Supporting information

Local and long-range helical structures of dendronized bisnaphthalimide mesogens with tunable torsional or planar configuration

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Content

1. Additional Experimental Data

1.1 Experimental techniques

A Mettler heating stage (Linkam T9) was used for polarizing optical microscopy (POM, Leica DM2700P) and DSC were recorded with a DSC 200 F3 Maia calorimeter (NETZSCH) at 10 K·min^{-1}.

SEM experiments were carried out on a ZEISS SIGMA 300 scanning electron microscopy (SEM, GER). All pictures were taken digitally. For the sample preparation, the gel was placed on an aluminium foil for some time until the gel became xerogel, then the sample was gold plated, finally the sample was put into the scanning electron microscopy for observation. XRD measurement of LC samples and xerogels was used the X-ray powder diffraction (XRD, Rigaku Co., Tokyo, Japan), analysis was conducted on a D/max-3B spectrometer with Cu Kα radiation.

For Electron density reconstruction. Fourier reconstruction of the electron density was carried out using the general formula for 2D periodic systems:

$$E(xy) = \Sigma_{hk}\,\mathrm{sqrt}[I(hk)]\,\exp[i2\pi(hx+ky)+\phi_{hk}]$$

For the centro-symmetric structures considered in this work the phase angle ϕ can take up the values of 0 or π. The choice of a phase combination was initially made on the merit of each reconstructed electron density map obtained using the most intense reflections, combined with the additional knowledge of the molecules (molecular shape, length, volume of each part and the distribution of electron density among the different moieties).

The UV-vis absorption and fluorescence spectra were carried out on UV2600A UV-vis absorption spectrometer (UNICO, China) and Hitachi F-7000 fluorescence spectrometer (Hitachi, Japan), IR spectra were carried out on Nicolet is10 (Thermo Fisher, US).

The quantum yields of compounds in THF were determined using quinolinium hydrogen sulphate in H_2SO_4 (Φ_F=0.55) as standard and applying the following equation.[S1]

$$\Phi_{F_x} = \Phi_{F_{st}} \frac{A_{st} F_x {n_x}^2}{A_x F_{st} {n_{st}}^2}$$

Φ_{F_x}= the quantum yields of the samples; $\Phi_{F_{st}}$= the quantum yield of the standard; A = the absorbance of the solution; F = the integration of corrected fluorescence spectrum; n = the average refractive index of the solution.

Molecular models were built using Materials Studio (Accelrys). Geometry optimization were performed using Gaussian 09 with B3LYP/6-31G (d) level.

1.2 Additional DSC traces and XRD data

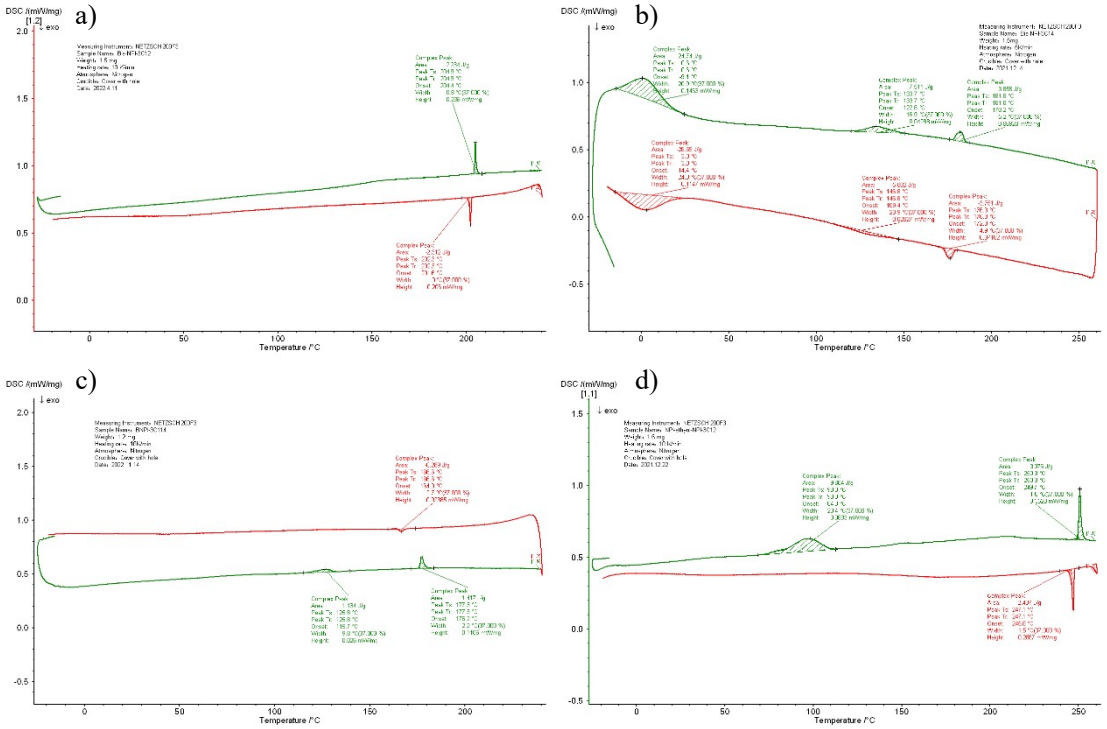


Fig. S1 DSC curves of (a) compound **BNI-3C12** (10 K·min^{-1}, first scan); (b) compound **BNI-3C14** (5 K·min^{-1}, first scan); (c) compound **BNI-3C114** (10 K·min^{-1}, first scan); compound **ABNI-3C12** (10 K·min^{-1}, first scan).

Table S1 Experimental and calculated *d*-spacings of the observed XRD reflections of the Col$_h$/*p6mm* phase in compound **BNI-3C12** at 100 °C. All intensity values are Lorentz and multiplicity corrected.

(*hk*)	d_{obs}-spacing(nm)	d_{cal}-spacing(nm)	*intensity*
10	3.20	3.23	100
11	1.87	1.87	0.5
20	1.63	1.62	0.7
a_{hex} = 3.73 nm			

Table S2 Experimental and calculated *d*-spacings of the observed XRD reflections of the Col$_h$/*p6mm* phase in compound **BNI-3C14** at 70 °C. All intensity values are Lorentz and multiplicity corrected.

(*hk*)	d_{obs}-spacing(nm)	d_{cal}-spacing(nm)	*intensity*
10	3.48	3.48	100
11	2.03	2.01	1.2
20	1.74	1.74	2.8
a_{hex} = 4.02 nm			

Table S3 Experimental and calculated *d*-spacings of the observed XRD reflections of the Col_h/*p6mm* phase in compound **BNI-3C14** at 160 °C. All intensity values are Lorentz and multiplicity corrected.

(*hk*)	d_{obs}-spacing(nm)	d_{cal}-spacing(nm)	*intensity*
10	3.74	3.74	100
a_{hex} = 4.32 nm			

Table S4 Experimental and calculated *d*-spacings of the observed XRD reflections of the Cub_I/*Pm*$\bar{3}$*n* phase in compound **BNI-3C114** at 160 °C. All intensity values are Lorentz and multiplicity corrected.

(*hkl*)	d_{obs}-spacing(nm)	d_{cal}-spacing(nm)	*intensity*
110	5.31	5.34	-
200	3.87	3.78	-
210	3.29	3.38	-
211	3.07	3.08	
310	2.40	2.38	
410	1.83	1.83	
a_{cub} = 7.55 nm			

Table S5 Experimental and calculated *d*-spacings of the observed XRD reflections of the Col_h/*p6mm* phase in compound **ABNI-3C12** at 150 °C. All intensity values are Lorentz and multiplicity corrected.

(*hk*)	d_{obs}-spacing(nm)	d_{cal}-spacing(nm)	*intensity*
10	3.68	3.72	100
11	2.13	2.15	0.4
20	1.86	1.86	1.3
30	1.25	1.24	0.2
a_{hex} = 4.29 nm			

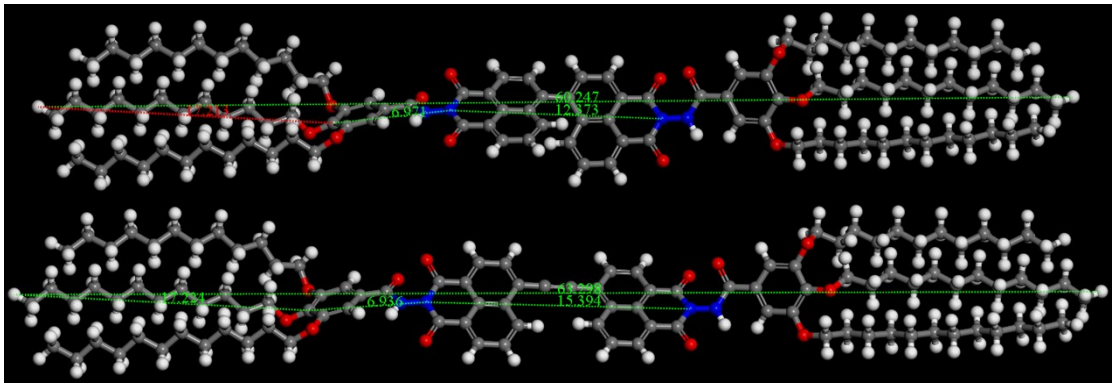


Fig. S2 Molecular structures under stretched molecular configuration for compounds **BNI-3C12** (top) and **ABNI-3C12** (bottom) based on the simulation of Materials Studio 8.0. The length of the

molecules (L_{mol}) as well as per incompatible parts, including aliphatic chains (L_1), benzoylhydrazine spacer (L_2) and aromatic binaphthalimide core (L_3) were measured as 6.02, 1.72, 0.70 and 1.24 nm for **BNI-3C12** and 6.33, 1.72, 0.70 and 1.54 nm for **ABNI-3C12**.

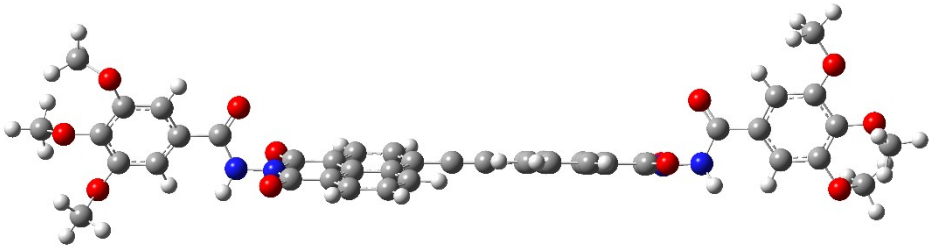


Fig. S3 Optimized molecular geometry of **BNIA-3C1** calculated by density functional theory (DFT) calculations under the B3LYP/6-31G (d) level with Gaussian 09. In order to substantially reduce calculation time, the terminal alkoxy long chains of **ABNI-3C12** were replaced by methoxy groups to be **BNIA-3C1**.

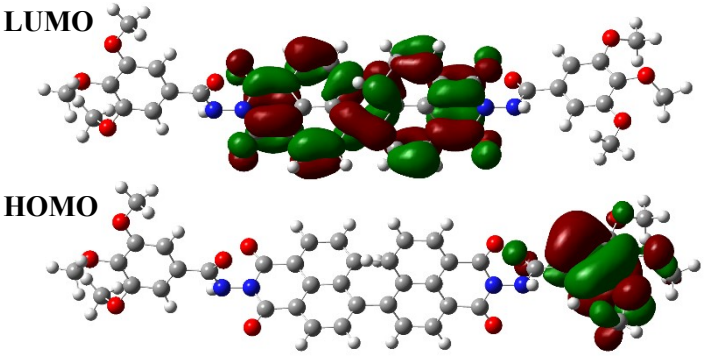


Fig. S4 The frontier molecular orbitals and HOMO-LUMO energy levels for compound **BNI-3C1 (**the methoxy homologs of **BNI-3C*n*)**.

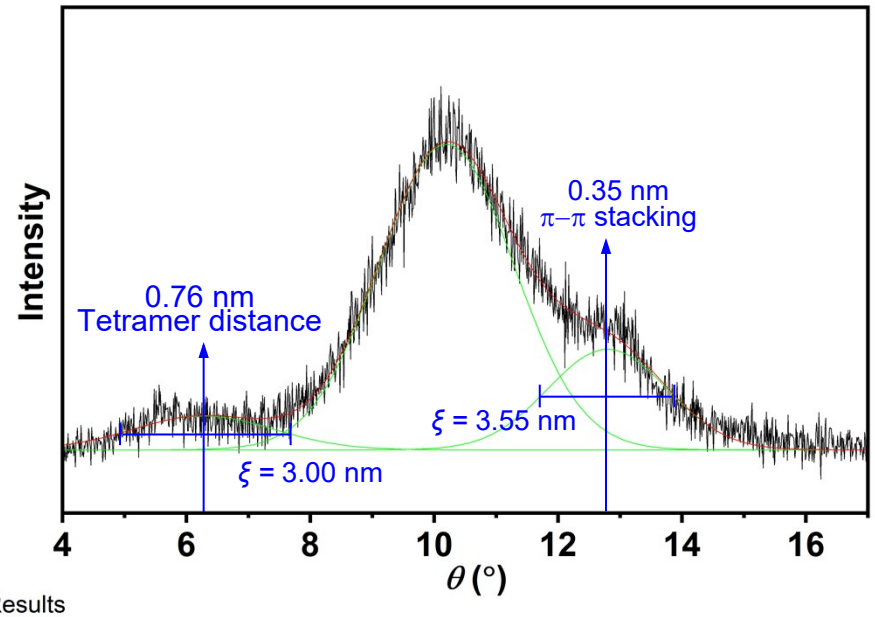
Peak Analysis

Data Set:[Book1]Sheet1!B Date:2023/8/11

BaseLine:Spline

Chi^2=1.58478E+02 Adj. R-Square=9.72174E-01 # of Data Points=1376

SS=2.16639E+05 Degrees of Freedom=1367



Fitting Results

Peak Index	Peak Type	Area Intg	FWHM	Max Height	Center Grvty	Area IntgP
1	Gaussian	75.02453	2.66921	26.75339	6.27378	8.09663
2	Gaussian	659.65349	2.5654	241.56184	10.19431	71.18964
3	Gaussian	191.93648	2.26998	79.43338	12.7982	20.71374

Fig. S5 The representative diffraction plot at wide-angle region with peak deconvolution. The correlation length ξ is estimated from the full width at half-maximum (FWHM).

1.3 Additional gel properties

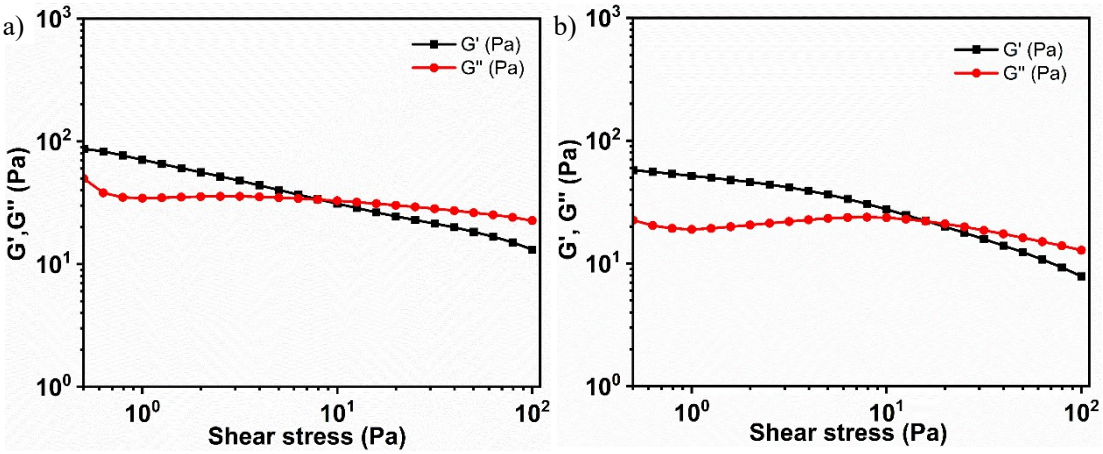
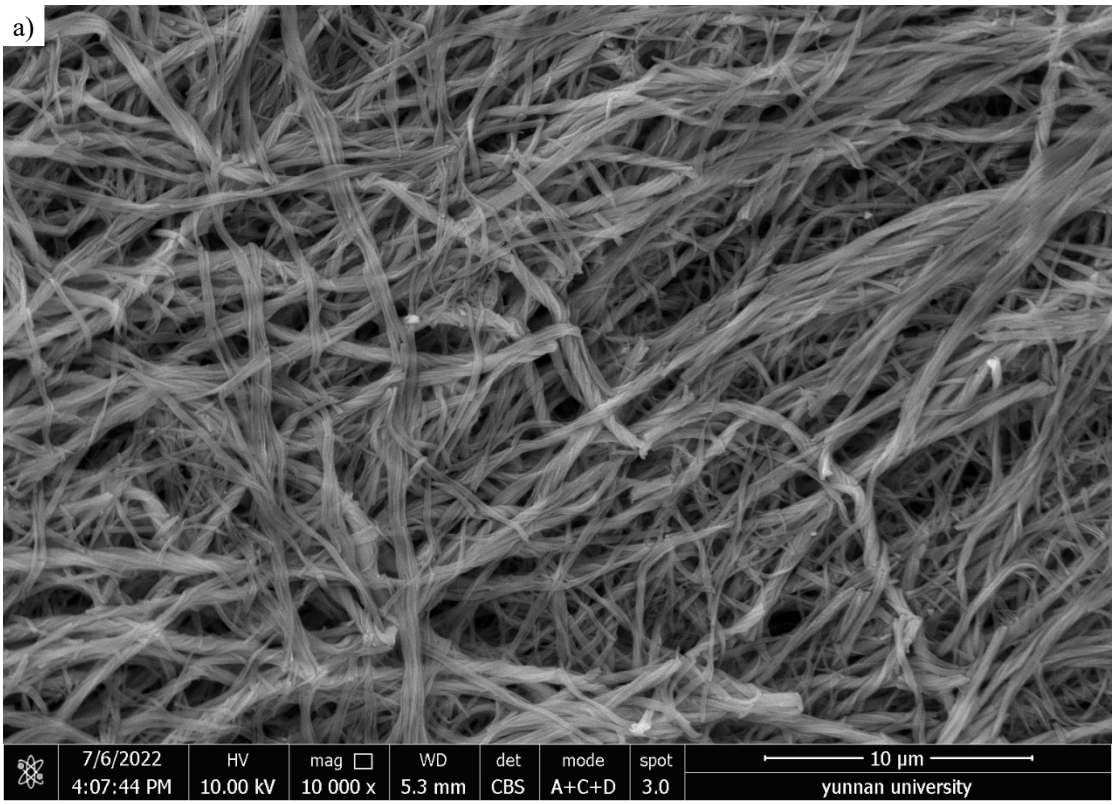


Fig. S6 Variation of storage modulus (G') and loss modulus (G'') with shear stress



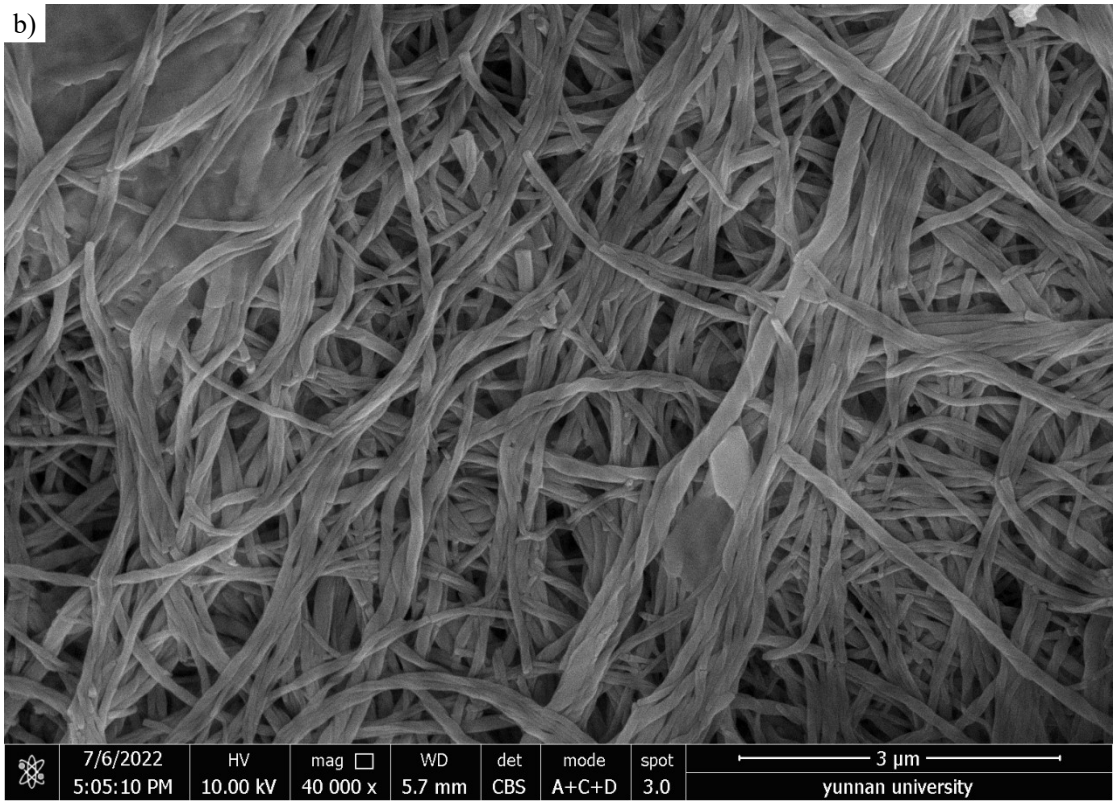


Fig. S7 The large-scale SEM images with supramolecular nanohelix morphologies for the gels of a) **BNI-3C12** in DMF and b) **ABNI-3C12** in acetone, corresponding to that in Fig. 6.

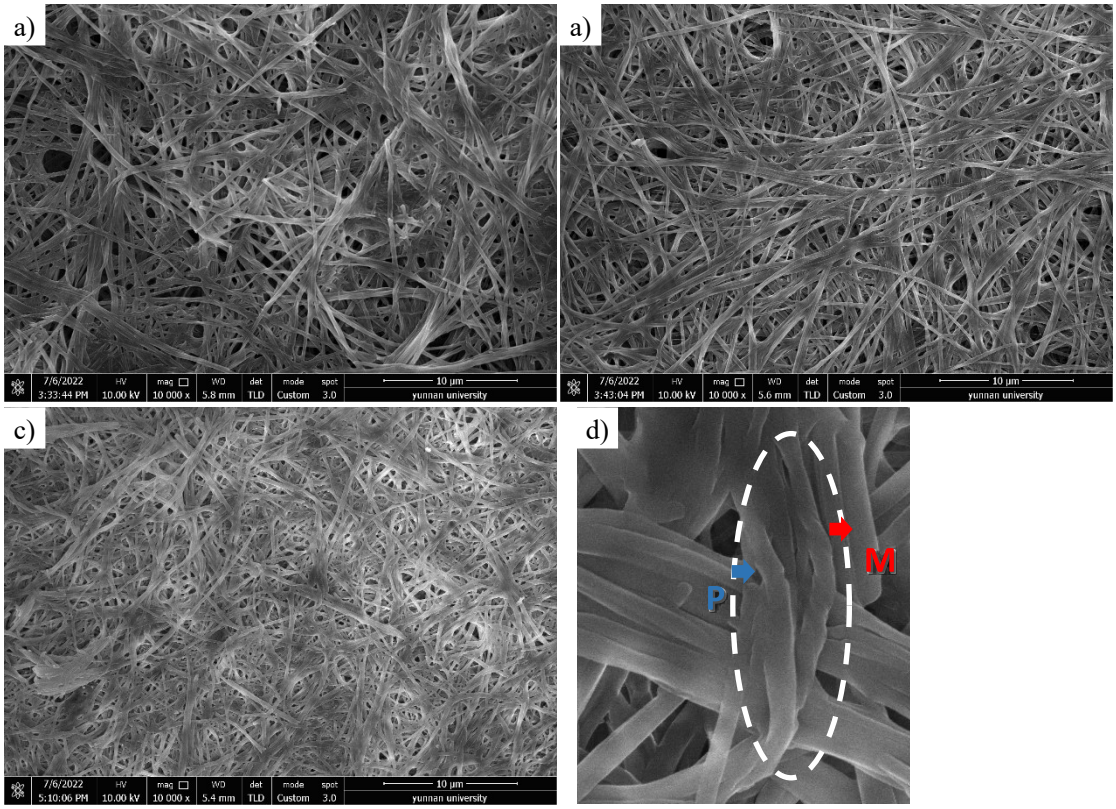


Fig. S8 SEM images of the xerogel of **BNI-3C12** formed in a) ethyl acetate and b) n-butanol, and c) **ABNI-3C12** formed in DMF. Figure S6d is the partial enlarged detail of figureS6c with helical

morphologies containing *P*- and *M*-helix.

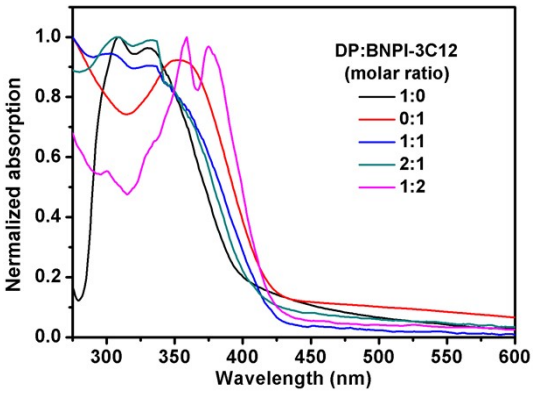
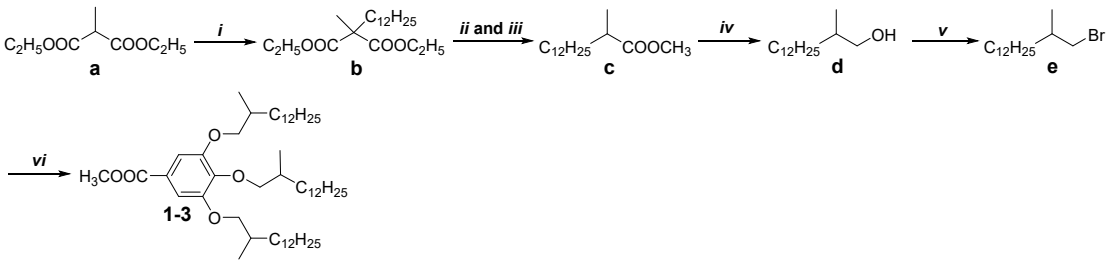


Fig. S9 UV-Vis absorption spectra **BNPI-3C12/DP** mixture in film.

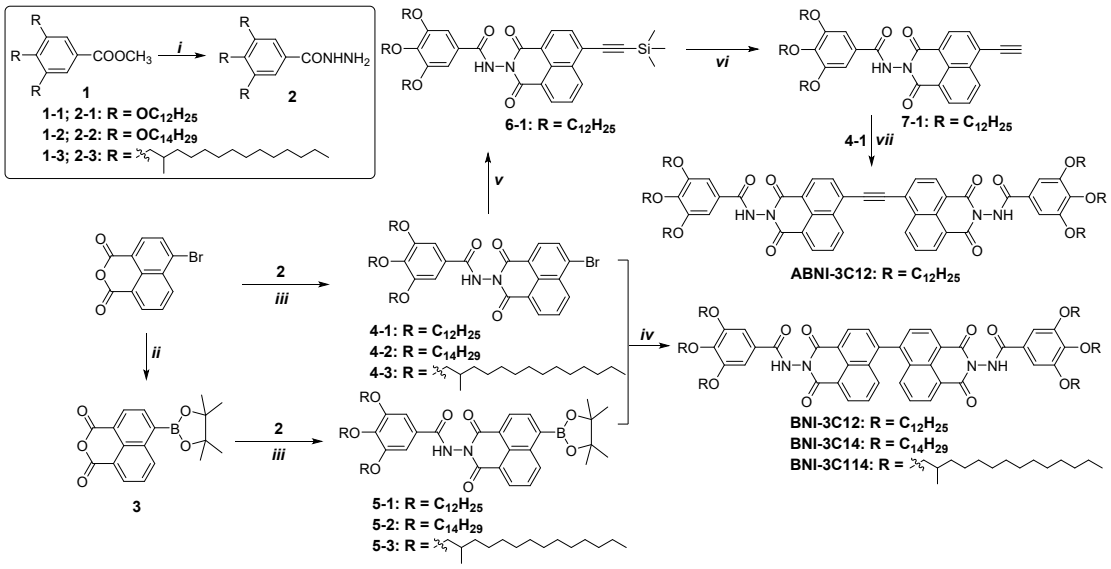
2. Synthesis and analytical data

2.1 General remarks

For the structures of the compounds were showed in Scheme 1 in the main text. Reactions requiring an inert gas atmosphere were conducted under nitrogen and the glassware was oven-dried (140 °C). Tetrahydrofuran (THF) was distilled from sodium prior to use. Commercially available chemicals were used as received. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker-DRX-400 and 100M. ESI-TOF-MS were performed on an Agilent 1100HPLC/TOF instrument. Thin-layer chromatography was performed on aluminum plates precoated with 5735 silica gel 60 PF254 (Merck). Column chromatography was performed on Merck silica gel 60 (230-400 mesh).



Scheme S1. Synthesis of the methyl 3,4,5-tris(dodecyloxy)benzoate with methyl branched chains (compound **1-3** in **Scheme S2**); Reagents and conditions: *i)* $C_{12}H_{25}Br$, NaH, THF, 0 to 75 °C; *ii*) KOH, EtOH, reflux; *iii*) a) N_2, 200 °C; b) *p*-toluenesulfonic acid, MeOH, reflux; *iv*) $LiAlH_4$, THF, 0 °C to RT; *v*) HBr, H_2SO_4(98%), tetrabutylammonium bromide, reflux; *vi*) methyl gallate, K_2CO_3, DMF, N_2, 90 °C.



Scheme S2. Synthesis of the 3,4,5-tri(alkoxy)benzoylhydrazine-functionalized binaphthalimide polycatenar compounds **BNI-3C*n*** and **ABNI-3C*n***; Reagents and conditions: (*i*) hydrazine hydrate, EtOH, reflux, 48 h; (*ii*) bis(pinacolato)diboron, $PdCl_2$(dppf), CH_3COOK, 1,4-dioxane, N_2, 90 °C, 24 h; (*iii*) glacial acetic acid, 130 °C, 48 h; (*iv*) $Pd(PPh_3)_4$, K_2CO_3, THF/H_2O, 70 °C, 24 h; (*v*) TMSA, $Pd(PPh_3)Cl_2$, CuI, diisopropylamine, THF, N_2, 70 °C, 12h; (*vi*) K_2CO_3, THF/MeOH, R.T, 2h; (*vii*) $Pd(PPh_3)Cl_2$, PPh_3, CuI, THF, diisopropylamine, N_2, 70 °C, 12h.

2.2 Synthesis and Characterization of Compounds

For the synthesis of compounds **1**, in which the straight chain analogues **1-1** and **1-2** could be prepared according to the previous literature,[S2] whereas the methyl branched chains analogue **1-3** is a new compound that can be prepared according the **Scheme S1**.

General procedures in scheme S1

Diethyl 2-dodecyl-2-methylmalonate (b)

At 0 °C, sodium hydride (NaH, 3.1 g, 129 mmol) was added to a solution of diethyl methylmalonate (**a**, 15g, 86.1 mmol) in dry THF (150 mL) in batches within 30 min, then this mixture was returned to room temperature and stirred for 4 h. 1-bromododecane ($C_{12}H_{25}Br$, 21.5g, 86.1 mmol) was dropped into the reaction solution within 30 min at room temperature followed by stirring at 75 °C for 12h. After the reaction was completed (monitored by TLC), the mixture was cooled to room temperature and a large number of solids were precipitated. The precipitate was directly filtered out and washed with THF. Then the collected filtrate was evaporated in vacuum to remove solvent. The residue was purified by a silica gel column chromatography (petroleum ether) to produce compound **b** as colorless liquid. Yield: 12g, 40 %. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 4.19-4.13 (q, J = 7.2 Hz, 4H, 2COOC**H$_2$**CH_3), 1.84-1.80 (m, 2H, $(CO)_2C(CH_3)$C**H$_2$**CH_2), 1.38 (s, 3H, $(CO)_2C$(C**H$_3$**) CH_2CH_2), 1.23 (s, 20H, 10C**H$_2$**), 1.25-1.21 (t, J = 6.8 Hz, 6H, 2$COOCH_2$C**H$_3$**), 0.88-0.85 (t, J = 7.2 Hz, 3H, C**H$_3$**).

Methyl 2-methyltetradecanoate (c)

A mixture of compound **b** (19.6 g, 57.2 mmol), KOH (32 g, 572 mmol) and EtOH (150 mL) was

stirred and refluxed at 80 ºC for 12h. After the reaction was completed (monitored by TLC), dilute hydrochloric acid (200 mL, 2M) was slowly poured into the reaction mixture under ice bath, then the mixture solution was stirred for 2 h and kept the pH = 1. The resulting solution was extracted with ethyl acetate (3 × 100 mL). The combined organic layer was washed by saturated salt water then dried over anhydrous $MgSO_4$, and the solvent was evaporated in vacuum to afford a white solid. The obtained white solid was further heated to 200 ºC without solvent under nitrogen atmosphere and the liquid was stirred for 12 h. After CO_2 bubbles were no longer visible, the system was cooled to room temperature, MeOH (100 mL) and *p*-toluenesulfonic acid (436 mg, 2.3 mmol) were added to the reaction system and the resulting solution was refluxed for 12 h. After the reaction was completed (monitored by TLC), the mixture was extracted with EA. The organic phase was separated, dried with anhydrous $MgSO_4$, then the mixture was filtered to remove $MgSO_4$, and the filtrate is concentrated on a rotary evaporator to remove the solvent. The residue was purified by a silica gel column chromatography (petroleum ether/CH_2Cl_2 = 4/1) to afford compound **c** as a pale yellow liquid. Yield: 12.5 g, 85 %. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 3.65 (s, 3H, COOC**H$_3$**), 2.45-2.39 (m, 1H, C**H**COOCH_3), 1.64-1.54 (m, 2H, C**H$_2$**CHCH_3), 1.24 (s, 20H, 10C**H$_2$**), 1.14-1.12 (d, J = 7.2 Hz, 3H, CH_2CHC**H$_3$**), 0.88-0.85 (t, J = 6.8 Hz, 3H, CH_2C**H$_3$**).

2-Methyltetradecan-1-ol (d)

To a stirred solution of compound **c** (12.5 g, 48.7 mmol) in dry THF (75 mL) at 0 ºC, lithium aluminium hydride ($LiAlH_4$, 2.8 g, 73.1 mmol) was added in batches. The reaction mixture was returned to room temperature and stirred for 4 h. After the reaction was completed (monitored by TLC), the mixture was cooled down to 0 ºC and the cold water was added very slowly to quench the reaction. Afterwards the solution was extracted with ethyl acetate. The organic phase was separated, dried with anhydrous $MgSO_4$, then the mixture was filtered to remove $MgSO_4$, and the filtrate is concentrated on a rotary evaporator to remove the solvent. The residue was purified by a silica gel column chromatography (petroleum ether/CH_2Cl_2 = 1/1) to afford compound **d** as a pale yellow liquid. Yield: 6.0 g, 54%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 3.52-3.48 (q, J = 5.6 Hz, 1H, C**H$_2$**OH), 3.42-3.38 (q, J = 6.8Hz, 1H, C**H$_2$**OH), 1.62-1.47 (m, 2H, CHC**H$_2$**CH_2), 1.41-1.37 (m, 2H, $CHCH_2$C**H$_2$**), 1.25 (s, 18H, 9C**H$_2$**), 1.09-1.03 (m, 1H, C**H**CH_2OH), 0.91-0.90 (d, J = 6.8 Hz, 3H, CH_2CHC**H$_3$**), 0.89-0.85 (t, J = 6.8 Hz, 3H, CH_2C**H$_3$**).

1-Bromo-2-methyltetradecane (e)

To a stirred solution of compound **d** (6.0 g, 26.3 mmol) and tetrabromomethane (CBr_4, 17.4g, 52.5 mmol) in dry CH_2Cl_2 (100 mL) at 0 ºC, triphenylphosphine (PPh_3, 13.8 g, 52.5 mmol) was added in batches within 1h, and the reaction mixture was returned to room temperature and stirred for 4 h. After the reaction was completed (monitored by TLC), the mixture was directly concentrated on a rotary evaporator. The hexane was added to the obtained residues to resolve the crude product while the insoluble substances were filtered. The collected organic solution was concentrated on a rotary evaporator again. The product was purified by a silica gel column chromatography (petroleum ether) to produce compound **e** as colorless liquid. Yield: 6.5 g, 85%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 3.41-3.38 (q, J = 4.8 Hz, 1H, C**H$_2$**Br), 3.34-3.30 (q, J = 6.4 Hz, 1H, C**H$_2$**Br), 1.82-1.74 (m, 1H, C**H**CH_2Br), 1.55-1.40 (m, 2H, C**H$_2$**$CH(CH_3)CH_2$Br), 1.26 (s, 20H, 10C**H$_2$**), 1.01-1.00 (d, J = 6.4 Hz, 3H, CH_2CH(**C**H_3)CH_2Br), 0.89-0.86 (t, J = 6.8 Hz, 3H, C**H$_3$**).

Methyl 3,4,5-tris(alkoxy)benzoate with methyl branched chains (1-3)

A mixture of methyl gallate (913 mg, 5.0 mmol), compound **e** (6.5 g, 22.3mmol) and K_2CO_3 (6.9 g, 50.0 mmol) in DMF was stirred at 90 ºC overnight under nitrogen atmosphere. After the reaction was completed (monitored by TLC), the reaction mixture was cooled to room temperature, then was extracted with CH_2Cl_2 (3 × 30 mL). The organic phase was separated, dried with anhydrous $MgSO_4$, filtered, and concentrated on a rotary evaporator. The residue was purified by a silica gel column chromatography (petroleum ether/CH_2Cl_2 = 3/1) to afford compound **1-3** as a colorless liquid. Yield: 3.0 g, 75%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 7.23 (s, 2H, 2Ar**H**), 3.89 (s, 3H, COOC**H$_3$**), 3.87-3.83 (m, 3H, 3OC**H**$_2$CH), 3.80-3.75 (m, 3H, 3OC**H**$_2$CH), 1.98-1.87 (m, 3H, 3OCH$_2$C**H**), 1.38-1.26 (m, 6H, 3C**H$_2$**CH(CH$_3$)CH$_2$O), 1.26 (s, 60H, 30C**H$_2$**), 1.04-1.03 (d, J = 5.6 Hz, 9H, 3CH$_2$CH(**CH$_3$**)CH$_2$O), 0.89-0.86 (t, J = 6.8 Hz, 9H, 3C**H$_3$**).

General procedures in scheme S2

General procedure for the synthesis of 3,4,5-tri(alkoxy)benzoylhydrazine (2)

Compounds **2** were synthesized according to literature procedure in ref S3. The ^{1}H NMR date of **2-1** and **2-2** were consistent with the literatures.

2-3. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 6.91 (s, 2H, 2Ar**H**), 4.08 (s, 2H, NHN**H$_2$**), 3.87-3.82 (m, 3H, 3OC**H**$_2$CH), 3.77-3.69 (m, 3H, 3OC**H**$_2$CH), 1.99-1.87 (m, 3H, 3OCH$_2$C**H**), 1.40-1.35 (m, 6H, 3C**H$_2$**CH(CH$_3$)CH$_2$O), 1.26 (s, 60H, 30C**H$_2$**), 1.05-1.04 (d, J = 5.2 Hz, 3H, CH$_2$CH(**CH$_3$**)CH$_2$O), 1.04-1.02 (d, J = 6.4 Hz, 6H, 2CH$_2$CH(**CH$_3$**)CH$_2$O), 0.89-0.86 (t, J = 6.8 Hz, 9H, 3C**H$_3$**).

4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-1,8-naphthalic anhydride (3)

A previously degassed 1,4-dioxane (60 mL), 4-bromo-1,8-naphthalic anhydride (2 g, 7.2 mmol), bis(pinacolato)diboron (3.7 g, 14.4 mmol), $PdCl_2$(dppf) (264 mg, 0.37 mmol) and CH_3COOK (2.13g, 21.7 mmol) were added to a flask, and the mixture was stirred at 100 ºC for 24 h under an argon atmosphere. After the reaction was complete (monitored by TLC), the mixture was cooled to room temperature and the dioxane was removed under vacuum, then the residue was extracted with CH_2Cl_2. The organic phase was separated, dried with anhydrous $MgSO_4$, then the mixture was filtered, and the filtrate is concentrated on a rotary evaporator to remove the solvent. The residue was purified by a silica gel column chromatography (pure CH_2Cl_2) to afford compound **3** as a white solid. Yield: 1.8 g, 77%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 9.24-9.21 (d, J = 9.6 Hz, 1H, Ar**H**), 8.63-8.61 (d, J = 8.4 Hz, 1H, Ar**H**), 8.59-8.57 (d, J = 7.2 Hz, 1H, Ar**H**), 8.36-8.34 (d, J = 7.6 Hz, 1H, Ar**H**), 7.86-7.82 (t, J = 8.4 Hz, 1H, Ar**H**), 1.46 (s, 12H, 4C**H**$_3$).

General procedure for the synthesis of the 3,4,5-tri(alkoxy)benzoylhydrazine-functionalized 4-bromo-naphthalimide (4)

The appropriate compounds **2** (0.36 mmol), 4-bromo-1,8-naphthalicanhydride (100 mg, 0.36 mmol) and glacial acetic acid (10 mL) were added into a flask and stirred at 130 ºC for 48 h. After reaction was complete (monitored by TLC), the reaction mixture was cooled to room temperature and water was added to get a large number of white precipitate, which was further filtered and washed by MeOH to afford relatively pure compounds **4** as white solid.

3,4,5-Tri(dodecyloxy)benzoylhydrazine-functionalized 4-bromo-naphthalimide (4-1): yield: 273 mg, 80%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 8.68-8.66 (d, J = 6.4 Hz, 1H, Ar**H**), 8.62-8.60

(d, J = 8.4 Hz, 1H, Ar**H**), 8.53 (s, 1H, N**H**), 8.43-8.41 (d, J = 7.6 Hz, 1H, Ar**H**), 8.06-8.05 (d, J = 7.6 Hz, 1H, Ar**H**), 7.88-7.84 (t, J = 7.6 Hz, 1H, Ar**H**), 7.18 (s, 2H, 2Ar**H**), 4.02-3.97 (m, 6H, 3OC$\mathbf{H_2}$), 1.83-1.72 (m, 6H, 3OCH$_2$C$\mathbf{H_2}$CH$_2$), 1.50-1.40 (m, 6H, 3OCH$_2$CH$_2$C$\mathbf{H_2}$), 1.25 (s, 48H, 24C$\mathbf{H_2}$), 0.89-0.86 (t, J = 6.8 Hz, 9H, 3C$\mathbf{H_3}$). ^{13}C NMR (100 MHz, CDCl$_3$), δ (ppm): 165.69, 161.95, 153.06, 141.79, 134.02, 132.86, 131.94, 131.28, 131.15, 130.56, 128.66, 128.09, 125.67, 122.43, 121.55, 106.03, 73.37, 69.07, 31.91, 30.36, 29.77-29.33 (multi carbons in alkyl chain), 26.07, 22.67, 14.09.

3,4,5-Tri(tetradecyloxy)benzoylhydrazine-functionalized 4-bromo-naphthalimide (4-2): yield: 300 mg, 81%. ^{1}H NMR (400 MHz, CDCl$_3$), δ (ppm): 8.69-8.67 (d, J = 7.2 Hz, 1H, Ar**H**), 8.63-8.61 (d, J = 8.4 Hz, 1H, Ar**H**), 8.62 (s, 1H, N**H**), 8.45-8.43 (d, J = 8.0 Hz, 1H, Ar**H**), 8.07-8.05 (d, J = 7.6 Hz, 1H, Ar**H**), 7.89-7.85 (t, J = 8.0 Hz, 1H, Ar**H**), 7.18 (s, 2H, 2Ar**H**), 4.02-3.93 (m, 6H, 3OC$\mathbf{H_2}$), 1.81-1.72 (m, 6H, 3OCH$_2$C$\mathbf{H_2}$CH$_2$), 1.49-1.40 (m, 6H, 3OCH$_2$CH$_2$C$\mathbf{H_2}$), 1.25 (s, 60H, 30C$\mathbf{H_2}$), 0.89-0.86 (t, J = 6.8 Hz, 9H, 3C$\mathbf{H_3}$). ^{13}C NMR (150 MHz, CDCl$_3$), δ (ppm): 165.76, 161.99, 153.06, 141.80, 134.06, 132.89, 131.97, 131.30, 131.18, 130.63, 128.78, 128.11, 125.69, 122.51, 121.62, 106.09, 73.39, 69.08, 31.91, 30.36, 29.72-29.33(multi carbons in alkyl chain), 26.08, 22.67, 14.09.

3,4,5-Tri(2-methyl-tetradecyloxy)benzoylhydrazine-functionalized 4-bromo-naphthalimide (4-3): yield: 317 mg, 82%. ^{1}H NMR (400 MHz, CDCl$_3$), δ (ppm): 8.67-8.65 (d, J = 7.2 Hz, 1H, Ar**H**), 8.61-8.59 (d, J = 8.4 Hz, 1H, Ar**H**), 8.56 (s, 1H, N**H**), 8.43-8.41 (d, J = 8.0 Hz, 1H, Ar**H**), 8.06-8.04 (d, J = 8.0 Hz, 1H, Ar**H**), 7.87-7.83 (t, J = 8.0 Hz, 1H, Ar**H**), 7.18 (s, 2H, 2Ar**H**), 3.88-3.82 (m, 3H, 3OC$\mathbf{H}_2$), 3.79-3.48 (m, 3H, 3OC$\mathbf{H}_2$), 1.98-1.88 (m, 3H, 3OCH$_2$C**H**), 1.54 (s, 6H, 3CH$_2$CHC$\mathbf{H_2}$), 1.26 (s, 60H, 30C$\mathbf{H_2}$), 1.07-1.05 (d, J = 6.4 Hz, 3H, OCH$_2$CHC$\mathbf{H_3}$), 1.02-1.01 (d, J = 6.4 Hz, 6H, 2OCH$_2$CHC$\mathbf{H_3}$), 0.87-0.86 (t, J = 6.8 Hz, 9H, 3C$\mathbf{H_3}$). ^{13}C NMR (100 MHz, CDCl$_3$), δ (ppm): 165.89, 161.93, 153.24, 141.84, 134.08, 132.91, 131.99, 131.31, 131.20, 130.62, 128.77, 128.13, 125.66, 122.48, 121.61, 105.80, 78.48, 74.10, 34.29, 33.45, 33.29, 31.92, 30.00, 29.79-29.37(multi carbons in alkyl chain), 27.18, 26.98, 22.68, 17.00, 14.10.

General procedure for the synthesis of the 3,4,5-tri(alkoxy)benzoylhydrazine-functionalized 4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)naphthalimide (5)

Compounds **5** were prepared by the similar methods to compounds **4.** Reactants: compounds **2** (0.31 mmol), compounds **3** (100 mg, 0.31 mmol) and glacial acetic acid (10 mL)

3,4,5-Tri(dodecyloxy)benzoylhydrazine-functionalized 4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)naphthalimide (5-1): yield: 200 mg, 65%. ^{1}H NMR (600 MHz, CDCl$_3$), δ (ppm): 9.14-9.13 (d, J = 8.4 Hz, 1H, Ar**H**), 8.74 (s, 1H, N**H**), 8.59-8.58 (d, J = 7.2 Hz, 1H, Ar**H**), 8.57-8.56 (d, J = 6.6 Hz, 1H, Ar**H**), 8.29-8.28 (d, J = 7.2 Hz, 1H, Ar**H**), 7.77-7.75 (t, J = 7.2 Hz, 1H, Ar**H**), 7.20 (s, 2H, 2Ar**H**), 4.01-3.97 (m, 6H, 3OC$\mathbf{H_2}$), 1.81-1.72 (m, 6H, 3OCH$_2$C$\mathbf{H_2}$CH$_2$), 1.49-1.40 (m, 6H, 3OCH$_2$CH$_2$C$\mathbf{H_2}$), 1.45 (s, 12H, 4CC$\mathbf{H_3}$), 1.25 (s, 48H, 24C$\mathbf{H_2}$), 0.88-0.86 (t, J = 7.2 Hz, 9H, 3C$\mathbf{H_3}$). ^{13}C NMR (150 MHz, CDCl$_3$), δ (ppm): 165.97, 162.56, 153.11, 141.81, 135.83, 135.73, 135.33, 131.78, 130.65, 127.78, 127.11, 126.06, 124.14, 122.02, 106.17, 84.68, 73.44, 69.16, 31.92, 30.34, 29.76-29.33(multi carbons in alkyl chain), 26.06, 24.95, 22.67, 14.10.

3,4,5-Tri(tetradecyloxy)benzoylhydrazine-functionalized 4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)naphthalimide (5-2): yield: 250 mg, 75%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 9.14 (s, 1H, N**H**), 9.11-9.09 (d, J = 8.4 Hz, 1H, Ar**H**), 8.55-8.53 (d, J = 7.6 Hz, 1H, Ar**H**), 8.53-8.52 (d, J = 7.2 Hz, 1H, Ar**H**), 8.26-8.25 (d, J = 7.2 Hz, 1H, Ar**H**), 7.75-7.71 (t, J = 7.6 Hz, 1H, Ar**H**), 7.21 (s, 2H, 2Ar**H**), 4.00-3.93 (m, 6H, 3OC**H$_2$**), 1.79-1.70 (m, 6H, 3OCH$_2$C**H$_2$**CH$_2$), 1.45 (s, 12H, 4CC**H$_3$**), 1.42-1.37 (m, 6H, 3OCH$_2$CH$_2$C**H$_2$**), 1.25 (s, 60H, 30C**H$_2$**), 0.89-0.85 (t, J = 6.8 Hz, 9H, 3C**H$_3$**). ^{13}C NMR (100 MHz, $CDCl_3$), δ (ppm): 165.85, 162.63, 153.03, 141.66, 135.77, 135.68, 135.24, 131.73, 130.59, 127.70, 127.05, 125.96, 124.08, 121.96, 106.09, 84.64, 73.37, 69.06, 31.90, 30.35, 29.77-29.32 (multi carbons in alkyl chain), 26.06, 24.92, 22.66, 14.08.

3,4,5-Tri(2-methyl-tetradecyloxy)benzoylhydrazine-functionalized 4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)naphthalimide (5-3): yield: 232 mg, 67%. ^{1}H NMR (600 MHz, $CDCl_3$), δ (ppm): 9.15-9.14 (d, J = 9.0 Hz, 1H, Ar**H**), 8.69 (s, 1H, N**H**), 8.60-8.59 (d, J = 7.2 Hz, 1H, Ar**H**), 8.58-8.57 (d, J = 7.2 Hz, 1H, Ar**H**), 8.30-8.28 (d, J = 7.2 Hz, 1H, Ar**H**), 7.78-7.76 (t, J = 7.8 Hz, 1H, Ar**H**), 7.20 (s, 2H, 2Ar**H**), 3.88-3.83 (m, 3H, 3OC**H$_2$**), 3.79-3.75 (m, 3H, 3OC**H$_2$**), 1.96-1.90 (m, 3H, 3OCH$_2$C**H**), 1.58-1.49 (m, 6H, 3CH$_2$CHC**H$_2$**), 1.46 (s, 12H, 4CC**H$_3$**), 1.25 (s, 60H, 30C**H$_2$**), 1.07-1.06 (d, J = 6.6 Hz, 3H, OCH$_2$CHC**H$_3$**), 1.02-1.01 (d, J = 6.6 Hz, 6H, 2OCH$_2$CHC**H$_3$**), 0.88-0.86 (t, J = 6.6 Hz, 9H, 3C**H$_3$**). ^{13}C NMR (150 MHz, $CDCl_3$), δ (ppm): 166.07, 162.57, 153.25, 141.80, 135.82, 135.74, 135.35, 131.78, 130.64, 127.79, 127.11, 126.02, 124.17, 122.05, 105.88, 84.68, 78.52, 74.16, 34.29, 33.52, 33.47, 33.31, 31.92, 30.08-29.36 (multi carbons in alkyl chain), 27.18, 26.98, 24.95, 22.68, 17.04, 14.10.

3,4,5-Tri(dodecyloxy)benzoylhydrazine-functionalized and TMS substituted naphthalimide derivative (6-1)

Compound **4-1** (300 mg, 0.32 mmol) and trimethylsilylacetylene (TMSA, 47 mg, 0.47 mmol) was dissolved in dry THF (10 mL) and dry diisopropylamine (5 mL) under a nitrogen atmosphere. Then $Pd(PPh_3)Cl_2$ (12mg, 0.016 mmol), CuI (3 mg, 0.016 mmol) was added, and the mixture was stirred at 40 ºC overnight. After the reaction was complete (monitored by TLC), dichloromethane (20 mL) was added, and the mixture was washed with brine (3×20 mL). Then the organic phase was dried over anhydrous Na_2SO_4 and the solvent was removed in vacuo. The residue was purified by neutral aluminum (III) oxide (200-300 mesh) filled column chromatography (CH_2Cl_2) to afford **6-1** as brown liquid. Yield: 265 mg, 87%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 8.69-8.67 (d, J = 8.4 Hz, 1H, Ar**H**), 8.66-8.65 (d, J = 8.0 Hz, 1H, Ar**H**), 8.56-8.54 (d, J = 7.6 Hz, 1H, Ar**H**), 8.47 (s, 1H, N**H**), 7.91-7.89 (d, J = 7.6 Hz, 1H, Ar**H**), 7.86-7.82 (t, J = 8.0 Hz, 1H, Ar**H**), 7.19 (s, 2H, 2Ar**H**), 4.02-3.98 (m, 6H, 3OC**H$_2$**), 1.83-1.73 (m, 6H, 3OCH$_2$C**H$_2$**CH$_2$), 1.48-1.41 (m, 6H, 3OCH$_2$CH$_2$C**H$_2$**), 1.26 (s, 48H, 24C**H$_2$**), 0.89-0.86 (t, J = 6.8 Hz, 9H, 3C**H$_3$**), 0.37 (s, 9H, Si(C**H$_3$**)$_3$).

3,4,5-Tri(dodecyloxy)benzoylhydrazine-functionalized and ethynyl group substituted naphthalimide derivative (7-1)

Compound **6-1** (215 mg, 0.22 mmol) and K_2CO_3 (215 mg, 1.6 mmol) was added into the solvents of THF-MeOH (1:1) and stirred at room temperature for 2h. After the reaction was complete (monitored by TLC), the mixture was extracted with CH_2Cl_2, the organic phase was separated, dried with anhydrous $MgSO_4$, filtered, and concentrated on a rotary evaporator. The residue was purified by neutral aluminum (III) oxide (200-300 mesh) filled column chromatography (CH_2Cl_2) to afford

7-1 as yellow solid. Yield: 169 mg, 85%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 8.72-8.70 (d, J = 8.4 Hz, 1H, Ar**H**), 8.67-8.65 (d, J = 7.6 Hz, 1H, Ar**H**), 8.57-8.55 (d, J = 7.6 Hz, 1H, Ar**H**), 8.50 (s, 1H, N**H**), 7.96-7.94 (d, J = 7.6 Hz, 1H, Ar**H**), 7.86-7.83 (t, J = 8.4 Hz, 1H, Ar**H**), 7.19 (s, 2H, 2Ar**H**), 4.02-3.98 (m, 6H, 3OC$\mathbf{H_2}$), 3.79 (s, 1H, C≡C**H**), 1.81-1.71 (m, 6H, 3OCH$_2$C$\mathbf{H_2}$CH$_2$), 1.48-1.41 (m, 6H, 3OCH$_2$CH$_2$C$\mathbf{H_2}$), 1.26 (s, 48H, 24C$\mathbf{H_2}$), 0.89-0.86 (t, J = 6.8 Hz, 9H, 3C$\mathbf{H_3}$). ^{13}C NMR (100 MHz, $CDCl_3$), δ (ppm): 165.86, 162.25, 161.97, 153.21, 141.75, 133.00, 132.55, 131.93, 131.58, 131.01, 127.74, 127.70, 127.12, 125.69, 122.33, 122.11, 105.78, 87.26, 80.01, 73.32, 69.02, 31.92, 30.09, 30.01, 29.80-29.37(multi carbons in alkyl chain), 26.03, 22.68, 14.11.

3,4,5-Tri(dodecyloxy)benzoylhydrazine-functionalized and alkyne-bridged binaphthalimide (ABNI-3C12)

Compound **7-1** (160 mg, 0.18 mmol) and compound **4-1** (169 mg, 0.18 mmol) were dissolved in dry THF (10 mL) and dry diisopropylamine (5 mL) under a nitrogen atmosphere. Then Pd(PPh$_3$)Cl$_2$ (13 mg, 0.018 mmol), CuI (4 mg, 0.018 mmol) and PPh$_3$ (5mg, 0.018 mmol) were added, and the mixture was stirred at 70 ºC overnight. After the reaction was complete (monitored by TLC), the solvents were directly removed in vacuo. The residues was purified by neutral aluminum (III) oxide (200-300 mesh) filled column chromatography (CH_2Cl_2) to afford **ABNI-3C12** as yellow solid. Yield: 254 mg, 81%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 8.78 (s, 1H, N**H**), 8.72 (s, 1H, N**H**), 8.69-8.67 (d, J = 8.0 Hz, 1H, Ar**H**), 8.61-8.58 (d, J = 10.8 Hz, 2H, Ar**H**), 8.57-8.55 (d, J = 7.6 Hz, 1H, Ar**H**), 8.46-8.44 (d, J = 7.2 Hz, 1H, Ar**H**), 8.41-8.39 (d, J = 7.2 Hz, 1H, Ar**H**), 7.99-7.97 (d, J = 7.6 Hz, 2H, Ar**H**), 7.88-7.85 (t, J = 7.6 Hz, 1H, Ar**H**), 7.78-7.75 (t, J = 7.6 Hz, 1H, Ar**H**), 7.24 (s, 2H, 2Ar**H**), 7.21 (s, 2H, 2Ar**H**), 4.03-3.94 (m, 12H, 6OC$\mathbf{H_2}$), 1.77-1.69 (m, 12H, 6OCH$_2$C$\mathbf{H_2}$CH$_2$), 1.49-1.42 (m, 12H, 6OCH$_2$CH$_2$C$\mathbf{H_2}$), 1.25 (s, 96H, 48C$\mathbf{H_2}$), 0.88-0.85 (t, J = 7.2 Hz, 18H, 6C$\mathbf{H_3}$). ^{13}C NMR (150 MHz, $CDCl_3$), δ (ppm): 165.91, 165.78, 162.03, 161.84, 161.49, 161.38, 153.12, 141.86, 132.41, 132.19, 131.56, 131.43, 131.12, 131.03, 130.65, 127.96, 127.59, 127.50, 125.67, 122.69, 122.53, 122.37, 106.13, 95.25, 73.41, 69.06, 31.88, 30.38, 29.76-29.34(multi carbons in alkyl chain), 26.08, 22.64, 14.06. HRMS (ESI-TOF): m/z calcd for $C_{112}H_{166}N_4O_{12}$ $[M+Na]^+$ 1783.2428; found 1783.2385.

3,4,5-Tri(dodecyloxy)benzoylhydrazine-functionalized and single bond bridged binaphthalimide (BNI-3C12)

A mixture of the compound **4-1** (381 mg, 0.40 mmol), compound **5-1** (400 mg, 0.40mmol), K_2CO_3 (555 mg, 4.0 mmol), Pd(PPh$_3$)$_4$ (19 mg, 0.016mmol), THF (15 mL) and H_2O (5 mL) was refluxed at 85 °C for 48 h under an argon atmosphere. After the reaction was complete (monitored by TLC), the mixture was cooled to room temperature and extracted with CH_2Cl_2. The organic phase was separated, dried with anhydrous $MgSO_4$, filtered, and concentrated in vacuo. The residues was purified by neutral aluminum (III) oxide (200-300 mesh) chromatography (CH_2Cl_2) to afford **BNI-3C12** as pale yellow solid. Yield: 209 mg, 30%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 8.83-8.82 (d, J = 7.6 Hz, 1H, Ar**H**), 8.82-8.80 (d, J = 6.8 Hz, 1H, Ar**H**), 8.73-8.71 (d, J = 6.8 Hz, 1H, Ar**H**), 8.71-8.69 (d, J = 6.4 Hz, 1H, Ar**H**), 8.47 (s, 2H, 2N**H**), 7.86-7.85 (d, J = 6.0 Hz, 1H, Ar**H**), 7.85-7.83 (d, J = 6.8 Hz, 1H, Ar**H**), 7.77-7.75 (d, J = 8.4 Hz, 1H, Ar**H**), 7.75-7.73 (d, J = 7.2 Hz, 1H, Ar**H**), 7.69-7.65 (t, J = 8.0 Hz, 1H, Ar**H**), 7.67-7.63 (t, J = 8.0 Hz, 1H, Ar**H**), 7.22 (s, 4H, 4Ar**H**), 4.05-4.02 (t, J = 6.0 Hz, 12H, 6OC$\mathbf{H_2}$), 1.86-1.72 (m, 12H, 6OCH$_2$C$\mathbf{H_2}$CH$_2$), 1.49-1.44 (m, 12H, 6OCH$_2$CH$_2$C$\mathbf{H_2}$), 1.26 (s, 96H, 48C$\mathbf{H_2}$), 0.88-0.86 (t, J = 6.8 Hz, 18H, 6C$\mathbf{H_3}$). ^{13}C NMR (150 MHz, $CDCl_3$), δ (ppm): 166.06, 165.94, 162.60, 162.31, 162.12, 153.09, 143.49, 141.83, 132.92, 132.72,

132.55, 132.36, 131.42, 131.32, 130.97, 130.91, 129.11, 129.04, 128.81, 128.71, 128.39, 127.78, 127.50, 125.88, 125.83, 122.86, 122.74, 122.67, 106.19, 73.43, 69.14, 31.89, 30.35, 29.75-29.34(multi carbons in alkyl chain), 26.09, 22.65, 14.08. HRMS (ESI-TOF): m/z calcd for $C_{110}H_{166}N_4O_{12}$ $[M+Na]^+$ 1759.2428; found 1759.2445.

3,4,5-Tri(tetradecyloxy)benzoylhydrazine-functionalized and single bond bridged binaphthalimide (BNI-3C14)

A mixture of the compound **4-2** (191 mg, 0.19 mmol), compound **5-2** (200 mg, 0.19mmol), K_2CO_3 (256 mg, 1.9 mmol), $Pd(PPh_3)_4$ (9 mg, 0.0074mmol), THF (15 mL) and H_2O (5 mL) was refluxed at 85 °C for 48 h under an argon atmosphere. After the reaction was complete (monitored by TLC), the mixture was cooled to room temperature and extracted with CH_2Cl_2. The organic phase was separated, dried with anhydrous $MgSO_4$, filtered, and concentrated in vacuo. The residues was purified by neutral aluminum (III) oxide(200-300 mesh) filled column chromatography (CH_2Cl_2) to afford **BNI-3C14** as pale yellow solid. Yield: 100 mg, 28%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 8.83-8.82 (d, J = 7.2 Hz, 1H, Ar**H**), 8.82-8.80 (d, J = 6.8 Hz, 1H, Ar**H**), 8.73-8.71 (d, J = 6.8 Hz, 1H, Ar**H**), 8.71-8.69 (d, J = 7.2 Hz, 1H, Ar**H**), 8.47 (s, 2H, 2N**H**), 7.86-7.85 (d, J = 6.0 Hz, 1H, Ar**H**), 7.85-7.83 (d, J = 7.2 Hz, 1H, Ar**H**), 7.77-7.75 (d, J = 7.2 Hz, 1H, Ar**H**), 7.75-7.73 (d, J = 7.6 Hz, 1H, Ar**H**), 7.69-7.65 (t, J = 8.0 Hz, 1H, Ar**H**), 7.67-7.63 (t, J = 8.0 Hz, 1H, Ar**H**), 7.22 (s, 4H, 4Ar**H**), 4.05-4.02 (t, J = 6.0 Hz, 12H, 6OC$\mathbf{H_2}$), 1.86-1.73 (m, 12H, 6OCH$_2$C$\mathbf{H_2}$CH$_2$), 1.49-1.44 (m, 12H, 6OCH$_2$CH$_2$C$\mathbf{H_2}$), 1.27 (s, 120H, 60C$\mathbf{H_2}$), 0.89-0.86 (t, J = 6.4 Hz, 18H, 6C$\mathbf{H_3}$). ^{13}C NMR (150 MHz, $CDCl_3$), δ (ppm): 166.14, 166.04, 162.29, 162.10, 153.16, 143.45, 141.94, 132.95, 132.76, 132.58, 132.42, 131.45, 131.36, 131.00, 130.94, 129.11, 129.06, 128.81, 128.73, 128.41, 127.81, 127.55, 125.93, 122.88, 122.75, 122.68, 106.23, 73.47, 69.21, 31.92, 30.36, 29.76-29.36(multi carbons in alkyl chain), 26.10, 22.67, 14.10. HRMS (ESI-TOF): m/z calcd for $C_{122}H_{190}N_4O_{12}$ $[M+Na]^+$ 1927.4306; found 1927.4273.

3,4,5-Tri(2-methyl-tetradecyloxy)benzoylhydrazine-functionalized and single bond bridged binaphthalimide (BNI-3C114)

A mixture of the compound **4-3** (479 mg, 0.45 mmol), compound **5-3** (500 mg, 0.19mmol), K_2CO_3 (616 mg, 4.5 mmol), $Pd(PPh_3)_4$ (21 mg, 0.018mmol), THF (15 mL) and H_2O (5 mL) was refluxed at 85 °C for 48 h under an argon atmosphere. After the reaction was complete (monitored by TLC), the mixture was cooled to room temperature and extracted with CH_2Cl_2. The organic phase was separated, dried with anhydrous $MgSO_4$, filtered, and concentrated in vacuo. The residues was purified by neutral aluminum (III) oxide (200-300 mesh) filled column chromatography (CH_2Cl_2) to afford **BNI-3C114** as pale yellow solid. Yield: 302 mg, 34%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 8.83-8.82 (d, J = 6.0 Hz, 1H, Ar**H**), 8.82-8.80 (d, J = 6.8 Hz, 1H, Ar**H**), 8.71-8.70 (d, J = 6.4 Hz, 2H, 2Ar**H**), 8.55 (s, 2H, 2N**H**), 7.86-7.85 (d, J = 6.4 Hz, 1H, Ar**H**), 7.85-7.83 (d, J = 7.2 Hz, 1H, Ar**H**), 7.75-7.73 (d, J = 7.6 Hz, 2H, 2Ar**H**), 7.70-7.66 (t, J = 6.0 Hz, 1H, Ar**H**), 7.66-7.62 (t, J = 7.6 Hz, 1H, Ar**H**), 7.22 (s, 4H, 4Ar**H**), 3.88-3.87 (m, 3H, 3OC$\mathbf{H_2}$), 3.82-3.79 (m, 3H, 3OC$\mathbf{H_2}$), 2.00-1.93 (m, 6H, 6OCH$_2$C**H**CH$_2$), 1.58-1.52 (m, 6H, 6OCH$_2$CHC$\mathbf{H_2}$CH$_2$), 1.39-1.37 (m, 6H, 6OCH$_2$CHC$\mathbf{H_2}$CH$_2$), 1.27 (s, 120H, 60C$\mathbf{H_2}$), 1.08-1.06 (d, J = 7.2 Hz, 6H, 2OCH$_2$CHC$\mathbf{H_3}$), 1.05-1.04 (d, J = 6.8 Hz, 12H, 4OCH$_2$CHC$\mathbf{H_3}$), 0.89-0.85 (t, J = 6.4 Hz, 18H, 6C$\mathbf{H_3}$). ^{13}C NMR (150 MHz, $CDCl_3$), δ (ppm): 166.21, 166.10, 162.30, 162.12, 153.30, 143.51, 141.96, 132.92, 132.71, 132.62, 132.47, 131.48, 131.36, 131.01, 130.95, 129.09, 129.03, 128.80, 128.72, 128.44, 127.83,

127.59, 125.83, 125.77, 122.92, 122.79, 122.70, 105.93, 78.56, 74.19, 34.30, 33.48, 33.33, 31.91, 30.08, 30.01, 29.78-29.35(multi carbons in alkyl chain), 27.18, 26.98, 22.67, 17.06, 14.10. HRMS (ESI-TOF): m/z calcd for $C_{128}H_{202}N_4O_{12}$ $[M+Na]^+$ 2011.5245; found 2011.5213.

2.3 ^{1}H and ^{13}C NMR spectra for representative compounds

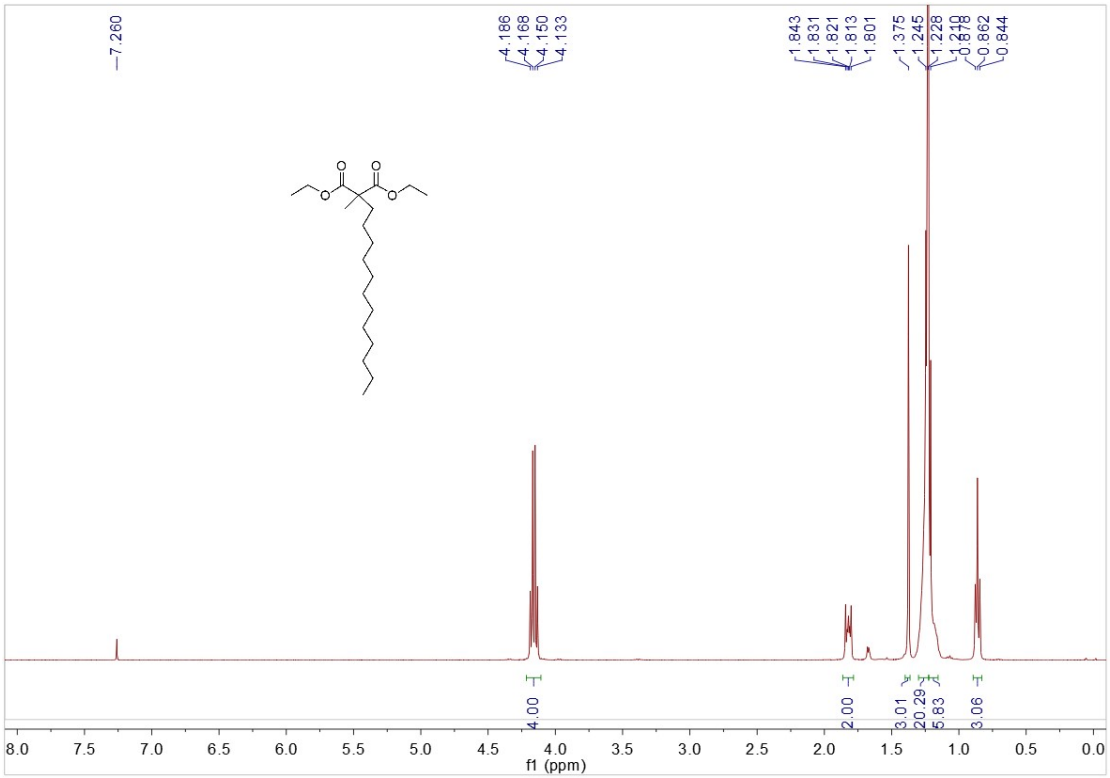


Fig. S10 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **b**.

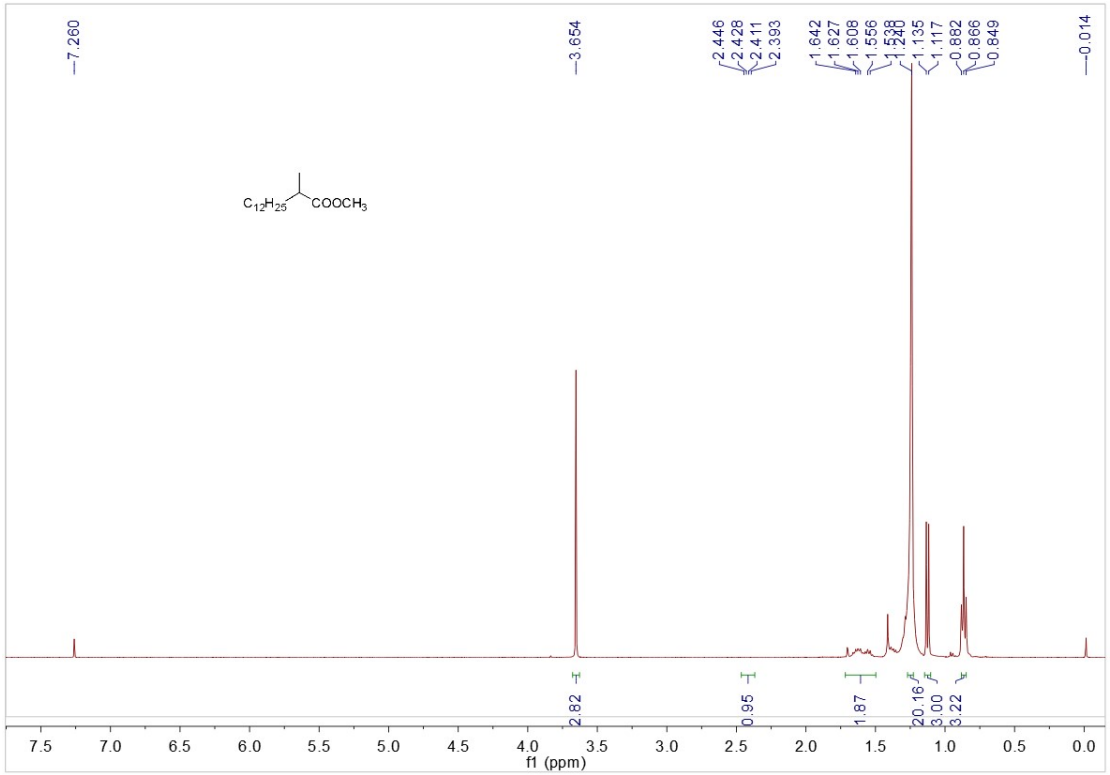


Fig. S11 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **c**.

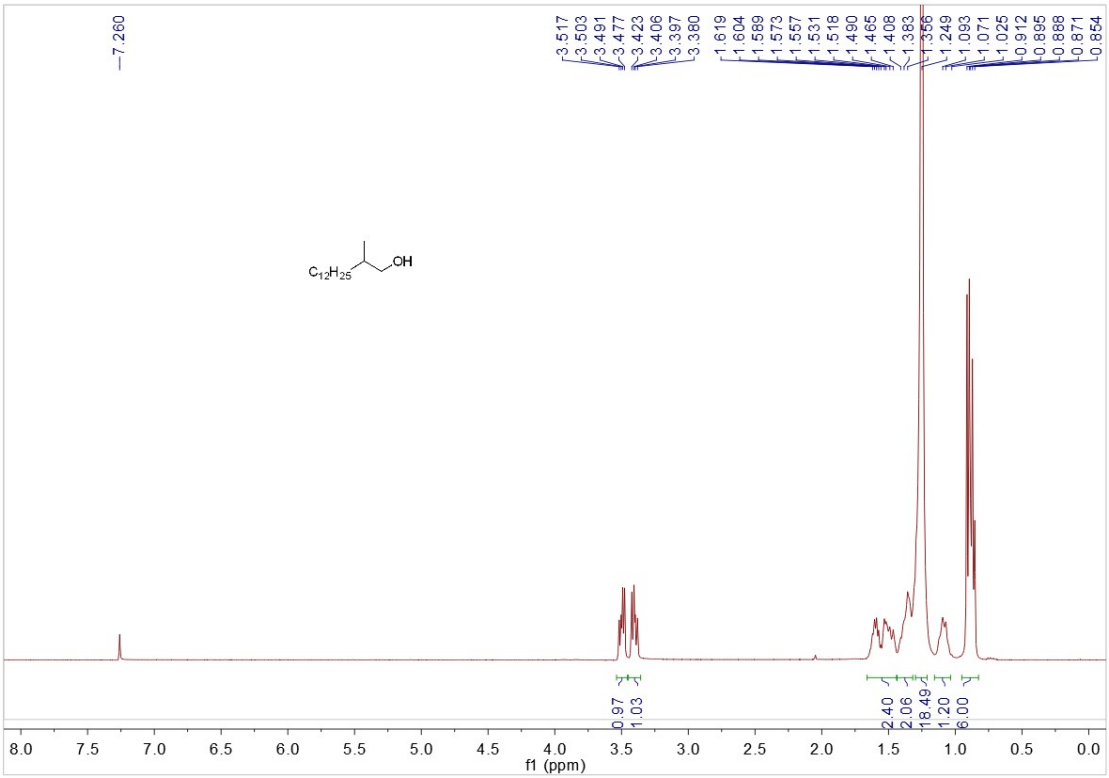


Fig. S12 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **d**.

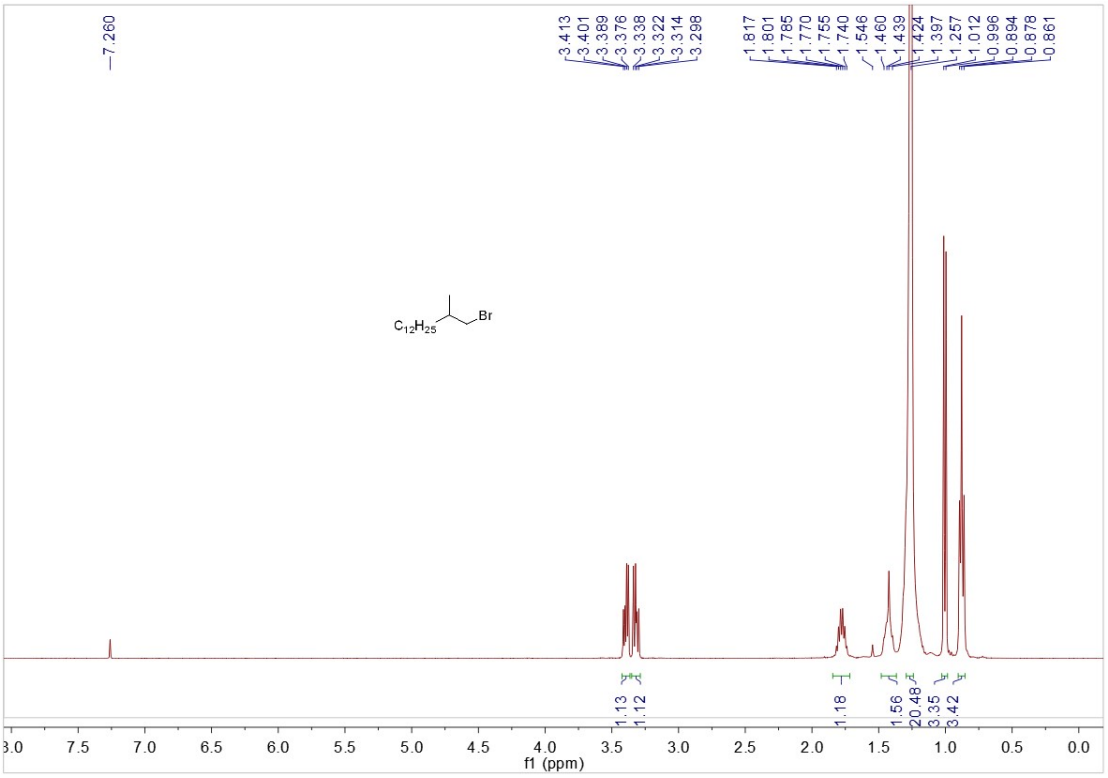


Fig. S13 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **e**.

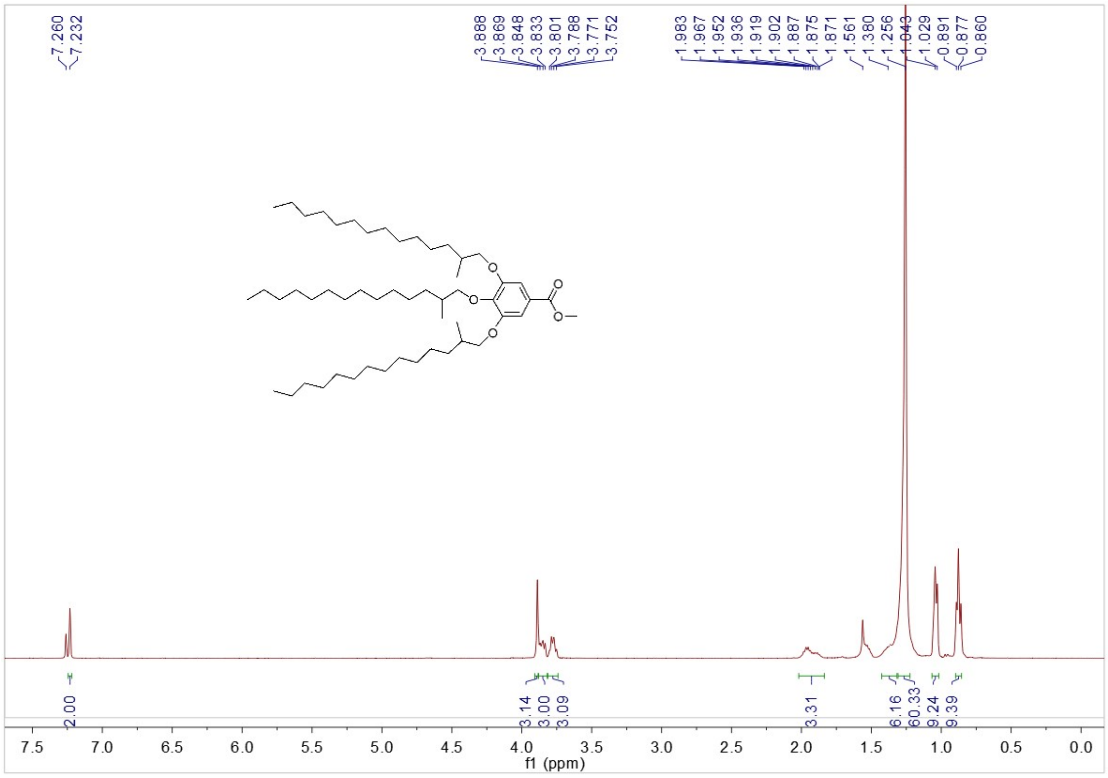


Fig. S14 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **1-3**.

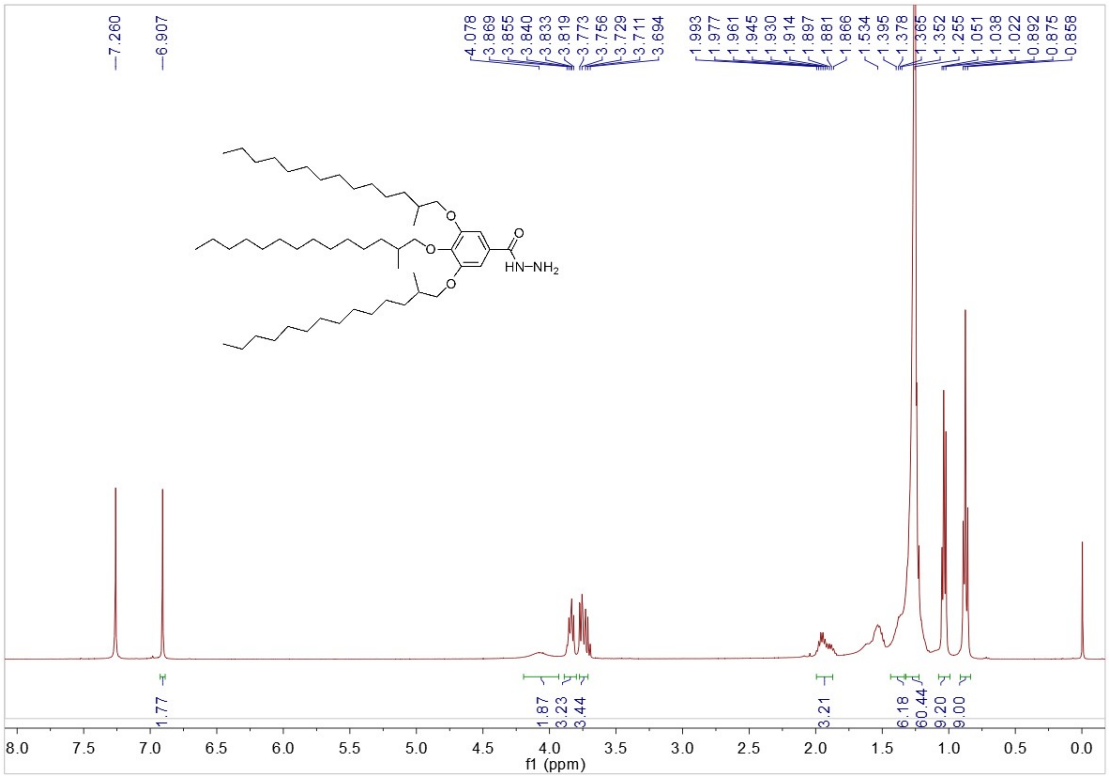


Fig. S15 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **2-3**.

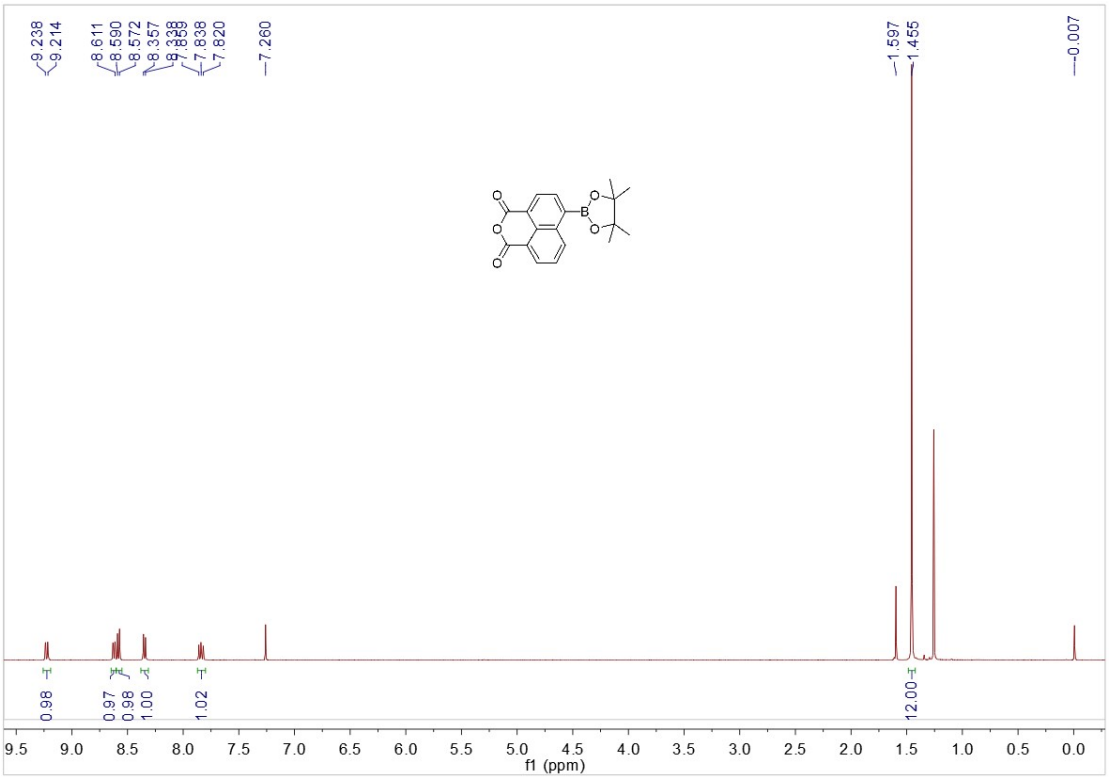


Fig. S16 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **3**.

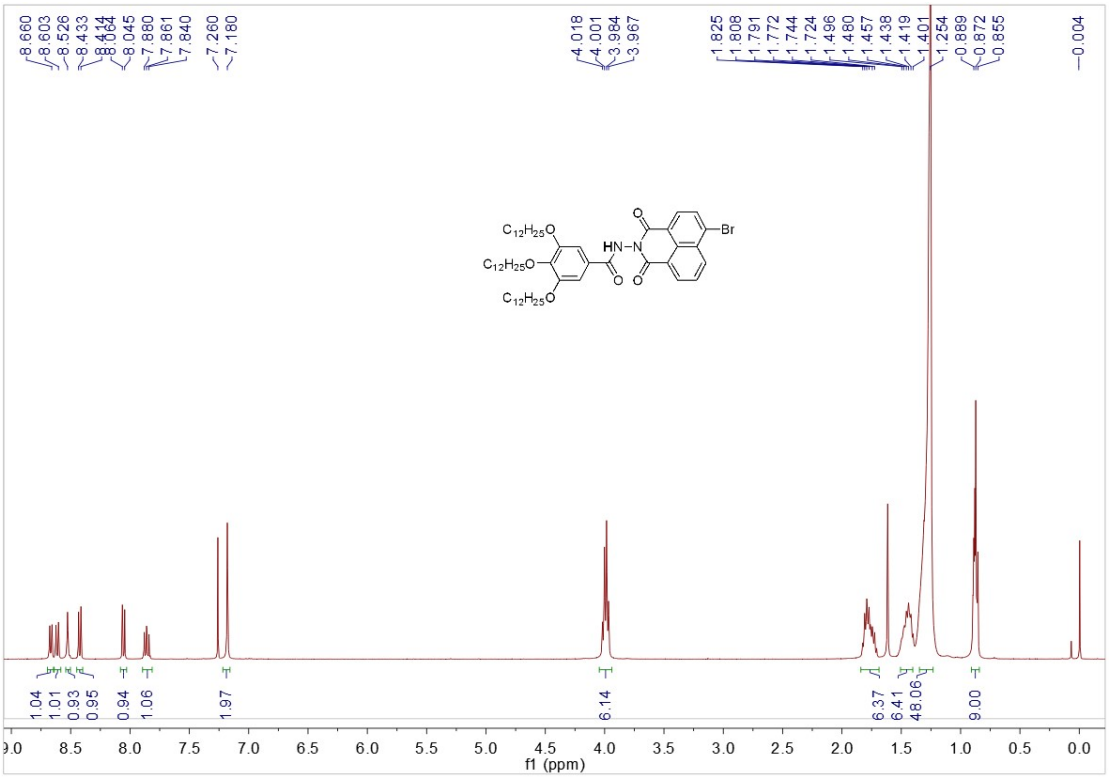


Fig. S17 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **4-1**.

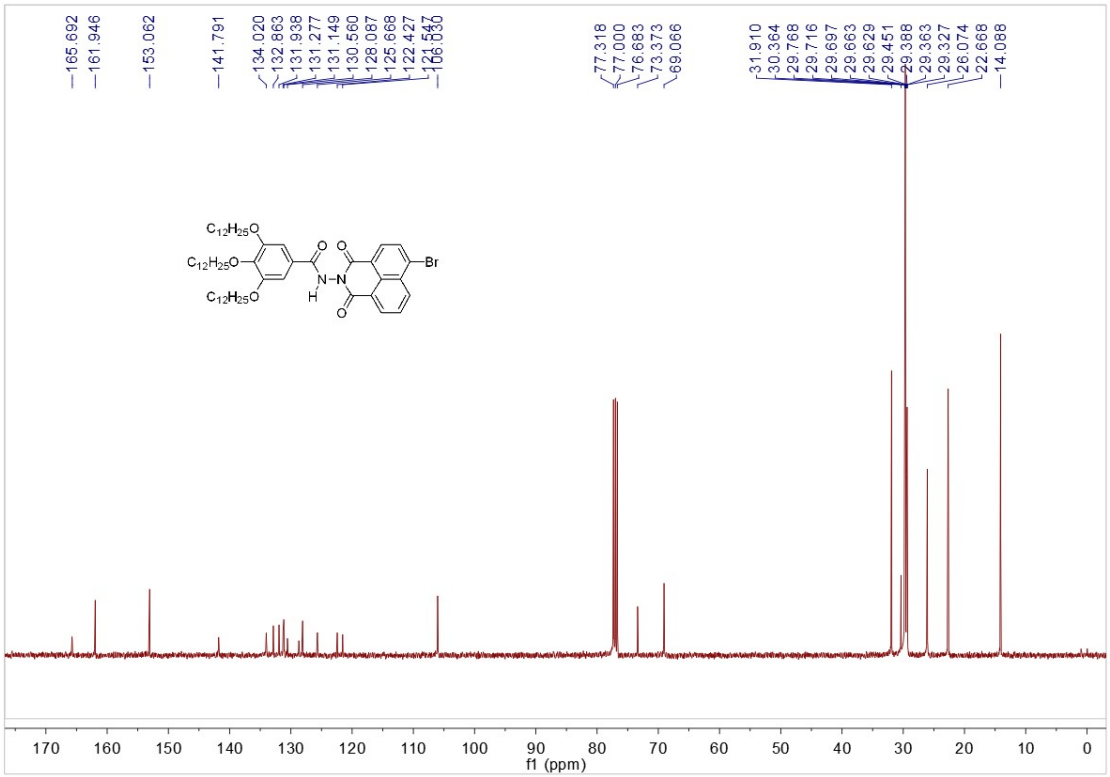


Fig. S18 ^{13}H NMR ($CDCl_3$, 100 MHz, ppm) spectra of compound **4-1**.

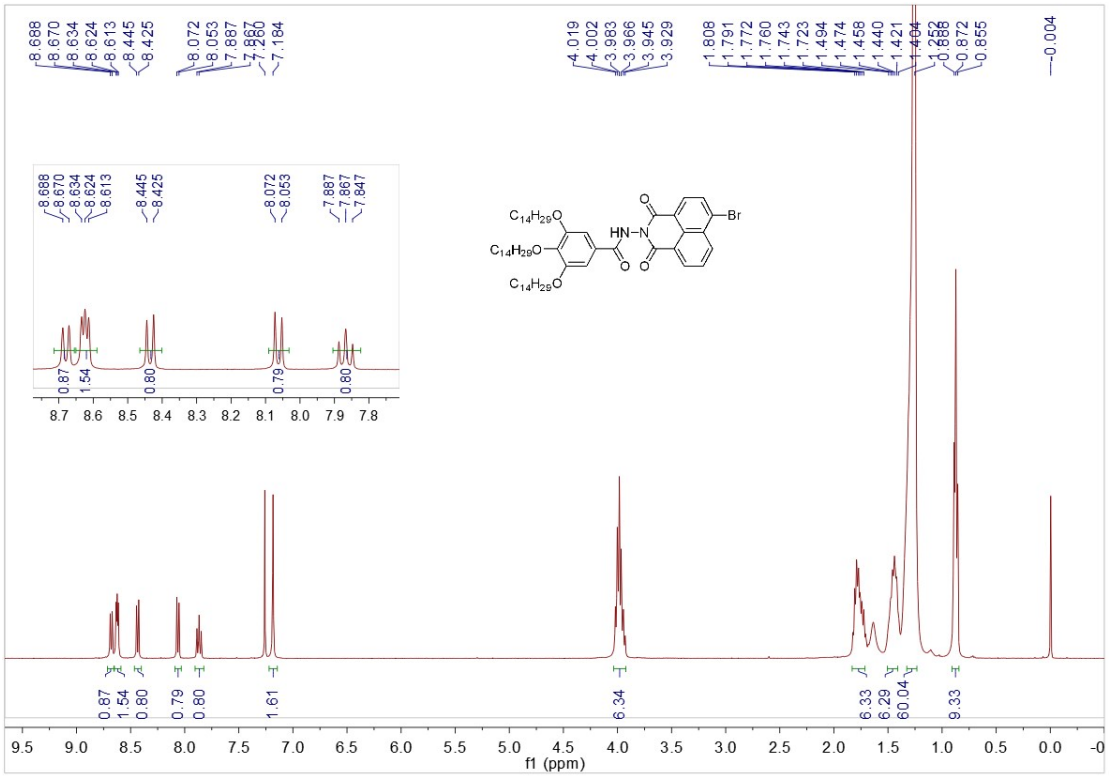


Fig. S19 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **4-2**.

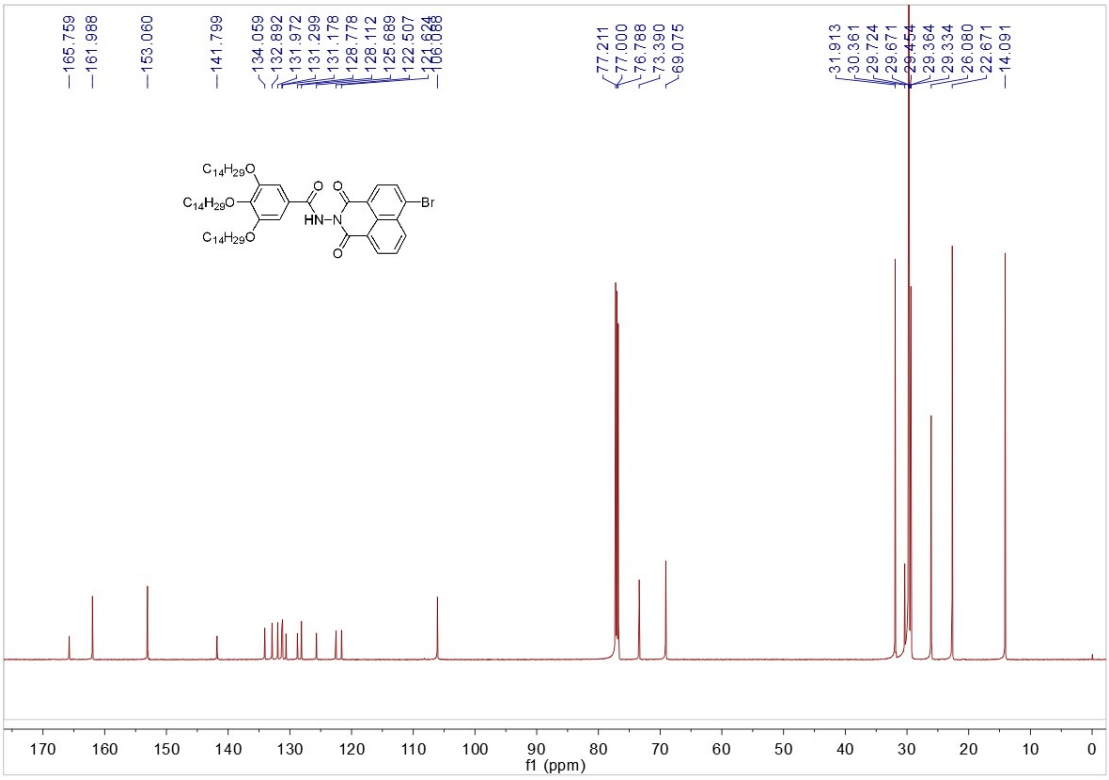


Fig. S20 ^{13}C NMR ($CDCl_3$, 150 MHz, ppm) spectra of compound **4-2**.

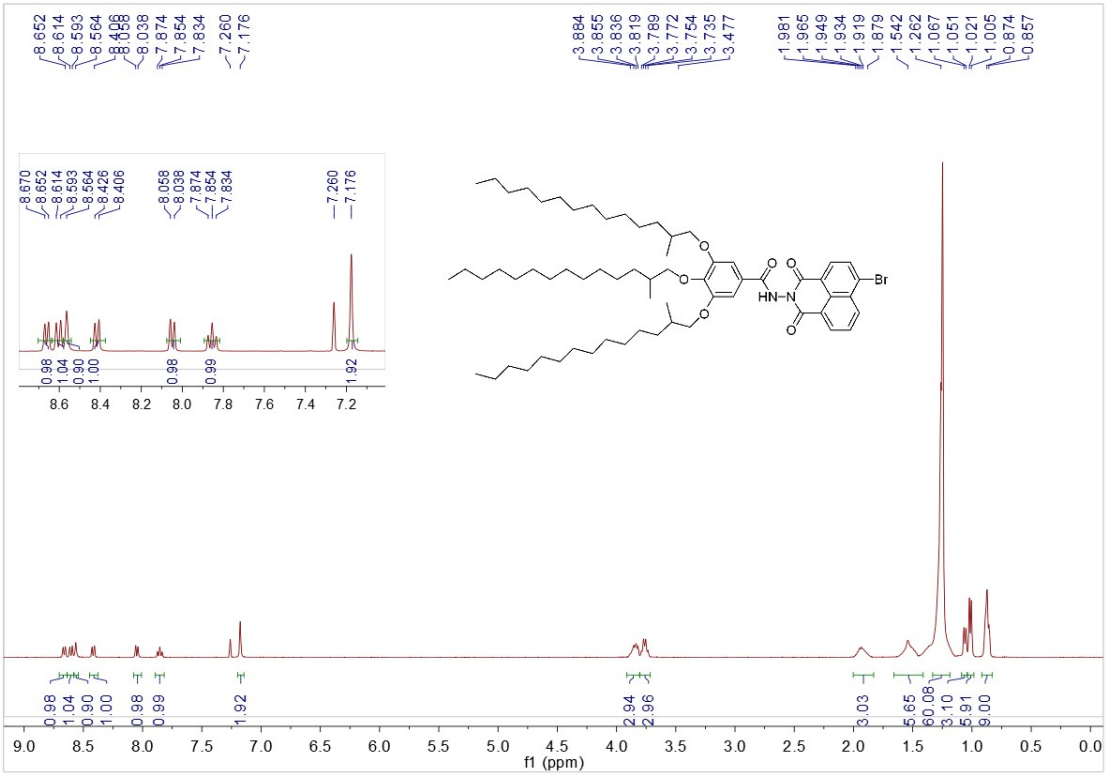


Fig. S21 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **4-3**.

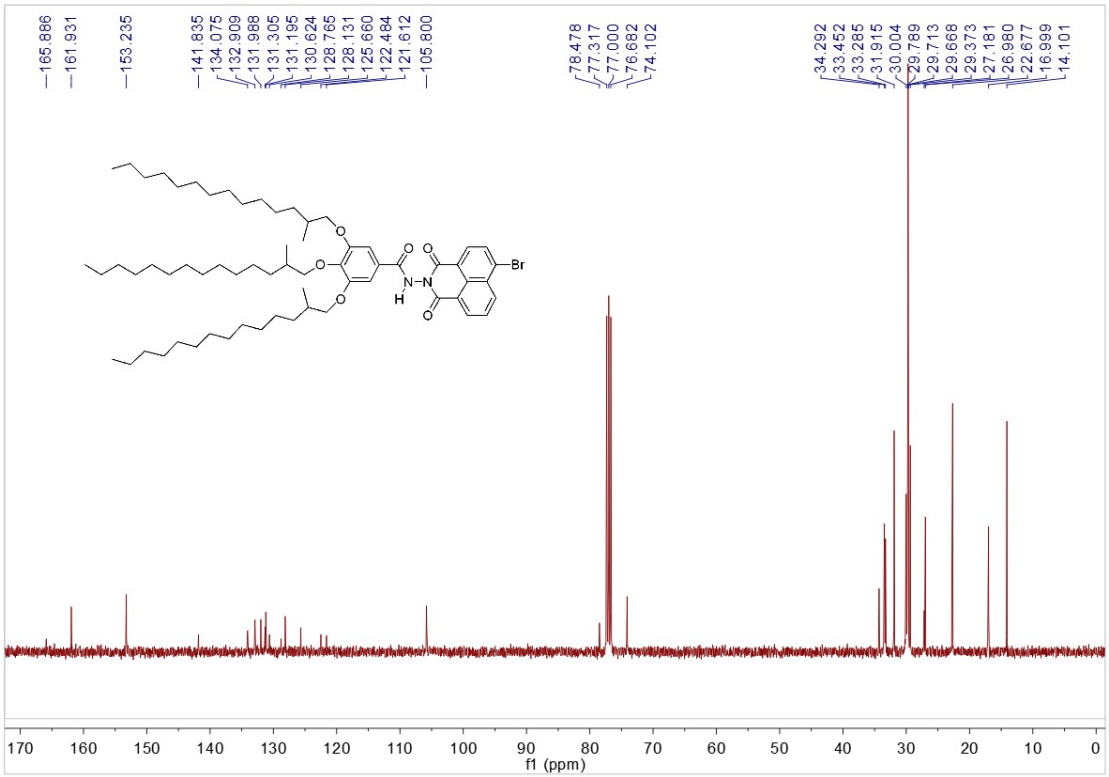


Fig. S22 ^{13}C NMR ($CDCl_3$, 100 MHz, ppm) spectra of compound **4-3**.

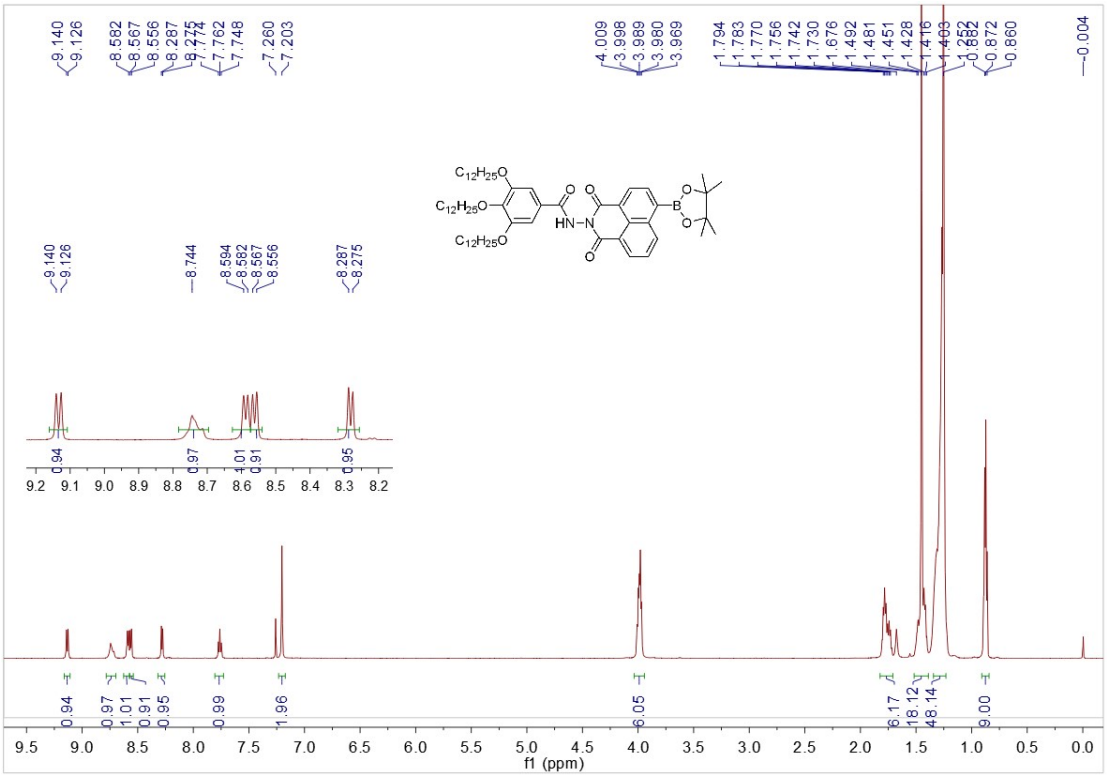


Fig. S23 ^{1}H NMR ($CDCl_3$, 600 MHz, ppm) spectra of compound **5-1**.

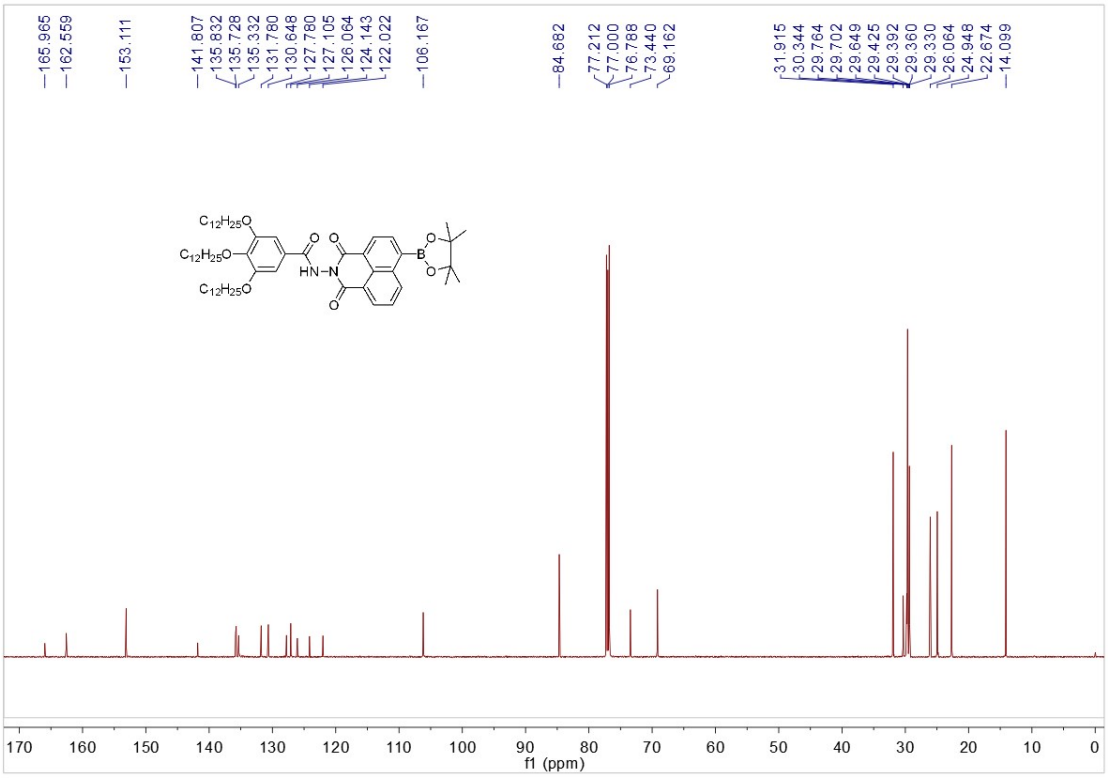


Fig. S24 ^{13}C NMR ($CDCl_3$, 150 MHz, ppm) spectra of compound **5-1**.

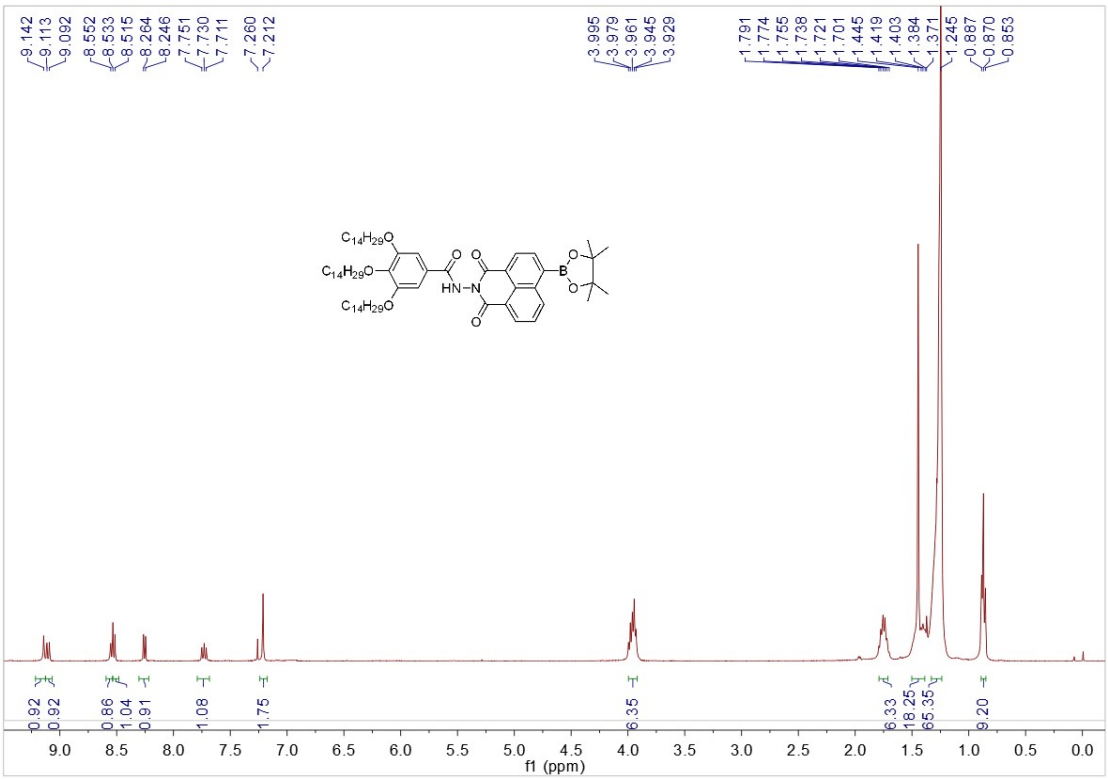


Fig. S25 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **5-2**.

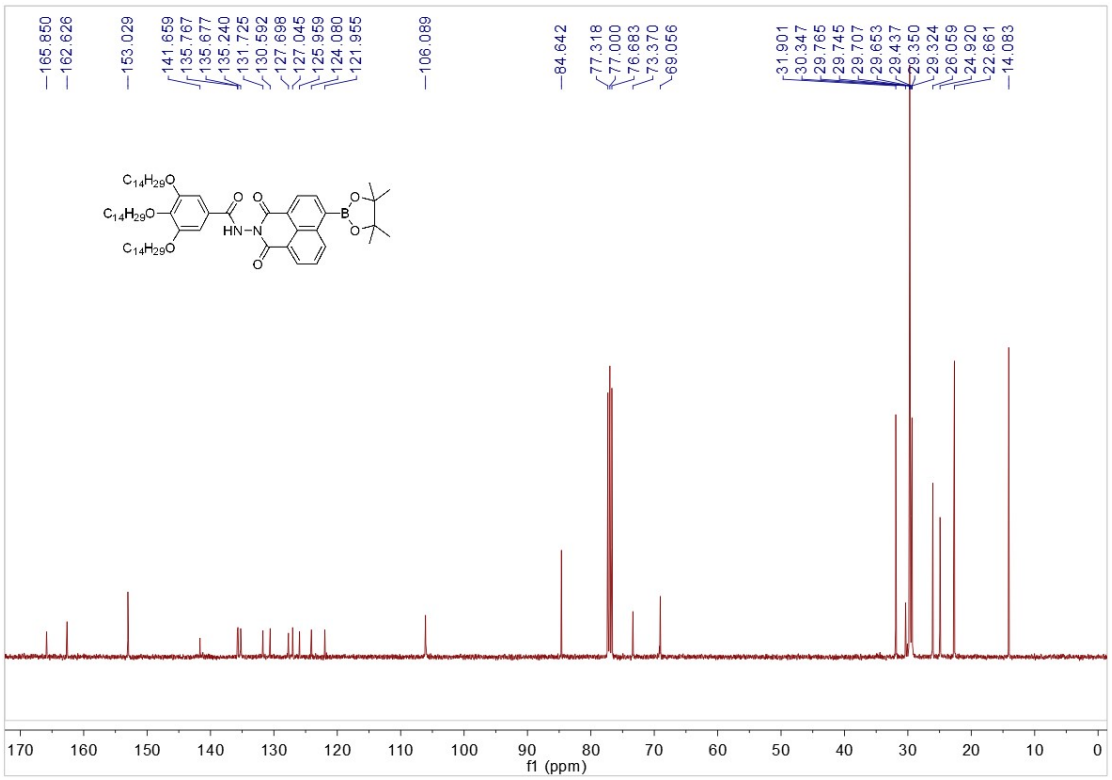


Fig. S26 ^{13}C NMR ($CDCl_3$, 100 MHz, ppm) spectra of compound **5-2**.

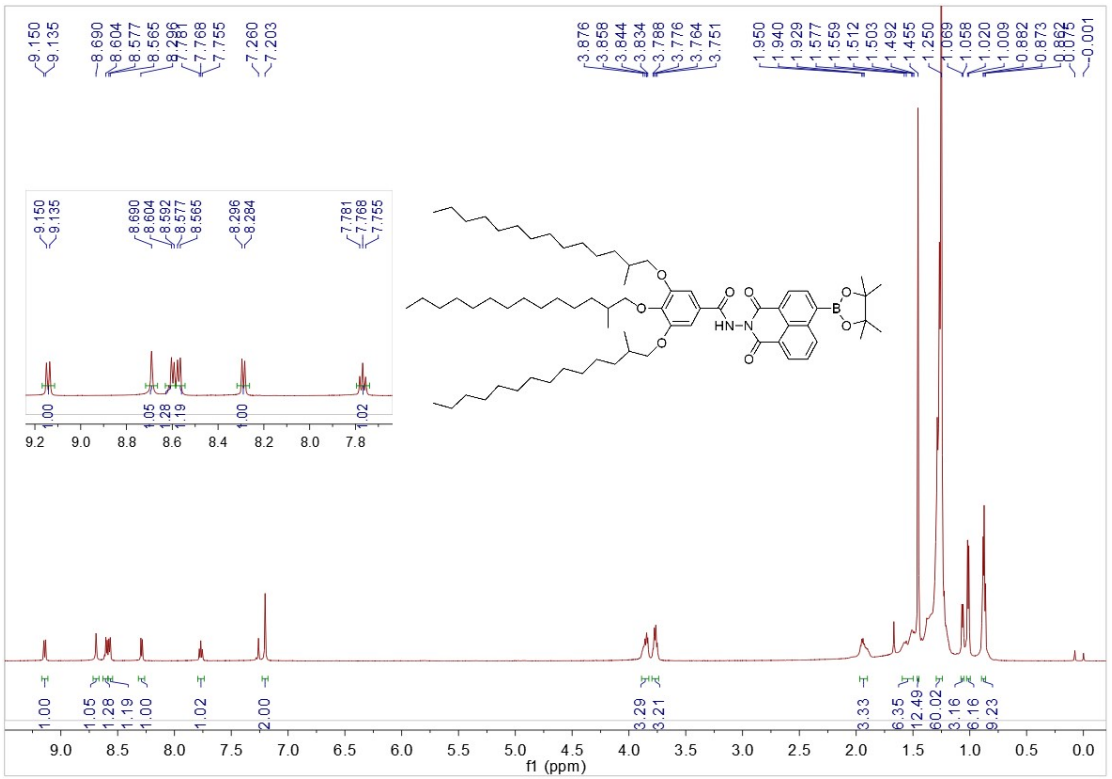


Fig. S27 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **5-3**.

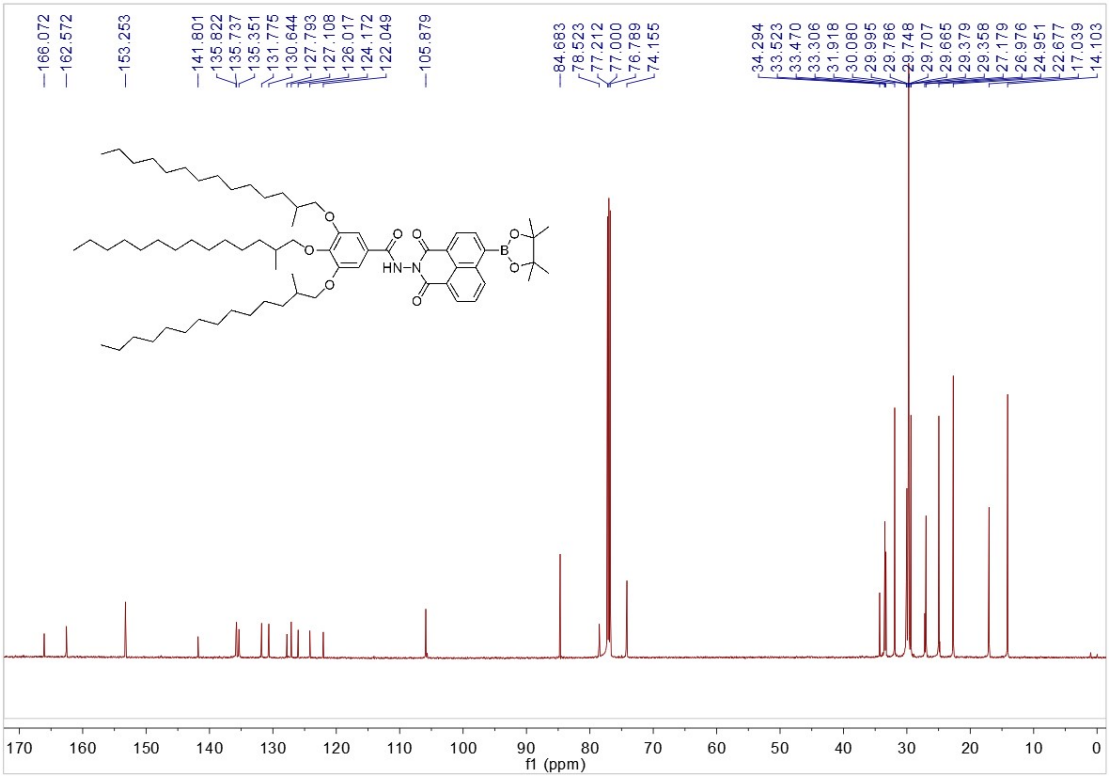


Fig. S28 ^{13}C NMR ($CDCl_3$, 150 MHz, ppm) spectra of compound **5-3**.

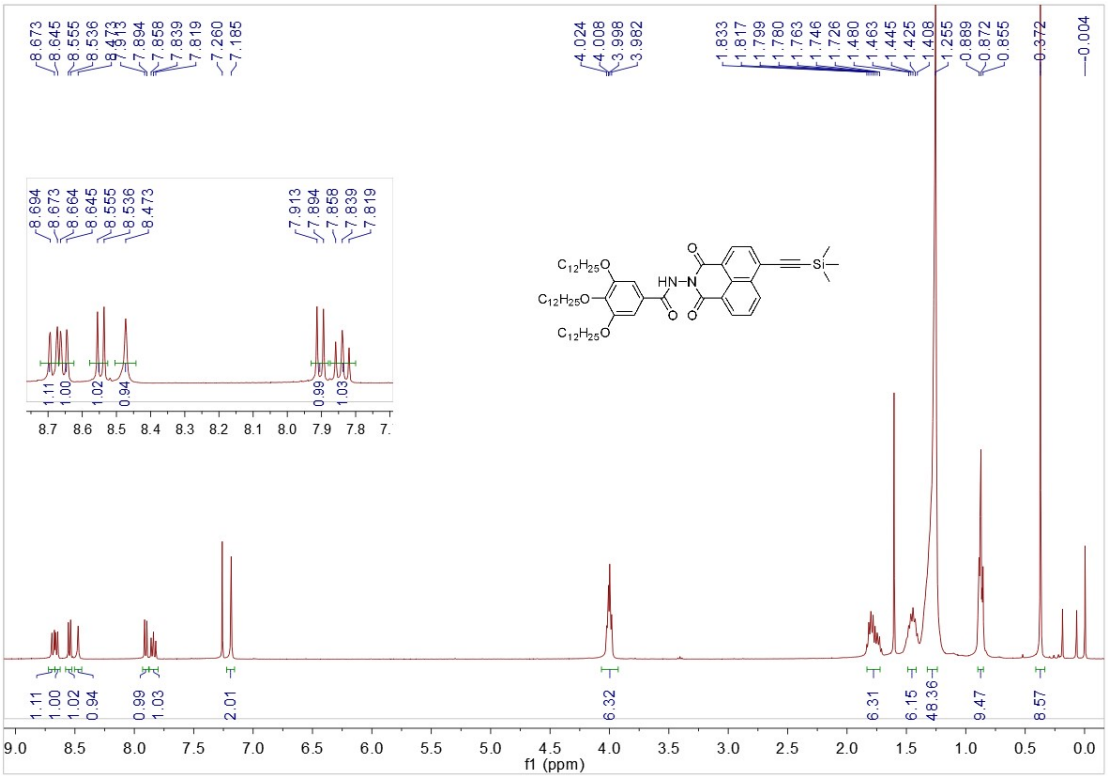


Fig. S29 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **6-1**.

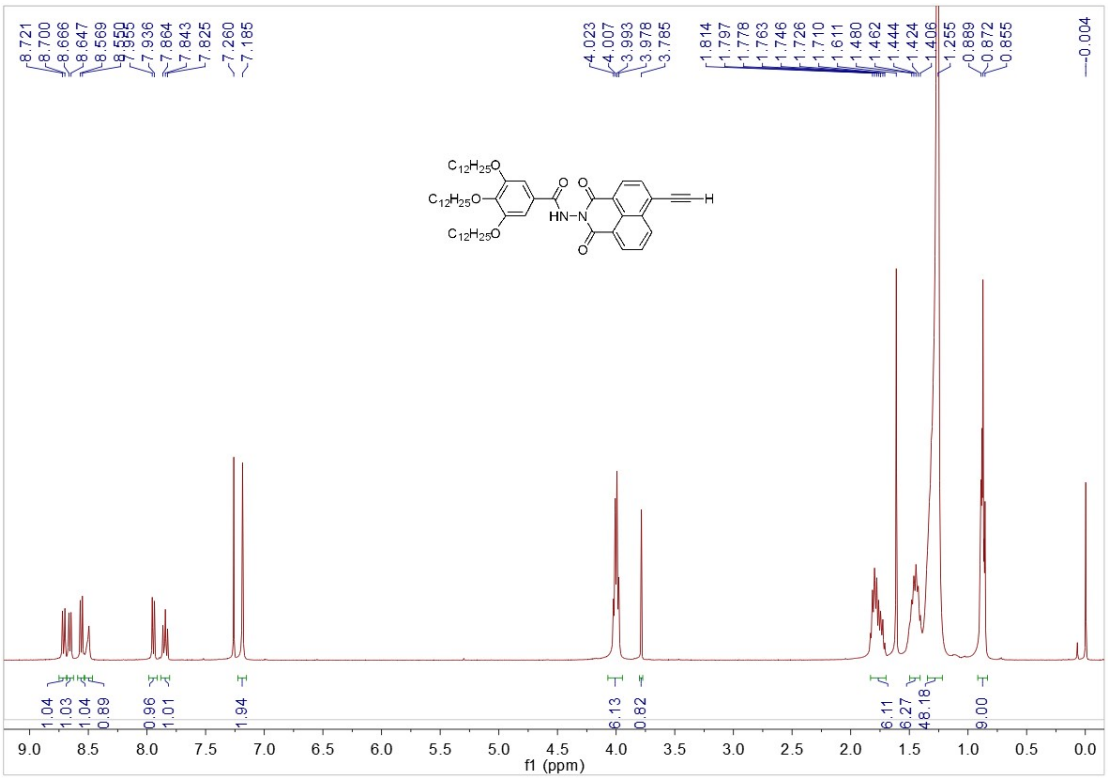


Fig. S30 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **7-1**.

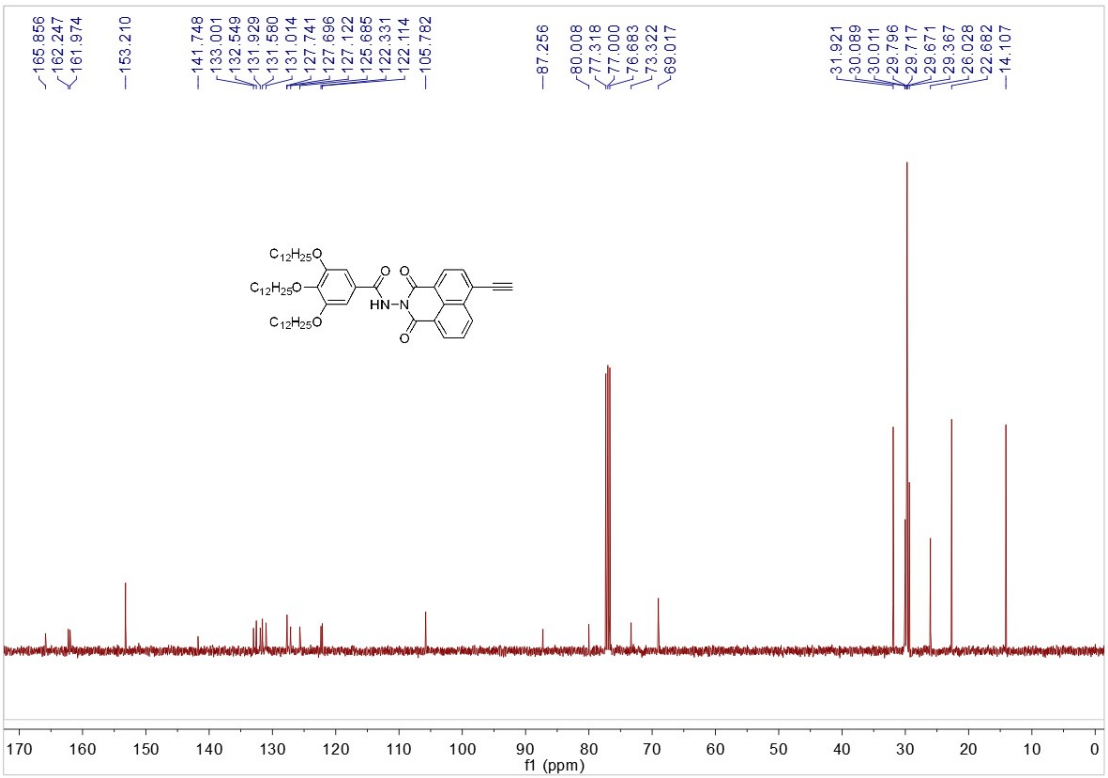


Fig. S31 ^{13}C NMR ($CDCl_3$, 100 MHz, ppm) spectra of compound **7-1**.

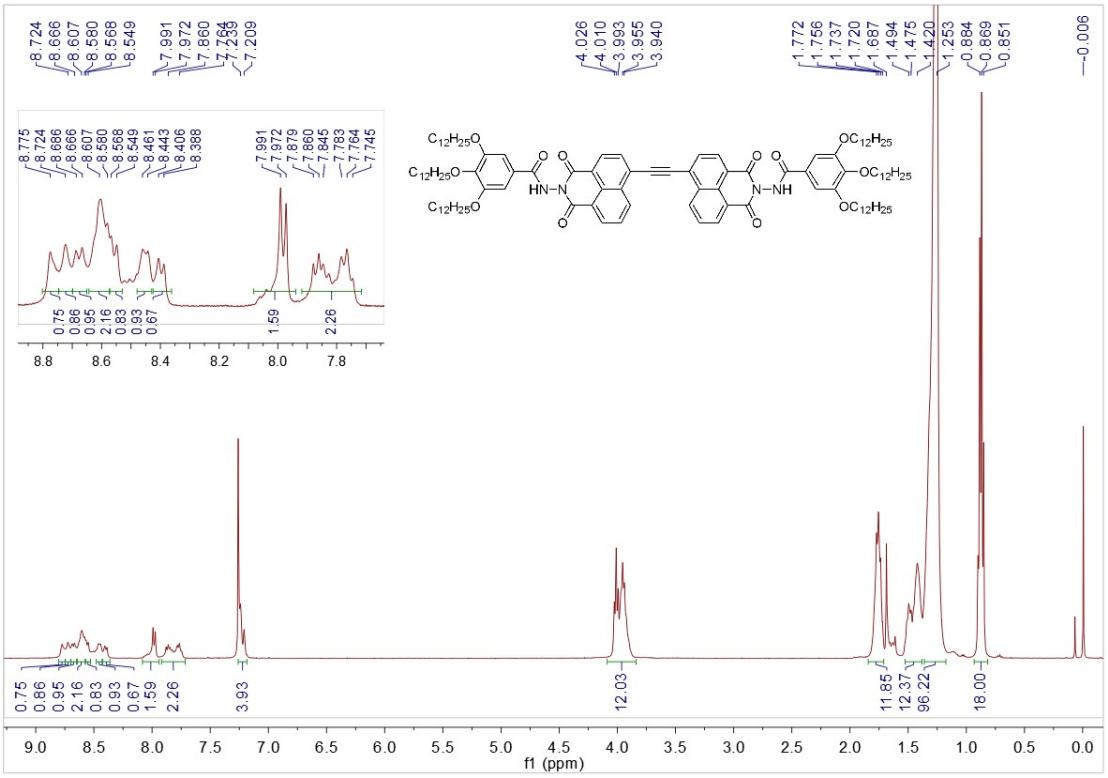


Fig. S32 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **ABNI-3C12**.

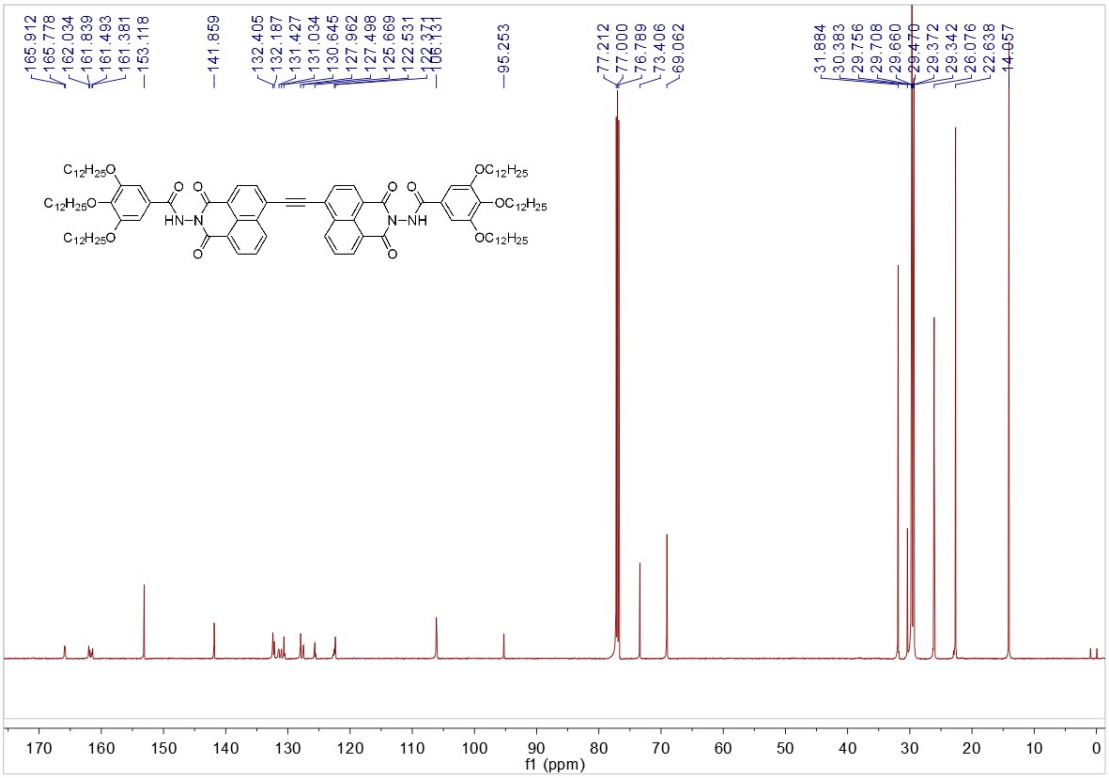


Fig. S33 ^{13}C NMR ($CDCl_3$, 150 MHz, ppm) spectra of compound **ABNI-3C12**.

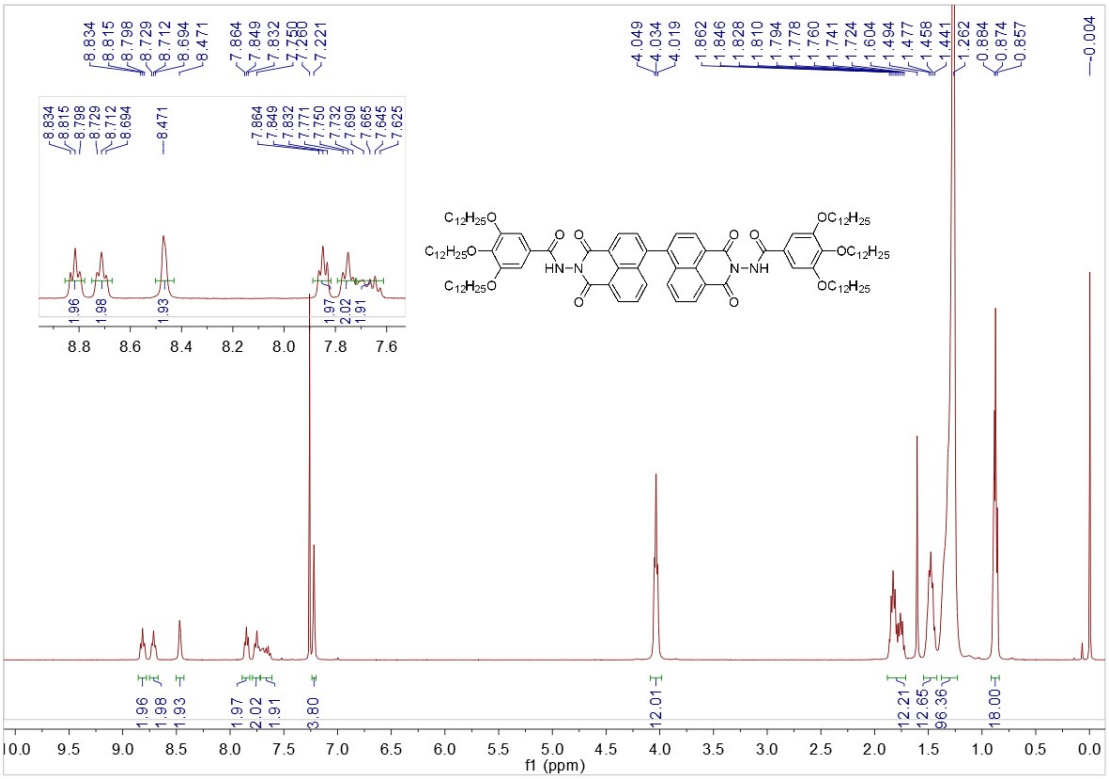


Fig. S34 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **BNI-3C12**.

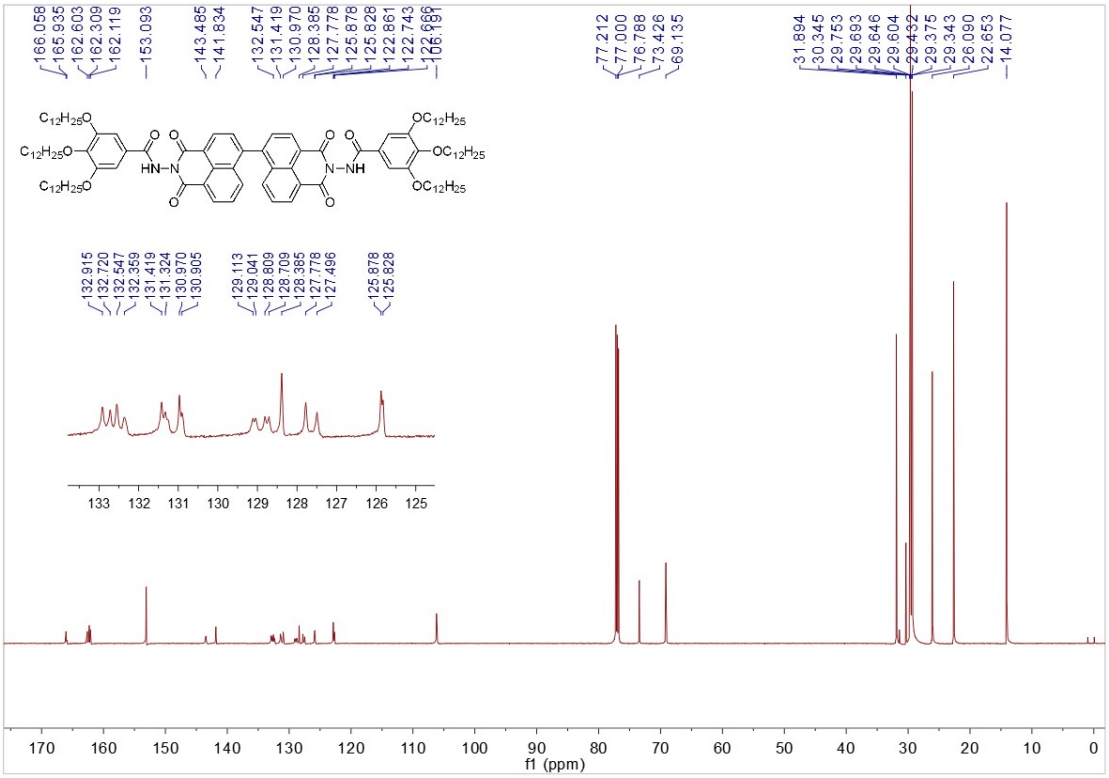


Fig. S35 ^{13}C NMR ($CDCl_3$, 150 MHz, ppm) spectra of compound **BNI-3C12**.

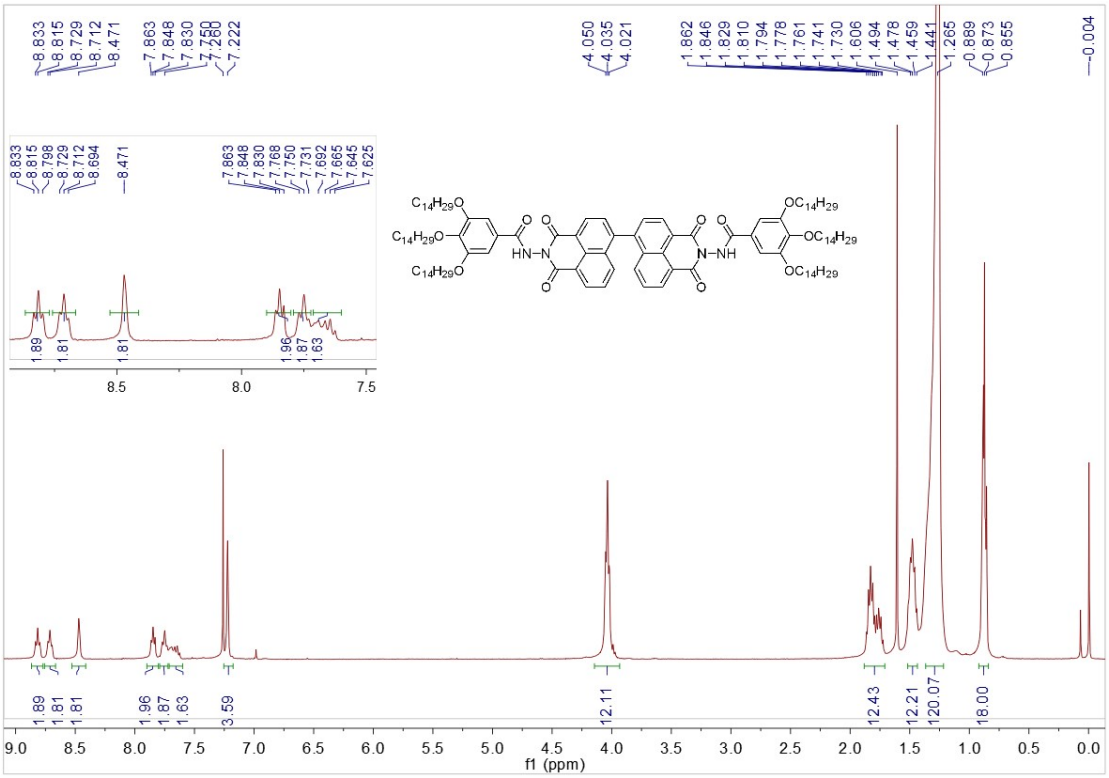


Fig. S36 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **BNI-3C14**.

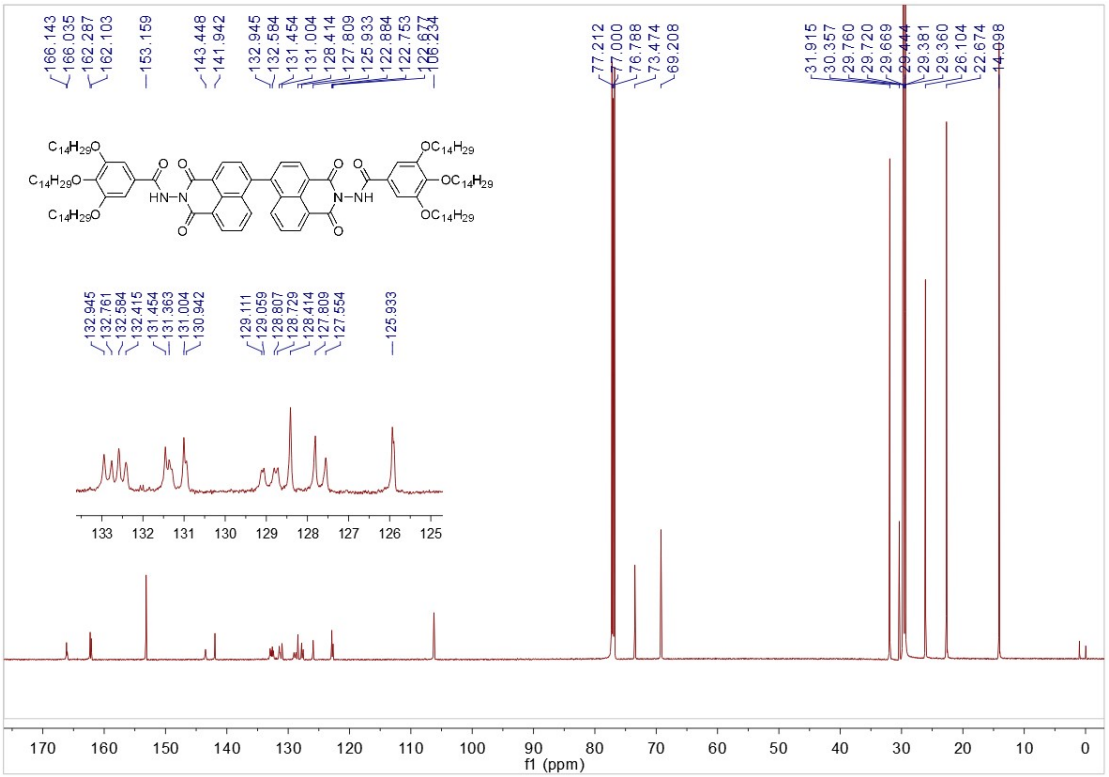


Fig. S37 ^{13}C NMR ($CDCl_3$, 150 MHz, ppm) spectra of compound **BNI-3C14**.

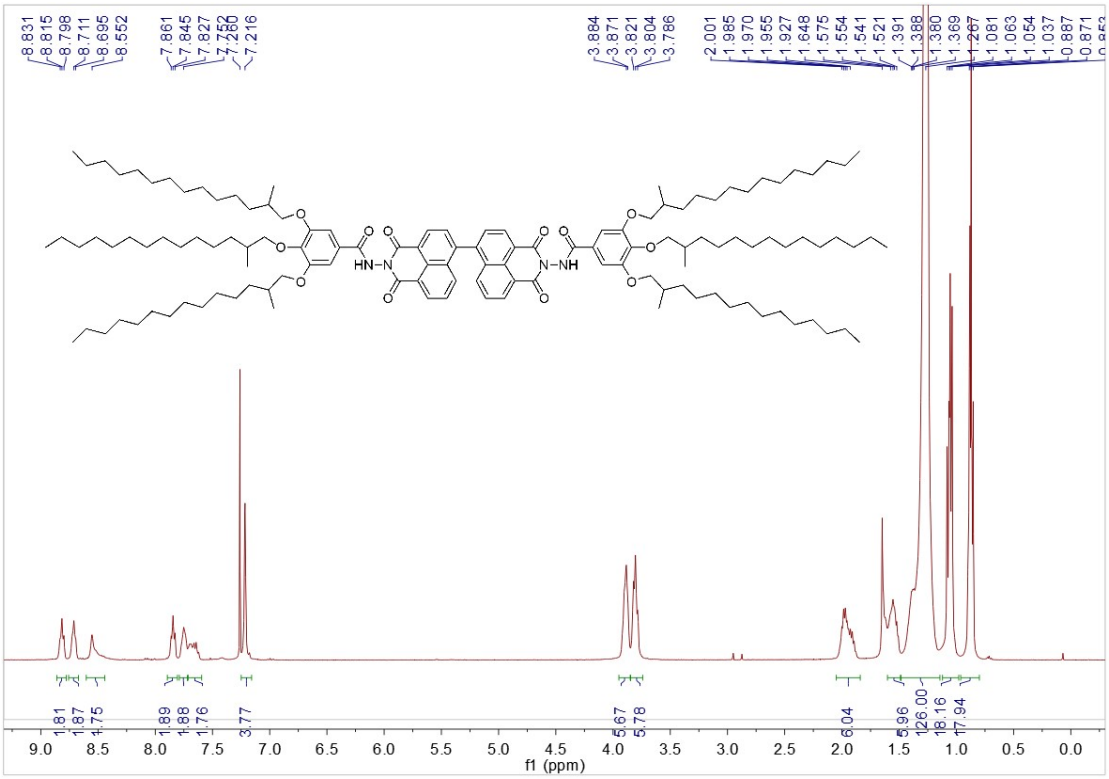


Fig. S38 ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) spectra of compound **BNI-3C114**.

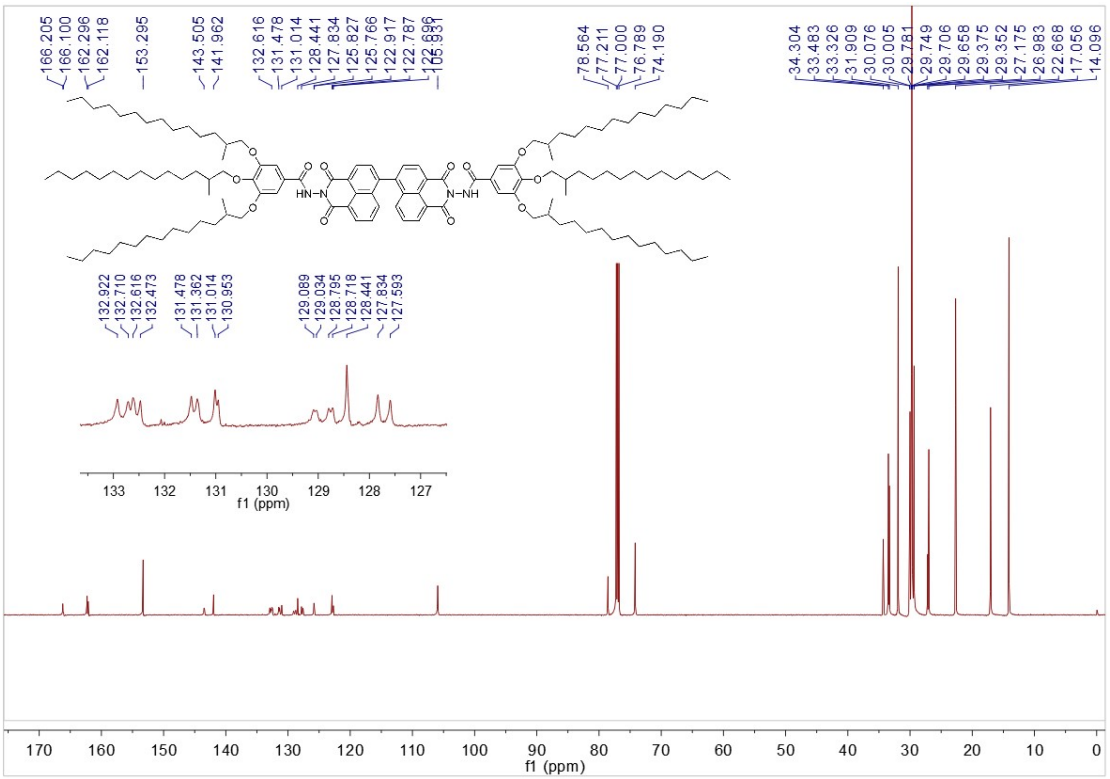


Fig. S39 ^{13}C NMR ($CDCl_3$, 150 MHz, ppm) spectra of compound **BNI-3C114**.

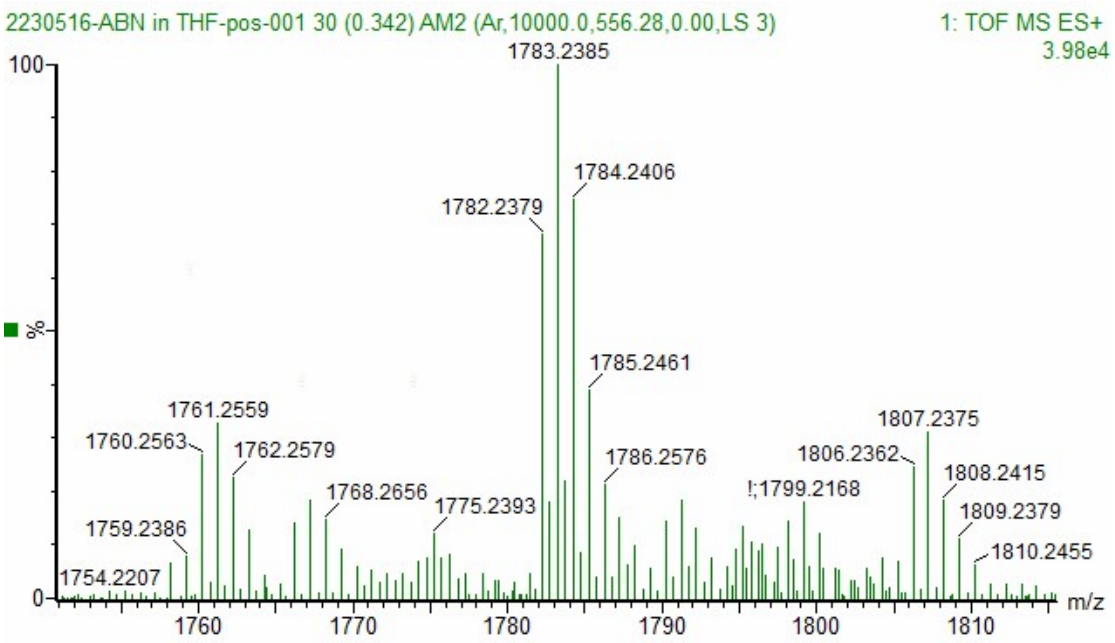


Fig. S40 HRMS (ESI-TOF) spectra of **ABNI-3C12**.

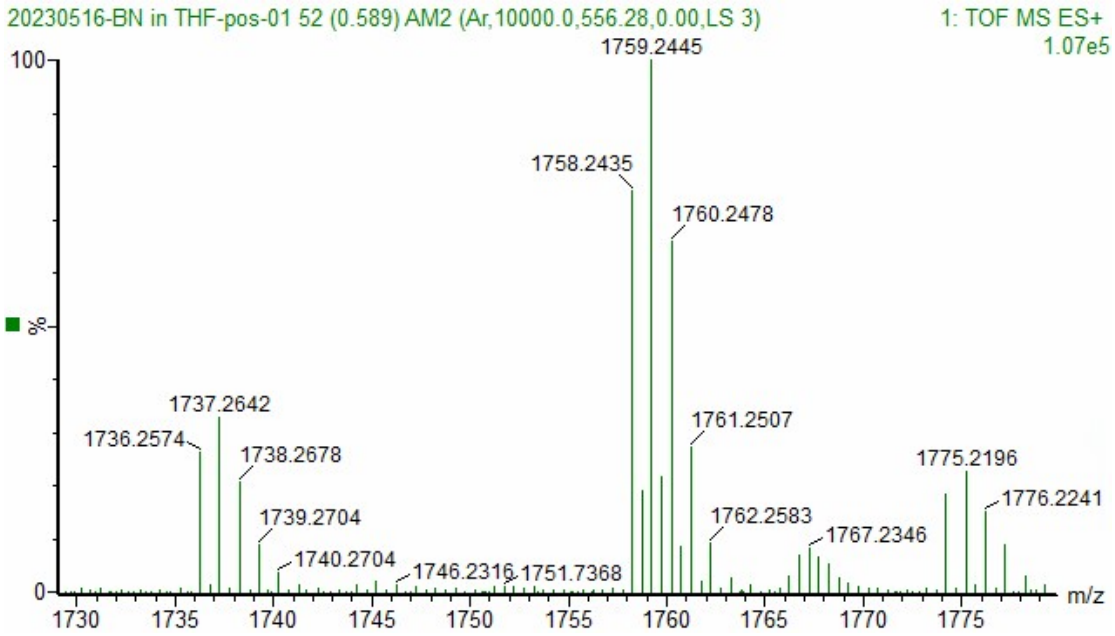


Fig. S41 HRMS (ESI-TOF) spectra of **BNI-3C12**.

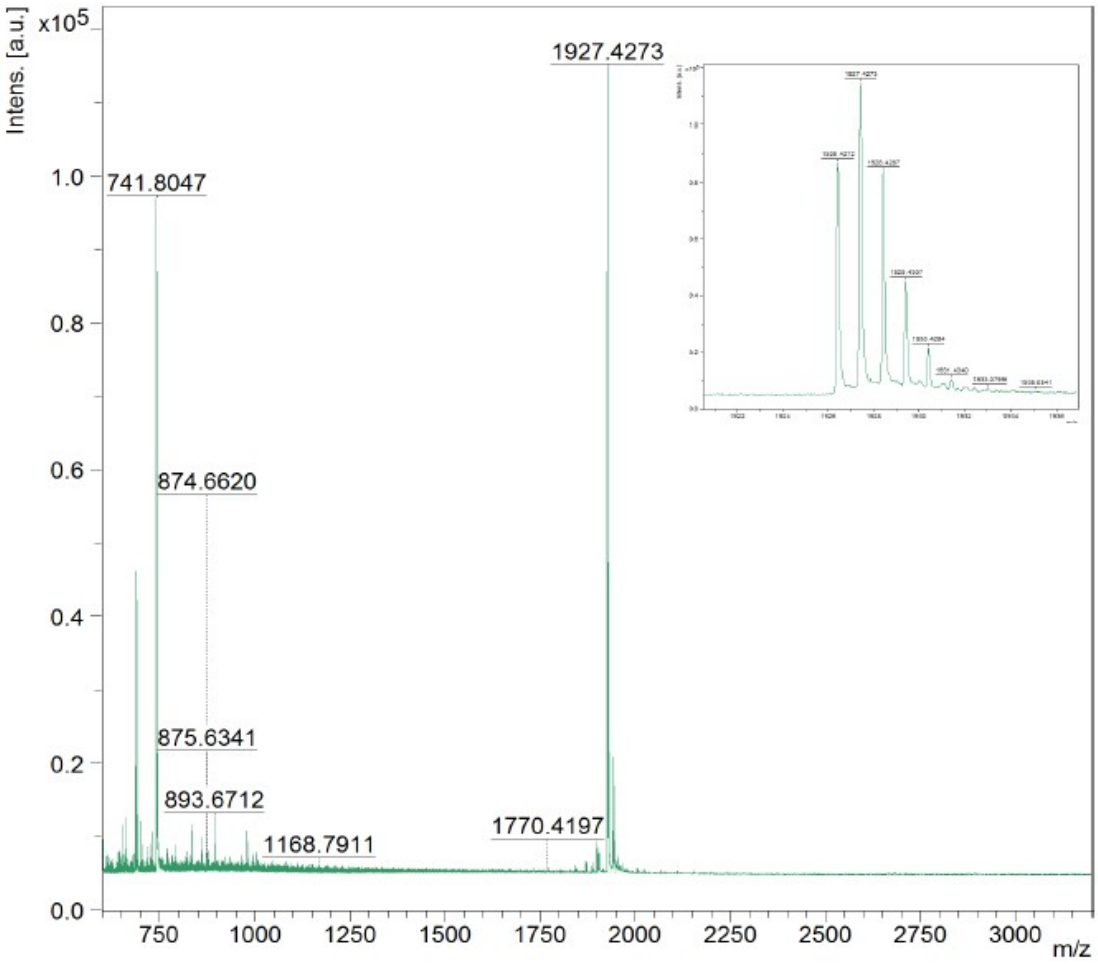


Fig. S42 HRMS (ESI-TOF) spectra of **BNI-3C14**.

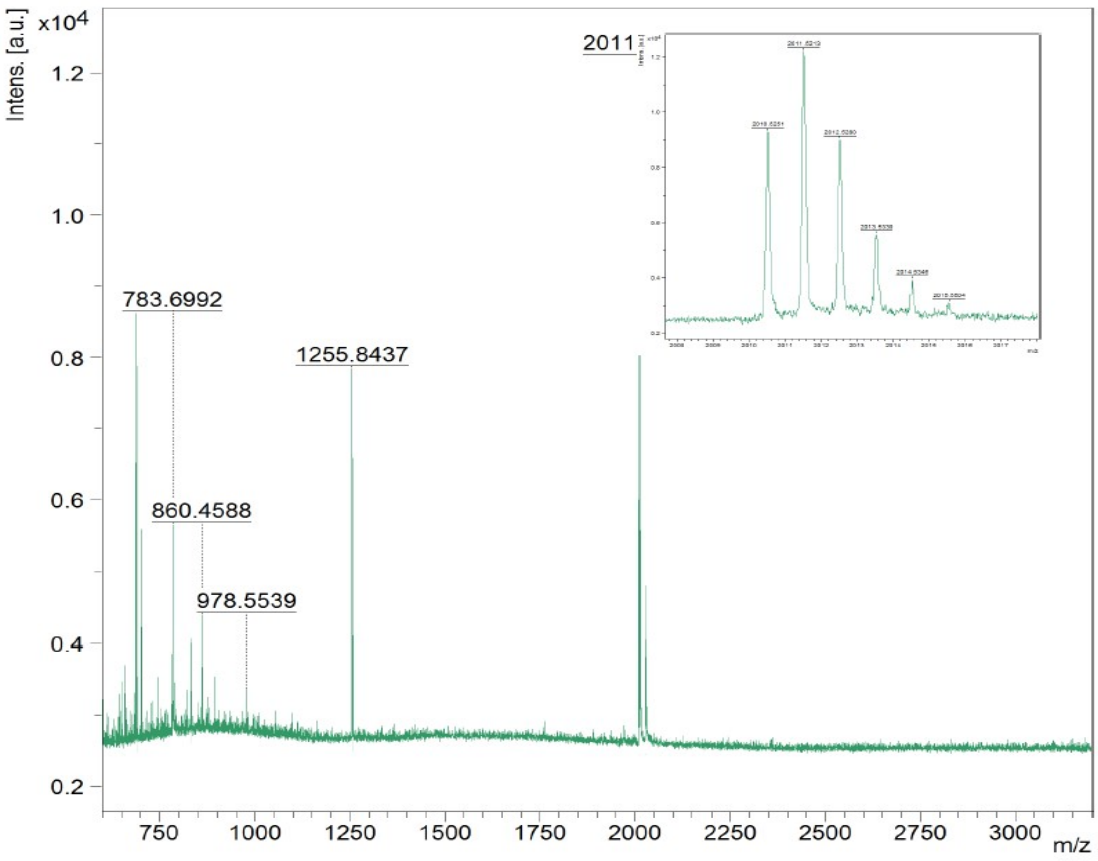


Fig. S43 HRMS (ESI-TOF) spectra of **BNI-3C114**.

References

[S1] a) Parker C A, Rees W T, Correction of Fluorescence Spectra and Measurement of Fluorescence Quantum Efficiency, Analyst, 1960; 85: 587-600. b) Williams A T R, Winfield S A, Miller J N, Relative Fluorescence Quantum Yields Using a Computer-controlled Luminescence Spectrometer, Analyst, 1983; 108: 1067-1071.

[S2] Xing W, Xiao Y, Zhao K, Kong T, Cheng X H, Control of Supramolecular Nano- assemblies by Tuning the Polarities of Linkages and Solvents, J. Mol. Liq., 2018; 272: 1-7.

[S3] a) New J. Chem., 2017,41, 2004-2012; b) Chem. Sci., 2022,13, 7892-7899