Dinaphthotetrathiafulvalene Bisimides: A New Member of the Family of \(\pi\)-Extended TTF Stable p-Type Semiconductors


Abstract: Air-stable organic semiconductors based on tetrathiafulvalene (TTF) were developed by synthesising a series of dinaphthotetrathiafulvalene bisimides (DNTTF-Im) using electron-donating TTF, \(\pi\)-extended naphthalene, and electron withdrawing imide. Electron spin resonance spectroscopy and X-ray single crystal structure analysis of aryI-substituted DNTTF-Im radical cations confirmed that localization of the spin resides on the electron donating TTF moiety. The organic field-effect transistor properties derived from the use of highly crystalline n-butyl(C4) and n-hexyl(C6)-substituted DNTTF-Im were assessed. The hole carrier mobility of C6-DNTTF-Im was improved from \(3.7 \times 10^{-3} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}\) to \(0.30 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}\) in ambient conditions. This is attributed to the raise of the substrate temperature from 25 °C to 200 °C during sublimation. The XRD and microscopy analysis suggested that increasing the substrate temperature accelerates the end-on packing resulting in larger grains suitable for hole charge transport parallel to the substrate.

Tetrathiafulvalenes (TTFs) are one of the most studied organic semiconductor materials serving as building blocks in charge-transfer salts with applications in organic conductors and superconductors. In 2004, a single-crystal organic field-effect transistor (SC-OFET) was fabricated from dithiophene-fused TTF (DT-TTF). This was found to be a promising p-type semiconductor with good hole mobility \(\mu_h\) of 1.4 cm² V⁻¹ s⁻¹, owing to the intermolecular S=S interaction and the \(\pi\)-\(\pi\) interaction of the \(\pi\)-electron-\(\pi\)-conjugated flat framework in single crystals. Since then, new families of \(\pi\)-extended TTF derivatives (Figure 1) have been under extensive study for use as p-type organic semiconductors for OFETs. DibenzoTTF (DB-TTF), an isoelectronic compound of DT-TTF, exhibited acceptable \(\mu_h\) of up to 1 cm² V⁻¹ s⁻¹ with suitable packing structure for charge transport. Despite the level of mobility in the single crystal, thin films of DT-TTF and DB-TTF did not show similar behaviour due to a strong electron donating effect of TTF moieties rendering the films susceptible to oxygen intrusion.

In order to improve the OFET performance of TTF derivatives, several ways of modifying the chemical structure of DB-TTF have been investigated. One of the methods involves the introduction of fused benzene rings, such as dinaphthalene (DN) and dianthrene (DA) to extend the \(\pi\)-conjugation in DN- and DA-TTFs. This resulted in improved bottom contact OFET performance of the DB-TTF films (0.06 cm² V⁻¹ s⁻¹) to 0.42 cm² V⁻¹ s⁻¹ for DN-TTF. The equivalent value for DA-TTF with top contact devices was increased to 2.2 cm² V⁻¹ s⁻¹. Synthesis of tetracene-fused TTF was also attempted by our research group but limited solubility prevented synthesis completion. Tetraphenylene-substituted derivatives were solely successfully obtained. An alternative method to improve oxygen stability of TTFs was reported by Yamashita et al. who introduced electron deficient nitrogen heterocycles, such as pyrazine and quinoxaline, instead of hydrocarbon rings. The mobility of the bottom contact OFET dipyrazino(DB)-TTF was only 3.3 \(\times 10^{-5}\) cm² V⁻¹ s⁻¹. On the contrary, the diquinoloxalino(DQ)-TTF exhibited a hole mobility of 0.2 cm² V⁻¹ s⁻¹ in a top contact configuration with an Al₂O₃ substrate and 6.3 \(\times 10^{-3}\) cm² V⁻¹ s⁻¹ when deposited on a SiO₂ substrate. DQ-TTF was sufficiently stable in air but the orientation of DQ-TTF on the SiO₂ substrate did not favour enhanced mobility. Finally, Jen and coworkers investigated the synthesis of benzothiadiazole(BT)-TTF and they reported carrier mobilities of \(10^{-2}\) cm² V⁻¹ s⁻¹ for thin film transistors and up to 0.34 cm² V⁻¹ s⁻¹ for microcrystal transistors. However, the stability of this compound was not assessed.

Bismide is another well-known soluble moiety with electron withdrawing functionality. Various acene and nylene bisimides have been reported to be good p- or ambipolar semiconductors. In the TTF family, only n-butyl- and n-hexyl-substituted dibenzo TTF bismide (C4- and C6-DDBTTF-Im) have been proposed for use as air-stable p-type materials (Figure 2). Hole mobility of up to 0.40 cm² V⁻¹ s⁻¹ in air was reported for C6-

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Supporting information for this article is given via a link at the end of the document.
In this study, the combination of electron donating TTF and electron withdrawing imide with a larger \( \pi \)-conjugated framework was investigated in an attempt to improve both the charge transport and chemical stability of semiconductors developed. Hence, novel dinaphthoimide bisimides (DNTTF-Im's) were synthesised and the charge transport properties of this new semiconductor were evaluated (Figure 2). In order to control the solubility and molecular packing structures, a series of alkyl substituted derivatives (\( \text{C}_n \)-DNTTF-Im's: alkyl = \( n \)-butyl (C4), \( n \)-hexyl (C6), \( n \)-octyl (C8), \( n \)-decyl (C10), and \( n \)-dodecyl (C12)) were prepared. 2,6-Diisopropylphenyl-substituted DNTTF-Im (Ph-DNTTF-Im) was also prepared to investigate the electric and optical properties of both the neutral DNTTF-Im and its radical cation (DNTTF-Im\(^{+} \)) in solution.

The highest occupied molecular orbitals (HOMOs) and the lowest unoccupied molecular orbitals (LUMOs) of DNTTF, C1-DNTTF-Im, and C1-DBTTF-Im were calculated using Gaussian 09 at the B3LYP/6-31G(d) level (Figure 3).\(^{[25]}\) The alkyl groups attached to nitrogen are simplified to methyl groups. The molecular orbitals of HOMO of these compounds are localized on the electron donating TTF moiety, while those of LUMO are localized on naphthalene moiety for DNTTF and the electron withdrawing naphtho- and benzo-imide moieties for C1-DNTTF-Im and C1-DBTTF-Im. The HOMO energy levels of C1-DNTTF-Im and C1-DBTTF-Im are \(-5.51\) and \(-5.53\) eV, LUMO energy levels are \(-2.23\) and \(-2.50\) eV, and the band gap energy values \( (E_{\text{gap}}) \) are 3.28 and 3.03 eV, respectively. Due to the similar HOMO level of C1-DNTTF-Im and C1-DBTTF-Im, DNTTF-Im is expected to be stable in air similar to DBTTF-Im.

The synthesis of C4-DNTTF-Im is presented in Scheme 1. 4,5-Dibromoxylene was brominated with N-bromosuccinimide (NBS) and azobisisobutyronitrile (AIBN) to produce 1,2-dibromo-4,5-bis(dibromomethyl)benzene (1) in 61% yield. Hexabromoxylene 1 was subsequently reacted with \( N \)-butylimaleimide to give \( N \)-butyl-dibromonaphthalenelimide (2a) in 59% yield. Imide 2a was converted to thione 3a in 75% yield by carbon disulfide (CS\(_2\)) and potassium sulfide (K\(_2\)S). Thione 3a was then converted to ketone 4a by mercury(II) acetate (Hg(OAc)\(_2\)) in 88% yield. A coupling reaction of 3a and 4a by triethylphosphite (P(OEt)\(_3\)) gave C4-DNTTF-Im in 62% yield. C6-DNTTF-Im, C8-DNTTF-Im, C10-Ph-DNTTF-Im, and C12-Ph-DNTTF-Im were synthesised in a similar way (Scheme 1). The reference compound, 2,6-diisopropylphenyl-substituted dibenzotz topoTTF bisimide (Ph-DBTTF-Im), was also prepared as shown in Scheme S1.

Ph-DNTTF-Im and Ph-DBTTF-Im were characterized by \(^1\)H and \(^{13}\)C NMR spectras, MALDI-TOF-mass spectra (Figure S1), and X-ray single crystal structure analyses (Figures S2 and S3 and Tables S1 and S2). Given that all of the synthesised Cn-DNTTF-Im’s are insoluble in common organic solvents, purification of the compounds was achieved by sublimation. The compounds were identified by HR Spiral TOF-MS spectroscopy. The purity of the compounds was assessed by elemental analysis and only C4- and C6-DNTTF-Im exhibited adequate levels of purity within a 0.3% error margin between observed and calculated values. The OFET measurement was carried out only for the C4- and C6-DNTTF-Im compounds.

To investigate the redox properties of DNTTF-Im, cyclic voltammograms (CVs) and differential pulse voltammograms...
(DPVs) of Ph-DNTTF-Im were measured and compared against the Ph-DBTC-Im (Figure S5). Ph-DNTTF-Im exhibits two reversible oxidation potentials at 0.45 and 0.85 V (vs. Fc/Fc\(^{-}\)) in dichloromethane containing 0.1 M tetrabutylammonium hexafluorophosphate (n-Bu\(_4\)NPF\(_6\)). These values are similar to 0.48 and 0.85 V (vs. Fc/Fc\(^{+}\)) for Ph-DNTTF-Im. Similarity in oxidation potentials of Ph-DNTTF-Im and Ph-DBTC-Im coincides with their corresponding HOMO energy levels estimated by DFT calculations (Figure 3). The reduction peaks were not observed for Ph-DNTTF-Im and Ph-DNTTF-Im in dichloromethane.

The UV-VIS absorption spectra of Ph-DNTTF-Im and Ph-DBTC-Im are depicted in Figure 4a. The absorption peaks are observed at 281, 308, 356, and 431 nm for Ph-DNTTF-Im and 315 and 376 nm for Ph-DBTC-Im in dichloromethane. The optical gap (\(E_{\text{opt}}\)) between HOMO and LUMO energy levels was estimated, based on the edges of the absorption spectra, to be 2.48 and 2.25 eV for Ph-DNTTF-Im and Ph-DBTC-Im, respectively. The HOMO and LUMO energy levels estimated by DPV and UV-VIS absorption spectra are summarized in Table S4, which also includes the theoretical values obtained by DFT calculation.

Absorption spectra of Ph-DNTTF-Im in the presence of one-electron oxidant, tris(4-bromophenyl)ammoniumyl hexachloroantimonate ((p-BrC\(_6\)H\(_4\))\(_3\)N\(^+\)SbCl\(_6\)^--), were also obtained. When 1 eq. of (p-BrC\(_6\)H\(_4\))\(_3\)N\(^+\)SbCl\(_6\)^-- was added by 0.2 eq. in five equal portions, the absorption spectra of Ph-DNTTF-Im was modified as shown in Figure 4b: the peaks at 356 and 431 nm decreased while new peaks at 303, 480, and 630 nm increased with isosbestic points at 333, 372, 446 nm. Upon excess addition of oxidant, the spectrum did not show further change. A similar phenomenon was observed for Ph-DNTTF-Im (Figure S6a) by addition of (p-BrC\(_6\)H\(_4\))\(_3\)N\(^+\)SbCl\(_6\)^--. The spectrum of the radical cation of Ph-DNTTF-Im (Ph-DNTTF-Im\(^{+}\)) had the peaks at 304, 435 (br), and 629 nm with isosbestic points at 310, 358, and 380 nm. Spectroelectrochemical analysis was also carried out in order to simultaneously observe the redox behaviour and the spectral signatures. When a potential of 0.6 V (vs. Fc/Fc\(^{+}\)) was applied to Ph-DNTTF-Im in dichloromethane containing 0.1 M n-Bu\(_4\)NPF\(_6\), the peaks at 480 and 630 nm appeared while the peaks at 356 and 431 nm decreased (Figure 4c). The spectrum was restored to the original one by adjusting the applied potential back to 0 V (vs. Fc/Fc\(^{+}\)). The spectrum obtained at 0.6 V is similar to the one obtained in the presence of 1 eq. of (p-BrC\(_6\)H\(_4\))\(_3\)N\(^+\)SbCl\(_6\)^--. This suggests that the spectrum is assigned to Ph-DNTTF-Im\(^{+}\). The radical cation of Ph-DNTTF-Im (Ph-DNTTF-Im\(^{+}\)) also showed a similar trend (Figure S6b).

The existence of the radical cation was confirmed by electron spin resonance (ESR) spectroscopy of Ph-DNTTF-Im (Figure S7a). The ESR spectrum of Ph-DNTTF-Im (2.0 \(\times\) 10\(^{-4}\) M) in dichloromethane in the presence of (p-BrC\(_6\)H\(_4\))\(_3\)N\(^+\)SbCl\(_6\)^-- was measured at \(-90\) °C. A broad spectrum of Ph-DNTTF-Im\(^{+}\) was obtained. The radical cation Ph-DNTTF-Im\(^{+}\) had a similar spectrum (Figure S7b). The similar broad signals were also observed at 77 K in CD\(_2\)Cl\(_2\). DFT calculations suggest the spin density of Ph-DNTTF-Im\(^{+}\) and Ph-DNTTF-Im\(^{+}\) mainly locates on TTF moiety and expands to the imide moieties (Figure S8).

The single crystals of Ph-DNTTF-Im\(^{+}\) SbCl\(_6\)^-- required for the X-ray single crystal structure analysis, were obtained in the use of dichloromethane. The single crystal structure of the radical cation is presented in Figures 5 and S4 and Table S3. Both the radical cation of Ph-DNTTF-Im\(^{+}\) as well as the neutral Ph-DNTTF-Im (Figure S2) had flat structures with Ph groups almost perpendicular to imide planes: 88.5° and 86.7° for Ph-DNTTF-Im\(^{+}\) and 79.8° for Ph-DNTTF-Im. Ph-DNTTF-Im is slightly sigmoidal and packed in parallel. A toluene molecule is confined between two Ph-DNTTF-Im molecules. In contrast, the Ph-DNTTF-Im\(^{+}\) is slightly bow-shaped with three dichloromethane molecules and a counter anion, SbCl\(_6\)^-- included per cation radical. The counter anion, SbCl\(_6\)^--, is located over one of the dithiapentene rings. Because of the alternate structure of Ph-DNTTF-Im\(^{+}\) and SbCl\(_6\)^-- the SbCl\(_6\)^-- on the other side is close to the other dithiapentene ring (point-symmetric movement). The bond lengths of Ph-DNTTF-Im and Ph-DNTTF-Im\(^{+}\), i.e. the C–C bond length at the centre of the molecules, are 1.35(1) Å (typical double bond) in the case of the neutral molecule and 1.38(2) Å in the case of the cation radical. The C-S bond lengths of TTF moiety of Ph-DNTTF-Im\(^{+}\) are
shorter than those of Ph-DNTTF-Im (Figures S2 and S4). This is a typical characteristic of the TTF radical cations.[26] The bond lengths of Ph-DNTTF-Im and its radical cation are summarized in Figure S2d and S4d.

Figure 5. X-ray crystal structure of Ph-DNTTF-Im**: (a) top view and (b) side view. Thermal ellipsoids were scaled to 50% probability.

In order to exploit the stability of the radical cation structure, the OFET performance of DNTTF-Im was investigated. Instead of 2,6-disopropylphenyl groups, normal alkyl groups were introduced in order to achieve the strong packing between DNTTF-Im frameworks. Given that solely C4- and C6-DNTTF-Im were satisfactorily purified by sublimation, further experiments were performed using these two derivatives. The thermal stability of C4- and C6-DNTTF-Im was assessed by thermogravimetric analysis (Figure S9). Both derivatives were stable up to around 300 °C and a weight loss was observed at 310 °C for C4-DNTTF-Im and 299 °C for C6-DNTTF-Im due to the decomposition.

The HOMO energy levels of thin films of C4-DNTTF-Im and C6-DNTTF-Im were derived from ionisation energies. These were determined by the onset of photoelectron spectra obtained by a photoelectron spectrometer operating in air. The ionisation energies were 5.76 and 5.74 eV for the C4-DNTTF-Im and C6-DNTTF-Im, respectively (Figure S10 and Table S4). The E_{gap} was obtained by analysing the edge of the absorption spectra. The C4-DNTTF-Im and C6-DNTTF-Im films exhibited an E_{gap} of 2.40 and 2.39 eV, respectively (Figure S12). The LUMO energy levels were estimated combining the HOMO energy levels and E_{gap}. LUMO energy values of C4-DNTTF-Im and C6-DNTTF-Im were 3.36 and 3.35 eV, respectively. Hence, a marginal difference between the two DNTTFs investigated was observed.

The carrier-transport characteristics of the C4- and C6-DNTTF-Im thin films were evaluated in bottom-gate-top-contact OFETs. The active layer (50 nm) was prepared on octadecyltrichlorosilane (OTS)-treated Si/SiO2. Molybdenum trioxide (MoO3) (7 nm) followed by gold electrode (50 nm) was deposited on the active layer with channel length and width of 50 µm and 1 mm, respectively. When C6-DNTTF-Im was deposited on the substrate at 25 °C, the maximum hole mobility (µh) attained was 3.7 x 10^{-3} cm^2 V^{-1} s^{-1} and the on/off ratio was 2.3 x 10^{4} (Figure S12 and Table 1). Raising the substrate temperature to 150 °C during the deposition stage increased the magnitude of µh to 3.1 x 10^{-2} cm^2 V^{-1} s^{-1}. Further increase of the µh to 3.0 x 10^{-1} cm^2 V^{-1} s^{-1} was attained when the temperature of the substrate was 200 °C. The on/off ratio was also increased with the increase of the temperature: 6.1 x 10^{5} at 150 °C and 1.1 x 10^{6} at 200 °C. Applying the optimum conditions used for the synthesis C6-DNTTF-Im, an FET device of C4-DNTTF-Im was also developed. The optimum µh value obtained was 2.3 x 10^{-1} cm^2 V^{-1} s^{-1} and the on/off ratio was 9.2 x 10^{5} (Figure 6). These properties were comparable with those of the C6-DNTTF-Im OFET.

Table 1. Summary of OFET characteristics of C4- and C6-DNTTF-Im

<table>
<thead>
<tr>
<th>Cn-DNTTF-Im</th>
<th>T_{onset}</th>
<th>µh (max) / cm^2 V^{-1} s^{-1}</th>
<th>µh (average) / cm^2 V^{-1} s^{-1}</th>
<th>V_{th} / V</th>
<th>on/off ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>C6-</td>
<td>25</td>
<td>3.7 x 10^{-3}</td>
<td>3.3 x 10^{-3}</td>
<td>-43</td>
<td>2.3 x 10^{4}</td>
</tr>
<tr>
<td></td>
<td>150</td>
<td>3.1 x 10^{-2}</td>
<td>2.5 x 10^{-2}</td>
<td>-40</td>
<td>6.1 x 10^{5}</td>
</tr>
<tr>
<td></td>
<td>200</td>
<td>3.0 x 10^{-1}</td>
<td>1.7 x 10^{-1}</td>
<td>-47</td>
<td>1.1 x 10^{6}</td>
</tr>
<tr>
<td>C4-</td>
<td>200</td>
<td>2.3 x 10^{-1}</td>
<td>1.5 x 10^{-1}</td>
<td>-45</td>
<td>9.2 x 10^{5}</td>
</tr>
</tbody>
</table>

*Temperature of substrates during the vacuum deposition. **Average of 5 devices.

Figure 6. (a) Output curves and (b) transfer curves of bottom-gate-top-contact OFET of C4-DNTTF-Im thin film fabricated by sublimation at 200 °C.

The effect of substrate temperature during sublimation on film morphology was investigated by X-ray diffraction (XRD) of the films prepared on OTS-treated Si/SiO2 substrates (Figure 7 and S13). At 25 °C, the C6-DNTTF-Im film exhibited broad peaks at 2.9, 15.6, and 26.9°. At 150 °C, the peak at 2.9° was sharp and the peaks at 15.6 and 26.9° were weaker. At 200 °C, these trends were further promoted: the peak at 3.2° was sharp and the peaks at 15.6 and 26.9° were weaker. The peaks at 2.9, 15.6, and 26.9° correspond to 27.5, 5.7 and 3.3 Å, respectively. The optimized C6-DNTTF-Im had long- and short-axis lengths of 31.4 and 4.9 Å, respectively (Figure S14). X-ray single crystal structure of C4-DNTTF-Im showed columnar π-stacking along the crystallographic a-axis direction with an interplanar separation of 3.4 Å.[24] The intermolecular S=O and S=S contacts were observed between columns, displaying a one-dimensional sheet-
like structure along the crystallographic b-axis direction. The centre-to-centre distance between neighbouring columns can be estimated to be 5.7 Å, which is comparable to the 5.7 Å observed for the C6-DNTTF-Im. Although DNTTF-Im has more benzene rings compared to C4-DBTTF-Im, similar interplanar and intercolumnar distances in both TTFs can be attained provided that the packing structures are also similar. As it can be inferred from the above findings, the 27.5, 5.7, and 3.3 Å peaks correspond to the inclined end-on, inclined edge-on, and face-on packing structures, respectively. By increasing the temperature of the substrate, the edge-on and face-on structures dwindled in favour of the end-on structures. The end-on structure promotes the charge-carrier movement in parallel to the substrate surface and hence, the FET performance was enhanced. The XRD patterns of C4-DNTTF-Im at 200 ºC showed a strong sharp peak at 3.51° (25.2 Å), which corresponds to the inclined end-on packing of the molecules.

AFM images of these four type of films are shown in Figure 8. The film sublimated at 25 ºC consisted of smaller grains. However, increasing the substrate temperature resulted in larger grain sizes. Root-mean square (RMS) values are 14.4, 24.2, and 33.9 for 25, 150 and 200 ºC. The grain size of C4-DNTTF-Im at 200 ºC was also higher and the RMS value is 26.7. Findings suggest that crystal grain growth and charge-carrier mobility were favoured at higher temperatures of sublimation.

In summary, naphthalene-fused TTF imides were successfully synthesised. The charge transport properties in films prepared were investigated. Based on X-ray single crystal structure analysis and DFT calculations, it was established that the cation radical is localized on the TTF moieties. The charge-carrier mobility of OFET devices was improved by increasing the substrate sublimation temperature to 200 ºC as follows: 3.0 × 10⁻³ cm² V⁻¹ s⁻¹ for C6-DNTTF-Im and 2.3 × 10⁻³ cm² V⁻¹ s⁻¹ for C4-DNTTF-Im. These values were comparable to a previously reported mobility of C6-DBTTF-Im.¹¹ Interestingly, the hole mobility of C6-DBTTF-Im was one order of magnitude higher than that of C4-DBTTF-Im. The mobilities of C4- and C6-DNTTF-Im were comparable. Introduction of additional benzene rings can potentially improve the crystallinity of the C4-compound. XRD and microscopy analysis suggested that the end-on packing structure, being the optimum one for FET devices, was obtained during sublimation of the substrate at 200 ºC. In conclusion, the performance of FET devices was enhanced owing to the enhanced properties of TTF derivatives developed from this study.

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**Keywords:** TTF • imide • organic semiconductor • organic field effect transistor • radical cation

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[25] Gaussian 09, Revision C.01. See Supplementary Material for the full citation of Gaussian 09.
Stable p-type semiconductors: Hole mobility as high as 0.3 cm² V⁻¹ s⁻¹ in ambient condition was realized by a film of dinaphthotetrathiafulvalene bisimide by raising the substrate temperature to 200 °C during sublimation.

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