Supporting Information

Construction of highly efficient near-IR solid emitter based on naphthalene diimide with AIE-active tetraphenylethene periphery

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General Methods & Materials: Naphthalene tetracarboxy dianhydride, octyl amine, chloroform, methanol, dichloromethane, tetrahydrofuran and hexane were purchased from Aldrich and used without purification, unless otherwise specified. All reagents were purchased either from Sigma Aldrich Chemical Co. or Merck and used as such without any further purification. All the solvents were received from commercial sources and purified by standard methods. The UV-vis absorption spectra were recorded on a Perkin Elmer Lambda 40p spectrometer. ^{1}H NMR, ^{13}C-NMR spectra were recorded on a Bruker spectrometer using chloroform-d as solvent and tetramethylsilane as an internal standard. The solvents for spectroscopic studies were of spectroscopic grade and used as received. Mass spectra (MS) were obtained by using Bruker AutoFlex Matrix Assisted Laser Desorption/Ionisation (MALDI) Time of Flight (TOF)- Mass Spectrometer (MALDI-TOF-MS). The X-ray diffraction (XRD) pattern spectra were performed on a Bruker D8 FOCUS diffractometer using a Cu target radiation source (λ= 0.15418 nm). **TArcNDI** already described in our previous publication and used as is.[S1]

UV-vis absorption spectroscopy: A 0.2 mL aliquot of the stock solution of **TTPEcNDI** and **TArcNDI** (conc. = 10^{-3} M) was transferred to various ratios of $CHCl_3$/hexane, $CHCl_3$/MeOH and THF/water in different volumetric flasks, and made up to 2 mL volume. The solutions were allowed to equilibrate for 2 h prior to the spectroscopic measurements.

Fluorescence Spectroscopy: Fluorescence emission spectra were recorded on a Horiba Jobin Yvon FluoroMax®-4–Spectrofluorometer. All experiments were performed in a quartz cell with a 1 cm path length with 365 nm excitation wavelength. All the solutions were prepared in a similar manner to that in the UV–Vis study.

Scanning Electron Microscopy (SEM): A 0.2 mL aliquot of the stock solution of **TTPEcNDI** was transferred separately to four different volumetric flasks of (i) THF/water (1:4, *v/v*), (ii) $CHCl_3$/MeOH (1:7, *v/v*), (iii) $CHCl_3$/hexane (1:9, *v/v*) and made up to 2 mL volume with respective solvents. The solutions were allowed to equilibrate for 2 h prior to the Scanning Electron Microscopy (SEM) imaging. Samples of **TTPEcNDI (**$1.2x10^{-5}$ M) in $CHCl_3$/hexane (1:9, *v/v*), $CHCl_3$/MeOH (1:7, *v/v*) and THF/water (1:4, *v/v*) were drop coated on a silica wafer substrate and sputter coated with gold for 10 s at 0.016 mA Ar plasma (SPI, West Chester, USA) for SEM imaging using a Nova SEM (Hillsboro, USA) operating at high vacuum,

X-ray crystlography: CCDC 1406389. Single crystal X-ray diffraction intensities were collected on a Bruker APEX II diffractometer with a PHOTON 100 CCD and Mo- Kα radiation (λ = 0.71073 Å).[20] The data were corrected for Lorentz and polarization effects. Absorption corrections were carried out based on multiple-scanned reflections. The crystal structures were solved by direct methods using SHELX-2014/7 and refined by full-matrix least square refinement on F2 using SHELX-2014/7. All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydrogen atoms were all placed at geometrically calculated positions and refined with a site occupancy factor of 0.5 while allowing them to ride on the parent atom. The chloroforms were refined using SADI, DFIX and SIMU restraints. SADI and DFIX restraints were applied to the to ensure the Cl-C bond lengths were consistent. The SIMU restraints were applied to similar chloroform molecules to ensure logical displacement parameters.

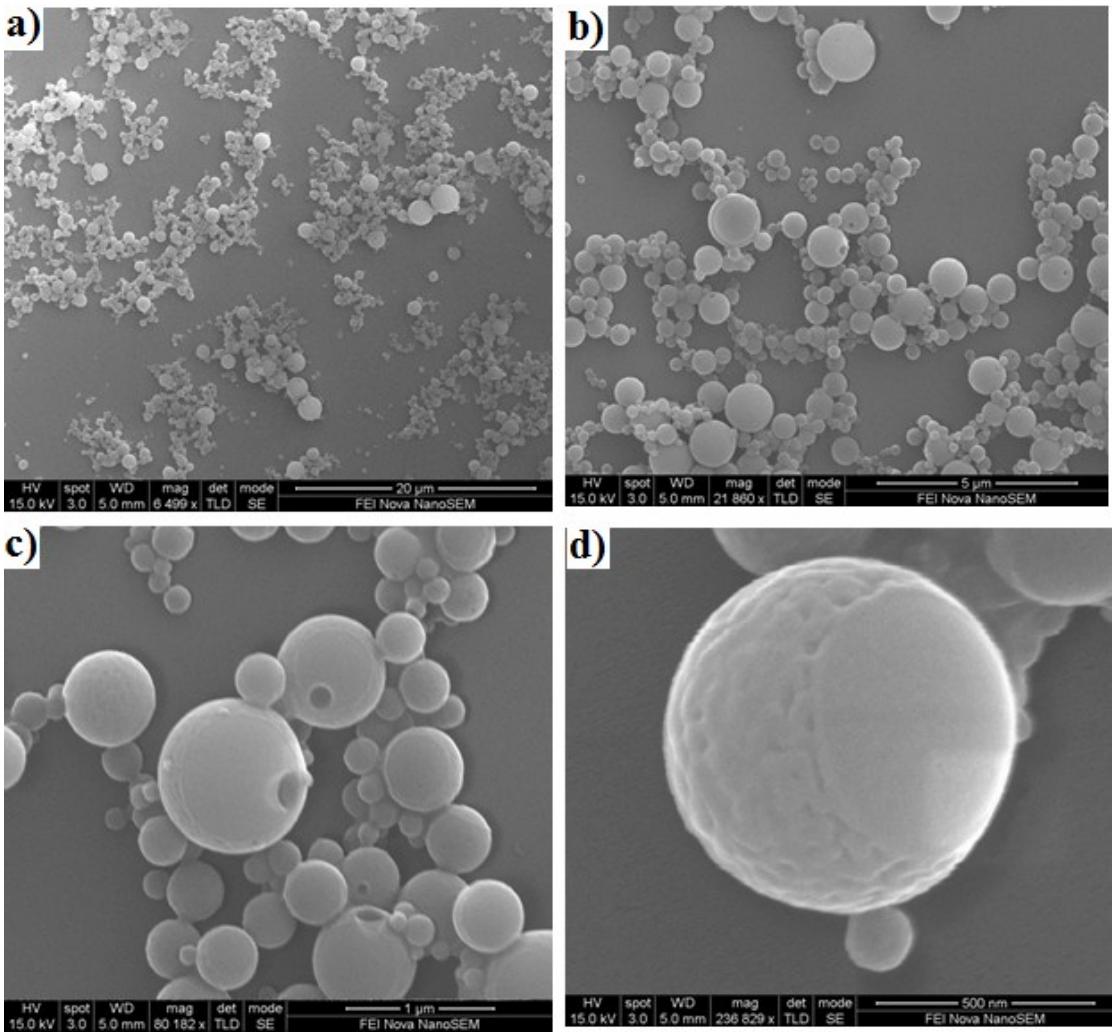


Fig. S1 SEM of **TTPEcNDI** in the $CHCl_3$/hexane with different fraction of hexane (a) f_h = 60%, (b) f_h = 90%, (c and d) enlarge images of "b".

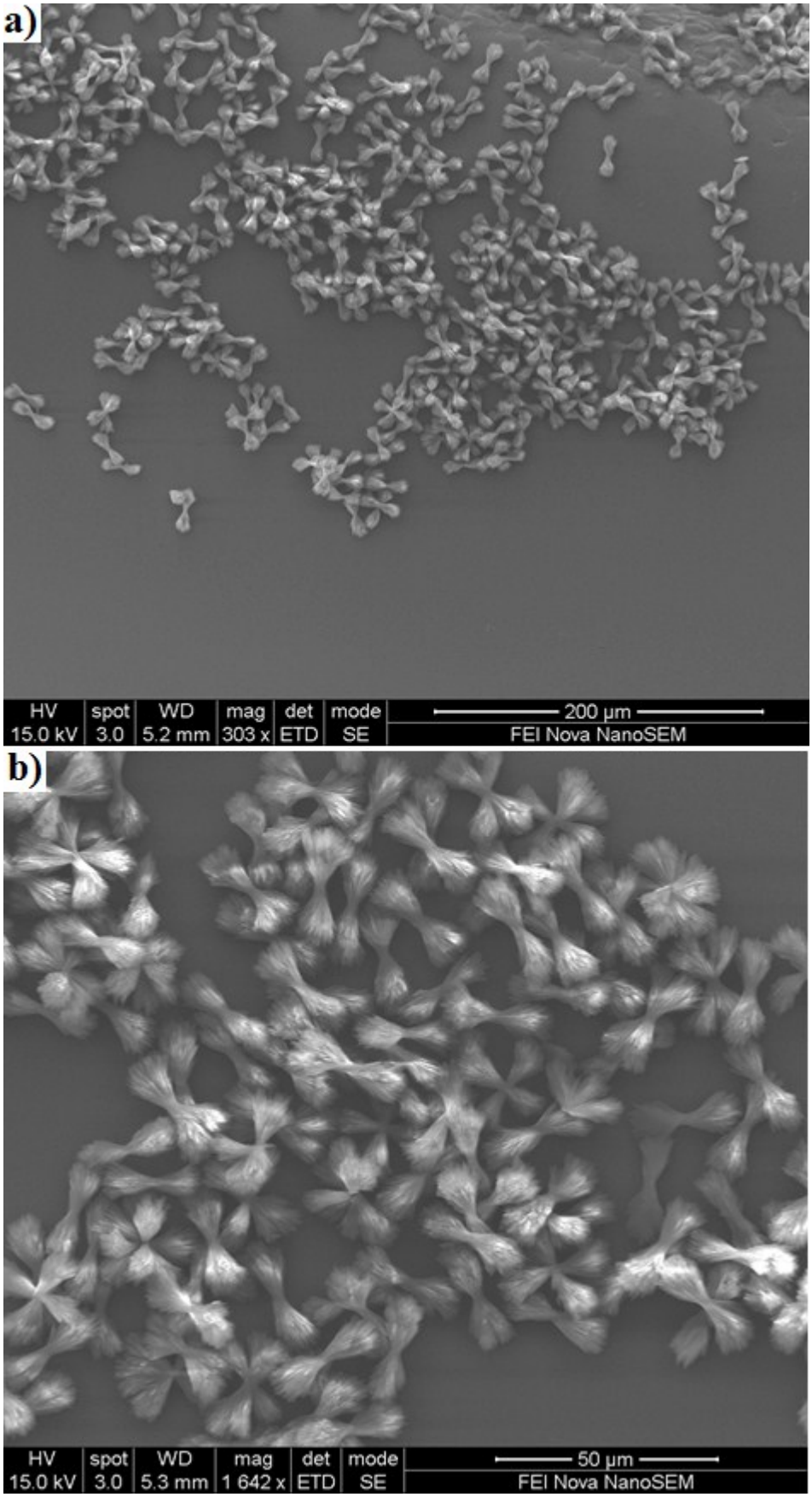


Fig. S2 SEM of **TTPEcNDI** in the $CHCl_3$/MeOH (f_m = 90%).

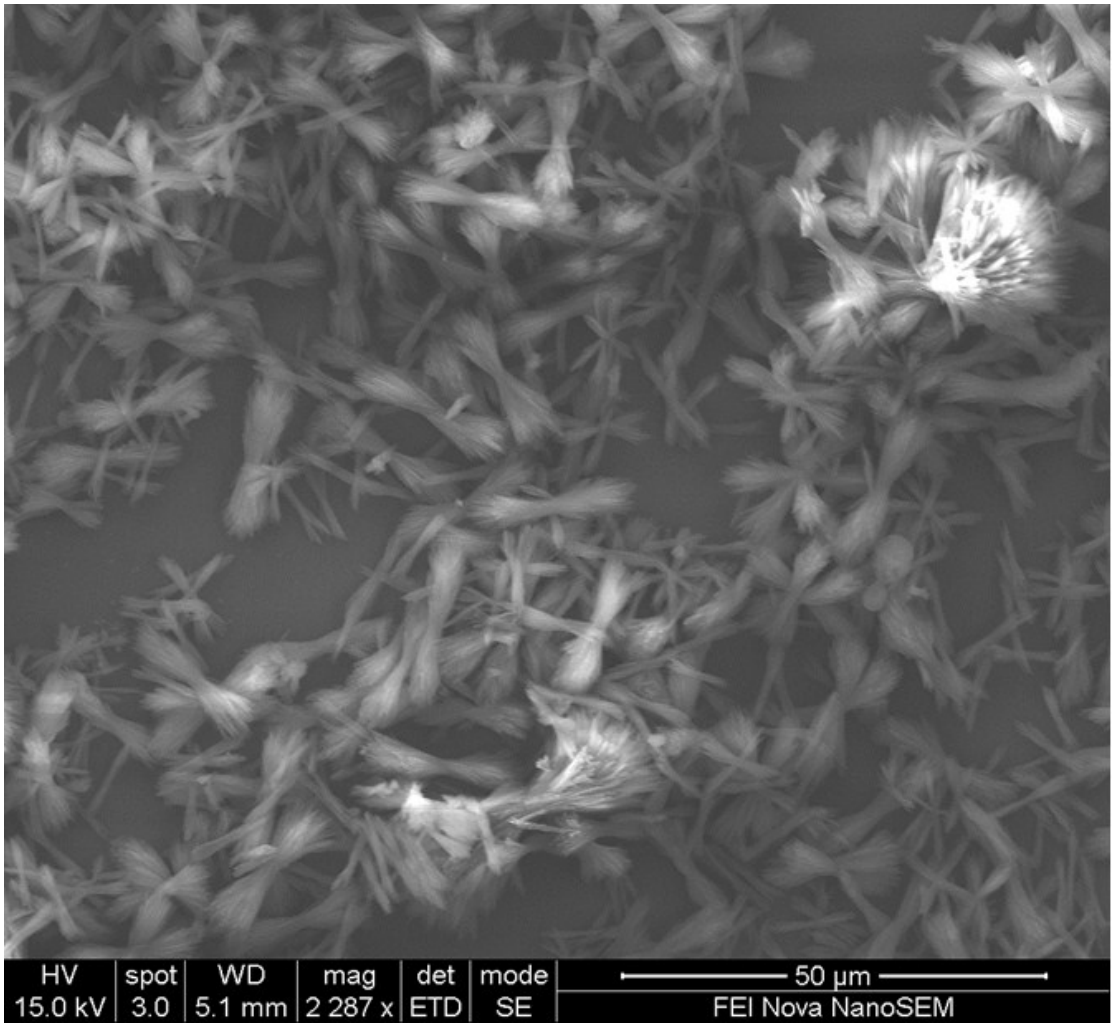


Fig. S3 SEM of **TTPEcNDI** in the $CHCl_3$/MeOH (f_m = 80%).

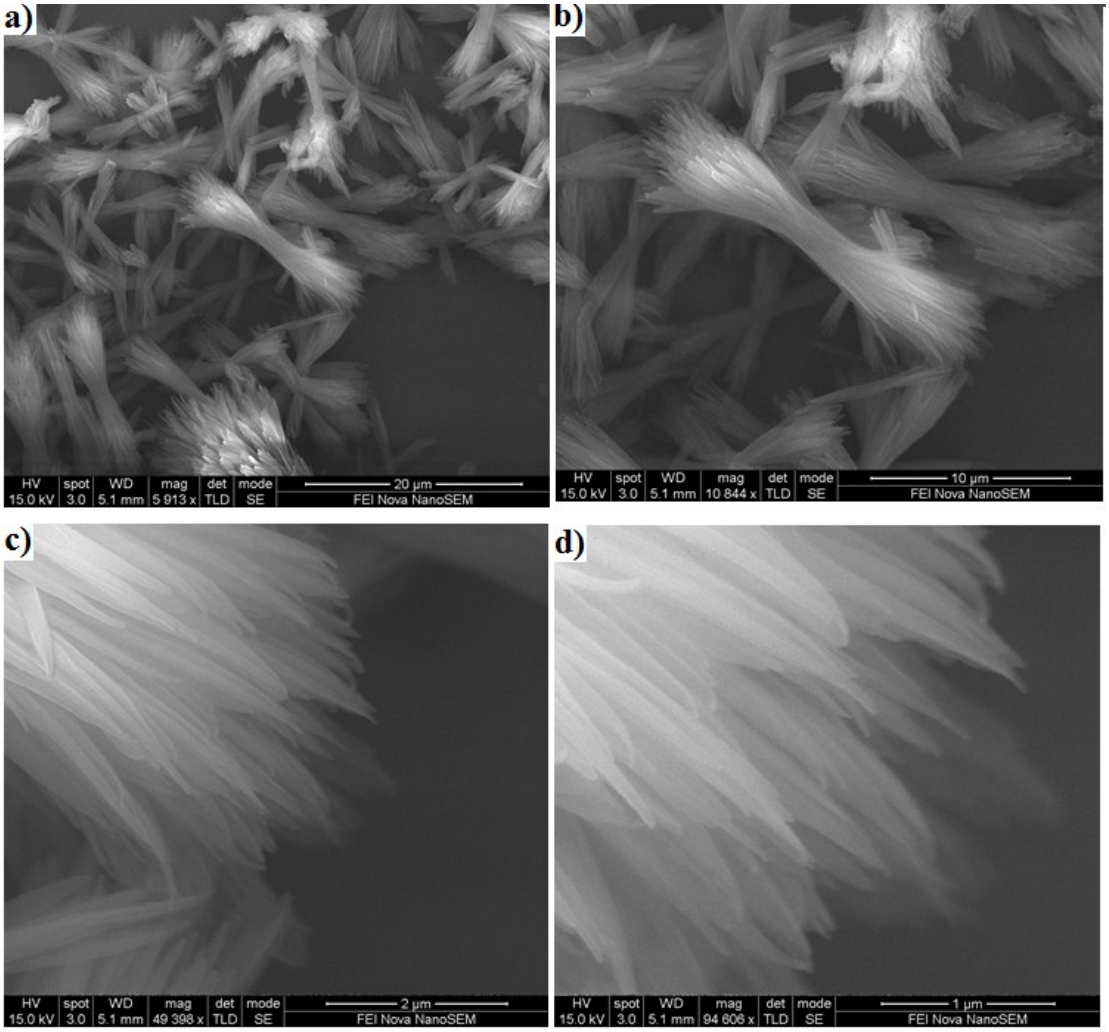


Fig. S4 SEM of **TTPEcNDI** in the $CHCl_3$/MeOH (f_m = 70%).

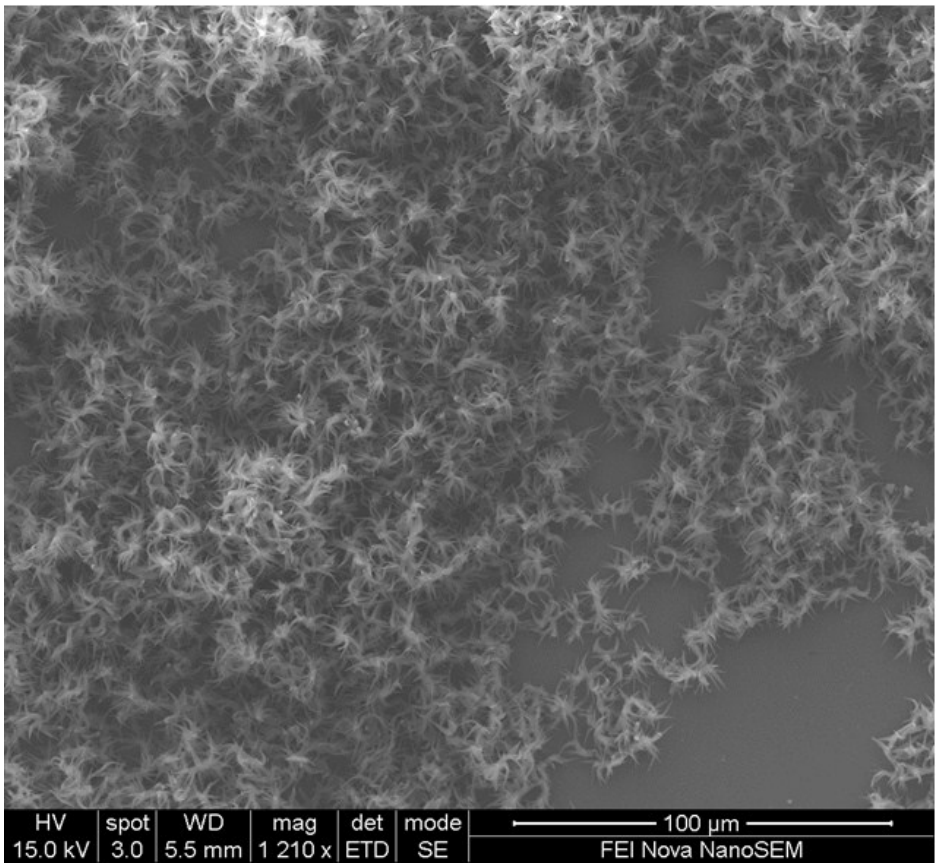


Fig. S5 SEM of **TTPEcNDI** in the THF/water (f_w = 80%).

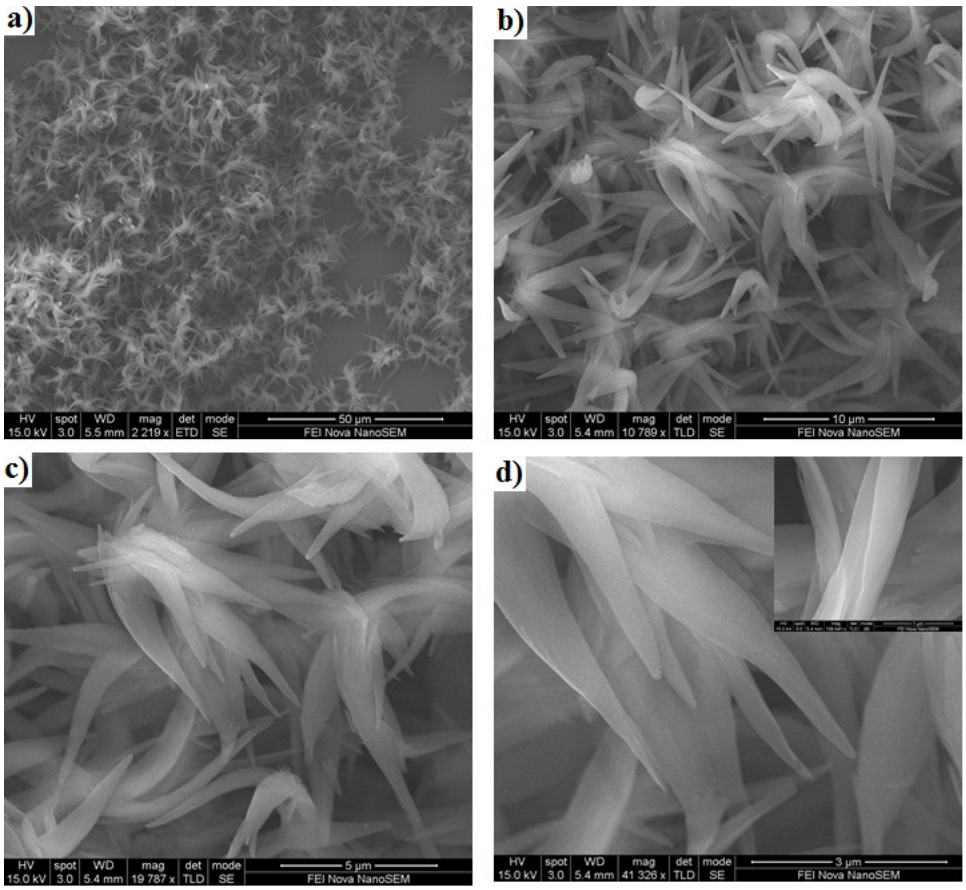


Fig. S6 SEM of **TTPEcNDI** in the THF/water (f_w = 70%).

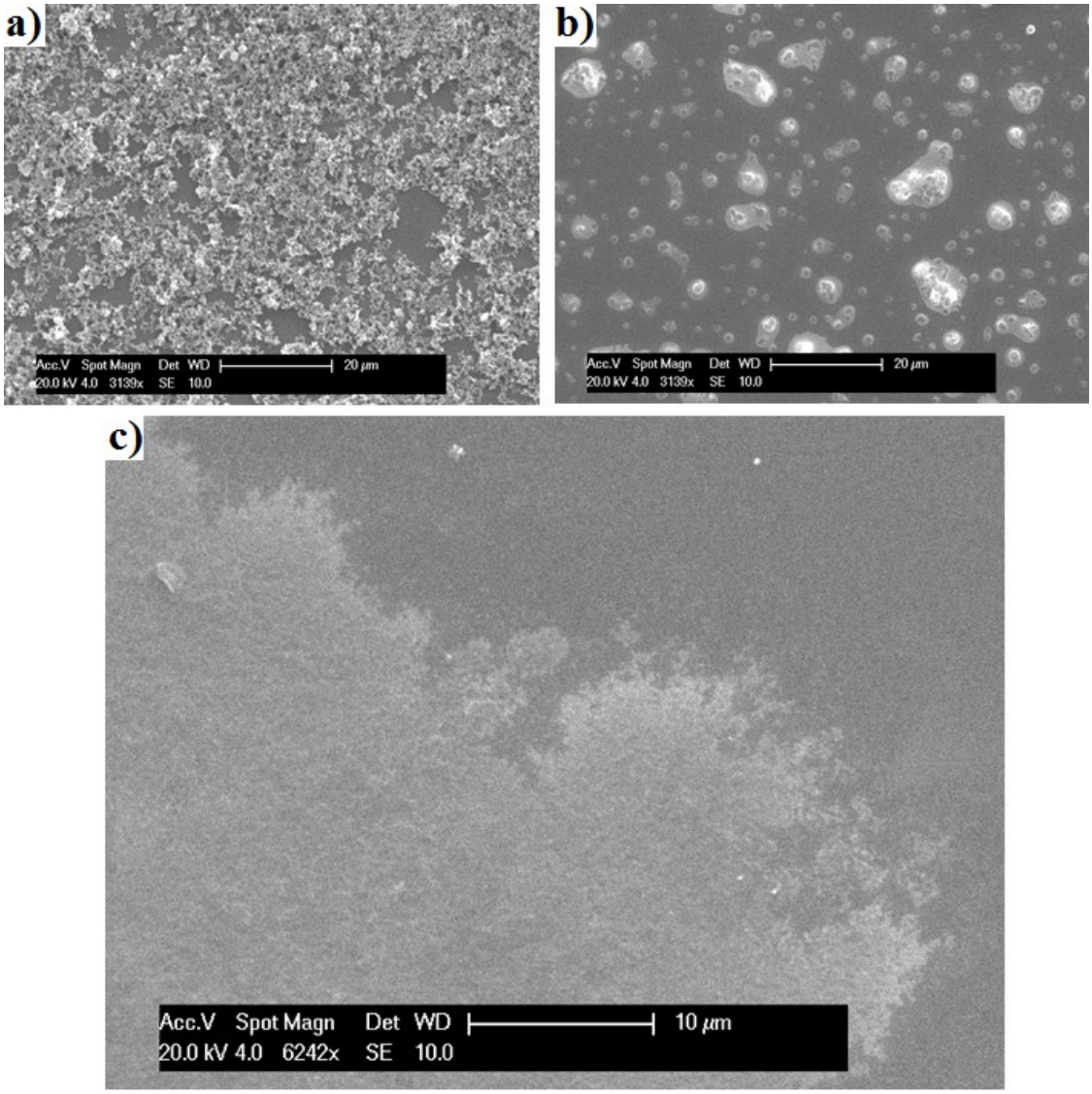


Fig. S7 SEM of **TArcNDI** in the (a) $CHCl_3$/hexane (f_h = 90%), (b) $CHCl_3$/MeOH (f_m = 70%) and (c) THF/water (f_w = 70%), respectively.

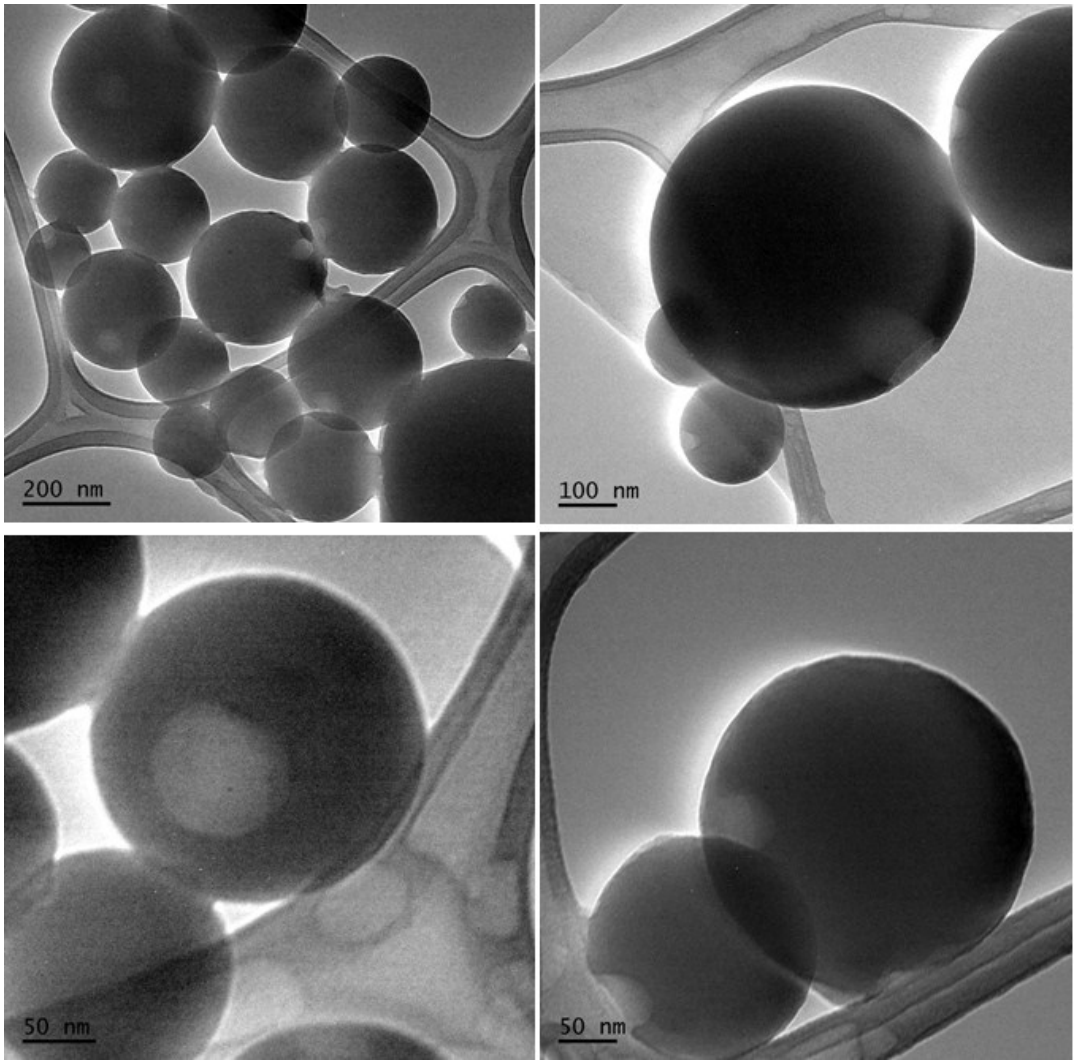


Fig. S8 TEM images of **TTPEcNDI** in the $CHCl_3$/hexane (f_h = 90%).

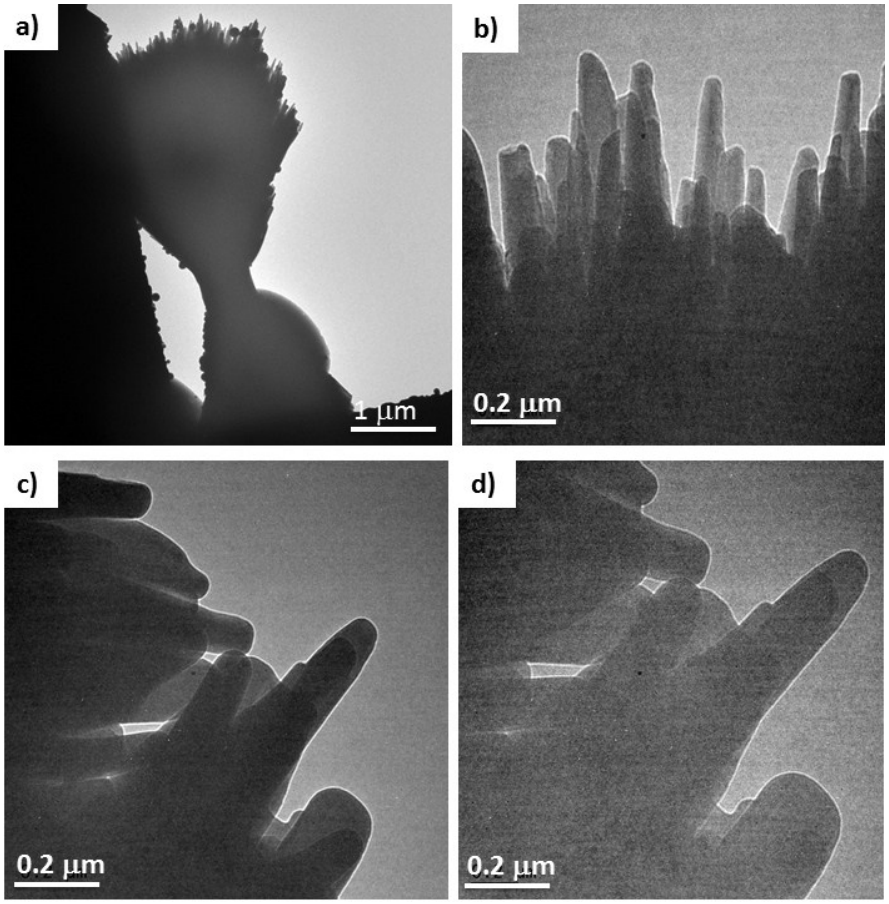


Fig. S9 TEM images of **TTPEcNDI** in the (a & b) (b) $CHCl_3$/MeOH (f_m = 70%) and (c & d) THF/water (f_w = 40%).

X-ray diffraction (XRD):

To examine the mode of self-assembly within the nanostructures, X-ray diffraction (XRD) measurements were carried out. The low angle XRD display the strongest sharp diffraction peaks at around 36° and 43.5° demonstrate that the resulted materials are highly crystalline (Fig. S10).[20]

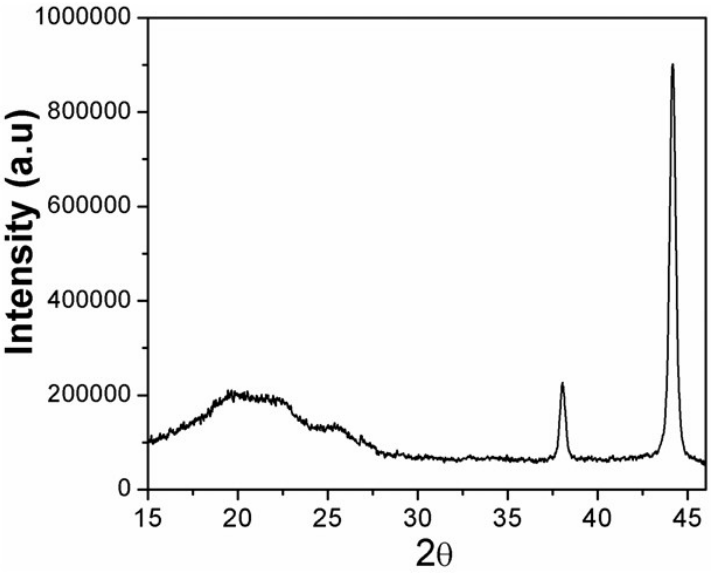


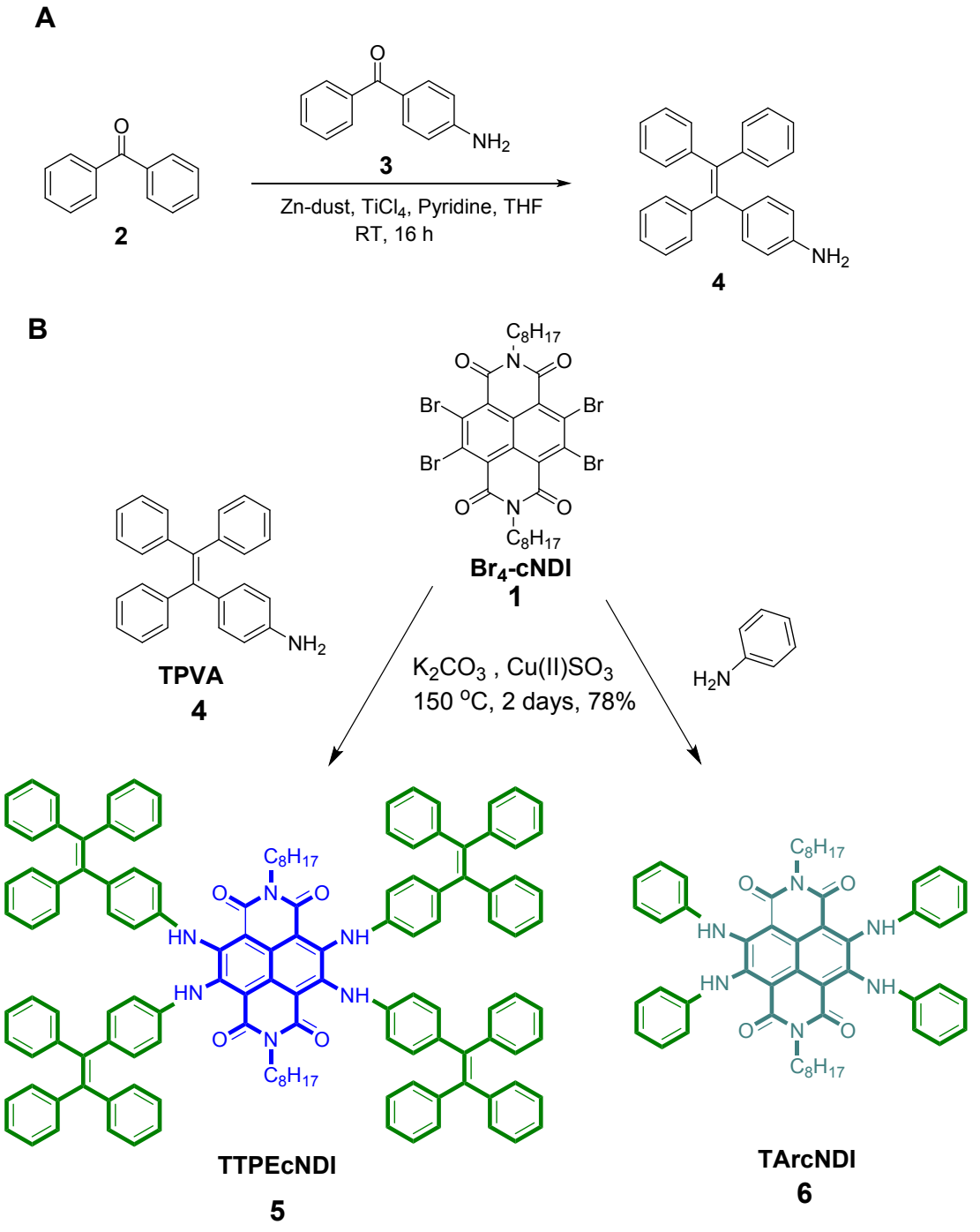
Fig. S10 XRD pattern of nanobelts (films prepared by evaporating premixed solution of **TTPEcNDI** from THF/water (f_w = 40%) on a silicon wafer for 2 h and plotted against the angle 2θ at 298 K).

Table S1. Optical properties[a]

	Solvent	**UV-vis (λ_{max})**	**PL (λ_{max})**	**Φ_F (%)**
TTPEcNDI				
	Chloroform	729	569 789	0.07
	DCM	728	568 787	0.08
	Toluene	719	571 792	19.3
	n-hexane	713	567 789	30.1
	THF	707	575 793	0.09
	DMF	707	579 791	17.8
	Ethanol	703	618 795	25.7
	Acetonitrile	704	512 773	3.01

a = UV-vis absorption peaks at concentration = 10^{-5} M and PL λ_{max} was measured at λ_{ex} for 2-TPEcNDI at 501 and for **TTPEcNDI** at 710 nm, respectively. The fluorescence quantum efficiency (Φ_F) of the samples with absorption (intensity ~0.05) was estimated using fluorescein in ethanol (Φ_F = 70%) as standard solution.

Synthesis of target derivatives:



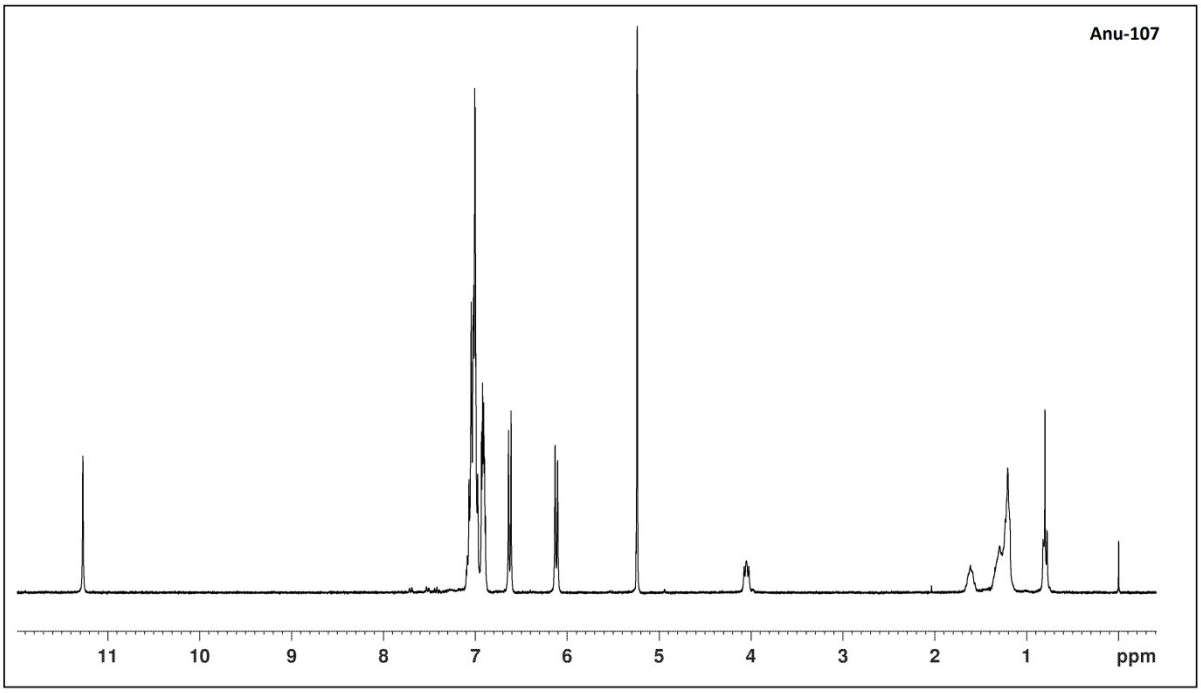
Scheme S1. Synthesis of **TTPEcNDI** (**5**) and **TArcNDI** (**6**).

Synthesis of 4-(1, 2, 2-triphenylvinyl) aniline (**TPAV**) **4:** We have reported synthesis of **TPAV** earlier, however, make it easy to followers, herein we outline the synthetic procedure.[S2] A 2-necked round bottom flask was charged with Zn powder (8.04 g, 123.37 mmol) and THF (460 mL) under an argon atmosphere. The mixture was cooled to -5 °C to 0 °C, and $TiCl_4$ (6.72 mL, 61.68 mmol) was added slowly by syringe with the temperature maintained under 10 °C. The suspended mixture was warmed to r.t. and stirred for 0.5 h, then heated to reflux for 2.5 h. The mixture was again cooled to -5 °C to 0 °C, charged with pyridine (4 mL), and stirred for 10 min. The solution of **2** (4.0 g, 22 mmol) and **3** (3.5 g, 17.78 mmol) in THF (60 mL) was added slowly. When the addition was complete, the mixture was heated to reflux for 16 h. Reaction completion was checked by TLC analysis. After completion, reaction was quenched by addition of 10% aq K_2CO_3 and extracted with DCM. Organic layer was dried over sodium sulphate and evaporated on rotary evaporator to obtain crude compound which was purified by flash chromatography to afford pure compound **4** as a dark yellow solid. (5 g yield 65%). M.P. = 230 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 7.19–6.98 (m, 15H), 6.86–6.77 (m, 2H), 6.48–6.40 (m, 2H), 3.59 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.78, 144.35, 144.22, 144.18, 140.96, 139.33, 134.04, 132.50, 131.48, 131.41, 131.36, 127.67, 127.54, 127.51, 126.24, 126.06, 114.33; HRMS (m/z): [M^+] calcd for $C_{26}H_{21}N$: 347.1674, found: 347.1675. Elemental Analysis for $C_{26}H_{21}N$: calcd. C, 89.88; H, 6.09; N, 4.03 and found C, 89.81; H, 6.03; N, 4.01.

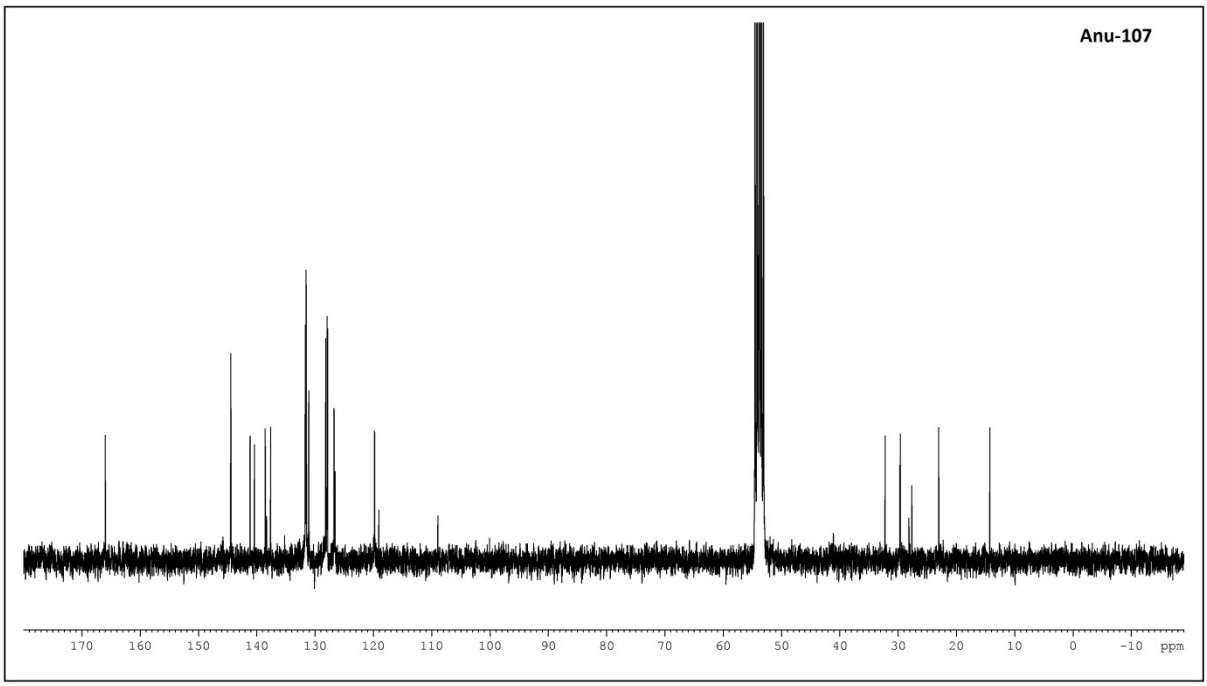
Synthesis of **TTPEcNDI** (**5**): A mixture of Br_4-cNDI[S3] **1** (0.15g, 0.186 mmol), TPAV **3** (0.107 g, 0.932 mmol), anhydrous K_2CO_3 (0.127 g, 0.932 mmol) and $CuSO_4$ (0.15 g) was grinded properly for 10 min and then resultant mixture was heated at 150 ^oC for 3 days. Reaction completion was checked by TLC analysis. After completion, resultant residue was dissolved in water and extracted with $CHCl_3$ (3 x 20 mL). Organic layer were separated, dried over sodium sulphate and evaporated on rotary evaporator to give crude residue which was further purified by silica gel flash column chromatography to give 2,7-dioctyl-4-((4-(1,2,2-triphenylvinyl) phenyl)amino)benzo[lmn] [3,8]phenanthroline-1,3,6,8(2H,7H)-tetraone (**5**) as a green solid (0.110 g, yield 31.6%). M.P. >300 ^oC. ^1H NMR (300 MHz, CD_2Cl_2) δ 11.27 (s, 4H), 7.12–6.84 (m, 64H), 6.62 (d, J = 8.5 Hz, 8H), 6.12 (d, J = 8.4 Hz, 8H), 4.13–3.95 (m, 4H), 1.61 (m, 4H), 1.40–1.10 (m, 20H), 0.80 (t, J = 6.8 Hz, 8H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 165.94, 157.28, 144.51, 144.43, 141.01, 140.44, 131.69, 131.56,

131.52, 131.10, 128.19, 127.96, 127.88, 126.76, 126.60, 119.81, 32.22, 29.70, 29.61, 23.03, 16.95, 16.45, 14.26; IR (KBr, cm^{-1}): 3262, 2967, 2939, 2853, 1647, 1633, 1581, 1529, 1453, 1281, 1175, 1147, 1122, 849, 791, 727; FT-IR (cm^{-1}): 2923, 2850, 1649, 1631, 1569, 1545, 1510, 1467, 1320, 1307, 1267, 750; MALDI-TOF (m/z): [M^+] calcd for $C_{134}H_{114}N_6O_4$: 1870.890, found: 1870.901. HRMS calcd for $C_{134}H_{114}N_6O_4$: 1870.8902, found: 1870.8901. Elemental Analysis for $C_{26}H_{21}N$: calcd. C, 85.96; H, 6.14; N, 4.49 and found C, 85.98; H, 6.13; N, 4.46.

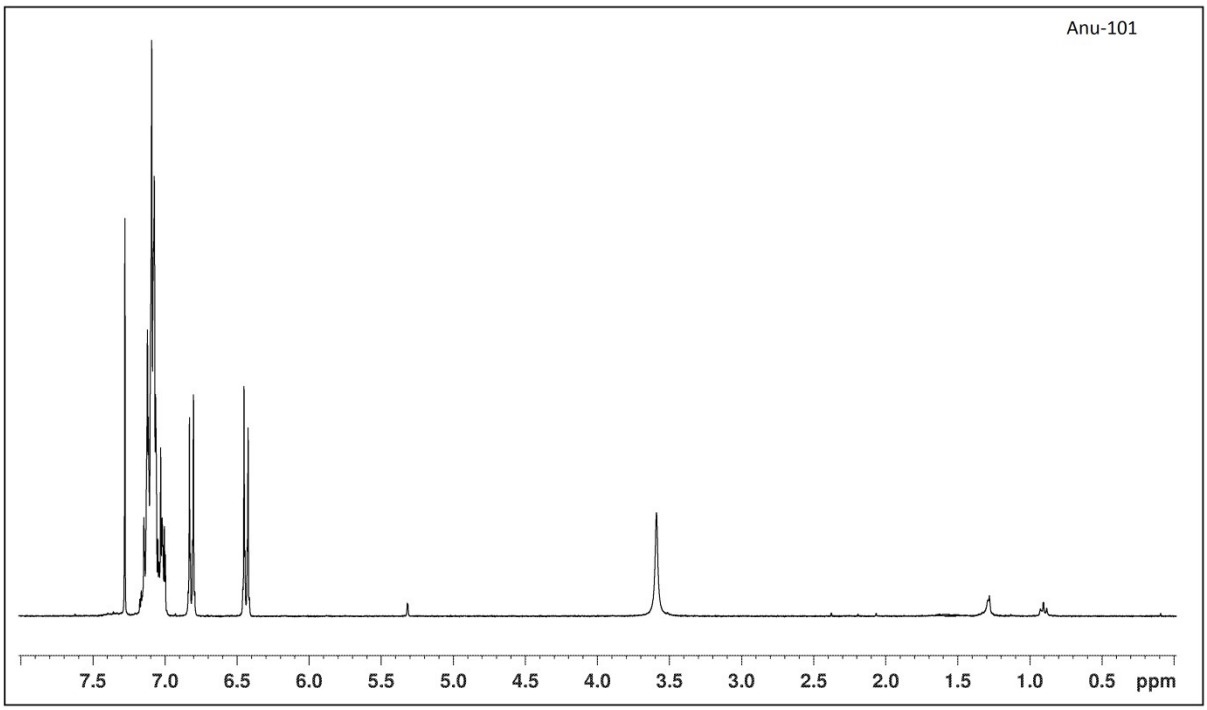
Spectroscopic characterisation:



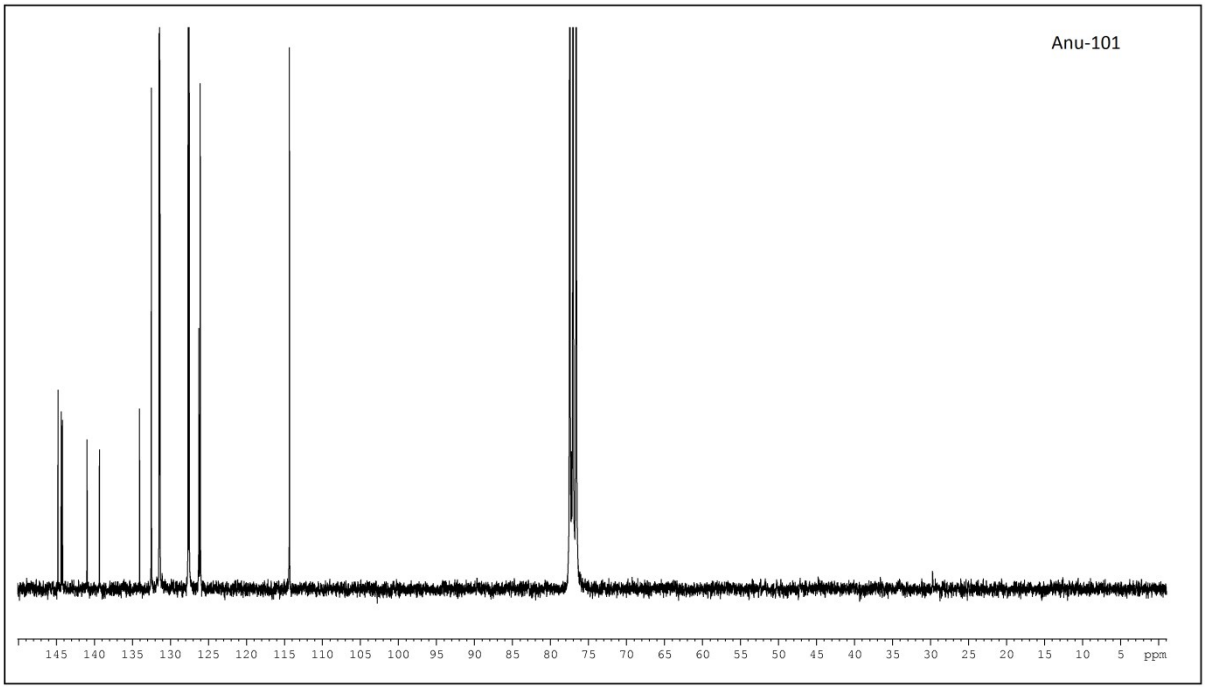
^{1}H NMR of **TTPEcNDI** (**5**)



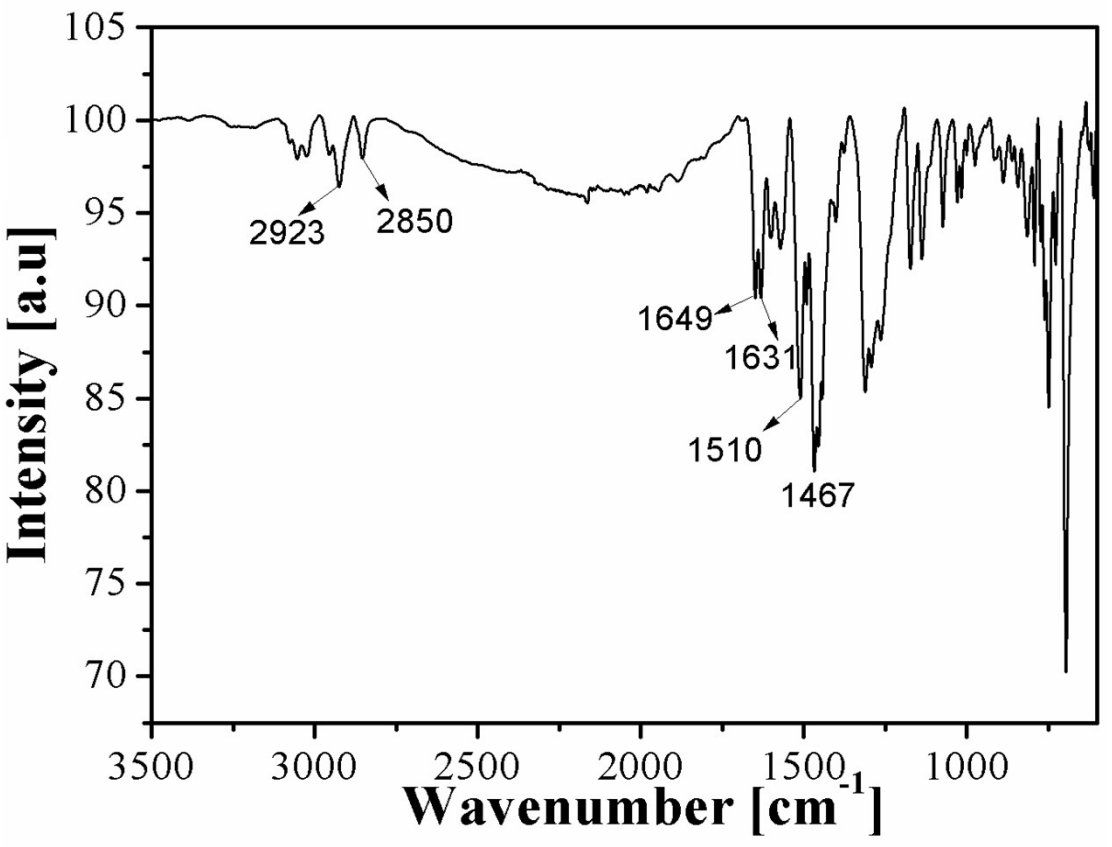
^{13}C NMR of **TTPEcNDI** (**5**)



^{1}H NMR of 4-(1, 2, 2-triphenylvinyl) aniline (**TPAV**) **4**



^{13}C NMR of 4-(1, 2, 2-triphenylvinyl) aniline (**TPAV**) **4**



FTIR of **TTPEcNDI** (**5**)

X-ray crystlography

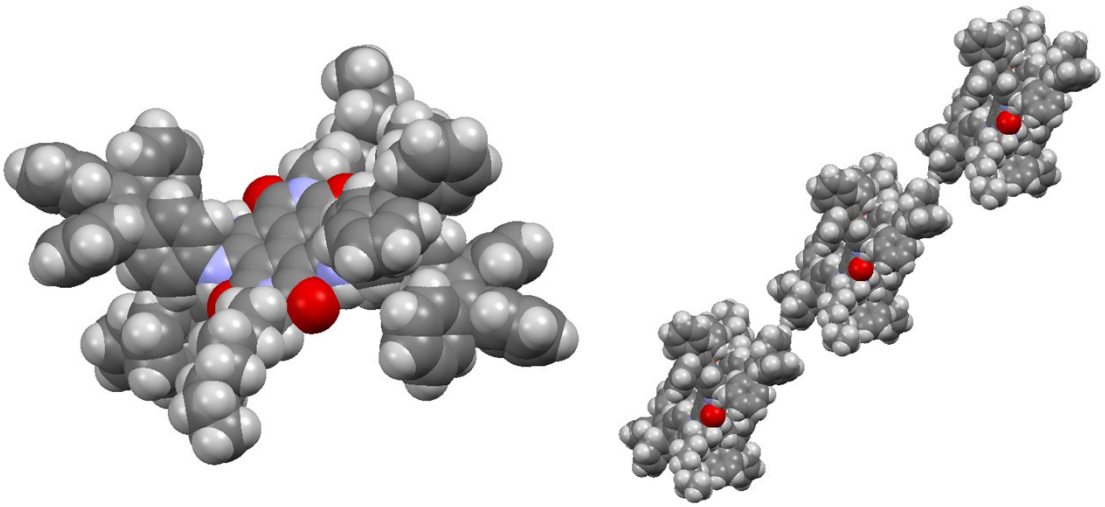


Figure S11. Space filling model of **TTPEcNDI 1**

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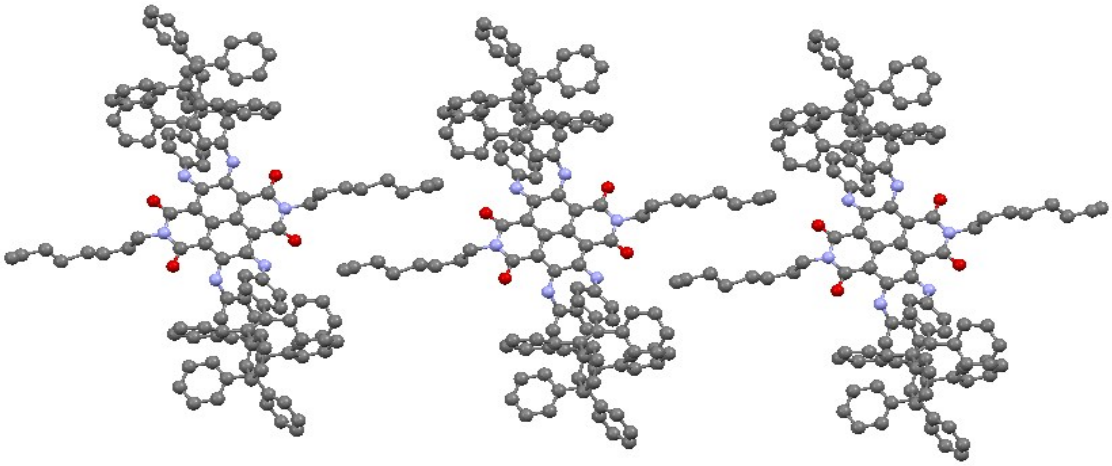

Figure S12. TTPEcNDI 1 viewed along the *a* axis to show the alkyl chain interactions. Hydrogen removed for clarity

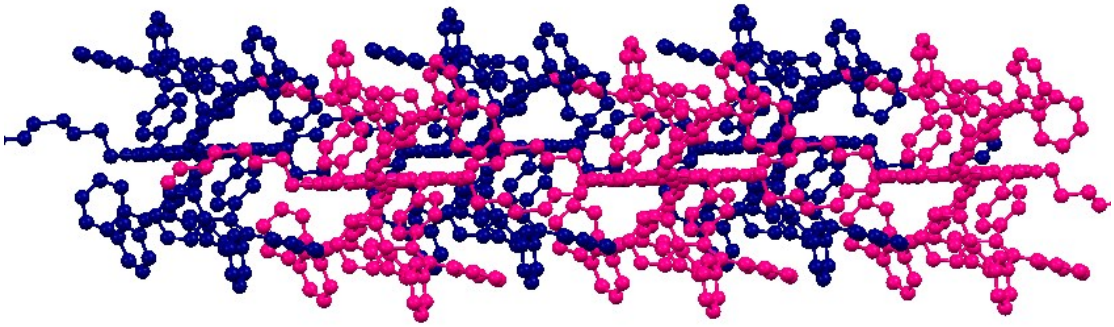

Figure S13. TTPEcNDI 1 viewed along the *b* axis. False colour has been used to view phenyl ring interactions. Hydrogen removed for clarity.

Bond Lengths

C11 C14 1.521(12)
C11 C12 1.372(12)
C11 C10 1.382(12)
C8 C13 1.383(12)
C8 C9 1.369(11)
C8 N3 1.422(10)
C14 C15 1.328(12)
C14 C28 1.495(14)
C9 C10 1.393(11)
C15 C22 1.531(12)
C15 C16 1.484(12)
C26 C27 1.405(14)
C26 C25 1.388(16)
C27 C22 1.396(13)
C22 C23 1.366(12)
C23 C24 1.358(14)
C24 C25 1.350(17)
C33 C28 1.350(15)
C33 C32 1.366(16)
C28 C29 1.390(14)
C29 C30 1.377(16)
C30 C31 1.37(2)
C31 C32 1.36(2)
C42 C40 1.513(13)
C42 C43 1.417(13)
C42 C47 1.342(13)
C48 C53 1.419(13)
C48 C49 1.395(12)
C48 C41 1.454(13)
C53 C52 1.359(14)
C52 C51 1.348(14)
C51 C50 1.386(15)
C50 C49 1.354(13)
C40 C41 1.323(13)
C40 C37 1.512(13)
C43 C44 1.383(15)
C41 C54 1.539(14)
C47 C46 1.310(16)
C46 C45 1.352(18)
C55 C54 1.335(14)
C55 C56 1.329(16)
C54 C59 1.395(13)
C59 C58 1.353(16)
C58 C57 1.383(18)
C57 C56 1.343(18)
C37 C36 1.392(11)
C37 C38 1.383(11)
C36 C35 1.347(11)
C35 C34 1.401(11)
C34 C39 1.387(11)

C34 N2 1.414(10)
C39 C38 1.376(12)
N2 C6 1.374(10)
N3 C7 1.370(10)
C6 C7 1.419(11)
C6 C2 1.386(11)
C7 C4 1.434(11)
O1 C5 1.224(9)
C3 C3 1.396(15)
C3 C4 1.398(11)
C3 C2 1.435(11)
C4 C7 1.434(11)
C4 C5 1.434(11)
C5 N1 1.403(10)
N1 C1 1.374(10)
N1 C60 1.470(10)
C45 C44 1.427(18)
O2 C1 1.250(9)
C1 C2 1.443(11)
C17 C16 1.401(12)
C17 C18 1.368(13)
C16 C21 1.339(12)
C21 C20 1.386(13)
C20 C19 1.394(13)
C19 C18 1.359(14)
Cl5 C72 1.776(14)
Cl5 C71 1.675(13)
Cl4 C72 1.800(12)
Cl4 C71 1.717(13)
Cl6 C72 1.814(13)
Cl6 C71 1.716(13)
Cl3 C68 1.701(14)
Cl2 C68 1.699(12)
C68 Cl1 1.721(15)
C60 C61 1.513(13)
C62 C61 1.502(14)
C62 C63 1.387(16)
C64 C65 1.41(2)
C64 C63 1.485(18)
C65 C66 1.457(18)
C66 C67 1.308(16)
Cl11 C70 1.79(2)
Cl10 C70 1.80(2)
Cl9 C70 1.79(2)
Cl7 C69 1.80(2)
Cl8 C69 1.79(2)
C69 Cl12 1.79(2)

Bond Angles

C12 C11 C14 118.2(9) .

C12 C11 C10 117.9(8) .
C10 C11 C14 123.9(8) .
C13 C8 N3 115.3(8) .
C9 C8 C13 120.5(8) .
C9 C8 N3 124.1(9) .
C15 C14 C11 123.0(9) .
C15 C14 C28 122.7(8) .
C28 C14 C11 114.4(8) .
C8 C13 H13 120.8 .
C8 C13 C12 118.5(9) .
C12 C13 H13 120.8 .
C11 C12 C13 122.4(9) .
C11 C12 H12 118.8 .
C13 C12 H12 118.8 .
C8 C9 H9 120.1 .
C8 C9 C10 119.8(9) .
C10 C9 H9 120.1 .
C14 C15 C22 118.8(9) .
C14 C15 C16 127.6(8) .
C16 C15 C22 113.6(7) .
C11 C10 C9 121.0(8) .
C11 C10 H10 119.5 .
C9 C10 H10 119.5 .
C27 C26 H26 120.0 .
C25 C26 H26 120.0 .
C25 C26 C27 119.9(11) .
C26 C27 H27 121.2 .
C22 C27 C26 117.5(10) .
C22 C27 H27 121.2 .
C27 C22 C15 117.0(8) .
C23 C22 C15 122.9(9) .
C23 C22 C27 120.1(9) .
C22 C23 H23 119.0 .
C24 C23 C22 121.9(11) .
C24 C23 H23 119.0 .
C23 C24 H24 120.3 .
C25 C24 C23 119.3(11) .
C25 C24 H24 120.3 .
C26 C25 H25 119.5 .
C24 C25 C26 120.9(11) .
C24 C25 H25 119.5 .
C28 C33 H33 118.6 .
C28 C33 C32 122.8(14) .
C32 C33 H33 118.6 .
C33 C28 C14 122.5(11) .
C33 C28 C29 118.6(11) .
C29 C28 C14 118.9(11) .
C28 C29 H29 120.6 .
C30 C29 C28 118.7(13) .
C30 C29 H29 120.6 .

C29 C30 H30 119.6 .
C31 C30 C29 120.8(15) .
C31 C30 H30 119.6 .
C30 C31 H31 119.9 .
C32 C31 C30 120.2(15) .
C32 C31 H31 119.9 .
C33 C32 H32 120.7 .
C31 C32 C33 118.6(14) .
C31 C32 H32 120.7 .
C43 C42 C40 118.5(9) .
C47 C42 C40 121.9(10) .
C47 C42 C43 119.3(10) .
C53 C48 C41 119.8(10) .
C49 C48 C53 114.9(9) .
C49 C48 C41 125.0(9) .
C48 C53 H53 119.3 .
C52 C53 C48 121.4(10) .
C52 C53 H53 119.3 .
C53 C52 H52 119.3 .
C51 C52 C53 121.4(10) .
C51 C52 H52 119.3 .
C52 C51 H51 120.3 .
C52 C51 C50 119.3(11) .
C50 C51 H51 120.3 .
C51 C50 H50 120.2 .
C49 C50 C51 119.7(11) .
C49 C50 H50 120.2 .
C48 C49 H49 118.4 .
C50 C49 C48 123.1(10) .
C50 C49 H49 118.4 .
C41 C40 C42 123.3(10) .
C41 C40 C37 124.2(10) .
C37 C40 C42 112.5(8) .
C42 C43 H43 120.6 .
C44 C43 C42 118.7(11) .
C44 C43 H43 120.6 .
C48 C41 C54 117.7(9) .
C40 C41 C48 124.3(11) .
C40 C41 C54 118.0(10) .
C42 C47 H47 119.6 .
C46 C47 C42 120.8(13) .
C46 C47 H47 119.6 .
C47 C46 H46 117.4 .
C47 C46 C45 125.2(14) .
C45 C46 H46 117.4 .
C54 C55 H55 118.9 .
C56 C55 H55 118.9 .
C56 C55 C54 122.3(12) .
C55 C54 C41 123.4(10) .
C55 C54 C59 120.0(11) .

C59 C54 C41 116.5(10) .
C54 C59 H59 121.0 .
C58 C59 C54 118.1(11) .
C58 C59 H59 121.0 .
C59 C58 H58 120.2 .
C59 C58 C57 119.7(13) .
C57 C58 H58 120.2 .
C58 C57 H57 119.5 .
C56 C57 C58 120.9(13) .
C56 C57 H57 119.5 .
C55 C56 C57 119.0(13) .
C55 C56 H56 120.5 .
C57 C56 H56 120.5 .
C36 C37 C40 117.5(8) .
C38 C37 C40 125.0(8) .
C38 C37 C36 117.2(8) .
C37 C36 H36 118.9 .
C35 C36 C37 122.1(8) .
C35 C36 H36 118.9 .
C36 C35 H35 119.9 .
C36 C35 C34 120.2(8) .
C34 C35 H35 119.9 .
C35 C34 N2 123.2(7) .
C39 C34 C35 118.7(9) .
C39 C34 N2 118.0(8) .
C34 C39 H39 120.1 .
C38 C39 C34 119.7(8) .
C38 C39 H39 120.1 .
C37 C38 H38 119.1 .
C39 C38 C37 121.8(8) .
C39 C38 H38 119.1 .
C34 N2 H2 117.3 .
C6 N2 C34 125.3(7) .
C6 N2 H2 117.3 .
C8 N3 H3 116.0 .
C7 N3 C8 128.1(8) .
C7 N3 H3 116.0 .
N2 C6 C7 119.0(8) .
N2 C6 C2 121.2(7) .
C2 C6 C7 119.8(7) .
N3 C7 C6 123.2(7) .
N3 C7 C4 118.6(8) .
C6 C7 C4 118.0(8) .
C3 C3 C4 121.3(9)
C3 C3 C2 118.6(10)
C4 C3 C2 119.9(7) .
C3 C4 C7 119.6(8)
C3 C4 C5 120.6(7) .
C5 C4 C7 119.8(8)
O1 C5 C4 125.3(8)

O1 C5 N1 116.1(8) .
N1 C5 C4 118.5(9) .
C5 N1 C60 117.7(8) .
C1 N1 C5 122.3(8) .
C1 N1 C60 119.9(7) .
C46 C45 H45 122.0 .
C46 C45 C44 116.0(12)
C44 C45 H45 122.0 .
C43 C44 C45 119.9(12) .
C43 C44 H44 120.0 .
C45 C44 H44 120.0 .
N1 C1 C2 119.8(8) .
O2 C1 N1 117.6(8) .
O2 C1 C2 122.6(8) .
C16 C17 H17 119.8 .
C18 C17 H17 119.8 .
C18 C17 C16 120.4(10) .
C17 C16 C15 122.6(9) .
C21 C16 C15 119.9(8) .
C21 C16 C17 117.5(9) .
C16 C21 H21 118.8 .
C16 C21 C20 122.5(9) .
C20 C21 H21 118.8 .
C21 C20 H20 120.0 .
C21 C20 C19 119.9(10) .
C19 C20 H20 120.0 .
C20 C19 H19 121.3 .
C18 C19 C20 117.4(11) .
C18 C19 H19 121.3 .
C17 C18 H18 118.9 .
C19 C18 C17 122.2(10) .
C19 C18 H18 118.9 .
Cl3 C68 H68 107.8 .
Cl3 C68 Cl1 108.9(7) .
Cl2 C68 Cl3 114.4(9) .
Cl2 C68 H68 107.8 .
Cl2 C68 Cl1 110.0(8) .
Cl1 C68 H68 107.8 .
N1 C60 H60A 109.7 .
N1 C60 H60B 109.7 .
N1 C60 C61 110.0(7) .
H60A C60 H60B 108.2 .
C61 C60 H60A 109.7 .
C61 C60 H60B 109.7 .
H62A C62 H62B 105.8 .
C61 C62 H62A 105.0 .
C61 C62 H62B 105.0 .
C63 C62 H62A 105.0 .
C63 C62 H62B 105.0 .
C63 C62 C61 129.3(16) .

H64A C64 H64B 107.1 .
C65 C64 H64A 107.6 .
C65 C64 H64B 107.6 .
C65 C64 C63 119(3) .
C63 C64 H64A 107.6 .
C63 C64 H64B 107.6 .
C64 C65 H65A 109.8 .
C64 C65 H65B 109.8 .
C64 C65 C66 109(3) .
H65A C65 H65B 108.3 .
C66 C65 H65A 109.8 .
C66 C65 H65B 109.8 .
C60 C61 H61A 110.1 .
C60 C61 H61B 110.1 .
C62 C61 C60 107.9(10) .
C62 C61 H61A 110.1 .
C62 C61 H61B 110.1 .
H61A C61 H61B 108.4 .
C65 C66 H66A 107.5 .
C65 C66 H66B 107.5 .
H66A C66 H66B 107.0 .
C67 C66 C65 119(2) .
C67 C66 H66A 107.5 .
C67 C66 H66B 107.5 .
C62 C63 C64 118.5(18) .
C62 C63 H63A 107.7 .
C62 C63 H63B 107.7 .
C64 C63 H63A 107.7 .
C64 C63 H63B 107.7 .
H63A C63 H63B 107.1 .
Cl5 C72 Cl4 107.7(8) .
Cl5 C72 Cl6 107.4(9) .
Cl5 C72 H72 112.3 .
Cl4 C72 Cl6 104.6(8) .
Cl4 C72 H72 112.3 .
Cl6 C72 H72 112.3 .
Cl5 C71 Cl4 116.6(10) .
Cl5 C71 Cl6 117.1(9) .
Cl5 C71 H71 102.4 .
Cl4 C71 H71 102.4 .
Cl6 C71 Cl4 112.8(9) .
Cl6 C71 H71 102.4 .
C6 C2 C3 120.4(7) .
C6 C2 C1 120.8(8) .
C3 C2 C1 118.6(8) .
C66 C67 H67A 109.5 .
C66 C67 H67B 109.5 .
C66 C67 H67C 109.5 .
H67A C67 H67B 109.5 .
H67A C67 H67C 109.5 .

H67B C67 H67C 109.5 .
Cl11 C70 Cl10 105.9(15) .
Cl11 C70 Cl9 93.5(15) .
Cl11 C70 H70 117.4 .
Cl10 C70 H70 117.4 .
Cl9 C70 Cl10 101.7(19) .
Cl9 C70 H70 117.4 .
Cl7 C69 H69 118.8 .
Cl8 C69 Cl7 100.0(15) .
Cl8 C69 H69 118.8 .
Cl12 C69 Cl7 97.6(14) .
Cl12 C69 Cl8 98.7(16) .
Cl12 C69 H69 118.8 .

References

S1. Villamaina, D.; Kelson, M. M. A.;Bhosale S. V.; Vauthey, E. *Phys. Chem. Chem. Phys.* **2014**, *16*, 5188.

S2. Rananaware, A.; La, D. D.; Bhosale S. V. *RSC. Adv.* 2015, **5**, 63130.

S3. Bhosale, S. V.; Al-Kobaisi, M.; Bhosale, R. S.; Bhosale, S. V. *RSC Adv.* **2013**, *3*, 19840.