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Synthesis and characterization of 1,3-diarylbenzo[*c*]selenophenes

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ABSTRACT

A series of 1,3-diarylbenzo[*c*]selenophenes (symmetrical/unsymmetrical) have been synthesized involving a selenium transfer reaction of keto-alcohol/benzo[*c*]furan using Woollins reagent. The optical and electrochemical studies of these diarylbenzo[*c*]selenophenes are correlated with their structures.

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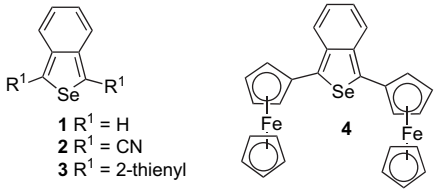
1. Introduction

Conjugated macromolecules attracted considerable interest in the past two decades due to their unique electrical and optical properties.[1] Among the various conjugated systems developed, poly(thiophene) derivatives have been paid much attention due to their electronic properties, stability, and structural versatility.[2] The synthesis and electrochemical properties of benzo[*c*]thiophene analogs have been well explored due to the low band-gap properties associated with their polymer.[3,4] However, the unstable benzo[*c*]-selenophene **1** and its polymer have received only scant attention, Scheme 1. Hence, the synthesis of stable 1,3-diarylbenzo[*c*]-selenophene analogs will be highly useful for further understanding of their physical properties.



Scheme 1.

The synthesis of dicyanobenzo[*c*]thiophene and dicyanobenzo[*c*]selenophene **2** analogs starting from *o*-xylene dicyanide using $SOCl_2$ and $SeOCl_2$ as sulfur and selenium transfer reagents have been reported,[5] Scheme 1. The synthesis of 1,3-disubstituted benzo[*c*]selenophene analogs using the procedure established for naphtho[*c*]thiophenes[6] is also achieved. Cava and co-workers have reported 3,4-ethylenedioxyselenophene as a novel building block for electron rich π-conjugated polymers.[7] The same group also reported the synthesis of benzo[*c*]selenophene via bromination–dehydrobromination as well as oxidation of the 1,3-dihydrobenzo[*c*]selenophene.[8] Aqad and co-workers reported the synthesis and electrochemical studies of seleno[3,4-*b*]quinoxalines.[9] The synthesis of methylthiocapped bi-isothianaphthalene and bi-isoselenophene derivatives[10] was also achieved. Larock and co-workers recently reported 2,3-disubstituted benzo[*b*]-selenophenes.[11]

Satoshi Ogawa and co-workers outlined the synthesis and electrochemical studies of 1,3-diferrocenyl benzo[*c*]selenophene **4**.[12]

2. Results and discussion

The incredible properties of conducting poly(benzo[*c*]-thiophene) led us to explore the synthesis of the corresponding selenium analog namely benzo[*c*]selenophenes, which will be useful for further understanding of its optical properties. We have recently communicated the synthesis of 1,3-diarylbenzo-[*c*]thiophenes[13] as well as benzo[*c*]selenophene[14] analogs.

A survey of the literature revealed that Woollins reagent $[PhP(Se)(\mu\text{-}Se)]_2$, a selenium analog of well-known sulfur transfer Lawesson reagent, has been widely employed for the preparation of selenoamides.[15] The usual ring opening of the lactones **5a–d** with freshly prepared aryl/hetero-arylmagnesium bromides followed by aqueous NH_4Cl quenching and subsequent interaction of the resulting keto-alcohols **6a–f** with 0.25 equiv of Woollins reagent at

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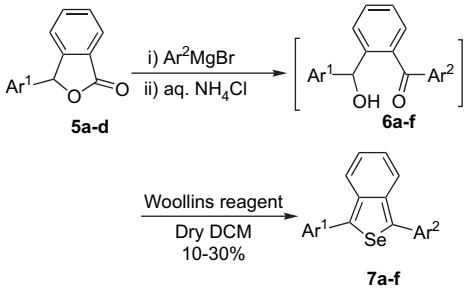
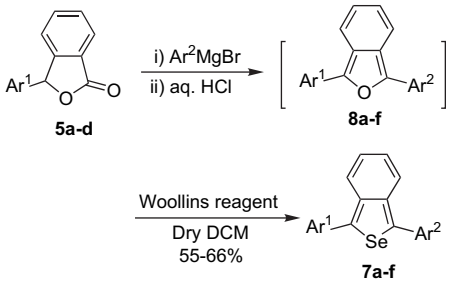
room temperature furnished the expected benzo[*c*]selenophenes **7a–f**. However, the yields of the various 1,3-disubstituted benzo-[*c*]selenophenes **7a–f** prepared using this procedure were only around 10–30%, Scheme 2.



Scheme 2.

The formation of the selenophenes **7a–f** can be visualized through selenation of carbonyl group followed by intramolecular cyclization analogous to that of benzo[*c*]thiophenes.[4a] The recovery of keto-alcohols **6a–f** confirmed that the selenation of keto group was not facile as that of thionation. Hence, it was decided to explore an alternative route to the preparation of benzo[*c*]selenophenes. It



Scheme 3.

should be mentioned that the dithienylbenzo[*c*]furan on treatment with Lawesson reagent led to the formation of the 1,3-dithienybenzo[*c*]thiophene[16] in good yields. To our delight, the interaction of known 1,3-dithienylbenzo[*c*]furan **8a** with 0.25 equiv of Woollins reagent at room temperature in dry DCM for 4 h followed by mass spectral analysis of the crude product indicated the absence of the M^+ ion peak at *m*/*z* 282 corresponding to the dithienylbenzo[*c*]furan. The presence of M^+ ion peak at *m*/*z* 345 confirmed the complete transformation of **8a** into the required dithienylbenzo[*c*]selenophene **7a** (67%).

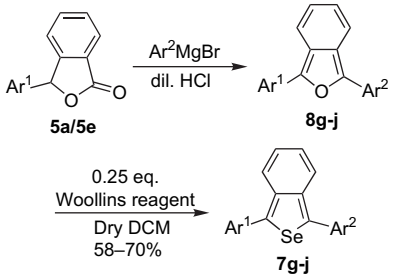
Table 1
Synthesis of 1,3-diarylbenzo[*c*]selenophenes using keto-alcohol/benzo[*c*]furan

Entry	Phthalide	Grignard	Benzo[*c*]selenophene	Yield (%)[a]
1	**5a**	MgBr	**7a**	10,[b] 67[c]
2	**5a**	*n*-C_6H_{13}, MgBr	**7b**	25,[b] 61[c]
3	**5a**	*n*-C_6H_{13}, MgBr	**7c**	22,[b] 66[c]
4	**5b** R^1 = O-*n*-C_6H_{13}	MgBr	**7d**	30,[b] 55[c]
5	**5c**	MgBr	**7e**	20,[b] 62[c]
6	**5d**	*n*-C_6H_{13}, MgBr	**7f**	15,[b] 58[c]

[a] Isolated yield after column chromatography.
[b] Isolated yield from keto-alcohol.
[c] Isolated yield from benzo[*c*]furan.

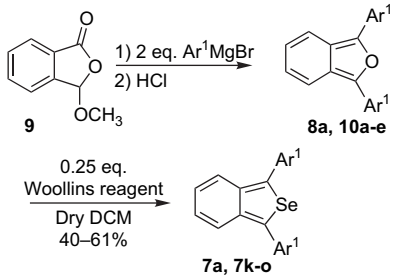
Hence, following this procedure, the unsymmetrical benzo[*c*]furans **8b**–**f** are smoothly converted into the corresponding benzo[*c*]selenophenes **7b**–**f** in 55–66% yields, Scheme 3. Obviously, the interaction of the benzo[*c*]furans **8a**–**f** with Woollins reagent will lead to the formation of hetero Diels–Alder adduct between furan and P=Se. The unstable adduct on rearrangement will furnish the selenophenes **7a**–**f**. The yields of the various benzo[*c*]selenophenes prepared via interaction of keto-alcohol/benzo[*c*]furan with Woollins reagent are listed in Table 1.

Thus, the conversion of symmetrical/unsymmetrical benzo[*c*]furans **8a**–**f** into the corresponding selenium analogs **7a**–**f** could be easily carried out via interaction with Woollins reagent. The ring opening of the lactones **5a**/**5e** with Grignards followed by acidic workup led to the isolation of stable benzo[*c*]furan analogs **8g**–**j** in 51–72% yields. As expected, the benzo[*c*]furans **8g**–**j** on interaction with Woollins reagent afforded respective benzo[*c*]selenophene analogs **7g**–**j** in 58–70% of yields, Scheme 4. The details of the aryl Grignards used and the yield of the benzo[*c*]selenophene analogs **7g**–**j** obtained from the respective benzo[*c*]furans **8g**–**j** are presented in Table 2.



Scheme 4.

The additional diarylbenzo[*c*]furans were prepared adopting the published procedure.[17] The interaction of 3-methoxyphthalide with 2 equiv of aryl/hetero-aryl Grignards followed by acidic workup afforded the required diarylbenzo[*c*]furans **8a**/**10a**–**e** in 45–55% yields. As expected, these symmetrical diarylbenzo[*c*]furans on reaction with 0.25 equiv of Woollins reagent led to the isolation of the corresponding diarylbenzo[*c*]selenophene analogs **7a**/**7k**–**o** in 40–61% yields, Scheme 5.

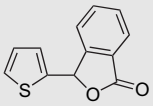
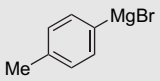
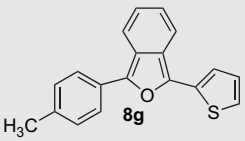
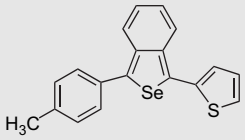
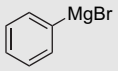
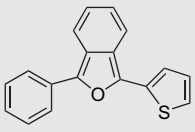
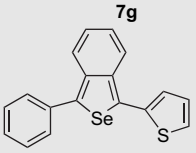
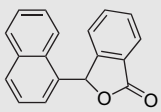
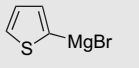
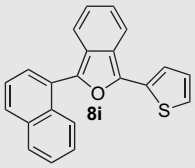
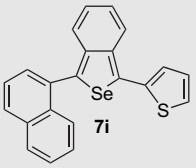
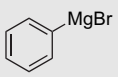
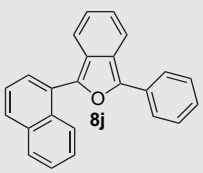
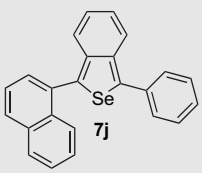


No	Ar^1	Yield (%)
7a	2-Thienyl	61
7k	*P*-Anisyl	52
7l	*P*-Tolyl	51
7m	*o*-Tolyl	46
7n	Phenyl	40
7o	1-Naphthyl	43

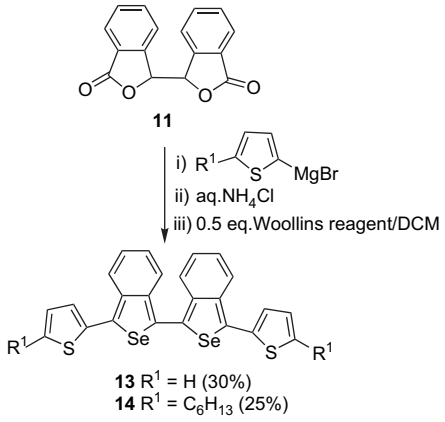
Scheme 5.

Finally, the bis-benz-annelated benzo[*c*]selenophenes **13**/**14** were synthesized via ring opening reaction of commercially available diphthalide **11** with thienylmagnesium bromide followed by neutral workup and subsequent selenation of the resulting keto-alcohol using 0.5 equiv of Woollins reagent in 30 and 25% yields, respectively, Scheme 6. Since, the furan analogs of **13**/**14** were found to be unstable, an alternative route for selenophenes **13**/**14** from the respective benzo[*c*]furan could not be explored.

Table 2
Synthesis of benzo[*c*]selenophene from benzo[*c*]furan

Entry	Phthalide	Grignard	Benzo[*c*]furan	Benzo[*c*]selenophene	Yield (%)[a]
1	 **5a**		 **8g**	 **7g**	66
2	**5a**		 **8h**	 **7h**	62
3	 **5e**		 **8i**	 **7i**	58
4	**5a**		 **8j**	 **7j**	70

[a] Isolated yield after column chromatography.



Scheme 6.

3. Photo physical and electrochemical studies

The UV–vis spectra of the benzo[*c*]selenophenes exhibited strong absorption in the region 410–447 nm corresponding to the π–π* electron transfer of the entire conjugated backbone. The exact λ_{max} values of the benzo[*c*]selenophene are listed in Table 3. On comparison with 1,3-dithienylbenzo[*c*]thiophene (λ_{max}=432 nm),[4d] benzo[*c*]selenophene **7a** exhibited λ_{max} at 447 nm, which confirms the enhancement of conjugation. The introduction of alkyl substituent (*n*-C_6H_{13}) in the α/β positions of **7a** lowers the long-wavelength absorption to 422 nm. In general, the introduction of alkyl/alkoxy groups into the benzo[*c*]selenophene system reduced the π-conjugation, which was reflected through the bathochromic shift of its λ_{max} value. The bis-benzo[*c*]selenophenes **13** and **14** exhibited λ_{max} at 535 and 525 nm, respectively. Thus, the introduction of hexyl substituents at both the ends of benzo[*c*]selenophene **13** has decreased the π-conjugation. Increasing the ratio of benzo[*c*]selenophene/thiophene (**7a**→**13**/**14**) led to the enhancement of conjugation, which was reflected by an increased λ_{max} value.

Qualitative fluorescence spectral data of the benzo[*c*]selenophenes are recorded in DCM solution and the values are presented in Table 3. Compounds **7a**–**k** containing one benzo[*c*]selenophene unit emitted light in the region 485–570 nm. As in the case of absorption, the emission value of benzo[*c*]selenophenes **13**/**14** also red shifted with respect to the increase of π-conjugation.

The HOMO and LUMO energy levels of selected benzo[*c*]selenophenes were calculated from the absorption onset and the onset oxidation potential, and the values are presented in Table 3. All the benzo[*c*]selenophene analogs exhibited E_g value in the range of 2–2.8 eV. Increasing the ratio of benzo[*c*]selenophene to thiophene significantly reduced the E_g value (**7a**→**13**/**14**). Among the benzo[*c*]selenophene analogs, compound **13** exhibited lowest E_g value (~2 eV). The increase of the number of benzo[*c*]selenophene unit significantly increased the LUMO energy level. It should be mentioned that the corresponding HOMO energy levels of **7a** and **13** are almost identical (~5 eV). The higher HOMO energy levels of benzo[*c*]selenophenes (~5.2 eV) greatly reduces energy barrier for hole injection from ITO (φ=4.8 eV) to emissive Alq_3 (φ=5.8 eV). Hence these benzo[*c*]selenophenes can be explored as hole transport material for double-layer OLEDS.

Table 3
Summary of physical measurements for some selected benzo[*c*]selenophenes

Compound	λ_{max}[a] (nm)	λ_{onset} (nm)	λ_{lum}[b] (nm)	E_g[c] (eV)	E_{ox}^{onset} (eV)	HOMO[d] (eV)	LUMO[e] (eV)
7a	447	470	550	2.64	0.60	5.04	2.40
7b	422	465	530	2.66	0.77	5.21	2.55
7c	432	460	538	2.69	0.75	5.19	2.50
7e	421	453	570	2.74	0.75	5.19	2.45
7j	412	437	495	2.84	0.72	5.16	2.32
7k	420	452	517	2.74	0.70	5.14	2.40
7m	410	450	510	2.75	0.78	5.22	2.47
7n	420	455	485	2.72	0.81	5.25	2.53
13	535	610	615	2.03	0.57	5.01	2.98
14	525	595	610	2.08	0.61	5.05	2.97

[a] Measured in dilute dichloromethane solution.
[b] Excited at the absorption maxima.
[c] Estimated from the onset of absorption (E_g=1240/λ_{onset}).
[d] Calculated using empirical equation: HOMO=(4.44+E_{ox}^{onset}).
[e] Calculated from LUMO=HOMO−E_g.

Table 4
Redox behavior of some selected benzo[*c*]selenophene analogs

Compound	$^1E_{pa}$[a] (V)	$^1E_{pc}$[b] (V)	$^1\Delta E_p$[c] (mV)	$^2E_{pa}$[a] (V)	$^2E_{pc}$[b] (V)	$^2\Delta E_p$[c] (mV)
7a	+0.71[e]					
7c	+0.88[e]					
7e	+0.80	+0.47	330[e]			
7i	+0.86					
7g	+0.63	+0.40	230[e]	+1.00	+0.56	440[e]
7k	+0.77[e]			+1.10	+0.67	430[e]
7n	+0.98[e]					
7j	+0.86[e]					
13	+0.86[e]					

[a] $^1E_{pa}$ and $^2E_{pa}$ are the anodic peak potentials of the first and second redox processes, respectively.
[b] $^1E_{pc}$ and $^2E_{pc}$ are the cathodic peak potentials of the first and second redox processes, respectively.
[c] $^1\Delta E_p$ and $^2\Delta E_p$ are the differences between the cathodic and anodic peak potentials of the first and second redox processes, respectively.
[d] Quasireversible.
[e] Irreversible.
[f] Reversible.

The electrochemical properties of benzo[*c*]selenophenes are summarized in Table 4. The benzo[*c*]selenophene **7a** showed a irreversible anodic peak potential at +0.71 V at scan rate of 100 mV S^{-1}. The anodic peak potential for **7a** and the corresponding 1,3-dithienylbenzo[*c*]thiophene (~0.71)[4d] was found to be the same. The introduction of alkyl side chain (*n*-C_6H_{13}) in the α/β positions of **7a** significantly diminished its electro-oxidation behavior. Surprisingly, the increase of the number of benzo[*c*]selenophene unit (**7a**→**13**) does not influence on its anodic peak potential value.

4. Conclusions

The synthesis of several 1,3-diarylbenzo[*c*]selenophenes involving a selenium transfer reaction of keto-alcohol or diarylbenzo[*c*]furans using Woollins reagent was achieved. The bis-annelated benzo[*c*]selenophenes were also prepared using this procedure. The benzo[*c*]selenophenes were obtained in relatively better yields from the respective benzo[*c*]furan rather than the keto-alcohol. Results from the optical and electrochemical studies of benzo[*c*]selenophenes are also presented.

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 JEOL 400 and Bruker-300 spectrometers, respectively. Mass spectra were recorded on a JEOL DX 303 HF spectrometer. Elemental analyses were carried out on Perkin–Elmer (Layolla College, Chennai) and Vario EL III (CDRI, Lucknow) equipments. 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 diarylbenzo[*c*]selenophenes was carried out on a CHI 600C electrochemical analyzer. All the measurements were carried out under oxygen free condition using three-electrode

cell in which glassy carbon electrode was working electrode, saturated Ag/AgCl electrode was reference electrode, and platinum wire was used as an auxiliary electrode. Tetrabutylammoniumhexafluoro phosphate ($TBAPF_6$) was used as supporting electrolyte and its concentration was 10^{-1} M.

5.2. A representative procedure for the preparation of benzo[*c*]selenophene from keto-alcohol (procedure A)

To a solution of phthalide **5a** (2 g, 9.25 mmol) in anhydrous THF (25 mL), 2-thienylmagnesium bromide [prepared from 2-bromothiophene (1.81 g, 11.10 mmol) and magnesium turnings (0.29 g, 12.08 mmol)] was added at 0 °C under N_2. The reaction mixture was slowly raised to room temperature and stirred for 4 h. It was then quenched with aqueous ammonium chloride solution, extracted with DCM (2×20 mL), and dried (Na_2SO_4). The pale yellow solution of keto-alcohol in DCM was stirred with Woollins reagent (1.23 g, 2.31 mmol) for 4 h. After removal of the solvent, the residue was purified by column chromatography (neutral alumina, hexane) to afford the benzo[*c*]selenophene **7a** as an orange solid (0.32 g, 10%).

*5.2.1. 2-(1-(Thiophen-2-yl)benzo[c]selenophen-3-yl)thiophene (**7a**)*

Mp 85 °C. [Found: C, 55.81; H, 3.15; S, 18.81. $C_{16}H_{10}S_2Se$ requires: C, 55.65; H, 2.92; S, 18.57%.] R_f (hexane) 0.82; δ_H (300 MHz, $CDCl_3$) 7.82 (2H, dd, *J* 3.88, 2.96 Hz, ArH), 7.39–7.37 (2H, m, ArH), 7.30–7.28 (2H, m, ArH), 7.17–7.14 (2H, m, ArH), 7.01 (2H, dd, *J* 3.88, 3.2 Hz, ArH); δ_c (75.6 MHz, $CDCl_3$) 137.7, 127.9, 127.8, 126.1, 125.8, 124.4, 122.0, 119.8; *m*/*z* (EI) 345 (M^+, 13).

5.3. A representative procedure for the preparation of benzo[*c*]selenophenes from benzo[*c*]furans (procedure B)

To a solution of phthalide **5a** (1 g, 4.62 mmol) in anhydrous THF (25 mL) was added 2-thienylmagnesium bromide [prepared from 2-bromothiophene (0.90 g, 5.55 mmol) and magnesium turnings (0.14 g, 5.83 mmol)] at 0 °C under N_2. The reaction mixture was slowly raised to room temperature and stirred for 4 h. It was then quenched with crushed ice (5 g) containing HCl (10 mL), extracted with DCM (2×20 mL), and dried (Na_2SO_4). The greenish-yellow fluorescent solution of benzo[*c*]furan was then stirred with Woollins reagent (0.62 g, 1.15 mmol) at room temperature for 4 h. After removal of the solvent, the residue was purified by column chromatography (neutral alumina, hexane) to afford the benzo[*c*]selenophene **7a** as an orange solid (1.07 g, 67%).

*5.3.1. 2-(1-(Thiophen-2-yl)benzo[c]selenophen-3-yl)thiophene (**7a**)*

Mp 85 °C. [Found: C, 55.81; H, 3.15; S, 18.81. $C_{16}H_{10}S_2Se$ requires: C, 55.65; H, 2.92; S, 18.57%.] R_f (hexane) 0.82; ν_{max} (KBr) 1600, 1510, 785, 465 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 7.82 (2H, dd, *J* 3.88, 2.96 Hz, ArH), 7.39–7.37 (2H, m, ArH), 7.30–7.28 (2H, m, ArH), 7.17–7.14 (2H, m, ArH), 7.01 (2H, dd, *J* 3.88, 3.2 Hz, ArH); δ_c (75.6 MHz, $CDCl_3$) 137.7, 127.9, 127.8, 126.1, 125.8, 124.4, 122.0, 119.8; *m*/*z* (EI) 345 (M^+, 13).

*5.3.2. 3-Hexyl-2-(1-(thiophen-2-yl)benzo[c]selenophen-3-yl)-thiophene (**7b**)*

Following the above-mentioned procedure B, compound **7b** was obtained using the phthalide **5a** (1 g, 4.63 mmol), 3-hexyl-2-thienylmagnesium bromide [prepared from 2-bromo-3-hexylthiophene (1.37 g, 5.55 mmol) and Mg (0.14 g, 5.83 mmol)] and Woollins reagent (0.61 g, 1.15 mmol) as a thick orange liquid (1.20 g, 61%). [Found: C, 61.22; H, 5.36; S, 15.22. $C_{22}H_{22}S_2Se$ requires: C, 61.52; H, 5.16; S, 14.93%.] R_f (hexane) 0.84; ν_{max} (KBr) 3010, 1596, 1491, 1200, 908, 764, 740, 689 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.76 (1H, d, *J* 8.8 Hz, ArH), 7.30–7.26 (4H, m, ArH), 7.22 (1H, dd, *J* 2.4, 1.2 Hz, ArH), 7.05 (1H, dd, *J* 3.6, 1.4 Hz, ArH), 6.97 (1H, d, *J* 5.3 Hz, ArH), 6.90 (1H, d, *J* 7.8 Hz, ArH), 2.53 (2H, t, *J* 7.8 Hz, CH_2), 1.31–1.60 (8H, m, CH_2), 0.74 (3H, t, *J* 6.8 Hz, CH_3); δ_c (75.6 MHz, $CDCl_3$) 143.9, 132.1, 131.8, 131.4, 130.4, 130.2, 129.1, 127.3, 126.8, 126.5, 125.3, 124.9, 124.0, 122.7, 122.3, 120.3, 31.9, 31.8, 30.1, 29.0, 22.8, 14.3; *m*/*z* (EI) 430 (M^{+1}, 100).

*5.3.3. 2-Hexyl-5-(1-(thiophen-2-yl)benzo[c]selenophen-3-yl)-thiophene (**7c**)*

Following the above-mentioned procedure B, compound **7c** was obtained using the phthalide **5a** (1 g, 4.63 mmol), 5-hexyl-2-thienylmagnesium bromide [prepared from 2-bromo-5-hexylthiophene (1.37 g, 5.54 mmol) and Mg (0.14 g, 5.83 mmol)] and Woollins reagent (0.61 g, 1.15 mmol) as a thick orange liquid (1.30 g, 66%). [Found: C, 61.32; H, 5.32; S, 15.15. $C_{22}H_{22}S_2Se$ requires: C, 61.52; H, 5.16; S, 14.93%.] R_f (hexane) 0.86; δ_H (400 MHz, $CDCl_3$) 7.82–7.83 (2H, m, ArH), 7.27 (1H, d, *J* 1.2 Hz, ArH), 7.26 (1H, d, *J* 1.2 Hz, ArH), 7.10–7.13 (2H, m, ArH), 6.97–7.08 (2H, m, ArH), 6.78 (1H, d, *J* 3.6 Hz, ArH), 2.81 (2H, t, *J* 7.5 Hz, CH_2), 1.72 (2H, quintet, CH_2), 1.25–1.34 (6H, m, CH_2), 0.90 (3H, t, *J* 3.3 Hz, CH_3); δ_c (100 MHz, $CDCl_3$) 147.0, 137.9, 137.8, 135.1, 129.0, 127.8, 127.5, 127.1, 125.9, 125.8, 125.6, 124.9, 124.4, 124.1, 122.3, 122.0; *m*/*z* (EI) 429 (M^+, 05).

*5.3.4. 2-(1-(4-(Hexyloxy)phenyl)benzo[c]selenophen-3-yl)-thiophene (**7d**)*

Following the above-mentioned procedure B, compound **7d** was obtained using the phthalide **5b** (1 g, 3.22 mmol), 2-thienylmagnesium bromide [prepared from 2-bromothiophene (0.63 g, 3.86 mmol) and Mg (0.10 g, 4.16 mmol)] and Woollins reagent (0.43 g, 0.81 mmol) as yellow solid (0.78 g, 55%). Mp 63 °C. [Found: C, 65.81; H, 5.30; S, 7.41. $C_{24}H_{24}OSSe$ requires: C, 65.59; H, 5.50; S, 7.30%.] R_f (hexane) 0.80; δ_H (400 MHz, $CDCl_3$) 7.83 (1H, d, *J* 8.8 Hz, ArH), 7.59 (1H, d, *J* 9.3 Hz, ArH), 7.51 (2H, d, *J* 8.8 Hz, ArH), 7.35 (1H, dd, *J* 4.1, 0.9 Hz, ArH), 7.26 (1H, dd, *J* 2.9, 0.9 Hz, ArH), 7.12 (1H, dd, *J* 3.7, 1.4 Hz, ArH), 6.95–7.01 (4H, m, ArH), 4.01 (2H, t, *J* 6.6 Hz, OCH_2), 1.81 (2H, quintet, *J* 7.1 Hz, CH_2), 1.45–1.55 (4H, m, CH_2), 1.34–1.38 (2H, m, CH_2), 0.90 (3H, t, *J* 7.1 Hz, CH_3); δ_c (100 MHz, $CDCl_3$) 159.7, 141.4, 138.2, 137.1, 136.5, 135.2, 132.5, 130.0, 128.6, 125.2, 124.5, 122.7, 124.4, 121.9, 121.2, 115.2, 68.3, 31.6, 29.2, 25.8, 22.62, 14.11; *m*/*z* (EI) 439 (M^+, 54).

*5.3.5. 2-(1-(Anthracen-10-yl)benzo[c]selenophen-3-yl)thiophene (**7e**)*

Following the above-mentioned procedure B, compound **7e** was obtained using the phthalide **5c** (1 g, 3.22 mmol), 2-thienylmagnesium bromide [prepared from 2-bromothiophene (0.63 g, 3.86 mmol) and Mg (0.10 g, 4.16 mmol)] and Woollins reagent (0.43 g, 0.81 mmol) as orange solid (0.87 g, 62%). Mp 162 °C. [Found: C, 71.00; H, 3.50; S, 7.40. $C_{26}H_{16}SSe$ requires: C, 71.06; H, 3.67; S, 7.30%.] R_f (hexane) 0.82; δ_H (400 MHz, $CDCl_3$) 8.62 (1H, s, ArH), 8.09 (2H, d, *J* 8.1 Hz, ArH), 7.88 (2H, t, *J* 9.1 Hz, ArH), 7.42–7.55 (6H, m), 7.35 (1H, d, *J* 4.5 Hz), 7.15–7.17 (1H, m, ArH), 7.08–7.10 (2H, m, ArH), 6.91–6.93 (1H, m, ArH); δ_c (100 MHz, $CDCl_3$) 147.0, 137.9, 137.8, 135.1, 129.0, 127.8, 127.5, 127.1, 125.9, 125.8, 125.6, 124.9, 124.4, 124.1, 122.3, 122.0, 119.5; *m*/*z* (EI) 440 (M^+, 35).

*5.3.6. 2-Hexyl-5-(1-(5-(thiophen-2-yl)thiophen-2-yl)benzo[c]-selenophen-3-yl)thiophene (**7f**)*

Following the above-mentioned procedure B, compound **7f** was obtained using the phthalide **5d** (1 g, 3.35 mmol), 5-hexyl-2-thienylmagnesium bromide [prepared from 2-bromo-5-hexylthiophene (0.99 g, 4.01 mmol) and Mg (0.10 g, 4.16 mmol)] and Woollins reagent (0.44 g, 0.83 mmol) as a thick orange liquid (0.99 g, 58%). [Found: C, 60.92; H, 4.61; S, 18.92. $C_{26}H_{24}S_3Se$ requires: C, 61.04; H, 4.73; S, 18.80%.] R_f (hexane) 0.78; ν_{max} (KBr) 1596, 1509, 1263, 954, 787, 753, 635 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.81–7.90 (1H, m, ArH), 7.70–7.76 (1H, m, ArH), 7.61–7.67 (1H, m,

ArH), 7.00–7.08 (2H, m, ArH), 7.11–7.12 (1H, d, *J* 6.6 Hz, ArH), 7.27–7.33 (5H, m, ArH), 2.80 (2H, t, *J* 7.6 Hz, CH_2), 1.23–1.54 (8H, m, CH_2), 0.83 (3H, t, *J* 6.6 Hz, CH_3); δ_c (75.6 MHz, $CDCl_3$) 145.0, 132.2, 131.8, 130.5, 130.3, 130.1, 129.2, 128.8, 127.8, 127.4, 127.2, 127.1, 126.9, 126.6, 125.4, 125.0, 124.2, 123.0, 122.8, 122.5, 32.1, 31.9, 30.2, 29.1, 23.0, 14.5; *m*/*z* (EI) 511 (M^+, 21).

5.4. Preparation of unsymmetrical benzo[*c*]furans (procedure C)

*5.4.1. 1-(Thiophen-2-yl)-3-p-tolylisobenzofuran (**8g**)*

To a solution of phthalide **5a** (2 g, 9.26 mmol) in anhydrous THF (25 mL), *p*-tolylmagnesium bromide [prepared from *p*-bromotoluene (1.90 g, 11.11 mmol) and magnesium turnings (0.29 g, 12.08 mmol)] was added at 0 °C under N_2. The reaction mixture was slowly raised to room temperature and stirred overnight. The reaction mixture was hydrolyzed by adding dropwise a solution of concd HCl (20 mL) and H_2O (20 mL) at 0 °C. The reaction mixture turned to bright orange with green fluorescence. Stirring was maintained under nitrogen atmosphere for 0.5 h. Then, Et_2O (50 mL) was added and the organic phase separated. The organic phase was washed with brine (2×50 mL), satd $NaHCO_3$ (50 mL), and dried (Na_2SO_4). After removal of the solvent, the residue was purified by column chromatography (silica gel, hexane) to afford the benzo[*c*]furan **8g** as yellow solid (1.92 g, 72%). Mp 98 °C. [Found: C, 78.47; H, 4.92; S, 11.00. $C_{19}H_{14}OS$ requires: C, 78.59; H, 4.86; S, 11.04%.] R_f (hexane) 0.96; ν_{max} (KBr) 1605, 1500, 755, 685 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 7.78 (2H, d, *J* 8.2 Hz, ArH), 7.69–7.76 (2H, m, ArH), 7.47 (1H, d, *J* 3.58 Hz, ArH), 7.17–7.27 (3H, m, ArH), 7.11–7.13 (1H, m, ArH), 6.95–6.99 (2H, m, ArH), 2.39 (3H, s, CH_3); δ_c (75 MHz, $CDCl_3$) 137.0, 133.9, 130.2, 129.8, 129.2, 128.9, 127.9, 125.3, 125.2, 124.8, 123.9, 122.1, 121.7, 121.5, 120.3, 120.1, 21.5; *m*/*z* (EI) 290 (M^+, 100).

*5.4.2. 1-Phenyl-3-(thiophene-2-yl)isobenzofuran (**8h**)*

Following the above-mentioned procedure C, **8h** was obtained using the phthalide **5a** (2 g, 9.25 mmol) and phenylmagnesium bromide [prepared from bromo benzene (1.74 g, 11.08 mmol) and Mg (0.29 g, 12.08 mmol)] as a thick yellow liquid (1.30 g, 51%). [Found: C, 78.13; H, 4.17; S, 11.72. $C_{18}H_{12}OS$ requires: C, 78.23; H, 4.38; S, 11.60%.] R_f (hexane) 0.92; ν_{max} (KBr) 1600, 1505, 741, 691 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 7.62–7.64 (3H, m, ArH), 7.55–7.58 (3H, m, ArH), 7.40–7.47 (3H, m, ArH), 7.28–7.30 (2H, m, ArH), 6.98 (1H, t, *J* 4.2 Hz, ArH); δ_c (75.6 MHz, $CDCl_3$) 144.1, 139.8, 139.6, 137.2, 134.9, 134.8, 133.2, 132.4, 130.8, 130.4, 129.8, 129.7, 129.0, 128.8, 128.3; *m*/*z* (EI) 276 (M^+, 100).

*5.4.3. 1-(Napthalen-8-yl)-3-(thiophen-2-yl)isobenzofuran (**8i**)*

Following the above-mentioned procedure C, **8i** was obtained using the phthalide **5e** (2 g, 7.69 mmol) and 2-thienyl magnesium bromide [prepared from 2-bromothiophene (1.50 g, 9.20 mmol) and Mg (0.24 g, 10.00 mmol)] as a yellow solid (1.55 g, 62%). Mp 87 °C. [Found: C, 80.87; H, 4.23; S, 9.92. $C_{22}H_{14}OS$ requires: C, 80.95; H, 4.32; S, 9.82%.] R_f (hexane) 0.90; δ_H (300 MHz, $CDCl_3$) 8.41 (1H, m, ArH), 7.80–7.96 (4H, m, ArH), 7.55–7.62 (5H, m, ArH), 7.32 (1H, d, *J* 4.89 Hz, ArH), 7.15–7.18 (1H, m, ArH), 6.97–7.09 (2H, m, ArH); δ_c (75.6 MHz, $CDCl_3$) 134.2, 133.9, 130.8, 128.7, 128.5, 128.1, 127.9, 127.3, 126.6, 126.2, 126.1, 125.4, 125.3, 125.1, 123.9, 123.7, 122.1, 121.0, 120.8, 119.7; *m*/*z* (EI) 326 (M^+, 100).

*5.4.4. 1-(Naphthalen-1-yl)-3-phenylisobenzofuran (**8j**)*

Following the above-mentioned procedure C, **8j** was obtained using phthalide **5e** (2 g, 7.69 mmol) and phenylmagnesium bromide [prepared from bromo benzene (1.45 g, 9.23 mmol) and Mg (0.24 g, 10.00 mmol)] as a yellow solid (1.50 g, 61%). [Found: C, 89.87; H, 4.96. $C_{24}H_{16}O$ requires: C, 89.97; H, 5.03; S, 9.82%.] R_f (hexane) 0.92; ν_{max} (KBr) 1595, 1495, 735, 451 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 8.22–8.21 (1H, m, ArH), 7. 81 (1H, d, *J* 7.1 Hz, ArH), 7.73–7.70 (1H, m, ArH), 7.59–7.52 (4H, m, ArH), 7.48–7.44 (3H, m, ArH), 7.40–7.36 (3H, m, ArH), 7.34–7.32 (3H, m, ArH); δ_c (75.6 MHz, $CDCl_3$) 141.0, 140.5, 137.3, 135.2, 133.6, 133.1, 132.8, 131.4, 130.9, 130.8, 130.4, 130.0, 129.5, 129.0, 128.3, 128.1, 127.7, 126.4, 125.7, 123.9; *m*/*z* (EI) 320 (M^+, 70).

5.5. A representative procedure for the preparation of unsymmetrical benzo[*c*]selenophene from corresponding benzo[*c*]furan (procedure D)

Compound **8g** (0.2 g, 0.69 mmol) in dry DCM (10 mL) along with Woollins reagent (0.09 g, 0.17 mmol) was stirred for 4 h at room temperature. After removal of the solvent, the residue was purified by column chromatography (silica gel, hexane) to afford the benzo[*c*]selenophene **7g** as a thick yellow liquid (0.16 g, 66%).

*5.5.1. 2-(1-p-Tolylbenzo[c]selenophen-3-yl)thiophene (**7g**)*

Found: C, 64.30; H, 4.19; S, 9.20. $C_{19}H_{14}SSe$ requires: C, 64.58; H, 3.99; S, 9.07%. R_f (hexane) 0.86; δ_H (400 MHz, $CDCl_3$) 7.85 (1H, d, *J* 8.8 Hz, ArH), 7.63 (1H, d, *J* 8.8 Hz, ArH), 7.51 (2H, d, *J* 8.3 Hz, ArH), 7.36 (1H, d, *J* 4.8 Hz, ArH), 7.02–7.14 (3H, m, ArH), 6.94–7.00 (3H, m, ArH), 2.42 (3H, s, CH_3); δ_c (100 MHz, $CDCl_3$) 146.5, 141.4, 138.0, 137.0, 136.4, 135.6, 130.4, 130.0, 129.2, 124.8, 124.6, 121.9, 121.1, 21.29; *m*/*z* (EI) 354 (M^+, 21).

*5.5.2. 2-(1-Phenylbenzo[c]selenophen-3-yl)thiophene (**7h**)*

Following the above-mentioned procedure D, **7h** was obtained using benzo[*c*]furan **8h** (0.2 g, 0.72 mmol) and Woollins reagent (0.10 g, 0.18 mmol) as thick yellow liquid (0.15 g, 62%). [Found: C, 63.60; H, 3.50; S, 9.51. $C_{18}H_{12}SSe$ requires: C, 63.71; H, 3.56; S, 9.45%.] R_f (hexane) 0.82; ν_{max} (KBr) 1597, 1500, 1227, 735, 681 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.84 (1H, d, *J* 8.8 Hz, ArH), 7.61–7.71 (2H, m, ArH), 7.45 (2H, t, *J* 7.32 Hz, ArH), 7.35–7.38 (2H, m, ArH), 7.29–7.30 (1H, m, ArH), 7.13–7.15 (1H, m, ArH), 6.94–7.03 (3H, m, ArH); δ_c (100 MHz, $CDCl_3$) 143.7, 137.2, 132.9, 131.6, 130.3, 129.8, 129.6, 128.8, 128.3, 126.8, 125.1, 124.7, 122.1, 120.1; *m*/*z* (EI) 340 (M^{+1}, 100).

*5.5.3. 2-(1-(Naphthalen-1-yl)benzo[c]selenophen-3-yl)thiophene (**7i**)*

Following the above-mentioned procedure D, **7i** was obtained using benzo[*c*]furan **8i** (0.2 g, 0.61 mmol) and Woollins reagent (0.08 g, 0.15 mmol) as a yellow solid (0.14 g, 58%). Mp 92 °C. [Found: C, 67.70; H, 3.52; S, 8.33. $C_{22}H_{14}SSe$ requires: C, 67.86; H, 3.62; S, 8.24%.] R_f (hexane) 0.82; δ_H (400 MHz, $CDCl_3$) 7.93 (2H, d, *J* 8.2 Hz, ArH), 7.88 (2H, m, ArH), 7.63 (1H, d, *J* 6.8 Hz, ArH), 7.56 (1H, d, *J* 7.8 Hz, ArH), 7.50–7.54 (1H, m, ArH), 7.41–7.45 (2H, m, ArH), 7.33–7.39 (1H, m, ArH), 7.14–7.17 (1H, m, ArH), 7.02–7.04 (2H, m, ArH), 6.84–6.99 (1H, m, ArH); δ_c (100 MHz, $CDCl_3$) 143.8, 141.0, 134.2, 133.8, 130.8, 128.6, 128.4, 128.1, 127.8, 127.2, 126.6, 126.1, 126.1, 125.78, 125.3, 125.2, 125.0, 123.9, 122.1, 120.2, 119.6; *m*/*z* (EI) 390 (M^+).

*5.5.4. 1-(Naphthalene-1-yl)-3-phenylbenzo[c]selenophene (**7j**)*

Following the above-mentioned procedure D, **7j** was obtained using benzo[*c*]furan **8j** (0.2 g, 0.62 mmol) and Woollins reagent (0.08 g, 0.15 mmol) as a thick yellow liquid (0.17 g, 70%). [Found: C, 74.85; H, 4.35. $C_{24}H_{16}Se$ requires: C, 75.20; H, 4.21%.] R_f (hexane) 0.80; δ_H (400 MHz, $CDCl_3$) 8.57 (1H, dd, *J* 3.42, 2.92 Hz, ArH), 8.17 (2H, d, *J* 7.32 Hz, ArH), 8.08 (2H, d, *J* 8.08 Hz, ArH), 7.98 (1H, d, *J* 7.36 Hz, ArH), 7.61–7.90 (5H, m, ArH), 7.41–7.58 (2H, m, ArH), 7.11–7.21 (3H, m, ArH); δ_c (100 MHz, $CDCl_3$) 144.6, 144.4, 134.2, 131.8, 130.9, 129.6, 129.4, 128.9, 128.4, 128.3, 127.3, 126.81, 126.6, 126.1,

125.4, 125.3, 124.6, 124.6, 123.9, 121.5, 120.3, 119.8; *m*/*z* (EI) 384 (M^+, 20).

5.6. A representative procedure for the preparation of symmetrical benzo[*c*]furans 10a–e

Symmetrical benzo[*c*]furans **8a**/**10a**–**e** were prepared using the published procedure.[17]

5.6.1. 1,3-Bis(4-methoxyphenyl)benzo[c]selenophene (**7k**)

Following the above-mentioned procedure D, compound **7k** was obtained using the benzo[*c*]furan **10a** (0.2 g, 0..61 mmol) and Woollins reagent (0.08 g, 0.15 mmol) as a thick yellow liquid (0.12 g, 50%). [Found: C, 67.25; H, 4.57. $C_{22}H_{18}O_2Se$ requires: C, 67.18; H, 4.61%.] R_f (hexane) 0.65; δ_H (400 MHz, $CDCl_3$) 7.76 (4H, d, *J* 9.0 Hz, ArH), 7.65 (2H, dd, *J* 2.7, 4.2 Hz, ArH), 6.91–6.95 (6H, m, ArH), 3.79 (6H, s, OCH_3); δ_c (100 MHz, $CDCl_3$) 137.3, 135.0, 134.0, 131.5, 129.2, 123.9, 121.4, 55.6; *m*/*z* (EI) 394 (M^+, 33).

5.6.2. 1,3-Di-p-tolylbenzo[c]selenophene (**7l**)

Following the above-mentioned procedure D, **7l** was obtained using the benzo[*c*]furan **10b** (0.2 g, 0.67 mmol) and Woollins reagent (0.09 g, 0.17 mmol) as a thick yellow liquid (0.12 g, 51%). [Found: C, 73.03; H, 5.10. $C_{22}H_{18}Se$ requires: C, 73.13; H, 5.02%.] R_f (hexane) 0.82; δ_H (400 MHz, $CDCl_3$) 7.55–7.57 (2H, m, ArH), 7.40 (4H, d, *J* 7.6 Hz, ArH), 7.14 (4H, d, *J* 7.6 Hz, ArH), 6.86–6.88 (2H, m, ArH), 2.30 (6H, s, CH_3); δ_c (100 MHz, $CDCl_3$) 137.2, 137.2, 136.6, 130.4, 129.1, 126.7, 123.5, 121.7, 21.2; *m*/*z* (EI) 362 (M^+, 54).

5.6.3. 1,3-Di-o-tolylbenzo[c]selenophene (**7m**)

Following the above-mentioned procedure D, **7m** was obtained using the benzo[*c*]furan **10c** (0.2 g, 0.67 mmol) and Woollins reagent (0.09 g, 0.17 mmol) as thick yellow liquid (0.11 g, 46%). [Found: C, 73.01; H, 5.10. $C_{22}H_{18}Se$ requires: C, 73.13; H, 5.02%.] R_f (hexane) 0.86; ν_{max} (KBr) 3060, 1595, 1500, 1230, 743, 695 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.78–7.68 (4H, m, ArH), 7.47–7.40 (6H, m, ArH), 7.11–7.09 (2H, m, ArH), 2.71 (6H, s, CH_3); δ_c (75.6 MHz, $CDCl_3$) 145.3, 136.5, 131.3, 130.6, 129.4, 128.0, 125.9, 124.5, 122.4, 120.0, 21.3; *m*/*z* (EI) 362 (M^+, 14).

5.6.4. 1,3-Diphenylbenzo[c]selenophene (**7n**)

Following the above-mentioned procedure D, **7n** was obtained using the benzo[*c*]furan **10d** (0.2 g, 0.74 mmol) and Woollins reagent (0.10 g, 0.19 mmol) as thick yellow liquid 0.096 g, (40%). [Found: C, 72.00; H, 4.18. $C_{20}H_{14}Se$ requires: C, 72.07; H, 4.23%.] R_f (hexane) 0.80; δ_H (400 MHz, $CDCl_3$) 7.97 (4H, d, *J* 7.8 Hz, ArH), 7.82 (2H, dd, *J* 3.92, 2.96 Hz, ArH), 7.48 (4H, t, *J* 7.8 Hz, ArH), 7.29 (2H, t, *J* 6.52 Hz, ArH), 7.02 (2H, dd, *J* 3.92, 2.98 Hz, ArH); δ_c (100 MHz, $CDCl_3$) 143.6, 131.6, 128.5, 127.0, 126.8, 124.7, 122.03, 120.0; *m*/*z* (EI) 334 (M^+, 31).

5.6.5. 1,3-Di(naphthalene-1-yl)selenophene (**7o**)

Following the above-mentioned procedure D, compound **7o** was obtained using the benzo[*c*]furan **10e** (0.2 g, 0.54 mmol) and Woollins reagent (0.07 g, 0.13 mmol) as thick yellow liquid (0.10 g, 43%). [Found: C, 77.70; H, 4.11. $C_{28}H_{18}Se$ requires: C, 77.60; H, 4.19%.] R_f (hexane) 0.72; ν_{max} (KBr) 1596, 1479, 762, 722 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.72–7.68 (6H, m, ArH), 7.48–7.33 (8H, m, ArH), 7.20–7.18 (2H, m, ArH), 6.86–6.84 (2H, m, ArH); δ_c (100 MHz, $CDCl_3$) 143.1, 133.1, 129.8, 128.7, 128.3, 127.3, 126.4, 126.3, 125.3, 124.8, 123.6, 121.6, 120.1; *m*/*z* (EI) 434 (M^+, 11).

5.6.6. 2-(1-(1-(Thiophen-2-yl)benzo[c]selenophen-3-yl)benzo[c]-selenophen-3-yl)thiophene (**13**)

Following the above-mentioned procedure A, **13** was obtained using diphthalide **11** (0.5 g, 1.87 mmol), 2-thienylmagnesium bromide [prepared from 2-bromothiophene (0.67 g, 4.11 mmol) and Mg (0.11 g, 4.58 mmol)] and Woollins reagent (0.50 g, 0.94 mmol) as red solid (0.29 g, 30%). [Found: C, 54.81; H, 2.75; S, 12.35. $C_{24}H_{14}S_2Se_2$ requires: C, 54.97; H, 2.69; S, 12.23%.] R_f (hexane) 0.72; ν_{max} (KBr) 1598, 1502, 1227, 753, 695 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 8.03 (2H, d, *J* 8.4 Hz, ArH), 7.87 (2H, d, *J* 8.1 Hz, ArH), 7.35–7.42 (4H, m, ArH), 7.16–7.23 (6H, m, ArH); δ_c (75.6 MHz, $CDCl_3$) 136.9, 135.5, 135.1, 128.5, 127.9, 125.7, 125.0, 124.8, 124.2, 121.8, 121.4; *m*/*z* (EI) 525 (M^+, 11).

5.6.7. 2-Hexyl-5-(1-(1-(5-hexylthiophen-2-yl)benzo[c]selenophen-3-yl)benzo[c]selenophen-3-yl)thiophene (**14**)

Following the above-mentioned procedure A, **14** was obtained using diphthalide **11** (0.5 g, 1.87 mmol), 5-hexyl-2-thienylmagnesium bromide [prepared from 2-bromo-5-hexylthiophene (1.02 g, 4.12 mmol) and Mg (0.11 g, 4.58 mmol)] and Woollins reagent (0.50 g, 0.94 mmol) as a thick orange liquid (0.32 g, 25%). [Found: C, 62.50; H, 5.43; S, 9.30. $C_{36}H_{38}S_2Se_2$requires: C, 62.42; H, 5.53; S, 9.26%.] R_f (hexane) 0.75; δ_H (300 MHz, $CDCl_3$) 8.01–8.07 (2H, m, ArH), 7.86 (2H, d, *J* 9.0 Hz, ArH), 7.15–7.20 (6H, m, ArH), 6.85 (2H, d, *J* 3.3 Hz, ArH), 1.77 (4H, t, *J* 7.35 Hz, CH_2), 1.33–1.47 (12H, m, CH_2), 0.90–0.95 (10H, m, CH_2CH_3); δ_c (75.6 MHz, $CDCl_3$) 146.76, 136.83, 134.74, 129.11, 125.33, 124.92, 124.66, 123.69, 121.88, 121.62, 31.58, 30.27, 28.83, 22.58, 14.08; *m*/*z* (EI) 694 (M^+, 15).

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