

# Acceptor-Donor-Acceptor Type Small Molecular Low Band Gap Organic Semiconductors Containing 2-Dicyanomethylen-3-cyano-4,5,5-trimethyl-dihydrofuran

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Acceptor-donor-acceptor type compounds **W1**–**W3** were designed and synthesized. These compounds had the same donor moiety of 2,6-di(thiophen-2-yl)dithieno[3,2-b:2',3'-d]thiophene and different acceptor groups of 2-dicyanomethylen-3-cyano-4,5,5-trimethyl-2,5-dihydrofuran (TCF), dicyanovinyl (DCV) and 3-ethyl-2-thioxothiazolidin-4-one. Experimental results showed all compounds had high thermal stability and strong absorption in the visible light region. Among them, compound **W1** with TCF as acceptor group displayed the lowest LUMO energy level of  $-3.74$  eV and the smallest HOMO-LUMO band gap of  $1.74$  eV, suggesting the potential applications of TCF unit in low band gap organic semiconductors.

**Keywords** low band gap compounds, organic semiconductors, donor-acceptor type conjugated molecules

## Introduction

Low band gap organic semiconductors have attracted great attentions due to their applications in solar cell, field-effect transistors and sensors.<sup>[1-4]</sup> Currently, most of the reported low band gap organic semiconductors are donor-acceptor (D-A) type molecules. To achieve the low band gap property of D-A type molecules, the acceptors used must possess strong electron withdrawing property.<sup>[5-8]</sup> Till now, the common acceptors used are aromatic units such as aromatic diimines (such as NDI and PDI),<sup>[9-12]</sup> benzobis(thiodiazole),<sup>[13]</sup> benzothiodiazole,<sup>[14,15]</sup> thioazole and aromatic rings with cyano, carbonyl and halogen substituents.<sup>[16-20]</sup> Some non-aromatic building blocks such as dicyanovinyl (DCV) and 3-ethyl-2-thioxothiazolidin-4-one have also been used as acceptors into D-A type molecules which displayed high mobility in thin film transistors and high power conversion efficiency in solar cells.<sup>[21-26]</sup> Comparing with electron-rich donor units, the acceptors with strong electron withdrawing properties are still rare. Hence it is requirable and important to develop new type of acceptor units for D-A type low band gap molecules.

2-Dicyanomethylen-3-cyano-4,5,5-trimethyl-2,5-dihydrofuran (TCF) is recognized as one of the best sorts of electron acceptor and has been used into unsymmet-

rical D-A type non-linear optical materials.<sup>[27-29]</sup> Though the strong electron withdrawing property of TCF suggested its potential applications in low band gap organic semiconductor, few studies on the TCF containing low band gap organic semiconductors have been reported so far. Herein, A-D-A type organic semiconductor **W1** (chemical structures see Scheme 1) with TCF as electron acceptor was synthesized and characterized. For comparison, compounds **W2** and **W3** (chemical structures see Scheme 1) which have the same donor unit as that of **W1** and different acceptor groups of DCV and 3-ethyl-2-thioxothiazolidin-4-one respectively were also prepared. Experimental results showed **W1** displayed the lowest LUMO energy levels and the smallest HOMO-LUMO band gap among **W1**–**W3**. Moreover, the thin film absorption of **W1** was in the range of 470–890 nm, reaching the near infrared ray region. All these results demonstrated the unique merits of TCF unit in D-A type low band gap organic semiconductors.

## Experimental

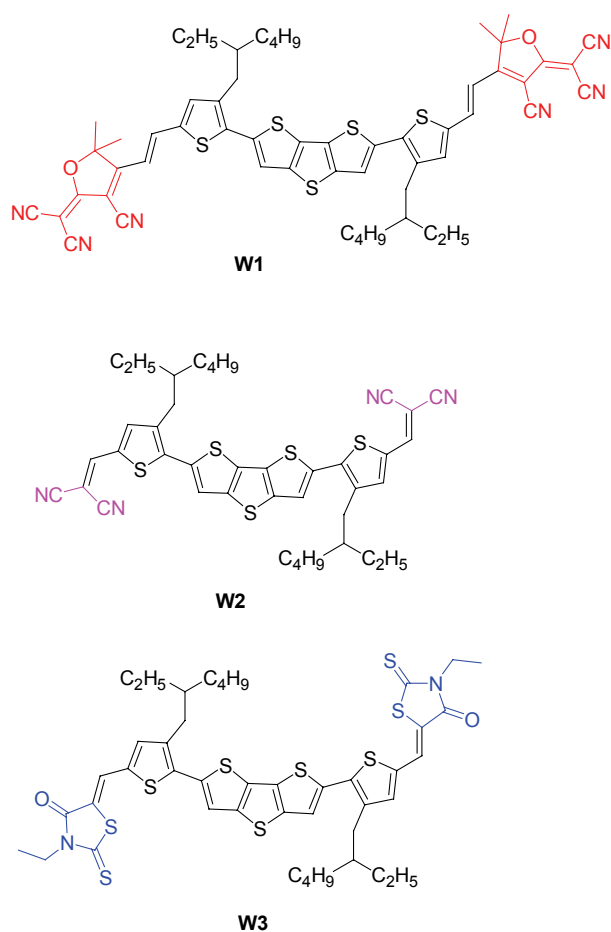
### Synthesis of compound 3

A three neck flask was charged with **1** (3.03 g, 5 mmol), **2** (3.87 g, 10 mmol), (PPh<sub>3</sub>)<sub>4</sub>Pd (578 mg, 0.5 mmol) and toluene (60 mL). The reaction mixture

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**Scheme 1** The chemical structures of compounds **W1**–**W3**

was degassed three times and heated to 110 °C for 40 h. After cooling to room temperature, the reaction mixture was poured into water (100 mL) and extracted with CH<sub>2</sub>Cl<sub>2</sub> (30 mL × 3). The organic layer was washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub> and filtered. The solvent was removed, and the residue was purified by column chromatography on silica gel [eluent: *V*(dichloromethane)/*V*(petroleum ether)=2/1], affording compound **3** as red solid. Yield 1.84 g (57.5%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 0.81–0.94 (m, 12H), 1.29–1.72 (m, 18H), 2.87 (d, *J*=7.2 Hz, 4H), 7.49 (s, 2H), 7.64 (s, 2H), 9.86 (s, 2H); MS (MALDI-TOF) *m/z*: 640.5.

#### Synthesis of compound **4**

Compound **4** was synthesized by the similar procedure as that of compound **W2** and obtained as black solid. Yield 41.3%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 0.87–0.92 (m, 12H), 1.29–1.34 (m, 18H), 1.78 (s, 6H), 2.05 (d, *J*=7.2 Hz, 4H), 6.66 (d, *J*=15.7 Hz, 1H), 7.36 (s, 1H), 7.47 (s, 1H), 7.48 (s, 1H), 7.63 (s, 1H), 7.74–7.79 (m, 1H), 9.81 (s, 1H); MS (MALDI-TOF) *m/z*: 822.6.

#### Synthesis of compound **5**

A mixture of **2** (0.607 g, 2.0 mmol) and 2-dicyanomethylen-3-cyano-4,5,5-trimethyl-2,5-dihydrofuran

(TCF) (0.44 g, 2.2 mmol) in 20 mL of THF was added 0.4 mL of piperidine/acetic acid (1 : 3 by *V/V*). The reaction mixture was refluxed for 8 h. The precipitate was collected and dissolved with CH<sub>2</sub>Cl<sub>2</sub>. The organic layer was washed with water and then dried over Na<sub>2</sub>SO<sub>4</sub>. After removal of solvent, the crude product was purified by column chromatography on silica gel using a mixture of dichloromethane and petroleum ether (2 : 1, *V* : *V*) as eluant to afford compound **5** (0.53 g, 54.8%) as orange solid. <sup>1</sup>H NMR (300 MHz, C<sub>3</sub>D<sub>6</sub>O) δ: 0.87–0.92 (m, 6H), 1.29–1.34 (m, 9H), 1.78 (s, 6H), 2.05 (d, *J*=6.4 Hz, 2H), 6.90 (d, *J*=16.2 Hz, 1H), 7.60 (s, 1H), 8.09 (d, *J*=16.2 Hz, 1H). MS (EI) *m/z*: 483.0.

#### Synthesis of compound **W1**

A reaction mixture of **1** (0.261 g, 0.5 mmol), **5** (0.48 g, 1 mmol) and (PPh<sub>3</sub>)<sub>4</sub>Pd (60 mg, 0.05 mmol) in toluene (10 mL) was heated to 110 °C and stirred for 24 h under nitrogen. After cooling to room temperature, the reaction mixture was poured into water (100 mL) and extracted with CH<sub>2</sub>Cl<sub>2</sub> (30 mL × 3). The organic layer was washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub> and filtered. The solvent was removed under vacuum. The residue was purified by column chromatography on silica gel [eluent: *V*(dichloromethane)/*V*(petroleum ether)=3/1] and further purified by recrystallization from methanol/CHCl<sub>3</sub> solution for four times, affording **W1** as a black solid. Yield, 210 mg (41.9%). M.p. 266–268 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 0.85–0.90 (m, 12H), 1.26–1.36 (m, 18H), 1.76 (s, 12H), 2.79 (d, *J*=7.2 Hz, 4H), 6.65 (d, *J*=15.8 Hz, 2H), 7.32 (s, 2H), 7.46 (s, 2H), 7.77 (d, *J*=15.7 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ: 11.0, 12.6, 14.4, 23.7, 26.1, 29.0, 32.9, 34.0, 40.3, 120.5, 121.5, 124.9, 128.2, 131.7, 136.4, 136.6, 137.0, 138.0, 139.9, 140.8, 141.2, 142.6, 167.5, 192.0. MS (MALDI-TOF) *m/z*: 1002.3. Anal. calcd for C<sub>56</sub>H<sub>54</sub>N<sub>6</sub>O<sub>2</sub>S<sub>5</sub>: C 67.03, H 5.42, N 8.38; found C 67.12, H 5.38, N 8.10.

#### Synthesis of compound **W2**

A solution of **3** (0.32 g, 0.5 mmol) and malononitrile (0.132 g, 4 mmol) in dry CHCl<sub>3</sub> (50 mL) was added 0.5 mL of piperidine. The reaction mixture was heated to reflux and stirred for 12 h under nitrogen. After cooling to room temperature, the solvent was removed under vacuum. The residue was purified by column chromatography on silica gel [eluent: *V*(dichloromethane)/*V*(petroleum ether)=3/1] and further recrystallized with methanol/CHCl<sub>3</sub> solution for four times, affording **W2** as a black solid (0.2 g, 54.3%), M.p. 212–213 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 0.85–0.87 (m, 12H), 1.26–1.27 (m, 18H), 2.82 (d, *J*=7.2 Hz, 4H), 7.54 (s, 2H), 7.58 (s, 2H), 7.78 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ: 10.8, 14.2, 23.2, 25.9, 28.8, 32.7, 33.6, 40.1, 113.4, 114.3, 121.9, 132.5, 133.1, 135.8, 140.9, 142.2, 143.2, 143.5, 150.0. MS (MALDI-TOF) *m/z*: 736.3. Anal. calcd for C<sub>40</sub>H<sub>40</sub>N<sub>4</sub>S<sub>5</sub>: C 65.18, H 5.47, N 7.60; found C 64.88, H 5.29, N 7.58.

Synthesis of compound **W3**

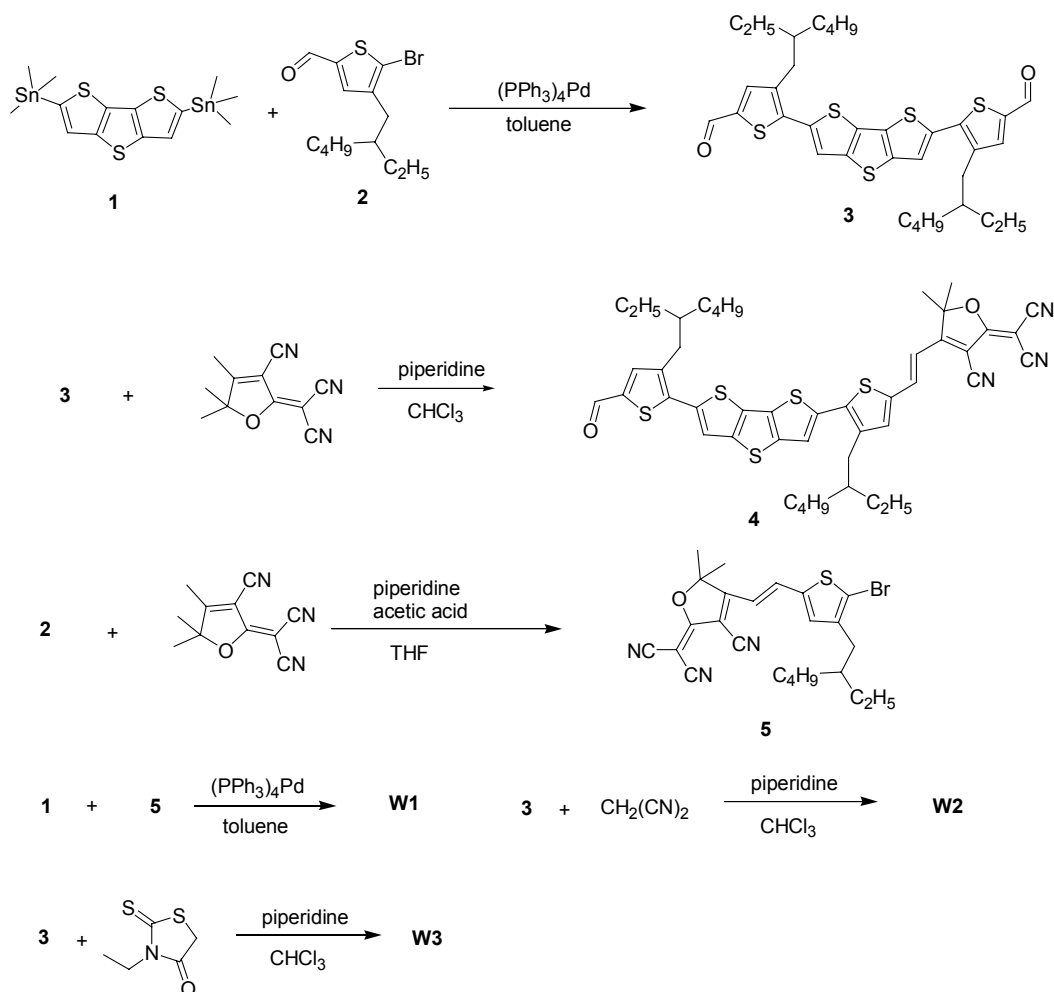
Compound **W3** was synthesized by the similar procedure as that of compound **W2** and obtained as black solid. Yield, 56.1%. M.p. 180–184 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 0.85–0.90 (m, 18H), 1.29–1.72 (m, 18H), 2.78 (d, *J*=7.2 Hz, 4H), 4.19 (q, *J*=6.9 Hz, 4H), 7.21 (s, 2H), 7.44 (s, 2H), 7.78 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ: 11.1, 12.6, 14.4, 23.4, 26.1, 29.0, 32.9, 34.0, 40.3, 120.5, 121.5, 124.9, 128.2, 131.7, 136.4, 136.6, 138.0, 139.9, 140.8, 141.2, 142.6, 167.5, 192.2. MS (MALDI-TOF) *m/z*: 926.7. Anal. calcd for C<sub>44</sub>H<sub>50</sub>N<sub>2</sub>O<sub>2</sub>S<sub>9</sub>: C 56.98, H 5.43, N 3.02; found C 57.13, H 5.67, N 2.93.

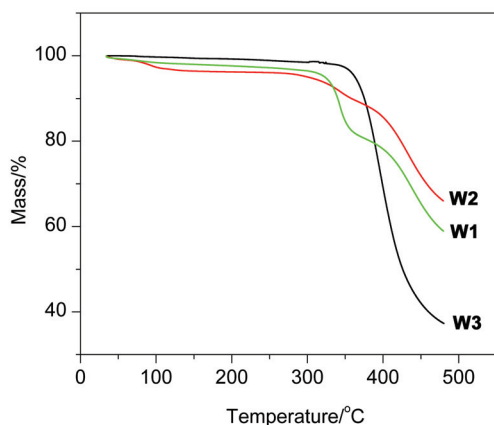
## Results and Discussion

The synthetic route of compounds **W1–W3** was shown in Scheme 2. The dialdehyde compound **3** was the key intermediate for syntheses of compounds **W2** and **W3**. Compound **3** was prepared by a Stille coupling reaction between the stannyl reagent of dithieno[3,2-b:2,3-d]thiophene **1** and compound **2**, and obtained in moderate yield. Compounds **W2** and **W3** were obtained by

reacting compound **3** with malononitrile and 3-ethyl-2-thioxothiazolidin-4-one in the presence of base, respectively. Surprisingly, compound **4** instead of **W1** was obtained when 2 equiv. or even 5 equiv. of TCF was added to the reaction solution of **3** and pyridine. Compound **4** will be an useful intermediate for the synthesis of unsymmetrical A-D-A' type molecules. Thus **W1** was prepared by another synthetic route. In this procedure, TCF was firstly reacted with compound **2** to obtain intermediate **5**. Compound **W1** was prepared by a Pd-catalyzed coupling reaction between **1** and **5**. All target compounds were soluble in organic solvents and were fully characterized by MS, <sup>1</sup>H NMR, <sup>13</sup>C NMR, and elemental analysis.

The thermal stability of organic semiconductors is an important parameter for their applications. The thermal stabilities of **W1–W3** were tested by thermogravimetric analysis (TGA) under a heating rate of 10 °C/min in a nitrogen atmosphere (Figure 1). The temperatures at 5% weight loss were 350 °C for **W1**, 315 °C for **W2** and 375 °C for **W3**, indicating the high thermal stability of **W1–W3** and proving the introduction of TCF units did not lower the thermal stability of compounds.

Scheme 2 The synthetic route of compounds **W1–W3**

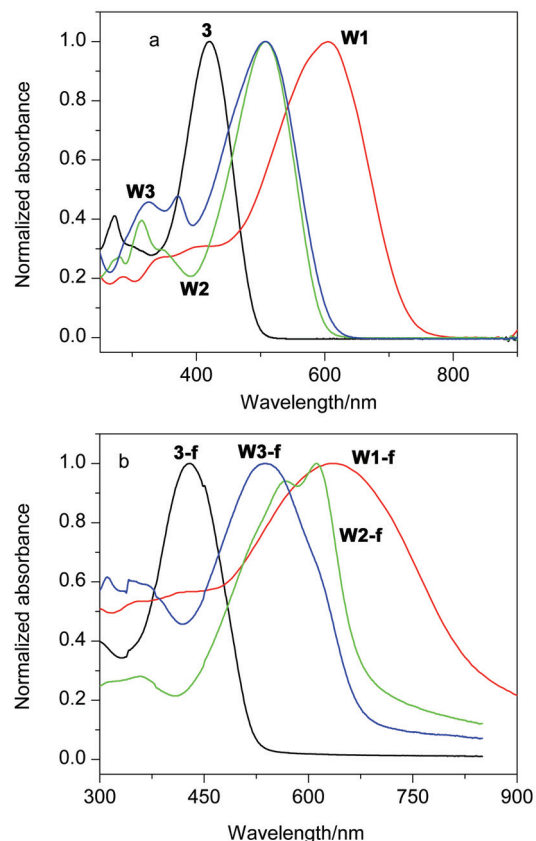


**Figure 1** Thermogravimetric analysis of compounds **W1**, **W2** and **W3**. The compounds were heated to 500 °C at 10 °C/min under N<sub>2</sub> atmosphere.

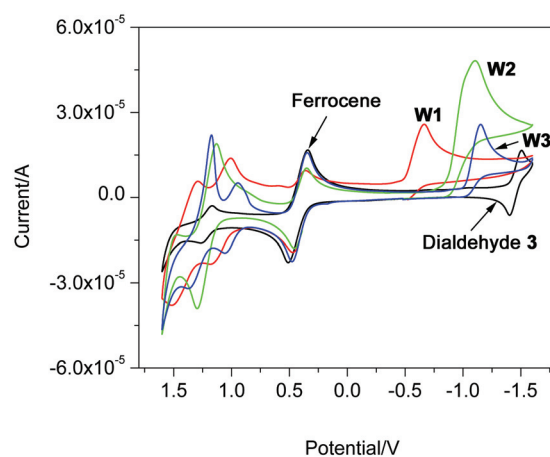
Figure 2 illustrates the absorption spectra of **W1**–**W3** and dialdehyde intermediate **3** in diluted CH<sub>2</sub>Cl<sub>2</sub> solutions and spin-coated thin films. Benefited from their A-D-A type molecular structures and intermolecular charge transfer, all compounds exhibited strong absorption bands in the visible light region (Figure 2a). The maximum absorption peaks of **W1**–**W3** and dialdehyde intermediate **3** were 607, 508, 508 and 420 nm respectively. And the largest absorption extinction coefficients were  $4.4 \times 10^5 \text{ mol}^{-1} \cdot \text{L} \cdot \text{cm}^{-1}$  for **W1**,  $2.6 \times 10^5 \text{ mol}^{-1} \cdot \text{L} \cdot \text{cm}^{-1}$  for **W2**,  $2.5 \times 10^5 \text{ mol}^{-1} \cdot \text{L} \cdot \text{cm}^{-1}$  for **W3** and  $1.7 \times 10^5 \text{ mol}^{-1} \cdot \text{L} \cdot \text{cm}^{-1}$  for **3**. **W1** exhibited the longest wavelength absorption with initial absorption at 766 nm. **W2** and **W3** displayed the nearly same maximum absorption and the initial absorption of **W3** was slightly red shifted by about 18 nm compared with that of **W2**. The energy band gaps calculated from the initial absorptions were 1.62 eV for **W1**, 1.97 eV for **W2**, 1.94 eV for **W3** and 2.43 eV for **3**. The lowest HOMO-LUMO energy band gap of **W1** suggested the strong electron withdrawing property of TCF unit.

The thin film absorptions of **W1**–**W3** and dialdehyde derivative **3** were all red shifted compared with their solution absorptions, indicating the existence of strong intermolecular interactions in the thin films. The red-shifted wavelengths estimated from the initial absorption were 160 nm for **W1**, 140 nm for **W2**, 100 nm for **W3** and 35 nm for **3**. More interestingly, the thin film of **W1** exhibited a broad absorption in the range of 470–890 nm, which reached near IR region. The large absorption changes of **W1** between solution and thin film demonstrated that strong intermolecular interactions exist among **W1** molecules in thin film and further confirmed the unique property of TCF unit.

The electrochemical properties of **W1**–**W3** were test by cyclic voltammetry (CV). The CV measurements were performed in dichloromethane solutions by using Bu<sub>4</sub>NPF<sub>6</sub> as electrolyte and ferrocene as an internal standard. As shown in Figure 3, all compounds exhibited reversible oxidation peaks and irreversible reduc-



**Figure 2** The absorption spectra of compounds **4**, **W1**, **W2** and **W3** in diluted chloromethane solutions (a) and in thin films on the quartz plate (b).



**Figure 3** The cyclic voltammetry of compounds **W1**, **W2**, **W3** and **3** in dichloromethane solution. Condition: Bu<sub>4</sub>NPF<sub>6</sub> as electrolyte, SCE as reference electrode, Au as working electrode, Pt wire as counter electrode, scan rate of 50 mV·s<sup>-1</sup>, ferrocene as internal standard.

tion peaks, and their redox potentials were collected in Table 1. The HOMO-LUMO energy gaps of **W1**–**W3** calculated from cyclic voltammetry were in the range of 1.70–2.25 eV, similar to the optical band gaps estimated from the initial absorption of solutions. **W1** dis-

played the lowest LUMO energy levels among **W1**–**W3**, indicating TCF was a stronger electron withdrawing group than DCV and 3-ethyl-2-thioxothiazolidin-4-one. Interestingly, **W1** also showed higher HOMO energy level which is just slightly lower than that of **W3**. The high HOMO energy level of **W1**, we believe, was attributed its extended conjugation length with the introduction of TCF unit. The lowest LUMO energy level of **W1** further demonstrated the potential applications of TCF units for low band gap organic semiconductors.

**Table 1** The electrochemistry data of compounds **W1**–**W3** and **3**

Compd.	$E_{1/2\text{Ox}}^{1a}/\text{V}$	$E_{1/2\text{Ox}}^{2a}/\text{V}$	$E_{1/2\text{Red}}^{1a}/\text{V}$	HOMO/eV	LUMO/eV
<b>3</b>	1.21		−1.45	−5.60	−2.94
<b>W1</b>	1.08	1.40	−0.66	−5.47	−3.73
<b>W2</b>	1.22	1.52	−1.10	−5.61	−3.20
<b>W3</b>	1.03	1.27	−1.15	−5.42	−3.24

<sup>a</sup>The redox potentials were determined as the midpoints between peak potentials in the forward and reverse scan.

## Conclusions

Acceptor-donor-acceptor type conjugated molecules **W1**–**W3** with 2-dicyanomethylen-3-cyano-4,5,5-trimethyl-2,5-dihydrofuran (TCF), dicyanovinyl (DCV) and 3-ethyl-2-thioxothiazolidin-4-one as electron withdrawing units respectively were designed and synthesized. Their physicochemical properties were thoroughly investigated. All compounds displayed high thermal stability and strong absorption in visible light region. Among them, compound **W1** with TCF as acceptor unit displayed the lowest LUMO energy level of −3.73 eV and the smallest HOMO-LUMO band gap of 1.74 eV. More interestingly, the thin film absorption of **W1** was in the range of 470–890 nm, reaching near IR region. All these results suggested the potential applications of TCF unit in D-A type low band gap organic semiconductors.

## Acknowledgement

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