Supporting Information

# A heptacyclic carbon-oxygen-bridged ladder-type building block for A-D-A acceptors

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#### 1. General characterization

<sup>1</sup>H and <sup>13</sup>C NMR spectra were measured on a Bruker Avance-400 spectrometer. Absorption spectra were recorded on a Shimadzu UV-1800 spectrophotometer. Cyclic voltammetry was done by using a Shanghai Chenhua CHI620D voltammetric analyzer under argon in an anhydrous acetonitrile solution of tetra-n-butylammonium hexafluorophosphate (0.1 M). A glassy-carbon electrode was used as the working electrode, a platinum-wire was used as the counter electrode, and a Ag/Ag<sup>+</sup> electrode was used as the reference electrode. The donor or acceptor was coated onto glassy-carbon electrode and all potentials were corrected against  $Fc/Fc^+$ . AFM was performed on a Multimode microscope (Veeco) using tapping mode.

## 2. Synthesis

All reagents were purchased from J&K Co., Aladdin Co., Innochem Co., Derthon Co., SunaTech Co. and other commercial suppliers. All reactions dealing with air- or moisture-sensitive compounds were carried out by using standard Schlenk techniques. 2,2'-(2,5-Dimethoxy-1,4-phenylene)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane)<sup>[1]</sup> and ethylhexyl 2-bromothieno[3,2-b]thiophene-3-carboxylate<sup>[2]</sup> were prepared according to literature.



Scheme S1 The synthetic routes for CO<sub>i</sub>7IC and CO<sub>i</sub>7DFIC.

**Compound 1.** To a solution of 2,2'-(2,5-dimethoxy-1,4-phenylene)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (500 mg, 1.2 mmol) and ethylhexyl 2-bromothieno[3,2-b]thiophene-3-carboxylate (1.06 g, 2.6 mmol) and in DMF (20 mL) were added Pd(PPh<sub>3</sub>)<sub>4</sub> (148 mg, 0.1 mmol) and K<sub>3</sub>PO<sub>4</sub> (1.36 g, 6.4 mmol) under argon. The mixture was heated to reflux for 12 h. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using CHCl<sub>3</sub> as eluent to give **compound 1** as a yellow solid (652 mg, 73%). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz,  $\delta$ /ppm): 7.47 (d, *J* = 5.3 Hz, 2H), 7.28 (d, *J* = 5.3 Hz, 2H), 6.97 (s, 2H), 4.17 (qd, *J* = 11.0, 5.2 Hz, 4H), 3.73 (s, 6H) 1.51-1.53 (m, 2H), 1.25-1.27 (m, 16H), 0.83-0.89 (m, 12H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz,  $\delta$ /ppm): 162.51, 150.63, 147.34, 139.93, 136.59, 128.44, 124.42, 123.08, 118.80, 114.53, 66.84, 56.18, 38.69, 30.27, 28.93, 23.62, 22.93, 14.06, 11.03. MALDI-TOF MS (m/z): 748.9 (M + Na<sup>+</sup>).

**Compound 2.** To a solution of compound 1 (500 mg, 0.72 mmol) in dry  $CH_2Cl_2$  (20 mL) was added BBr<sub>3</sub> (3.59 mL, 1.75 M, 6.28 mmol) at room temperature under argon. The mixture was stirred for 3 h and was quenched by adding MeOH (50 mL). The precipitate was collected and dried under vacuum to give **compound 2** as a yellow solid (305 mg, 97%). NMR data were not acquired due to the extremely low solubility of compound 2.

CO<sub>*i*</sub>7. To compound 2 (300 mg, 0.68 mmol) was added (4-hexylphenyl)magnesium bromide (12 mL, 0.6 M in THF, 7.2 mmol) at room temperature under argon. The mixture was stirred at 60 °C for 12 h. After cooling to room temperature, hydrochloric acid (3 mL, 36%) was added to the solution. The mixture was stirred at room temperature for 1 h and was poured into MeOH. The precipitate was collected and purified via column chromatography (silica gel) by using CHCl<sub>3</sub>:petroleum ether (1:2) as eluent to give CO<sub>*i*</sub>7 as a yellow solid (440 mg, 61%). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz,  $\delta$ /ppm): 7.09-7.19 (m, 20H), 6.97 (s, 2H), 2.56-2.60 (m, 8H), 1.55-1.64 (m, 8H), 1.28-1.33 (m, 24H), 0.87 (t, *J* = 6.7 Hz, 12H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz,  $\delta$ /ppm): 146.29, 143.04, 139.65, 139.57, 136.99, 134.46, 130.10, 128.34, 128.05, 127.96, 121.13, 119.23, 111.09, 85.63, 35.62, 31.69, 31.21, 28.98, 22.59, 14.08. MALDI-TOF MS (m/z): 1051.3 (M + H<sup>+</sup>).

**CO**<sub>*i*</sub>**7-CHO.** To a solution of CO<sub>*i*</sub>7 (392 mg, 0.37 mmol) in dry THF (20 mL) was added *n*-BuLi (2.33 mL, 1.6 M, 3.73 mmol) at -78 °C under argon. The resulting mixture was warmed to -50 °C and stirred for 1.5 h. To the mixture was added N,N-dimethylformamide (147  $\mu$ L, 1.9 mmol). The resulting mixture was stirred for 1 h and was then quenched by water

followed by extraction with CH<sub>2</sub>Cl<sub>2</sub> three times. The combined organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using CH<sub>2</sub>Cl<sub>2</sub>:petroleum ether (1:2) as eluent to give **CO**<sub>i</sub>**7**-**CHO** as an orange solid (304 mg, 74%). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz,  $\delta$ /ppm): 9.83 (s, 2H), 7.85 (s, 2H), 7.11-7.17 (m, 16H), 7.02 (s, 2H), 2.57-2.61 (m, 8H), 1.55-1.66 (m, 8H), 1.29-1.34 (m, 24H), 0.87 (t, *J* = 6.6 Hz, 12H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz,  $\delta$ /ppm): 182.89, 146.92, 145.97, 145.40, 143.62, 140.27, 138.70, 136.88, 130.69, 128.63, 128.31, 128.18, 121.65, 112.01, 85.69, 35.61, 31.67, 31.12, 28.99, 22.57, 14.08. MALDI-TOF MS (m/z): 1107.2 (M + H<sup>+</sup>).

CO<sub>i</sub>7IC. To a solution of CO<sub>i</sub>7-CHO (100 mg, 0.09 mmol) in CHCl<sub>3</sub> (10 mL) was added 2-(3-oxo-2,3-dihydro-1H-inden-1-ylidene)malononitrile (IC) (88 mg, 0.45 mmol) and pyridine (0.5 mL) at room temperature. The mixture was heated to reflux for 1 h. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using CHCl<sub>3</sub> as eluent to give CO<sub>i</sub>7IC as a black solid (124 mg, 95%). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz,  $\delta$ /ppm): 8.72 (s, 2H), 8.66 (d, J = 7.5 Hz, 2H), 8.17 (s, 2H), 7.85 (m, 2H), 7.74 (m, 4H), 7.18-7.25 (m, 16H), 7.06 (s, 2H), 2.61 (t, J = 7.7 Hz, 8H), 1.55-1.65 (m, 8H), 1.24-1.31 (m, 24H), 0.81 (t, J = 6.9 Hz, 12H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz,  $\delta$ /ppm): 187.34, 160.42, 152.57, 147.47, 143.75, 142.57, 140.46, 139.85, 138.65, 137.80, 136.83, 135.10, 135.04, 134.40, 131.48, 128.42, 128.29, 125.21, 123.79, 123.53, 122.32, 114.49, 114.38, 112.30, 85.83, 69.69, 35.67, 31.72, 31.24, 29.00, 22.57, 14.07. MALDI-TOF MS (m/z): 1460.5 (M + H<sup>+</sup>). Single crystals of CO<sub>i</sub>7IC were obtained by slowly diffusing methanol into its chloroform solution. Formula: C<sub>94</sub>H<sub>82</sub>N<sub>4</sub>O<sub>4</sub>S<sub>4</sub>; formula weight: 1459.87; crystal system: triclinic; space group: *P*-1; color of crystal: black; unit cell parameters: a = 10.952(2) Å, b = 13.127(3) Å, c = 13.687(3)Å,  $\alpha = 85.865(7)^{\circ}$ ,  $\beta = 80.518(7)^{\circ}$ ,  $\gamma = 80.36(3)^{\circ}$ , V = 1911.5(7) Å<sup>3</sup>; temperature for data collection: 100(2) K; Z = 1; final R indices [I > 2 $\sigma$ (I)]: RI = 0.0686, wR2 = 0.1436; GOF on F<sup>2</sup>: 1.155. The crystallographic data have been deposited in Cambridge Crystallographic Data Centre (CCDC-1837067).

**CO**<sub>*i*</sub>**7DFIC.** To a solution of CO<sub>*i*</sub>**7**-CHO (102 mg, 0.092 mmol) in CHCl<sub>3</sub> (10 mL) was added DFIC (94 mg, 0.46 mmol) and pyridine (0.5 mL) at room temperature. The mixture was heated to reflux for 1 h. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using CHCl<sub>3</sub> as eluent to give **CO**<sub>*i*</sub>**7DFIC** as a black solid (96 mg, 70%). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz,  $\delta$ /ppm): 8.72 (s, 2H), 8.51 (dd, *J* = 9.8, 6.4

Hz, 2H), 8.15 (s, 2H), 7.61 (t, J = 7.4 Hz, 2H), 7.18-7.23 (m, 16H), 7.06 (s, 2H), 2.61 (t, J = 7.7 Hz, 8H), 1.57-1.63 (m, 8H), 1.24-1.32 (m, 24H), 0.81-0.84 (m, 12H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz,  $\delta$ /ppm): 185.07, 158.25, 155.71, 153.27, 153.08, 147.58, 143.85, 143.32, 140.30, 138.66, 138.54, 138.10, 136.43, 135.77, 134.50, 131.56, 128.45, 128.27, 122.44, 115.00, 114.79, 114.09, 113.93, 112.72, 112.54, 112.43, 85.83, 70.23, 35.67, 31.73, 31.25, 29.01, 22.57, 14.05. MALDI-TOF MS (m/z): 1532.8 (M + H<sup>+</sup>).

**3. NMR** 



560560	58	$\begin{array}{c} 31\\ 28\\ 11\\ 88\\ 88\\ 85\\ 85\end{array}$	00
27.2	-i_	0.00	00

 $\begin{bmatrix} 7.26\\ 7.21\\ 7.19\\ 7.16\\ 7.16\\ 7.15\\ 7.12\\ 7.09\\ 6.97 \end{bmatrix}$ 





-9. 83	-7. 85 7. 26 7. 17 7. 15 7. 15 7. 11 7. 02	$\begin{array}{c} 2.55\\ -1.29\\ -1.29\\ 0.07\\ 0.08 \end{array}$	0.00
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72 551 551 551 15 15 61 61	22 22 11 22 06 06	$63 \\ 61 \\ 61 \\ 61 \\ 61 \\ 61 \\ 61 \\ 61 \\ $	32 26 24	81	.00
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Fig. S9 <sup>1</sup>H NMR spectrum of  $CO_i7DFIC$ .



4. UV-Vis



Fig. S11 Absorption spectra of CO<sub>i</sub>7IC and CO<sub>i</sub>7DFIC in solution and as films



Fig. S12 Cyclic voltammograms for PTB7-Th, CO<sub>i</sub>7IC and CO<sub>i</sub>7DFIC.



Fig. S13 Energy level diagram.

#### 6. Device fabrication and measurements

## **Inverted solar cells**

The ZnO precursor solution was prepared according to literature.<sup>[3]</sup> It was spin-coated onto ITO glass (4000 rpm for 30 s). The films were annealed at 200 °C in air for 30 min. ZnO film thickness is ~30 nm. A PTB7-Th:NFA blend in *o*-dichlorobenzene (ODCB) with DIO additive was spin-coated onto ZnO layer. MoO<sub>3</sub> (~6 nm) and Ag (~80 nm) was successively evaporated onto the active layer through a shadow mask (pressure ca.  $10^{-4}$  Pa). The effective area for the devices is 4 mm<sup>2</sup>. The thicknesses of the active layers were measured by using a KLA Tencor D-120 profilometer. *J-V* curves were measured by using a computerized Keithley 2400 SourceMeter and a Xenon-lamp-based solar simulator (Enli Tech, AM 1.5G, 100 mW/cm<sup>2</sup>). The illumination intensity of solar simulator was determined by using a monocrystalline silicon solar cell (Enli SRC2020,  $2\text{cm}\times2\text{cm}$ ) calibrated by NIM. The external quantum efficiency (EQE) spectra were measured by using a QE-R3011 measurement system (Enli Tech).

### **Hole-only devices**

The structure for hole-only devices is ITO/PEDOT:PSS/PTB7-Th:NFA/MoO<sub>3</sub>/Al. A 30 nm thick PEDOT:PSS layer was made by spin-coating an aqueous dispersion onto ITO glass (4000 rpm for 30 s). PEDOT substrates were dried at 150 °C for 10 min. A PTB7-Th:NFA blend in ODCB with DIO additive was spin-coated onto PEDOT layer. Finally, MoO<sub>3</sub> (~6 nm) and Al (~100 nm) was successively evaporated onto the active layer through a shadow mask (pressure ca.  $10^{-4}$  Pa). *J-V* curves were measured by using a computerized Keithley 2400 SourceMeter in the dark.

## **Electron-only devices**

The structure for electron-only devices is Al/PTB7-Th:NFA/Ca/Al. Al (~80 nm) was evaporated onto a glass substrate. A PTB7-Th:NFA blend in ODCB with DIO additive was spin-coated onto Al. Ca (~5 nm) and Al (~80 nm) were successively evaporated onto the active layer through a shadow mask (pressure ca. 10<sup>-4</sup> Pa). *J-V* curves were measured by using a computerized Keithley 2400 SourceMeter in the dark.

## 7. Optimization of device performance

D/A	$V_{\rm oc}$	$J_{ m sc}$	FF	PCE
[w/w]	[V]	[mA/cm <sup>2</sup> ]	[%]	[%]
1:1	0.81	11.90	57.7	5.58 (5.42) <sup>b</sup>
1:1.2	0.82	12.13	59.0	5.86 (5.66)
1:1.4	0.82	12.38	60.4	6.12 (5.97)
1:1.6	0.81	12.25	58.7	5.85 (5.77)

Table S1 Optimization of D/A ratio for PTB7-Th:CO<sub>i</sub>7IC inverted solar cells.<sup>a</sup>

<sup>a</sup> Blend solution: 16 mg/mL in ODCB; spin-coating: 800 rpm for 85 s.

<sup>b</sup> Data in parentheses stand for the average PCEs for 10 cells.

**Table S2** Optimization of the active layer thickness for PTB7-Th:CO<sub>i</sub>7IC inverted solar cells.<sup>*a*</sup>

Thickness	V <sub>oc</sub>	$J_{ m sc}$	FF	PCE
[nm]	[V]	[mA/cm <sup>2</sup> ]	[%]	[%]
123	0.82	11.51	57.2	5.41 (5.23) <sup>b</sup>
105	0.82	12.63	60.9	6.29 (6.17)
96	0.82	12.38	60.4	6.12 (5.97)
86	0.82	11.99	58.7	5.79 (5.64)
80	0.82	10.59	59.8	5.19 (4.95)

<sup>*a*</sup> D/A ratio: 1:1.4 (w/w); blend solution: 16 mg/mL in ODCB.

<sup>b</sup> Data in parentheses stand for the average PCEs for 10 cells.

DIO	$V_{\rm oc}$	$J_{ m sc}$	FF	PCE
[vol%]	[V]	[mA/cm <sup>2</sup> ]	[%]	[%]
0	0.82	12.63	60.9	6.29 (6.17) <sup>b</sup>
0.2	0.82	13.12	58.9	6.36 (6.25)
0.5	0.82	12.80	59.5	6.25 (5.99)
1	0.81	11.94	59.4	5.79 (5.27)

Table S3 Optimization of DIO content for PTB7-Th:CO<sub>i</sub>7IC inverted solar cells.<sup>a</sup>

<sup>a</sup> D/A ratio: 1:1.4 (w/w); blend solution: 16 mg/mL in ODCB; spin-coating: 600 rpm for 85 s.
<sup>b</sup> Data in parentheses stand for the average PCEs for 10 cells.

Table S4 Optimization of D/A ratio for PTB7-Th:CO<sub>i</sub>7DFIC inverted solar cells.<sup>a</sup>

D/A	$V_{\rm oc}$	$J_{ m sc}$	FF	PCE
[w/w]	[V]	[mA/cm <sup>2</sup> ]	[%]	[%]
1:1	0.68	16.38	61.6	$6.85 (6.56)^b$
1:1.2	0.68	16.50	69.1	7.76 (7.49)
1:1.4	0.67	17.31	69.8	8.08 (7.81)
1:1.6	0.68	16.85	68.6	7.83 (7.48)

<sup>*a*</sup> Blend solution: 16 mg/mL in ODCB with 0.2 vol% DIO; spin-coating: 1000 rpm for 85 s. <sup>*b*</sup> Data in parentheses stand for the average PCEs for 10 cells.

Thickness	$V_{\rm oc}$	$J_{ m sc}$	FF	PCE
[nm]	[V]	[mA/cm <sup>2</sup> ]	[%]	[%]
117	0.65	16.48	66.3	7.13 (6.97) <sup>b</sup>
95	0.66	16.92	71.1	7.91 (7.68)
83	0.67	17.31	69.8	8.08 (7.81)
71	0.67	16.11	72.8	7.81 (7.58)
56	0.66	15.94	70.1	7.35 (7.32)

**Table S5** Optimization of the active layer thickness for PTB7-Th:CO<sub>i</sub>7DFIC inverted solar cells.<sup>*a*</sup>

<sup>a</sup> D/A ratio: 1:1.4 (w/w); blend solution: 16 mg/mL in ODCB with 0.2 vol% DIO.

<sup>b</sup> Data in parentheses stand for the average PCEs for 10 cells.

Table S6 Optimization of DIO content for PTB7-Th:CO<sub>i</sub>7DFIC inverted solar cells.<sup>a</sup>

DIO	$V_{\rm oc}$	$J_{ m sc}$	FF	PCE
[vol%]	[V]	[mA/cm <sup>2</sup> ]	[%]	[%]
0	0.69	17.79	61.8	7.54 (7.32) <sup>b</sup>
0.2	0.67	17.31	69.8	8.08 (7.81)
0.4	0.67	17.36	71.9	8.32 (8.00)
0.6	0.67	16.12	70.0	7.54 (7.36)

<sup>a</sup> D/A ratio: 1:1.4 (w/w); blend solution: 16 mg/mL in ODCB; spin-coating: 1000 rpm for 85 s.
<sup>b</sup> Data in parentheses stand for the average PCEs for 10 cells.

## 8. Exciton dissociation probabilities



Fig. S14  $J_{\rm ph}$ - $V_{\rm eff}$  plots.

## 9. SCLC

Charge carrier mobility was measured by SCLC method. The mobility was determined by fitting the dark current to the model of a single carrier SCLC, which is described by:

$$J = \frac{9}{8}\varepsilon_0\varepsilon_r\mu\frac{V^2}{d^3}$$

where *J* is the current density,  $\mu$  is the zero-field mobility of holes ( $\mu_h$ ) or electrons ( $\mu_e$ ),  $\varepsilon_0$  is the permittivity of the vacuum,  $\varepsilon_r$  is the relative permittivity of the material, *d* is the thickness of the blend film, and *V* is the effective voltage ( $V = V_{appl} - V_{bi}$ , where  $V_{appl}$  is the applied voltage, and  $V_{bi}$  is the built-in potential determined by electrode work function difference). Here,  $V_{bi} = 0.1$  V for hole-only devices,  $V_{bi} = 0$  V for electron-only devices.<sup>[4]</sup> The mobility was calculated from the slope of  $J^{1/2}$ -*V* plots.



**Fig. S15** *J-V* curves (a) and corresponding  $J^{1/2}$ -*V* plots (b) for the hole-only devices (in dark). The thicknesses for PTB7-Th:CO<sub>i</sub>7IC and PTB7-Th:CO<sub>i</sub>7DFIC blend films are 100 nm and 95 nm, respectively.



Fig. S16 *J-V* curves (a) and corresponding  $J^{1/2}$ -*V* plots (b) for the electron-only devices (in dark). The thicknesses for PTB7-Th:CO<sub>i</sub>7IC and PTB7-Th:CO<sub>i</sub>7DFIC blend films are 100 nm and 110 nm, respectively.

**Table S7**. Hole and electron mobilities for PTB7-Th:NFA blend films.

PTB7-Th:NFA	$\mu_{ m h}$ [cm <sup>2</sup> V <sup>-1</sup> s <sup>-1</sup> ]	$\mu_{ m e}$ [cm <sup>2</sup> V <sup>-1</sup> s <sup>-1</sup> ]	$\mu_{ m h}/\mu_{ m e}$
PTB7-Th:CO <sub>i</sub> 7IC	2.58×10 <sup>-4</sup>	1.43×10 <sup>-5</sup>	18.0
PTB7-Th:CO <sub>i</sub> 7DFIC	4.73×10 <sup>-4</sup>	6.95×10 <sup>-5</sup>	6.8

## 10. Bimolecular recombination



**Fig. S17**  $J_{sc}$ - $P_{light}$  plots.

## 11. AFM



**Fig. S18** AFM height (left) and phase (right) images for the blend films. (a) and (b), PTB7-Th:CO<sub>i</sub>7IC film with 0.2 vol% DIO ( $R_{\rm rms} = 1.40$  nm); (c) and (d), PTB7-Th:CO<sub>i</sub>7DFIC film with 0.4 vol% DIO ( $R_{\rm rms} = 4.82$  nm).  $R_{\rm rms}$ : root-mean-square roughness.



Fig. S19 XRD profiles for the pure and blend films.

## References

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