Synthesis of Novel Organic Dyes Containing Coumarin Moiety for Solar Cell†

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Novel organic dyes, JK-34, JK-35 and JK-36 containing bis-dimethylfluorenyl amino coumarin unit are designed and synthesized. Nanocrystalline TiO$_2$ dye-sensitized solar cells were fabricated using these dyes. Under standard global AM 1.5 solar condition, the JK-34 sensitized cell gave a short circuit photocurrent density of 10.05 mA cm$^{-2}$, open circuit voltage of 0.65 V, and a fill factor of 0.68, corresponding to an overall conversion efficiency $\eta$ of 4.54%. We found that the power conversion efficiency was shown to be quite sensitive to the structural modifications of bridging thiophene moiety.

Key Words : Fluorenyl, Coumarin, Organic dyes, Solar cell

Introduction

Increasing energy demands and concerns over global warming have led to a greater focus on renewable energy sources in recent years. The conversion of solar energy to electricity appears as one of the technologies that can replace fossil fuels. Dye-sensitized solar cells (DSSCs) based on mesoporous nanocrystalline TiO$_2$ films have attracted significant attention as low-cost devices and can reach high power conversion efficiencies of 8-11%. In these cells, the sensitizer is one of the key elements for high power conversion efficiency. Some polypyrpyridyl ruthenium (II) complexes have been used as efficient sensitizers because they have an intense metal-to-ligand charge transfer transition in the visible region. Although the ruthenium complexed dyes exhibited high efficiency and long-term stability, they contain a limited precious metal and they are hard to purify. Recently, impressive photocatalytic performance has been obtained with some organic coumarin, indoline, oligoene, merocyanine, and hemicyanine dyes having efficiencies in the range of 5-9%. Very recently, we reported highly efficient and stable organic dyes containing bis-dimethylfluorenyl amino donor unit. The bis-dimethylfluorenyl moiety was introduced to prevent aggregation via molecular stacking by the bulky nonplanar structure and to ensure greater resistance to degradation when exposed to light and high-temperature. A major factor responsible for the high photoconversion efficiency of an organic dye-sensitized solar cell can be tuned by the structural modification. In our previous works, we adapted the (9,9-dimethylfluoren-2-yl)amino phenyl, benzo[b]furan, benzo[b]thiophene and N-aryl carbazole moiety as the donor moiety. Another promising design strategy is replacement of the above moieties with the coumarin electron donor. Changing the electron donor would affect the highest occupied molecular orbital and lowest unoccupied molecular orbital levels of the dye and consequently, the absorption properties.

In this article, we report three new organic dyes containing [bis(9,9-dimethylfluoren-2-yl)amino]coumarin as electron donor and cyano acrylic acid as electron acceptor bridged by thiophene unit or vinylene thiophene unit (Figure 1).

Results and Discussion

The novel organic dyes JK-34–36 were prepared by the stepwise synthetic protocol illustrated in Scheme 1. The reaction started with 7-amino-3-bromo-4-methyl coumarin.

![Figure 1. Structure of JK-34, JK-35, JK-36 and N3.](image-url)
The coumarin mono- and bithiophenes 1 and 2 were synthesized by coupling reaction of 7-amino-3-bromo-4-methylcoumarin with stannylthiophene units in the presence of a catalytic amount of tetraakis(triphenylphosphine)palladium(0) according to the Stille reaction conditions. Fluorene substituted coumarin derivatives 3 and 4 were obtained by Ullmann coupling reaction. These thiophene derivatives were converted into their corresponding thiophene aldehydes 5 and 6 by dedioxanylation with trifluoroacetic acid (TFA). An acetonitrile solution of thiophene aldehyde derivatives (5 and 6) and cyanoacetic acid were refluxed in the presence of piperidine for 8 h. Solvent removal followed by purification using chromatography yielded JK-34 and JK-35. We have also designed to insert the ethylene unit between two thiophenes in order to lengthen the π-conjugation. Introduction of ethylene unit augmented the absorption extinction coefficient and red-shifted the absorption peaks. Coupling reaction of aldehyde 5 with diethyl(5-(5,5-dimethyl-1,3-dioxan-2-yl)thiophen-2-yl)methylphosphonate under Horner-Emmons-Wittig coupling condition using potassium tert-butoxide in THF led to 7. This was then converted to the aldehyde 8, which yielded the dye JK-36 on treatment of cyanoacetic acid.

For the fabrication of DSSC, three sensitizers have been used to manufacture solar cell devices to explore current-voltage characteristics using 12 + 4 μm TiO₂ transparent layers. The first TiO₂ layer of 12 μm thickness was prepared by screen printing TiO₂ paste (Solaronix, 13 nm paste), and the second TiO₂ scattering layer of 4 μm thickness was coated with a different paste (CCIC, HWP-400) for the photo-scattering. The double layer film was treated with 40 mM TiCl₄ solution. The resulting layer was dried at 500 °C for 30 min. After cooling to 100 °C, the films were immersed into the JK-34, JK-35, and JK-36 solution (0.3 mM dyes in ethanol containing 10 mM of chenodeoxycholic acid). The stained TiO₂ electrode and Pt-counter electrode
The electrolyte was composed of 0.6 M 3-hexyl-1,2-dimethylimidazolium iodide, 0.04 M LiI, 0.05 M guanidinium thiocyanate and 0.28 M tetrabutylammonium hexafluorophosphate using TiO$_2$ film with adsorbed dyes as working electrode. The results are listed in Table 1. The three organic dyes absorbed on TiO$_2$ film showed quasi-reversible behaviors. The oxidation potentials of three organic dyes were measured to be 1.37-1.39 V vs. NHE, an oxidation potential energetically favorable for iodide oxidation. The reduction potentials of three dyes calculated from the oxidation potentials and the $E_{ox}$ determined from the intersection of absorption and emission spectra is listed in Table 1. The excited state oxidation potentials ($E_{ox}^*$) of the dyes (JK-34: -1.09 V vs. NHE; JK-35: -1.03 V vs. NHE; JK-36: -0.98 V vs. NHE) are much negative than the conduction band of TiO$_2$ at approximately -0.5 V vs. NHE.

were assembled into a sealed sandwich cell by heating with a hot-melt film (Surlyn 1702, 25 $\mu$m thickness) as a spacer between the electrodes. The electrolyte was composed of 0.6 M 3-hexyl-1,2-dimethylimidazolium iodide, 0.04 M I$_2$, 0.025 M LiI, 0.05 M guanidinium thiocyanate and 0.28 M tetrabutylpyridine in acetonitrile.

The UV-vis and emission spectra of JK-34 and JK-36 in ethanol are shown in Figure 2 and listed Table 1, together with the UV-vis spectra of the corresponding dyes absorbed on TiO$_2$ film. The absorption spectra of the JK-34 and JK-35 display visible band at 429 nm and 431 nm, respectively, which is due to the $\pi$-$\pi^*$ transition of the conjugated molecule. A slight red-shifted band at about 2 nm and a large extinction coefficient of JK-35 compared to those of JK-34 are due to the increase of the $\pi$-conjugation system by thiophene unit. The introduction of ethylene unit in JK-36 also induced the red-shifted band at about 20 nm compared to JK-34 and JK-35. The absorption spectra of all the dyes JK-34--JK-36 on TiO$_2$ film are broadened due to the interaction of the anchoring group with the organic dyes in TiO$_2$-electrodes. Such broadening of the absorption spectrum is desirable for harvesting the solar spectrum and leads to a large photocurrent. The dyes on TiO$_2$ film have red-shifted bands via J-aggregation. We also observed that the dyes JK-34--JK-36 exhibited strong luminescence maxima of 578-643 nm when they are excited within their $\pi$-$\pi^*$ bands in an air-equilibrated solution at 298 K.

Electrochemical properties of the dyes JK-34--JK-36 were evaluated by cyclic voltametry in acetonitrile with 0.1 M tertbutyl ammonium hexafluorophosphate using TiO$_2$ film with adsorbed dyes as working electrode. The results are listed in Table 1. The three organic dyes absorbed on TiO$_2$ film showed quasi-reversible behaviors. The oxidation potentials of three organic dyes were measured to be 1.37-1.39 V vs. NHE, an oxidation potential energetically favorable for iodide oxidation. The reduction potentials of three dyes calculated from the oxidation potentials and the $E_{ox}$ determined from the intersection of absorption and emission spectra is listed in Table 1. The excited state oxidation potentials ($E_{ox}^*$) of the dyes (JK-34: -1.09 V vs. NHE; JK-35: -1.03 V vs. NHE; JK-36: -0.98 V vs. NHE) are much negative than the conduction band of TiO$_2$ at approximately -0.5 V vs. NHE.

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**Table 1. Optical, redox and DSSC performance parameters of dyes**

<table>
<thead>
<tr>
<th>Dye</th>
<th>$\lambda_{abs}$/nm ($\varepsilon$ M$^{-1}$cm$^{-1}$)</th>
<th>$E_{ox}$/V</th>
<th>$E_{red}$/V</th>
<th>$E_{LUMO}$/V</th>
<th>$J_{sc}$ (mAcm$^{-2}$)</th>
<th>$V_{oc}$ (V)</th>
<th>FF</th>
<th>$\eta$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>JK-34</td>
<td>429 (22900)</td>
<td>1.38</td>
<td>2.47</td>
<td>-1.09</td>
<td>10.05</td>
<td>0.65</td>
<td>0.68</td>
<td>4.54</td>
</tr>
<tr>
<td>JK-35</td>
<td>431 (39000)</td>
<td>1.37</td>
<td>2.40</td>
<td>-1.03</td>
<td>9.44</td>
<td>0.65</td>
<td>0.70</td>
<td>4.34</td>
</tr>
<tr>
<td>JK-36</td>
<td>450 (25000)</td>
<td>1.39</td>
<td>2.37</td>
<td>-0.98</td>
<td>9.87</td>
<td>0.64</td>
<td>0.69</td>
<td>4.41</td>
</tr>
<tr>
<td>N3</td>
<td>13.13</td>
<td>0.73</td>
<td>0.66</td>
<td>6.32</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

$^a$Absorption spectra were measured in ethanol solution. $^b$Oxidation potential of dyes on TiO$_2$ were measured in CH3CN with 0.1 M (N,N,N,N,N-pentamethyldenedenediamine) with a scan rate of 50 mV s$^{-1}$ (vs NHE). $^c$$E_{red}$ was determined from intersection of absorption and emission spectra in ethanol. $^d$E$_{LUMO}$ was calculated by $E_0- E_{ox}$. $^e$Performances of DSSCs were measured with 0.18 cm$^2$ working area. $^f$Absorption coefficient; $E_{ox}$: oxidation potential; $E_{red}$: voltage of intersection point between absorption and emission spectra; $J_{sc}$: short-circuit photocurrent density; $V_{oc}$: open-circuit photovoltage; FF: fill factor; $\eta$: total power conversion efficiency.
To gain insight into the geometrical configuration and photophysical properties, molecular orbital calculations of the three sensitizers were performed with the TD-DFT on B3LYP/3-21G* (Figure 3). The calculation illustrates that the HOMO of JK-34 and JK-35 is delocalized over the \( \pi \)-conjugated system through the phenyl amino group and the LUMO is delocalized over the cyanoacrylic unit through thiophene. On the other hand, the HOMO of JK-36 is heavily delocalized over the conduit channel from phenyl amino unit to thiophene group. Examination of the HOMO and LUMO of these dyes indicates that HOMO-LUMO excitation move the electron distribution from the phenyl amino unit to thiophene group. From the same condition, the power conversion efficiency of the DSSCs sensitized by JK-34, JK-35, and JK-36 sensitizers reaches 4.34-4.54%. The power conversion efficiency was shown to be sensitive to the structural modifications of bridging moieties. Contrary to our expectation, the JK-36 sensitizer with a high molar extinction coefficient and red-shifted absorption band showed slightly lower conversion efficiency than JK-34 due probably to the aggregation and interaction between the dye and TiO\(_2\) film. Expanding the \( \pi \)-conjugation is necessary for the high molar extinction coefficient, but the introduction of methine unit is worse in our case because the non-rigid conformation of bridging moiety occurs the energy loss via photoisomerization. Accordingly, the introduction of rigid \( \pi \)-conjugated expansion without a significant lowering of LUMO should be attained for the high efficiency of dye.

**Experimental Section**

**General methods.** All reactions were carried out under an argon atmosphere. Solvents were distilled from appropriate reagents. All reagents were purchased from Sigma-Aldrich. 7-Amino-3-bromo-4-methylocoumarin\(^{18}\) was synthesized using a modified procedure of previous reference. \(^{19}\)H and

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**Figure 4.** A photocurrent voltage curve obtained with a DSSC based on JK-34 (solid line), JK-35 (dashed line), JK-36 (dot line) and N3 (dashed dot-dot line) under AM 1.5 radiation (100 mWcm\(^{-2}\)).

**Figure 5.** Spectra of monochromatic incident photon-to-current conversion efficiency (IPCE) for the DSSCs based on the three dyes. The IPCE data of JK-34 and JK-35 sensitizers in the peak maximum region are about 72% at 445 nm. However, the maximum IPCE value (60%) of JK-36 is lower than those of JK-34 and JK-35. A likely region for this may be attributed to the aggregation of the dye molecule on the TiO\(_2\) electrode. Although the DCA as co-adsorbent to prevent the aggregation of dye on TiO\(_2\) film was used, their red shifts of maximum absorbance between in solution and on TiO\(_2\) film were observed with 13 nm for JK-34, 9 nm for JK-35 and 8 nm for JK-36. In summary, we have designed and synthesized three organic dyes containing coumarin moiety bridged by thiophene units. The power conversion efficiency of the DSSCs based on the JK-34–JK-36 sensitizers reaches 4.34–4.54%. The power conversion efficiency was shown to be sensitive to the structural modifications of bridging moieties. Contrary to our expectation, the JK-36 sensitizer with a high molar extinction coefficient and red-shifted absorption band showed slightly lower conversion efficiency than JK-34 due probably to the aggregation and interaction between the dye and TiO\(_2\) film. Expanding the \( \pi \)-conjugation is necessary for the high molar extinction coefficient, but the introduction of methine unit is worse in our case because the non-rigid conformation of bridging moiety occurs the energy loss via photoisomerization. Accordingly, the introduction of rigid \( \pi \)-conjugated expansion without a significant lowering of LUMO should be attained for the high efficiency of dye.
13C NMR spectra were recorded on a Varian Mercury 300 spectrometer. Elemental analyses were performed with a Carlo Elba Instruments CHNS-O EA 1108 analyzer. Mass spectra were recorded on a JEOL JMS-SX102A instrument. The absorption and photoluminescence spectra were recorded on a Perkin-Elmer Lambda 25 UV-visible spectrometer and a Perkin LS fluorescence spectrometer, respectively.

**Cyclovoltamogram**: Cyclic voltammetry was carried out with a BAS 100B (Bioanalytical Systems, Inc.). A three-electrode system was used and consisted of a gold disk, working electrode, a platinum wire electrode. Redox potentials of dyes on TiO2 were measured in CH3CN with 0.1M (n-C6H5)3N-PF6 as a scan rate between 50 mVs⁻¹ (v.s. Fe/Fe3⁺).

7-Amino-3-(5-(5,5-dimethyl-1,3-dioxan-2-yl)thiophen-2-yl)-4-methylcoumarin (1): A stirred mixture of 7-amino-3-bromo-4-methylcoumarin (2 g, 7.87 mmol), tributylation (5'-5,5-dimethyl-1,3-dioxan-2-yl)thiophen-2-yl)stannane (4.6 g, 9.44 mmol) and Pd(PPh3)4 (0.454 g, 0.39 mmol) in toluene (50 mL) was refluxed for 12 h. After cooling the solution, H2O (10 mL) and brine were added to the solution. The organic layer was separated and dried in MgSO₄. The solvent was removed in vacuo. The pure product 1 was obtained by chromatographic work-up (elucent EAHx = 1:3, Rf = 0.3) as a green solid in 70% yield. Mp: 210.0 °C. 1H NMR (DMSO-d₆): δ 7.50 (d, J = 8.4 Hz, 1H), 7.08 (d, J = 3.3 Hz, 1H), 6.94 (d, J = 3.3 Hz, 1H), 6.60 (d, J = 8.4 Hz, 1H), 6.43 (s, 1H), 6.22 (br, 2H), 5.69 (s, 1H), 3.65 (br, 4H), 2.32 (s, 3H), 1.16 (s, 3H), 0.75 (s, 3H). 13C [H] NMR (CDCl₃): δ 160.1, 154.4, 153.2, 150.9, 142.2, 135.7, 128.4, 127.2, 124.4, 112.1, 111.6, 108.9, 99.1, 74.7, 197.3, 29.7, 22.6, 21.3, 16.5. MS: m/z 371 [M⁺]. Anal. Calc. for C₂₇H₂₀N₂O₃S: C, 64.67; H, 5.07. Found: C, 64.32; H, 5.45.

7-Amino-3-(5-(5,5-dimethyl-1,3-dioxan-2-yl)-2,2'-bithiophen-5-yl)-4-methylcoumarin (2): Compound 2 was synthesized by a procedure similar to 1 except that tributylation (5'-5,5-dimethyl-1,3-dioxan-2-yl)-2,2'-bithiophen-5-yl)stannane (5.37 g, 9.44 mmol) was used in place of tributylation (5'-5,5-dimethyl-1,3-dioxan-2-yl)thiophen-2-yl)stannane. Yield: 76%. Mp: 208 °C. 1H NMR (DMSO-d₆): δ 7.53 (d, J = 8.4 Hz, 1H), 7.29 (d, J = 3.9 Hz, 1H), 7.18 (d, J = 3.3 Hz, 1H), 7.07 (d, J = 3.9 Hz, 1H), 7.06 (d, J = 3.3 Hz, 1H), 6.62 (d, J = 8.4 Hz, 1H), 6.44 (s, 1H), 6.28 (br, 2H), 5.68 (s, 1H), 3.65 (br, 4H), 2.40 (s, 3H), 1.17 (s, 3H), 0.75 (s, 3H). 13C [H] NMR (CDCl₃): δ 160.1, 154.3, 153.3, 150.8, 140.2, 137.0, 136.3, 135.8, 135.1, 130.5, 127.3, 126.3, 123.3, 123.0, 111.7, 109.9, 98.0, 97.1, 76.3, 29.8, 22.6, 21.3, 16.7. MS: m/z 453 [M⁺]. Anal. Calc. for C₂₃H₁₇N₂O₃S: C, 63.55; H, 5.11. Found: C, 63.98; H, 4.98.

7-(Bis[9,9-diethylfluorenyl-2-yl]-3-(5-(5,5-dimethyl-1,3-dioxan-2-yl)thiophen-2-yl)-4-methylcoumarin (3): A stirred mixture of 1 (2 g, 5.38 mmol), 2-iodo-9,9-diethylfluorene (5.17 g, 16.15 mmol), powdered anhydrous potassium carbonate (2.23 g, 16.15 mmol), copper bromide (1.02 g, 16.15 mmol) and 18-crown-6 (0.43 g, 1.615 mmol) in 1,2-dichlorobenzene (70 mL) was refluxed for 48 h. After cooling, the insoluble inorganic material was filtered off under suction and the dark brown filtrate was collected. The insoluble material was washed with dichloromethane (3 × 50 mL). The combined filtrate and organic phase were washed with diute aqueous ammonia and water and dried with magnesium sulfate. The solvent was removed under reduced pressure. The pure product 3 was obtained by silica gel chromatography (elucent EAHx = 1:3, Rf = 0.3) to afford 95 in 95% yield. Mp: 189 °C. 1H NMR (CDCl₃): δ 9.93 (s, 1H), 7.77 (d, J = 3.9 Hz, 1H), 7.69 (i, J = 8.1 Hz, 2H), 7.68 (d, J = 8.2 Hz, 2H), 7.50 (d, J = 8.7 Hz, 1H), 7.42 (d, J = 8.2 Hz, 2H), 7.39 (d, J = 8.7 Hz, 2H), 7.33 (i, J = 8.1 Hz, 2H), 7.26 (s, 2H), 7.21 (d, J = 3.9 Hz, 1H), 7.19 (d, J = 9.0 Hz, 2H), 7.06 (d, J = 9.0 Hz, 1H), 7.00 (s, 1H), 2.46 (s, 3H), 1.43 (s, 12H). 13C [H] NMR (CDCl₃): δ
(E)-3-(2-(5,5-dimethyl-2,2'-bithiophen-5-yl)-4-methylcoumarin-3-yl)-2-(5-(7-(Bis(9,9-dimethylfluoren-2-yl)amino)-4-methylocarmin-3-yl)thiophen-2-yl)-2-cyanoacrylic acid (JK-34): A mixture of 5 (0.25 g, 0.37 mmol) and cyanoacetic acid (0.06 g, 0.74 mmol) was vacuum-dried and added THF (30 mL). The solution was refluxed for 8 h. After cooling the solution, the organic layer was removed in vacuo. The pure product JK-34 was obtained by silica gel chromatography (eluent MeOH:CHCl₃ = 2:1, Rf = 0.6) to afford JK-34 in 51% yield. Mp: 231 °C. "H NMR (DMSO-d₆): δ 8.08 (s, 1H), 7.86 (d, J = 7.8 Hz, 2H), 7.78 (t, J = 7.8 Hz, 2H), 7.67 (d, J = 4.2 Hz, 1H), 7.53 (d, J = 7.8 Hz, 2H), 7.49-7.43 (m, 8H), 7.31 (s, J = 8.1 Hz, 2H), 7.21 (s, 2H), 6.96 (d, J = 4.2 Hz, 1H), 6.79 (s, 1H), 2.44 (s, 3H), 1.40 (s, 12H). "C NMR (DMSO-d₆): δ 162.8, 159.3, 155.2, 153.4, 151.3, 150.6, 144.9, 141.7, 140.5, 140.1, 138.3, 137.9, 135.8, 130.6, 129.5, 128.7, 128.1, 127.1, 124.9, 123.9, 123.1, 122.7, 121.4, 120.4, 119.9, 115.0, 113.1, 105.7, 46.5, 26.6, 17.1. MS: m/z 736 [M⁺]. Anal. Calc. for C₃₉H₂₅N₂O₅S: C, 78.24; H, 4.92. Found: C, 77.48; H, 4.81.

(5R)-3-(2-(5,5-dimethyl-2,2'-bithiophen-5-yl)-4-methylcoumarin-3-yl)-2-(5-(7-(Bis(9,9-dimethylfluoren-2-yl)amino)-4-methylocarmin-3-yl)thiophen-2-yl)-2-cyanoacrylic acid (JK-35): Compound JK-35 was synthesized by a procedure similar to JK-34. Yield: 58%. Mp: 239 °C. "H NMR (DMSO-d₆): δ 8.08 (s, 1H), 7.86 (d, J = 7.8 Hz, 2H), 7.78 (t, J = 7.8 Hz, 2H), 7.67 (d, J = 4.2 Hz, 1H), 7.53 (d, J = 7.8 Hz, 2H), 7.49-7.43 (m, 8H), 7.31 (s, J = 8.1 Hz, 2H), 7.21 (s, 2H), 6.96 (d, J = 4.2 Hz, 1H), 6.79 (s, 1H), 2.44 (s, 3H), 1.40 (s, 12H). "C NMR (DMSO-d₆): δ 162.2, 159.3, 155.2, 153.4, 151.3, 150.6, 144.9, 141.7, 140.5, 140.1, 138.3, 137.9, 135.8, 130.6, 129.5, 128.7, 128.1, 127.1, 124.9, 123.9, 123.1, 122.7, 121.4, 120.4, 119.9, 115.0, 113.1, 105.7, 46.5, 26.6, 17.3. MS: m/z 1819 [M⁺]. Anal. Calc. for C₃₉H₂₅N₂O₅S: C, 75.92; H, 4.68. Found: C, 75.47; H, 4.59.

(Z)-3-(2-(5,5-dimethyl-2,2'-bithiophen-5-yl)-4-methylcoumarin-3-yl)thiophen-2-yl)-2-cyanoacrylic acid (JK-36): Compound JK-36 was synthesized by a procedure similar to JK-34. Yield: 53%. Mp: 221 °C. "H NMR (DMSO-d₆): δ 8.11 (s, 1H), 7.83 (t, J = 8.1 Hz, 2H), 7.74 (d, J = 7.8 Hz, 2H), 7.63 (d, J = 3.6 Hz, 1H), 7.52 (d, J = 7.2 Hz, 2H), 7.46-7.27 (m, 10H), 7.24 (s, 2H), 7.20 (d, J = 3.6 Hz, 1H), 7.13 (s, 1H), 6.94 (d, J = 8.1 Hz, 1H), 6.74 (s, 1H), 2.45 (s, 3H), 1.39 (s, 12H). "C NMR (DMSO-d₆): δ 163.5, 159.7, 156.2, 153.4, 152.3, 150.3, 146.9, 141.7, 140.8, 140.1, 138.3, 137.2, 136.8, 130.6, 129.7, 128.1, 127.1, 127.1, 125.3, 124.9, 123.9, 123.1, 122.9, 122.7, 121.4, 120.8, 120.4, 119.9, 115.0, 114.1, 109.7, 46.5, 26.6, 17.3. MS: m/z 1819 [M⁺]. Anal. Calc. for C₃₉H₂₅N₂O₅S: C, 75.92; H, 4.68. Found: C, 75.47; H, 4.59.

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References


