Supporting information

Tetracyano isoindigo small molecules and their use in n-channel organic field-effect transistors

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Contents

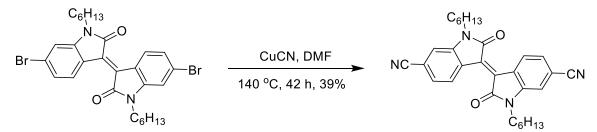
1.	Materials and General Methods	S2
2.	Synthesis Details	S3-S7
3.	OFET Device Fabrication and Characterisation	S 8
4.	Molecular Structures from Density Functional Theory Calculations	S9
5.	Cyclic Voltammetry and Organic Field-effect Transistor Figures	S10-S14
6.	Table of OFET properties of 7	S15
7.	References	S15

1. Materials and General Methods

General: All chemicals were purchased from commercial sources and used as received, unless stated otherwise. Synthesis of N,N'-Dihexyl-6,6'-dibromoisoindigo,¹ 5,6-dibromooxindole,² 5,6dibromoisatin³ and *N*-hexyl-3-aminothiophene⁴ were carried out according to modified literature procedures. Column chromatography was carried out using silica gel (46-63 µm, Sorbent) as the stationary phase, and thin-layer chromatography (TLC) was performed on precoated silica-gel plates (0.25 mm thick, 60F254, EMD, Germany) and visualised under UV light. The ¹H and ${}^{3}C{}^{1}H$ NMR spectra were measured on Varian Mercury 300 MHz and 400 MHz spectrometers. The chemical shift values were referenced with the solvent residual proton signal or the solvent carbon signal as internal standard. Mass spectra were measured on an Applied Biosystems 4700 Proteomics Analyzer using MALDI. Elemental analyses were carried out by Atlantic Microlabs using a LECO 932 CHNS elemental analyser. Electrochemical measurements were carried out under nitrogen in dry deoxygenated 0.1 M tetra-n-butylammonium hexafluorophosphate in dry tetrahydrofuran using a CH Instruments CHI620D Electrochemical Workstation CHI620D and a conventional three-electrode cell with a glassy carbon working electrode, platinum wire counter electrode, and an Ag wire coated with AgCl as the pseudoreference electrode. Potentials were referenced to ferrocenium/ferrocene by using internal ferrocene. Cyclic voltammograms were recorded at a scan rate of 50 mV s⁻¹.

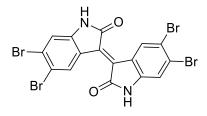
2. Synthesis Details

Synthesis of *N*,*N*'-dihexyl-6,6'-dicyanoisoindigo (1)



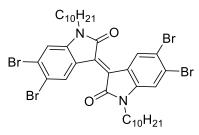
Copper cyanide (0.55 g, 6.10 mmol) was added to *N*,*N*'-dihexyl-6,6'-dibromoisoindigo (0.45 g, 0.76 mmol) in anhydrous DMF (15 mL) under nitrogen. The reaction mixture was stirred at 140 °C for 42 h. The reaction mixture was dropped into methanol (50 mL) and the precipitate was collected by filtration. The crude product was purified by column chromatography (silica, chloroform) to yield **1** as a purple powder (0.18 g, 39%). ¹H NMR (CDCl₃, 400 MHz) δ 9.31 (d, *J* = 8.4 Hz, 2H), 7.14 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.00 (d, *J* = 1.2 Hz, 2H), 3.76 (t, *J* = 7.4 Hz, 4H), 1.69-1.66 (m, 4H), 1.39-1.29 (m, 12H), 0.87 (t, *J* = 7.0 Hz, 6H). ¹³C{¹H} NMR (CDCl₃, 100 MHz) δ 166.8, 145.2, 134.3, 130.9, 126.5, 124.9, 118.5, 115.5, 110.5, 40.5, 31.4, 27.2, 27.6, 22.5, 14.0. HRMS (EI) calcd for C₃₀H₃₂N₄O₂ (M⁺), 480.2525; found 480.2528. Anal. Calcd for C₃₀H₃₂N₄O₂: C, 74.97; H, 6.71; N, 11.66. Found: C, 75.13; H, 6.99; N, 11.46.

Synthesis of 5,5',6,6'-tetrabromoisoindigo (10)



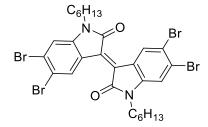
To a suspension of 6-bromooxindole (0.95 g, 3.29 mmol) and 6-bromoisatin (1.0 g, 3.3 mmol) in acetic acid (70 mL), conc. HCl (0.2 ml) was added. The mixture was heated at reflux for 24 h and then allowed to cool to room temperature; the resultant dark green precipitate was collected by filtration and washed with water, ethanol and ethyl acetate. The crude product was purified *via* recrystallisation from DMF (1.40 g, 81%). ¹H NMR (300 MHz, DMSO-*d*₆) δ 10.97 (s, 2H), 9.45 (s, 2H), 7.16 (s, 2H). ¹³C (¹H) NMR (DMSO-*d*₆, 75 MHz, 80 °C) δ 167.9, 143.9, 132.9, 127.0, 121.9, 114.5, 113.7 (one aromatic carbon resonance was not observed due to overlap). HRMS (EI) calcd for C₁₆H₆Br₄N₂O₂ (M⁺), 573.7163; found 573.7174. Anal. Calcd. for C₁₆H₆Br₄N₂O₂: C, 33.26; H, 1.05; N, 4.85. Found: C, 33.53; H, 1.22; N, 4.82.

Synthesis of *N*,*N*'-didecyl-5,5',6,6'-tetrabromoisoindigo (2)



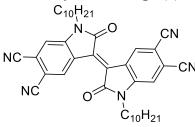
To a suspension of 5,5',6,6'-tetrabromoisoindigo (1.00 g, 1.74 mmol) and K₂CO₃ (1.20 g, 8.71 mmol) in anhydrous DMF (25 mL), 1-bromodecane (1.45 mL, 6.96 mmol) was added under nitrogen. The reaction mixture was stirred at reflux for 20 min. After cooling to room temperature, the reaction mixture was poured into water and a dark brown precipitate was collected by filtration. The precipitate was washed with hexanes and the crude product was purified by recrystallization from CH₂Cl₂, to yield **2** as a brown solid (1.07 g, 72%). ¹H NMR (300 MHz, 1,2-dichlorobenzene-*d*₄, 80 °C) δ 9.91 (s, 2H), 7.16 (s, 2H), 3.82 (t, *J* = 7.4 Hz, 4H), 1.82 (quint, *J* = 4.4 Hz, 4H), 1.52-1.41 (m, 28H), 1.05 (t, *J* = 6.6 Hz, 6H). ¹³C{¹H} NMR (75 MHz, 1,2-dichlorobenzene-d₄, 80 °C) δ 167.1, 144.9, 134.9, 129.0, 125.2, 122.2, 117.2, 112.6, 40.5, 32.0, 29.6, 29.4, 29.3, 27.5, 27.1, 22.8, 14.1 (one aliphatic carbon resonance was not observed due to overlap). HRMS (MALDI) calcd for C₃₆H₄₇Br₄N₂O₂ (MH⁺), 855.0371; found, 855.0330. Anal. Calcd. for C₃₆H₄₆Br₄N₂O₂: C, 50.37; H, 5.40; N, 3.26. Found: C, 50.56; H, 5.42; N, 3.30.

Synthesis of *N*,*N*'-dihexyl-5,5',6,6'-tetrabromoisoindigo (3)



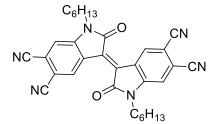
To a suspension of 5,5',6,6'-tetrabromoisoindigo (0.60 g, 1.04 mmol) and K₂CO₃ (0.72 g, 5.22 mmol) in anhydrous DMF (20 mL), 1-bromohexane (0.59 mL, 4.21 mmol) was added under nitrogen. The reaction mixture was stirred at reflux for 20 min. After cooling to room temperature, the reaction mixture was poured into water and a dark brown precipitate was collected by filtration, washed with hexanes, and purified by recrystallization from CH₂Cl₂ to yield **3** as a brown solid (0.69 g, 74%). ¹H NMR (300 MHz, 1,2-dichlorobenzene-*d*₄, 80 °C) δ 9.95 (s, 2H), 7.15 (s, 2H), 3.81 (t, *J* = 7.5 Hz, 4H), 1.79 (quint, *J* = 4.5 Hz, 4H), 1.51-1.41 (m, 12H), 1.02 (t, *J* = 6.9 Hz, 6H). ¹³C{¹H} NMR (75 MHz, 1,2-dichlorobenzene-*d*₄, 80 °C) δ 167.2, 144.9, 134.9, 132.3, 129.0, 122.2, 117.8, 112.6, 40.5, 31.5, 27.5, 26.8, 22.6, 13.9. HRMS (EI) calcd for C₂₈H₃₁Br₄N₂O₂ (MH⁺), 742.9119; found, 742.9132. Anal. Calcd. for C₂₈H₃₀Br₄N₂O₂: C, 45.07; H, 4.05; N, 3.75. Found: C, 45.10; H, 4.11; N, 3.79.

Synthesis of N,N'-didecyl-5,5',6,6'-tetracyanoisoindigo (4)



A 20 mL microwave tube was charged with copper(I) cyanide (0.88 g, 9.83 mmol) and **2** (0.70 g, 0.82 mmol). Under nitrogen, anhydrous DMF (8.0 mL) was added and heated at 180 °C for 1 h in a microwave reactor (Biotage). The reaction mixture was dropped into methanol (50 mL) and a brown precipitate was collected by filtration. The crude product was dissolved in hot chloroform; the solution was filtered to remove copper salts and the dark-coloured filtrate was evaporated under reduced pressure. The crude product was purified by column chromatography (silica, CH₂Cl₂) followed by a recrystallisation from ethyl acetate to afford the desired product as a brown solid (0.23 g, 44%). ¹H NMR (300 MHz, CDCl₃) δ 9.75 (s, 2H), 7.17 (s, 2H), 3.82 (t, *J* = 7.5 Hz, 4H), 1.69 (quint, *J* = 4.3 Hz, 4H), 1.42-1.31 (m, 34H), 0.85 (t, *J* = 6.6 Hz, 6H). ¹³C{¹H} NMR (75 MHz, CDCl₃, 60 °C) δ 166.4, 148.4, 135.3,133.6, 125.2, 124.1, 120.0, 115.4, 115.0, 112.2, 109.7, 41.5, 31.8, 29.4, 29.4, 29.2, 29.1, 27.3, 26.9 (two aliphatic carbon resonances were not observed due to overlap). HRMS (MALDI) calcd for C₄₀H₄₇N₆O₂ (MH⁺), 643.3761; found 643.3721. Anal. Calcd. for C₄₀H₄₆N₆O₂: C, 74.74; H, 7.21; N, 13.07. Found: C, 74.56; H, 7.34; N, 12.93.

Synthesis of N,N'-dihexyl-5,5',6,6'-tetracyanoisoindigo (5)



A 20 mL microwave tube was charged with copper(I) cyanide (0.72 g, 8.08 mmol) and *N*,*N*'-dihexyl-5,5',6,6'-tetrabromoisoindigo (0.50 g, 0.67 mmol). Under nitrogen, anhydrous DMF (8.5 mL) was added and heated at 160 °C for 1 h in a microwave reactor (CEM Corp.). The reaction mixture was dropped into methanol (100 mL) and a brown precipitate was collected by filtration. The crude product was dissolved in hot chloroform; the solution was filtered to remove copper salts and the dark-coloured filtrate was evaporated under reduced pressure. The crude product was purified by column chromatography (silica, CH₂Cl₂) followed by a recrystallisation from ethyl acetate afforded the desired product as a brown solid (0.15 g, 42%). ¹H NMR (300 MHz, CDCl₃) δ 9.80 (s, 2H), 7.16 (s, 2H), 3.83 (t, *J* = 6.8 Hz, 4H), 1.7 (quint, *J* = 4.4 Hz, 4H), 1.45-1.27 (m, 12H), 0.89 (t, *J* = 6.8 Hz, 6H). ¹³C{¹H} NMR (75 MHz, CDCl₃, 60 °C) δ 166.5, 148.4, 135.3, 133.7, 124.2, 119.9, 115.5, 115.1, 112.2, 109.7, 41.2, 31.3, 27.2, 26.5, 22.4, 13.8. HRMS

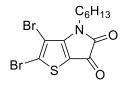
(EI) calcd for $C_{32}H_{30}N_6O_2$, (M⁺) 530.2430; found, 530.2413. Anal. Calcd. for $C_{32}H_{30}N_6O_2$: C, 72.43; H, 5.70; N, 15.84. Found: C, 72.70; H, 5.87; N, 15.61.

Synthesis of 4-hexyl-4*H*-thieno[3,2-*b*]pyrrole-5,6-dione (11)



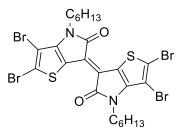
To a solution of dichloromethane (55 mL) and oxalyl chloride (8.32 mL, 65.6 mmol), was added dropwise a solution of *N*-hexyl thiophen-3-amine (10.0 g, 54.6 mmol) in dichloromethane (65 mL) at 0 °C. After 30 min stirring at 0 °C, a solution of triethylamine (34 mL) in dichloromethane (35 mL) was added dropwise over a period of 2h, after which the reaction was allowed to warm to room temperature and stirred for 12 h. The reaction was quenched by the slow addition of water (20 mL); the mixture was then extracted with dichloromethane (300 mL). The solvent was removed from the organic extracts under reduced pressure and the residue was passed through a silica plug (CH₂Cl₂). The crude product was further purified by column chromatography (silica, 50:50 v/v CH₂Cl₂/hexanes) to yield **11** as an orange solid (7.07 g, 63%). ¹H NMR (300 MHz, CDCl₃) δ 8.01 (d, *J* = 3 Hz, 1H), 6.79 (d, *J* = 6 Hz, 1H), 3.60 (t, *J* = 6 Hz, 2H), 1.60 (quint, *J* = 6.7 Hz, 2H), 1.35-1.18 (m, 6H), 0.80 (t, *J* = 6 Hz, 3H). ¹³C{¹H} NMR (75 MHz, CDCl₃) δ 173.0, 165.2, 161.5, 144.4, 113.2, 110.8, 42.0, 31.2, 27.5, 26.3, 22.4, 13.9. HRMS (EI) m/z calcd for C₁₂H₁₅NO₂S (M⁺), 238.0896; found, 238.0894. Anal. Calcd. for C₁₂H₁₅NO₂S: C, 60.73; H, 6.37; N, 5.90; found: C, 60.90; H, 6.25; N, 5.84.

Synthesis of 2,3-dibromo-4-hexyl-4*H*-thieno[3,2-*b*]pyrrole-5,6-dione (12)

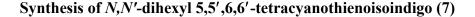


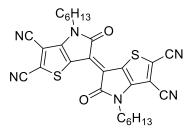
Bromine (45.9 g, 287 mmol, 20 eq.) was added to 4-hexyl-4*H*-thieno[3,2-*b*]pyrrole-5,6-dione (3.40 g, 14.3 mmol) at room temperature and the resulting mixture stirred for 20 min. Saturated aqueous Na₂S₂O₃ solution was then added at 0 °C and the mixture was extracted into CH₂Cl₂. Solvent was removed from the organic extracts under reduced pressure and the crude product was purified by column chromatography (silica, 40:60 v/v CH₂Cl₂/hexanes) to give **12** as an orange solid (6.63 g, 58%). ¹H NMR (300 MHz, CDCl₃) δ 3.81 (t, *J* = 9 Hz, 2H), 1.61 (quint, *J* = 7.5 Hz, 2H), 1.34-1.19 (m, 6H), 0.79 (t, *J* = 6 Hz, 3H). ¹³C{¹H} NMR (75 MHz, CDCl₃.) δ 171.1, 160.3, 161.1, 134.2, 111.2, 102.1, 40.8, 31.3, 29.5, 26.1, 22.4, 14.0. HRMS (EI) calcd for C₁₂H₁₃NO₂S (M⁺), 393.9103; found, 393.9104. Anal. Calcd. for C₁₂H₁₃NO₂S: C, 36.48; H, 3.32; N, 3.55; found: C, 36.51; H, 3.36; N, 3.48.

Synthesis of *N*,*N*'-dihexyl 5,5',6,6'-tetrabromothienoisoindigo (6)



Lawesson's reagent (1.08 g, 2.67 mmol) and 2,3-dibromo-4-hexyl-4*H*-thieno[3,2-*b*]pyrrole-5,6dione (2.10 g, 5.34 mmol) was combined under nitrogen in dry toluene (7.5 mL). The reaction mixture was heated at 65 °C for 20 min. After cooling, reaction mixture was passed through a silica plug (50:50 v/v CH₂Cl₂/hexanes). Solvent was removed under reduced pressure and the crude product was purified multiple times by column chromatography (silica, 30:70 v/v CH₂Cl₂/hexanes), and then further purified by recrystallisation from ethyl acetate to give a blue solid (1.03 g, 25%). ¹H NMR (300 MHz, CDCl₃) δ 3.97 (t, *J* = 7.5 Hz, 4H), 1.71 (quint, *J* = 4.6 Hz, 4H), 1.45-1.25 (m, 12H), 0.88 (t, *J* = 6.6 Hz, 6H). ¹³C{¹H} NMR (75 MHz, CDCl₃, 60 °C) δ 170.3, 146.6, 123.2, 119.8,115.2, 100.1, 40.9, 31.5, 29.9, 26.3, 22.5, 13.9. HRMS (EI) calcd for C₂₄H₂₆Br₄N₂O₂S₂ (M⁺), 753.8164; found, 753.8155. Anal. Calcd. for C₂₄H₂₆Br₄N₂O₂S₂: C, 38.02; H, 3.46; N, 3.69; found: C, 38.28; H, 3.63; N, 3.64.





A 10 mL microwave tube was charged with copper(I) cyanide (0.20 g, 2.21 mmol) and **6** (0.21 g, 0.27 mmol). Under nitrogen, anyhydrous DMF (4.1 mL) was added and the mixture was heated at 180 °C for 1 h in a microwave reactor (Biotage). The reaction mixture was dropped into water (100 mL) and a dark purple precipitate was collected by filtration. The crude product was dissolved in hot chloroform; the solution was filtered to remove copper salts and the dark-coloured filtrate was evaporated under reduced pressure. The crude product was purified by column chromatography (silica, 1.5:98.5 v/v ethylacetate/chloroform) to yield a purple solid (0.05 g, 36%). ¹H NMR (300 MHz, CDCl₃) δ 4.00 (t, J = 7.2 Hz, 4H), 1.75 (quint, J = 7.3 Hz, 4H), 1.53-1.31 (m, 12H), 0.87 (t, J = 5.7 Hz, 6H). ¹³C{¹H} NMR (75 MHz, CDCl₃) δ 169.0, 150.6, 123.3, 122.3, 119.2, 111.2, 109.9, 102.4, 42.0, 31.2, 29.0, 26.1, 22.4, 13.8. HRMS (EI) calcd for C₂₈H₂₆N₆O₂S₂ (M⁺), 542.1559; found, 542.1571. Anal. Calcd. for C₂₈H₂₆N₆O₂S₂: C, 61.97; H, 4.83; N, 15.49. Found: C, 62.01; H, 4.96; N, 15.44.

3. OFET device fabrication and characterisation

Device fabrication

OFETs with top-gate and bottom-contact geometry were fabricated on glass (Corning® Eagle^{2000TM}) and flexible polyethersulfone (PES, i-components) substrates. For spin-coated OFETs, Au bottom contact source / drain (S/D) electrodes were deposited on glass substrates by e-beam evaporation through a shadow mask (channel length and width are 180 and 2550 µm, respectively). Organic semiconductor layers were formed on the substrates by spin-coating with a solution prepared from solutions in 1,1,2,2-tetrachloroethane, TCE, (12 mg m L^{-1}) at 500 rpm for 10 s and at 2000 rpm for 20 s in a N₂-filled dry box. Samples were then annealed at 110-130 °C. For printed OFETs, Ag electrodes were printed with a Dimatix DMP 2831 inkjet printing system using Cabot Conductive Ink 300 (CCI-300), a commercial dispersion of silver nano-particles from Cabot Corporation. Liquid-crystal polymer (LCP) grade cartridges were used because of their resistance to more aggressive solvents. Ag bottom contact source / drain (S/D) electrodes were deposited on glass and polyethersulfone (PES) substrates by e-beam evaporation through a shadow mask (channel lengths and width are 40-200 and 2000 µm, respectively). The semiconductor ink was formulated with DCB (5 mg mL⁻¹) in ambient conditions. The semiconductor material was printed in air. The drop spacing and platen temperature were optimized as 15 µm and 45 °C, respectively. Printed samples were annealed in air for 10 min on a hot plate at 110-130 °C. A CYTOP (45 nm)/Al₂O₃ (50 nm) bi-layer was used as top-gate dielectric for both spin-coated and printed OFETs. The CYTOP solution (CTL-809M, 9 wt.%) was purchased from Asahi Glass, and diluted with solvent (CT-solv.180) to have a solution:solvent volume ratio of 1:3.5. The 45 nm thick CYTOP layers were then deposited by spin coating at 3000 rpm for 60 s and annealed at 100 °C for 10 min. Al₂O₃ (50 nm) films were deposited on the top of CYTOP layers by atomic layer deposition (ALD) using a Cambridge NanoTech Savannah 100 system at 110 °C using alternating exposures of trimethyl aluminum and H₂O vapor at a deposition rate of approximately 0.1 nm per cycle. Finally, Ag top gate electrodes were deposited by thermal evaporation through a shadow mask.

Device characterisation

The electrical characterisation of the OFETs was carried out in a N₂-filled glove box (O₂, $H_2O < 0.1$ ppm). Current-voltage (*I-V*) characteristics of OFETs were measured with an Agilent E5272A source/monitor unit.

4. Molecular structures from density functional theory calculations

Density functional theory (DFT) calculations were carried out with Spartan 10 program. The (gas-phase) neutral and anion ground-state electronic structures were obtained from DFT calculations using B3LYP functional and 6-31G** basis set. Alkyl groups on the tetracyano substituted isoindigo and thienoisoindigo units were replaced by methyl groups to reduce the amount of time for calculations. The LUMO and HOMO energies for tetracyano isoindigo are - 4.2 and -7.0 eV and for tetracyano thienoisoindigo are -4.2 and -6.5 eV, respectively.

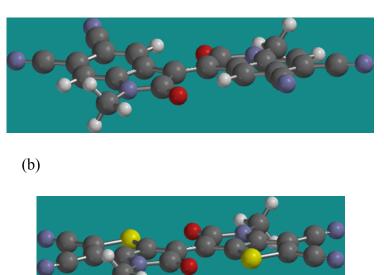




Fig. S1 Molecular structures of (a) N,N'-dimethyl-5,5',6,6'-tetracyanoisoindigo and (b) N,N'-dimethyl-5,5',6,6'-tetracyanothienoisoindigo from density functional theory calculations.

5. Cyclic voltammetry traces and OFET device figures

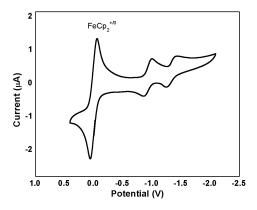


Fig. S2 Cyclic voltammetric trace of *N*,*N'*-dihexyl-6,6'-dicyanoisoindigo (1). The feature at $E_{1/2} = 0$ V is due to the internal ferrocene (FeCp₂) reference.

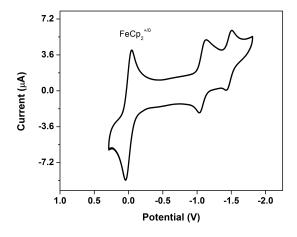


Fig. S3 Cyclic voltammetric trace of *N*,*N'*-didecyl-5,5',6,6'-tetrabromoisoindigo (2). The feature at $E_{1/2} = 0$ V is due to the internal ferrocene (FeCp₂) reference.

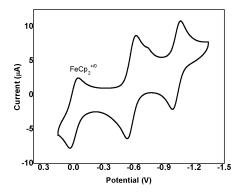


Fig. S4 Cyclic voltammetric trace of *N*,*N'*-didecyl-5,5',6,6'-tetracyanoisoindigo (4). The feature at $E_{1/2} = 0$ V is due to the internal ferrocene (FeCp₂) reference.

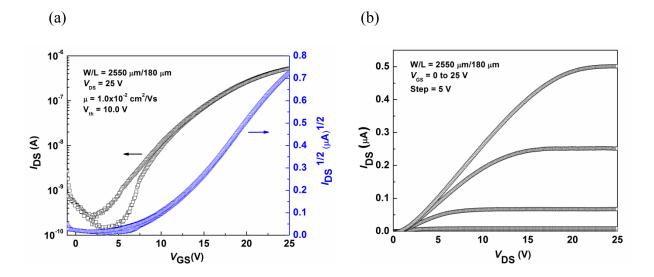


Fig. S5 Representative (a) transfer and (b) output charcterstics of spin-coated OFETs of 1.

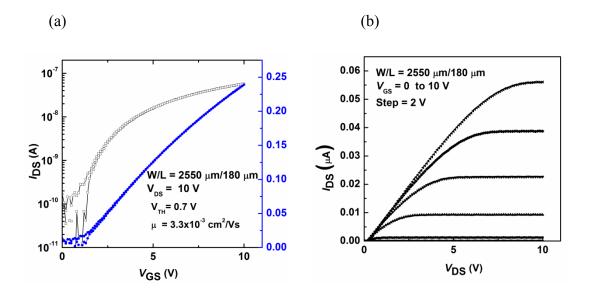


Fig. S6 Representative (a) transfer and (b) output charcterstics of spin-coated OFETs of 4.

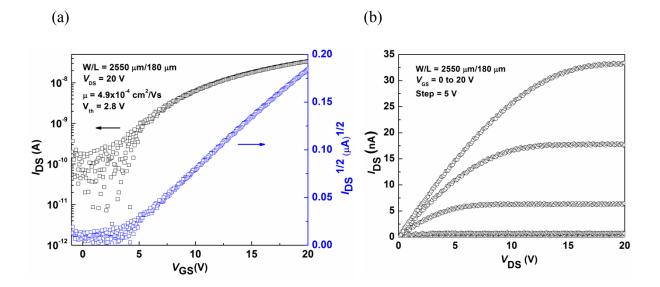


Fig. S7 Representative (a) transfer and (b) output charcterstics of spin-coated OFETs of 5.

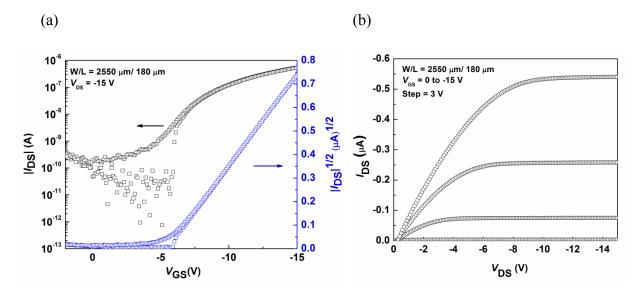


Fig. S8 Representative (a) transfer and (b) output charcterstics of spin-coated OFETs of 6.

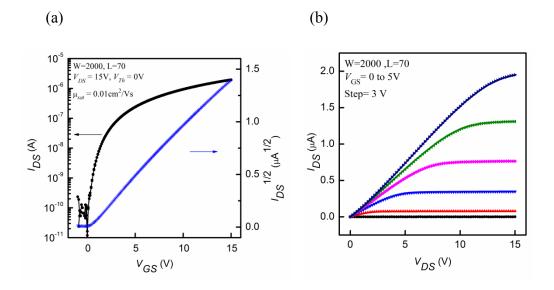


Fig. S9 Representative (a) transfer and (b) output charcterstics of inkjet-printed OFETs of 4.

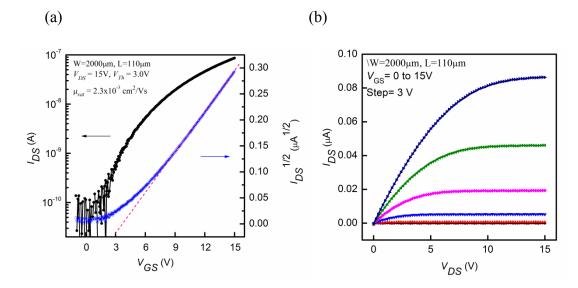


Fig. S10 Representative (a) transfer and (b) output charcterstics of inkjet-printed OFETs of 5.

Device ^a	W/L	S/D	$\mu_e / cm^2 V^{-1} s^{-1}$	$V_{ m Th}$ / V	$I_{ m on}/I_{ m off}$
measurements		electrode			
In N ₂	2550 mm/180 mm	Au	4.3×10^{-2}	0.6	1.5×10^{3}
	Best device				
In air 2 days	2550 mm/180 mm	Au	3.8×10^{-2}	1.3	$4.5 imes 10^2$
	Best device				
In air 7 days	2550 mm/180 mm	Au	3.5×10^{-2}	1.4	$5.9 imes 10^2$
	Best device				
In N ₂	2550 mm/180 mm	Au	$3.0(\pm 1.1) \times 10^{-2}$	0.5 (±0.1)	$8.7 imes 10^2$
	Average (3 devices)				
In air 2 days	2550 mm/180 mm	Au	$2.6 (\pm 1.0) \times 10^{-2}$	1.1 (±0.2)	3.2×10^{2}
	Average (3 devices)				
In air 7 days	2550 mm/180 mm	Au	$2.5 (\pm 0.9) \times 10^{-2}$	1.2 (±0.2)	4.1×10^2
	Average (3 devices)				

 Table S1. OFET properties of 7 measured in air and nitrogen.

^a Capacitance, $C_{\rm in} = 35.2 \text{ nF cm}^{-2}$.

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