

Top-ranked efficiency under indoor light of DSSC enabled by iodide-based DES-like solvents electrolyte

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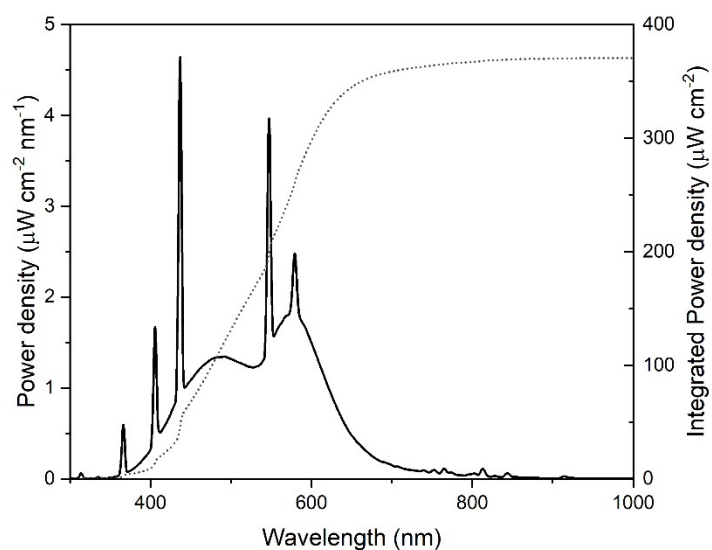
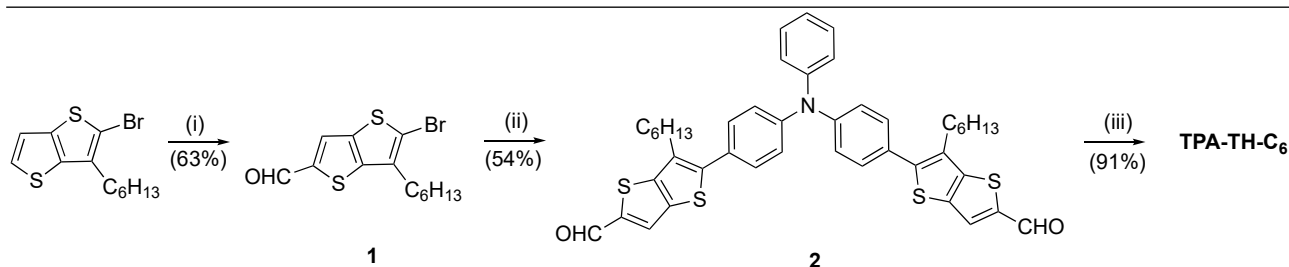


Fig. S1. Emission spectrum of Osram L 8W/765 T5 lamp used for indoor light DSSC characterization at 1200 lux (solid line) and corresponding integrated power density (dotted line).

Table S1. Averaged values of three independent cells with standard errors of the investigated dyes and electrolyte solutions.

Dye/coads.	DES solution ^[a]	J_{sc} (mA cm ⁻²)	V_{oc} (mV)	FF	PCE (%)
PTZ-Th-EG/GlcA	1	4.0 ± 0.3	488 ± 13	64 ± 3	1.23 ± 0.02
	2	$24 \times 10^{-5} \pm 1 \times 10^{-5}$	495 ± 1	55 ± 1	0.07 ± 0.01
	3	2.3 ± 0.1	496 ± 4	65 ± 1	0.73 ± 0.07
	4	1.6 ± 0.1	474 ± 3	59 ± 1	0.46 ± 0.04
PTZ-Th-EG/CDCA	1	5.1 ± 0.1	515 ± 6	60 ± 1	1.58 ± 0.01
	3	4.0 ± 0.2	512 ± 4	60 ± 6	1.25 ± 0.20
	4	2.1 ± 0.1	485 ± 7	59 ± 1	0.55 ± 0.10
PTZ-Th-C ₈ /CDCA	1	6.6 ± 0.1	561 ± 7	63 ± 1	2.37 ± 0.01
	3	8.1 ± 0.3	567 ± 2	66 ± 2	3.04 ± 0.07
	4	4.2 ± 0.2	524 ± 1	63 ± 1	1.40 ± 0.10
PTZ-Th-C ₈ /CDCA, different PMII conc.	1 (2 M)	6.6 ± 0.1	561 ± 7	63 ± 1	2.37 ± 0.01
	2.7 M	7.2 ± 0.2	510 ± 13	63 ± 2	2.41 ± 0.20
	1 M	3.7 ± 0.2	464 ± 3	61 ± 1	1.05 ± 0.05
TPA-TTh/CDCA	1	5.5 ± 0.4	562 ± 12	72 ± 2	2.20 ± 0.05
	3	8.1 ± 0.5	523 ± 7	62 ± 3	2.63 ± 0.33
	4	7.3 ± 0.1	515 ± 15	60 ± 5	2.20 ± 0.30
TPA-TTh-C ₆ /CDCA	1	5.8 ± 0.8	540 ± 26	69 ± 5	2.11 ± 0.32
	3	7.4 ± 0.3	599 ± 55	69 ± 2	3.06 ± 0.26
	4	8.2 ± 0.2	651 ± 10	69 ± 2	3.57 ± 0.20



Scheme S1. Synthesis of TPA-TTh-C₆: Reagents and condition: (i) POCl₃, anhydrous DMF, N₂, -10 °C to reflux, 8 h; (ii) N-Phenyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-N-(4-(4,4,5,5-tetramethyl-1,3,2-

dioxaborolan-2-yl)phenyl)aniline, Pd(dppf)Cl₂, K₂CO₃, N₂, Toluene, MeOH, 70 °C microwave heating 70 W, 1 h; (iii) cyanoacetic acid, piperidine, CHCl₃, reflux 4 h.

Synthetic procedures

Reactions performed under inert atmosphere were carried out in oven-dried glassware and a nitrogen atmosphere was made with Schlenk technique. All reagents were obtained from commercial suppliers at the highest purity grade and used without further purification. Anhydrous solvents were purchased from Akros and kept over molecular sieves, then used without further purification. Organic extracts were dried with Na₂SO₄ and filtered before removal of the solvent by evaporation. Chromatography was performed with Merck grade 9385 silica gel 230–400 mesh (60 Å). High-resolution mass spectra were recorded with an Agilent 6230B Time of Flight (TOF) equipped with an electrospray (Dual ESI) source. NMR spectra were recorded with a Bruker AMX-500 spectrometer operating at 500.13 MHz (¹H) and 125.77 MHz (¹³C). Coupling constants are given in Hz. Absorption spectra were recorded with a V-570 Jasco spectrophotometer. Reactions were monitored by thin-layer chromatography by using UV light (254 and 365 nm) as a visualizing agent.

PTZ-Th-C₈, **PTZ-Th-EG**, and **TPA-Th** were prepared according to a previously published procedure.⁶⁰⁻⁶² 2-bromo-3-hexylthieno[3,2-b]thiophene and *N*-Phenyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-*N*-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)aniline were prepared according to literature.^{63, 64, 65} The synthetic pathway to **TPA-Th-C₆** is depicted in **Scheme S1**.

Synthesis of 5-bromo-6-hexylthieno[3,2-b]thiophene-2-carbaldehyde (1). Under inert atmosphere, freshly distilled POCl₃ (1.84 g, 12.0 mmol) was added to anhydrous DMF (873 mg, 12.0 mmol) keeping temperature below 0 °C. The reaction mixture was kept at 0 °C for 30 min until a transparent glassy solid was formed. Then, anhydrous DMF (10 mL) was added and the reaction mixture was warmed to room temperature. Then, a solution of 2-bromo-3-hexylthieno[3,2-b]thiophene (363 mg, 1.20 mmol) in anhydrous DMF (10 mL) was added to the reaction mixture, and then heated at reflux for 8 h. The reaction mixture was then diluted with dichloromethane (50 mL) and poured in water saturated with CH₃COONa (100 mL) and stirred for 1 h. The organic layer was then separated, and the aqueous layer extracted with EtO₂ (3 x 20 mL). The collected organic layers were dried, filtered and the solvent evaporated under reduced pressure, yielding a dark yellow oil. 5-bromo-6-hexylthieno[3,2-b]thiophene-2-carbaldehyde (**1**) was obtained as a dark yellow oil (250 mg, 0.75 mmol, yield 63%) after flash chromatography over silica gel with a mixture of petroleum ether- AcOEt 15:1. ¹H NMR (500 MHz, CDCl₃) δ 9.94 (s, 1H), 7.82 (s, 1H), 2.76 (t, *J* = 7.6 Hz, 2H), 1.73 – 1.66 (m, 2H), 1.39 – 1.27 (m, 6H), 0.88 (t, *J* = 7.0 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 183.19, 145.05, 143.80, 136.92, 134.93, 128.60, 117.59, 31.46, 29.02, 28.90, 27.74, 22.49, 14.02.

Synthesis of 5,5'-((phenylazanediyl)bis(4,1-phenylene))bis(6-hexylthieno[3,2-b]thiophene-2-carbaldehyde) (2). 5-bromo-6-hexylthieno[3,2-b]thiophene-2-carbaldehyde (**1**) (250 mg, 0.75 mmol) was dissolved in a mixture of toluene/methanol (1/1 v/v, 6 mL), then *N*-Phenyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-*N*-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)aniline⁶⁵ (150 mg, 0.30 mmol), Pd(dppf)Cl₂ (25 mg, 0.03 mmol) were added under nitrogen bubbling. After complete dissolution, K₂CO₃ (3.43 g, 24.8 mmol) was added, and the reaction mixture was heated at 70 °C for 1 h under μw irradiation (70 W). The mixture was diluted with dichloromethane (20 mL) and then poured in a NH₄Cl (sat. sol., 100 mL). The two layers were filtered over a pad of Celite and then separated. The aqueous layer was extracted with dichloromethane (3 x 50 mL). The combined organic layers were washed with H₂O (50 mL), dried, filtered and then the solvent was removed. The crude was purified by flash chromatography (petroleum ether/ethyl acetate from 20:1 to 15:1), yielding 5,5'-((phenylazanediyl)bis(4,1-phenylene))bis(6-hexylthieno[3,2-b]thiophene-2-carbaldehyde) (**2**) (120 mg, 0.16 mmol, 54%) as orange solid. ¹H NMR (500 MHz, CDCl₃) δ 9.96 (s, 2H), 7.91 (s, 2H), 7.38 (d, *J* = 8.6 Hz, 4H), 7.36 (t, *J* = 6.0 Hz, 2H), 7.23 (d, *J* = 7.6 Hz, 2H), 7.20 (d, *J* = 8.6 Hz, 2H), 7.16 (t, *J* = 7.4 Hz, 1H), 2.91 – 2.77 (m, 4H), 1.76 (m, 4H), 1.41 – 1.24 (m, 12H), 0.87 (t, *J* = 6.9 Hz, 6H). ¹³C NMR (126 MHz, CDCl₃) δ 183.18, 148.02, 147.59, 146.74, 146.35, 143.66, 136.17, 130.99, 130.11, 129.68, 129.31, 128.30, 125.76, 124.44, 123.44, 31.42, 29.15, 28.63, 28.25, 22.50, 14.03.

Synthesis of TPA-TTh-C₆. 5,5'-((phenylazanediyl)bis(4,1-phenylene))bis(6-hexylthieno[3,2-b]thiophene-2-carbaldehyde) (**2**) (80 mg, 0.11 mmol) was dissolved in CHCl₃ (10 mL). Cyanoacetic acid (91 mg, 1.07 mmol) and piperidine (100 mg, 1.18 mmol) were added and the resulting red mixture was refluxed for 8 h. The reaction was then concentrated and 10% HCl aqueous solution (40 mL) was added, and the formation of a dark precipitate was observed. The resulting dark solid was then filtered under reduced pressure washed with H₂O (100 mL). **TPA-TTh-C₆** (86 mg, 0.97 mmol, 72%) was recovered as dark red solid. m.p. > 230 °C (dec). ¹H NMR (500 MHz, DMSO) δ 8.59 (s, 2H), 8.34 (s, 2H), 7.49 (d, *J* = 8.5 Hz, 4H), 7.42 (t, *J* = 7.9 Hz, 2H), 7.21 (t, *J* = 8.0 Hz, 3H), 7.17 (d, *J* = 8.6 Hz, 4H), 2.84 (t, *J* = 7.5 Hz, 4H), 1.73 – 1.67 (m, 4H), 1.34 – 1.27 (m, 4H), 1.24 (s, 8H), 0.83 (t, *J* = 6.5 Hz, 6H). ¹³C NMR (126 MHz, CDCl₃) δ 164.11, 148.03, 148.00, 147.64, 146.70, 146.56, 136.47, 136.40, 133.18, 130.83, 130.57, 130.48, 128.07, 126.16, 123.70, 116.95, 98.29, 44.19, 31.15, 28.72, 28.39, 27.89, 22.39, 14.35. HRMS (Dual-ESI) *m/z*: [M-H]⁻ calcd for C₅₀H₄₅N₃O₄S₄, 879.2293; found 879.2263.

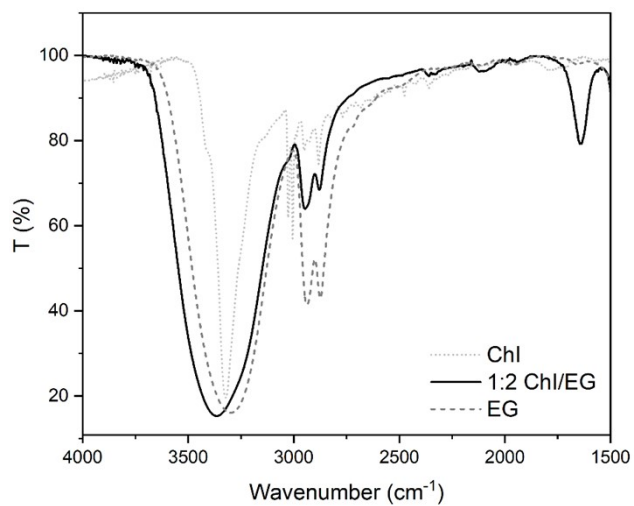


Fig. S2. FT-IR spectra of the investigated 1:2 Chl/EG mixture (solid line) and of the two pure components (dashed lines).

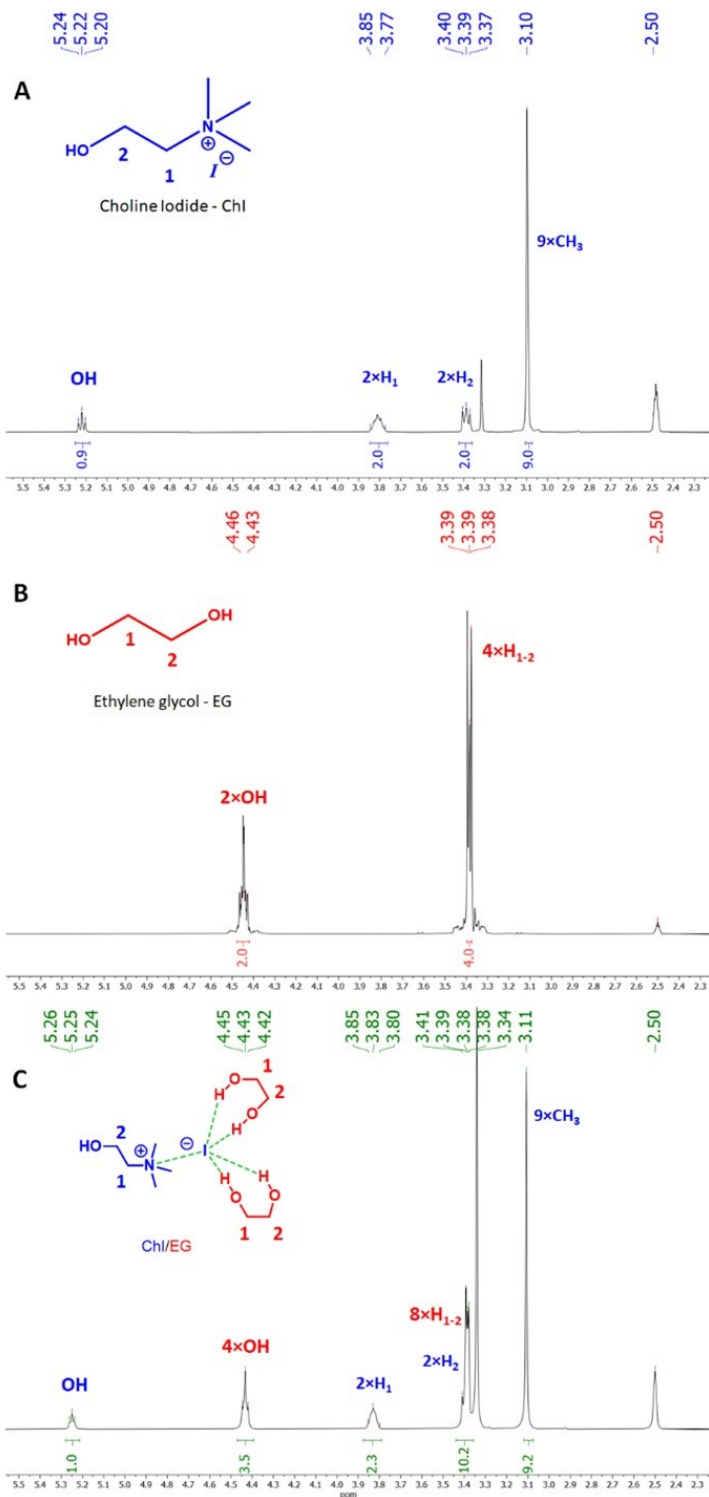


Fig. S3. ^1H -NMR spectra of (a) ChI, (b) EG, and (c) 1:2 ChI/EG mixture (^1H NMR spectra run after solubilizing 10 mg of ChI or EG or ChI/EG (1:2 mol mol⁻¹) in 0.5 mL of DMSO-d₆).

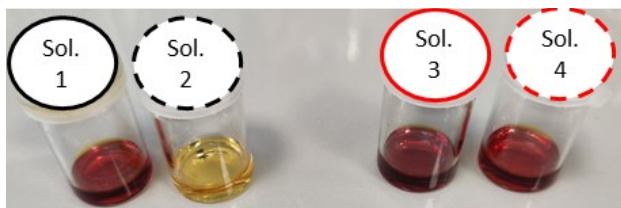


Fig. S4. Picture of the DES-based electrolyte solutions used in this work. Sol. 1: ChCl/EG 1:2 + PMII 2 M + I₂ 20 mM; Sol. 2: ChCl/EG 1:2 + I₂ 20 mM; Sol. 3: ChI/EG 1:2 + PMII 2 M + I₂ 20 mM; Sol. 4: ChI/EG 1:2 + I₂ 20 mM.

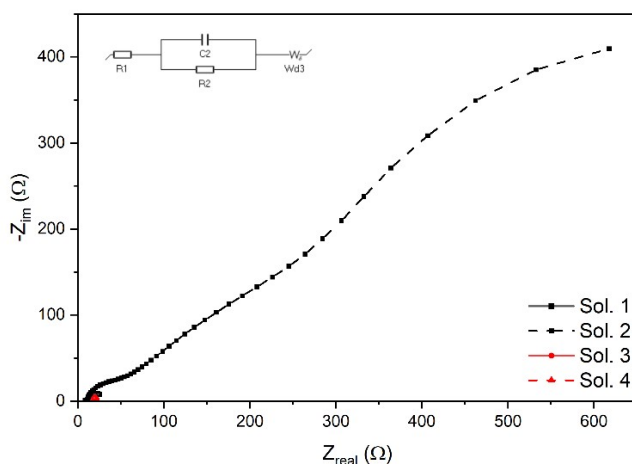


Fig. S5. Nyquist plots of dummy cells containing all the investigated DES-based solutions.

Table S2. Photovoltaic characteristics of DSSC prepared using **PTZ-Th-EG** as a sensitizer.

DES solution ^[a]	J_{sc} (mA cm ⁻²)	V_{oc} (mV)	FF	PCE (%)	J_{sc} (mA cm ⁻²)	V_{oc} (mV)	FF	PCE (%)
	GlcA 1:10 as co-adsorbent				CDCA 1:10 as co-adsorbent			
1	3.7	501	67	1.3	5.1	519	59	1.6
2	0.2	496	56	0.1	-	-	-	-
3	2.4	500	66	0.8	4.2	515	66	1.5
4	1.7	471	60	0.5	2.2	492	60	0.7

[a] I₂ concentration: 0.02 M; transparent TiO₂ thickness: 2.5 μm. Photovoltaic parameters were obtained from the champion cells. Averaged values with standard errors are presented in Table S1.

Table S3. Photovoltaic characteristics of DSSC using **PTZ-Th-C₈** as a sensitizer and **CDCA (10 CDCA:1 dye)** as a co-adsorbent.

DES solution ^[a]	J_{sc} (mA cm ⁻²)	V_{oc} (mV)	FF	PCE (%)
1	6.4	568	64	2.4
3	8.4	569	65	3.1
4	4.4	525	63	1.5

[a] I₂ concentration: 0.02 M; transparent TiO₂ thickness: 2.5 μm.

Table S4. Photovoltaic characteristics of DSSC with ChCl/EG DES using **PTZ-Th-C₈/CDCA 1:10**, with different concentrations of PMII.^[a]

PMII	J_{sc} (mA cm ⁻²)	V_{oc} (mV)	FF	PCE (%)
2 M	6.4	568	64	2.4
2.7 M	7.4	523	65	2.6
1 M	3.9	467	60	1.1

[a] I₂ concentration: 0.02 M; transparent TiO₂ thickness: 2.5 μm.

Table S5. Photovoltaic characteristics of DSSC using **TPA-TTh** as sensitizer and **CDCA** (10 CDCA:1 dye) as a co-adsorbent.

DES-like solution	J_{sc} (mA cm ⁻²)	V_{oc} (mV)	FF	PCE (%)
1	5.9	550	70	2.3
3	8.7	530	65	3.0
4	7.4	520	65	2.5

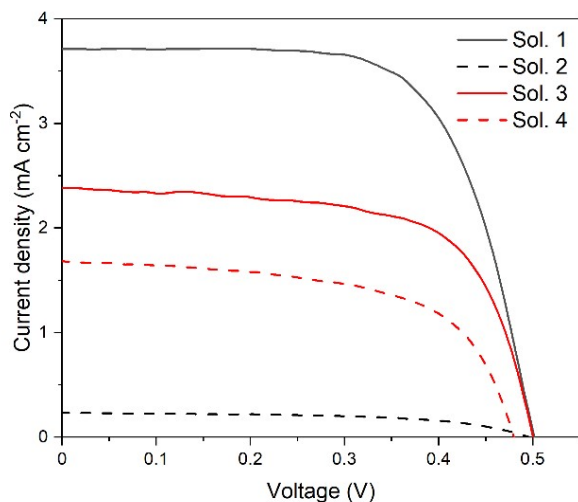


Fig. S6. J/V curves of DSSC with ChCl vs ChI DESs with **PTZ-Th-EG** as sensitizer with 1:10 **GlcA** as a co-adsorbent.

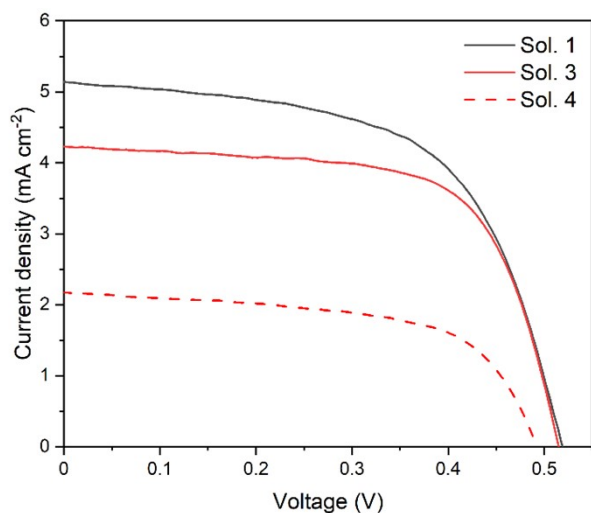


Fig. S7. J/V curves of DSSC with ChCl vs ChI DESs with **PTZ-Th-EG** as sensitizer with 1:10 **CDCA** as a co-adsorbent.

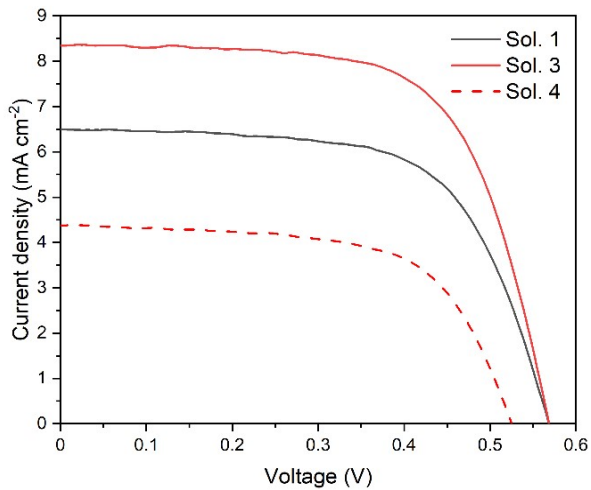


Fig. S8. J/V curves of DSSC with ChCl vs ChI DESs with $PTZ-Th-C_8$ as sensitizer with 1:10 CDCA as a co-adsorbent.

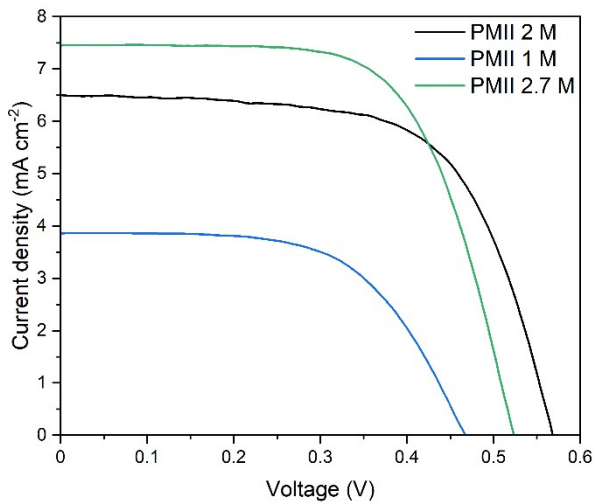


Fig. S9. J/V curves of DSSC with different concentrations of PMII in ChCl/EG 1:2 DES with $PTZ-Th-C_8$ as sensitizer with 1:10 CDCA as a co-adsorbent.

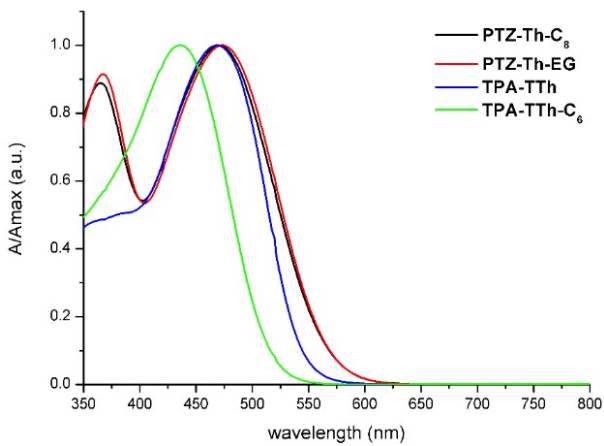


Fig. S10. Normalized UV-Vis absorption spectra of the investigated sensitizers.

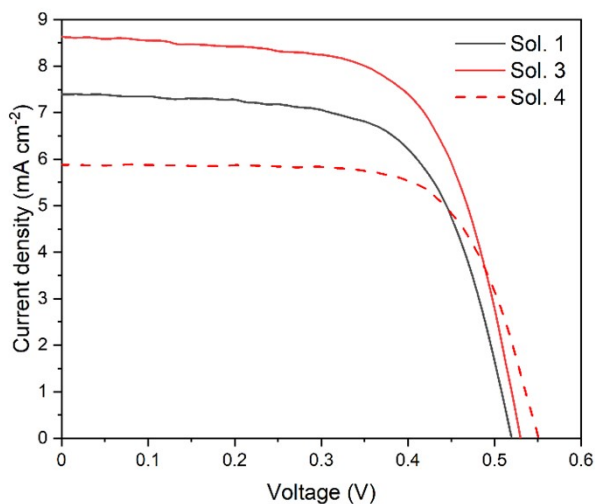


Fig. S11. J/V curves of DSSC with ChCl vs ChI DESs with TPA-TTh as sensitizer with 1:10 CDCA as a co-adsorbent.

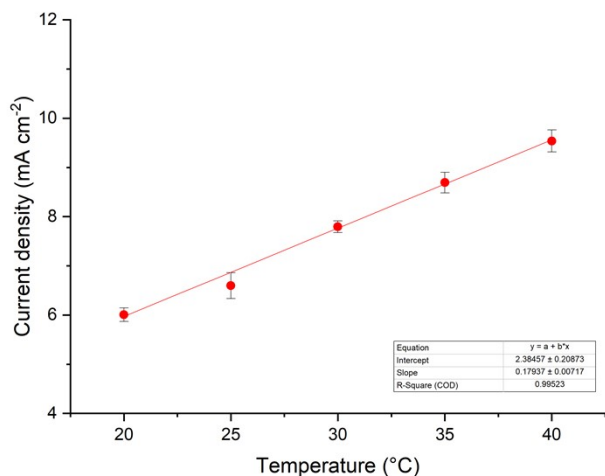


Fig. S12. Temperature dependency of the diffusion-limited current of solution 4.

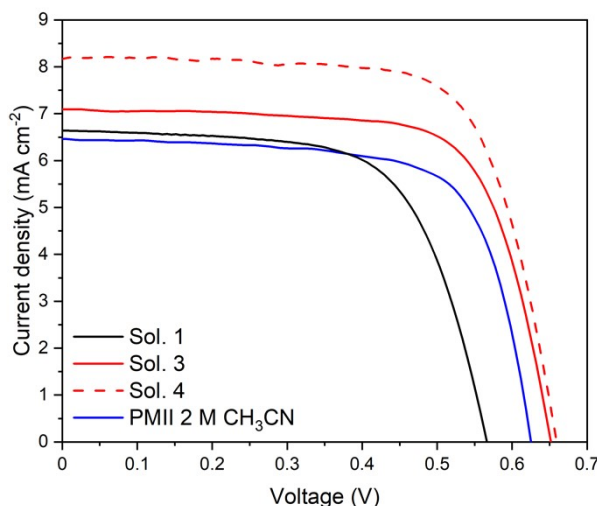


Fig. S13. J/V curves of DSSC with ChCl vs ChI DESs vs CH₃CN solution (blue line) with TPA-TTh-C₆ as sensitizer with 1:10 CDCA as a co-adsorbent.

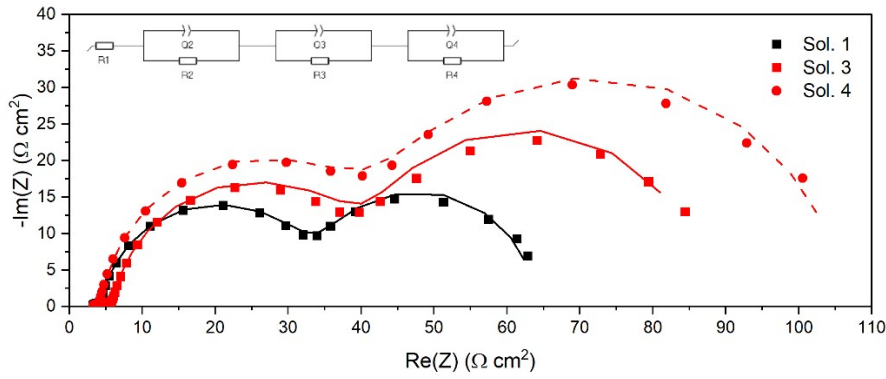


Fig. S14. EIS data plots of DSSCs described in **Table S5**. Lines represent the result of the fitting with the equivalent circuit in the inset.

Table S6. Electrochemical parameters obtained from Nyquist plots data fitting of DSSC using **TPA-TTh-C₆** as sensitizer and **CDCA** (10 CDCA:1 dye) as a co-adsorbent in dark.

DES solution	C_{μ} (mF cm ⁻²)	R_{rec} (Ω cm ²)	τ_n (ms)
1	0.40 ± 0.01	29.73 ± 0.18	11.8 ± 0.37
3	0.67 ± 0.03	35.30 ± 0.43	23.6 ± 1.34
4	0.63 ± 0.04	35.48 ± 1.49	22.4 ± 2.36

Table S7. Comparison of the electrochemical parameters obtained from Nyquist plots data fitting of DSSC using **TPA-TTh-C₆** as sensitizer and **CDCA** (10 CDCA:1 dye) as a co-adsorbent in dark and under 0.23 sun illumination. $R3=R_{rec}$ $R4=R_{diff}$. Pseudo capacitance can be calculated as $C = Q^{1/\alpha} R^{(1/\alpha) - 1}$.

Illumination	DES-like solution	R1 (Ω)	Q2 (μF · s ^{α2-1})	α2	R2 (Ω)	Q3 (μF · s ^{α3-1})	α3	R3 (Ω)	Q4 (μF · s ^{α4-1})	α4	R4 (Ω)
Dark	1	15.84 ± 0.44	159 ± 52	0.618 ± 0.002	6.84 ± 0.34	111 ± 2	0.920 ± 0.003	151.7 ± 0.9	2918 ± 48	0.990 ± 0.001	151.2 ± 1.1
	3	22.38 ± 1.63	126 ± 40	0.661 ± 0.146	8.74 ± 3.17	178 ± 5	0.918 ± 0.009	180.1 ± 2.2	3267 ± 43	1.000 ± 0.002	231.4 ± 1.5
	4	12.5 ± 4.9	15350 ± 3000	0.182 ± 0.106	23.8 ± 3.8	142 ± 10	0.963 ± 0.020	180.9 ± 7.6	1875 ± 65	0.943 ± 0.015	329.6 ± 10.7
0.23 sun	1	12.34 ± 0.46	15.9 ± 3.9	0.865 ± 0.081	5.00 ± 0.60	148 ± 3	0.905 ± 0.005	119.6 ± 0.7	15210 ± 270	0.970 ± 0.009	73.41 ± 0.81
	3	12.85 ± 0.57	56 ± 10.5	0.745 ± 0.084	6.81 ± 0.91	127 ± 4	0.902 ± 0.007	110.5 ± 1.0	9886 ± 250	0.976 ± 0.015	51.42 ± 1.05
	4	13.47 ± 0.64	40 ± 28.0	0.776 ± 0.104	5.04 ± 0.96	95.3 ± 3.0	0.908 ± 0.007	122.0 ± 1.2	9560 ± 380	0.972 ± 0.026	37.73 ± 1.57

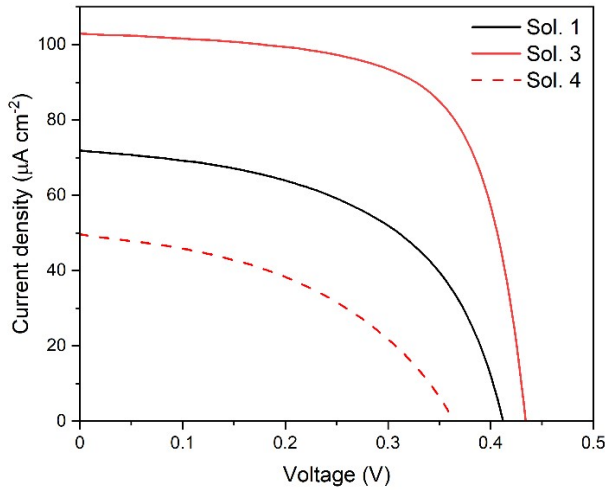


Fig. S15. J/V curves of DSSC with ChCl vs ChI DESs with **PTZ-Th-C₈** as sensitizer with 1:10 **CDCA** as a co-adsorbent under 1200 lux light.

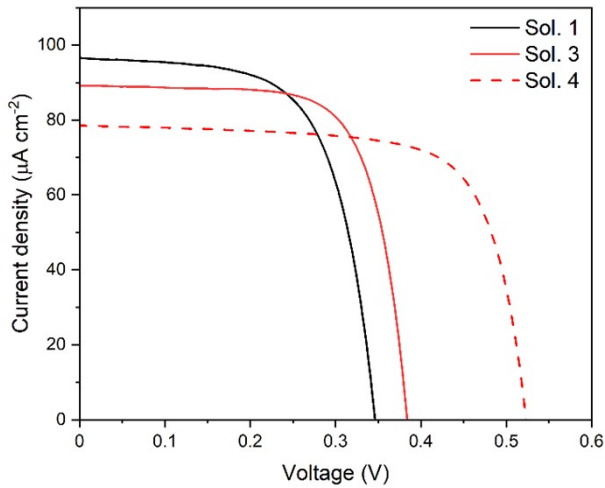


Fig. S16. J/V curves of DSSC with ChCl vs ChI DESs with **TPA-TTh-C₆** as sensitizer with 1:10 **CDCA** as a co-adsorbent under 1200 lux light.

Irradiance and illuminance determination

Since precisely measuring the power density generated by the light source is mandatory to calculate a reliable power conversion efficiency of the cells under investigation ($PCE = P_{out}/P_{in}$), we developed an easy way to measure it with instruments that are often present in solar research laboratories, avoiding the use of an expensive spectroradiometer. The only devices needed are a spectrometer able to measure the emission spectrum of the lamp in arbitrary units and a calibrated Si-photodiode, that is commonly used to calibrate External Quantum Efficiency stations.

The final aim is obtaining the irradiance spectrum $R(\lambda)$, so that, by integrating it over all the wavelengths, we can calculate the total power density of the lamp (Fig. S1). The irradiance spectrum can be seen as the product of a normalized spectrum $g(\lambda)$ (intensity between 0 and 1) and a normalization constant (K), $R(\lambda) = K g(\lambda)$. The photocurrent generated by a calibrated photodiode, when illuminated by the lamp under test at a given position, can be seen as the integral of the product of the irradiance $R(\lambda)$ and its calibration curve, $f(\lambda)$ (Fig. S16).

$$I_{mis} = \int R(\lambda)f(\lambda)d\lambda = K \int g(\lambda)f(\lambda)d\lambda$$

It is now easy to calculate K because I_{mis} can be measured, for example by connecting the photodiode to a Keithley SMU, $g(\lambda)$ can be obtained by normalizing the emission spectrum in arbitrary units and the calibration curve $f(\lambda)$ is given by the producer of the photodiode. Using software for data analysis, such as Origin, it is possible to calculate the integral value, obtain K and, finally, the desired irradiance spectrum.

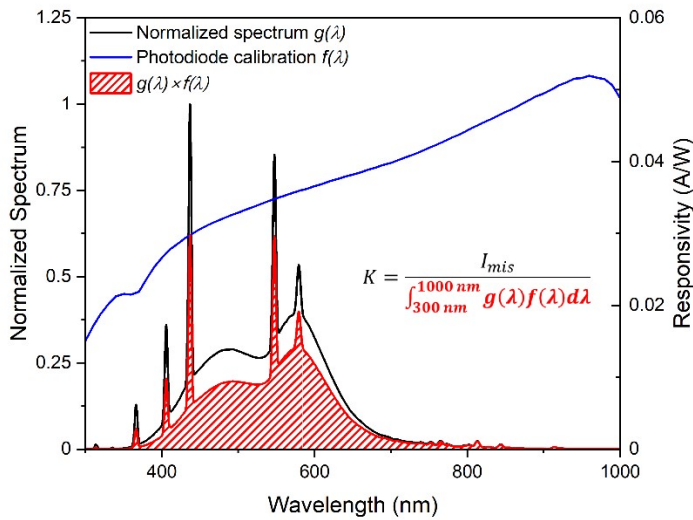


Fig. S17. Functions involved in K calculation. Lamp spectrum normalized between 0 and 1 $g(\lambda)$, black (left axis); responsivity calibration curve of the photodiode $f(\lambda)$, blue (right axis); product $g(\lambda) \times f(\lambda)$, red (right axis).

From the irradiance it is also possible to accurately calculate the illuminance in lux. It is enough to multiply the irradiance $R(\lambda)$ by the Photopic luminous efficiency function $V(\lambda)$, which is tabulated, then integrate the product and multiply it by the maximum spectral luminous efficacy for human photopic vision, that is 683 lm/W.

$$lux = 683 \frac{lm}{W} \int_{360 \text{ nm}}^{830 \text{ nm}} R(\lambda)V(\lambda)d\lambda$$

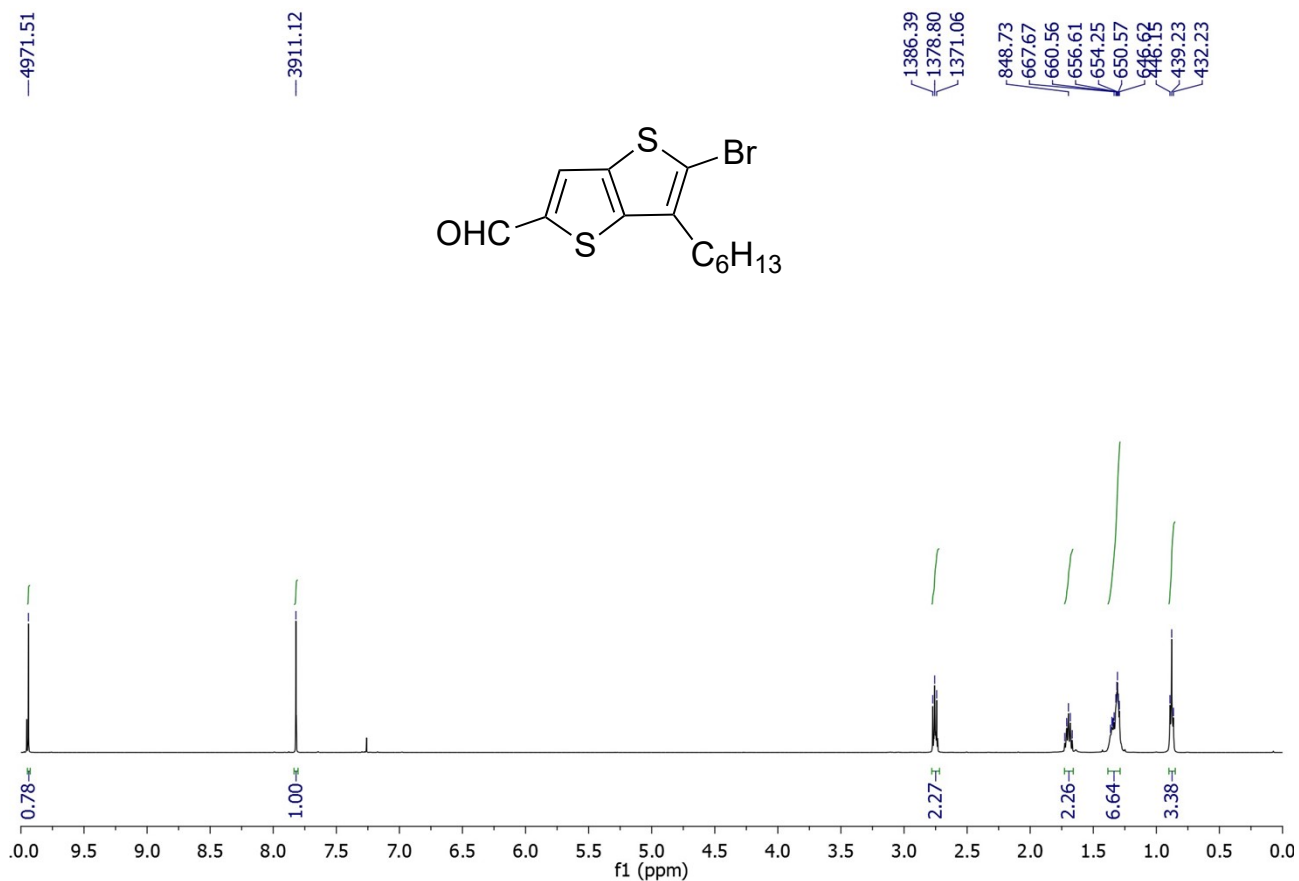


Fig. S18. $^1\text{H-NMR}$ of compound **1** in CDCl_3 .

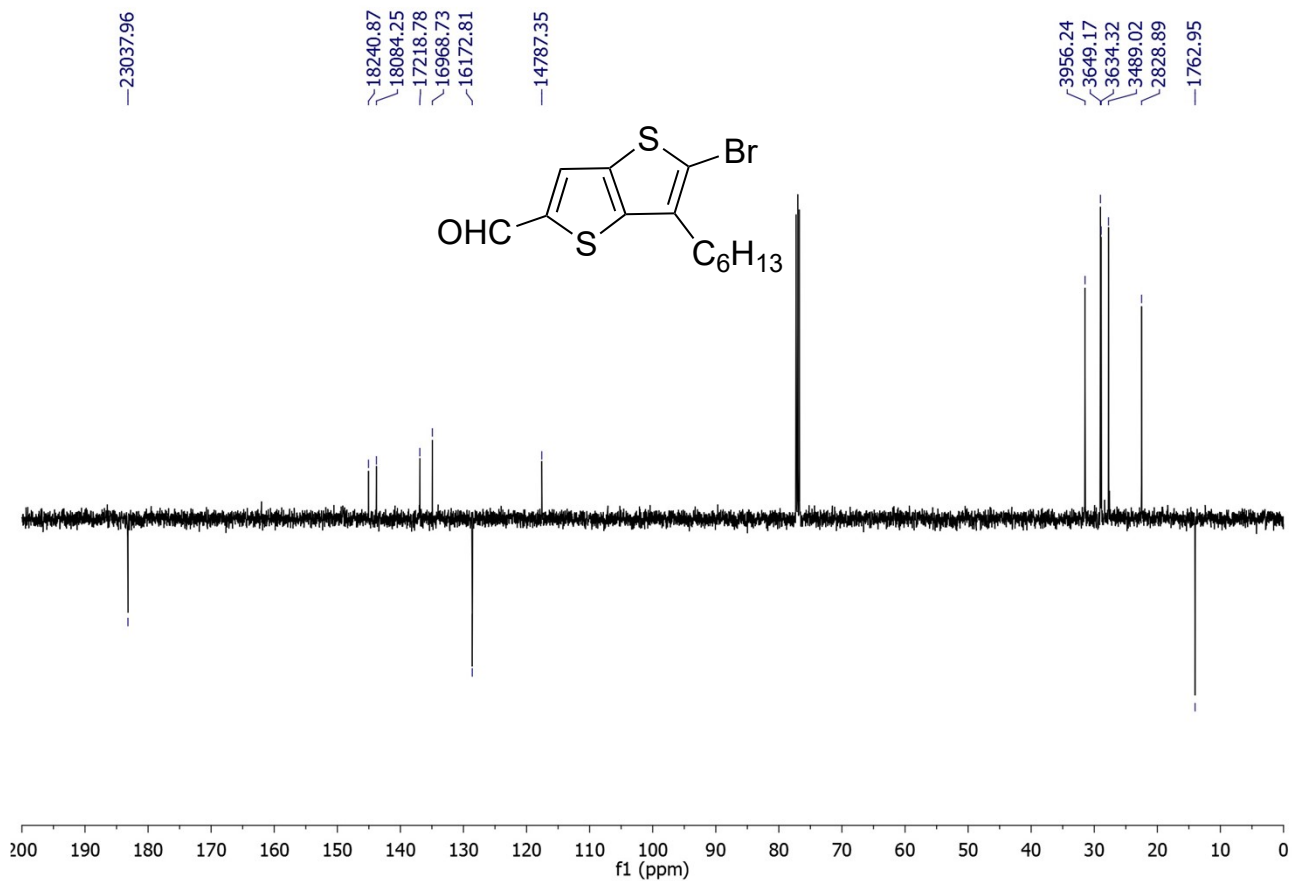


Fig. S19. $^{13}\text{C-NMR}$ of compound **1** in CDCl_3 .

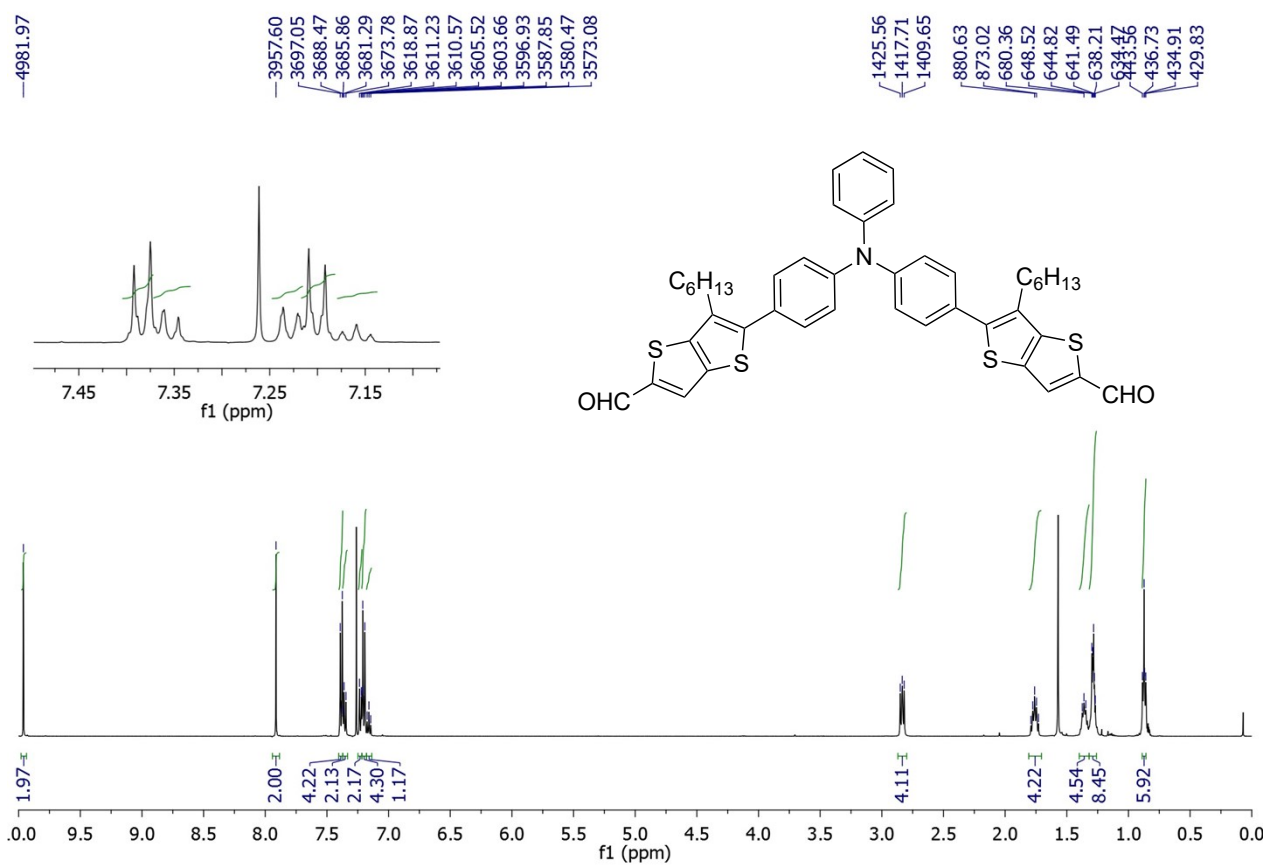


Fig. S20. ¹H-NMR of compound 2 in CDCl₃.

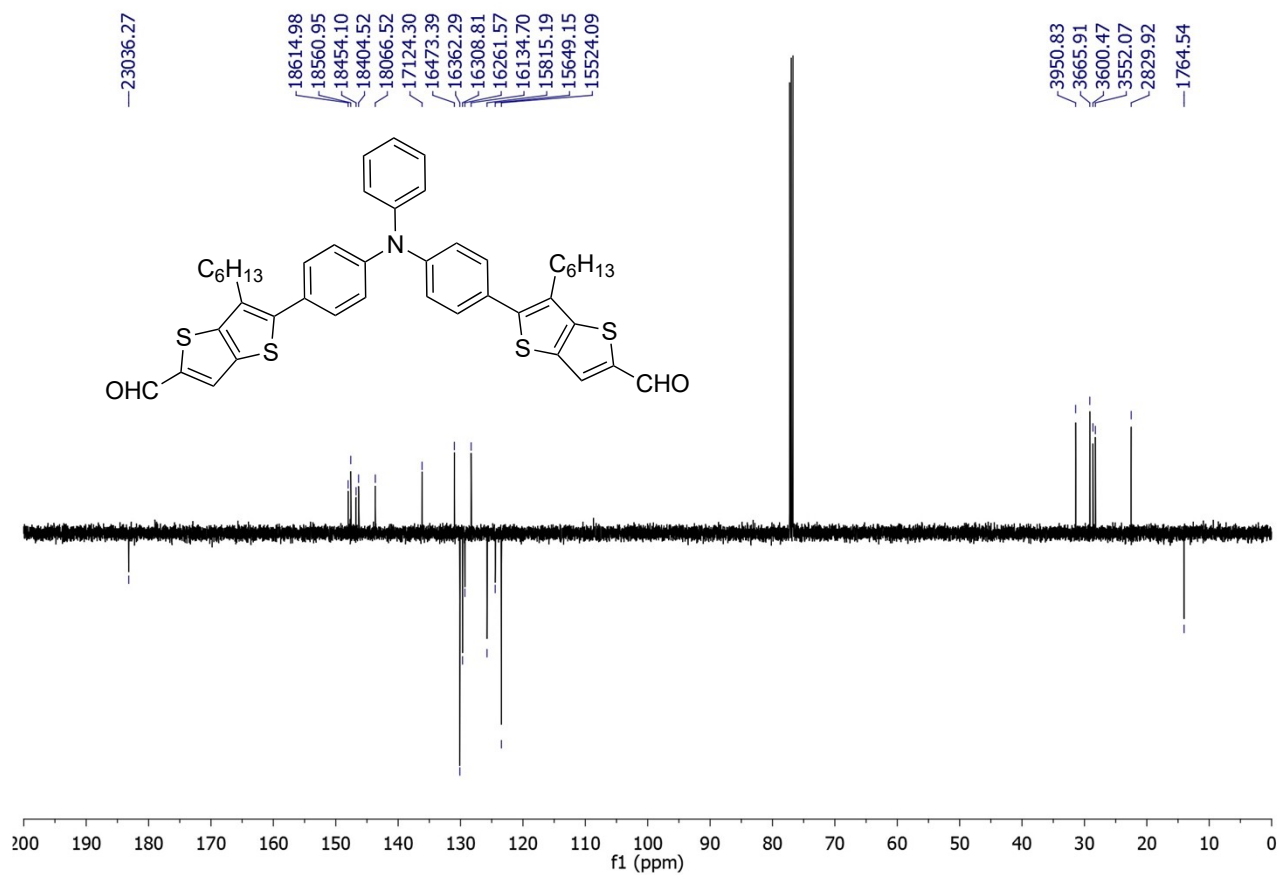


Fig. S21. ¹³C-NMR of compound 2 in CDCl₃.

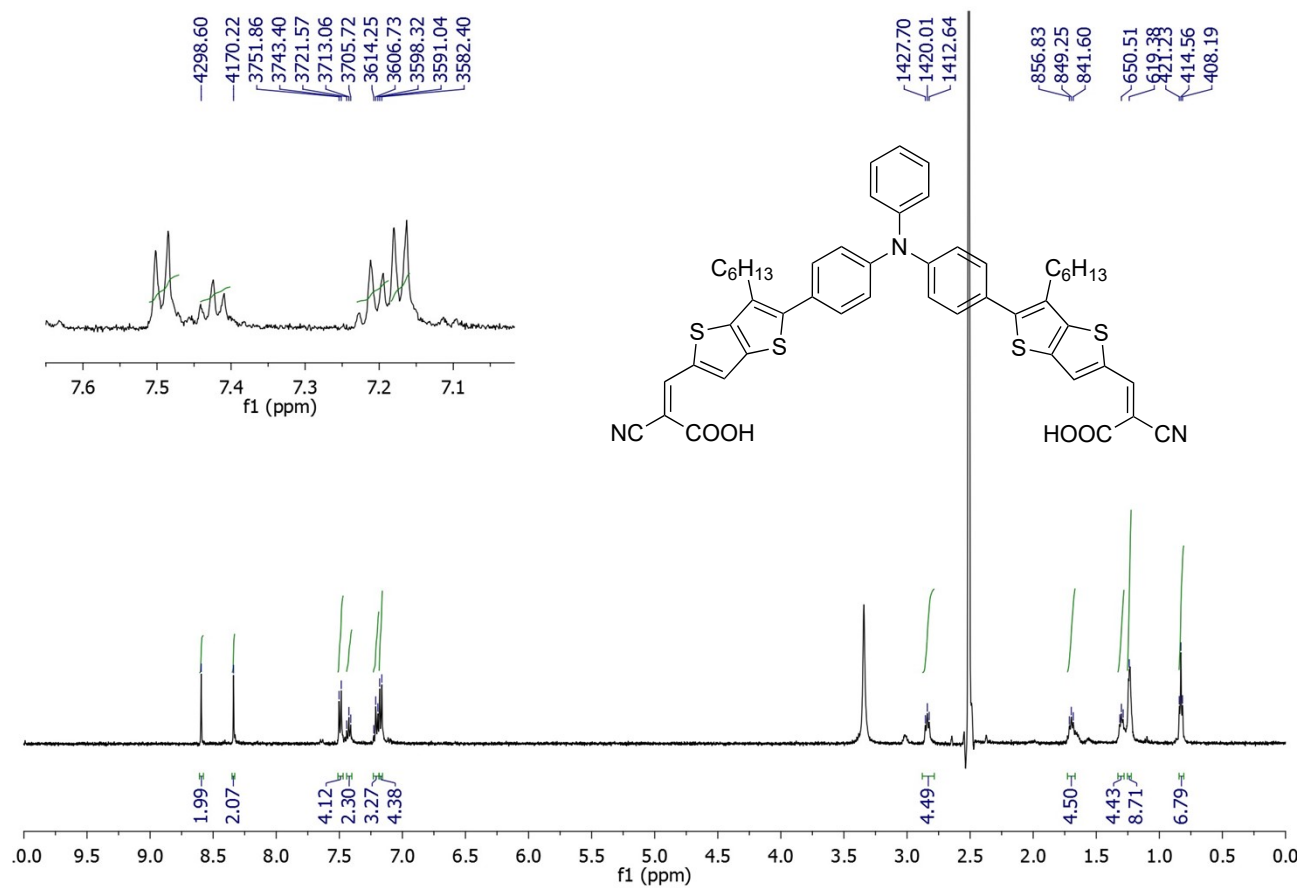


Fig. S22. ¹H-NMR of compound TPA-TTh-C₆ in DMSO-d₆.

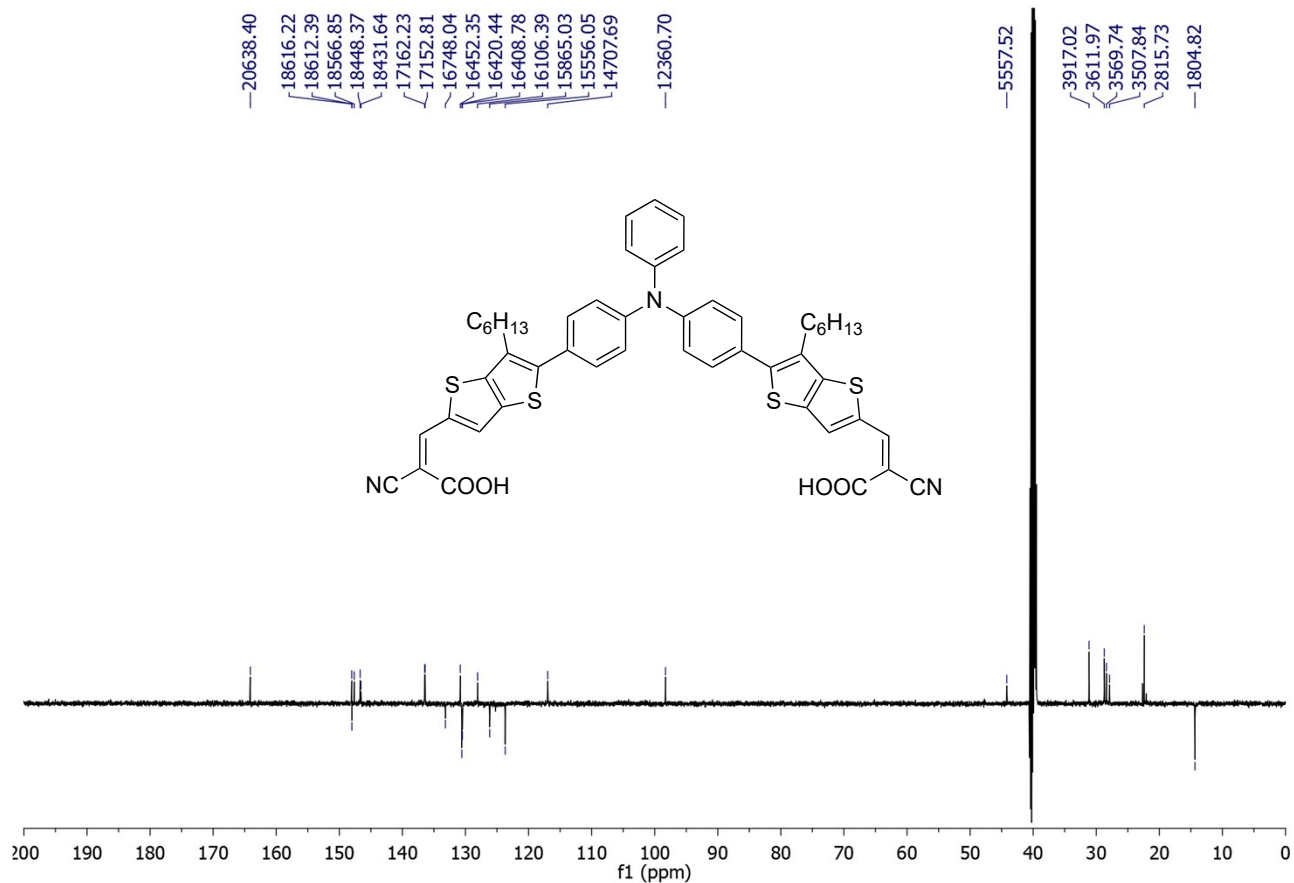


Fig. S23. ^{13}C -NMR of compound TPA-TTh- C_6 in $\text{DMSO-}d_6$.