

**Table 1**

| Entry | Bithiophene | R   | $\lambda_{\max}^a$ (nm) ( $\epsilon$ ) | Diazonium<br>salt | Azo<br>bithiophene | Yield<br>(%) | $\lambda_{\max}^a$<br>(nm) ( $\epsilon$ ) | IR<br>$\nu$ (cm <sup>-1</sup> ) <sup>b</sup> |
|-------|-------------|-----|--|-------------------|--------------------|--------------|---|--|
| 1     | <b>1a</b>   | MeO | 319.0 (14,994)                         | <b>2a</b>         | <b>3a</b>          | 47           | 467.0 (21,260)                            | 1679 (C=O)<br>2360-3430 (OH)                 |
| 2     | <b>1b</b>   | EtO | 319.5 (14,517)                         | <b>2a</b>         | <b>3b</b>          | 48           | 471.5 (24,020)                            | 1693 (C=O)<br>2360-3420 (OH)                 |
| 3     | <b>1a</b>   | MeO | ----                                   | <b>2b</b>         | <b>4a</b>          | 37           | 496.0 (23,300)                            | 2220 (CN)                                    |
| 4     | <b>1b</b>   | EtO | ----                                   | <b>2b</b>         | <b>4b</b>          | 48           | 499.0 (22,960)                            | 2220 (CN)                                    |
| 5     | <b>1a</b>   | MeO | ----                                   | <b>2c</b>         | <b>5a</b>          | 27           | 509.0 (20,240)                            | ----   |

**Table 2**

| Solvents          | $\pi^*$ | <b>3a</b>                |                                     | <b>3b</b>                |                                     | <b>4a</b>                |                                     | <b>4b</b>                |                                     | <b>5a</b>                |                                     |
|-------------------|---------|--------------------------|-------------------------------------|--------------------------|-------------------------------------|--------------------------|-------------------------------------|--------------------------|-------------------------------------|--------------------------|-------------------------------------|
|                   |         | $\lambda_{\max}$<br>[nm] | $\nu_{\max}$<br>[cm <sup>-1</sup> ] | $\lambda_{\max}$<br>[nm] | $\nu_{\max}$<br>[cm <sup>-1</sup> ] | $\lambda_{\max}$<br>[nm] | $\nu_{\max}$<br>[cm <sup>-1</sup> ] | $\lambda_{\max}$<br>[nm] | $\nu_{\max}$<br>[cm <sup>-1</sup> ] | $\lambda_{\max}$<br>[nm] | $\nu_{\max}$<br>[cm <sup>-1</sup> ] |
| ethanol           | 0.54    | 467.0                    | 21,413                              | 471.5                    | 21,208                              | 496.0                    | 20,161                              | 499.0                    | 20,040                              | 509.0                    | 19,646                              |
| dimethylformamide | 0.88    | 476.0                    | 21,008                              | 481.5                    | 20,768                              | 505.0                    | 19,801                              | 509.0                    | 19,646                              | 521.0                    | 19,193                              |
| dimethylsulfoxide | 1.00    | 488.0                    | 20,491                              | 486.0                    | 20,576                              | 515.0                    | 19,417                              | 515.0                    | 19,417                              | 528.0                    | 18,939                              |