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Alternating Tetrafluorobenzene and Thiophene Units by Direct Arylation for Organic Electronics

Xiaoping Cui,[a],[c] Chengyi Xiao,*[b] Wei Jiang,[d],[e] and Zhaohui Wang*[a],[c]

Dedication ((optional))

Abstract: Direct arylation represents an attractive alternative to the conventional cross-coupling methods because of its step-economic and eco-friendly advantages. A set of simple D-A oligomeric molecules (**F-3**, **F-5**, and **F-7**) by integrating thiophene (T) and tetrafluorobenzene (F4B) alternatively units through direct arylation strategy is presented to obtain high-performance charge transporting materials. Single crystal analysis revealed their herringbone packing arrangements driven by intensive C-H...π interactions. Excellent hole transporting efficiency based on single-crystalline micro-plates/ribbons was witnessed that larger π-conjugation and D-A constitution give higher mobilities. Consequently, an average mobility of 1.31 cm^2 V^{-1} s^{-1} and a maximum mobility of 2.44 cm^2 V^{-1} s^{-1} for **F-7** were achieved, providing an effective way for excellently performing materials by designing simple D-A oligomeric systems.

There is increasing attention in the use of discrete molecules as electroactive and photoactive chromophores for organic electronics.[1] Because of having better-defined chemical structures and synthetic reproducibility, monodisperse oligomers instead of polymer counterparts have recently been explored in attempts to extrapolate a structure-properties-device performance relationship.[2] The "push-pull" systems with alternating donor-acceptor (D-A) structures show remarkable properties like strong charge transfer character, broadened absorptions, and narrowed band-gaps, which are excellently performing materials in organic solar cells.[3]

Direct arylation represents an attractive alternative to the conventional cross-coupling methods because of its step-economic and eco-friendly advantages.[4] Up to now, there are many examples of small molecules and polymers by transition-metal-catalyzed direct arylation using heteroarenes as orienting and activating components.[5] However, the rigorous application of such molecular systems as photoelectronic materials is still in its infancy.[6] Herein, we report a set of D-A oligomeric molecules with alternating thiophene (T) and tetrafluorobenzene (F4B) units (**F-3**, **F-5**, and **F-7**) by direct arylation strategy as presented in Figure 1. As the smallest analogue, **F-3** has been previously designed and synthesized by several methods that depend heavily on metallization of thiophene (Figure 1a).[7] Using these three model oligomers, we investigate the influences of electronic structures, molecular packing, and charge transport performances by the chromophore elongation. As the simplest heterocycle, thiophene is among the most interesting and widely investigated electron-rich building block for constructing conjugated systems.[8] By combining the F4B unit, the additional S…F nonbonding interactions can be induced between molecules that would favour for molecular planarity and thus enhance charge transfer properties significantly.[9] As a comparison, the corresponding thiophene-benzene alternating oligomers without fluorine substituents (**B-3**, **B-5**, and **B-7**) have been synthesized by Suzuki cross-coupling detailed in Scheme S1.

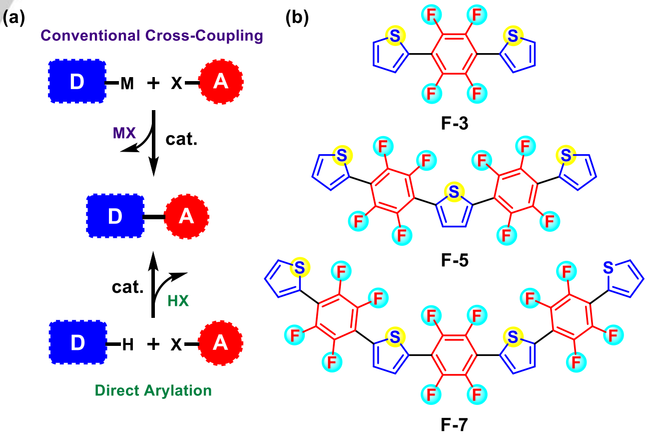

Figure 1. (a) Conventional cross-coupling versus direct arylation for the synthesis of D-A structural systems, M = SnR$_3$, B(OR)$_2$, MgX, ZnX, etc.; X = Cl, Br, I, etc.. (b) Chemical structures of thiophene-tetrafluorobenzene alternating oligomers (**F-3**, **F-5**, and **F-7**).

The synthetic routes to these three oligomers relying on the direct arylation approach are illustrated in Scheme 1. In our method, just pristine thiophene and 1,4-

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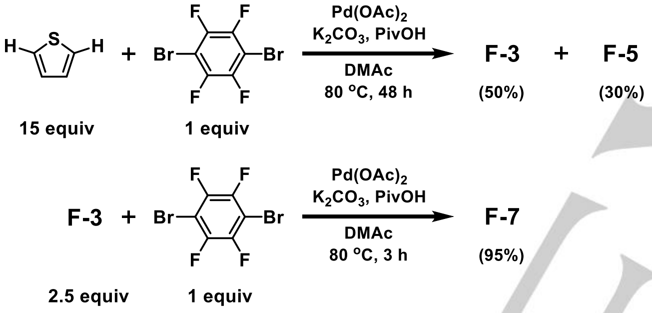
dibromotetrafluorobenzene are used as the key starting materials. Under simple procedure, using Pd(OAc)$_2$ as the catalyst, potassium carbonate (K$_2$CO$_3$) and pivalic acid (PivOH) as the base and the additive, and dimethylacetamide (DMAc) as the solvent, the two precursors with the amount ratio of 15:1 were coupled at 80 °C for 48 h.[10] As a consequence, **F-3** with three units and **F-5** with five units were formed in one pot, furnishing a total yield of about 80%. Then, **F-3** (2.5 equiv) further reacts with 1,4-dibromotetrafluorobenzene to afford **F-7** in a very high yield of about 95% within 3 hours. While the synthesis of fluorine-free counterparts (**B-3**, **B-5**, and **B-7**) combining the advantages of divergent and convergent strategies, are shown in Scheme S1. These three oligomers were prepared by a standard Suzuki coupling reaction because of the lack of activating fluorine atoms on benzene.[11] As the chain length increases, the solubility of the oligomers is less and less, whether in fluorine or fluorine-free molecules. Therefore, both **F-7** and **B-7** just have limited solubility in common organic solvents. All the oligomers possess good thermal stability except for **F-3** and **B-3** having low molecular weight which are easy to sublimate (Figure S1 and Table S1) in thermogravimetric analysis (TGA) measurements performed under nitrogen.



Scheme 1. Synthetic routes to **F-3**, **F-5**, and **F-7**.

The absorption spectra of three homogeneous oligomers with different length units in toluene are shown in Figure 2; the series showed one intense band that shifted bathochromically with the increasing number of units with the longest maximum at 325, 366, and 389 nm for **F-3**, **F-5**, and **F-7**, respectively, as a reflection of extended conjugation length of the D-A structure. We carried out electrochemistry measurements of these oligomer films to corroborate the HOMO values measured in THF solution; their HOMO levels are estimated from the oxidation onsets (0.72, 0.74, and 0.77 V for **F-3**, **F-5**, and **F-7**, respectively) to be about -5.5 eV, indicating good environmental stability as electronic materials.[12] And the optical band gaps determined from the onset absorptions are 3.49, 3.00, and 2.78 eV for **F-3**, **F-5**, and **F-7**, respectively.

Crystals suitable for single-crystal X-ray diffraction structure analysis were obtained by physical vapor transport technique by using a two-zone horizontal tube furnace.[13] As revealed by Figure 3a and 3c, **F-3** and **F-7** exhibit almost planar skeleton, and thiophene rings adopt an *anti* orientation slightly flap up and down around the tetrafluorobenzene centre with a twisting angle of about 6.04° and 6.46°, respectively. While **F-5** displays a more distorted and disordered structure, in which two isomers with two or three thiophene rings orientated in the same direction are observed; in other words, no *anti* isomer was formed in **F-5** crystals under the same crystallization conditions (Figure 3b). The crystal structures of all three oligomers show herringbone arrangements driven by C-H...π interactions (2.89 Å for **F-3**, 2.71-2.73 Å for **F-5**, and 2.87 Å for **F-7** shown in Figure 3). In addition, intensive F…F (2.47-2.93 Å) and C-H…F interaction (2.41-2.45 Å) networks are observed between neighbouring π-stacks in all single crystals, thus decrease the slip distance along the short molecular axis, which should be contributed to compact π−π stacking arrangements. It is worthy noting that the adjacent **F-3** and **F-7** molecules are observed to align in cofacial overlapping fashion with π-π distances of 3.51 Å and 3.49 Å, respectively. Whereas **F-5** showed shorter and intensive π−π interactions ranging from 3.26 to 3.39 Å (Figure 3b).

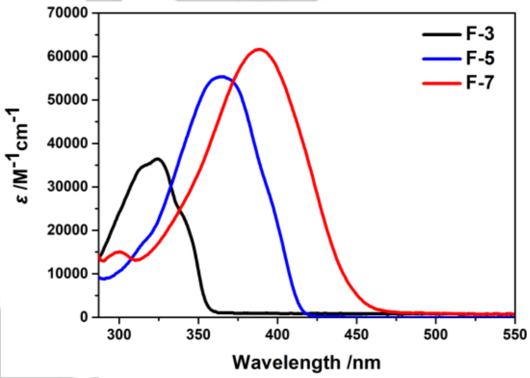


Figure 2. Room-temperature UV-vis absorption spectra of **F-3** (black), **F-5** (blue) and **F-7** (red) in dilute toluene (1.0 × 10^{-5} M).

The single-crystalline micro-plates/ribbons of oligomers **B-5/7**, **F-5/7** were prepared by physical vapor transport (PVT) method under an argon atmosphere with optimized temperature and flow rates onto the *n*-octadecyltrichlorosilane (OTS) modified SiO$_2$/Si substrates. Optical microscope (OM) images of the obtained micro-plates/ribbons with excellent long-range regularity are presented in Figure S4. Well-aligned hexagonal crystals were obtained for **B-5/7** and ribbon-like crystals were obtained for **F-5/7** on the micron scale. The change in crystal intensities in the cross-polarized optical microscopy (CPOM) images indicated the single crystal nature of the micro-plates/ribbons (Figure S5-S6).[14] The atomic force microscopy (AFM) images in Figure S7 reveal that all the crystals of the oligomers with thickness of 10-100 nm have very smooth surface for good interface contact with electrodes in OFET devices.

The out of plane X-ray diffraction patterns (XRD) with strong and sharp multiple Bragg diffractions in Figure S8 indicate high degree crystalline structures and highly ordered lamellar structure of micro-plates/ribbons of both oligomers **B-5/7** and **F-5/7**. Five strong diffraction peaks at $2\theta = 4.06°$, 8.09°, 12.14°, 20.26°, 28.48° are observed in the XRD analysis for **F-7** (Figure S8d), which could be assigned as [002], [004], [006], [0010] and [0014] lattice plane, respectively, based on the information from the single-crystal structure. All the peaks in this pattern are well indexed along the [001] crystal orientation, suggesting the molecules stand up along the *c*-axis with a tilt angle of ~51.2° on the substrate. The diffraction peaks in the XRD profiles of **F-5** (Figure

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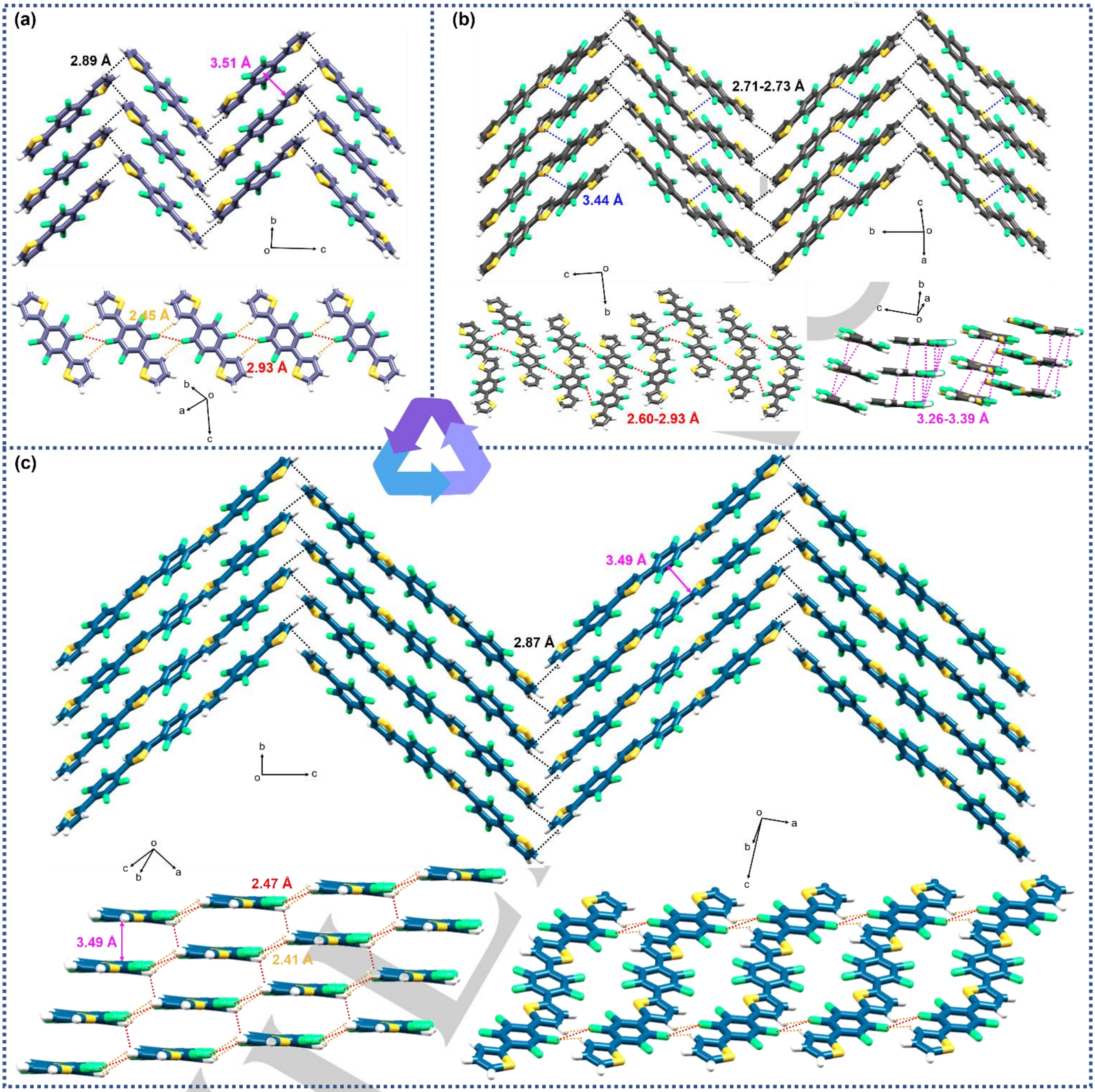

Figure 3. Single crystal structures and packing arrangements of **F-3** (a), **F-5** (b), and **F-7** (c). **F-3** and **F-7** molecules pack along *b*-axis, while **F-5** packs along *a*-axis. The purple/dark gray/cyanine -, yellow-, green-, and white-colored atoms represent C, S, F, and H, respectively. The dotted lines represent the intermolecular interactions (black: C-H...π interactions; magenta: π-π interactions; red: F...F interactions; orange: C-H...F interactions; blue: S...π interactions).

S8c) can also be indexed on basis of the prior single-crystal X-ray analysis.

Bright field transmission electron microscopic (TEM) observation of the micro-plates/ribbons of oligomers **B-5/7**, **F-5/7** (Figure S9 and Figure 4) reveal that the micro-plates/ribbons exhibit long-range regularity. The corresponding selected area electron diffraction (SAED) patterns show ordered and bright diffractions of the micro-plates/ribbons at different places, indicating the single-crystal nature of these plates/ribbons. The SAED diffraction patterns of **F-7** are equal to a repeating period of 5.1 Å along the fastest growth direction and a repeating period of 6.5 Å along the vertical direction, which can be assigned as the [010] direction and [100] direction according to the prior single-crystal X-ray analysis. The out-of-plane XRD data together with

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SAED pattern and single-crystal XRD data reveal that the micro-ribbons possess the same phase with previously obtained single crystals and the fastest growth direction of the micro-ribbons is along π-π stacking direction ([010] direction). The above analysis suggests that the **F-7** molecules in the single-crystalline micro-ribbons are nearly oriented with a specific tilt angle to the substrate and the π-π stacking direction is parallel to the substrate. The fastest growth direction of **F-5** micro-ribbons can also be assigned as the molecules π-π stacking direction ([100] direction) based on the SAED analysis. Unexpectedly, different from the **F-7** micro-ribbons, vertical direction of the **F-5** micro-ribbons is indexed as [010] direction, which was the long axis direction of the **F-5** molecules. Together with the out-of-plane XRD data, **F-5** molecules exhibited a different packing motif from **F-7** molecules, in which both the long axis direction of the **F-5** molecules and the π-π stacking direction are parallel to the substrate.

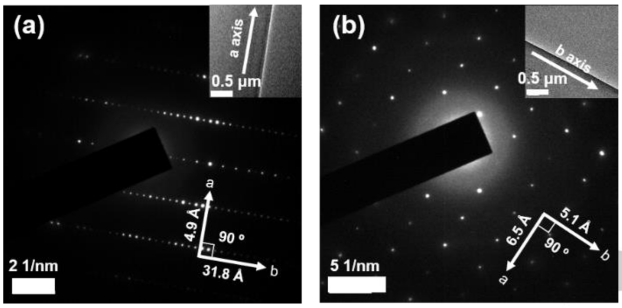

Figure 4. The corresponding SAED patterns of the single-crystalline micro-ribbons of **F-5** (a) and **F-7** (b). The insets show their TEM images.

Finally, we evaluated the intrinsic charge transport properties of the oligomeric micro-plates/ribbons. The field-effect transistors were fabricated by a "gold trips sticking" technique[15] on the OTS treated substrate with Au as the electrodes. Figure S10 and Figure 5 show the output and transfer characteristics of **B-5/7** micro-plates, and **F-5/7** micro-ribbons tested under ambient conditions. The average/maximum hole mobilities of these oligomers and corresponding important characters including width/length ratio of the devices, on/off ratio (I_{on}/I_{off}) and threshold voltage (V_{th}) are summarized in Table 1. OFET devices based on **B-7** show an average mobility of 0.032 cm^2 V^{-1} s^{-1} and a maximum mobility of 0.058 cm^2 V^{-1} s^{-1}. The **F-7** devices exhibit an average mobility of 1.31 cm^2 V^{-1} s^{-1} and a maximum mobility of 2.44 cm^2 V^{-1} s^{-1}, which is nearly two orders of magnitude higher than those of **B-7** devices. We also observe that the mobility of **F-5** is 2 grades higher than that of **B-5**. This result can be attributed to the fluorination of the backbone, which introduce an intense intermolecular interaction for compact stacks. In addition, **F-7** devices show one order of magnitude higher than those of **F-5** devices, which not only attributed to the larger extended D-A structure, but also to the more planar and ordered molecular backbone in **F-7**.

Table 1. OFET device parameters based on single crystalline transistors for **B-5/7** and **F-5/7**.

	W/L [μm]	V_{th} [V]	I_{on}/I_{off}	μ_{ave} [cm^2 V^{-1} s^{-1}] [a]	μ_{max} [cm^2 V^{-1} s^{-1}]
B-5	17.4/18.9	-15.4	2.2×10^7	0.0047	0.0062
B-7	24.1/26.9	-10.4	1.3×10^6	0.032	0.058
F-5	2.88/13.5	-6.7	7.2×10^4	0.27	0.57
F-7	1.36/17.3	-20.8	1.2×10^6	1.31	2.44

[a] Each based on more than 20 devices.

In conclusion, we have presented an effective way for excellently performing materials by designing simple D-A oligomeric systems. The oligomeric molecules (**F-3**, **F-5**, and **F-7**) were designed by integrating thiophene (T) and tetrafluorobenzene (F4B) alternatively units through direct arylation strategy. The absorption spectra of the three homogeneous oligomers showed bathochromic shift with the increasing number of units as a reflection of extended conjugation length of the D-A structure, and their HOMO level values indicated good environmental stability as electronic materials. Single crystal analysis revealed their herringbone packing arrangements driven by intensive C-H...π interactions. In comparison with fluorine-free oligomers (**B-5** and **B-7**), hole transporting efficiency based on single-crystalline micro-plates/ribbons was witnessed that larger π-conjugation and D-A constitution give higher mobilities. Consequently, an average mobility of 1.31 cm^2 V^{-1} s^{-1} and a maximum mobility of 2.44 cm^2 V^{-1} s^{-1} for **F-7** were achieved.

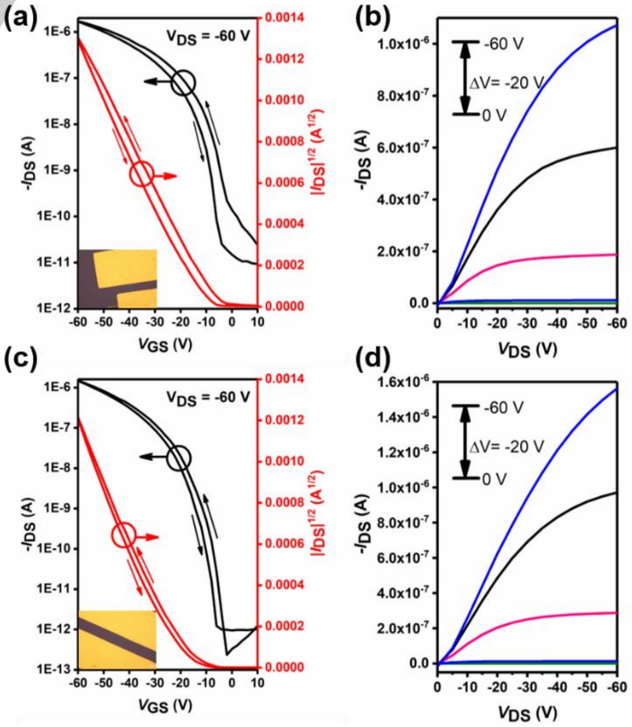

Figure 5. Typical transfer (a, c) and output curves (b, d) of representative single crystalline OFET devices of **F-5** (top) and **F-7** (bottom). The insets in (a) and (c) show the OM images of the corresponding single-crystalline OFET devices.

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Keywords: direct arylation • donor-acceptor systems • oligomeric molecules • crystal engineering • organic electronics

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Layout 1:

COMMUNICATION

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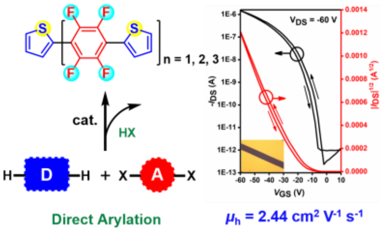

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