

Electronic Supplementary Information to

**Highly electrochemically and thermally stable donor- $\pi$ -acceptor triphenylamine-based  
hole-transporting homopolymers via oxidative polymerization**

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## 1. Experimental part

### 1.1 Materials

Tetrakis(triphenylphosphine)palladium (0) Pd(PPh<sub>3</sub>)<sub>4</sub>, (4-bromophenyl)diphenylamine, malononitrile, FeCl<sub>3</sub> were obtained from Sigma–Aldrich Co. and used without further purification. THF, toluene, pyridine, nitrobenzene were dried and purified according to the known techniques. (4-{5-[1,1-dicyano(4-fluorophenyl)methyl]-2-thienyl}phenyl)diphenylamine (TA-TDCV-PF) was obtained as described in Ref. [1]. 2-hexyl-5,5-dimethyl-2-[5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2-thienyl]-1,3-dioxane (2) was obtained as described in Ref. [2]. All reactions, unless stated otherwise, were carried out under an inert atmosphere.

### 1.2 General characterization

<sup>1</sup>H NMR spectra were recorded at a “Bruker WP-250 SY” spectrometer, working at a frequency of 250.13 MHz and utilizing CDCl<sub>3</sub> signal (7.25 ppm) or Acetone-D<sub>6</sub> (2.09 ppm) as the internal standard. <sup>13</sup>C NMR spectra were recorded using a “Bruker Avance II 300” spectrometer at 75 MHz. In the case of <sup>1</sup>H NMR spectroscopy, the compounds to be analyzed were taken in the form of 1% solutions in CDCl<sub>3</sub>. In the case of <sup>13</sup>C NMR spectroscopy, the compounds to be analyzed were taken in the form of 5% solutions in CDCl<sub>3</sub>. The spectra were then processed using the ACD Labs software.

Elemental analysis of C, N and H elements was carried out using a CHN automatic analyzer (CE 1106). The settling titration using BaCl<sub>2</sub> was applied to analyze Sulfur. The experimental error for elemental analysis is 0.30-0.50%. Mass-spectra (MALDI) were registered on the Autoflex II Bruker (resolution FWHM 18000), equipped with a nitrogen laser (work wavelength 337 nm) and time-of-flight mass-detector working in reflections mode. The accelerating voltage was 20 kV. Samples were applied to a polished stainless steel substrate. Spectrum was recorded in the positive ion mode. The resulting spectrum was the sum of 300 spectra obtained at different points of sample. 2,5-Dihydroxybenzoic acid (DHB) (Acros, 99%) and α-cyano-4-hydroxycinnamic acid (HCCA) (Acros, 99%) were used as matrices. Knövenagel condensation was carried out in the microwave (Discovery, CEM corporation), using a standard method with the open vessel option, 50 watts. In the case of column chromatography, silica gel 60 (Merck) was taken. Absorption profiles were recorded with an absorption spectrometer (Shimadzu UV 2501 PC) at room temperature in diluted THF solutions (10<sup>-5</sup> M) and films cast from THF solutions with concentration 10 g/L on quartz substrates.

GPC analysis was performed on a Shimadzu instrument with a RID10AVP refractometer and a SPD-M10AVP diode matrix as detector using 7.8 × 300 mm<sup>2</sup> Phenomenex columns (USA) filled with the Phenogel sorbent with pore size of 500 Å and THF as the eluent. Light scattering experiments were performed on Shimadzu HPLC System, equipped with DGU20A3 degasser

unit, LC-20AD pump, CTO-20A column oven with a Phenomenex column (USA) with a size of  $7.8 \times 300 \text{ mm}^2$  filled with the Phenogel sorbent with a pour size of  $10^3 \text{ \AA}$ , RID-10A refractometer, Viscotek 270 Dual detector (RALS and LALS) and OmniseC 4.5 Software. The sample loop was  $100 \text{ }\mu\text{L}$ , angle RALS was  $90^\circ$ , angle LALS was  $7^\circ$ . The system was calibrated using polystyrene standards with  $M_w = 11.6 \times 10^3$  and  $M_w = 68 \times 10^3$ ,  $dn/dc = 0.185$  (Polymer Laboratories). The molar weights of the standards were chosen in the range of  $M_w$  of the samples under investigation for better accuracy of the measurements. Using the exact concentrations of the samples, close to  $5.00 \text{ mg/mL}$ , the values of  $dn/dc$  were determined from RI data. Then this value was used by OmniseC 4.5 software to calculate the  $M_w$  from the light scattering (RALS and LALS) data.

Cyclic voltammetry measurements were carried out using IPC-ProM potentiostat in 1,2-dichlorobenzene/acetonitrile (4:1) mixture of solvents for  $10^{-3} \text{ M}$  solutions in a standard three-electrode cell equipped with a glassy carbon working electrode ( $s = 2 \text{ mm}^2$ ), platinum plate as the counter electrode, and SCE (saturated calomel electrode) as the reference electrode. The scan rate was  $200 \text{ mV s}^{-1}$ .  $\text{Bu}_4\text{NPF}_6$  was used as supporting electrolyte. The highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) energies were evaluated using the first standard oxidation ( $\phi_{\text{ox}}$ ) and reduction ( $\phi_{\text{red}}$ ) potentials obtained from CV experiments as  $E(\text{HOMO}) = -e(\phi_{\text{ox}} + 4.40) \text{ (eV)}$  and  $E(\text{LUMO}) = -e(\phi_{\text{red}} + 4.40) \text{ (eV)}$ , where  $e$  is the elementary charge [3, 4].

Thermogravimetric analysis was carried out in a dynamic mode within the temperature range of  $30\text{--}700 \text{ }^\circ\text{C}$  using a “Mettler Toledo TG50” system equipped with a microbalance (M3). Heating/cooling rate was chosen to be  $10 \text{ }^\circ\text{C/min}$ . Every compound was studied in air and nitrogen flow of  $200 \text{ mL/min}$ . Differential scanning calorimetry (DSC) scans were obtained with a “Mettler Toledo DSC30” system with  $20 \text{ }^\circ\text{C/min}$  heating/cooling rate in the temperature range of  $+20\text{--}290 \text{ }^\circ\text{C}$  for all compounds.  $\text{N}_2$  flow of  $50 \text{ mL/min}$  was used.

The solubility of molecules was measured using their saturated solutions in chloroform, which were prepared by stirring of an excess of solid material in the solvent [5]. For this purpose, the compounds were added in small portions to  $1 \text{ mL}$  of pure solvent. As prepared, the saturated solution was filtered through  $0.25\text{-mm}$  PTFE syringe filters, and the solvent was evaporated using a rotary evaporator. Afterwards the residue was dried in vacuum at  $130^\circ\text{C}$  until reaching its constant weight, which was used to calculate the solubility value.

### 1.3 Monomer Synthesis

**{4-[5-(2-hexyl-5,5-dimethyl-[1,3]-dioxane-2-yl)-phenyl]-diphenylamine (3)}**. In an inert atmosphere, degassed solutions of 2-hexyl-5,5-dimethyl-2-[5-(4,4,5,5-tetramethyl-[1,3,2]-

dioxyborolan-2-yl)thiophen-2-yl]-[1,3]-dioxane (**2**) (5.6 g, 13.6 mmol) and (4-bromophenyl)diphenylamine (**1**) (3.5 g, 10.8 mmol) in toluene/ethanol mixture (70/7 mL) and 2M solution of aq. Na<sub>2</sub>CO<sub>3</sub> (10 mL) were added to Pd(PPh<sub>3</sub>)<sub>4</sub> (400 mg, 0.0034 mmol). The reaction mixture was stirred under reflux for 10 h, and then it was cooled to room temperature and poured into 100 mL of water and 70 mL of toluene. The organic phase was separated, washed with water, dried over sodium sulfate and filtered. The solvent was evaporated in vacuum and the residue was dried at 1 Torr. The product was purified by column chromatography on silica gel (eluent toluene: petroleum ether, 1:2) to give pure compound **3** (3.40 g, 60%) as a viscous yellow liquid. <sup>1</sup>H NMR (250 MHz, acetone-d<sub>6</sub>): δ [ppm] 0.63 (s, 3H), 0.84 (t, 3H, *J* = 6.7 Hz), 1.19 (s, 3H), 1.21-1.33 (overlapping peaks, 6H), 1.37-1.53 (overlapping peaks, 2H), 1.76-1.89 (overlapping peaks, 2H), 3.42 (d, 6H, *J* = 11.0 Hz), 3.72 (d, 2H, *J* = 10.7 Hz), 6.90 (d, 1H, *J* = 3.7 Hz), 6.96-7.15 (overlapping peaks, 8H), 7.21-7.36 (overlapping peaks, 5H), 7.52 (d, 2H, *J* = 8.9 Hz). <sup>13</sup>C NMR (125 MHz, acetone-d<sub>6</sub>): δ [ppm] 14.99, 22.70, 23.68, 23.90, 24.53, 30.67, 30.94, 33.23, 46.43, 72.90, 101.60, 123.58, 124.76, 125.01, 125.91, 127.78, 128.80, 130.00, 130.94, 144.72, 145.48, 148.80, 149.01. Calcd (%) for C<sub>34</sub>H<sub>39</sub>NO<sub>2</sub>S: C, 77.67; H, 7.48; S, 6.10; N, 2.66. Found C, 77.81; H, 7.61; S, 6.03; N, 2.53.

**1-[5-(4-diphenylamine-phenyl)-thiophen-2-yl]-hexen-1-one (4).** 1M HCl (7 mL) was added to a solution of compound **2** (2.7 g, 5.1 mmol) in THF (50 mL) and then the reaction mixture was stirred for 4 hours at reflux. After completion of the reaction the organic phase was separated using diethyl ether, washed with water and filtered off to give pure compound **4** (2.0 g, 91%) as a yellow powder. <sup>1</sup>H NMR (250 MHz, CDCl<sub>3</sub>): δ [ppm] 0.94 (t, 3H, *J* = 6.9 Hz), 1.27-1.53 (overlapping peaks, 6H), 1.73-1.92 (overlapping peaks, 2H), 2.91 (t, 2H, *J* = 7.3 Hz), 7.05-7.21 (overlapping peaks, 8H), 7.24 (d, 1H, *J* = 3.9 Hz), 7.27-7.39 (overlapping peaks, 4H), 7.52 (d, 2H, *J* = 8.8 Hz), 7.67 (d, 1H, *J* = 3.7 Hz). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ [ppm] 13.61, 22.06, 24.52, 28.60, 31.17, 38.54, 122.21, 122.24, 123.18, 124.52, 126.28, 126.56, 128.97, 132.42, 141.51, 146.62, 148.18, 152.01, 192.87. Calcd (%) for C<sub>29</sub>H<sub>29</sub>NOS: C, 79.23; H, 6.65; S, 7.29; N, 3.19. Found C, 79.37; H, 6.77; S, 7.26; N, 3.09.

**{4-[5-(2-hexyl-1,1-dicyanoet-1-en-2-yl)-2-thienyl]phenyl}diphenylamine (TA-TDCV-H).** Compound **3** (1.8 g, 4.2 mmol), malononitrile (1.1 g, 16.5 mmol) and dry pyridine (37 mL) were placed in a reaction vessel and stirred under argon atmosphere for 11 hours at reflux using the microwave heating. After completeness of the reaction, pyridine was evaporated in vacuum and the residue was dried at 1 Torr. The crude product was purified by column chromatography on silica gel (eluent: methylene chloride). Further purification included precipitation of the product from its THF solution with toluene and hexane to give pure product as a red solid (1.7 g, 87%). <sup>1</sup>H NMR (250 MHz, CDCl<sub>3</sub>): δ [ppm] 0.88 (t, 3H, *J* = 6.7 Hz), 1.25-1.38 (overlapping peaks, 4H),

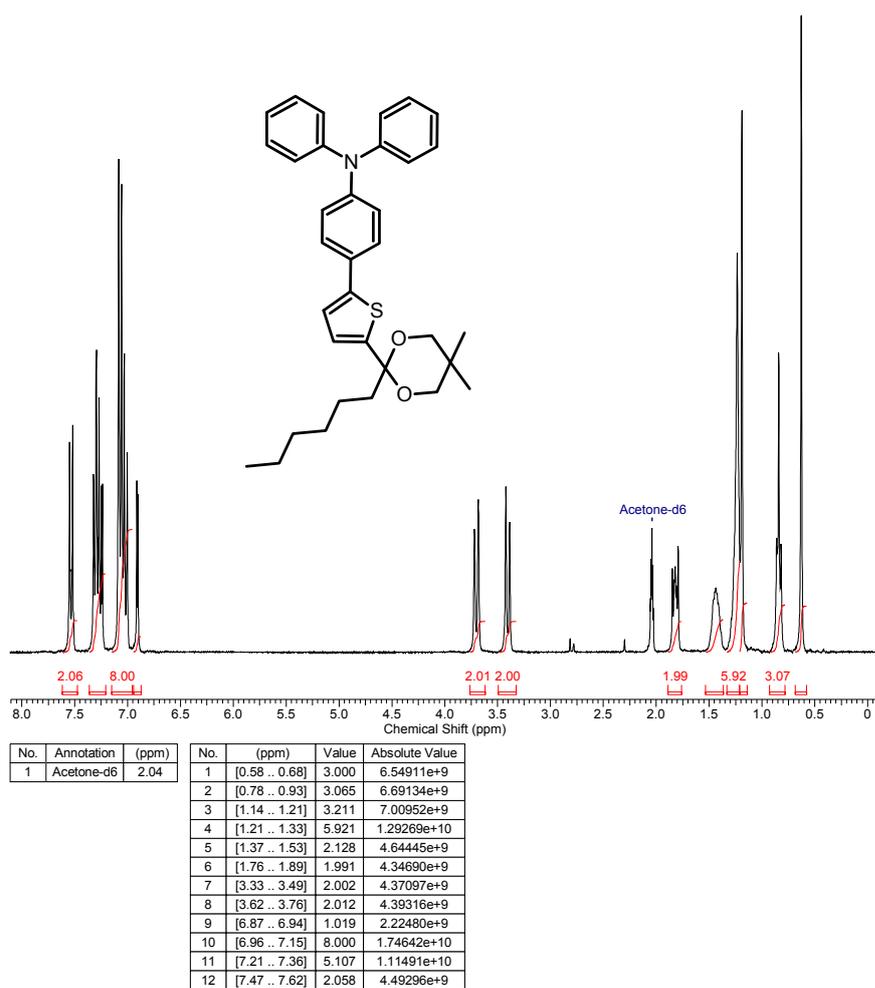
1.39-1.52 (overlapping peaks 2H), 1.70 (m, M = 5, 2H,  $J = 6.7$  Hz), 2.92 (t, 2H,  $J = 7.3$  Hz), 6.96-7.22 (overlapping peaks, 8H), 7.25-7.37 (overlapping peaks, 5H), 7.48 (d, 2H,  $J = 8.8$  Hz), 7.99 (d, 1H,  $J = 3.7$  Hz).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  [ppm] 13.53, 21.98, 28.75, 30.04, 30.82, 37.09, 75.24, 113.66, 114.43, 121.59, 123.39, 123.63, 124.75, 124.84, 126.83, 129.06, 134.46, 134.92, 146.30, 148.97, 153.77, 166.22. Calcd (%) for  $\text{C}_{32}\text{H}_{29}\text{N}_3\text{S}$ : C, 78.81; H, 5.99; S, 6.57; N, 8.62. Found C, 78.93; H, 6.09; S, 6.51; N, 8.51. MALDI-MS: found  $m/z$  486.99; calculated for  $[\text{M}]^+$  487.67.

#### 1.4 Polymer Synthesis

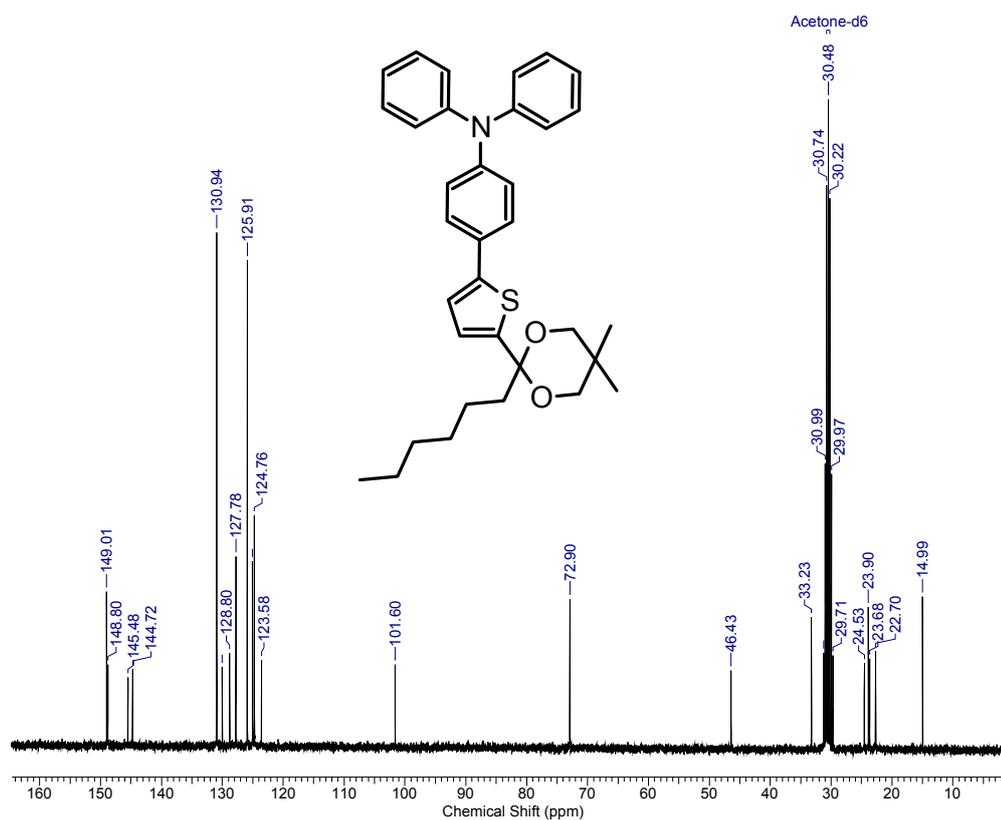
**Poly{4-[5-(2-hexyl-1,1-dicyanoet-1-en-2-yl) -2-thienyl] phenyl}diphenylamine (P(TA-TDCV-H)).**  $\text{FeCl}_3$  (0.722 g, 3.59 mmol) and nitrobenzene (5.3 ml) were added to **TA-TDCV-H** (0.7 g, 1.44 mmol). The solution was stirred at room temperature in an inert atmosphere for 108 h and then poured into a mixture of methanol and 1M hydrochloric acid in a ratio of 4:1. The precipitate was collected and washed thoroughly with aqueous ammonium hydroxide. Precipitations from chloroform solutions with ethanol and the further purification by the method of Soxhlet extraction in toluene gave rise to the polymer as a red solid (0.263 g, 36%).  $^1\text{H}$  NMR (250 MHz,  $\text{CDCl}_3$ ):  $\delta$  [ppm] 0.89 (t, 3H,  $J = 6.7$  Hz), 1.20-1.42 (overlapping peaks, 4H), 1.42-1.57 (overlapping peaks, 2H), 1.73 (m, M = 5, 2H,  $J = 6.7$  Hz), 2.96 (t, 3H,  $J = 6.7$  Hz), 7.11-7.41 (overlapping peaks 7H), 7.45-7.74 (overlapping peaks 6H), 8.01 (d, 1H,  $J = 3.7$  Hz). Calcd (%) for  $(\text{C}_{32}\text{H}_{27}\text{N}_3\text{S})_n$ : C, 79.14; H, 5.60; S, 6.60; N, 8.65. Found (%) for  $(\text{C}_{32}\text{H}_{18}\text{F}_1\text{N}_3\text{S})_n$ : C, 78.52; H, 5.38; S, 5.88; N, 8.58.

**Poly[{5-[4-(diphenylamino)phenyl]-2-thienyl}(4-fluorophenyl)methylene]malononitrile (P(TA-TDCV-PF)).** The polymer was obtained by the method described above for **P(TA-TDCV-Hex)** using  $\text{FeCl}_3$  (0.31 g, 1.90 mmol), nitrobenzene (2.9 ml) and **TA-TDCV-PF** (0.38 g, 0.76 mmol). The solution was stirred at room temperature for 120 h and poured into ethanol. The precipitate was collected and washed thoroughly with aqueous ammonium hydroxide. Precipitations from chloroform solution with ethanol were carried out for further purification. Then the product was washed successively with 1N hydrochloric acid solution (3x5ml) and ammonia water (3x5ml). Precipitations from chloroform solutions with ethanol and the further purification by the method of Soxhlet extraction in toluene gave rise to afford the polymer as a burgundy solid (0.304 g, 80%).  $^1\text{H}$  NMR (250 MHz,  $\text{CDCl}_3$ ):  $\delta$  [ppm] 7.02-7.24 (overlapping peaks, 7H), 7.30 (s, 2H,  $J = 7.33$  Hz), 7.41-7.70 (overlapping peaks, 9H). Calcd (%) for  $(\text{C}_{32}\text{H}_{18}\text{F}_1\text{N}_3\text{S})_n$ : C, 77.56; H, 3.66; S, 6.47; N, 8.48. Found (%) for  $(\text{C}_{32}\text{H}_{18}\text{F}_1\text{N}_3\text{S})_n$ : C, 77.16; H, 3.86; S, 6.26; N, 8.16.

## 2. $^1\text{H}$ and $^{13}\text{C}$ NMR Spectra

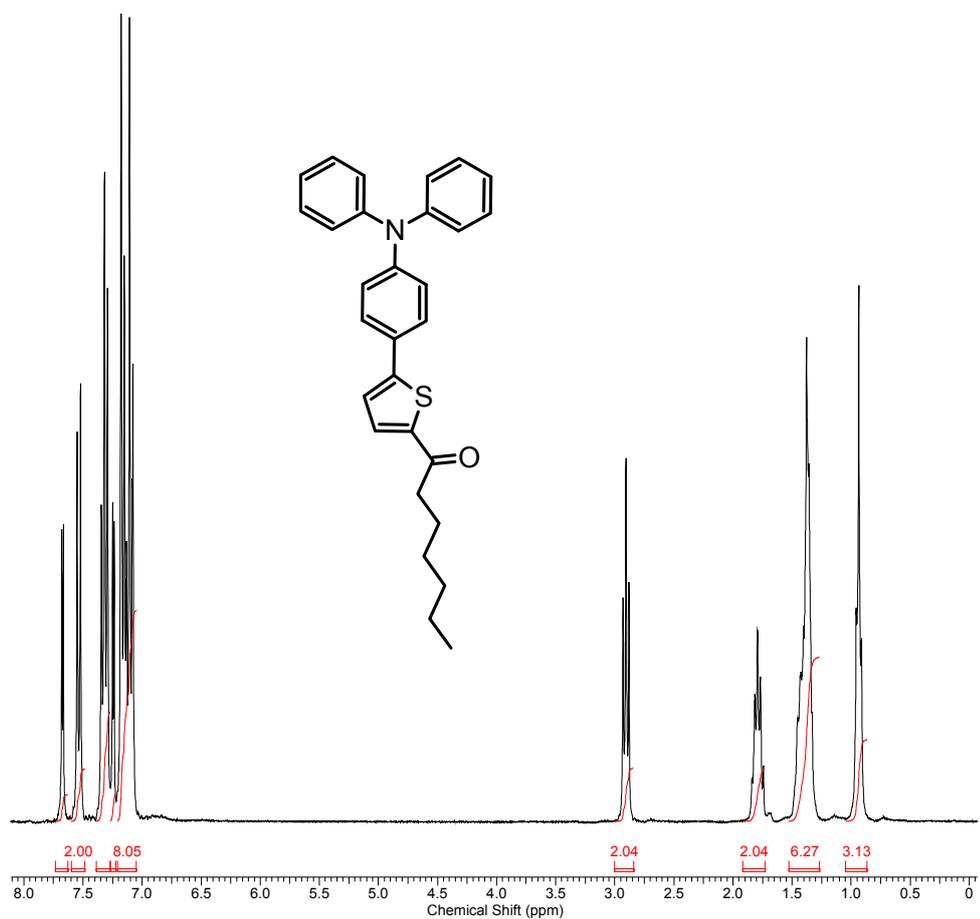


**Figure S2.1.**  $^1\text{H}$  NMR spectrum of compound **3** in acetone-D<sub>6</sub>.



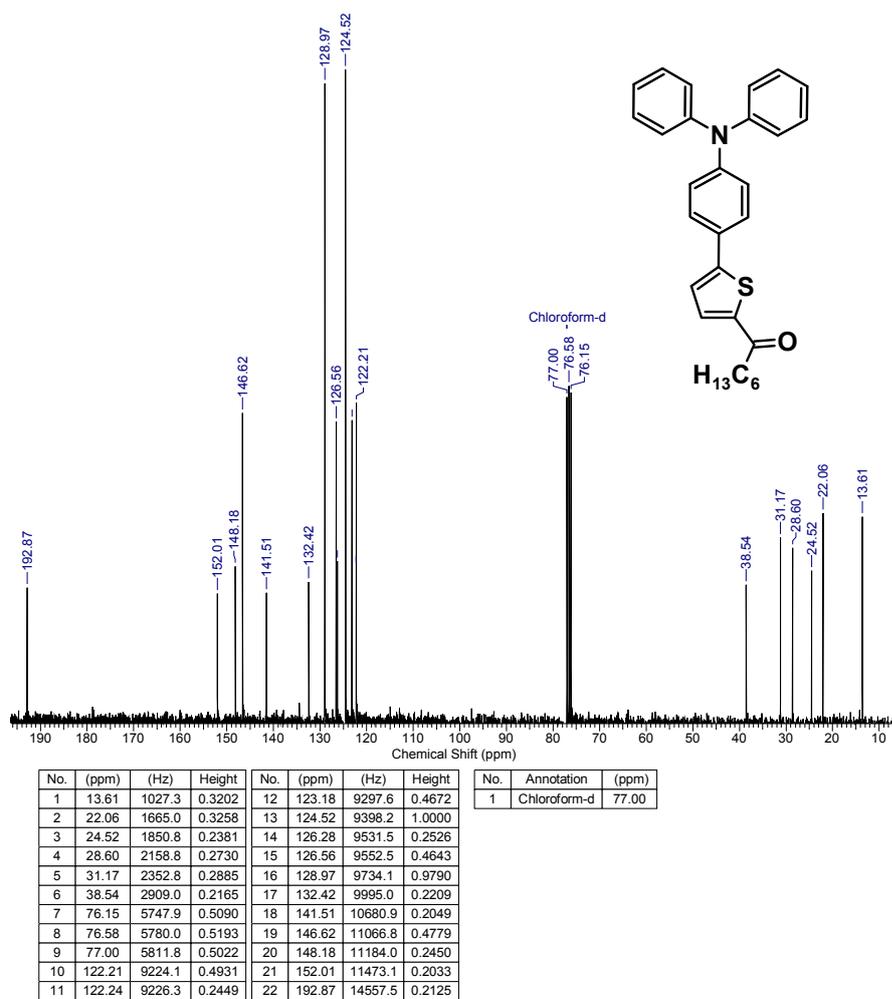
No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)
1	14.99	1131.2	0.1952	16	46.43	3504.1	0.0977	1	Acetone-d6	[30.22 .. 30.74]
2	22.70	1713.3	0.1232	17	72.90	5502.4	0.1924			
3	23.68	1787.4	0.1134	18	101.60	7668.5	0.1060			
4	23.90	1803.7	0.1816	19	123.58	9327.4	0.1115			
5	24.53	1851.8	0.1073	20	124.76	9416.4	0.3021			
6	29.71	2242.4	0.1180	21	125.01	9435.7	0.2424			
7	29.97	2261.8	0.3569	22	125.91	9503.2	0.6388			
8	30.22	2281.1	0.7208	23	127.78	9644.4	0.2485			
9	30.48	2300.5	0.8513	24	128.80	9721.3	0.1205			
10	30.67	2315.1	0.1521	25	130.00	9812.0	0.1028			
11	30.74	2319.8	0.7381	26	130.94	9882.7	0.6757			
12	30.94	2335.6	0.1688	27	144.72	10923.2	0.0998			
13	30.99	2339.2	0.3705	28	145.48	10980.7	0.0889			
14	31.25	2358.5	0.1212	29	148.80	11231.4	0.1056			
15	33.23	2507.8	0.1682	30	149.01	11246.6	0.2019			

Figure S2.2.  $^{13}\text{C}$  NMR spectrum of compound 3 in acetone-D6.

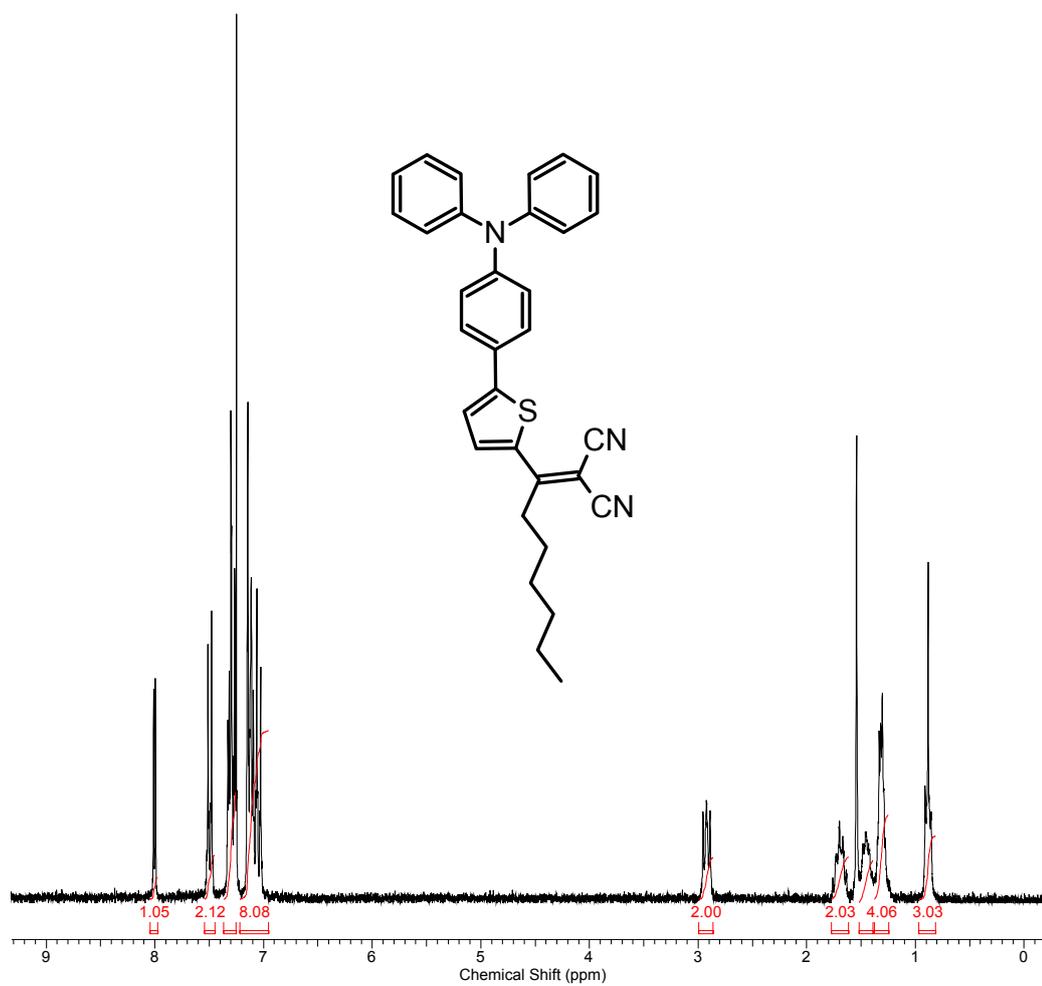


No.	Annotation	(ppm)	No.	(ppm)	Value	Absolute Value
1	Chloroform-d	7.25	1	[0.86 .. 1.05]	3.128	1.77747e+10
			2	[1.27 .. 1.53]	6.270	3.56297e+10
			3	[1.73 .. 1.92]	2.041	1.15990e+10
			4	[2.84 .. 3.00]	2.036	1.15684e+10
			5	[7.05 .. 7.21]	8.055	4.57713e+10
			6	[7.22 .. 7.27]	1.042	5.92245e+9
			7	[7.27 .. 7.39]	4.175	2.37251e+10
			8	[7.48 .. 7.60]	2.000	1.13650e+10
			9	[7.63 .. 7.73]	1.024	5.82043e+9

Figure S2.3.  $^1\text{H}$  NMR spectrum of compound 4 in  $\text{CDCl}_3$ .



**Figure S2.4.**  $^{13}\text{C}$  NMR spectrum of compound **4** in  $\text{CDCl}_3$ .



No.	Annotation	(ppm)	No.	(ppm)	Value	Absolute Value
1	H <sub>2</sub> O	1.54	1	[0.82 .. 0.97]	3.029	2.93954e+7
2	Chloroform-d	7.25	2	[1.25 .. 1.38]	4.060	3.93973e+7
			3	[1.39 .. 1.52]	2.058	1.99752e+7
			4	[1.61 .. 1.77]	2.028	1.96831e+7
			5	[2.86 .. 3.00]	2.000	1.94080e+7
			6	[6.96 .. 7.22]	8.078	7.83870e+7
			7	[7.25 .. 7.37]	5.179	5.02529e+7
			8	[7.45 .. 7.55]	2.116	2.05381e+7
			9	[7.97 .. 8.05]	1.050	1.01860e+7

**Figure S2.5.** <sup>1</sup>H NMR spectrum of compound TA-TDCV-H in CDCl<sub>3</sub>.

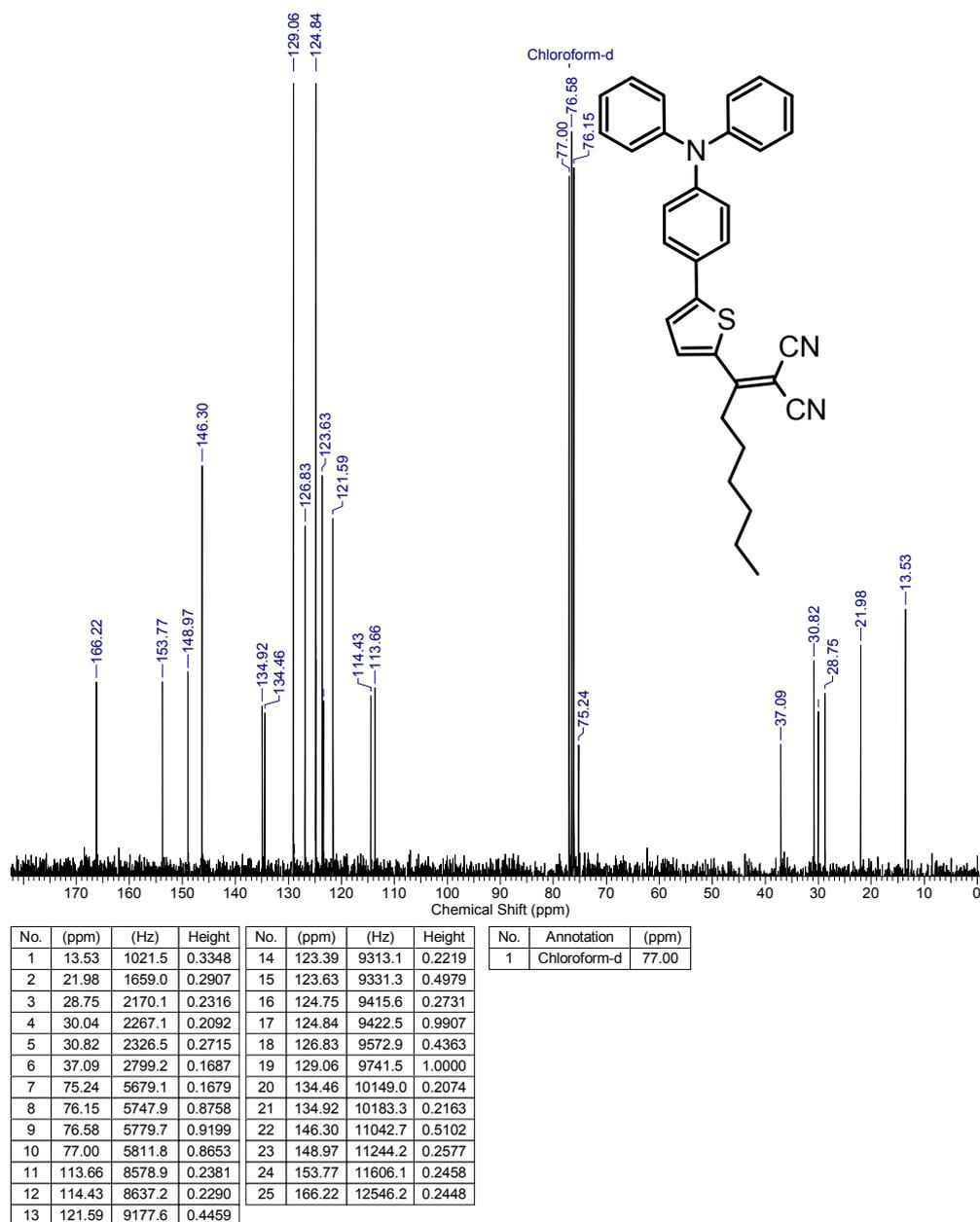
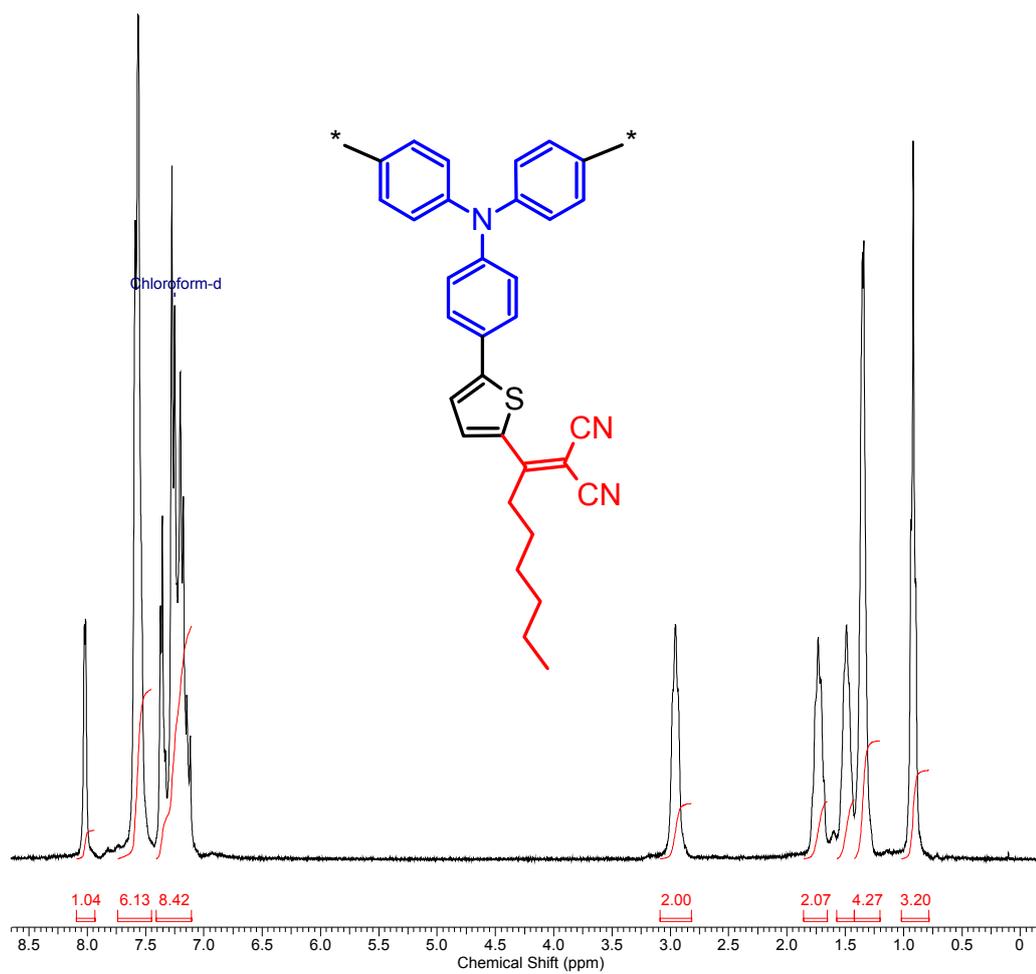
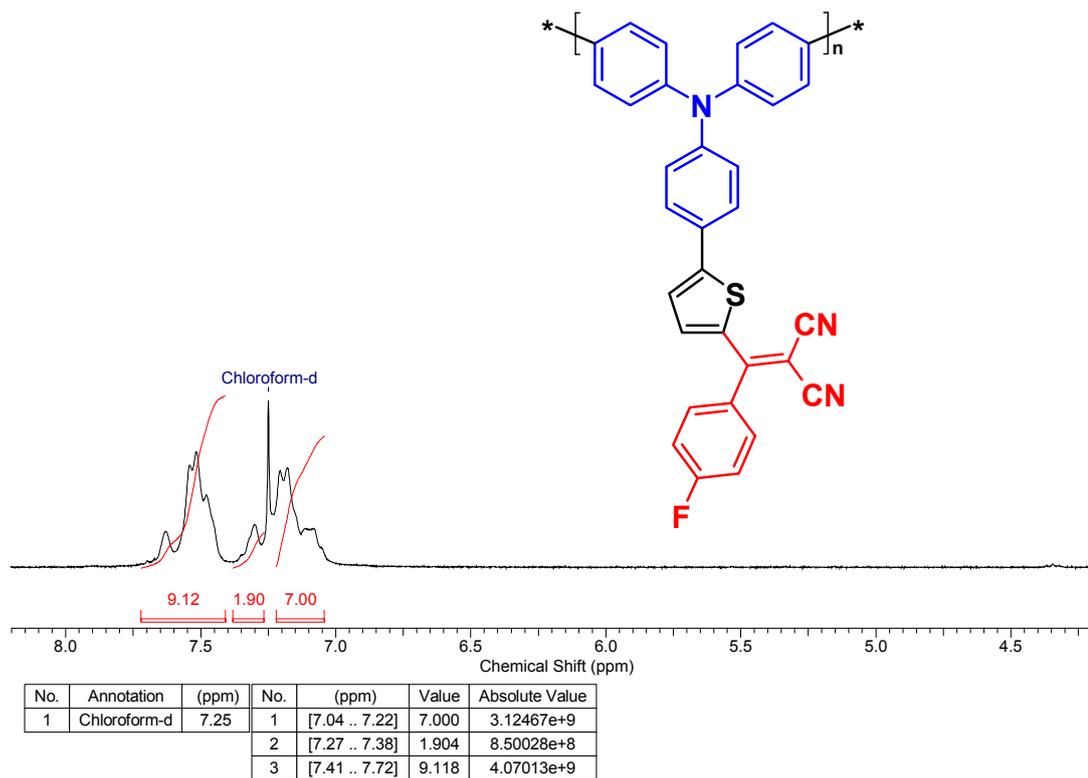


Figure S2.6.  $^{13}\text{C}$  NMR spectrum of compound TA-TDCV-H in  $\text{CDCl}_3$ .



No.	Annotation	(ppm)	No.	(ppm)	Value	Absolute Value
1	Chloroform-d	7.25	1	[0.78 .. 1.02]	3.199	2.11816e+10
			2	[1.20 .. 1.42]	4.272	2.82854e+10
			3	[1.42 .. 1.57]	2.153	1.42539e+10
			4	[1.66 .. 1.86]	2.070	1.37061e+10
			5	[2.82 .. 3.09]	2.000	1.32411e+10
			6	[7.11 .. 7.41]	8.422	5.57584e+10
			7	[7.45 .. 7.74]	6.131	4.05909e+10
			8	[7.94 .. 8.09]	1.038	6.86946e+9

Figure S2.7.  $^1\text{H}$  NMR spectrum of compound P(TA-TDCV-H) in  $\text{CDCl}_3$ .



**Figure S2.8.** <sup>1</sup>H NMR spectrum of compound P(TA-TDCV-PF) in CDCl<sub>3</sub>.

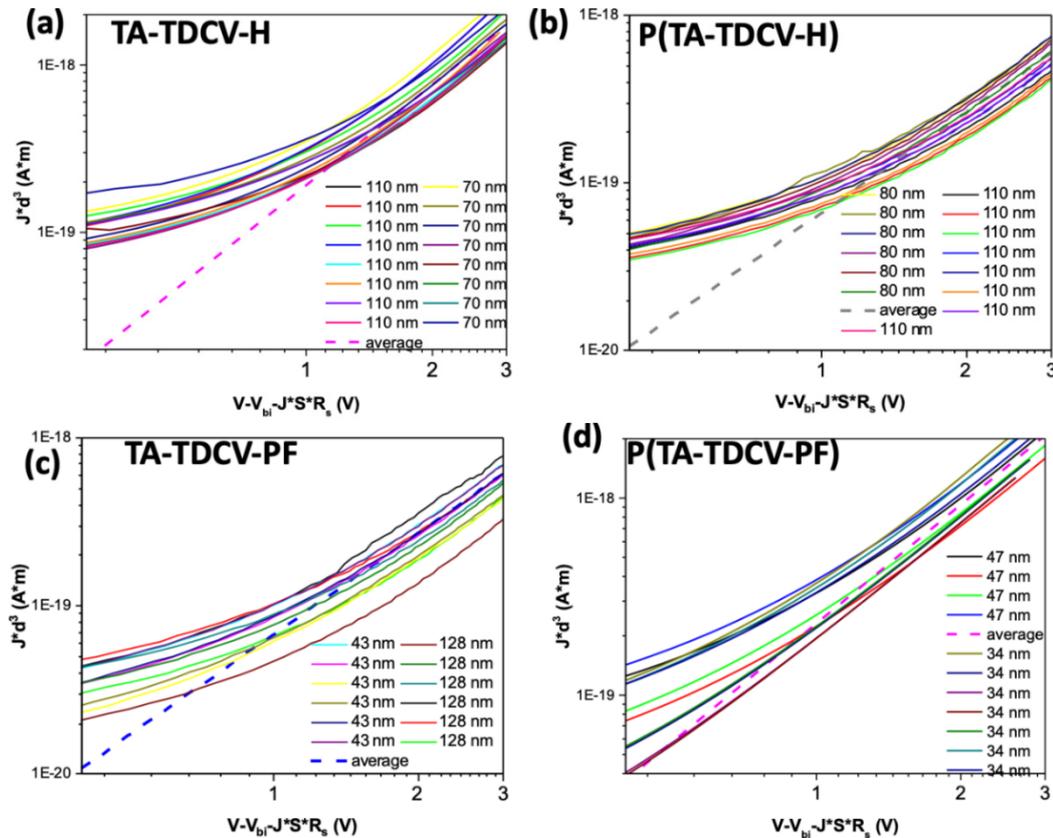
### 3. Charge-carrier mobility

Hole mobility was determined from space charge limited current (SCLC) measurements on unipolar thin-film devices of several thicknesses. To achieve the SCLC regime, an ohmic contact between the injection electrode and the semiconductor is required. The structure of the hole-only devices was ITO/PEDOT:PSS/active layer/Ag. The hole mobilities were extracted by fitting the current–voltage ( $J$ - $V$ ) characteristics of the corresponding devices with the simplest SCLC model.

According to the simplest SCLC model and taking into account the series ( $R_s$ ) and shunt ( $R_{sh}$ ) resistances (as fitting parameters), the charge mobility was calculated by approximating the dark  $J$ - $V$  curves of unipolar devices using the following equation:

$$J = \frac{9}{8} \varepsilon \varepsilon_0 \mu \frac{(V - V_{BI} - JSR_s)^2}{d^3} + \frac{V - JSR_s}{R_{sh}},$$

where  $\varepsilon_0 = 8.85 \cdot 10^{-12}$  F/m,  $\varepsilon$  is the dielectric permittivity (taken as 3),  $d$  is the active layer thickness (measured by AFM),  $V_{BI}$  is the built-in voltage (fitting parameter).  $J$ - $V$  characteristics were measured using a source-meter (SourceMeter 2400, Keithley) in dark and under light of a solar simulator (Newport 67005) with an intensity of 100 mW/cm<sup>2</sup> (AM1.5G).



**Figure S3.1**  $J$ - $V$  characteristics of the monomer and polymer hole only devices (solid lines).  $Jd^3$  is plotted for two different layer thicknesses against the voltage, corrected for the built-in voltage and the electrode series resistance. Dashed line represents SCLC-fit, from which the hole mobility is extracted.

## References

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