



Synthesis and characterization of fluorene tethered benzo[c]thiophene/benzo[c]-selenophene analogs

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ABSTRACT

Synthesis of 9,9-dialkyl/diarylfluorene based benzo[c]thiophene/benzo[c]selenophenes is presented. The synthesis of benzo[c]thiophene analogs is also realized with fluorene containing one or two thiophene units. The optical and electrochemical studies of fluorenyl benzo[c]heterocycles are correlated with their structures.

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1. Introduction

Conjugated thienyl oligomers attract much attention by their unique optical and semiconducting properties.¹ They can be used as Organic Light Emitting Diodes (OLEDs)² and Organic Field Effect Transistors (OFETs).³ Oligomers can be easily tuned to the desired properties by changing the structure, by inserting various functional groups like 2-*n*-hexylfluorene, 9,9-diphenyl fluorene,⁴ and oligothiophene/functionalized truxene.⁵ Polythiophene and its derivatives are often used as optoelectronic materials because of their high electrical conductivity and thermal stability in both neutral and doped states.⁶ The fluorene incorporated polythiophene materials exhibit relatively high photo and electroluminescence quantum efficiency than polythiophene materials.⁷

Perepichka and co-workers synthesized poly star shaped tris-EDOT coupled terthienobenzene, which showed switching between red- and blue-colored stable states.⁸ Now a days plenty of fluorene incorporated materials have been synthesized and used in electronic application especially *ortho*-linked spirobifluorene have unique photophysical properties and high triplet energy because of the π -conjugation interruption between spirobifluorene units.⁹ Bao and co-workers reported the synthesis of fluorene coupled oligothiophenes as high performance semiconductors for organic thin

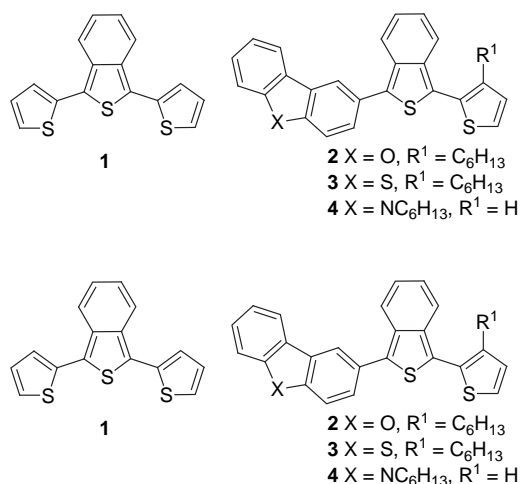
film transistors.¹⁰ Huang and co-workers outlined the synthesis of 2,7-dipyrenyl-9-phenyl-9-pyrenyl fluorene moiety as blue emitters in nondoped OLEDs without the need for hole-injection layers for simplifying device fabrication.¹¹

Ding and co-workers reported the synthesis of copolymers of fluorene and oxadiazole as Polymer Light Emitting Diodes (PLEDs).¹² Lee and co-workers outlined the synthesis of oligo(9,9-dihexyl-2,7-fluoreneethynylene)s as blue light emitting diodes.¹³ Beifield and Yao reported the synthesis of two-photon absorbing unsymmetrical branched chromophores containing fluorene moiety.¹⁴ Recently, Yang and co-workers synthesized novel oligo-9,9'-spirobifluorenes through *ortho*-linkage as full hydrocarbon phosphorescent OLEDs.¹⁵ Wong and co-workers reported the synthesis of novel (9,9-di-*p*-tolylfluorene) analogs containing thiophene and pyridine rings.¹⁶

Rodriguez and co-workers reported the synthesis and optical characterization of thiophene and fluorene derivatives containing ethynyl spacers.¹⁷ Promarak and co-workers reported the synthesis of conjugated fluorenyl oligothiophenes and showed the decrease in oxidation potential and increase in absorption maxima with increasing the number of thiophene rings on fluorene moiety.¹⁸ They have also synthesized *N*-carbazole end-capped oligothiophene-fluorenes as potential hole-transporting and light-emitting layers in OLED devices.¹⁹ Indenofluorenes are known to have good fluorescence properties and have also been investigated for their OLED applications.²⁰ Recently, linear and star shaped rigid conjugated fluorene and truxene molecules containing terpyridine units are

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reported, and their photophysical properties showed that they emit strong blue color in solutions, which provides potential for such materials as the hosts to construct novel functionalized materials for understanding the energy and electron transfer processes.²¹



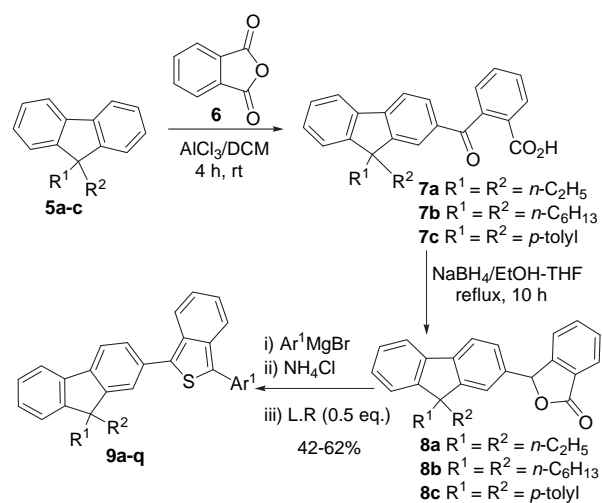
The smaller band gap of 1,3-dithienylbenzo[c]thiophene **1** is a result of the increased contribution of the quinoid resonance structure due to the stabilization achieved by the aromatization of the benzene ring.²² Over the years, the synthesis and characterization of benzo[c]thiophene analogs are well explored due to the low band gap properties associated with their polymer.²³ The benzo[c]thiophene analogs are explored as fluorescent labels,²⁴ components in OLEDs,²⁵ as well as photovoltaics.²⁶ Recently, Kiselev and Thelakkat achieved the synthesis of polymer containing benzo[c]thiophene and triaryl amine groups as a new class of light harvesting dyes.²⁷ Very recently, Swager and co-workers reported the synthesis and characterization of push/pull-type near-IR fluorophores containing benzo[c]furan/benzo[c]thiophene units.²⁸ Thus, the exciting photophysical properties along with enormous application potential of benzo[c]thiophenes prompted us to explore the synthesis and characterization of various types of symmetrical as well as unsymmetrical 1,3-diarylbenzo[c]thiophenes.²⁹ In continuation of the preliminary report on synthesis of fluorene capped benzo[c]thiophenes³⁰ and dibenzoheterocycle tethered benzo[c]thiophenes **2–4**,^{29e} we reported herein a detailed study on synthesis and characterization of 9,9-disubstituted fluorenyl benzo[c]thiophenes.

2. Results and discussion

Cava and Lorcy achieved the synthesis of 1,3-dithienylbenzo[c]thiophene **1** via ring-opening of 2-thienylphthalide with 2-thienylmagnesium bromide followed by thionation.^{23b} The required 2-thienylphthalide was prepared using Friedel–Crafts phthaloylation of thiophene with phthalic anhydride followed by reductive cyclization using NaBH₄ in aq NaHCO₃ solution. Following the Cava's procedure, 9,9-dialkyl/diarylfluorene **5a–c**^{12,31} upon Friedel–Crafts phthaloylation with phthalic anhydride in the presence of anhydrous AlCl₃ led to keto-acids **7a–c**.

The usual NaBH₄ reduction of the keto-acids **7a–c** in aq NaHCO₃ found to be problematic, producing only trace amount of lactones **8a–c**. However, the reduction of keto-acids, using NaBH₄ in THF/EtOH (2:5) at reflux for 10 h afforded required lactones in 37–60% yields. Next, ring-opening of the lactones **8a–c** using freshly prepared 2-thienyl/arylmagnesium bromides followed by aq NH₄Cl

quenching led to the isolation of respective keto-alcohols. The DCM solution of keto-alcohols upon thionation using 0.5 equiv of Lawesson's reagent (LR) followed by workup and column chromatographic purification afforded respective benzo[c]thiophene analogs **9a–q** in 42–62% yields (Scheme 1). As expected, the transformation of the lactones **8a–c** into the respective benzo[c]thiophenes can be smoothly performed via ring-opening of the lactones with variety of aryl as well as heteroaryl Grignards to afford the respective heterocycles in moderate yields. With the exception of heterocycles **9a** and **9d–f**, all other benzo[c]thiophenes are found to be either orange or yellow liquids. The fluorenyl benzo[c]thiophenes **9a–q** are found to be highly soluble in common organic solvents.

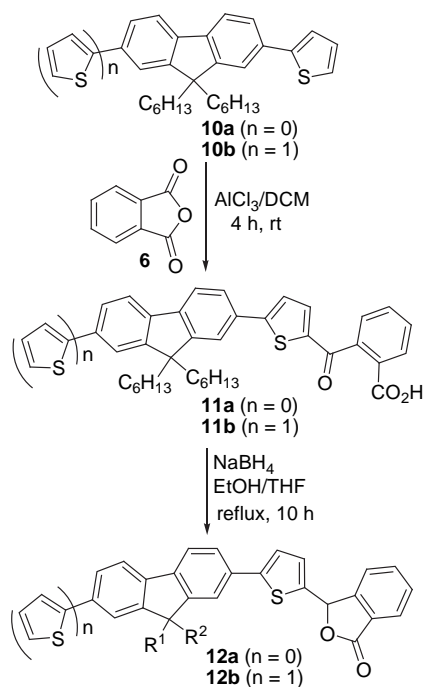


| Compound | R ¹ /R ² | Ar ¹ | Yield (%) |
|-----------|--------------------------------|----------------------|-----------|
| 9a | ethyl | thiophen-2-yl | 57 |
| 9b | ethyl | 3-hexylthiophen-2-yl | 57 |
| 9c | ethyl | 5-hexylthiophen-2-yl | 45 |
| 9d | ethyl | phenyl | 50 |
| 9e | ethyl | <i>p</i> -anisyl | 47 |
| 9f | ethyl | <i>p</i> -tolyl | 52 |
| 9g | ethyl | 1-naphthyl | 52 |
| 9h | hexyl | thiophen-2-yl | 62 |
| 9i | hexyl | 3-hexylthiophen-2-yl | 57 |
| 9j | hexyl | 5-hexylthiophen-2-yl | 50 |
| 9k | hexyl | phenyl | 57 |
| 9l | hexyl | <i>p</i> -anisyl | 58 |
| 9m | hexyl | <i>p</i> -tolyl | 57 |
| 9n | hexyl | 1-naphthyl | 55 |
| 9o | <i>p</i> -tolyl | thiophen-2-yl | 45 |
| 9p | <i>p</i> -tolyl | 3-hexylthiophen-2-yl | 42 |
| 9q | <i>p</i> -tolyl | 5-hexylthiophen-2-yl | 47 |

Scheme 1.

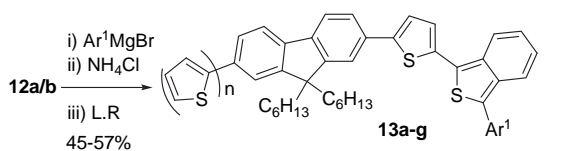
Having achieved the synthesis of fluorene capped benzo[c]thiophenes **9a–q**, next the preparation of similar heterocycles with 2-(2-thienyl)-9,9-dihexyl fluorene **10a** as well as 2,7-bis(2-thienyl)-9,9-dihexyl fluorene **10b** was planned. The known oligothiophene/fluorene **10a** and **10b** were prepared using a Pd-mediated Stille coupling of respective mono/dibromo-9,9-dihexyl fluorene with 2-tributylstannylthiophene. As expected, Friedel–Crafts phthaloylation of **10a** followed by reductive cyclization produced the lactone **12a** in 35% overall yield. Similarly, Friedel–Crafts phthaloylation of

10b with phthalic anhydride in the presence of anhydrous AlCl_3 led to keto-acid **11b** in 51% yield. Reductive cyclization of the keto-acid **11b** using NaBH_4 in THF/EtOH at reflux led to the isolation of lactone **12b** in 69% yield as a thick yellow liquid (Scheme 2).



Scheme 2.

Interaction of the fluorene/oligothiophene lactones **12a** and **12b** with freshly prepared aryl/heteroarylmagnesium bromides followed by thionation and subsequent column chromatographic purification led to the isolation of benzo[*c*]thiophene analogs **13a–g** (Scheme 3).

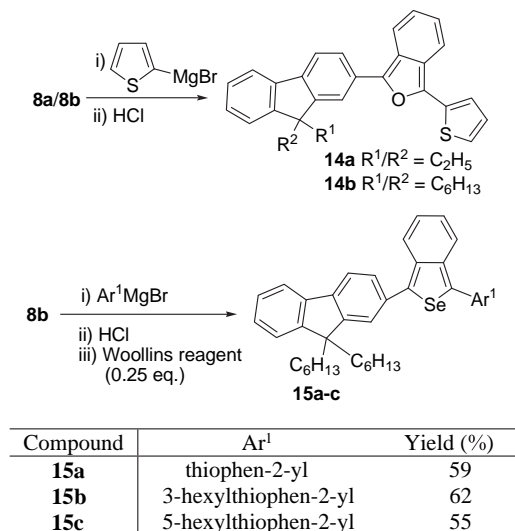


| Compound | n | Ar | Yield (%) |
|------------|---|----------------------|-----------|
| 13a | 0 | thiophen-2-yl | 51 |
| 13b | 0 | 3-hexylthiophen-2-yl | 47 |
| 13c | 0 | 5-hexylthiophen-2-yl | 45 |
| 13d | 0 | phenyl | 49 |
| 13e | 1 | thiophen-2-yl | 55 |
| 13f | 1 | 3-hexylthiophen-2-yl | 57 |
| 13g | 1 | 5-hexylthiophen-2-yl | 50 |

Scheme 3.

Treatment of phthalide **8a/8b** with freshly prepared 2-thienylmagnesium bromides followed by acidic workup and column chromatographic purification gave the respective benzo[*c*]furans **14a/14b** as an orange solid. The ^1H NMR spectral analysis of bright yellow fluorescent solution of benzo[*c*]furans **14a/14b** confirmed that these compounds are highly unstable as the solutions turned red when exposed to white light. Our earlier observation has confirmed that always benzo[*c*]selenophenes can be prepared in

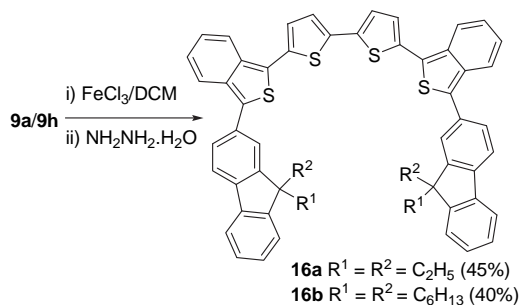
comparatively better yields from the corresponding benzo[*c*]furans rather than the keto-alcohols.³¹ Hence, the ring-opening of the lactone **8b** with various 2-thienylmagnesium bromides followed by acidic workup, and subsequent selenation of crude benzo[*c*]furans with 0.25 equiv of Woollins reagent in DCM afforded benzo[*c*]selenophene derivatives **15a–c** in 55–62% yields, (Scheme 4).



Scheme 4.

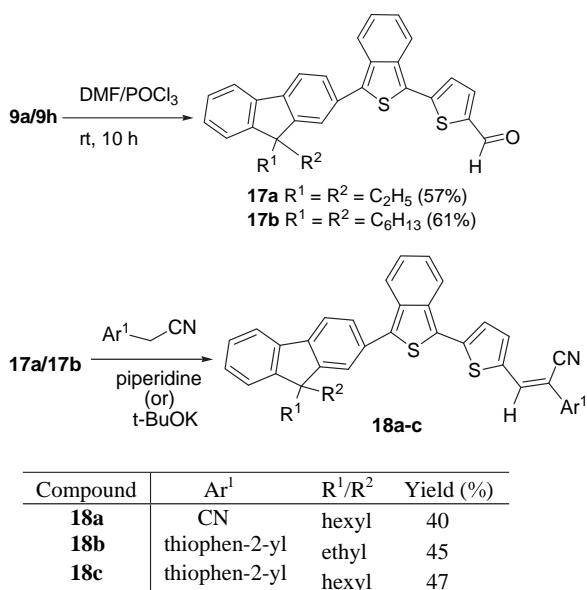
Interaction of monomeric benzo[*c*]thiophene **9a/9h** with anhydrous FeCl_3 in DCM at room temperature for 6 h followed by quenching with dilute solution of hydrazine hydrate and subsequent column chromatographic purification led to the isolation of dimer **16a/16b** (Scheme 5). Similar dimerization of benzo[*c*]thiophene **9i** using anhydrous FeCl_3 did form the expected dimer. However, the attempted purification of the dimer was unsuccessful. It should be noted that the dimerization of **9a/9h** proceeded selectively at the thiophene-2-position rather than the fluorene-7-position.

Next, the synthesis of push/pull-type benzo[*c*]thiophene analogs is planned. Accordingly, Vilsmeier/Haack formylation of **9a/9h**



Scheme 5.

at room temperature for 10 h followed by workup and column chromatographic purification afforded aldehyde **17a/17b** as red solid and dark red liquids in 57% and 61% yields, respectively. It should be noted that formylation has taken place selectively at thiophene rather than the fluorene portion. As expected, condensation of the aldehyde **17a/17b** with malononitrile/thiophene-2-acetonitrile in the presence of piperidine or *t*-BuOK afforded the corresponding conjugated cyano-vinylenes **18a–c** as black solids (Scheme 6).



Scheme 6.

3. Photophysical and electrochemical studies

The UV–vis spectra of monomeric benzo[c]thiophenes exhibited a strong absorption in the region of 415–455 nm due to the π – π^* electronic transition of the conjugated backbone system. The exact absorption λ_{max} values of some representative benzo[c]thiophenes as well as benzo[c]selenophenes are given in Table 1. As expected, the replacement of thiophene ring by 9,9-disubstituted fluorene (**1**→**9a/9h**) has increased its λ_{max} value. Similar to earlier observation,^{29d} the presence of β -hexyl group on the thiophene unit of the fluorenyl benzo[c]thiophene (**9a/9h**→**9b/9i**) reduced its π -conjugation due to the twist in their

Table 1
Physical data for some selected benzo[c]thiophenes

| Compound | $\lambda_{\text{max}}^{\text{a}}$ (nm) | $\lambda_{\text{lum}}^{\text{b}}$ (nm) | E_{g}^{c} (V) | $E_{\text{ox}}^{\text{onsetd}}$ (eV) | HOMO ^e (eV) | LUMO ^f (eV) |
|------------|---|---|-------------------------------|---|---------------------------|---------------------------|
| 9a | 455 | 550 | 2.72 | 0.81 | 5.25 | 2.53 |
| 9b | 430 | 520 | 2.88 | 0.85 | 5.29 | 2.41 |
| 9c | 435 | 525 | 2.85 | 0.83 | 5.27 | 2.42 |
| 9h | 445 | 539 | 2.78 | 0.82 | 5.26 | 2.48 |
| 9i | 425 | 512 | 2.92 | 0.91 | 5.35 | 2.43 |
| 9j | 430 | 515 | 2.88 | 0.85 | 5.29 | 2.41 |
| 9o | 430 | 500 | 2.88 | 0.89 | 5.33 | 2.45 |
| 9p | 415 | 490 | 2.99 | 0.93 | 5.37 | 2.38 |
| 13a | 460 | 555 | 2.69 | 0.80 | 5.24 | 2.55 |
| 13b | 450 | 545 | 2.75 | 0.81 | 5.25 | 2.50 |
| 13e | 475 | 570 | 2.61 | 0.71 | 5.15 | 2.54 |
| 13f | 455 | 540 | 2.72 | 0.72 | 5.16 | 2.44 |
| 15a | 465 | 553 | 2.66 | 0.72 | 5.17 | 2.51 |
| 15b | 445 | 541 | 2.78 | 0.76 | 5.21 | 2.43 |
| 15c | 435 | 545 | 2.85 | 0.77 | 5.22 | 2.37 |
| 16a | 495 | 597 | 2.50 | 0.67 | 5.12 | 2.62 |
| 16b | 480 | 592 | 2.58 | 0.71 | 5.16 | 2.58 |
| 17a | 510 | 605 | 2.43 | 0.69 | 5.14 | 2.71 |
| 17b | 500 | 600 | 2.48 | 0.72 | 5.17 | 2.69 |
| 18a | 545 | 635 | 2.27 | 0.65 | 5.10 | 2.83 |
| 18c | 530 | 625 | 2.33 | 0.67 | 5.12 | 2.79 |

^a Measured in dilute dichloromethane solution.

^b Excited at the absorption maxima.

^c Estimated from the absorption ($E_{\text{g}}=1240/\lambda_{\text{max}}$).

^d The onset of oxidation potential determined using CV.

^e Calculated using the empirical equation: HOMO=(4.44+ $E_{\text{ox}}^{\text{onset}}$).

^f Calculated from LUMO=HOMO– E_{g} .

backbone. Surprisingly, the incorporation of α -hexyl unit in the case of **9a/9h** also reduced the π -conjugation, which indicates that the planarity of skeletal framework may be perturbed. The λ_{max} values of fluorenyl benzo[c]thiophenes **9a/9h** as well as heterocycles **2–4**^{29e} were more or less same. This confirms that the non-bonded electron pair of heterocycles **2–4** is not significantly contributing for the observed π -conjugation. The addition of one or two thiophene unit into the benzo[c]thiophene (**9a**→**13a/13e**) induced red shift in the λ_{max} value. As observed earlier,³² comparatively benzo[c]selenophenes **15a–c** displayed higher λ_{max} values than the corresponding benzo[c]thiophenes. The presence of electron-withdrawing aldehyde function at one end of benzo[c]thiophene **9a/9h** caused 55 nm red shift of absorption, which confirmed the enhancement in the π -electron delocalization. The dimerization of benzo[c]thiophene (**9a/9h**→**16a/16b**) also red shifted the λ_{max} value (Table 1).

The qualitative emission data of fluorenyl heterocycles were recorded in DCM solution and the emission values are also given in Table 1. The monomeric benzo[c]thiophenes showed emission values in the range of 490–550 nm. As observed in the case of absorption spectra, the emission values of these benzo[c]thiophenes are also red shifted with increasing π -conjugation.

A complete summary on physical properties of representative fluorenyl heterocycles are listed in Table 1. The HOMO and LUMO energy levels of these heterocycles were calculated from the absorption and the onset oxidation potential. The E_{g} , HOMO, and LUMO values obtained for representative benzo[c]thiophenes and benzo[c]selenophenes are presented in Table 1. The monomeric benzo[c]thiophenes **9a–c**, **9h–j**, **9o**, and **9p** showed E_{g} values in the range of 2.7–3.0 eV. The replacement of one thiophene ring of parent 1,3-dithienylbenzo[c]thiophene **1** with 9,9-disubstituted fluorene has slightly increased the band gap value (**1**→**9a–c**, **9h–j**, **9o**, **9p**). The observed band gap values of **9a/9h** and heterocycles **2–4** are in the same range, which indicated the minimum non-bonded electron pair influence in the heterocycles **2–4**. The incorporation of one or two thiophene unit into the benzo[c]thiophene (**9a**→**13a/13e**) has not caused any appreciable change in its band gap value. As observed earlier,³¹ the E_{g} values for benzo[c]selenophenes **15a–c** are less compared to the respective benzo[c]thiophenes **9h–j**. The dimerization of benzo[c]thiophenes (**9a/9h**→**16a/16b**) has reduced its band gap value. Introduction of electron withdrawing aldehyde function at one end of the benzo[c]thiophene (**9a/9h**→**17a/17b**) has also reduced band gap value. The monomeric benzo[c]thiophenes displayed HOMO energy level in the range of 5.2–5.4 eV and the corresponding LUMO energy level lie in the region of 2.4–2.5 eV. It is clear that during the dimerization, the HOMO energy levels are more or less unaffected. However, the dimerization has significantly increased the LUMO energy levels, which led to the decrease in their band gap value. Introduction of electron withdrawing aldehyde function at one end of the benzo[c]thiophenes portrayed similar effect on the HOMO and LUMO energy levels.

4. Conclusion

In summary, the synthesis of a variety of fluorenyl benzo[c]thiophene analogs has been achieved in reasonable yields. The physical data of representative fluorenyl benzo[c]thiophenes are correlated with their structure. The synthesis of fluorenyl benzo[c]thiophene based push/pull system was also achieved. The highly soluble nature of the fluorenyl benzo[c]thiophenes may make them suitable for transistor applications through spin-coating techniques. Additionally, the higher-lying HOMO energy levels of these benzo[c]thiophenes (~5.1–5.3 eV) may find them as suitable

candidates for application as hole-transporting materials in double-layer OLEDs.

5. Experimental

5.1. General

All melting points are uncorrected. IR spectra were recorded on a SHIMADZU FT-IR 8300 instrument. ^1H and ^{13}C NMR spectra were recorded in CDCl_3 using TMS as an internal standard on a Bruker-300 spectrometer. Mass spectra were recorded on a JEOL DX 303 HF spectrometer. Elemental analyses were carried out on a Perkin–Elmer series II 2400 (IIT Madras) instrument. All UV–vis spectra were recorded in CH_2Cl_2 solution. The emission spectra were recorded on Perkin–Elmer LS-45 spectrophotometer. The cyclic-Voltammogram of 10^{-3} M solution of benzo[*c*]thiophenes was carried out on a CHI 600C electrochemical analyzer. All the measurements were carried out under oxygen free condition using three electrode cells in which glassy carbon electrode was working electrode, saturated Ag/AgCl electrode was reference electrode, and platinum wire was used as an auxiliary electrode. Tetrabutylammonium hexafluoro phosphate (TBAF_6) was used as supporting electrolyte and its concentration was 10^{-1} M .

5.1.1. Preparation of compounds 10a/b. Compound **10a** was synthesized in 63% yield from 2-bromo-9,9-dihexyl fluorene¹² (1 equiv), tributylstannylthiophene (1 equiv), and 1 mol% of Pd(PPh_3)₄ in dry toluene at reflux for 24 h. Similarly compound **10b** was also synthesized in 50% yield from 2,7-dibromo-9,9-dihexyl fluorene¹² (1 equiv), tributylstannylthiophene (2 equiv), and 2 mol% of Pd(PPh_3)₄ in dry toluene at reflux for 24 h.

5.2. General procedure(A) for preparation of lactones (8a–c and 12a/b)

To a stirred suspension of phthalic anhydride (1.2 equiv) in dry DCM (100 mL), anhydrous aluminum chloride (1.5 equiv with respect to phthalic anhydride) was added and stirred for 0.5 h at room temperature to get yellow solution. To this solution fluorene (**5a–c** and **10a/b**) (1 equiv) in DCM (25 mL) was slowly added (20 min) at 0 °C. The resulting dark-green solution was stirred for 4 h at room temperature. Then it was poured into ice water containing concd HCl. The organic layer was separated and dried over Na_2SO_4 . Removal of solvent in vacuo afforded crude keto-acid (**7a–c** and **11a/b**), which was dissolved in THF/EtOH (2:5). To this solution NaBH_4 (5 equiv) was added in portions and refluxed for 10 h. The reaction mixture was poured into water and concd HCl was added dropwise under stirring (pH=1–2). The precipitated solid was washed with ethanol to afford the required lactones **8a–c** and **12a/b**. (only **7b**, **11b** compounds were isolated and other keto-acids were used as such in reduction step. Compounds **12a/b** were obtained by column chromatography).

5.2.1. 2-(9,9-Dihexyl fluorenyl)benzoic acid (7b). Following the above-mentioned procedure (A), keto-acid **7b** was obtained using 9,9-dihexyl fluorene **5b** (5 g, 14.9 mmol), phthalic anhydride (2.66 g, 17.9 mmol), and AlCl_3 (3.60 g, 27.02 mmol) as a colorless solid (5.19 g, 71%); mp 142 °C; [found: C, 81.9; H, 7.7. $\text{C}_{33}\text{H}_{38}\text{O}_3$ requires C, 82.12; H, 7.94%]; R_f (20% EA/hexane) 0.22; ν_{max} (KBr) 3365, 2911, 1691, 1672, 1595 cm^{-1} ; δ_{H} (300 MHz, CDCl_3) 8.06–8.03 (1H, m, ArH), 7.96–7.91 (1H, m, ArH), 7.75–7.72 (1H, m, ArH), 7.69–7.64 (2H, m, ArH), 7.57–7.48 (2H, m, ArH), 7.44–7.34 (4H, m, ArH), 2.06–1.97 (4H, m, CH_2), 1.13–1.06 (12H, m, CH_2), 0.79–0.61 (10H, m, CH_2CH_3); δ_{C} (75.6 MHz, CDCl_3) 197.06, 170.35, 152.16, 151.01, 146.17, 143.03, 139.76, 136.00, 135.65, 133.01, 130.85, 129.86, 129.33, 128.43,

127.85, 126.98, 125.69, 123.21, 123.08, 120.75, 55.26, 40.08, 31.43, 29.59, 23.71, 22.54, 13.97; MS (EI): m/z (%)=482 [$\text{M}]^+$.

5.2.2. 3-((5-(9,9-Dihexyl-2-thiophen-2-yl)-9H-fluoren-7-yl)thiophen-2-yl)benzoic acid (11b). Following the above-mentioned procedure (A), keto-acid **11b** was obtained using 2,7-bis(2-thienyl)-9,9-dihexyl fluorene **10b** (5 g, 10.00 mmol), phthalic anhydride (1.78 g, 12.02 mmol), and AlCl_3 (2.40 g, 18.02 mmol) as a thick yellow liquid (3.30 g, 51%); [found: C, 75.9; H, 6.4; S, 10.1. $\text{C}_{41}\text{H}_{42}\text{O}_3\text{S}_2$ requires C, 76.12; H, 6.54; S, 9.91%]; R_f (20% EA/hexane) 0.31; ν_{max} (KBr) 3365, 2911, 1681, 1667, 1595 cm^{-1} ; δ_{H} (300 MHz, CDCl_3) 8.10 (1H, d, J 7.2 Hz, ArH), 7.70–7.48 (10H, m, ArH), 7.40–7.38 (1H, m, ArH), 7.32–7.29 (2H, m, ArH), 7.26–7.25 (1H, m, ArH), 7.13 7.10 (1H, m, ArH), 2.04–1.99 (4H, m, CH_2), 1.08–1.04 (12H, m, CH_2), 0.76–0.71 (10H, m, CH_2CH_3); δ_{C} (75.6 MHz, CDCl_3) 188.89, 170.02, 154.52, 151.96, 151.93, 144.98, 142.40, 141.92, 141.73, 139.75, 135.66, 133.83, 132.86, 132.07, 131.07, 129.79, 128.10, 127.86, 125.50, 125.10, 124.74, 123.72, 123.09, 120.58, 120.43, 120.26, 120.21, 55.40, 40.35, 31.43, 29.62, 23.75, 22.53, 13.96; MS (EI): m/z (%)=646 [$\text{M}]^+$.

5.2.3. 3-(9,9-Diethyl-9H-fluoren-2-yl)isobenzofuran-1(3H)-one (8a). Following the above-mentioned procedure (A), lactone **8a** was obtained using 9,9-diethyl fluorene **5a** (5 g, 22.5 mmol), phthalic anhydride (4 g, 27.02 mmol), AlCl_3 (5.4 g, 40.5 mmol), and NaBH_4 (4.28 g, 112 mmol) as a colorless solid (4.0 g, 50%); mp 102 °C; [found: C, 84.5; H, 6.5. $\text{C}_{25}\text{H}_{22}\text{O}_2$ requires C, 84.72; H, 6.26%]; R_f (10% EA/hexane) 0.41; ν_{max} (KBr) 2857, 1759, 1600, 1511 cm^{-1} ; δ_{H} (300 MHz, CDCl_3) 7.77 (1H, d, J 12 Hz, ArH), 7.47–7.41 (2H, m, ArH), 7.36–7.27 (2H, m, ArH), 7.10–7.09 (4H, m, ArH), 7.04–6.97 (2H, m, ArH), 6.26 (1H, m, ArH), 1.83–1.73 (4H, m, CH_2), 0.22–0.10 (6H, m, CH_3); δ_{C} (75.6 MHz, CDCl_3) 170.70, 150.79, 150.15, 150.01, 142.80, 140.61, 135.00, 134.37, 129.36, 127.60, 126.97, 125.89, 125.73, 125.64, 122.97, 122.87, 121.65, 120.04, 119.94, 83.29, 56.24, 32.61, 8.51; MS (EI): m/z (%)=354 [$\text{M}]^+$.

5.2.4. 3-(9,9-Dihexyl-9H-fluoren-2-yl)isobenzofuran-1(3H)-one (8b). Following the above-mentioned procedure (A), lactone **8b** was obtained using **7b** (5 g, 22.5 mmol) and NaBH_4 (4.28 g, 112 mmol) as a colorless solid (2.2 g, 60.1%); mp 98 °C; [found: C, 84.7; H, 8.4. $\text{C}_{33}\text{H}_{38}\text{O}_2$ requires C, 84.94; H, 8.21%]; R_f (10% EA/hexane) 0.42; ν_{max} (KBr) 2875, 1759, 1600, 1500 cm^{-1} ; δ_{H} (300 MHz, CDCl_3) 7.91 (1H, d, J 7.2 Hz, ArH), 7.59–7.56 (2H, m, ArH), 7.52 (1H, d, J 7.39 Hz, ArH), 7.40 (1H, t, J 7.37 Hz, ArH), 7.27–7.19 (4H, m, ArH), 7.14–7.10 (2H, m, ArH), 6.40 (1H, s, CH), 1.89–1.78 (4H, m, CH_2), 1.09–0.82 (12H, m, CH_2), 0.71–0.43 (10H, m, CH_2CH_3); δ_{C} (75.6 MHz, CDCl_3) 170.70, 151.60, 150.94, 150.12, 142.40, 140.21, 134.95, 134.24, 129.31, 127.56, 126.87, 125.74, 125.73, 125.67, 122.92, 122.80, 121.56, 120.06, 119.94, 83.26, 55.21, 40.24, 31.33, 29.60, 23.71, 22.53, 13.98; MS (EI): m/z (%)=466 [$\text{M}]^+$.

5.2.5. 3-(9,9-Dip-tolyl-9H-fluoren-2-yl)isobenzofuran-1(3H)-one (8c). Following the above-mentioned procedure (A), lactone **8c** was obtained using 9,9-dip-tolyl fluorene **5c** (5 g, 14.45 mmol), phthalic anhydride (2.57 g, 17.36 mmol), AlCl_3 (3.47 g, 26.05 mmol), and NaBH_4 (2.74 g, 72.10 mmol) as a colorless solid (2.6 g, 37%); mp 115 °C; [found: C, 88.1; H, 5.7. $\text{C}_{35}\text{H}_{26}\text{O}_2$ requires C, 87.84; H, 5.48%]; R_f (10% EA/hexane) 0.41; ν_{max} (KBr) 2925, 1759, 1611, 1500 cm^{-1} ; δ_{H} (300 MHz, CDCl_3) 7.89–7.87 (1H, m, ArH), 7.65–7.47 (4H, m, ArH), 7.32–7.07 (8H, m, ArH), 6.95–6.89 (6H, m, ArH), 6.31 (1H, s, CH), 2.23 (6H, s, CH_3); δ_{C} (75.6 MHz, CDCl_3) 170.22, 151.60, 150.97, 150.12, 147.17, 145.95, 142.40, 141.55, 140.21, 134.95, 134.24, 129.31, 127.56, 126.87, 125.74, 125.73, 125.67, 122.92, 122.80, 121.56, 120.06, 119.94, 83.26, 62.91, 21.01; MS (EI): m/z (%)=478 [$\text{M}]^+$.

5.2.6. 3-(5-(9,9-Dihexyl-9H-fluoren-2-yl)thiophen-2-yl)isobenzofuran-1(3H)-one (12a). Following the above-mentioned procedure (A), lactone **12a** was obtained using 2-(2-thienyl)-9,9-

dihexyl fluorene **10a** (3 g, 7.2 mmol), phthalic anhydride (1.28 g, 8.65 mmol), AlCl₃ (1.73 g, 13.00 mmol), and NaBH₄ (1.37 g, 36.00 mmol) as a thick yellow liquid (1.4 g, 35%); [found: C, 80.7; H, 7.6; S, 5.7. C₃₇H₄₀O₂S requires C, 80.98; H, 7.35; S, 5.84%]; R_f (10% EA/hexane) 0.47; ν_{max} (KBr) 2875, 1755, 1600, 1500 cm⁻¹; δ_H (300 MHz, CDCl₃) 7.99 (1H, d, J 7.5 Hz, ArH), 7.71–7.67 (2H, m, ArH), 7.57–7.51 (3H, m, ArH), 7.45–7.35 (2H, m, ArH), 7.28–7.26 (2H, m, ArH), 7.16–7.08 (3H, m, ArH), 6.67 (1H, s, CH), 2.03–1.96 (4H, m, CH₂), 1.11–1.03 (12H, m, CH₂), 0.75–0.63 (10H, m, CH₂CH₃); δ_C (75.6 MHz, CDCl₃) 170.11, 151.79, 151.75, 148.61, 145.06, 140.85, 139.98, 137.61, 134.41, 133.55, 132.53, 129.89, 128.10, 126.00, 125.77, 125.04, 124.64, 123.21, 122.99, 122.61, 120.23, 120.15, 78.27, 55.33, 40.40, 31.45, 29.64, 23.73, 22.56, 14.00; MS (EI): m/z (%)=548 [M]⁺.

5.2.7. 3-((5-(9,9-Dihexyl-2-thiophen-2-yl)-9H-fluoren-7-yl)thiophen-2-yl)isobenzofuran-1(3H)-one (**12b**). Following the above-mentioned procedure (A), lactone **12b** was obtained using **11b** (3.3 g, 5.11 mmol) and NaBH₄ (0.97 g, 25.5 mmol) as a thick yellow liquid (2.2 g, 68.7%); [found: C, 77.8; H, 7.0; S, 9.9. C₄₁H₄₂O₂S₂ requires C, 78.05; H, 6.71; S, 10.16%]; R_f (10% EA/hexane) 0.51; ν_{max} (KBr) 2875, 1759, 1600, 1500 cm⁻¹; δ_H (300 MHz, CDCl₃) 8.00 (1H, d, J 7.5 Hz, ArH), 7.74–7.63 (4H, m, ArH), 7.60–7.50 (4H, m, ArH), 7.47 (1H, s, ArH), 7.37 (1H, d, J 3.3 Hz, ArH), 7.28–7.26 (2H, m, ArH), 7.16 (1H, d, J 3.6 Hz, ArH), 7.10–7.08 (1H, m, ArH), 6.66 (1H, s, CH), 2.03–1.96 (4H, m, CH₂), 1.11–1.03 (12H, m, CH₂), 0.75–0.63 (10H, m, CH₂CH₃); δ_C (75.6 MHz, CDCl₃) 169.76, 151.80, 151.75, 148.61, 147.55, 145.06, 140.85, 139.98, 137.61, 134.41, 133.51, 132.53, 129.89, 129.16, 128.10, 126.00, 125.77, 125.04, 124.93, 124.64, 123.21, 122.99, 122.61, 120.23, 120.15, 78.27, 55.33, 40.40, 31.45, 29.64, 23.73, 22.56, 14.00; MS (EI): m/z (%)=630 [M]⁺.

5.3. A representative procedure for the preparation of benzo[c]thiophene (**9a**) from lactone (**8a**) (procedure B)

5.3.1. 1-(9,9-Diethyl-9H-fluoren-2-yl)-3-(thiophen-2-yl)benzo[c]thiophene (**9a**). To a solution of lactone **8a** (0.7 g, 1.97 mmol) in anhydrous THF (25 mL) was added 2-thienylmagnesium bromide [prepared from 2-bromothiophene (0.64 g, 3.92 mmol) and magnesium turnings (0.11 g, 4.58 mmol)] at 0 °C under N₂. The reaction mixture was slowly raised to room temperature and stirred for 4 h. It was then quenched with aq NH₄Cl solution, extracted with DCM (2×20 mL), and dried (Na₂SO₄). The DCM solution was then stirred with Lawesson's reagent (0.40 g, 1.00 mmol) at room temperature for 4 h. Solvent was evaporated in vacuo to give the crude product, which was purified by column chromatography (100% hexane) to give the title compound **9a** (0.49 g, 57%) as an orange solid; mp 56 °C; [found: C, 79.6; H, 5.6; S, 14.8. C₂₉H₂₄S₂ requires C, 79.77; H, 5.54; S, 14.69%]; R_f (100% hexane) 0.85; ν_{max} (KBr) 2891, 1601, 1500 cm⁻¹; δ_H (300 MHz, CDCl₃) 7.92–7.70 (6H, m, ArH), 7.53 (1H, d, J 3 Hz, ArH), 7.36–7.29 (5H, m, ArH), 7.14 (1H, t, J 3 Hz, ArH), 7.01 (1H, m, ArH), 2.15–2.06 (4H, m, CH₂), 0.40 (6H, t, J 3.6 Hz, CH₃); δ_C (75.6 MHz, CDCl₃) 150.70, 150.19, 144.21, 141.14, 140.58, 140.09, 133.74, 130.24, 127.88, 127.16, 126.99, 125.44, 125.22, 123.96, 123.84, 122.96, 122.25, 121.81, 120.33, 120.22, 120.06, 119.74, 118.97, 56.27, 32.86, 8.62; MS (EI): m/z (%)=436 [M]⁺.

5.3.2. 1-(9,9-Diethyl-9H-fluoren-2-yl)-3-(3-hexylthiophen-2-yl)benzo[c]thiophene (**9b**). Following the above-mentioned procedure (B), benzo[c]thiophene **9b** was obtained using the lactone **8a** (0.7 g, 1.97 mmol), 3-hexyl-2-thienylmagnesium bromide [prepared from 2-bromo-3-hexylthiophene (0.97 g, 3.95 mmol) and Mg (0.11 g, 4.58 mmol)], and Lawesson's reagent (0.40 g, 1.00 mmol) as a thick orange liquid (0.58 g, 57%); [found: C, 80.5; H, 6.8; S, 12.6. C₃₅H₃₆S₂ requires C, 80.72; H, 6.97; S, 12.31%]; R_f (100% hexane) 0.89; ν_{max} (KBr) 2915, 1595, 1500 cm⁻¹; δ_H (300 MHz, CDCl₃) 7.85–7.82 (1H, m, ArH), 7.74–7.73 (1H, m, ArH), 7.68–7.64 (2H, m, ArH), 7.60–7.57 (1H,

m, ArH), 7.38–7.35 (4H, m, ArH), 7.38–7.29 (1H, m, ArH), 7.11–7.06 (3H, m, ArH), 2.65 (2H, t, J 8.25 Hz, CH₂), 2.08 (4H, q, J 7.4 Hz, CH₂), 1.63–1.55 (2H, m, CH₂), 1.25–1.21 (6H, m, CH₂), 0.83–0.79 (3H, m, CH₃), 0.41 (6H, t, J 7.35 Hz, CH₃); δ_C (75.6 MHz, CDCl₃) 150.84, 150.21, 142.34, 141.14, 141.02, 139.23, 138.01, 136.24, 134.33, 132.84, 129.25, 128.22, 128.16, 127.28, 127.00, 125.85, 124.20, 124.05, 123.66, 123.00, 121.79, 121.02, 120.18, 119.83, 56.27, 32.78, 31.60, 30.84, 29.09, 29.03, 22.56, 14.05, 8.62; MS (EI): m/z (%)=520 [M]⁺.

5.3.3. 1-(9,9-Diethyl-9H-fluoren-2-yl)-3-(5-hexylthiophen-2-yl)benzo[c]thiophene (**9c**). Following the above-mentioned procedure (B), benzo[c]thiophene **9c** was obtained using the lactone **8a** (0.7 g, 1.97 mmol), 5-hexyl-2-thienylmagnesium bromide [prepared from 2-bromo-5-hexylthiophene (0.97 g, 3.95 mmol) and Mg (0.11 g, 4.58 mmol)], and Lawesson's reagent (0.40 g, 1.00 mmol) as a thick orange liquid (0.46 g, 45%); [found: C, 80.9; H, 7.1; S, 12.1. C₃₅H₃₆S₂ requires C, 80.72; H, 6.97; S, 12.31%]; R_f (100% hexane) 0.89; ν_{max} (KBr) 2915, 1595, 1500 cm⁻¹; δ_H (300 MHz, CDCl₃) 7.85–7.79 (1H, m, ArH), 7.74–7.73 (1H, m, ArH), 7.65–7.59 (2H, m, ArH), 7.55–7.49 (1H, m, ArH), 7.41–7.35 (4H, m, ArH), 7.31–7.29 (1H, m, ArH), 7.15–7.07 (3H, m, ArH), 2.67 (2H, t, J 8.17 Hz, CH₂), 2.05 (4H, q, J 7.4 Hz, CH₂), 1.59–1.51 (2H, m, CH₂), 1.25–1.21 (6H, m, CH₂), 0.83–0.79 (3H, m, CH₃), 0.45 (6H, t, J 7.29 Hz, CH₃); δ_C (75.6 MHz, CDCl₃) 150.55, 149.27, 142.30, 141.11, 141.00, 139.22, 138.05, 136.25, 134.37, 132.83, 129.37, 128.12, 127.32, 127.00, 125.98, 124.22, 124.01, 123.77, 123.03, 121.89, 121.00, 120.22, 119.88, 56.37, 32.86, 31.55, 30.79, 29.07, 22.61, 14.11, 8.62; MS (EI): m/z (%)=520 [M]⁺.

5.3.4. 1-(9,9-Diethyl-9H-fluoren-2-yl)-3-phenylbenzo[c]thiophene (**9d**). Following the above-mentioned procedure (B), benzo[c]thiophene **9d** was obtained using the lactone **8a** (0.7 g, 1.97 mmol), phenylmagnesiumbromide [prepared from bromobenzene (0.62 g, 3.95 mmol) and Mg (0.11 g, 4.58 mmol)], and Lawesson's reagent (0.40 g, 1.00 mmol) as a yellow solid (0.40 g, 50%); mp 55 °C; [found: C, 86.7; H, 6.3; S, 7.2. C₃₁H₂₆S requires C, 86.47; H, 6.09; S, 7.45%]; R_f (100% hexane) 0.81; ν_{max} (KBr) 2905, 1595, 1501 cm⁻¹; δ_H (300 MHz, CDCl₃) 7.99–7.89 (1H, m, ArH), 7.84–7.71 (4H, m, ArH), 7.61 (1H, s, ArH), 7.35–7.28 (5H, m, ArH), 7.15–7.09 (3H, m, ArH), 7.03–7.01 (2H, m, ArH), 2.10–2.03 (4H, m, CH₂), 0.43–0.37 (6H, m, CH₃); δ_C (75.6 MHz, CDCl₃) 150.70, 150.22, 144.25, 141.14, 140.60, 140.09, 133.80, 130.25, 127.90, 127.20, 127.00, 125.64, 125.44, 125.22, 124.00, 123.84, 123.00, 122.25, 121.81, 120.33, 120.22, 120.10, 119.74, 119.00, 56.30, 32.91, 8.62; MS (EI): m/z (%)=430 [M]⁺.

5.3.5. 1-(9,9-Diethyl-9H-fluoren-2-yl)-3-(4-methoxyphenyl)benzo[c]thiophene (**9e**). Following the above-mentioned procedure (B), benzo[c]thiophene **9e** was obtained using the lactone **8a** (0.7 g, 1.97 mmol), *p*-anisylmagnesiumbromide [prepared from 4-bromoanisole (0.74 g, 3.95 mmol) and Mg (0.11 g, 4.58 mmol)], and Lawesson's reagent (0.40 g, 1.00 mmol) as a yellow solid (0.47 g, 47%); mp 59 °C. [found: C, 83.2; H, 6.3; S, 7.2. C₃₂H₂₈OS requires C, 83.44; H, 6.13; S, 6.96%]; R_f (100% hexane) 0.71; ν_{max} (KBr) 2855, 1595, 1501 cm⁻¹; δ_H (300 MHz, CDCl₃) 7.97–7.85 (2H, m, ArH), 7.85–7.73 (3H, m, ArH), 7.39–7.27 (5H, m, ArH), 7.15–7.11 (3H, m, ArH), 7.03–6.95 (2H, m, ArH), 3.75 (3H, s, OCH₃), 2.01–1.95 (4H, m, CH₂), 0.45–0.39 (6H, m, CH₃); δ_C (75.6 MHz, CDCl₃) 151.01, 150.45, 144.25, 141.14, 140.60, 140.09, 133.80, 130.25, 127.90, 127.20, 127.05, 125.64, 125.44, 125.22, 124.00, 123.84, 123.00, 122.25, 121.81, 121.43, 120.22, 120.10, 119.74, 119.22, 56.31, 40.57, 32.91, 8.62; MS (EI): m/z (%)=460 [M]⁺.

5.3.6. 1-(9,9-Diethyl-9H-fluoren-2-yl)-3-(4-methylphenyl)benzo[c]thiophene (**9f**). Following the above-mentioned procedure (B), benzo[c]thiophene **9f** was obtained using the lactone **8a** (0.7 g, 1.97 mmol), *p*-tolylmagnesiumbromide [prepared from 4-bromotoluene (0.68 g, 3.95 mmol) and Mg (0.11 g, 4.58 mmol)], and

Lawesson's reagent (0.40 g, 1.00 mmol) as a yellow solid (0.46 g, 52%); mp 55 °C; [found: C, 86.2; H, 6.4; S, 7.0. C₃₂H₂₈S requires C, 86.44; H, 6.35; S, 7.21%]; *R_f* (100% hexane) 0.75; ν_{\max} (KBr) 2855, 1595, 1501 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 7.76–7.69 (3H, m, ArH), 7.67–7.56 (3H, m, ArH), 7.35–7.30 (7H, m, ArH), 7.19–6.93 (2H, m, ArH), 2.62 (3H, s, CH₃), 2.12–2.00 (4H, m, CH₂), 0.42–0.30 (6H, m, CH₃); δ_{C} (75.6 MHz, CDCl₃) 151.21, 151.00, 150.00, 146.63, 144.37, 140.19, 139.94, 136.01, 135.05, 134.80, 133.72, 130.72, 130.46, 129.86, 128.99, 128.45, 127.94, 127.07, 123.90, 123.10, 120.75, 119.24, 56.28, 32.50, 29.70, 8.44; MS (EI): *m/z* (%)=444 [M]⁺.

5.3.7. *1-(9,9-Diethyl-9H-fluoren-2-yl)-3-(naphthalen-1yl)benzo[c]thiophene (9g)*. Following the above-mentioned procedure (B), benzo[c]thiophene **9g** was obtained using the lactone **8a** (0.7 g, 1.97 mmol), 1-naphthalenylmagnesiumbromide [prepared from 1-bromonaphthalene (0.82 g, 3.95 mmol) and Mg (0.11 g, 4.58 mmol)], and Lawesson's reagent (0.40 g, 1.00 mmol) as a thick yellow liquid (0.49 g, 52%); [found: C, 87.2; H, 5.6; S, 6.9. C₃₅H₂₈S requires C, 87.46; H, 5.87; S, 6.67%]; *R_f* (100% hexane) 0.81; ν_{\max} (KBr) 2875, 1601, 1501 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 7.79–7.76 (4H, m, ArH), 7.75–7.72 (6H, m, ArH), 7.66 (1H, d, *J* 6 Hz, ArH), 7.60 (1H, d, *J* 4.8 Hz, ArH), 7.47–7.33 (5H, m, ArH), 7.05 (1H, t, *J* 4.2 Hz, ArH), 2.01–1.94 (4H, m, CH₂), 0.24 (6H, t, *J* 7.2 Hz, CH₃); δ_{C} (75.6 MHz, CDCl₃) 150.70, 150.22, 147.22, 145.71, 144.25, 143.11, 141.14, 140.60, 140.09, 133.80, 131.91, 130.25, 127.90, 127.20, 127.00, 125.64, 125.44, 125.22, 124.00, 123.84, 123.00, 122.25, 121.81, 120.33, 120.22, 120.10, 119.74, 119.00, 56.30, 32.91, 8.62; MS (EI): *m/z* (%)=480 [M]⁺.

5.3.8. *1-(9,9-Dihexyl-9H-fluoren-2-yl)-3-(thiophen-2-yl)benzo[c]thiophene (9h)*. Following the above-mentioned procedure (B), benzo[c]thiophene **9h** was obtained using the lactone **8b** (0.7 g, 1.50 mmol), 2-thienylmagnesium bromide [prepared from 2-bromothiophene (0.49 g, 3.00 mmol) and Mg (0.08 g, 3.61 mmol)], and Lawesson's reagent (0.30 g, 0.74 mmol) as a thick orange liquid (0.51 g, 62%); [found: C, 80.8; H, 7.3; S, 11.9. C₃₇H₄₀S₂ requires C, 80.97; H, 7.35; S, 11.68%]; *R_f* (100% hexane) 0.85; ν_{\max} (KBr) 2895, 1601, 1505 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 8.02–7.92 (3H, m, ArH), 7.88–7.80 (3H, m, ArH), 7.62–7.61 (1H, m, ArH), 7.47–7.36 (4H, m, ArH), 7.22–7.10 (3H, m, ArH), 2.18–2.10 (4H, m, CH₂), 1.24–1.08 (12H, m, CH₂), 0.89–0.79 (10H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 151.55, 151.07, 144.33, 140.78, 140.25, 140.15, 133.80, 130.26, 127.90, 127.20, 126.96, 125.47, 125.36, 125.25, 123.97, 123.90, 122.94, 122.24, 121.86, 120.35, 120.31, 120.10, 119.83, 119.03, 55.29, 40.55, 31.60, 29.81, 23.91, 22.66, 14.10; MS (EI): *m/z* (%)=548 [M]⁺.

5.3.9. *1-(9,9-Dihexyl-9H-fluoren-2-yl)-3-(3-hexylthiophen-2-yl)benzo[c]thiophene (9i)*. Following the above-mentioned procedure (B), benzo[c]thiophene **9i** was obtained using the lactone **8b** (0.7 g, 1.50 mmol), 3-hexyl-2-thienyl magnesiumbromide [prepared from 2-bromo-3-hexylthiophene (0.74 g, 3.00 mmol) and Mg (0.08 g, 3.61 mmol)], and Lawesson's reagent (0.30 g, 0.74 mmol) as a thick orange liquid (0.54 g, 57%); [found: C, 81.8; H, 8.0; S, 10.3. C₄₃H₅₂S₂ requires C, 81.59; H, 8.28; S, 10.13%]; *R_f* (100% hexane) 0.89; ν_{\max} (KBr) 2915, 1595, 1500 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 7.85–7.82 (1H, m, ArH), 7.81–7.75 (2H, m, ArH), 7.70–7.64 (2H, m, ArH), 7.45–7.39 (5H, m, ArH), 7.13–7.00 (3H, m, ArH), 2.73–2.68 (2H, m, CH₂), 2.10–2.05 (4H, m, CH₂), 1.32–1.27 (8H, m, CH₂), 1.20–1.15 (12H, m, CH₂CH₃), 0.90–0.80 (13H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 151.61, 151.10, 146.10, 142.00, 141.00, 140.61, 136.60, 134.81, 132.85, 130.63, 129.22, 128.70, 127.30, 127.00, 125.90, 124.01, 123.80, 123.77, 123.00, 122.44, 121.60, 120.20, 119.90, 55.27, 40.40, 31.65, 31.50, 30.80, 29.73, 29.14, 29.10, 23.90, 22.61, 14.10, 14.05; MS (EI): *m/z* (%)=632 [M]⁺.

5.3.10. *1-(9,9-Dihexyl-9H-fluoren-2-yl)-3-(5-hexylthiophen-2-yl)benzo[c]thiophene (9j)*. Following the above-mentioned procedure

(B), benzo[c]thiophene **9j** was obtained using the lactone **8b** (0.7 g, 1.50 mmol), 5-hexyl-2-thienyl magnesiumbromide [prepared from 2-bromo-5-hexylthiophene (0.74 g, 3.00 mmol) and Mg (0.08 g, 3.61 mmol)], and Lawesson's reagent (0.30 g, 0.74 mmol) as a thick orange liquid (0.47 g, 50%); [found: C, 81.8; H, 8.0; S, 10.4. C₄₃H₅₂S₂ requires C, 81.59; H, 8.28; S, 10.13%]; *R_f* (100% hexane) 0.85; ν_{\max} (KBr) 2885, 1595, 1500 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 7.81–7.64 (4H, m, ArH), 7.42–7.40 (2H, m, ArH), 7.19–7.14 (2H, m, ArH), 6.98–6.89 (3H, m, ArH), 6.84–6.83 (2H, m, ArH), 2.91–2.71 (2H, m, CH₂), 2.10–2.05 (4H, m, CH₂), 1.81–1.66 (8H, m, CH₂), 1.45–1.33 (12H, m, CH₂CH₃), 0.98–0.82 (13H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 151.52, 151.08, 147.66, 145.89, 140.75, 140.62, 137.56, 135.69, 135.40, 134.77, 129.41, 128.63, 127.29, 126.94, 126.64, 125.59, 124.94, 123.91, 122.72, 122.37, 121.92, 120.17, 119.87, 55.23, 40.38, 31.64, 31.57, 30.38, 29.98, 29.75, 28.93, 28.86, 28.71, 23.90, 22.64, 14.12; MS (EI): *m/z* (%)=632 [M]⁺.

5.3.11. *1-(9,9-Dihexyl-9H-fluoren-2-yl)-3-phenylbenzo[c]thiophene (9k)*. Following the above-mentioned procedure (B), benzo[c]thiophene **9k** was obtained using the lactone **8b** (0.7 g, 1.50 mmol), phenylmagnesiumbromide [prepared from bromobenzene (0.47 g, 3.00 mmol) and Mg (0.08 g, 3.61 mmol)], and Lawesson's reagent (0.30 g, 0.74 mmol) as a thick yellow liquid (0.46 g, 57%); [found: C, 86.5; H, 7.6; S, 5.7. C₃₉H₄₂S requires C, 86.29; H, 7.80; S, 5.91%]; *R_f* (100% hexane) 0.81; ν_{\max} (KBr) 2905, 1595, 1501 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 7.95–7.89 (4H, m, ArH), 7.84–7.66 (4H, m, ArH), 7.55–7.51 (2H, m, ArH), 7.43–7.33 (4H, m, ArH), 7.16–7.02 (2H, m, ArH), 2.12–2.01 (4H, m, CH₂), 1.20–1.18 (12H, m, CH₂), 0.82–0.78 (10H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 150.89, 150.41, 143.67, 140.13, 139.59, 139.49, 133.15, 129.60, 127.24, 126.54, 126.30, 124.81, 124.63, 124.59, 123.30, 123.24, 122.28, 121.58, 121.20, 119.69, 119.65, 119.45, 119.17, 118.37, 54.63, 39.89, 30.94, 29.15, 23.25, 22.00, 13.44; MS (EI): *m/z* (%)=542 [M]⁺.

5.3.12. *1-(9,9-Dihexyl-9H-fluoren-2-yl)-3-(4-methoxyphenyl)benzo[c]thiophene (9l)*. Following the above-mentioned procedure (B), benzo[c]thiophene **9l** was obtained using the lactone **8b** (0.7 g, 1.50 mmol), *p*-anisylmagnesiumbromide [prepared from 4-bromoanisole (0.56 g, 3.00 mmol) and Mg (0.08 g, 3.61 mmol)], and Lawesson's reagent (0.30 g, 0.74 mmol) as a thick yellow liquid (0.49 g, 58%); [found: C, 83.8; H, 7.8; S, 5.7. C₄₀H₄₄OS requires C, 83.87; H, 7.74; S, 5.60%]; *R_f* (100% hexane) 0.71; ν_{\max} (KBr) 2905, 1595, 1501 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 7.73–7.49 (8H, m, ArH), 7.24–7.20 (3H, m, ArH), 6.98–6.95 (4H, m, ArH), 3.71 (3H, s, OCH₃), 1.83–1.80 (4H, m, CH₂), 1.11–0.89 (12H, m, CH₂), 0.65–0.62 (10H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 158.85, 151.51, 151.01, 143.91, 140.86, 139.89, 130.64, 130.48, 127.06, 126.91, 126.36, 125.10, 124.87, 124.71, 123.69, 122.91, 122.03, 121.33, 120.33, 120.24, 119.86, 119.73, 118.87, 114.60, 55.40, 55.26, 40.56, 31.58, 29.79, 23.89, 22.64, 14.07; MS (EI): *m/z* (%)=572 [M]⁺.

5.3.13. *1-(9,9-Dihexyl-9H-fluoren-2-yl)-3-(4-methylphenyl)benzo[c]thiophene (9m)*. Following the above-mentioned procedure (B), benzo[c]thiophene **9m** was obtained using the lactone **8b** (0.7 g, 1.50 mmol), *p*-tolylmagnesiumbromide [prepared from 4-bromotoluene (0.51 g, 3.00 mmol) and Mg (0.08 g, 3.61 mmol)], and Lawesson's reagent (0.30 g, 0.74 mmol) as a thick yellow liquid (0.47 g, 57%); [found: C, 86.1; H, 8.2; S, 6.0. C₄₀H₄₄S requires C, 86.28; H, 7.96; S, 5.76%]; *R_f* (100% hexane) 0.75; ν_{\max} (KBr) 2855, 1595, 1501 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 7.85–7.79 (6H, m, ArH), 7.77–7.61 (2H, m, ArH), 7.33–7.29 (5H, m, ArH), 7.05–7.01 (2H, m, ArH), 2.41 (3H, s, CH₃), 2.10–2.00 (4H, m, CH₂), 1.11–1.01 (12H, m, CH₂), 0.74–0.72 (10H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 151.46, 151.00, 144.30, 143.94, 140.79, 140.01, 136.80, 130.51, 130.09, 129.68, 127.05, 126.86, 125.03, 124.90, 124.83, 123.78, 122.88, 122.01, 121.88,

120.36, 120.19, 119.70, 118.98, 55.23, 40.49, 31.52, 29.73, 23.82, 22.57, 21.36, 14.00; MS (EI): m/z (%)=556 [M]⁺.

5.3.14. 1-(9,9-Dihexyl-9H-fluoren-2-yl)-3-(naphthalen-1-yl)benzo[c]thiophene (9n). Following the above-mentioned procedure (B), benzo[c]thiophene **9n** was obtained using the lactone **8b** (0.7 g, 1.50 mmol), 1-naphthalenylmagnesiumbromide [prepared from 1-bromonaphthalene (0.62 g, 3.00 mmol) and Mg (0.08 g, 3.61 mmol)], and Lawesson's reagent (0.30 g, 0.74 mmol) as a thick yellow liquid (0.49 g, 55%); [found: C, 86.9; H, 7.6; S, 5.6. C₄₃H₄₄S requires C, 87.11; H, 7.48; S, 5.41%]; R_f (100% hexane) 0.81; ν_{\max} (KBr) 2855, 1595, 1500 cm⁻¹; δ_H (300 MHz, CDCl₃) 8.08–7.83 (9H, m, ArH), 7.60 (2H, t, J 7.5 Hz, ArH), 7.47–7.39 (5H, m, ArH), 7.15–7.12 (2H, m, ArH), 2.20–2.18 (4H, m, CH₂), 1.21–1.11 (12H, m, CH₂), 0.90–0.85 (10H, m, CH₂CH₃); δ_C (75.6 MHz, CDCl₃) 151.58, 151.10, 144.86, 144.77, 143.66, 142.57, 142.11, 140.84, 140.28, 139.77, 136.51, 131.85, 130.51, 129.04, 127.23, 127.00, 126.91, 125.33, 125.16, 124.88, 124.02, 122.97, 122.45, 122.17, 120.52, 120.33, 119.87, 119.18, 55.34, 40.60, 31.65, 29.85, 23.95, 22.70, 14.14; MS (EI): m/z (%)=592 [M]⁺.

5.3.15. 1-(9,9-Dip-tolyl-9H-fluoren-2-yl)-3-(thiophen-2-yl)benzo[c]thiophene (9o). Following the above-mentioned procedure (B), benzo[c]thiophene **9o** was obtained using the lactone **8c** (0.7 g, 1.48 mmol), 2-thienylmagnesium bromide [prepared from 2-bromothiophene (0.48 g, 2.97 mmol) and Mg (0.08 g, 3.61 mmol)], and Lawesson's reagent (0.30 g, 0.74 mmol) as a thick orange liquid (0.37 g, 45%); [found: C, 83.8; H, 5.2; S, 11.2. C₃₉H₂₈S₂ requires C, 83.53; H, 5.03; S, 11.44%]; R_f (100% hexane) 0.82; ν_{\max} (KBr) 2925, 1601, 1505 cm⁻¹; δ_H (300 MHz, CDCl₃) 7.70–7.66 (2H, m, ArH), 7.47–7.46 (2H, m, ArH), 7.37–7.31 (2H, m, ArH), 7.27–7.25 (3H, m, ArH), 7.10–6.98 (13H, m, ArH), 2.27 (6H, s, CH₃); δ_C (75.6 MHz, CDCl₃) 154.61, 152.45, 141.90, 141.25, 139.77, 136.79, 136.57, 136.27, 135.14, 134.95, 133.41, 130.61, 129.10, 128.98, 127.86, 127.76, 127.67, 127.55, 127.37, 126.17, 125.96, 125.47, 124.05, 122.95, 122.31, 121.90, 119.84, 119.28, 62.77, 20.99; MS (EI): m/z (%)=560 [M]⁺.

5.3.16. 1-(9,9-Dip-tolyl-9H-fluoren-2-yl)-3-(3-hexylthiophen-2-yl)benzo[c]thiophene (9p). Following the above-mentioned procedure (B), benzo[c]thiophene **9p** was obtained using the lactone **8b** (0.7 g, 1.48 mmol), 3-hexyl-2-thienyl magnesiumbromide [prepared from 2-bromo-3-hexylthiophene (0.73 g, 2.97 mmol) and Mg (0.08 g, 3.61 mmol)], and Lawesson's reagent (0.30 g, 0.74 mmol) as a thick orange liquid (0.40 g, 42%); [found: C, 83.6; H, 6.1; S, 10.1. C₄₅H₄₀S₂ requires C, 83.80; H, 6.25; S, 9.94%]; R_f (100% hexane) 0.87; ν_{\max} (KBr) 2900, 1595, 1501 cm⁻¹; δ_H (300 MHz, CDCl₃) 7.45–7.42 (5H, m, ArH), 7.38–7.16 (3H, m, ArH), 7.11–7.00 (13H, m, ArH), 2.72 (2H, t, J 7.5 Hz, CH₂), 2.28 (6H, s, CH₃), 1.63–1.59 (2H, m, CH₂), 1.35–1.25 (6H, m, CH₂), 0.85–0.75 (3H, m, CH₃); δ_C (75.6 MHz, CDCl₃) 154.59, 152.61, 147.15, 146.10, 145.61, 144.93, 142.00, 141.00, 140.61, 137.61, 136.60, 134.81, 132.85, 130.63, 129.22, 128.70, 127.30, 127.02, 125.90, 124.03, 123.80, 123.79, 123.01, 122.45, 121.60, 120.20, 119.90, 62.73, 40.40, 29.10, 23.95, 22.61, 20.93, 14.05; MS (EI): m/z (%)=644 [M]⁺.

5.3.17. 1-(9,9-Dip-tolyl-9H-fluoren-2-yl)-3-(5-hexylthiophen-2-yl)benzo[c]thiophene (9q). Following the above-mentioned procedure (B), benzo[c]thiophene **9q** was obtained using the lactone **8b** (0.7 g, 1.48 mmol), 5-hexyl-2-thienyl magnesiumbromide [prepared from 2-bromo-5-hexylthiophene (0.73 g, 2.97 mmol) and Mg (0.08 g, 3.61 mmol)], and Lawesson's reagent (0.30 g, 0.74 mmol) as a thick orange liquid (0.44 g, 47%); [found: C, 83.6; H, 6.1; S, 10.2. C₄₅H₄₀S₂ requires C, 83.80; H, 6.25; S, 9.94%]; R_f (100% hexane) 0.87; ν_{\max} (KBr) 2900, 1595, 1501 cm⁻¹; δ_H (300 MHz, CDCl₃) 7.65–7.62 (4H, m, ArH), 7.45–7.35 (3H, m, ArH), 7.32–7.25 (2H, m, ArH), 7.13–7.00 (12H, m, ArH), 2.73–2.68 (2H, m, CH₂), 2.27 (6H, s, CH₃), 1.63–1.57 (2H, m, CH₂), 1.30–1.23 (6H, m, CH₂), 0.82–0.71 (3H, m, CH₃); δ_C (75.6 MHz, CDCl₃) 154.60, 152.65, 147.13, 146.10, 145.60, 144.93,

142.01, 141.02, 140.61, 137.62, 136.62, 134.80, 132.87, 130.65, 129.23, 128.71, 127.32, 127.00, 125.91, 124.01, 123.82, 123.79, 123.00, 122.45, 121.62, 120.22, 119.60, 62.72, 40.41, 29.11, 23.97, 22.62, 20.91, 14.01; MS (EI): m/z (%)=644 [M]⁺.

5.3.18. 1-(5-(9,9-Dihexyl-9H-fluoren-2-yl)thiophen-2-yl)-3-(thiophen-2-yl)benzo[c]thiophene (13a). Following the above-mentioned procedure (B), benzo[c]thiophene **13a** was obtained using the lactone **12a** (0.6 g, 1.10 mmol), 2-thienylmagnesium bromide [prepared from 2-bromothiophene (0.53 g, 3.30 mmol) and Mg (0.09 g, 3.90 mmol)], and Lawesson's reagent (0.22 g, 0.55 mmol) as a thick red liquid (0.35 g, 51%); [found: C, 78.3; H, 7.0; S, 15.0. C₄₁H₄₂S₃ requires C, 78.05; H, 6.71; S, 15.25%]; R_f (100% hexane) 0.85; ν_{\max} (KBr) 2915, 1601, 1505 cm⁻¹; δ_H (300 MHz, CDCl₃) 8.43–8.41 (1H, m, ArH), 7.87 (1H, d, J 9 Hz, ArH), 7.80–7.75 (2H, m, ArH), 7.71–7.67 (2H, m, ArH), 7.64–7.54 (7H, m, ArH), 7.46–7.43 (3H, m, ArH), 1.91–1.86 (4H, m, CH₂), 1.25–0.97 (12H, m, CH₂), 0.76–0.53 (10H, m, CH₂CH₃); δ_C (75.6 MHz, CDCl₃) 152.17, 150.89, 146.03, 141.54, 139.71, 135.68, 135.33, 132.72, 131.09, 131.03, 130.78, 130.43, 129.97, 129.92, 129.09, 128.40, 127.89, 127.76, 127.01, 126.50, 125.96, 125.82, 123.94, 123.13, 120.73, 119.23, 55.18, 39.98, 31.48, 29.66, 23.77, 22.58, 14.01; MS (EI): m/z (%)=630 [M]⁺.

5.3.19. 1-(5-(9,9-Dihexyl-9H-fluoren-2-yl)thiophen-2-yl)-3-(3-hexylthiophen-2-yl)benzo[c]thiophene (13b). Following the above-mentioned procedure (B), benzo[c]thiophene **13b** was obtained using the lactone **12a** (0.6 g, 1.10 mmol), 3-hexyl-2-thienyl magnesiumbromide [prepared from 2-bromo-3-hexylthiophene (0.87 g, 3.30 mmol) and Mg (0.09 g, 3.90 mmol)], and Lawesson's reagent (0.22 g, 0.55 mmol) as a thick red liquid (0.37 g, 47%); [found: C, 78.7; H, 7.8; S, 13.7. C₄₇H₅₄S₃ requires C, 78.94; H, 7.61; S, 13.45%]; R_f (100% hexane) 0.85; ν_{\max} (KBr) 2915, 1601, 1505 cm⁻¹; δ_H (300 MHz, CDCl₃) 8.43–8.41 (1H, m, ArH), 7.87 (1H, d, J 9 Hz, ArH), 7.80–7.75 (2H, m, ArH), 7.71–7.67 (3H, m, ArH), 7.64–7.54 (3H, m, ArH), 7.46–7.43 (2H, m, ArH), 7.33–7.30 (3H, m, ArH), 2.77–2.71 (2H, m, CH₂), 1.92–1.85 (4H, m, CH₂), 1.69–1.60 (2H, m, CH₂), 1.35–1.12 (18H, m, CH₂), 0.90–0.81 (13H, m, CH₂CH₃); δ_C (75.6 MHz, CDCl₃) 152.93, 150.25, 147.57, 143.70, 139.99, 137.16, 132.94, 131.62, 130.30, 130.12, 129.80, 129.63, 129.33, 128.89, 128.29, 127.42, 126.88, 126.84, 126.07, 125.13, 124.74, 124.29, 122.82, 122.11, 121.10, 120.98, 120.15, 119.71, 55.32, 40.26, 31.49, 31.45, 31.39, 30.30, 29.62, 28.63, 23.64, 22.55, 14.06, 13.98; MS (EI): m/z (%)=714 [M]⁺.

5.3.20. 1-(5-(9,9-Dihexyl-9H-fluoren-2-yl)thiophen-2-yl)-3-(5-hexylthiophen-2-yl)benzo[c]thiophene (13c). Following the above-mentioned procedure (B), benzo[c]thiophene **13c** was obtained using the lactone **12a** (0.6 g, 1.10 mmol), 5-hexyl-2-thienyl magnesiumbromide [prepared from 2-bromo-5-hexylthiophene (0.87 g, 3.30 mmol) and Mg (0.09 g, 3.90 mmol)], and Lawesson's reagent (0.22 g, 0.55 mmol) as a thick red liquid (0.36 g, 45%); [found: C, 78.7; H, 7.9; S, 13.7. C₄₇H₅₄S₃ requires C, 78.94; H, 7.61; S, 13.45%]; R_f (100% hexane) 0.85; ν_{\max} (KBr) 2911, 1600, 1505 cm⁻¹; δ_H (300 MHz, CDCl₃) 8.42–8.41 (1H, m, ArH), 7.85 (1H, d, J 8.5 Hz, ArH), 7.82–7.75 (2H, m, ArH), 7.73–7.67 (3H, m, ArH), 7.63–7.51 (3H, m, ArH), 7.43–7.41 (2H, m, ArH), 7.33–7.30 (3H, m, ArH), 2.77–2.71 (2H, m, CH₂), 1.93–1.82 (4H, m, CH₂), 1.71–1.60 (2H, m, CH₂), 1.37–1.12 (18H, m, CH₂), 0.90–0.81 (13H, m, CH₂CH₃); δ_C (75.6 MHz, CDCl₃) 152.17, 150.89, 146.03, 141.54, 139.71, 135.68, 135.33, 132.72, 131.09, 131.03, 130.78, 130.43, 129.97, 129.92, 129.09, 128.40, 127.89, 127.76, 127.01, 126.50, 125.96, 125.82, 123.94, 123.13, 120.73, 119.23, 55.18, 39.98, 31.48, 31.43, 31.37, 30.22, 29.66, 28.61, 23.77, 22.58, 14.01, 13.97; MS (EI): m/z (%)=714 [M]⁺.

5.3.21. 1-(5-(9,9-Dihexyl-9H-fluoren-2-yl)thiophen-2-yl)-3-phenylbenzo[c]thiophene (13d). Following the above-mentioned procedure (B), benzo[c]thiophene **13d** was obtained using the lactone

12a (0.6 g, 1.10 mmol), phenylmagnesiumbromide [prepared from bromobenzene (0.51 g, 3.30 mmol) and Mg (0.09 g, 3.90 mmol)], and Lawesson's reagent (0.22 g, 0.55 mmol) as a thick orange liquid (0.33 g, 49%); [found: C, 82.5; H, 6.9; S, 10.5. C₄₃H₄₄S₂ requires C, 82.64; H, 7.10; S, 10.26%]; *R_f* (100% hexane) 0.81; ν_{\max} (KBr) 2905, 1601, 1505 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 7.92–7.89 (1H, m, ArH), 7.87–7.83 (2H, m, ArH), 7.79–7.74 (2H, m, ArH), 7.73–7.70 (2H, m, ArH), 7.67–7.52 (2H, m, ArH), 7.37–7.23 (4H, m, ArH), 7.16–7.03 (3H, m, ArH), 7.07–7.02 (2H, m, ArH), 2.07–2.00 (4H, m, CH₂), 1.14–1.06 (12H, m, CH₂), 0.77–0.72 (10H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 152.91, 150.22, 14.55, 143.71, 139.97, 137.11, 132.95, 131.62, 130.31, 130.15, 129.81, 129.63, 129.35, 128.91, 128.31, 127.41, 126.89, 126.85, 126.05, 125.11, 124.75, 124.27, 122.81, 122.11, 121.11, 120.97, 120.15, 119.72, 55.23, 40.48, 31.45, 29.74, 23.83, 22.59, 13.99; MS (EI): *m/z* (%)=624 [M]⁺.

5.3.22. 1-(5-(9,9-Dihexyl-2-(thiophen-2-yl)-9H-fluoren-2-yl)thiophen-2-yl)-3-(thiophen-2-yl)benzo[c]thiophene (**13e**). Following the above-mentioned procedure (B), benzo[c]thiophene **13e** was obtained using the lactone **12b** (0.7 g, 1.11 mmol), 2-thienylmagnesium bromide [prepared from 2-bromothiophene (0.54 g, 3.33 mmol) and Mg (0.09 g, 3.90 mmol)], and Lawesson's reagent (0.22 g, 0.55 mmol) as a thick red liquid (0.43 g, 55%); [found: C, 75.6; H, 6.5; S, 17.7. C₄₅H₄₄S₄ requires C, 75.79; H, 6.22; S, 17.99%]; *R_f* (100% hexane) 0.85; ν_{\max} (KBr) 2915, 1601, 1505 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 8.53 (1H, d, J 9 Hz, ArH), 7.89 (1H, d, J 9 Hz, ArH), 7.73–7.63 (4H, m, ArH), 7.49–7.41 (7H, m, ArH), 7.28–6.99 (5H, m, ArH), 1.91–1.85 (4H, m, CH₂), 1.11–1.03 (12H, m, CH₂), 0.78–0.56 (10H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 152.60, 140.85, 140.71, 140.19, 139.77, 139.65, 139.10, 136.77, 133.84, 130.19, 129.91, 127.97, 127.09, 126.20, 126.08, 125.40, 125.34, 125.04, 125.00, 124.34, 123.86, 122.85, 122.78, 122.29, 121.99, 121.53, 121.19, 120.90, 119.89, 119.79, 55.72, 40.21, 31.51, 29.62, 23.70, 22.63, 14.07; MS (EI): *m/z* (%)=712 [M]⁺.

5.3.23. 1-(5-(9,9-Dihexyl-2-(thiophen-2-yl)-9H-fluoren-2-yl)thiophen-2-yl)-3-(3-hexylthiophen-2-yl)benzo[c]thiophene (**13f**). Following the above-mentioned procedure (B), benzo[c]thiophene **13f** was obtained using the lactone **12b** (0.7 g, 1.11 mmol), 3-hexyl-2-thienyl magnesiumbromide [prepared from 2-bromo-3-hexylthiophene (0.82 g, 3.33 mmol) and Mg (0.09 g, 3.90 mmol)], and Lawesson's reagent (0.22 g, 0.55 mmol) as a thick red liquid (0.50 g, 57%); [found: C, 76.5; H, 6.9; S, 16.3. C₅₁H₅₆S₄ requires C, 76.83; H, 7.08; S, 16.09%]; *R_f* (100% hexane) 0.85; ν_{\max} (KBr) 2915, 1601, 1505 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 8.47–8.43 (1H, m, ArH), 7.87 (1H, d, J 8.5 Hz, ArH), 7.83–7.75 (3H, m, ArH), 7.71–7.67 (3H, m, ArH), 7.64–7.52 (4H, m, ArH), 7.46–7.41 (2H, m, ArH), 7.37–7.31 (3H, m, ArH), 2.77–2.69 (2H, m, CH₂), 1.95–1.85 (4H, m, CH₂), 1.69–1.61 (2H, m, CH₂), 1.35–1.15 (18H, m, CH₂), 0.92–0.81 (13H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 152.71, 150.22, 145.53, 143.77, 140.71, 140.19, 139.77, 139.65, 139.10, 136.77, 133.84, 130.19, 129.91, 129.39, 127.97, 127.09, 126.20, 126.08, 125.40, 125.34, 125.04, 124.34, 123.86, 122.85, 122.78, 122.29, 121.99, 121.53, 121.19, 120.90, 120.22, 119.79, 55.37, 40.22, 31.53, 31.47, 31.37, 30.32, 29.62, 23.71, 22.63, 14.01, 13.93; MS (EI): *m/z* (%)=796 [M]⁺.

5.3.24. 1-(5-(9,9-Dihexyl-2-(thiophen-2-yl)-9H-fluoren-2-yl)thiophen-2-yl)-3-(5-hexylthiophen-2-yl)benzo[c]thiophene (**13g**). Following the above-mentioned procedure (B), benzo[c]thiophene **13g** was obtained using the lactone **12b** (0.7 g, 1.11 mmol), 5-hexyl-2-thienyl magnesiumbromide [prepared from 2-bromo-5-hexylthiophene (0.82 g, 3.33 mmol) and Mg (0.09 g, 3.90 mmol)], and Lawesson's reagent (0.22 g, 0.55 mmol) as a thick red liquid (0.44 g, 50%); [found: C, 76.6; H, 6.9; S, 16.3. C₅₁H₅₆S₄ requires C, 76.83; H, 7.08; S, 16.09%]; *R_f* (100% hexane) 0.87; ν_{\max} (KBr) 2915, 1611, 1501 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 8.51 (1H, d, J 9 Hz, ArH), 7.81 (1H,

d, J 9 Hz, ArH), 7.73–7.63 (4H, m, ArH), 7.49–7.41 (7H, m, ArH), 7.28–6.99 (4H, m, ArH), 2.77–2.71 (2H, m, CH₂), 1.92–1.85 (4H, m, CH₂), 1.69–1.60 (2H, m, CH₂), 1.35–1.11 (18H, m, CH₂), 0.90–0.81 (13H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 152.97, 150.28, 147.56, 143.75, 137.21, 132.98, 131.67, 130.81, 130.33, 130.20, 129.93, 129.86, 129.67, 129.39, 128.96, 128.92, 128.34, 128.03, 127.86, 127.50, 126.97, 126.14, 125.22, 124.78, 124.66, 122.56, 122.20, 121.58, 121.18, 121.06, 120.21, 119.80, 55.37, 40.35, 31.57, 31.45, 31.39, 30.30, 29.71, 28.62, 23.63, 22.64, 14.10, 13.91; MS (EI): *m/z* (%)=796 [M]⁺.

5.4. A representative procedure for the preparation of benzo[c]selenophene (**15a**) from lactone (**8b**) (procedure C)

5.4.1. 1-(9,9-Dihexyl-9H-fluoren-2-yl)-3-(thiophen-2-yl)benzo[c]selenophene (**15a**). To a solution of lactone **8b** (0.7 g, 1.50 mmol) in anhydrous THF (25 mL) was added 2-thienylmagnesium bromide [prepared from 2-bromothiophene (0.49 g, 3.00 mmol) and magnesium turnings (0.08 g, 3.61 mmol)] at 0 °C under N₂. The reaction mixture was slowly raised to room temperature and stirred for 4 h. It was then quenched with 3 M HCl solution, extracted with DCM (2 × 20 mL), and dried (Na₂SO₄). The DCM solution of benzo[c]furan was then stirred with Woollins reagent (0.20 g, 0.37 mmol) at room temperature for 4 h. Solvent was evaporated in vacuo to give the crude product, which was purified by column chromatography (100% hexane) to give the title compound **15a** (0.53 g, 59%) as an orange liquid; [found: C, 74.4; H, 6.5; S, 5.5. C₃₇H₄₀SSe requires C, 74.60; H, 6.77; S, 5.38%]; *R_f* (100% hexane) 0.81; ν_{\max} (KBr) 2895, 1601, 1505 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 7.87 (1H, d, J 8.4 Hz, ArH), 7.77–7.66 (3H, m, ArH), 7.57–7.55 (2H, m, ArH), 7.36–7.29 (5H, m, ArH), 7.14–7.11 (1H, m, ArH), 7.04–6.94 (2H, m, ArH), 2.0 (4H, q, J 5.23 Hz, CH₂), 1.09–1.07 (12H, m, CH₂), 0.79–0.74 (10H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 151.55, 151.09, 144.31, 140.88, 140.55, 138.27, 137.90, 137.54, 134.58, 134.37, 128.63, 127.92, 127.32, 126.94, 125.89, 125.64, 124.36, 124.02, 123.97, 122.97, 122.14, 121.97, 120.18, 119.88, 55.23, 40.34, 31.52, 29.73, 23.87, 22.60, 14.04; MS (EI): *m/z* (%)=596 [M]⁺.

5.4.2. 1-(9,9-Dihexyl-9H-fluoren-2-yl)-3-(3-hexylthiophen-2-yl)benzo[c]selenophene (**15b**). Following the above-mentioned procedure (C), benzo[c]selenophene **15b** was obtained using the lactone **8b** (0.7 g, 1.50 mmol), 3-hexyl-2-thienyl magnesiumbromide [prepared from 2-bromo-3-hexylthiophene (0.74 g, 3.00 mmol) and Mg (0.08 g, 3.61 mmol)], and Woollins reagent (0.20 g, 0.37 mmol) as a thick orange liquid (0.63 g, 62%); [found: C, 76.1; H, 7.7; S, 4.7. C₄₃H₅₂SSe requires C, 75.96; H, 7.71; S, 4.72%]; *R_f* (100% hexane) 0.85; ν_{\max} (KBr) 2895, 1601, 1505 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 7.85–7.74 (3H, m, ArH), 7.66–7.63 (2H, m, ArH), 7.48–7.37 (5H, m, ArH), 7.12 (1H, d, J 5.4 Hz, ArH), 7.04–7.00 (2H, m, ArH), 2.71 (2H, t, J 7.8 Hz, CH₂), 2.10–2.05 (4H, m, CH₂), 1.68–1.61 (2H, m, CH₂), 1.32–1.15 (18H, m, CH₂), 0.90–0.81 (13H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 151.53, 151.08, 146.09, 141.96, 140.80, 140.60, 136.60, 134.81, 132.81, 130.62, 129.21, 128.65, 127.30, 126.94, 125.88, 124.01, 123.78, 123.74, 122.97, 122.43, 121.55, 120.16, 119.87, 55.24, 40.38, 31.65, 31.53, 30.79, 29.73, 29.14, 29.07, 23.88, 22.60, 14.09, 14.04; MS (EI): *m/z* (%)=680 [M]⁺.

5.4.3. 1-(9,9-Dihexyl-9H-fluoren-2-yl)-3-(5-hexylthiophen-2-yl)benzo[c]selenophene (**15c**). Following the above-mentioned procedure (C), benzo[c]selenophene **15c** was obtained using the lactone **8b** (0.7 g, 1.50 mmol), 5-hexyl-2-thienyl magnesiumbromide [prepared from 2-bromo-5-hexylthiophene (0.74 g, 3.00 mmol) and Mg (0.08 g, 3.61 mmol)], and Woollins reagent (0.20 g, 0.37 mmol) as a thick orange liquid (0.56 g, 55%); [found: C, 76.2; H, 7.9; S, 4.5. C₄₃H₅₂SSe requires C, 75.96; H, 7.71; S, 4.72%]; *R_f* (100% hexane) 0.85; ν_{\max} (KBr) 2895, 1601, 1505 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 7.84–7.73 (4H, m, ArH), 7.65–7.62 (2H, m, ArH), 7.47–7.37

(4H, m, ArH), 7.11 (1H, d, *J* 5.4 Hz, ArH), 7.04–7.00 (2H, m, ArH), 2.69 (2H, t, *J* 7.8 Hz, CH₂), 2.09–2.03 (4H, m, CH₂), 1.67–1.62 (2H, m, CH₂), 1.30–1.16 (18H, m, CH₂), 0.93–0.77 (13H, m, CH₂CH₃); δ_C (75.6 MHz, CDCl₃); 151.52, 151.07, 146.09, 141.95, 140.79, 140.59, 136.58, 134.79, 132.80, 130.61, 129.21, 128.64, 127.29, 126.93, 125.88, 124.00, 123.77, 123.74, 122.96, 122.42, 121.54, 120.15, 119.87, 55.23, 40.37, 31.64, 31.52, 30.79, 29.72, 29.13, 29.07, 23.87, 22.60, 14.07, 14.04; MS (EI): *m/z* (%)=680 [M]⁺.

5.5. A representative procedure for the preparation of compound 16a (procedure D)

To a stirred solution of **9a** (0.4 g, 0.92 mmol) in dry DCM (20 mL) was added FeCl₃ (0.30 g, 1.85 mmol) under N₂ atmosphere. The reaction mixture was stirred for 6 h at room temperature and quenched with dilute solution of N₂H₄·H₂O. Then, it was filtered through Celite, extracted with DCM, and dried (Na₂SO₄). Evaporation of solvent followed by column chromatographic purification afforded **16a** as a semi solid (0.18 g, 45%).

5.5.1. 5,5'-Bis(3-(9,9-diethyl-9H-fluoren-2-yl)benzo[c]thiophen-1-yl)-2,2'-bithiophene (16a). Semi solid; [found: C, 80.1; H, 5.4; S, 14.6. C₅₈H₄₆S₄ requires C, 79.96; H, 5.32; S, 14.72%]; R_f (100% hexane) 0.65; ν_{\max} (KBr) 2891, 1595, 1500 cm⁻¹; δ_H (300 MHz, CDCl₃) 7.98 (2H, d, *J* 8.7 Hz, ArH), 7.80–7.61 (4H, m, ArH), 7.52–7.51 (2H, m, ArH), 7.31–7.00 (14H, m, ArH), 6.89–6.86 (4H, m, ArH), 2.00 (8H, q, *J* 7.4 Hz, CH₂), 0.35 (12H, t, *J* 7.4 Hz, CH₃); δ_C (75.6 MHz, CDCl₃) 150.90, 150.22, 141.33, 140.93, 136.55, 135.44, 135.30, 135.15, 132.54, 128.15, 127.35, 127.01, 126.95, 126.30, 126.05, 125.77, 124.89, 124.53, 124.30, 123.65, 123.00, 121.69, 120.22, 119.86, 56.28, 32.77, 8.62; MS (EI): *m/z* (%)=870 [M]⁺.

5.5.2. 5,5'-Bis(3-(9,9-hexyl-9H-fluoren-2-yl)benzo[c]thiophen-1-yl)-2,2'-bithiophene (16b). Following the above-mentioned procedure (D), dimer **16b** was obtained using the benzo[c]thiophene **9h** (0.7 g, 1.50 mmol) and FeCl₃ (0.36 g, 2.29 mmol) as a red liquid (0.28 g, 40%); [found: C, 80.9; H, 7.4; S, 11.5. C₇₄H₇₈S₄ requires C, 81.12; H, 7.18; S, 11.71%]; R_f (100% hexane) 0.77; ν_{\max} (KBr) 2891, 1595, 1500 cm⁻¹; δ_H (300 MHz, CDCl₃); 7.67–7.64 (4H, m, ArH), 7.60–7.54 (10H, m, ArH), 7.37–7.36 (4H, m, ArH), 7.27 (4H, d, *J* 5.1 Hz, ArH), 7.10–7.07 (4H, m, ArH), 2.04–1.99 (8H, m, CH₂), 1.11–1.05 (24H, m, CH₂), 0.76–0.71 (20H, m, CH₂CH₃); δ_C (75.6 MHz, CDCl₃) 151.60, 151.07, 144.31, 140.78, 140.25, 140.15, 133.80, 130.26, 127.90, 127.20, 126.96, 125.47, 125.36, 125.25, 123.97, 123.90, 122.94, 122.24, 121.86, 120.35, 120.31, 120.10, 119.83, 119.03, 55.29, 40.55, 31.60, 29.81, 23.91, 22.66, 14.10; MS (EI): *m/z* (%)=1094 [M]⁺.

5.6. A representative procedure for the preparation of compound 17a (procedure E)

To a stirred solution of DMF (1 mL) in dry DCM, POCl₃ (0.37 g, 2.42 mmol) was added to get yellow solution. To this solution **9a** (0.7 g, 1.60 mmol) was added slowly and stirred overnight at room temperature. Then, the reaction mixture was quenched with water, extracted with DCM, and dried. Evaporation solvent followed by column chromatographic purification gave **17a** (0.42 g, 57%) as a red solid.

5.6.1. 5-((3-(9,9-Diethyl-9H-fluoren-2-yl)benzo[c]thiophen-1-yl)-thiophene-2-yl)carbaldehyde (17a). Mp 95 °C; [found: C, 77.3; H, 5.5; S, 13.6. C₃₀H₂₄OS₂ requires C, 77.55; H, 5.21; S, 13.80%]; R_f (5% EA/hexane) 0.63; ν_{\max} (KBr) 2891, 1670, 1601, 1500 cm⁻¹; δ_H (300 MHz, CDCl₃) 9.91 (1H, s, CHO), 8.09 (1H, d, *J* 8.7 Hz, ArH), 7.86–7.78 (3H, m, ArH), 7.69 (2H, d, *J* 6.0 Hz, ArH), 7.67–7.61 (3H, m, ArH), 7.47 (1H, d, *J* 4.2 Hz, ArH), 7.37–7.36 (3H, m, ArH), 2.09 (4H, q, *J* 7.35 Hz, CH₂), 0.41 (6H, t, *J* 7.35 Hz, CH₃); δ_C (75.6 MHz, CDCl₃)

182.28, 151.03, 150.25, 146.50, 141.99, 141.52, 140.72, 138.81, 137.13, 136.53, 135.65, 131.87, 128.33, 127.59, 127.07, 126.30, 124.91, 124.78, 123.77, 123.04, 121.93, 121.30, 120.31, 119.98, 56.33, 32.73, 8.59; MS (EI): *m/z* (%)=464 [M]⁺.

5.6.2. 5-((3-(9,9-Dihexyl-9H-fluoren-2-yl)benzo[c]thiophen-1-yl)-thiophene-2-yl)carbaldehyde (17b). Following the above-mentioned procedure (E), aldehyde **17b** was obtained using the benzo[c]thiophene **9h** (0.7 g, 1.27 mmol), DMF (1 mL), and POCl₃ (0.29 g, 1.90 mmol) as a red liquid (0.45 g, 61%); [found: C, 79.4; H, 7.2; S, 11.0. C₃₈H₄₀OS₂ requires C, 79.12; H, 6.99; S, 11.12%]; R_f (5% EA/hexane) 0.67; ν_{\max} (KBr) 2895, 1675, 1601, 1505 cm⁻¹; δ_H (300 MHz, CDCl₃) 9.61 (1H, s, CHO), 7.81 (1H, d, *J* 8.3 Hz, ArH), 7.81–7.67 (3H, m, ArH), 7.66–7.61 (5H, m, ArH), 7.41 (1H, d, *J* 3.9 Hz, ArH), 7.37–7.27 (3H, m, ArH), 2.22–2.17 (4H, m, CH₂), 1.31–1.15 (12H, m, CH₂), 0.95–0.81 (10H, m, CH₂CH₃); δ_C (75.6 MHz, CDCl₃) 181.71, 150.73, 147.15, 141.72, 141.50, 140.51, 138.47, 137.69, 136.11, 135.51, 133.87, 132.09, 128.53, 127.69, 126.91, 125.11, 124.67, 123.93, 123.11, 121.52, 121.42, 120.69, 119.12, 55.13, 40.67, 31.89, 29.49, 23.07, 22.71, 14.22; MS (EI): *m/z* (%)=576 [M]⁺.

5.6.3. 2-((5-(1-(9,9-Dihexyl-9H-fluoren-2-yl)benzo[c]thiophen-3-yl)thiophen-2-yl)methylene)malononitrile (18a). Few drops of piperidine was added to the solution of **17b** (0.3 g, 0.53 mmol), malononitrile (0.10 g, 1.56 mmol) in ethanol and stirred for 10 h. The reaction mixture was poured into water. The precipitated solid was filtered off and purified by column chromatography to afford **18a** (0.13 g, 40%) as a black solid. Mp 151 °C; [found: C, 79.0; H, 6.7; N, 4.8; S, 10.0. C₄₁H₄₀N₂S₂ requires C, 78.80; H, 6.45; N, 4.48; S, 10.26%]; R_f (5% EA/hexane) 0.55; ν_{\max} (KBr) 2895, 2215, 1675, 1601, 1505 cm⁻¹; δ_H (300 MHz, CDCl₃) 7.63–7.61 (2H, m, ArH), 7.59–7.49 (5H, m, ArH), 7.39–7.35 (1H, m, ArH), 7.31–7.23 (3H, m, ArH), 7.14–7.05 (3H, m, ArH), 2.15–2.03 (4H, m, CH₂), 1.30–1.17 (12H, m, CH₂), 0.91–0.81 (10H, m, CH₂CH₃); δ_C (75.6 MHz, CDCl₃) 152.13, 151.64, 151.04, 140.84, 140.56, 136.09, 135.49, 135.14, 134.93, 132.58, 130.09, 129.93, 129.02, 128.12, 127.89, 127.32, 126.93, 125.35, 125.29, 124.66, 124.37, 123.65, 122.96, 121.57, 121.43, 120.26, 119.89, 55.26, 40.38, 31.53, 29.73, 23.86, 22.62, 14.06; MS (EI): *m/z* (%)=624 [M]⁺.

5.7. A representative procedure for the preparation of compound 18b (procedure F)

To the solution of **17a** (0.3 g, 0.64 mmol) and thiophene-2-acetonitrile (0.16 g, 1.29 mmol) in dry THF, a solution of *t*-BuOK (0.14 g, 1.29 mmol) in THF was added dropwise and stirred for 10 h under nitrogen atmosphere. The reaction mixture was poured into water, extracted with DCM followed by column chromatographic purification gave **18b** (0.16 g, 45%) as a black solid.

5.7.1. 3-(5-(1-(9,9-Diethyl-9H-fluoren-2-yl)benzo[c]thiophen-3-yl)thiophen-2-yl)-2-(thiophen-2-yl)acrylonitrile (18b). Mp 137 °C; [found: C, 76.1; H, 4.9; N, 2.2; S, 17.1. C₃₆H₂₇N₃S₃ requires C, 75.88; H, 4.78; N, 2.46; S, 16.88%]; R_f (5% EA/hexane) 0.55; ν_{\max} (KBr) 2895, 2220, 1675, 1601, 1505 cm⁻¹; δ_H (300 MHz, CDCl₃) 8.52–8.49 (1H, m, ArH), 7.90–7.81 (3H, m, ArH), 7.80–7.73 (1H, m, ArH), 7.67–7.61 (4H, m, ArH), 7.55–7.51 (2H, m, ArH), 7.43–7.33 (4H, m, ArH), 7.23–7.15 (2H, m, ArH), 2.12–2.01 (4H, m, CH₂), 0.81–0.73 (6H, m, CH₃); δ_C (75.6 MHz, CDCl₃) 151.22, 151.05, 144.61, 144.17, 141.78, 140.11, 134.35, 131.07, 130.59, 128.66, 128.56, 127.90, 127.35, 127.22, 126.89, 126.61, 126.29, 126.22, 125.83, 125.50, 125.27, 124.82, 124.39, 123.73, 122.91, 121.47, 120.48, 120.27, 120.11, 119.24, 118.97, 55.25, 32.75, 8.61; MS (EI): *m/z* (%)=569 [M]⁺.

5.7.2. 3-(5-(1-(9,9-Dihexyl-9H-fluoren-2-yl)benzo[c]thiophen-3-yl)thiophen-2-yl)-2-(thiophen-2-yl)acrylonitrile (18c). Following the above-mentioned procedure (F), compound **18c** was prepared

using *t*-BuOK (0.12 g, 1.04 mmol), aldehyde **17b** (0.3 g, 0.53 mmol), and thiophene-2-acetonitrile (0.13 g, 1.04 mmol) as a black solid (0.17 g, 47%); mp 117 °C; [found: C, 77.3; H, 6.3; N, 2.2; S, 14.2. C₄₄H₄₃NS₃ requires C, 77.49; H, 6.35; N, 2.05; S, 14.10%]; *R*_f (5% EA/hexane) 0.57; ν_{max} (KBr) 2895, 2217, 1675, 1601, 1505 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 8.52–8.49 (1H, m, ArH), 7.90–7.81 (3H, m, ArH), 7.80–7.73 (1H, m, ArH), 7.66–7.59 (4H, m, ArH), 7.55–7.51 (2H, m, ArH), 7.43–7.33 (4H, m, ArH), 7.13 (1H, t, *J* 8.7 Hz, ArH), 7.10 (1H, t, *J* 8.4 Hz, ArH), 2.12–2.01 (4H, m, CH₂), 1.20–1.18 (12H, m, CH₂), 0.84–0.78 (10H, m, CH₂CH₃); δ_{C} (75.6 MHz, CDCl₃) 151.57, 151.05, 145.61, 144.17, 141.78, 140.16, 134.35, 131.07, 130.59, 128.66, 128.56, 127.90, 127.35, 127.11, 126.89, 126.61, 126.29, 126.22, 125.83, 125.50, 125.27, 124.82, 124.19, 123.69, 122.91, 121.47, 120.48, 120.27, 120.19, 119.74, 118.97, 55.25, 40.49, 31.54, 29.76, 23.85, 22.60, 14.01; MS (EI): *m/z* (%)=681 [M]⁺.

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