

# Supplementary Information

## Insight into effects of the anchoring groups on the photovoltaic performance of unsymmetrical phthalocyanine based dye-sensitized solar cells

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## 1. Materials

Chemicals were purchased from commercial suppliers and used without further purification unless stated otherwise. The monitoring of the reactions was carried out by thin layer chromatography (TLC), employing aluminum sheets coated with silica gel type 60 F254 (0.2 mm thick, E. Merck). Purification and separation of the synthesized products was performed by column chromatography using silica gel (230–400 mesh, 0.040– 0.063 mm, Merck). Eluents and relative proportions of the solvents are indicated for each particular case. Column chromatography was carried out on silica gel Merck-60 (230–400 mesh, 60 A), and TLC on aluminum sheets pre-coated with silica gel 60 F254 (E. Merck). Size exclusion chromatography was carried out on Bio-Rad Bio-Beads S-X1 beads (200–400 mesh) with tetrahydrofuran (THF) as the eluent. Fluorine-doped SnO<sub>2</sub> conducting glass (FTO, sheet resistance 15 Ω/sq), TiO<sub>2</sub> paste (Ti-nanoxide T/SP), platinum paste (Platisol T/SP), sealing film (Surlyn, Meltonix 1170–25) and redox electrolyte (Iodolyte HI-30) containing 30 mM iodide/tri-iodide in acetonitrile and additives of ionic liquid were purchased from Solaronix. Chenodeoxycholic acid (CDCA), tetrabutylammonium tetrafluoroborate (TBABF<sub>4</sub>), tetrahydrofuran (THF), ferrocene/ferrocenium (Fc/Fc<sup>+</sup>) were also purchased from Sigma-Aldrich.

## 2. Instruments

Elemental analyses were obtained using a Carlo Erba 1106 Instrument. Infrared spectra were recorded between 4000–650 cm<sup>-1</sup> using a Perkin Elmer FT-IR System Spectrum BX spectrometer with an attenuated total reflection (ATR) accessory featuring a zinc selenide (ZnSe) crystal. <sup>1</sup>H NMR spectra were recorded on Bruker AVANCE III-HD and Varian INNOVA 500 MHz spectrometers. MALDI-TOF- MS measurements were performed on a Bruker Daltonics MicrOTOF spectrometer. Positive ion and linear mode MALDI-TOF-MS spectra of the compounds were obtained in 2,5-dihydroxy benzoic acid (DHB) or dithranol (DIT) MALDI matrixes using nitrogen laser accumulating 50 laser shots. Mass spectra of some

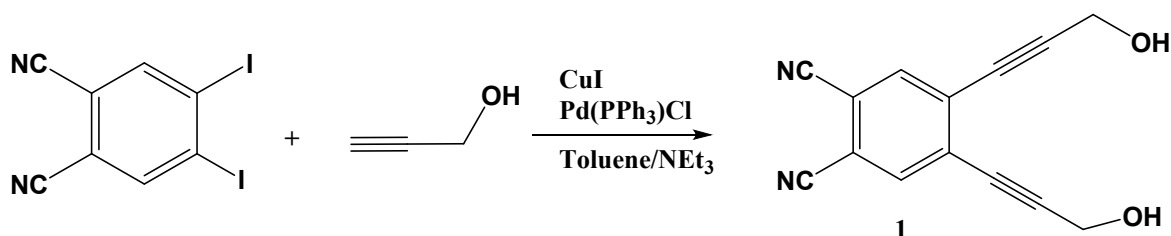
phthalonitrile derivatives were obtained using Thermo Scientific- TSQ Fortis mass spectrometer by using ESI technique. The absorption spectra of the dyes and the sensitized films were measured using Shimadzu UV 2600 spectrophotometer. The dye loading amount was calculated by the absorbance of the dyes desorbed from the dye-sensitized photoanode using a 0.1 M NaOH solution in THF/H<sub>2</sub>O (1/1, v/v). Cyclic voltammetry experiments for the dyes were performed by using a potentiostat/galvanostat (PARSTAT 2273, Princeton Applied Research) with a three-electrode cell with a glassy carbon as working electrode, Pt disk as reference electrode, Pt wire as counter electrode at a scan rate of 0.050 V s<sup>-1</sup>. The electrolyte solution contains 2 mM dye and 0.1 M TBABF<sub>4</sub> in DMSO, and the Fc/Fc<sup>+</sup> redox couple was used as an external standard. The half-wave potential ( $E_{1/2}$ ) of the Fc/Fc<sup>+</sup> was found to be 0.34 V versus the reference electrode. The onset oxidation potentials ( $E_{ox}$ ) measured versus the reference electrode were converted to Fc/Fc<sup>+</sup> by subtraction of 0.34 V. The  $E_{ox}$  calculated from intersection of two tangents drawn at rising current and background charging current of a cyclic voltammograms and these oxidation potentials are related to the HOMO levels<sup>1</sup>.

### **3. Preparation and characterization of DSSCs**

For DSSCs fabrication, two transparent TiO<sub>2</sub> pastes were deposited using Doctor Blade's method on the conductive side of FTO substrate. In order to prepare the dye-sensitized TiO<sub>2</sub> films (photoanodes), the coated FTO substrates were immersed into a 0.3 mM dye solution in EtOH/THF (1/1, v/v) for 5 h. 10 mM chenodeoxycholic acid (CDCA) as a coadsorbent was also introduced into the dye solution. For comparison, the TiO<sub>2</sub> film was also immersed in a solution made of 0.3 mM a zinc phthalocyanine dye (TT1) and 10 mM CDCA in EtOH/THF for 5 h. The Pt counter electrodes were prepared on the FTO substrates by casting platinum paste solution. Both the photoanode and counter electrode were sealed with Surlyn film. The redox electrolyte was injected between the electrodes through a drilled hole at the counter electrode. The current density-voltage ( $J-V$ ) characteristics were investigated by using the

potentiostat/galvanostat under AM 1.5 global one sun illumination ( $100 \text{ mW cm}^{-2}$ ) from a solar simulator (96000, Newport). During the measurements, a black mask of an area of  $0.196 \text{ cm}^2$  was covered on the cells. The incident photon-to-current conversion efficiency (IPCE) of the DSSCs were carried out a monochromator (74004, Oriel) from the region from 350 to 800 nm. The electrochemical impedance spectroscopy (EIS) of the solar cells were recorded with the potentiostat/galvanostat, under dark conditions, at a forward bias  $-0.45 \text{ V}$  in the frequency range  $0.1 \text{ Hz}-10^5 \text{ Hz}$ .

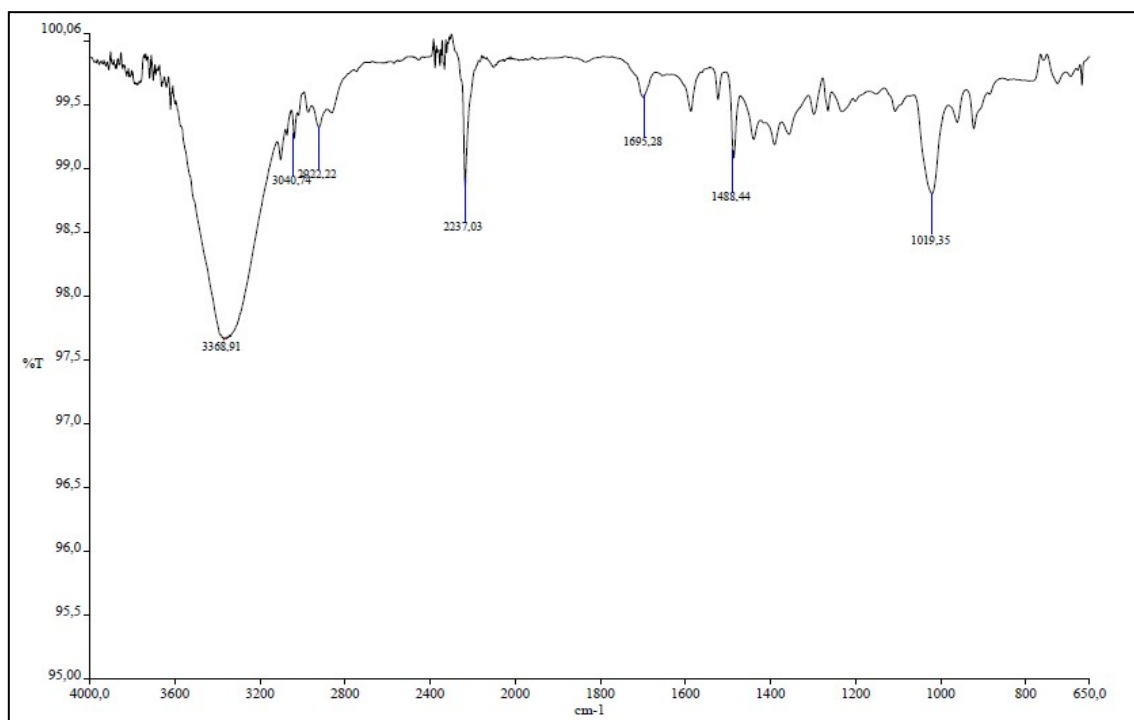
#### 4. Synthesis and characterization of compound 1



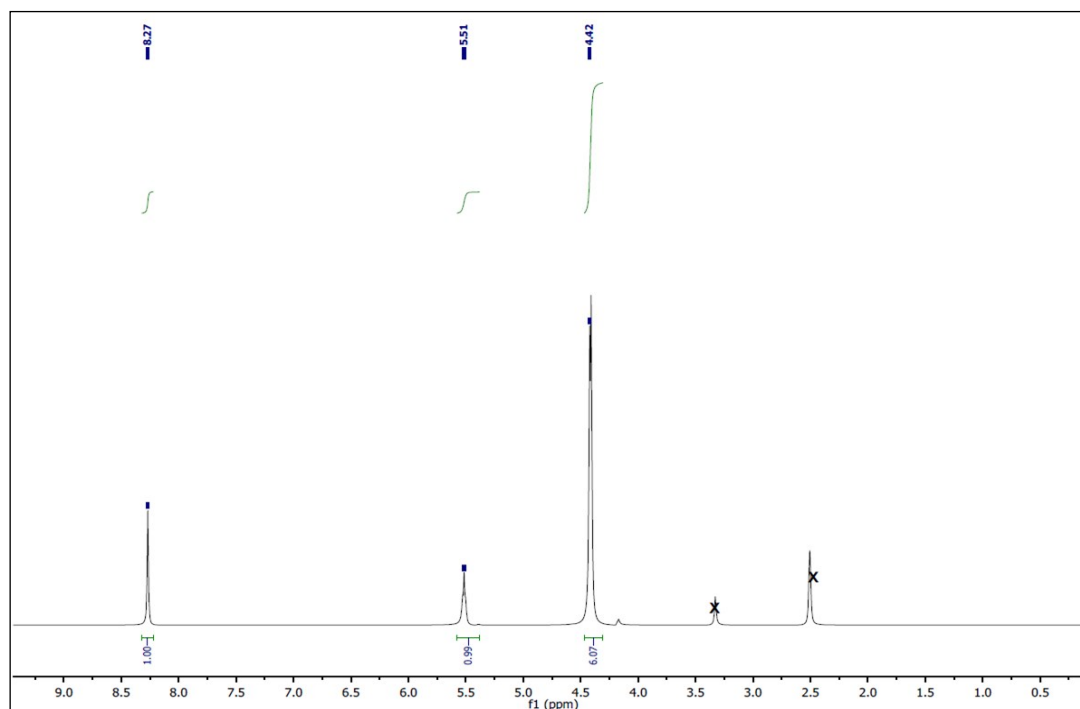
**Scheme S1.** Synthesis of propargylic alcohol substituted phthalonitrile **1**

**Synthesis of 4,5-bis(3-hydroxyprop-1-yn-1-yl) phthalonitrile (1):** Compound **1** synthesized in accordance the literatures.<sup>1,2</sup> 4,5-diiido-phthalonitrile, (500 mg, 1.31 mmol) CuI (15.81 mg, 0.083 mmol) and Pd(PPh<sub>3</sub>)Cl<sub>2</sub> (136.87 mg, 0.195 mmol) were dissolved in 10 ml dry toluene under argon atmosphere and added 5 ml triethyl amine (TEA) by stirring. Propargyl alcohol (295.18 mg, 5.26 mmol) was added and the reaction mixture was stirred at  $90^\circ\text{C}$  for 24h. Then, the reaction mixture was filtered and evaporated. After, the solid materials obtained was purified by column chromatography over silica gel using (CH<sub>2</sub>Cl<sub>2</sub>/EtOH (20/1) mixtures as eluent) to afforded **1** (120 mg, 31%) as a brown-green solid. FT-IR  $\nu_{\text{max}}$  (cm<sup>-1</sup>): 3368, (OH), 2237,03 (-C≡N), 1695, 1488, 1019. <sup>1</sup>H-NMR (500 MHz, DMSO-*d*<sub>6</sub>,  $\delta$  ppm): 8.27(s, 2H, ArH), 5.51 (br s, 2H, OH), 4.42 (d, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR (500 MHz, DMSO-*d*<sub>6</sub>,  $\delta$  ppm): 137.07 (ArC). 130.22 (ArC), 115.61 (ArC), 114.57(C≡N), 100.92 (C≡C), 80.40 (C≡C), 49.92 (-CH<sub>2</sub>). ESI-

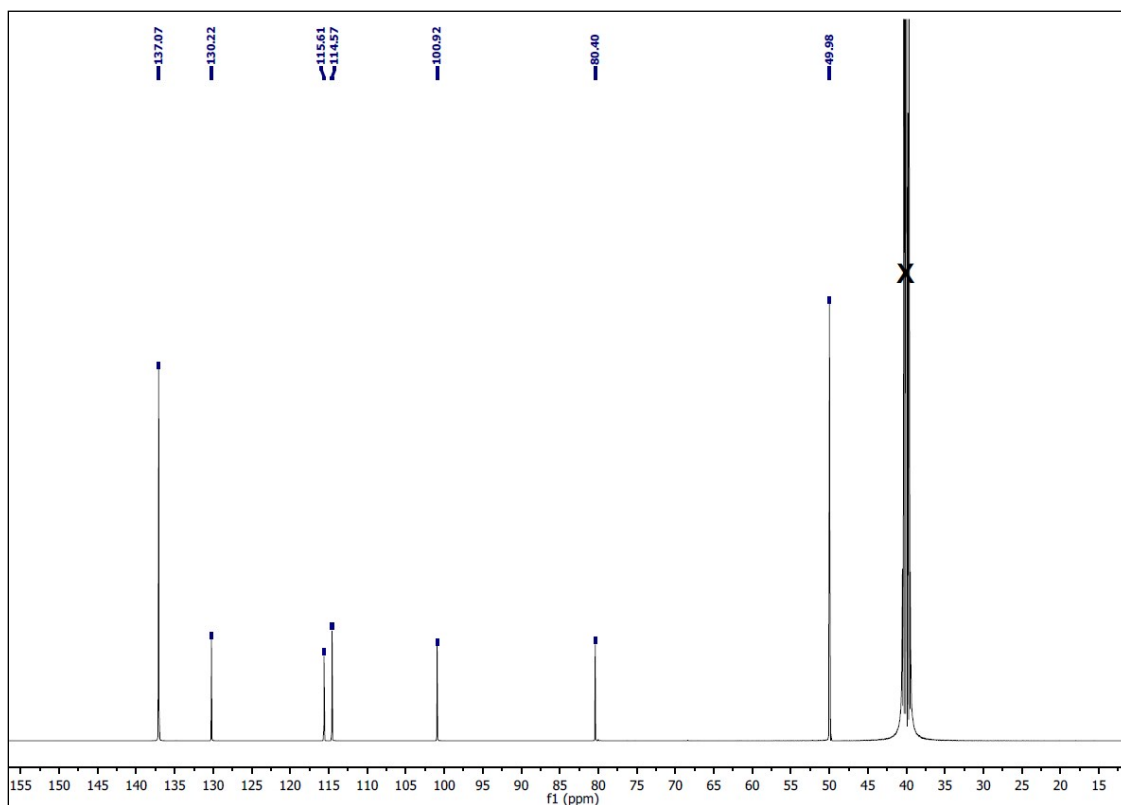
MS: calcd: 236.23; found:281.10  $[M+2Na]^+$ , 298.0  $[M+Na+K]^+$ ,315.0  $[M+2K]^+$ ,  
363 $[M+2Na+2K]^+$



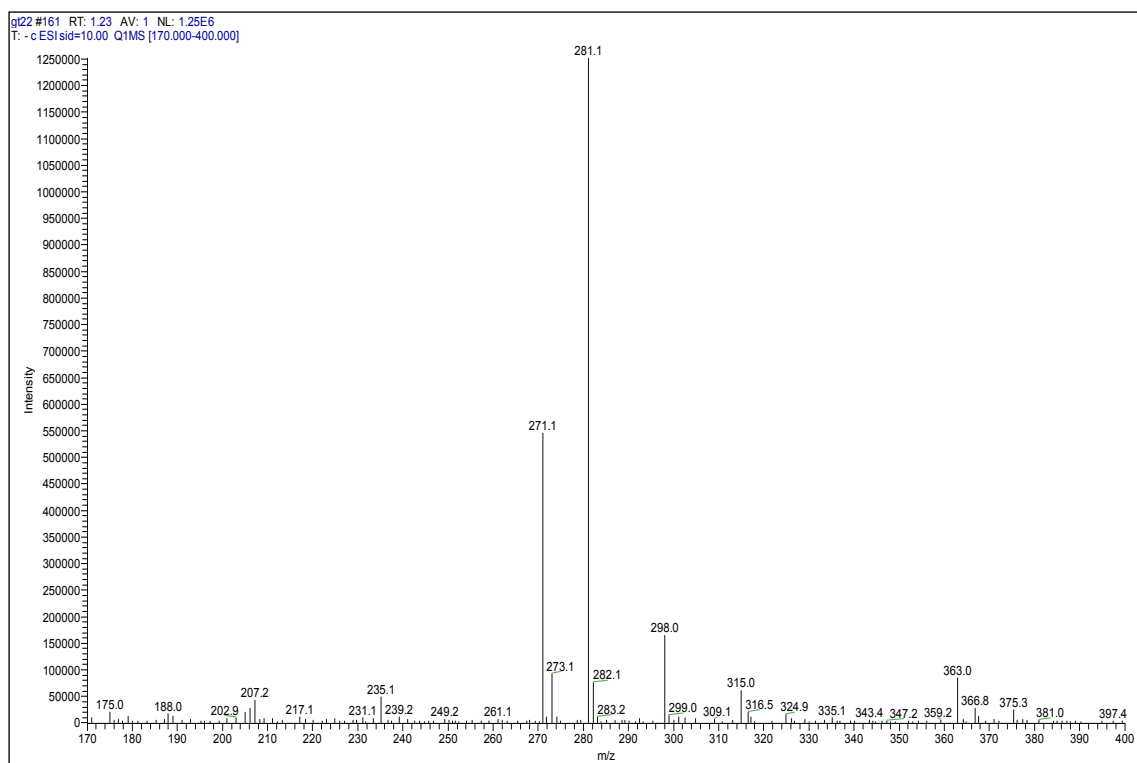
**Fig. S1.** FT-IR spectrum of compound **1**.



**Fig. S2.** <sup>1</sup>H-NMR spectrum of compound **1** (in DMSO-*d*<sub>6</sub>).



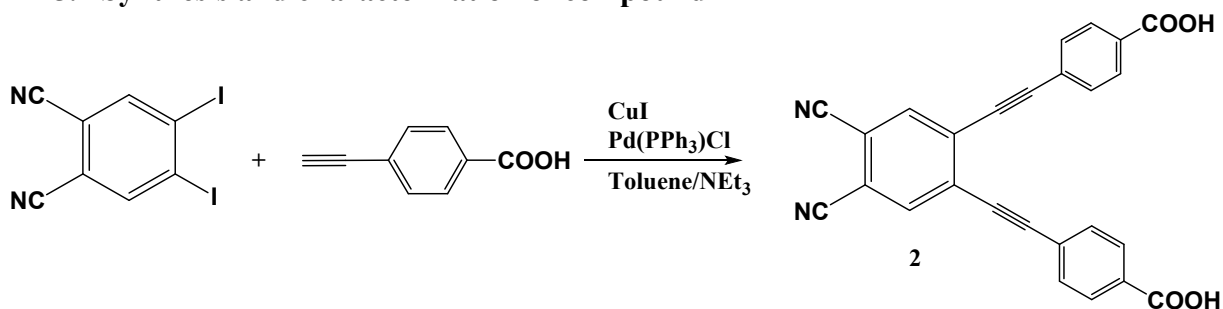
**Fig. S3.**  $^{13}\text{C}$ -NMR spectrum of compound **1** (in  $\text{DMSO}-d_6$ ).



**Fig. S4.** ESI-MS spectrum of compound **1**.

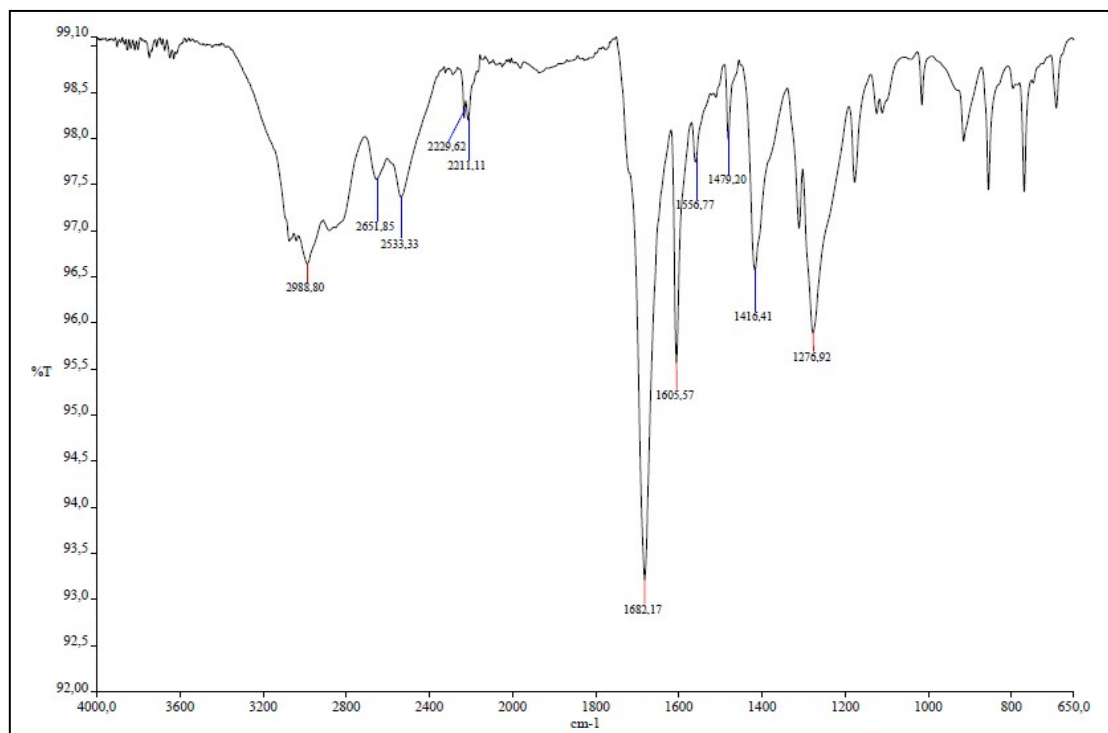


## 5. Synthesis and characterization of compound 2

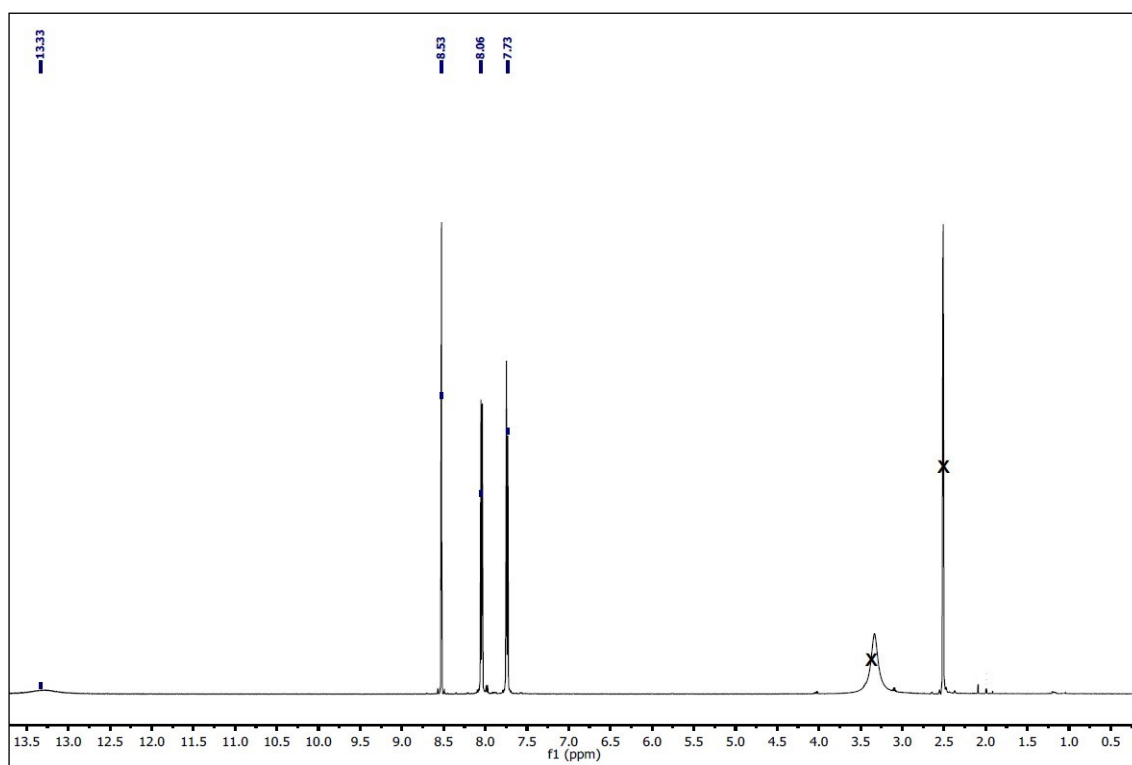


### Synthesis of 4,4'-((4,5-dicyano-1,2-phenylene) bis(ethyne-2,1-diyl))dibenzoic acid (2):

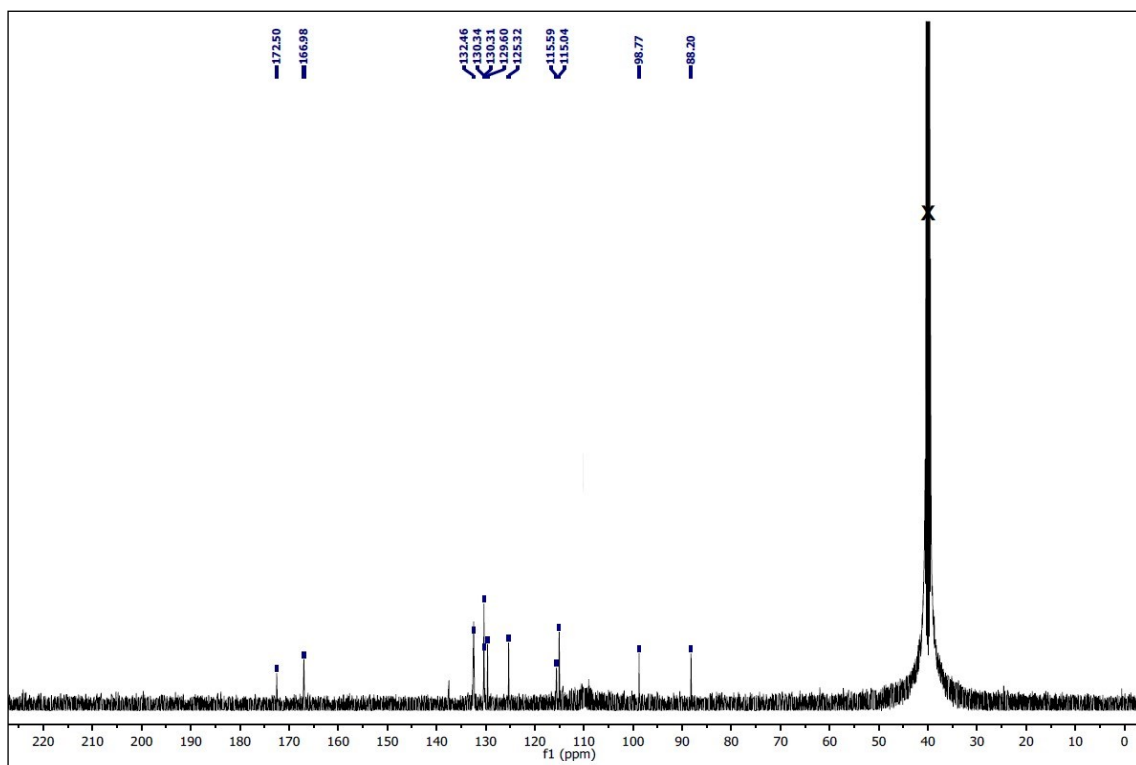
Compound 2 was synthesized in accordance with the literature.<sup>2,3</sup> 4,5-diiodo-phthalonitrile (500 mg, 1.31 mmol), CuI (15.81 mg, 0.083 mmol), and Pd(PPh<sub>3</sub>)Cl<sub>2</sub> (136.87 mg, 0.195 mmol) were dissolved in 10 ml dry toluene under argon atmosphere and 5 ml triethylamine (TEA) was added by stirring. 4-ethynyl benzoic acid (765.24 mg, 5.24 mmol) was added. The reaction mixture was stirred and refluxed at room temperature for 24 h. Then, the reaction mixture was added 20 ml dichloromethane and evaporated. After, the solid material obtained was extracted with Et<sub>2</sub>O (100 ml x 3). The organic layer was precipitated with a mixture of dichloromethane and hexane to obtain 2 (125 mg, 22%) as a yellow solid. FT-IR  $\nu_{\max}$  (cm<sup>-1</sup>): 3100-2500, (-OH broad), 2988, (ArCH) 2229 (-C≡N), 2211 (-C≡C), 1682 (C=O), 1605, 1556, 1479, 1416, 1276. <sup>1</sup>H-NMR (500 MHz, DMSO-d<sub>6</sub>,  $\delta$  ppm): 13.23 (br s, 2H, OH), 8.53 (s, 2H, ArH), 8.06 (d, 4H, ArH), 7.73 (d, 4H, ArH). <sup>13</sup>C NMR (500 MHz, DMSO-d<sub>6</sub>,  $\delta$  ppm): 172.50 (C=O), 166.98 (ArC), 132.46 (ArC), 130.34 (ArCH), 130.31 (ArCH), 129.60 (ArCH), 125.32 (ArC), 115.59 (C≡N), 115.04 (ArC), 98.77 (C≡C), 88.20 (C≡C). ESI-MS: calcd: 416.4; found: 415.2 [M-H]<sup>+</sup>



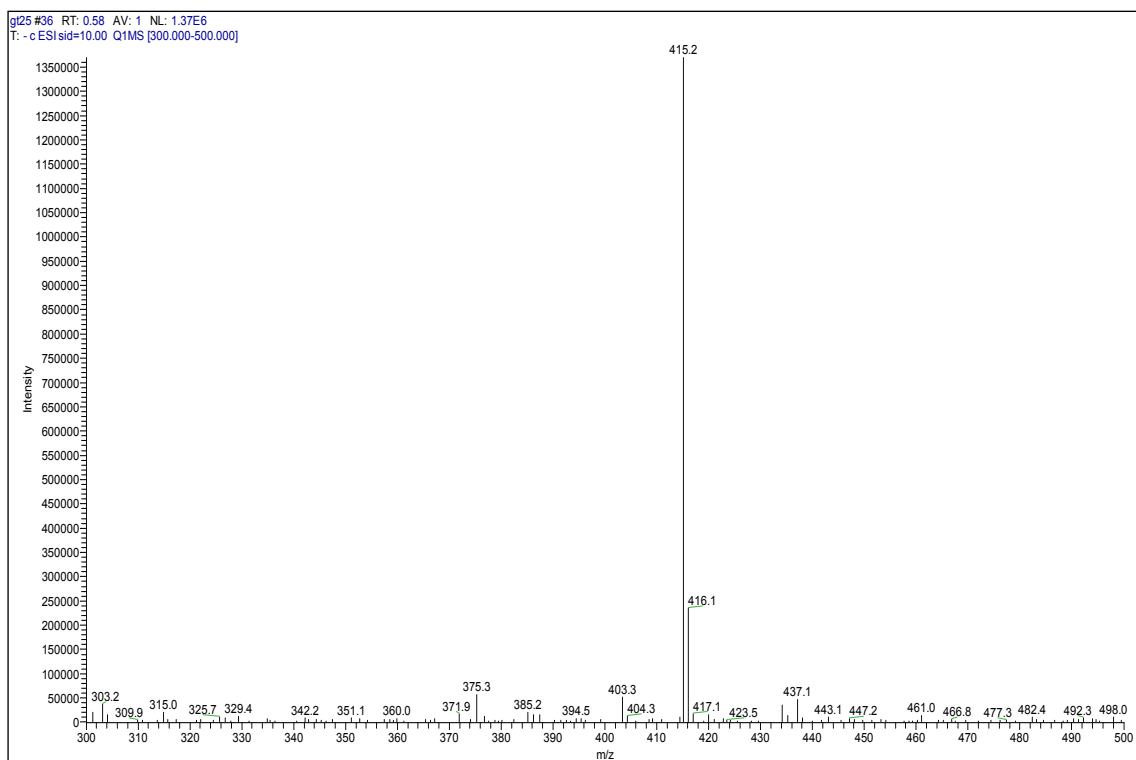
**Fig. S5.** FT-IR spectrum of compound **2**.



**Fig. S6.** <sup>1</sup>H-NMR spectrum of compound **2** (in DMSO-*d*<sub>6</sub>).

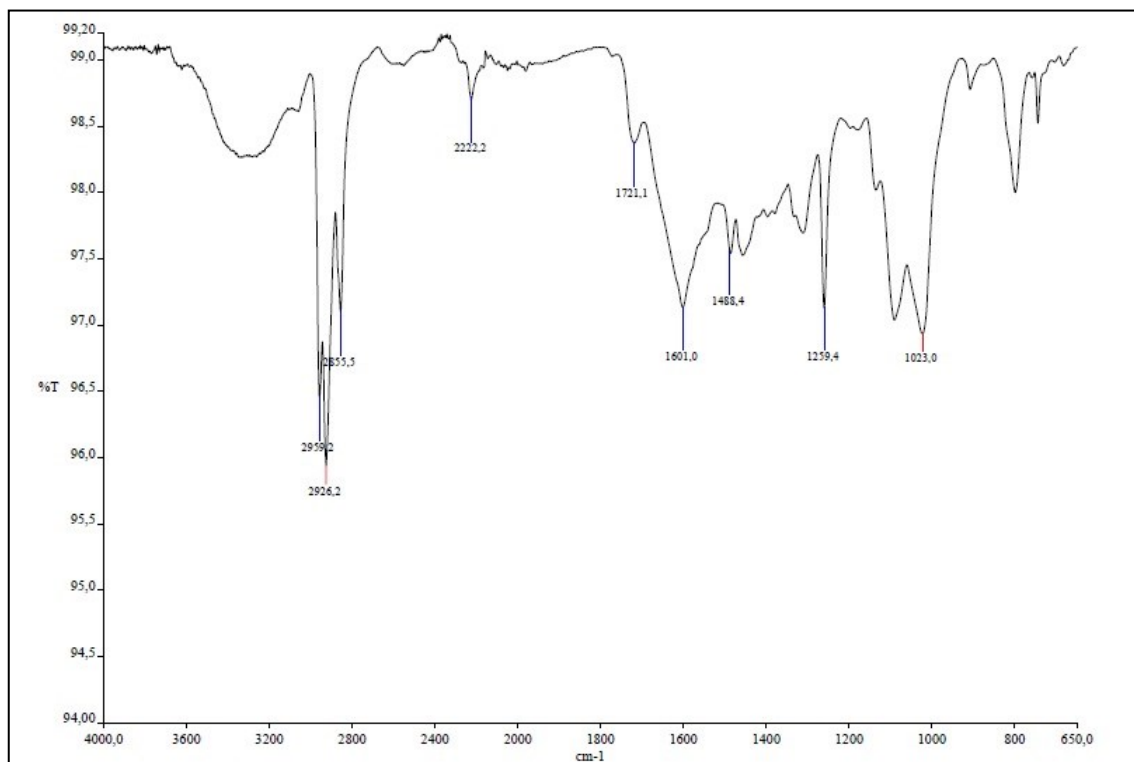


**Fig. S7.**  $^{13}\text{C}$ -NMR spectrum of compound **2** (in  $\text{DMSO}-d_6$ ).

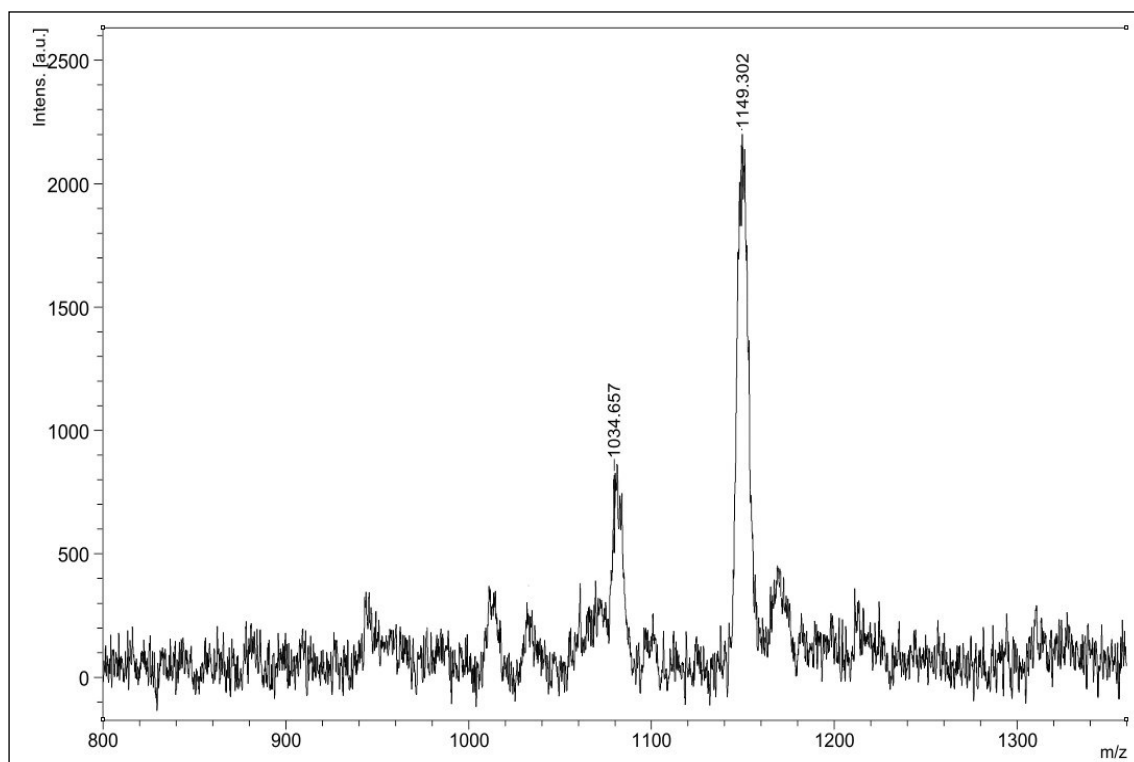


**Fig. S8.** ESI-MS spectrum of compound **2**.

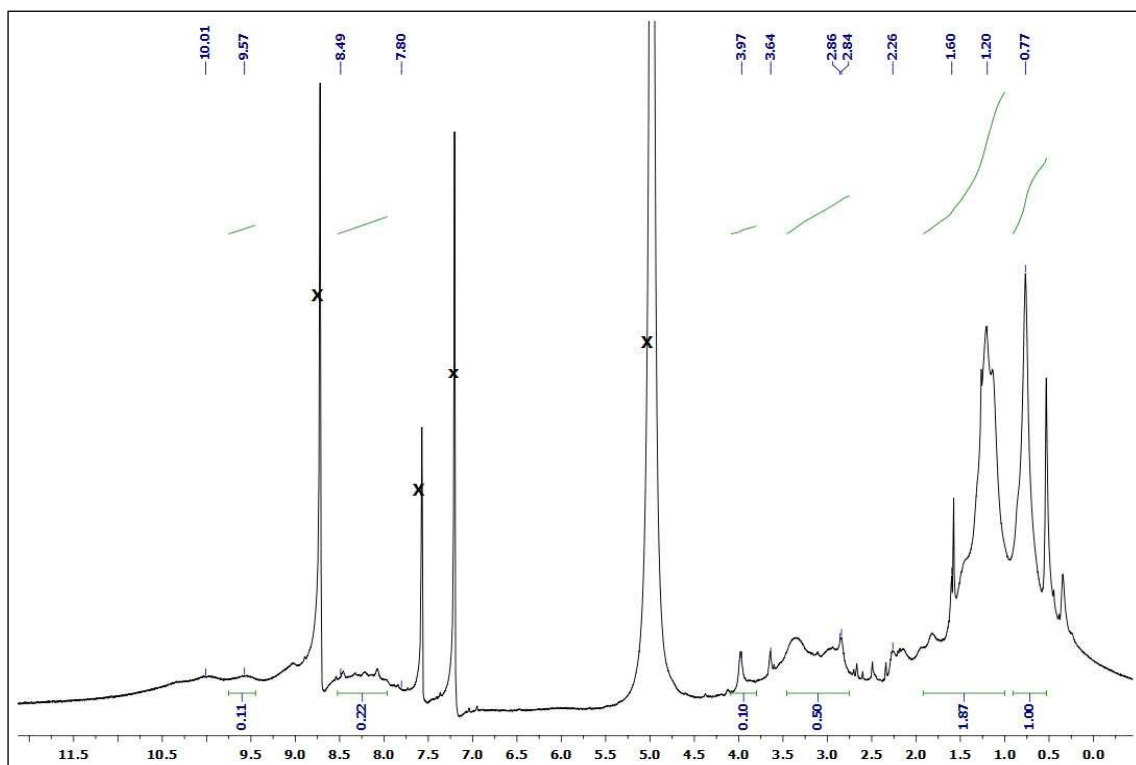
## 6. Characterization of compound 5



**Fig. S9.** FT-IR spectrum of compound 5.

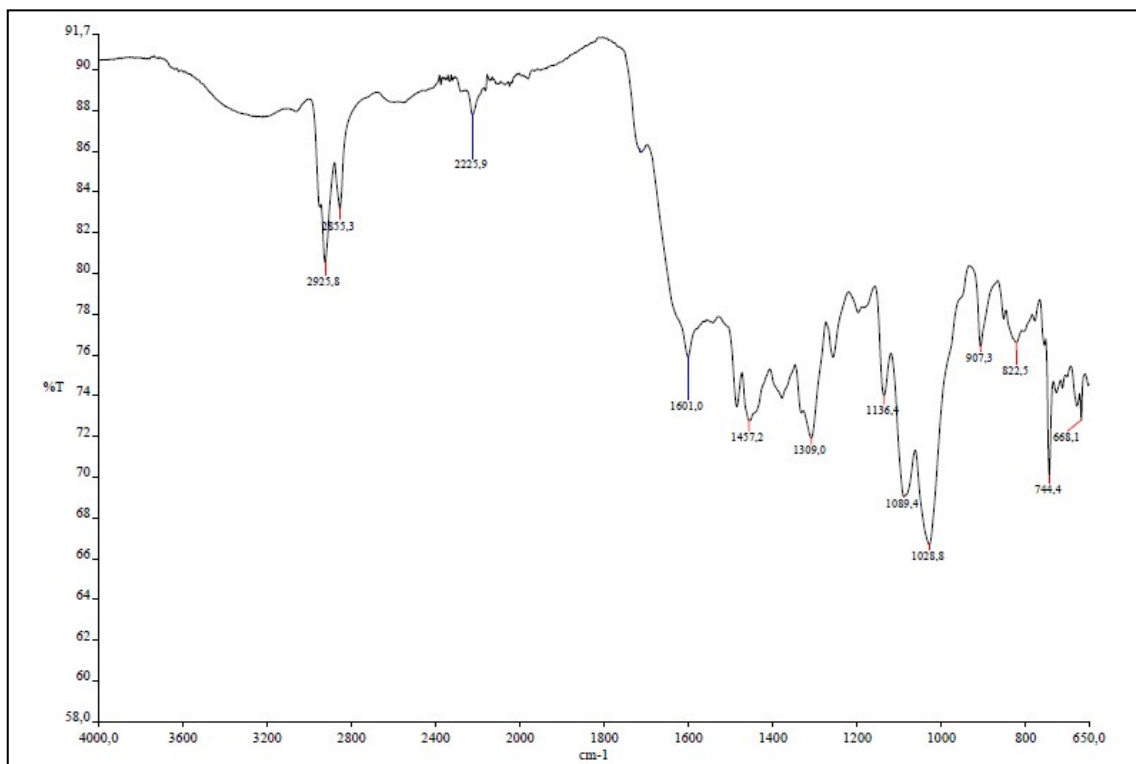


**Fig. S10.** MS (MALDI-TOF) spectrum of compound 5 (Matrix: DIT).

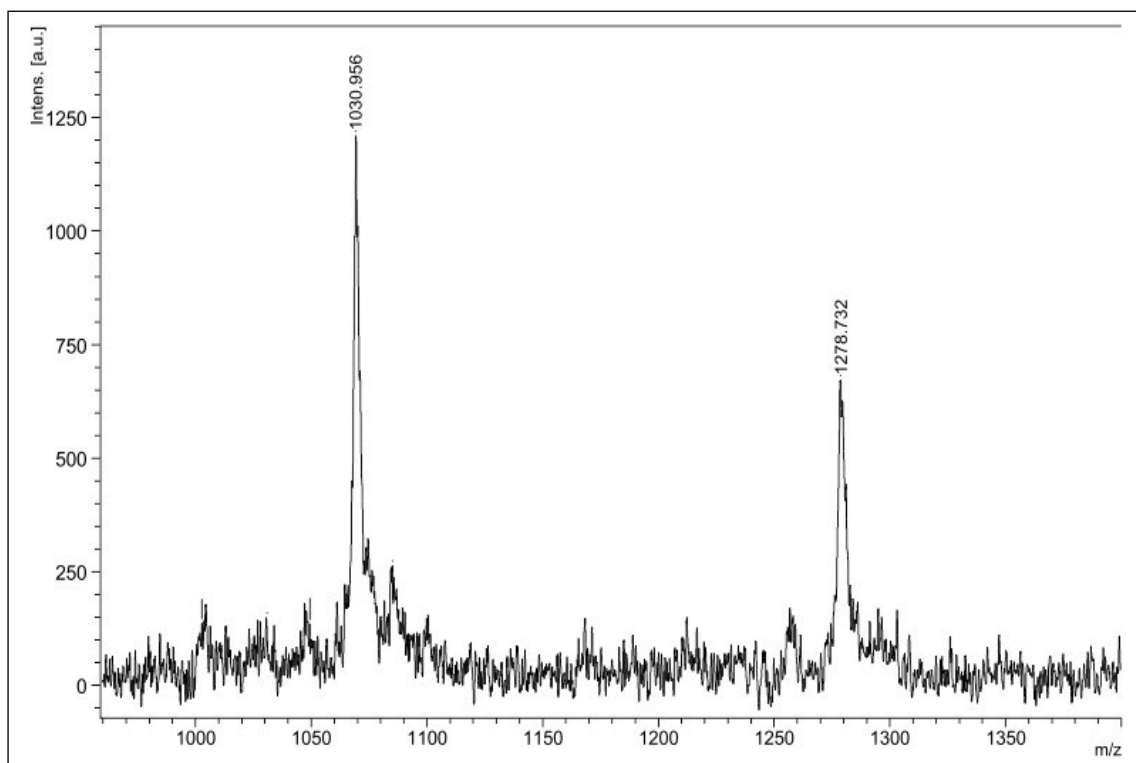


**Fig. S11.**  $^1\text{H-NMR}$  spectrum of compound **5** (in Pyridine- $d_5$ ).

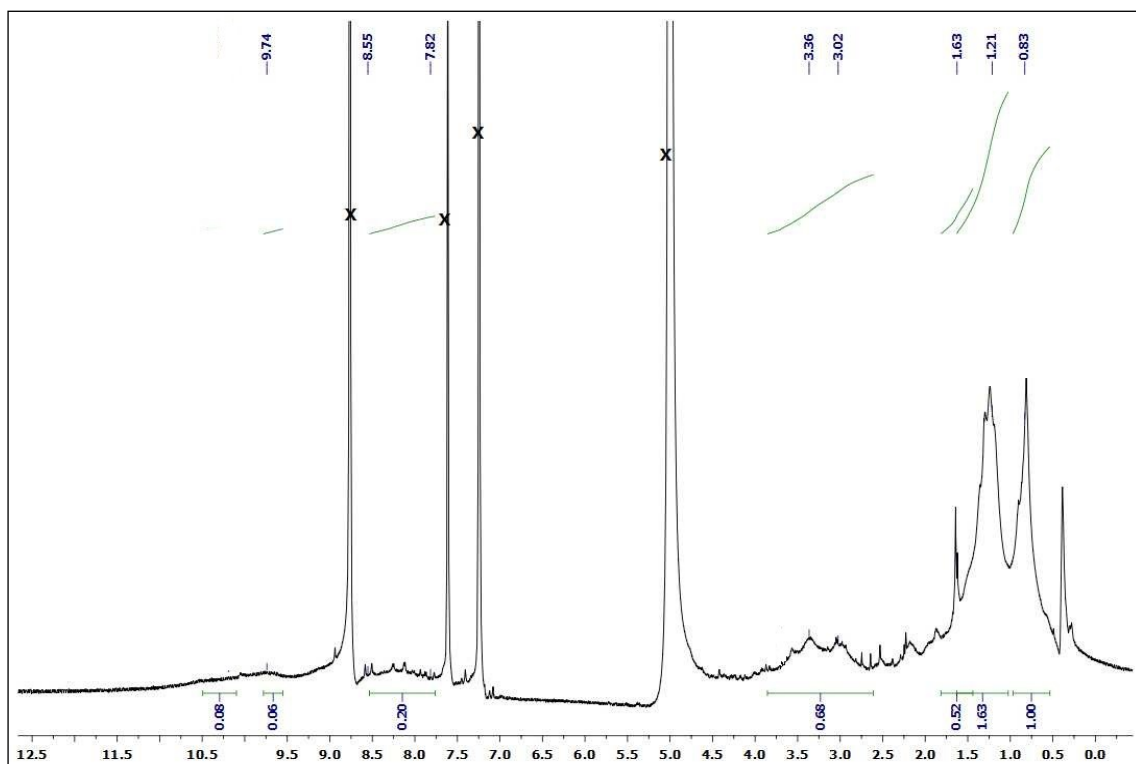
## 7. Characterization of compound **6**



**Fig. S12.** FT-IR spectrum of compound **6**.

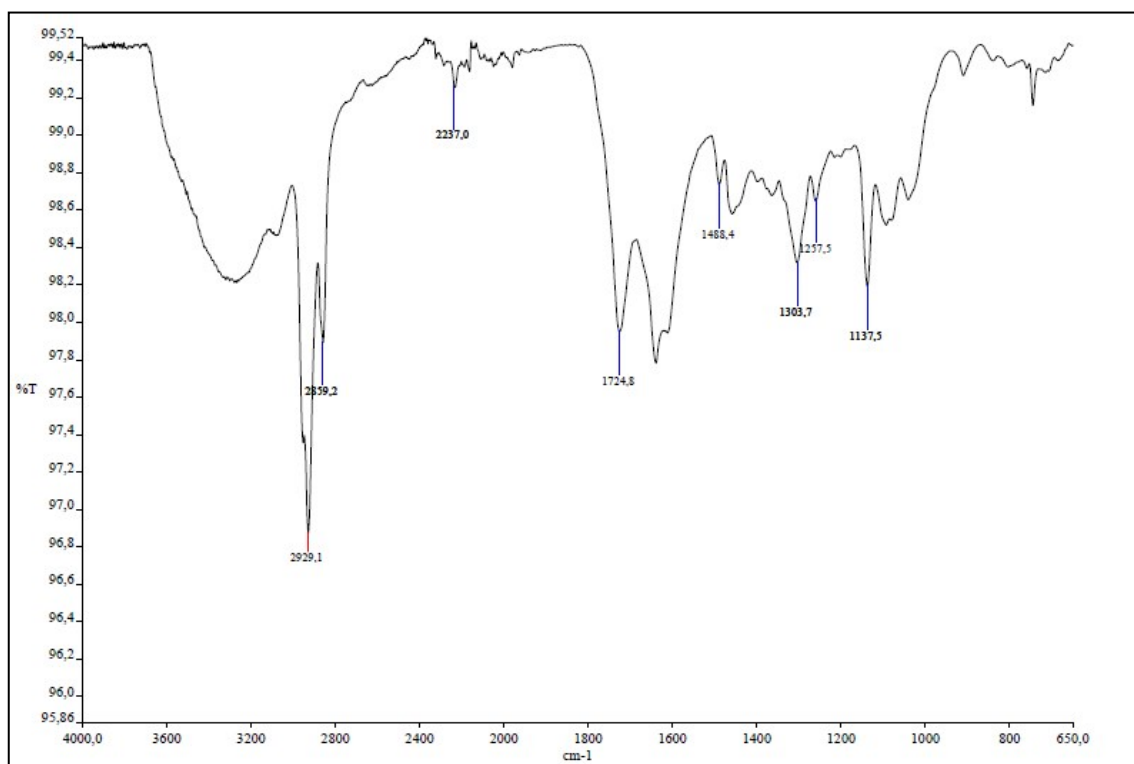


**Fig. S13.** MS (MALDI-TOF) spectrum of compound **6** (Matrix: DIT).

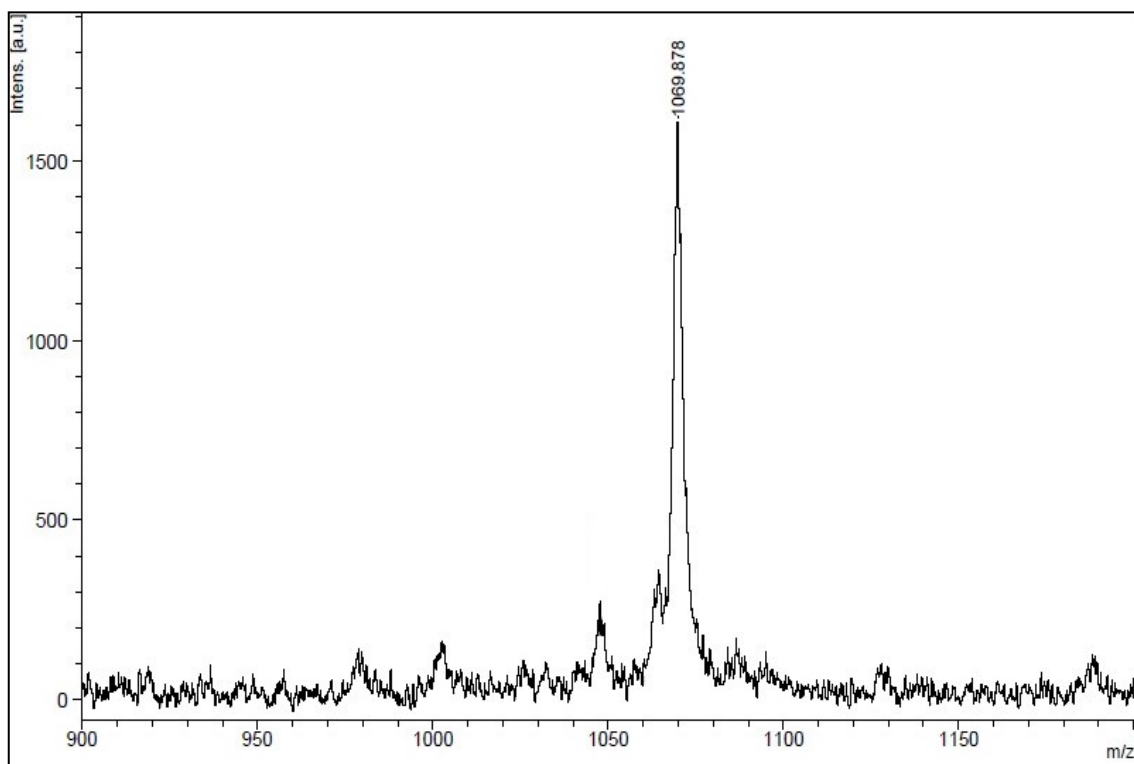


**Fig. S14.** <sup>1</sup>H-NMR spectrum of compound **6** (in Pyridine-*d*<sub>5</sub>).

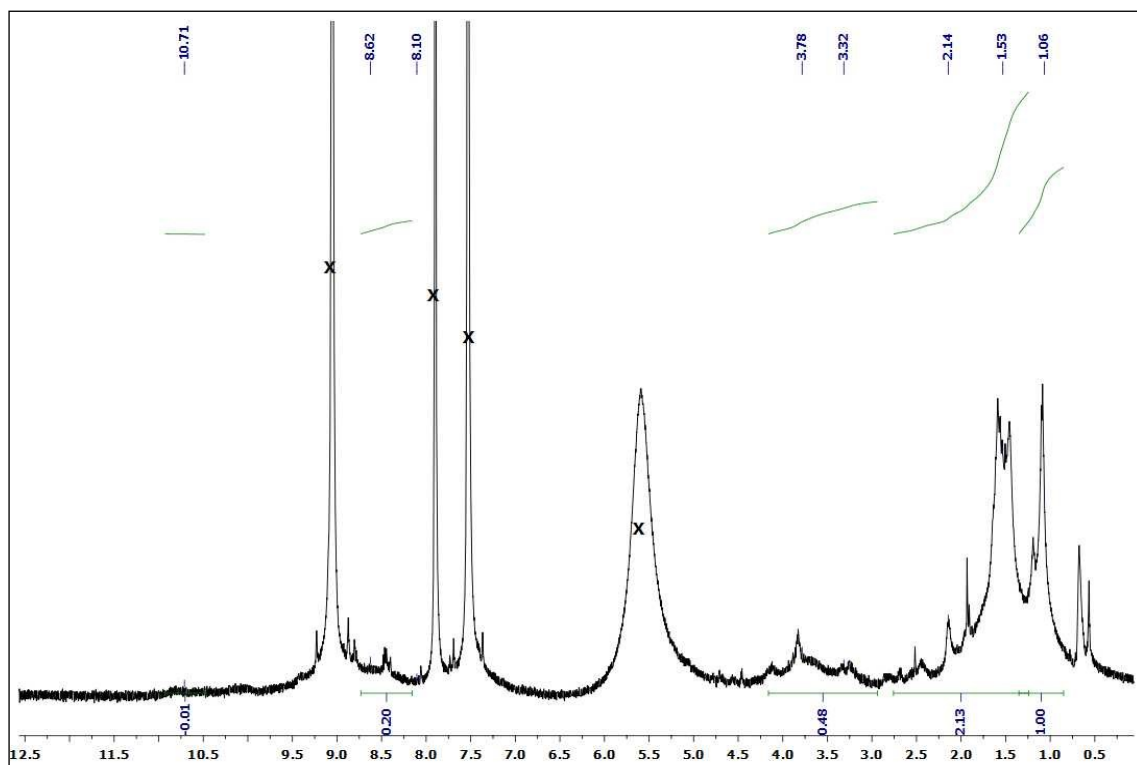
## 8. Characterization of compound GT32



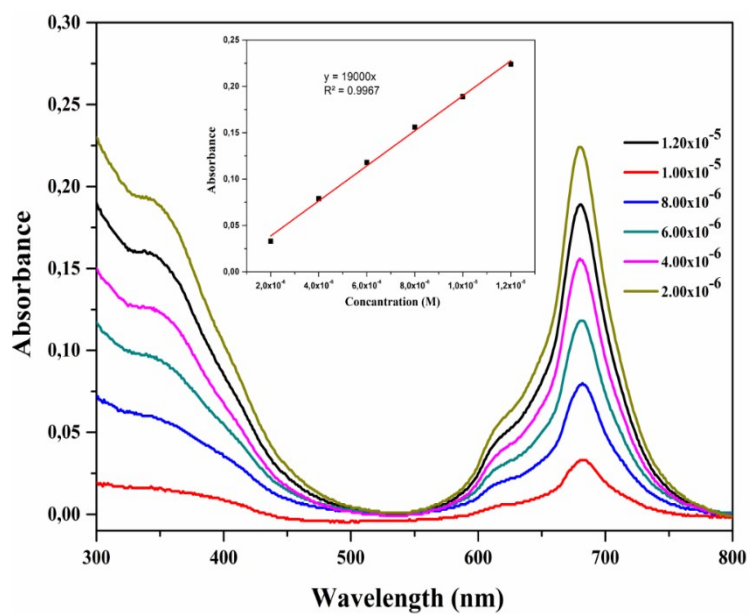
**Fig. S15.** FT-IR spectrum of compound GT32.



**Fig. S16.** MS (MALDI-TOF) spectrum of compound GT32 (Matrix: DHB).

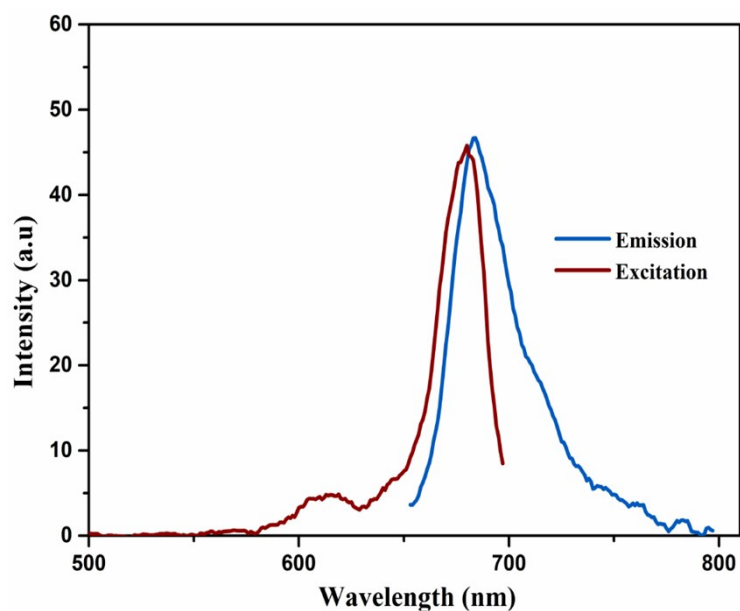


**Fig. S17.**  $^1\text{H-NMR}$  spectrum of compound **GT32** (in Pyridine- $d_5$ ).



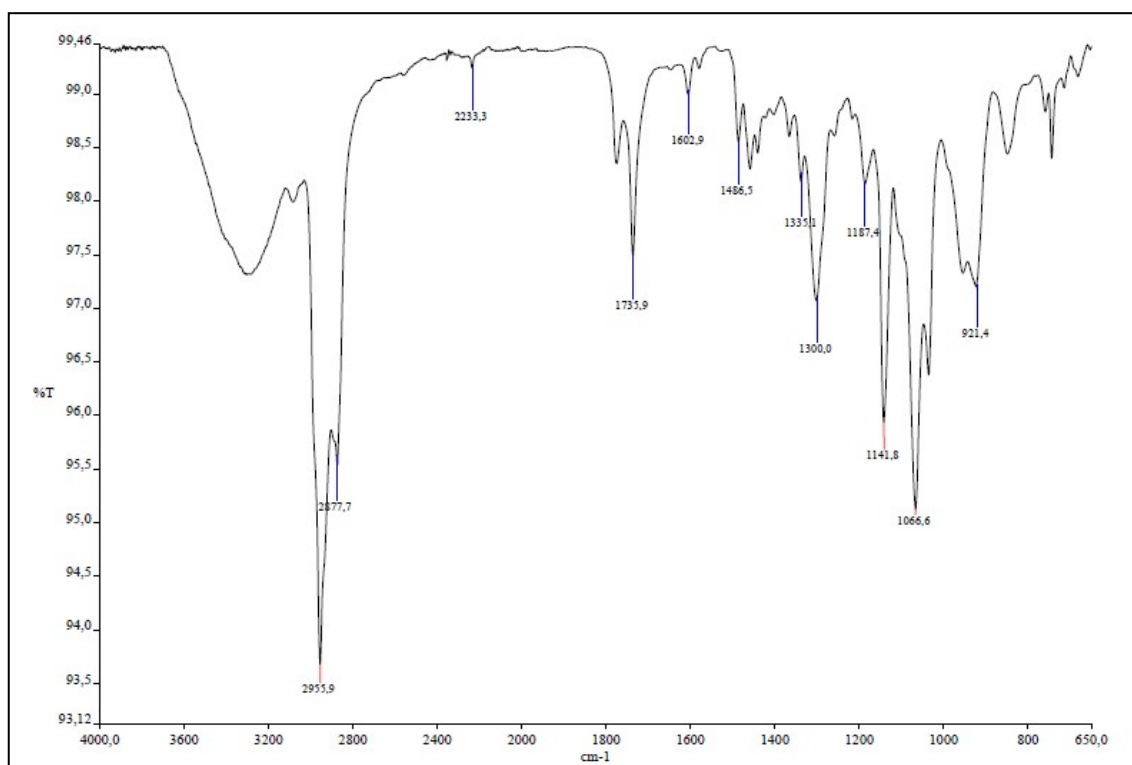
**Fig. S18.** UV-Vis spectra of **GT32** at the different concentrations in THF+Pyridine.



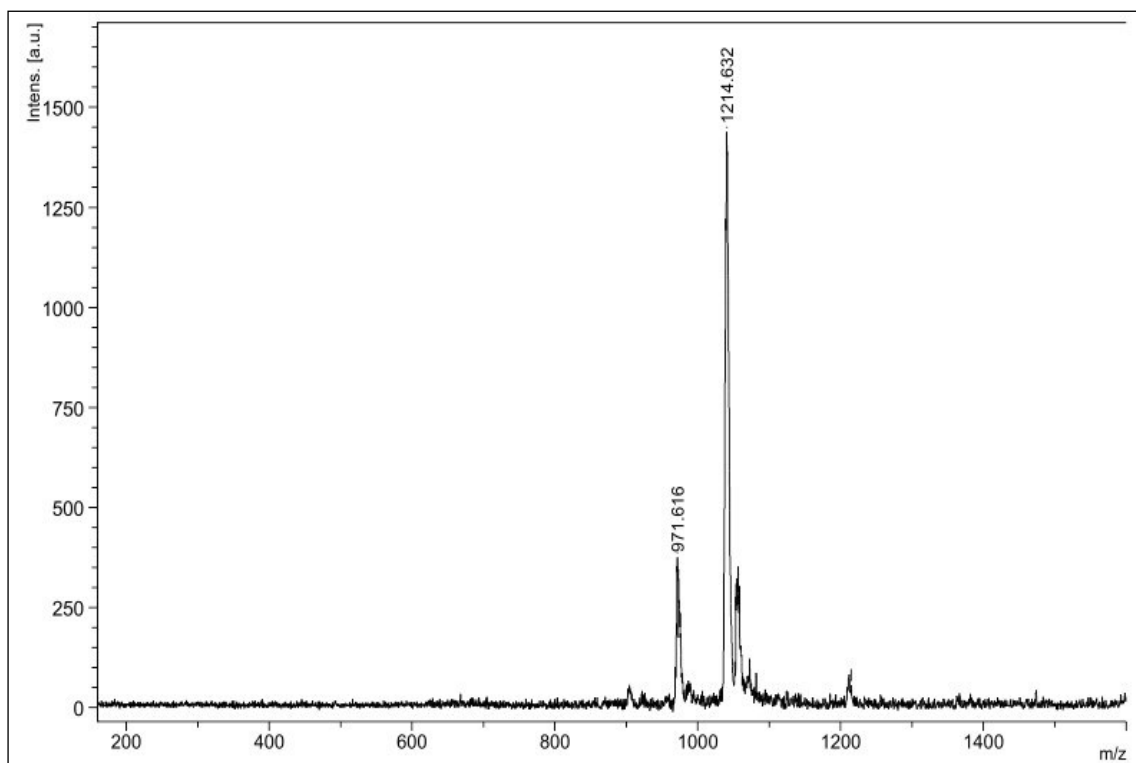


**Fig. S19.** Fluorescence excitation and emission spectra of **GT32**.

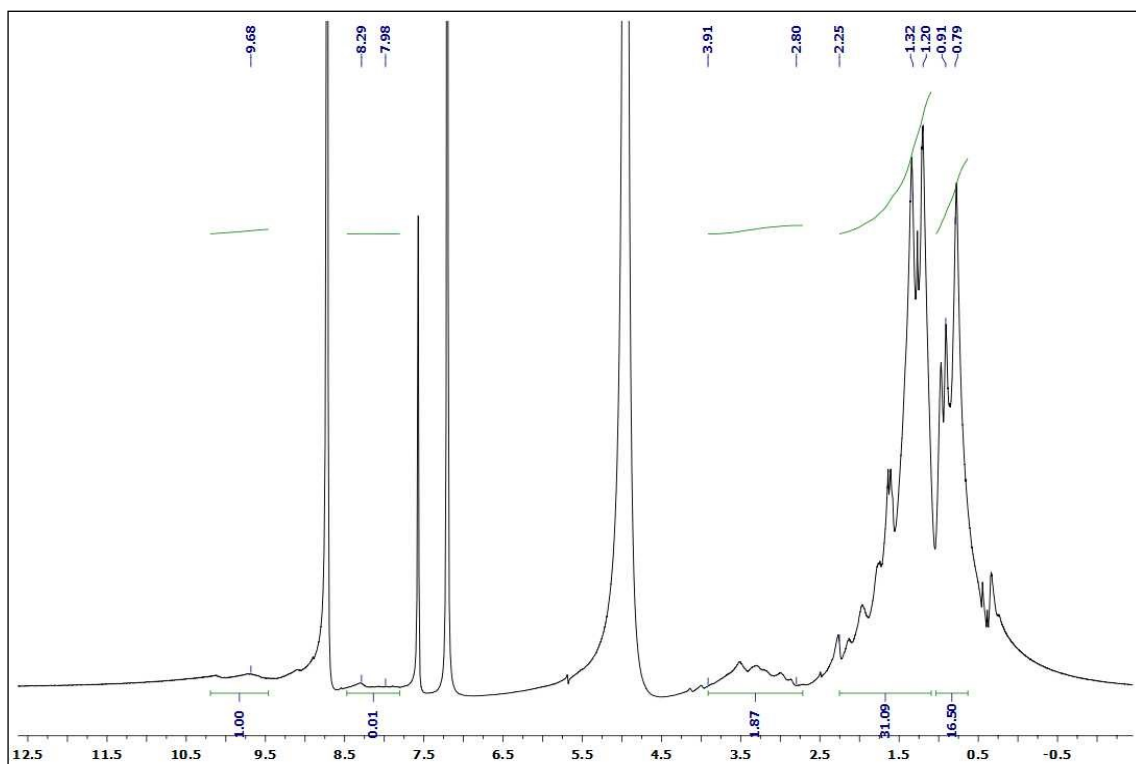
## 9. Characterization of compound **GT31**



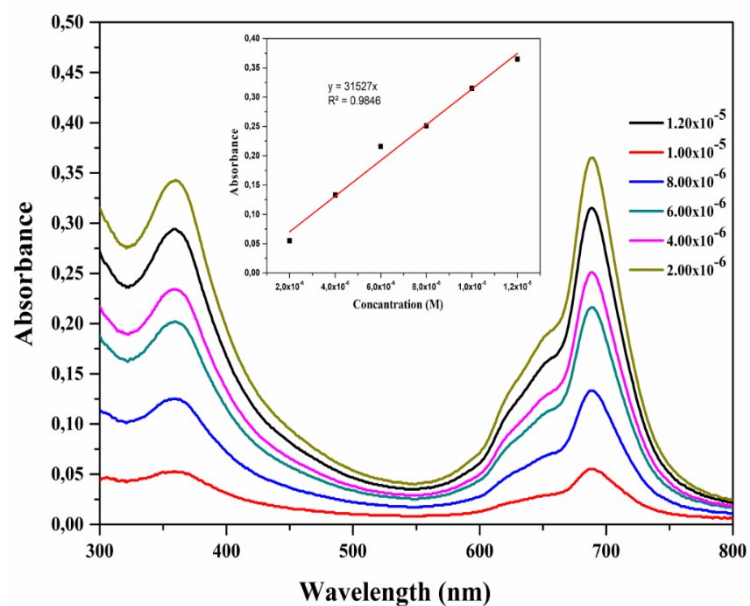
**Fig. S20.** FT-IR spectrum of compound **GT31**.



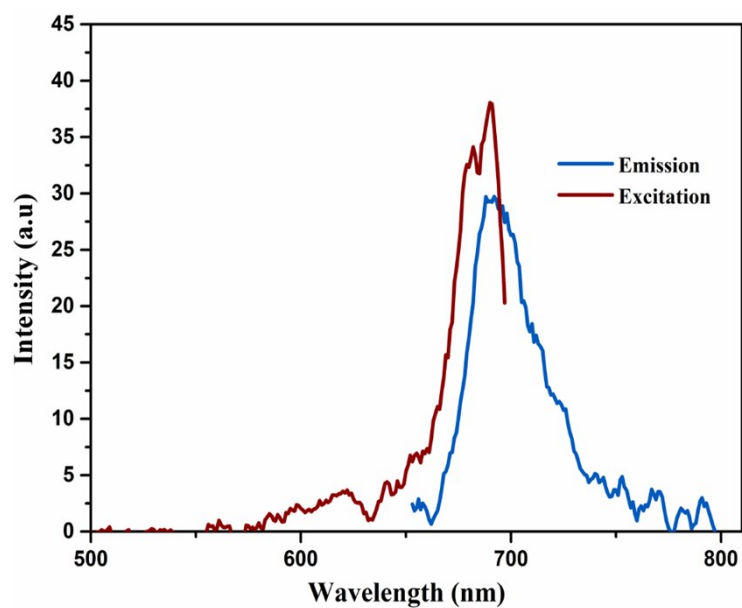
**Fig. S21.** MS (MALDI-TOF) spectrum of compound **GT31** (Matrix: DHB).



**Fig. S22.** <sup>1</sup>H-NMR spectrum of compound **GT31** (in Pyridine-*d*<sub>5</sub>).

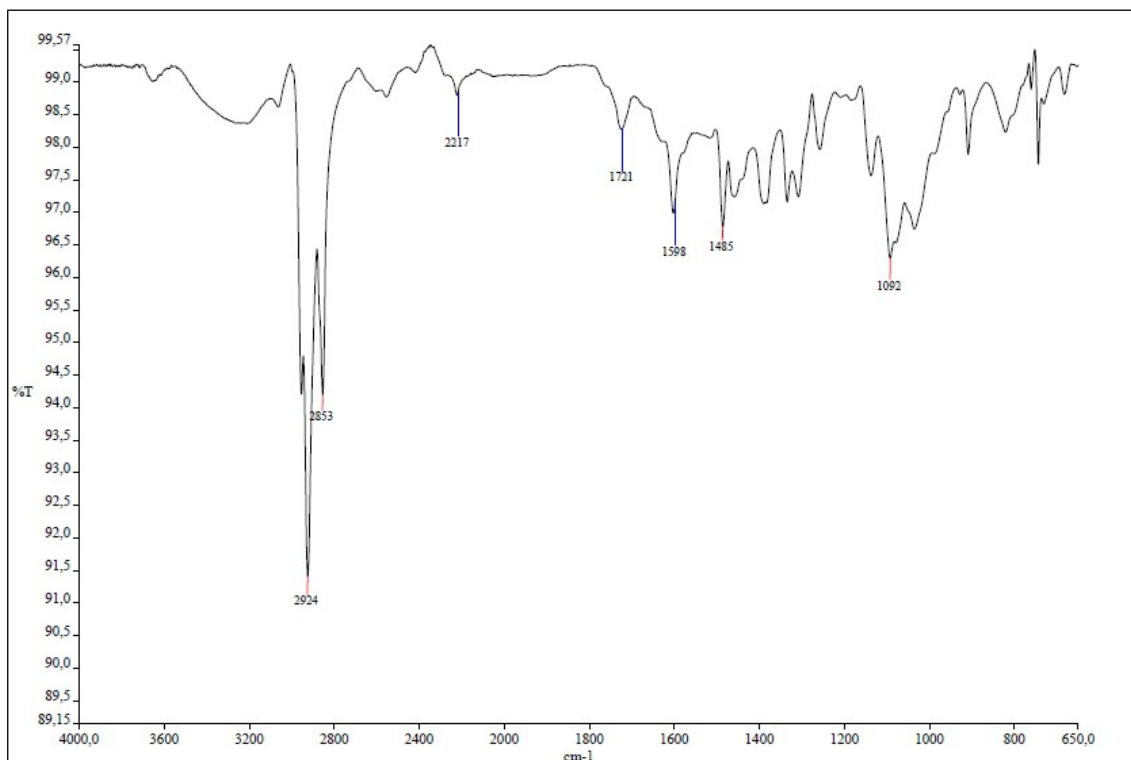


**Fig. S23.** UV-Vis spectra of **GT31** at the different concentrations in THF+Pyridine.

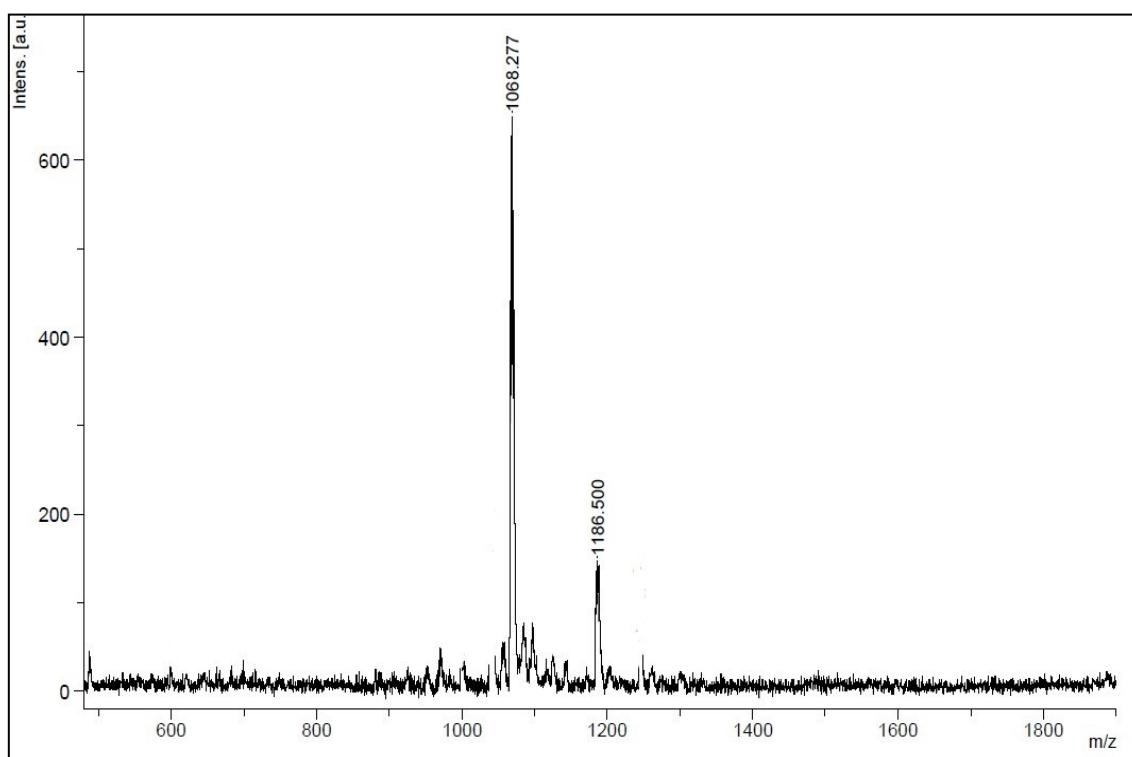


**Fig. S24.** Fluorescence excitation and emission spectra of **GT31**.

## 10. Characterization of compound GT5



**Fig. S25.** FT-IR spectrum of compound **GT5**.



**Fig. S26.** MS (MALDI-TOF) spectrum of compound **GT5** (Matrix: DHB).

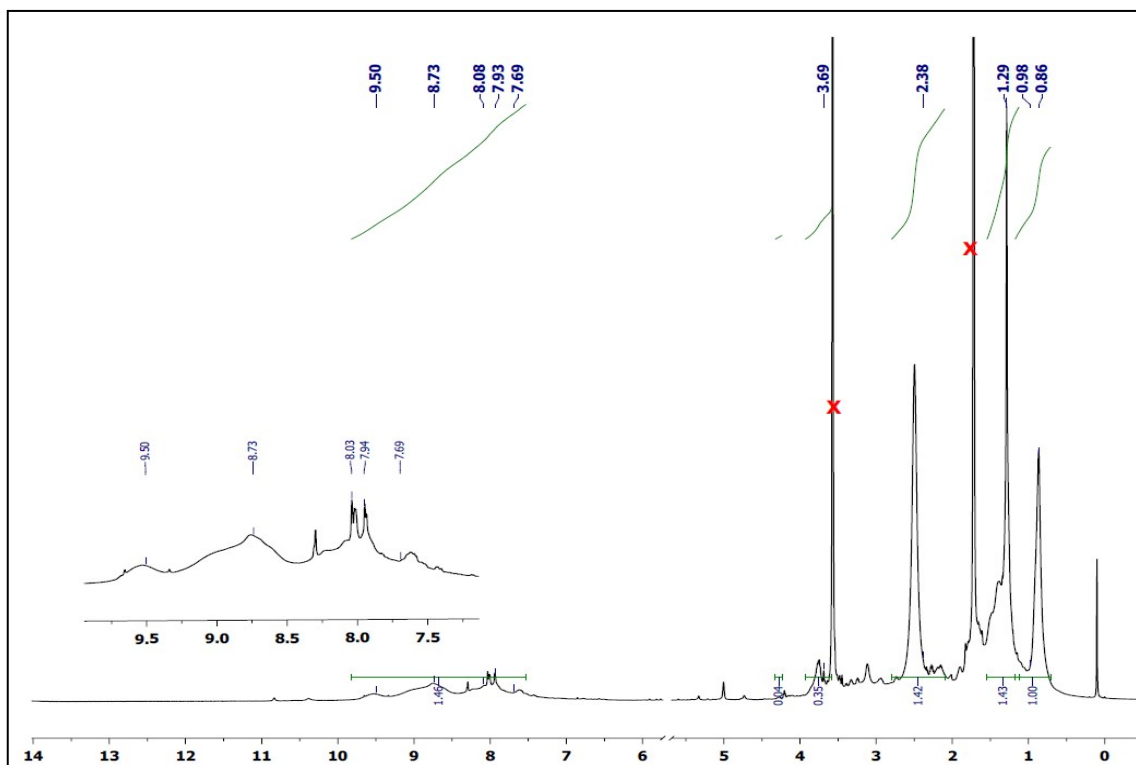


Fig. S27.  $^1\text{H-NMR}$  spectrum of compound **GT5** (in  $\text{THF-}d_8$ ).

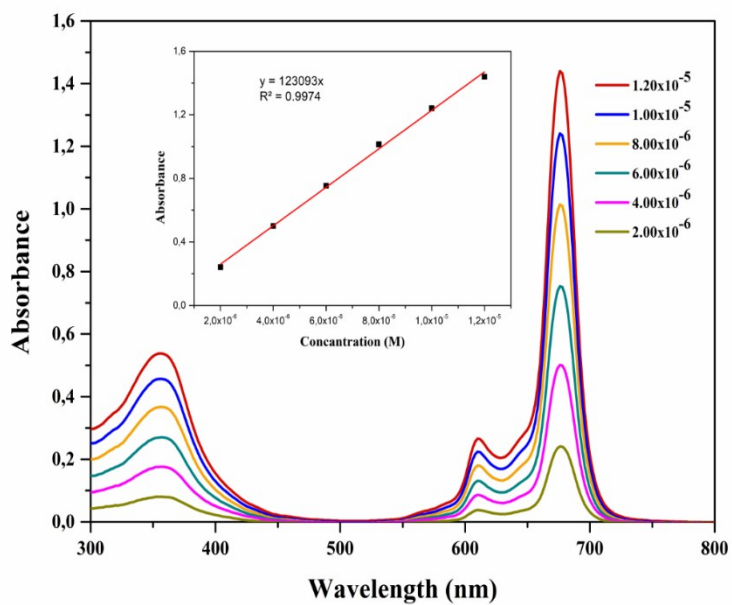
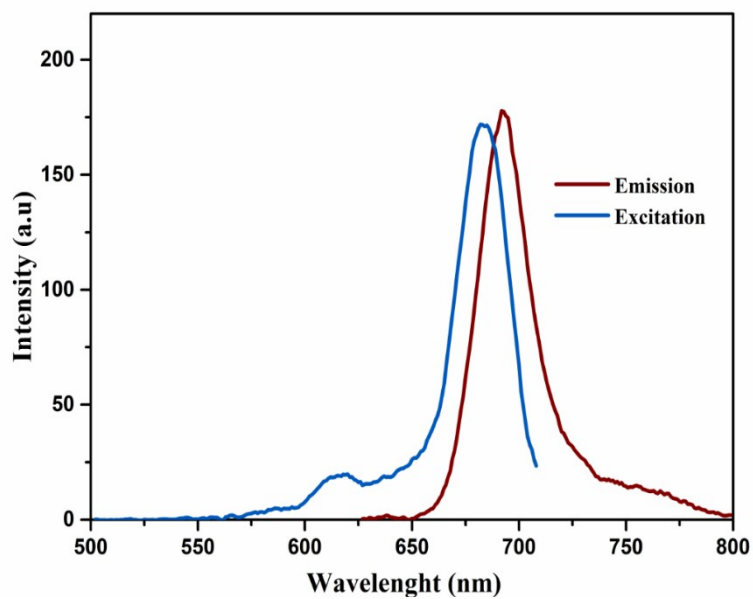
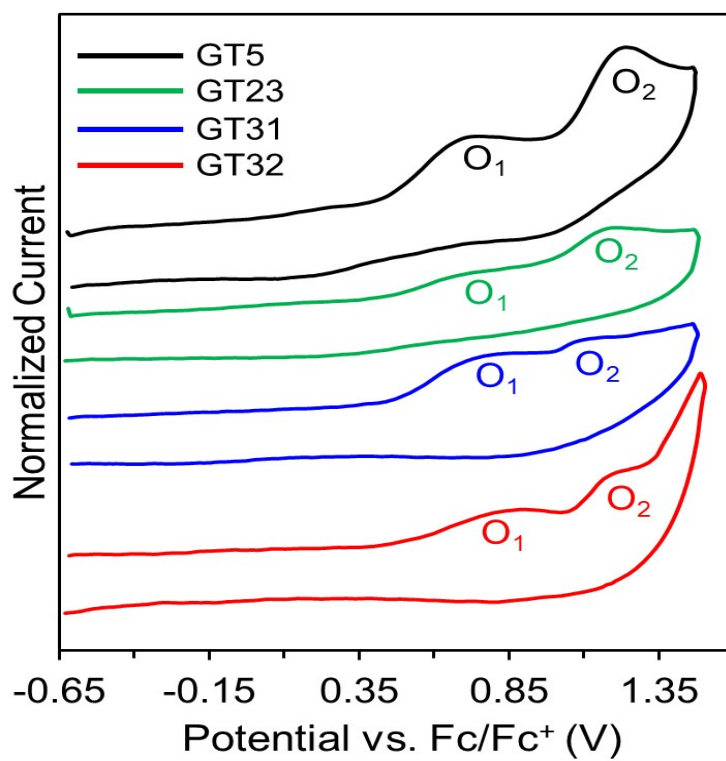


Fig. S28. UV-Vis spectra of **GT5** at the different concentrations in  $\text{THF+pyridine}$ .

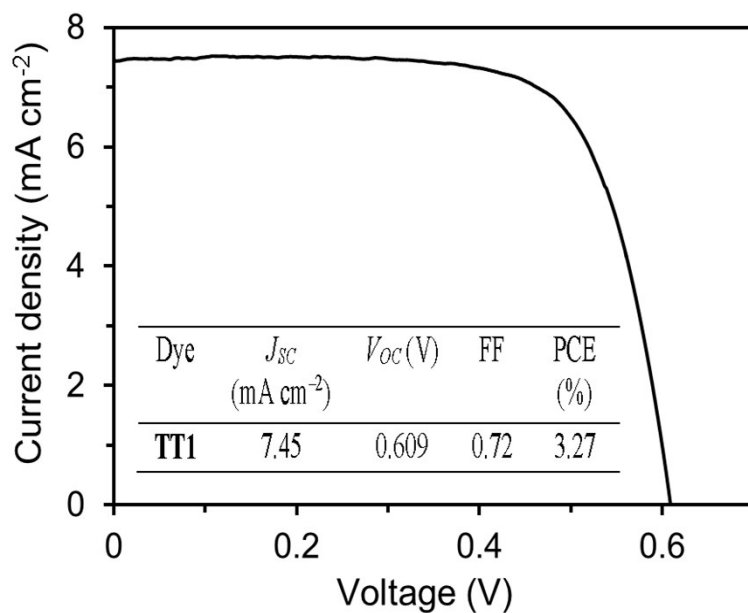


**Fig. S29.** Fluorescence excitation and emission spectra of **GT5**.

### 11. Electrochemical, photovoltaic and theoretical results



**Figure S30.** Cyclic voltammetry of the dyes in DMSO at a scan rate of  $0.050 \text{ V s}^{-1}$ .



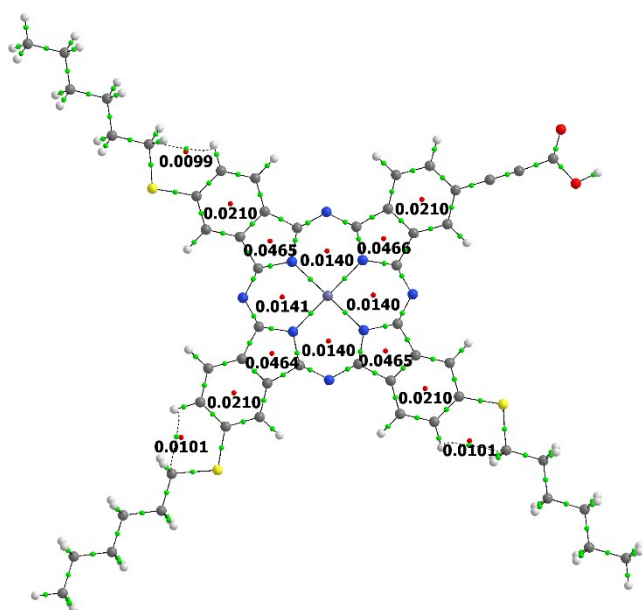
**Figure S31.**  $J$ - $V$  curve of DSSC based on **TT1** with CDCA.

**Table S1.** Absorption, excitation and emission spectral data for **GT5**, **G31**, **GT23**, **GT32** in THF.

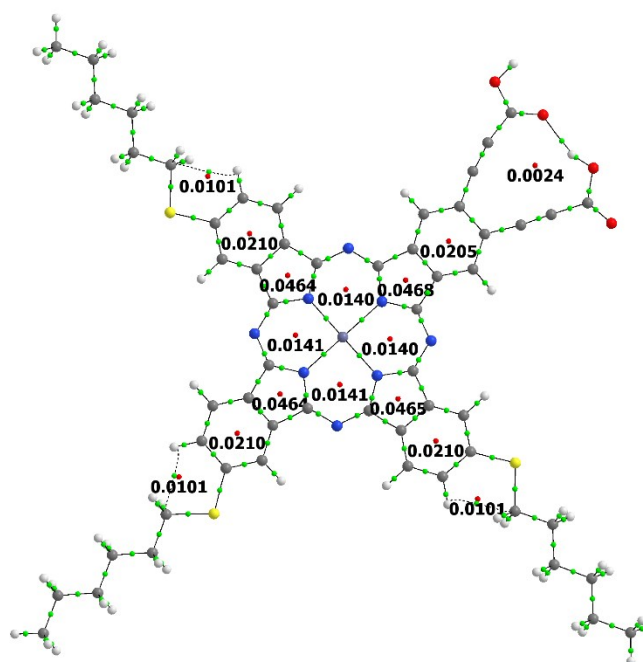
Compound	$\lambda_{\max}^{abs}$ (nm)	$\log \epsilon$	$\lambda_{\max}^{em}$ (nm)	$\lambda_{\max}^{ex}$ (nm)	$\Delta\lambda_{ST}$ (nm)
<b>GT5</b>	676	5.09	692	685	16
<b>GT31</b>	689	4.49	692	686	3
<b>GT23</b>	679	4.82	687	685	8
<b>GT32</b>	680	4.27	684	679	4

Maximum absorption wavelengths ( $\lambda_{\max}$ ), the logarithmic value of extinction coefficients( $\epsilon$ ),  $\lambda_{\max}^{em}$  : maximum emission wavelength,  $\lambda_{\max}^{ex}$  : maximum excitation wavelength and  $\Delta\lambda_{ST}$  : Stoke's shift.

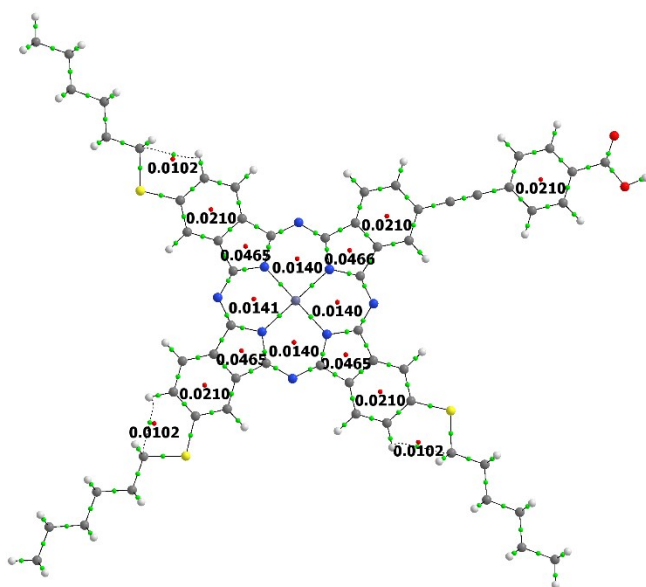
GT23-2



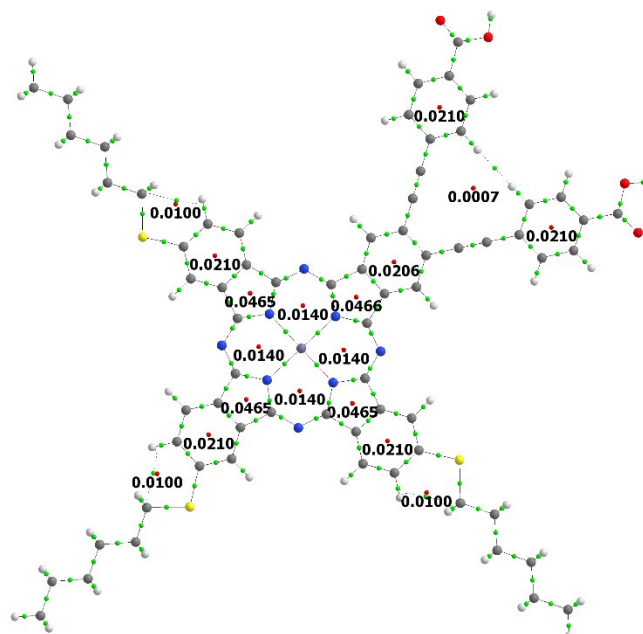
GT32-2



GT5-2



GT31-2

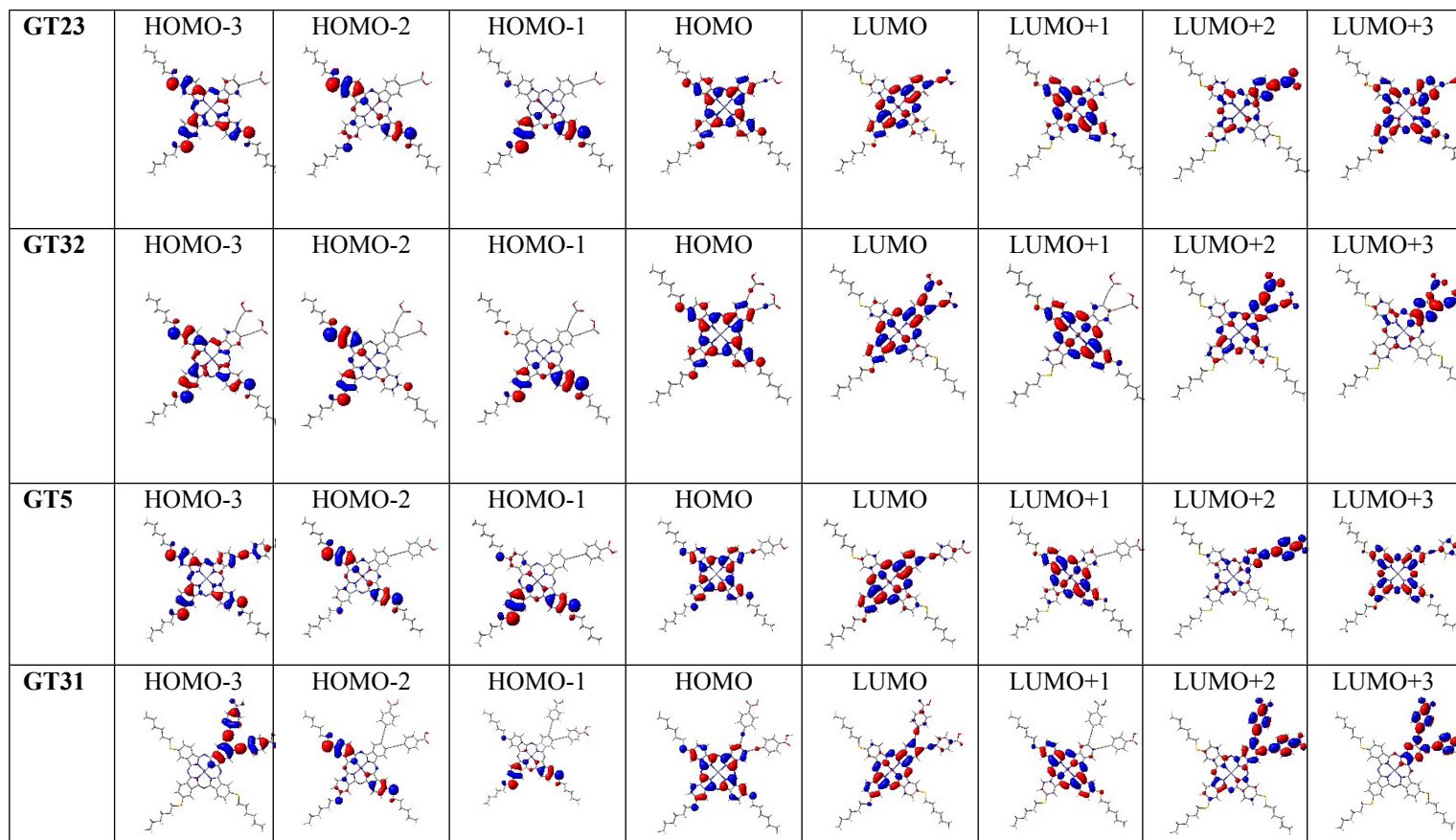


**Figure S32.** The bond (green) and ring (red) critical points calculated for all phthalocyanines at the PBE0/6-31G(d,p) level of theory.



**Table S2.** More relevant parameters of the electronic absorption bands calculated in the solution phase with the M06/6-31G(d,p) level of theory; wavelength ( $\lambda$ ), energy (E), oscillator strength ( $f$ ), deviation ( $\Delta E$ ) from the experimental data of this work. The assignment of the bands, and the molecular orbitals involved in the electronic transition are also included.

Molecule	$\lambda$ /nm	E/eV	$f$	$\Delta E$ /eV	% $\Delta E$	Assignment	Transition
<b>GT23-2</b>							
Exp	679	1.83				Q	
	615	2.02				Q	
M06	356	3.48				Soret(B)	
	700	1.77	0.816	-0.06	3.28	Q	H→L
	668	1.86	0.797	-0.16	7.92	Q	H→L+1
	441	2.81	0.296				H-3→L
	321	3.86	0.686	0.38	10.92	Soret(B)	NTO 24 occ→virt
<b>GT32-2</b>							
Exp	680	1.82				Q	
	616	2.01				Q	
	345	3.59				Soret(B)	
M06	734	1.69	0.997	-0.13	7.14	Q	H→L
	665	1.86	0.753	-0.15	7.46	Q	H→L+1
	457	2.71	0.197				H-3→L
	335	3.70	0.718	0.11	3.06	Soret(B)	NTO 23 occ→virt
<b>GT5-2</b>							
Exp	676	1.83				Q	
	610	2.03				Q	
	356	3.48				Soret(B)	
M06	700	1.77	0.914	-0.06	3.28	Q	H→L
	673	1.84	0.792	-0.19	9.36	Q	H→L+1
	438	2.83	0.444				H-3→L
	331	3.75	0.662	0.27	7.76	Soret(B)	NTO 21 occ→virt
<b>GT31-2</b>							
Exp	689	1.80					
	643	1.93					
	358	3.46					
M06	725	1.71	1.168	-0.09	5.00	Q	H→L
	676	1.84	0.729	-0.09	4.66	Q	H→L+1
	446	2.78	0.435				H-4→L
	331	3.74	0.594	0.28	8.09	Soret(B)	NTO 25 occ→virt



**Figure S33.** Surface of the molecular orbitals that participate in the electronic absorption bands obtained at the PBE0/6-31G(d,p) in the solution phase.

## References

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