, 11.950).

**2c** <sup>1</sup>H-NMR (CDCl<sub>3</sub>/TMS): δ 7.2–7.7 Complex m, 9H, four *o*-chlorophenyl protons, four aryl protons and one vinylic proton α-to heteryl ring), 7.88 (d, 1H, vinylic proton β-to heteryl ring); **MS: *m/z* (%I)**: 254 and 256 (13.4 and 1.5, M<sup>+</sup> corresponding to <sup>35</sup>Cl and <sup>37</sup>Cl), 253 and 255 (14.9 and 4.47, M–1), 220 (13.4), 219 (100, M-Cl), 218 (20.9), 129 (5.2), 77 (7.5), 65 (8.95), 64 (16.4) etc. (Anal. Calcd for C<sub>15</sub>H<sub>11</sub>N<sub>2</sub>Cl: %C, 70.731; %H, 4.352; %N, 10.997; Found: %C, 70.730; %H, 4.351; %N, 10.995).

**2d** <sup>1</sup>H-NMR (CDCl<sub>3</sub>/TMS): δ 7.14–7.72 (Complex m, 10H, four *p*-chlorophenyl protons, four aryl protons and two vinylic protons), 12.7 (bs, 1H, -NH); <sup>13</sup>C-NMR (CDCl<sub>3</sub>/TMS): δ 111.19, 118.55, 122.16, 128.72, 128.98, 132.92, 133.20, 134.74, 151.56, (six *p*-chlorophenyl carbons, six aryl carbons, one imidazole quaternary carbon and two vinylic carbons) etc; **MS: *m/z* (%I)**: 256, 254 (14.3, 42.9; M+2, M<sup>+</sup>) 255, 253 (39.3, 100; [(M+2)-1, M-1]), 219 (6), 218 (8), 109 (8.6). (Anal. Calcd for C<sub>15</sub>H<sub>11</sub>N<sub>2</sub>Cl: %C, 70.731; %H, 4.352; %N, 10.997; Found: %C, 70.730; %H, 4.351; %N, 10.995).

**2e** <sup>1</sup>H-NMR (CDCl<sub>3</sub>/TMS): δ 7.1–8.2 (Complex m, 10H, four *m*-nitrophenyl protons, four aryl protons and two vinylic protons); **MS: *m/z* (%I)**: 266 (6.63, M+1), 265 (55, M<sup>+</sup>), 264 (100, M–1), 236 (6.73), 235 (20.78), 234 (34.54), 219 (10.73), 218 (30.8), 217 (16.25), 43 (10.16), 42 (9.74); HRMS: calculated for C<sub>15</sub>H<sub>11</sub>N<sub>3</sub>O<sub>2</sub><sup>+</sup> = 265.085 amu; found = 265.0859 +/–0.0008 amu. (Anal. Calcd for C<sub>15</sub>H<sub>11</sub>N<sub>2</sub>O<sub>2</sub>: %C, 67.917; %H, 4.179; %N, 15.840; Found: %C, 67.916; %H, 4.178; %N, 15.839).

**2f** <sup>1</sup>H-NMR (CDCl<sub>3</sub>/TMS): δ 7.21 and 7.50 (complex m, 4H, aryl protons), 7.40, 7.56, 7.72 and 7.78 (AB quartet, 2H, vinylic protons, <sup>3</sup>J<sub>H-H</sub> = 16.55 Hz), 7.89, 7.93, 8.22 and 8.25 (A<sub>2</sub>B<sub>2</sub> quartet, 4H, *p*-nitrophenyl protons, J<sub>ortho</sub> = 8.2 Hz); <sup>13</sup>C-NMR (CDCl<sub>3</sub>/TMS): δ 115.13, 122.06, 122.57, 124.16, 128.00, 131.90, 142.46, 146.92 and 150.60 (six *p*-nitrophenyl carbons, six aryl carbons, one imidazole quaternary carbon and two vinylic carbons); **MS: *m/z* (%I)**: 266 (10, M+1), 265 (55, M<sup>+</sup>), 264 (100, M–1), 234 (10), 219 (21), 218 (67), 217 (30), 206 (8). (Anal. Calcd for C<sub>15</sub>H<sub>11</sub>N<sub>3</sub>O<sub>2</sub>: %C, 67.917; %H, 4.179; %N, 15.840; Found: %C, 67.916; %H, 4.178; %N, 15.839).

**3a** <sup>1</sup>H-NMR (CDCl<sub>3</sub>/TMS): δ 7.12–7.49 (complex m, 9H, four aryl and five phenyl protons), 7.57 (s, 1H, vinylic proton), 12.58 (s, 1H, -NH); <sup>13</sup>C-NMR (CDCl<sub>3</sub>/TMS): δ 15.30 (CH<sub>3</sub>), 122.00, 127.51, 127.59, 128.55, 129.32, 130.26, 136.52 and 154.00 (six phenyl carbons, six aryl carbons, one imidazole quaternary carbon and two vinylic carbons); **MS: *m/z* (%I)**: 236 (0.14, M+2), 235 (2.30, M+1), 234 (56.9, M<sup>+</sup>), 233 (100, M–1), 232 (10.2), 219 (8.44), 157 (44.32), 155 (8.36), 141 (5.34), 129



(12.7), 127 (10.42) and 117 (2.85); HRMS: found = 233.1079 amu; calculated for  $C_{16}H_{13}N_2^+$  ( $M-1$ ) is 233.1079 amu. (Anal. Calcd for  $C_{16}H_{14}N_2$ : %C, 82.020; %H, 6.022; %N, 11.956; Found: %C, 82.019; %H, 6.021; %N, 11.954).

**3b**  $^1H$ -NMR ( $CDCl_3/TMS$ ):  $\delta$  2.35–2.45 (two singlets, 6H,  $2 \times CH_3$ ), 7.1–7.8 (complex, 9H, four *p*-methylphenyl protons, four aryl protons and one vinylic protons), 12.6 (s, 1H, -NH);  $^{13}C$ -NMR ( $CDCl_3/TMS$ ):  $\delta$  20.27 (Phenyl  $CH_3$ ), 15.30 (allylic  $CH_3$ ), 111.01, 118.73, 121.08, 121.37, 122.48, 126.76, 129.16, 129.31, 130.25, 133.68, 134.96, 136.98, 143.60, 154.17 (six *p*-methylphenyl carbons, six aryl carbons, one quaternary imidazole carbon and two vinylic carbons); MS:  $m/z$  (%I): 249 (2,  $M+1$ ), 248 (19,  $M^+$ ), 247 (100,  $M-1$ ), 246 (3), 245 (1), 234 (2), 233 (3), 232 (2), 128 (0.7), 116 (07); HRMS calcd for  $C_{17}H_{15}N_2^+$  ( $M-1$ ) = 247.1235 amu; found = 247.1236 amu. (Anal. Calcd for  $C_{17}H_{16}N_2$ : %C, 82.222; %H, 6.494; %N, 11.280; Found: %C, 82.221; %H, 4.493; %N, 11.278).

**3c**  $^1H$ -NMR ( $CDCl_3/TMS$ ):  $\delta$  2.36 (s, 3H,  $CH_3$ ), 7.16–7.62 (complex m, 9H, four *o*-chlorophenyl protons, four aryl protons and one vinylic proton); MS:  $m/z$  (%I): 268 and 270 (2.23 and 3,  $M^+$  corresponding to  $^{35}Cl$  and  $^{37}Cl$ ), 267 and 269 (18.6 and 4.47,  $M-1$ ), 234 (14.18), 233 (100,  $M-C1$ ), 232 (10.45), 116 (6.95), 115 (14.92), 66 (10.44), 65 (9.7), 64 (14.92) etc. (Anal. Calcd for  $C_{16}H_{13}N_2Cl$ : %C, 71.508; %H, 4.875; %N, 10.423; Found: %C, 71.506; %H, 4.873; %N, 10.421).

**3d**  $^1H$ -NMR ( $CDCl_3/TMS$ ):  $\delta$  2.45 (s, 3H,  $CH_3$ ), 7.1–7.8 (complex m, 9H, four *p*-chlorophenyl protons, four aryl protons and one vinylic proton), 12.7 (s, 1H, NH);  $^{13}C$ -NMR ( $CDCl_3/TMS$ ):  $\delta$  15.25 ( $CH_3$ ), 111.11, 118.85, 121.51, 122.67, 128.37, 128.57, 128.93, 131.05, 132.03, 134.94, 135.37, 143.56, 153.75 (six *p*-chlorophenyl carbons, six aryl carbons, one imidazole quaternary carbon and two vinylic carbons); MS:  $m/z$  (%I): 268 and 270 (insignificant intensity, twin peaks,  $M^+$  corresponding to  $^{35}Cl$  and  $^{37}Cl$ ), 267 and 269 ( $M-1$  corresponding to  $^{35}Cl$  and  $^{37}Cl$ ). (Anal. Calcd for  $C_{16}H_{13}N_2Cl$ : %C, 71.508; %H, 4.875; %N, 10.423; Found: %C, 71.506; %H, 4.873; %N, 10.421).

**3e**  $^1H$ -NMR ( $CDCl_3/TMS$ ):  $\delta$  2.51 (s, 3H,  $CH_3$ ), 7.27–8.22 (complex m, 8H, four *m*-nitrophenyl protons and four aryl protons), 8.3 (s, 1H, vinylic proton); MS:  $m/z$  (%I): 279 (23.9,  $M^+$ ), 278 (100,  $M-1$ ), 248 (26.86), 233 (17.9), 232 (67.1), 231 (35.8), 218 (17.9), 140 (7.46), 115 (20.9), 103 (10.44), 102 (9.7), 78 (24.6), 76 (12.7), 66 (32), 65 (19.4), 64 (32) etc. (Anal. Calcd for  $C_{16}H_{13}N_2O_2$ : %C, 68.800; %H, 4.691; %N, 15.045; Found: %C, 68.800; %H, 4.690; %N, 15.044).

**3f**  $^1H$ -NMR ( $CDCl_3/TMS$ ):  $\delta$  2.45 (s, 3H,  $CH_3$ ), 7.1–8.4 (complex m, 9H, four *p*-nitrophenyl protons, four aryl protons and one vinylic proton);  $^{13}C$ -NMR ( $CDCl_3/TMS$ ):  $\delta$  15.5 (3), 122.35, 123.642, 128.134, 130.282,



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131.292, 143.335, 145.955, 153.229 (six *p*-nitrophenyl carbons, six aryl carbons, two vinylic carbons and one imidazole quaternary carbon); **MS: *m/z* (%I):** 279 (32.8, M<sup>+</sup>), 278 (100, M-1), 249 (10.4), 248 (41.8), 233 (35.8), 232 (91), 231 (40.3), 218 (16.4), 190 (23.9), 145 (7.46), 131 (10.44), 116 (46.2), 115 (58.2), 77 (41.8), 65 (25.4), 64 (14.9), 63 (34.32) etc. (Anal. Calcd for C<sub>16</sub>H<sub>13</sub>N<sub>3</sub>O<sub>2</sub>: %C, 68.800; %H, 4.691; %N, 15.045; Found: %C, 68.800; %H, 4.690; %N, 15.044).

**4 <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>):** δ 7.11–7.50 (complex m, 14H, ten phenyl protons and four aryl protons), 8.12 (sharp s, 1H, vinylic proton); **MS: *m/z* (%I):** 296 (100, M<sup>+</sup>), 219 (41.2), 218 (18), 181 (10), 178 (11.1), 176 (8.9), 169 (10), 148 (14.7), 147 (14.9), 146 (15), 131 (25), 119 (25), 92 (5.9), 77 (19.1), 69 (90), 65 (14.9), 64 (10), 63 (14), 51 (34), 44 (54.4). (Anal. Calcd for C<sub>21</sub>H<sub>16</sub>N<sub>2</sub>: %C, 85.106; %H, 5.441; %N, 9.452; Found: %C, 85.105; %H, 5.440; %N, 9.450).

**5 <sup>1</sup>H-NMR (CDCl<sub>3</sub>/TMS):** δ 2.8 (s, 3H, CH<sub>3</sub>), 6.82 (s, 1H, vinylic proton), 7.26–7.72 (complex m, 9H, five phenyl and four aryl protons), 12.4 (broad s, 1H, -NH); **<sup>13</sup>C-NMR (CDCl<sub>3</sub>/TMS):** δ 17.93 (CH<sub>3</sub>), 115.124, 121.996, 125.86, 128.146, 128.633, 142.532, 144.088, 150.998 (six phenyl carbons, six aryl carbons, two vinylic carbons and one imidazole quaternary carbon); **MS: *m/z* (%I):** 236 (4.2, M + 2), 235 (22.8, M + 1), 234 (46, M<sup>+</sup>), 233 (100, M - 1), 231 (4.6), 219 (8.85), 218 (7), 156 (4), 131 (3.5), 128 (3.5), 117 (3.5), 116 (8.85), 115 (10.6), 109 (4), 105 (4.4), 104 (5.3), 103 (8), 102 (4.4), 92 (4.4), 91 (6.2), 90 (3.5). (Anal. Calcd for C<sub>16</sub>H<sub>14</sub>N<sub>2</sub>: %C, 82.0220; %H, 6.022; %N, 11.956; Found: %C, 82.019; %H, 6.020; %N, 11.952).

**6 <sup>1</sup>H-NMR (CDCl<sub>3</sub>/TMS):** δ 7.1–7.7 (complex m, ten phenyl protons, four aryl protons and vinylic proton); **<sup>13</sup>C-NMR (CDCl<sub>3</sub>/TMS):** δ 117.327, 122.896, 127.316, 128.57, 128.71, 129.043, 129.662, 129.76, 139.00, 140.5, 146.4 and 150.00 (twelve phenyl carbons, six aryl carbons, two vinylic carbons and one imidazole quaternary carbon); **MS: *m/z* (%I):** 298 (3.7, M + 2), 297 (17.7, M + 1), 296 (29.7, M<sup>+</sup>), 295 (100, M - 1), 293 (7.1), 219 (9.7), 218 (4.9), 147 (10.6), 105 (2.6), 91 (3.1). (Anal. Calcd for C<sub>21</sub>H<sub>16</sub>N<sub>2</sub>: %C, 85.106; %H, 5.441; %N, 9.452; Found: %C, 85.105; %H, 5.440; %N, 9.450).

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