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ORIGINAL PAPER

Electron-rich ligands with multiple tetrathiafulvalene arms

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Abstract We have synthesized a series of oligodentate ligands, the structure of which consists of a central aromatic core and three or four tetrathiafulvalene-based arms. Their syntheses exploited a simple general protocol based on the reaction of a bromomethylated core with a thiolate generated in situ from an appropriate tetrathiafulvalene precursor. According to molecular modeling, proper positioning of the fulvalene arms over the central platform gives rise to an electron-rich cavity suitable for inclusion of spatial electron-deficient guests such as fullerenes.

Keywords Donor–acceptor effects · Supramolecular chemistry · Ligands · Tetrathiafulvalenes

Introduction

Tetrathiafulvalene (TTF) derivatives, well-known electron-rich redox-active components, have been widely used in numerous scientific areas, especially macrocyclic, supramolecular, and materials chemistry [1–4]. In order to study the interactions between individual TTF units and the properties of mixed-valence radical cation salts, several macrocyclic [5–7] and branched [8–16] TTF oligomers have been prepared during the past 10 years. Some of these molecules are also interesting as host molecules for inorganic cations or organic electron-deficient guests [17–21]. A photo-induced electron-transfer in TTF containing donor–acceptor dyads was also studied [22].

In recent decades, fullerenes have received much attention because of their unique properties. Many ligands, both cyclic and non-cyclic, were synthesized and the formation of their inclusion complexes with fullerenes was studied [23], but until recently, only a few of them featured dithiolthione or TTF units [24–30]. Lately, TTF-based molecular tweezers [31–33] or calixarene–porphyrin conjugates with C_{60}/C_{70} selectivity appeared [34–36]. Very recently, TTF–calixpyrroles and tripodal extended TTF–cyclotriveratrylenes were presented as efficient fullerene receptors [37, 38]. Herein we report on the synthesis of tri- and tetrapodal ligands containing TTF arms as prospective fullerene hosts.

Results and discussion

Syntheses of all new tri- and tetrapodal TTF ligands exploited a simple general protocol. Easily available hexasubstituted benzene derivatives bearing three bromomethyl groups were selected as the central units for the tripodal ligands. 1,3,5-Tris(bromomethyl)-2,4,6-trimethylbenzene (**1**) was prepared from mesitylene according to the procedure developed earlier by our group [39]. 1,3,5-Tris(bromomethyl)-2,4,6-triethylbenzene (**2**) was a commercial product and 1,3,5-tris(bromomethyl)-2,4,6-trimethoxybenzene (**3**) was obtained from 1,3,5-trimethoxybenzene by a slightly modified literature procedure [40]. The synthesis of the core of the tetrapodal ligand started from 4,4′-dihydroxybiphenyl, which was converted first into 4,4′-dibutoxybiphenyl (**4**) [41]. Then, bromomethylation of **4** under harsh conditions

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[39] smoothly yielded the target 3,3′,5,5′-tetrakis(bromomethyl)-4,4′-dibutoxybiphenyl (**5**).

3,3′-[[2-[4,5-Bis(butylthio)-1,3-dithiol-2-ylidene]-1,3-dithiole-4,5-diyl]bis(thio)]bis(propanenitrile) (**6**) [42] was chosen as the key intermediate for the attachment of TTF units. It was prepared in several steps from a stable salt, namely bis(tetraethylammonium) bis(thioxo-1,3-dithiole-4,5-dithiol) zincate [43], according to the original synthetic procedure [44]. One of the two cyanoethyl protecting groups in **6** was selectively removed [45] by a stoichiometric amount of $CsOH{\cdot}H_2O$ and the resulting mono-thiolate was alkylated with MeI affording 3-[[2-[4,5-bis(butylthio)-1,3-dithiol-2-ylidene]-5-(methylthio)-1,3-dithiol-4-yl]thio]propanenitrile (**7**) in 98% yield. Syntheses of the new structural blocks are shown in Scheme 1.

The remaining cyanoethyl moiety of TTF derivative **7** was then cleaved by the action of additional $CsOH{\cdot}H_2O$ and the in situ generated thiolate readily reacted with the bromomethyl groups located on the central core. Using the bromomethyl derivatives **1**, **2**, **3**, and **5**, we obtained the target ligands **8**, **9**, **10**, and **11**, respectively, in good yields (Scheme 2). Their structures were unambiguously confirmed by NMR and mass spectrometry. Notably, the reaction of 1,4,5,8-tetrakis(bromomethyl)naphthalene [46, 47] with TTF derivative **7** performed analogously failed totally.

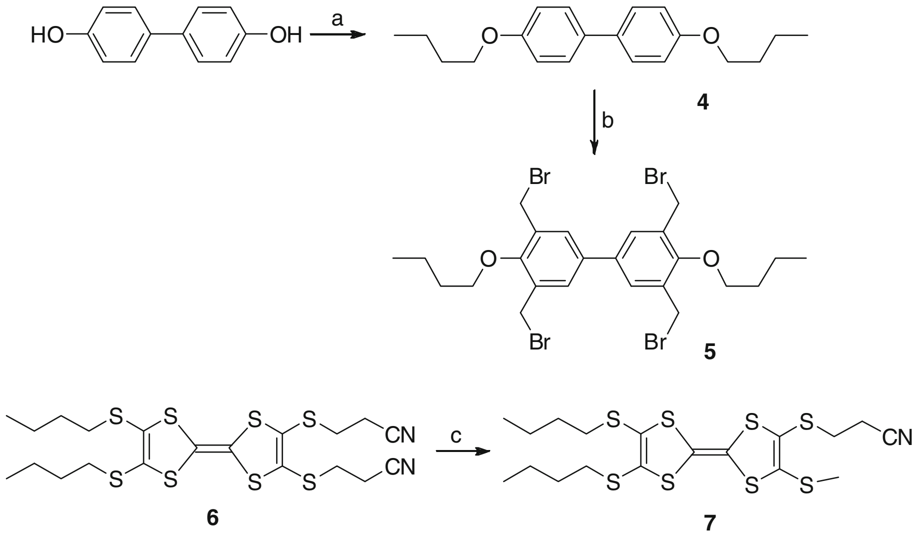
The study of interactions of the prepared ligands with electron-deficient guests is now in progress. Preliminary experiments proved remarkable changes in ^1H NMR spectra of the ligands after addition of fullerene C_{60} in excess. The spectral changes observed for the ligand **8** are shown in Fig. 1. Particularly noteworthy are the transformations of signals corresponding to $S{-}CH_2$ groups in the vicinity of TTF units (at 4.08 and 2.76 ppm for the free ligand **8**).

All new ligands were characterized by cyclic voltammetry. For a more precise evaluation of reversible formal redox potentials all voltammograms were baseline corrected, numerically transformed by semi-integration to neopolarograms, and analyzed by a standard method of log-plot analysis [48]. This procedure allowed the identification of two overlapped electron-transfer steps corresponding to the first oxidation of TTF units. The formal redox potentials $E_{1,1}$ and $E_{1,2}$ of the two overlapped waves of the first oxidation step and the potentials of the second oxidation step E_2 are summarized in Table 1. A representative cyclic voltammogram is shown in Fig. 2.

Experimental

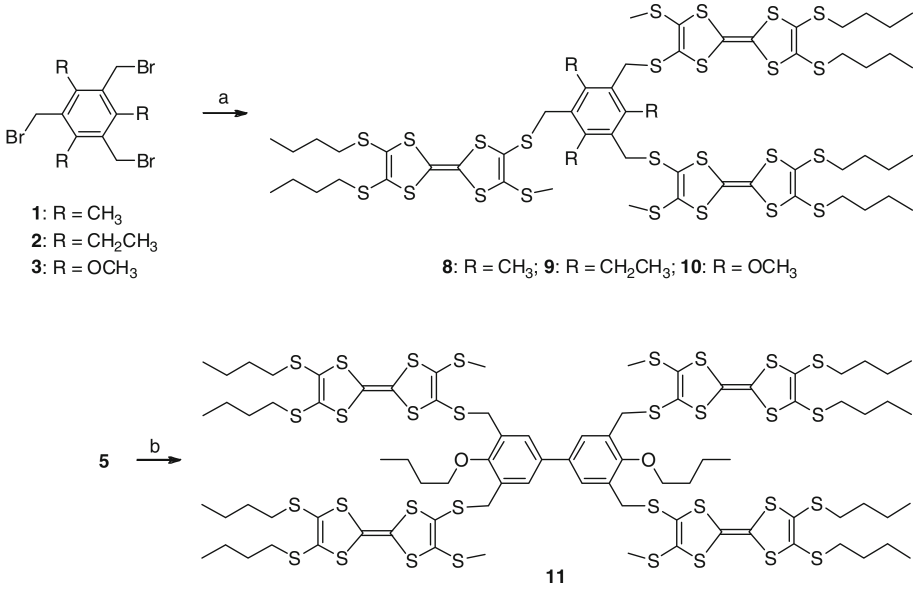
Melting points were determined on a Mikro-Heiztisch Polytherm A apparatus (Hund Wetzlar, Germany). NMR spectra were measured on Bruker Avance 400 (^1H at 400 MHz and ^{13}C at 101 MHz) and Bruker Avance 600 (^1H at 600 MHz and ^{13}C at 151 MHz) spectrometers. Chemical shifts (in ppm) were referenced to tetramethylsilane or residual solvent peaks as internal standards. Coupling constants (*J*) are given in hertz. Mass spectra were recorded on spectrometers LCQ Classic (Thermo Finnigan) and Q-Tof micro (Waters) with EI (70 eV) or ES ionization. UV–Vis spectra were measured on a double-beam spectrometer VARIAN Cary 5000 (1-cm quartz cell) at 25 °C. The reaction progress was monitored by thin-layer chromatography (TLC) on aluminum sheets

Scheme 1



Reagents and yields: (a) *n*-BuBr, K_2CO_3, MeCN, 85%; (b) $(CH_2O)_n$, KBr, $AcOH/H_2SO_4$, 53%; (c) $CsOH{\cdot}H_2O$, MeOH/DMF, CH_3I, 98%

Scheme 2



1: R = CH_3
2: R = CH_2CH_3
3: R = OCH_3

8: R = CH_3; **9**: R = CH_2CH_3; **10**: R = OCH_3

5

11

Reagents and yields: (a) **7** (3 equiv), $CsOH \cdot H_2O$ (3.3 equiv), MeOH/DMF, 65% of **8**, 65% of **9**, 71% of **10**; (b) **7** (4 equiv), $CsOH \cdot H_2O$ (4.4 equiv), MeOH/DMF, 63% of **11**

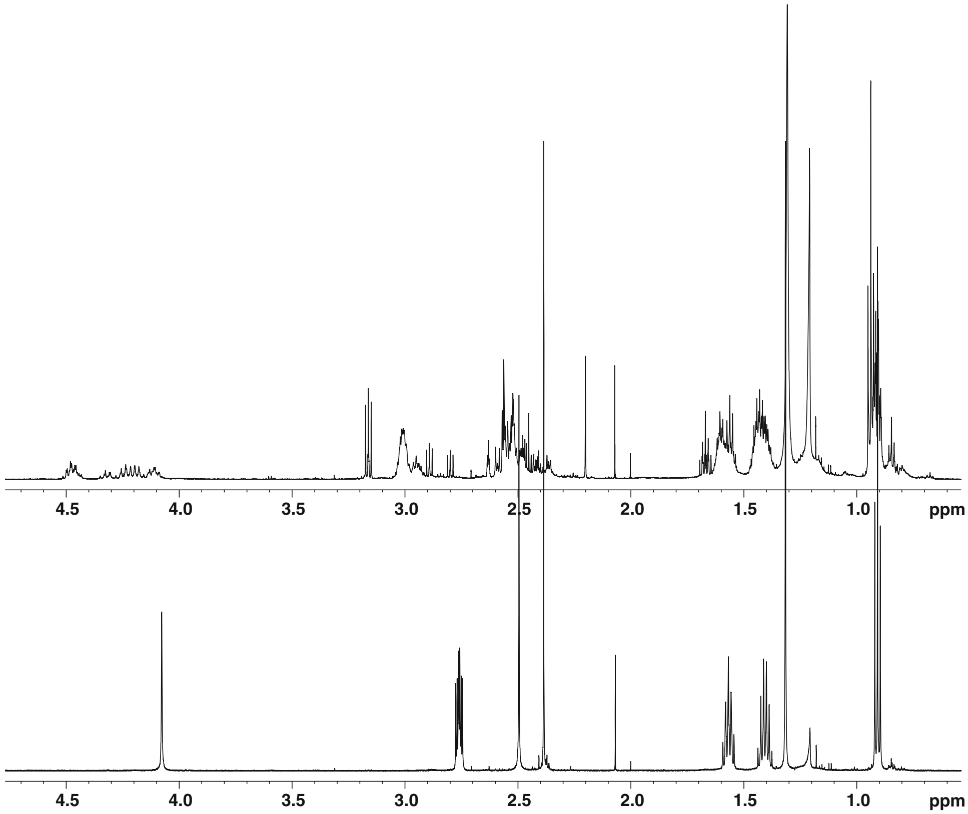


Fig. 1 600 MHz ^1H NMR spectra of the free ligand **8** (*bottom*) and its mixture with 4 equiv. of C_{60} (*top*), both recorded in a CS_2/CD_2Cl_2 2:1 mixture at 25 °C

pre-coated with silica gel 60 F254 (Merck), and column chromatography was carried out using silica gel 60 (Merck). Commercially available reagent grade chemicals and dry solvents were used as received. The term "hexanes" indicates a mixture of aliphatic hydrocarbons with b.p. 60–80 °C.

Table 1 Reversible redox potentials of compounds **8**–**11** versus the ferrocene/ferricenium couple in 1,2-dichloroethane

Compound	$E_{1,1}$ (V)	$E_{1,2}$ (V)	E_2 (V)
8	0.034	0.064	0.399
9	0.023	0.073	0.394
10	0.024	0.069	0.399
11	0.019	0.099	0.414

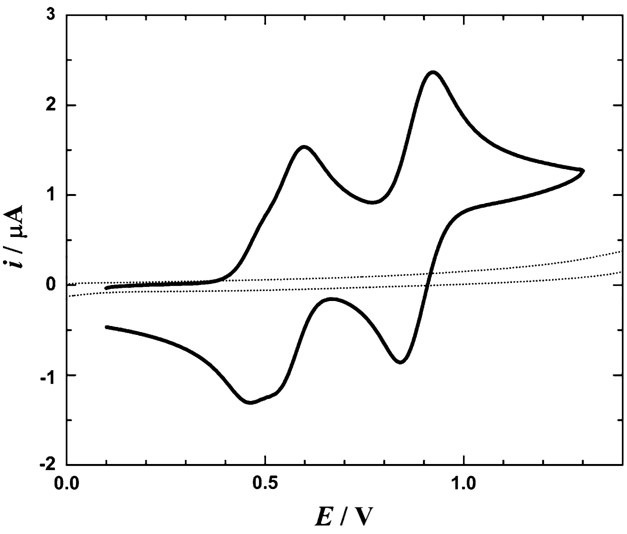


Fig. 2 Cyclic voltammogram of 0.56 mM ligand **8** and 0.1 M TBAPF$_6$ in 1,2-dichloroethane (glassy carbon electrode, scan rate 0.1 V s^{-1}, $E_{\mathrm{Fc/Fc+\cdot}} = 0.491$ V)

Elemental analyses (C, H, N, S) of the new starting compounds were conducted using a PE 2400 Series II CHNS/O Analyzer (Perkin Elmer, USA), and their results were found to be in good agreement (±0.2%) with the calculated values. On the contrary, the character of the target ligands in this study hampered the determination of elemental composition with satisfactory precision. We found that the substances in general bind solvent molecules very tightly in non-stoichiometric ratio and their thermal stability prevents long-term vacuum drying at an elevated temperature. Nevertheless, HRMS data provided adequate confirmation of their composition.

Electrochemical measurements were performed using a potentiostat/galvanostat Autolab PGSTAT30 equipped with a frequency response module (Eco Chemie, The Netherlands). A three-electrode electrochemical cell was used. The reference electrode, Ag|AgCl|3 M KCl, was separated from the test solution by a non-aqueous salt bridge. The working electrode was a glass-sealed glassy carbon disc of 0.5-mm diameter. The auxiliary electrode was a platinum wire. The scan rate of the applied DC potential was in the range 0.05–10 V/s. Tetrabutylammonium hexafluorophosphate (TBAPF$_6$) and 1,2-dichloroethane were supplied by Sigma-Aldrich. The indifferent electrolyte TBAPF$_6$ was recrystallized and dried under vacuum. 1,2-Dichloroethane was dried over activated molecular sieves. Oxygen was removed from the solution by passing through a stream of argon saturated with solvent vapors.

1,3,5-Tris(bromomethyl)-2,4,6-trimethoxybenzene (**3**)
Into a stirred mixture of 2.50 g 1,3,5-trimethoxybenzene (14.9 mmol) and 2.00 g paraformaldehyde (66.6 mmol) in 5 cm^3 acetic acid heated to 70 °C 11 cm^3 of a solution of hydrogen bromide in acetic acid (33% HBr) was dropped. The reaction mixture was kept at this temperature for 20 h, then 300 cm^3 water was added and the mixture was extracted with dichloromethane (3 × 120 cm^3). The combined extracts were washed with water (2 × 100 cm^3) and dried over MgSO$_4$. After evaporation of the solvent the crude product was purified by column chromatography on silica gel (eluent hexanes/acetone/diethyl ether 80:10:10). Recrystallization of relevant fractions from *n*-hexane afforded 1.55 g (23%) of **3**. M.p.: 130–132 °C (Ref. [40] 126 °C); ^{1}H and ^{13}C NMR spectra were found to be identical with those described in Ref. [40].

4,4′-Dibutoxy-1,1′-biphenyl (**4**)
A mixture of 9.31 g 4,4′-dihydroxy-1,1′-biphenyl (50.0 mmol), 16.4 g 1-bromobutane (120 mmol), and 13.8 g anhydrous potassium carbonate (100 mmol) in 300 cm^3 acetonitrile was refluxed for 40 h. After evaporation to dryness, 200 cm^3 water was added and the organic portions were extracted with toluene (3 × 150 cm^3). Combined extracts were dried (MgSO$_4$) and concentrated to 200 cm^3. Upon diluting the solution with 200 cm^3 *n*-hexane the product crystallized. Crystals were filtered, washed with *n*-hexane, and dried. Yield of **4** was 12.6 g (85%). M.p.: 151 °C (Ref. [41] 145–146 °C); ^{1}H NMR spectrum was found to be identical with that described in Ref. [41].

3,3′,5,5′-Tetrakis(bromomethyl)-4,4′-dibutoxy-1,1′-biphenyl (**5**, $C_{24}H_{30}Br_4O_2$)
To a suspension of 2.69 g **4** (9.00 mmol), 1.90 g paraformaldehyde (63.3 mmol), and 9.00 g potassium bromide (7.56 mmol) in 15 cm^3 acetic acid a mixture of 7 cm^3 sulfuric acid and 7 cm^3 acetic acid was added dropwise. The resulting mixture was heated to 95 °C for 6 h, diluted with 100 cm^3 water, and extracted with dichloromethane (3 × 120 cm^3). The combined extracts were washed with water (2 × 100 cm^3) and dried with MgSO$_4$, then the solvent was evaporated. The oily residue was dissolved in 300 cm^3 *n*-hexane, the brown solution was decolorized with silica gel (ca. 1 g), silica gel was filtered off, and the solution was concentrated to 100 cm^3. After standing in the fridge overnight the crystals were filtered, washed with *n*-hexane, and dried. Yield of **5** was 3.20 g (53%). M.p.:

167 °C; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 1.05$ (t, 3H, $J = 7.6$ Hz), 1.62 (m, 4H), 1.92 (m, 4H), 4.15 (t, 4H, $J = 6.6$ Hz), 4.60 (s, 8H), 7.54 (s, 4H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): $\delta = 13.97$, 19.22, 27.54, 32.43, 74.94, 130.58, 132.51, 136.33, 155.35 ppm; MS (70 eV): $m/z = 670$ (M^+), 614, 558, 477, 453, 387, 317, 165, 149, 80; HRMS (EI+): for $C_{24}H_{30}Br_4O_2$ [M^+] calcd. $m/z = 665.8979$, found 665.8999.

3,3′-[[2-[4,5-Bis(butylthio)-1,3-dithiol-2-ylidene]-1,3-dithiole-4,5-diyl]bis(thio)]bis(propanenitrile) (**6**) *[42]*

The coupling reaction [44] of 4.89 g 4,5-bis(cyanoethylthio)-1,3-dithiol-2-one [42] (17.0 mmol) and 5.27 g 4,5-bis-(butylthio)-1,3-dithiol-2-thione [42] (17.0 mmol) in 15 cm^3 triethyl phosphite gave 5.00 g (53%) of the TTF derivative **6**. M.p.: 95–96 °C (Ref. [42] 94–95 °C); ^{1}H and ^{13}C NMR spectra were found to be identical with those described in Ref. [42].

3-[[2-[4,5-Bis(butylthio)-1,3-dithiol-2-ylidene]-5-(methylthio)-1,3-dithiol-4-yl]thio]propanenitrile (**7**, $C_{18}H_{25}NS_8$)

To a solution of 2.20 g **6** (4.00 mmol) in 90 cm^3 *N,N*-dimethylformamide (DMF) a degassed solution of 0.739 g $CsOH\cdot H_2O$ (4.40 mmol) in 10 cm^3 MeOH was added under Ar, followed after 15 min by 0.682 g iodomethane (4.80 mmol). The reaction mixture was stirred for 5 h at r.t. Then, the mixture was diluted with 400 cm^3 water, the precipitate was filtered, washed with water, and dried. Crystallization from 300 cm^3 *n*-heptane afforded 2.01 g (98%) of **7**. M.p.: 89–91 °C; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 0.93$ (t, 6H, $J = 7.2$ Hz), 1.45 (m, 4H), 1.62 (m, 4H, $J = 7.6$ Hz), 2.48 (s, 3H), 2.71 (t, 2H, $J = 7.2$ Hz), 2.83 (t, 4H, $J = 7.6$ Hz), 3.03 (t, 2H, $J = 7.2$ Hz) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): $\delta = 13.58$, 18.75, 19.07, 21.65, 31.20, 31.77, 36.04, 112.89, 117.52, 120.10, 127.71, 127.99, 130.29, 135.11 ppm; MS (70 eV): $m/z = 511$ (M^+), 457, 322, 223, 88, 44; HRMS (EI+): for $C_{18}H_{25}NS_8$ [M^+] calcd. $m/z = 510.9753$, found 510.9743.

4,4′,4″-[(2,4,6-Trimethylbenzene-1,3,5-triyl)tris(methylenethio)]tris[2-[4,5-bis(butylthio)-1,3-dithiol-2-ylidene]-5-(methylthio)-1,3-dithiole] (**8**, $C_{57}H_{78}S_{24}$)

$CsOH\cdot H_2O$ (67.1 mg, 0.400 mmol) was dissolved in 3 cm^3 methanol and added dropwise under Ar to a solution of 169 mg TTF derivative **7** (0.330 mmol) in 9 cm^3 dry and degassed DMF over 10 min and the resulting solution was stirred for 15 min. Tris(bromomethyl) derivative **1** (40.2 mg, 0.100 mmol) in 7 cm^3 dry and degassed DMF was added dropwise during 2 h and the reaction mixture was stirred for 5 h at r.t. Then, the mixture was diluted with 150 cm^3 water, the precipitate was filtered, washed with water, and dried. Column chromatography of the crude product on silica gel (eluent hexanes/CH_2Cl_2 2:1) and crystallization from acetone afforded the ligand **8** (102 mg, 65%). M.p.: 66–69 °C; ^{1}H NMR (600 MHz, CD_2Cl_2): $\delta = 0.847$ (t, 9H, $J = 7.4$ Hz), 0.850 (t, 9H, $J = 7.4$ Hz), 1.36 (m, 12H), 1.54 (m, 12H), 2.34 (s, 9H), 2.47 (s, 9H), 2.76 (m, 12H), 4.08 (s, 6H) ppm; ^{13}C NMR (151 MHz, CD_2Cl_2): $\delta = 15.26$, 15.28, 17.92, 20.96, 23.51, 33.66, 33.68, 37.85, 38.62, 125.66, 129.61, 129.72, 132.88, 134.85, 139.14 ppm; UV–Vis (dichloromethane, $c = 9.79 \times 10^{-5}$ mol dm^{-3}): λ_{max} (ε) = 333 (61,600), 311 (65,600) nm (mol^{-1} dm^3 cm^{-1}); HRMS (ES+): for $C_{57}H_{78}S_{24}Na$ [$(M+Na)^+$] calcd. $m/z = 1{,}552.9293$, found 1,552.9270.

4,4′,4″-[(2,4,6-Triethylbenzene-1,3,5-triyl)tris(methylenethio)]tris[2-[4,5-bis(butylthio)-1,3-dithiol-2-ylidene]-5-(methylthio)-1,3-dithiole] (**9**, $C_{60}H_{84}S_{24}$)

The reaction procedure described for the ligand **8** was applied to 169 mg TTF derivative **7** (0.330 mmol) and 44.8 mg tris(bromomethyl) derivative **2** (0.100 mmol). Column chromatography of the crude product on silica gel (eluent hexanes/CH_2Cl_2 2:1) afforded the pure ligand **9** (103 mg, 65%). M.p.: 92–94 °C; ^{1}H NMR (600 MHz, CD_2Cl_2): $\delta = 0.847$ (t, 9H, $J = 7.4$ Hz), 0.851 (t, 9H, $J = 7.4$ Hz), 1.20 (t, 9H, $J = 7.5$ Hz), 1.36 (m, 6H), 1.37 (m, 6H), 1.54 (m, 12H), 2.37 (s, 9H), 2.76 (m, 12H), 2.93 (m, 6H), 4.04 (s, 6H) ppm; ^{13}C NMR (151 MHz, CD_2Cl_2): $\delta = 15.26$, 15.27, 17.88, 21.00, 23.51, 24.98, 33.66, 33.70, 37.38, 37.85, 111.61, 112.76, 125.86, 129.62, 129.74, 131.84, 134.62, 146.27 ppm; UV–Vis (dichloromethane, $c = 9.84 \times 10^{-5}$ mol dm^{-3}): λ_{max} (ε) = 333 (63,000), 312 (67,400) nm (mol^{-1} dm^3 cm^{-1}); HRMS (ES+): for $C_{60}H_{84}S_{24}Na$ [$(M+Na)^+$] calcd. $m/z = 1{,}594.9762$, found 1,594.9758.

4,4′,4″-[(2,4,6-Trimethoxybenzene-1,3,5-triyl)tris(methylenethio)]tris[2-[4,5-bis(butylthio)-1,3-dithiol-2-ylidene]-5-(methylthio)-1,3-dithiole] (**10**, $C_{57}H_{78}O_3S_{24}$)

The reaction conditions and purification procedure described for the ligand **8** was applied to 169 mg TTF derivative **7** (0.330 mmol) and 45.0 mg tris(bromomethyl) derivative **3** (0.100 mmol). Column chromatography of the crude product on silica gel (eluent hexanes/CH_2Cl_2 3:2) yielded the pure ligand **10** (112 mg, 71%). M.p.: 65–67 °C; ^{1}H NMR (600 MHz, CD_2Cl_2): $\delta = 0.847$ (t, 9H, $J = 7.4$ Hz), 0.849 (t, 9H, $J = 7.4$ Hz), 1.36 (m, 12H), 1.54 (m, 12H), 2.32 (s, 9H), 2.75 (m, 12H), 3.91 (s, 9H), 4.04 (s, 6H) ppm; ^{13}C NMR (151 MHz, CD_2Cl_2): $\delta = 15.26$, 15.28, 20.99, 23.51, 32.38, 33.67, 33.68, 37.84, 65.25, 111.89, 112.66, 122.64, 126.49, 129.62, 134.30, 161.22 ppm; UV–Vis (dichloromethane, $c = 9.84 \times 10^{-5}$ mol dm^{-3}): λ_{max} (ε) = 333 (56,200), 311 (59,600) nm (mol^{-1} dm^3 cm^{-1}); HRMS (ES+): for $C_{57}H_{78}O_3S_{24}Na$ [$(M+Na)^+$] calcd. $m/z = 1{,}600.9140$, found 1,600.9144.

4,4′,4″,4‴-[(4,4′-Dibutoxy-[1,1′-biphenyl]-3,3′,5,5′-tetrayl)tetrakis(methylenethio)]tetrakis[2-[4,5-bis(butylthio)-1,3-dithiol-2-ylidene]-5-(methylthio)-1,3-dithiole] (**11**, $C_{84}H_{114}O_2S_{32}$)

The reaction conditions and chromatographic purification described for the ligand **8** was applied to 225 mg TTF derivative **7** (0.440 mmol) and 67.2 mg tetrakis (bromomethyl) derivative **5** (0.100 mmol). Column chromatography of the crude product on silica gel (eluent hexanes/CH_2Cl_2 3:2) afforded the pure ligand **11** (137 mg, 63%, orange semicrystalline compound). ^{1}H NMR (600 MHz, CD_2Cl_2): $\delta = 0.84$ (t, 12H, $J = 7.4$ Hz), 0.85 (t, 12H, $J = 7.4$ Hz), 0.96 (t, 6H, $J = 7.4$ Hz), 1.36 (m, 16H), 1.52 (m, 4H), 1.53 (m, 16H), 1.81 (m, 4H), 2.75 (m, 16H), 4.00 (t, 4H, $J = 6.5$ Hz), 4.01 (s, 8H), 7.40 (s, 4H) ppm; ^{13}C NMR (151 MHz, CD_2Cl_2): $\delta = 15.30$, 15.89, 17.88, 20.86, 21.35, 23.54, 33.68, 33.70, 34.54, 37.01, 37.85, 37.88, 111.30, 112.26, 125.36, 129.55, 129.80, 130.84, 132.99, 135.95, 137.30, 157.12 ppm; UV–Vis (dichloromethane, $c = 9.95 \times 10^{-5}$ mol dm^{-3}): $\lambda_{\max}$ (ε) = 333 (71,700), 310 (80,100) nm (mol^{-1} dm^3 cm^{-1}); HRMS (ES+): for $C_{84}H_{114}O_2S_{32}$ [M^{2+}] calcd. $m/z =$ 1,088.9935; found 1,088.9928.

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