Electronic Supplementary Material (ESI) for Soft Matter.

Supplementary Information

for

Chiral three-dimensional supramolecular assemblies: colloidal onions, cubosomes, and hexosomes

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Contents

S1. Materials

1-aminopropyl-3,5,7,9,11,13,15-heptaisobutyl-POSS was purchased from Hybrid Plastics and used without further purification. 1,4,5,8-naphthalenetetracarboxylic dianhydride (AR, Innochem), *L*-phenylalanine (AR, Energy Chemical), *D*-phenylalanine (AR, Energy Chemical), *N*, *N*'-diisopropylethylamine (DIPEA, AR, Alfa Aesar), 2-ethoxy-1-ethoxycarbonyl-1,2-dihydroquinoline (EEDQ, 99%, Aladdin) and tris(hydroxyl-methyl)aminomethane (Tris, 99%, Alfa Aesar) were all used as received. $(Bu_4N)_6H_3P_2W_{15}V_3O_{62}$ (Wells-Dawson POM) was synthesized according to the literature procedure.[1] Other reagents were purchased from major chemical suppliers and used as received unless otherwise noted. All solvents were analytical grade and were dried and distilled prior to use according to standard procedures.

S2. Characterization Methods

Nuclear Magnetic Resonance Spectroscopy (NMR). ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra were performed on a BRUKER AVANCE III 400MHz spectrometer.

Matrix-Assisted Laser Desorption/Ionization Time-of-Flight Spectroscopy (MALDI-TOF). MALDI-TOF mass spectra were carried out on a Bruker Autoflex MALDI TOF-TOF III LRF200 CID. Trans-2-[3-(4-tert-butylphenyl)-2-methyl-2-propenylidene]-malononitrile (DCTB, Aldrich, >98%) was used as the matrix for MALDI-TOF measurements.

Transmission Electron Microscopy (TEM). The bright field TEM images were performed to use a FEI Tecnai G2 F20 operated at 200 kV with the image point resolution of 0.24 nm.

Scanning Electron Microscopy (SEM). The SEM characterization was performed on a ZEISS MERLIN Compact at an acceleration voltage of 5 kV.

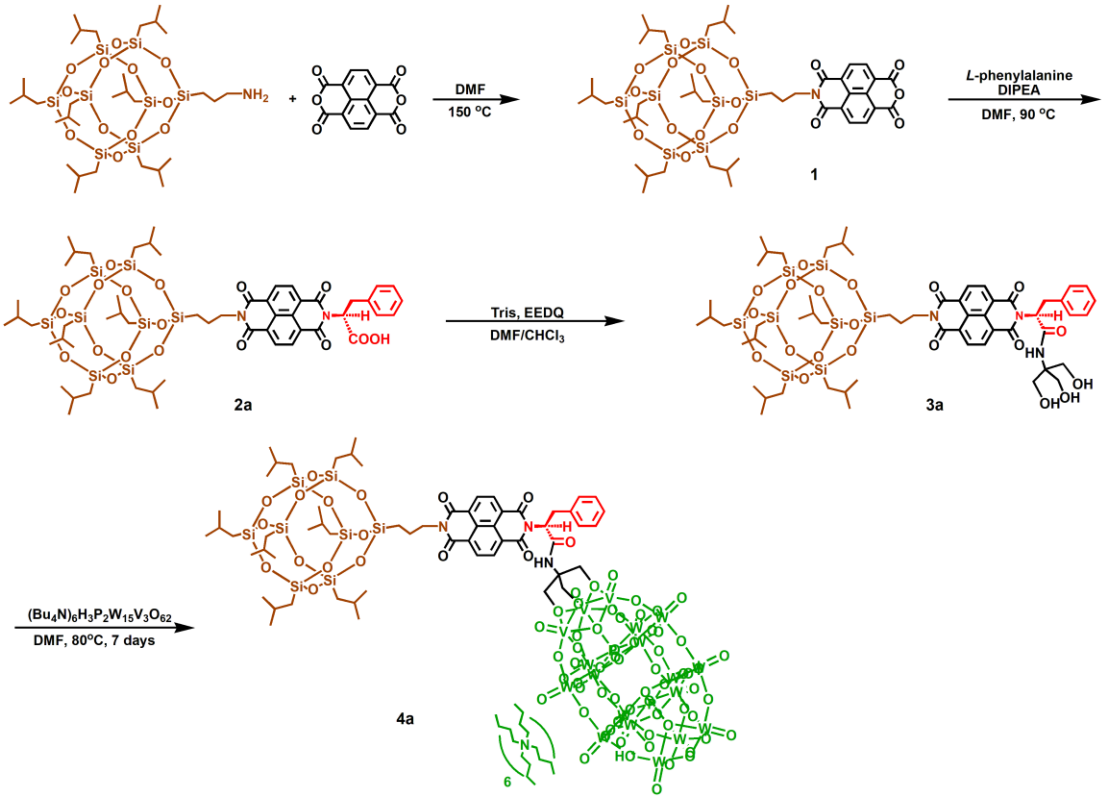
Synchrotron Radiation Small-Angle X-ray Scattering (SAXS). SAXS measurements were carried out on the 1W2A beam line at Beijing Synchrotron Radiation Facility (BSRF) and Xeuss system of Xenocs. The X-ray wavelength was 1.54 Å and the sample-to-detector distance was 1.5 m.

Ultraviolet-visible Spectrophotometer (UV-vis). The UV-vis absorption spectra were collected by Hitachi U-3900H ultraviolet-visible spectrophotometer. The slit width was 5 nm.

Circular Dichroism (CD). The CD spectra were collected by JASCO J-715 circular dichroism spectroscopy. The scanning speed was 100 nm/min, the wavelength range was 300~650 nm.

Linear dichroism (LD). The LD spectra were collected by JASCO J-1500 circular dichroism spectroscopy. The scanning speed was 50 nm/min and the wavelength range was 300~500 nm.

S3. Synthetic Procedures



Scheme S1. Synthetic route of POSS-NDI-*L*-Phe-POM

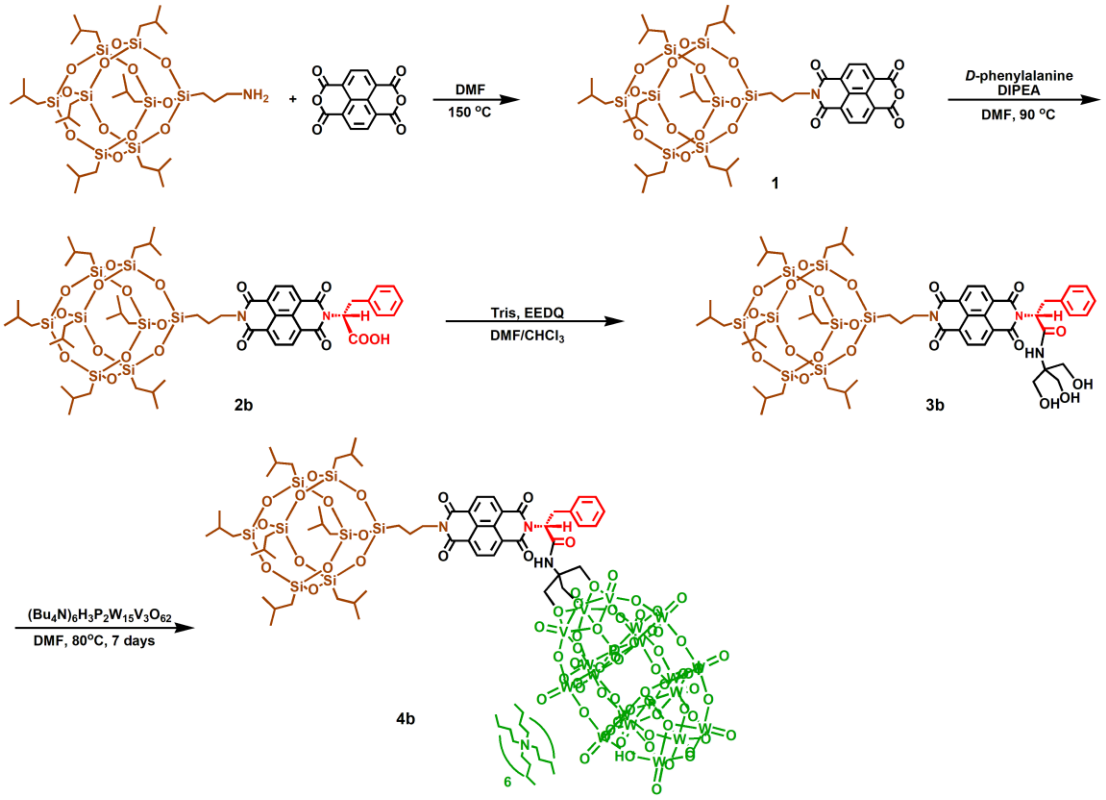
(1): 1-aminopropyl-3,5,7,9,11,13,15-heptaisobutyl-POSS (3.5 g, 4.0 mmol) and 1,4,5,8-naphthalenetetracarboxylic dianhydride (6.43 g, 24.0 mmol) were dissolved in 250 mL DMF, the reaction mixture was stirred for 12 h at 150 °C. After cooling to room temperature, 600 mL water was poured into the mixture, resulting in a large amount of precipitate. The precipitate was filtered and washed with 200 mL water, the resulting residue was purified by gel column chromatography to obtain **1** (yield: 3.82 g, 85 %). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.90 – 8.77 (m, 4H), 4.24 – 4.17 (m, 2H), 1.89 – 1.79 (m, 9H), 0.93 (t, J = 6.3 Hz, 42H), 0.77 – 0.67 (m, 2H), 0.59 (t, J = 5.8 Hz, 14H) (Figure S18).

(2a): 1 (1.0 g, 0.89 mmol) and *L*-phenylalanine (441 mg, 2.67 mmol) were dissolved in 100 mL DMF, DIPEA (465 μL, 4.84 mmol) was added. The reaction mixture was stirred for 12 h at 90 °C. After cooling to room temperature, the mixture was concentrated under reduced pressure and purified by gel column chromatography to obtain **2a** (yield: 0.92 g, 81%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.67 (dd, J = 21.7, 7.5 Hz, 4H), 7.17 – 6.99 (m, 5H), 6.04 (s, 1H), 4.21 – 4.13 (m, 2H), 3.67 (d, J = 14.0 Hz, 1H), 3.51 (d, J =

11.1 Hz, 1H), 1.90 – 1.76 (m, 9H), 0.93 (t, *J* = 6.3 Hz, 42H), 0.74 – 0.66 (m, 2H), 0.59 (t, *J* = 5.8 Hz, 14H) (Figure S19).

(3a): 2a (110 mg, 0.086 mmol), Tris (32 mg, 0.26 mmol) and EEDQ (64 mg, 0.26 mmol) were dissolved in 10 mL DMF and 10 mL $CHCl_3$, and the reaction mixture was stirred for 12 h at 100 °C. After cooling to room temperature, the mixture was concentrated under reduced pressure and purified by gel column chromatography to obtain **3a** (yield: 85 mg, 72%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.70 (dd, *J* = 21.3, 7.5 Hz, 4H), 7.20 – 7.03 (m, 5H), 6.09 (t, *J* = 7.7 Hz, 1H), 4.18 (t, *J* = 7.2 Hz, 2H), 3.75 (dd, *J* = 37.7, 12.0 Hz, 6H), 3.67 – 3.62 (m, 1H), 3.55 – 3.48 (m, 1H), 1.89 – 1.78 (m, 9H), 0.94 (t, *J* = 5.8 Hz, 42H), 0.74 – 0.69 (m, 2H), 0.59 (t, *J* = 5.8 Hz, 14H) (Figure S20).

(4a): 3a (63 mg, 0.045 mmol) and Wells-Dawson POM (205 mg, 0.038 mmol) were dissolved in 5 mL DMF, the reaction mixture was stirred for 7 days at 80 °C. After cooling to room temperature, the mixture was concentrated under reduced pressure. The resulting residue was dissolved in 5 mL acetonitrile, and was added dropwise into stirred diethyl ether (50 mL) to precipitate the product and remove the excess 3a, this procedure was repeated twice. The resulting solid was washed with 20 mL diethylether and was dried under vacuum at 45 °C for 12 h to obtain **4a** (yield: 90 mg, 35 %). ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.66 – 8.56 (m, 4H), 7.41 (br 1H), 7.12 – 6.94 (m, 5H), 5.80 – 5.76 (m, 1H), 5.57 (dd, *J* = 18.2, 11.9 Hz, 6H), 4.07 – 4.00 (m, 2H), 3.64 (d, *J* = 11.8 Hz, 2H), 3.24 – 3.10 (m, 48H), 1.85 – 1.74 (m, 9H), 1.64 – 1.47 (m, 48H), 1.40 – 1.27 (m, 48H), 1.02 – 0.86 (m, 114H), 0.72 – 0.64 (m, 2H), 0.59 (t, *J* = 5.8 Hz, 14H) (Figure S21). ^{13}C NMR (101 MHz, DMSO-d_6): δ 129.22, 128.07, 126.71, 126.29, 57.68, 54.97, 54.75, 48.91, 34.70, 25.45, 25.39, 23.58, 23.19, 22.07, 21.93, 19.31, 13.62 (Figure S22). MS (MALDI-TOF, m/z): $[M–2TBA+3H]^+$: Calcd: 6261.20 Da, Found: 6261.45 Da. $[M–TBA+2H]^+$: Calcd: 6502.67 Da, Found: 6502.53 Da. $[M–TBA+Na+K]^+$: Calcd: 6562.76 Da, Found: 6562.82 Da. $[M+H]^+$: Calcd: 6744.14 Da, Found: 6744.10 Da. $[M+Na+K–H]^+$: Calcd: 6804.23 Da, Found: 6804.22 Da. $[M+TBA]^+$: Calcd: 6985.61 Da, Found: 6985.16 Da. $[M+2TBA–H]^+$: Calcd: 7227.08 Da, Found: 7227.56 Da (Figure S23).

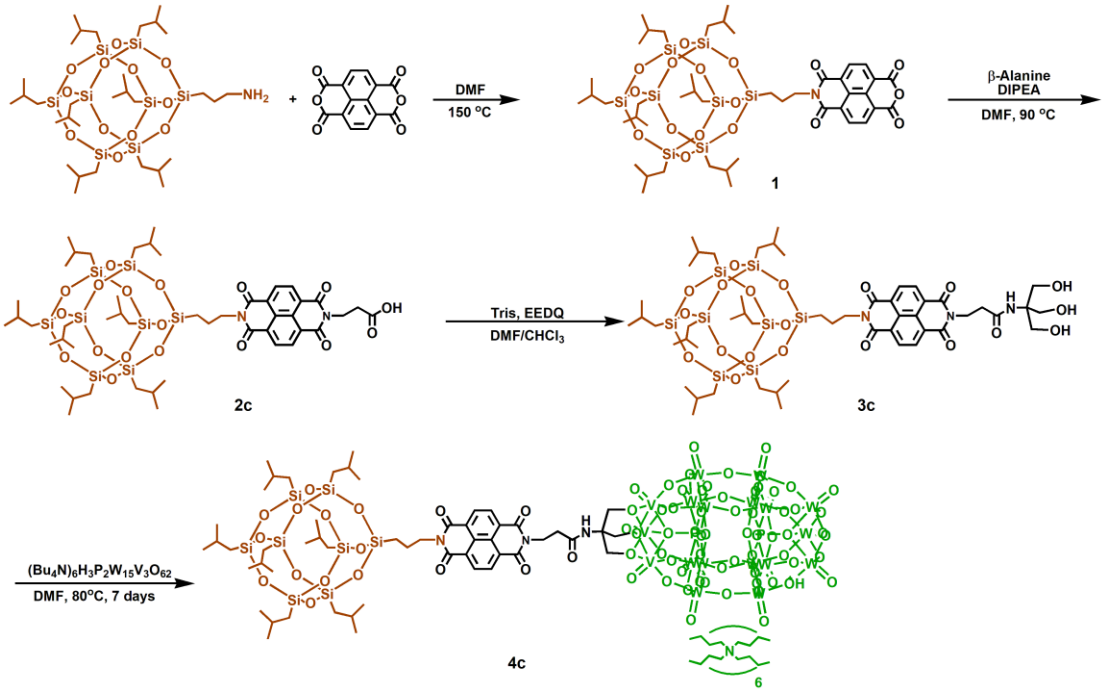


Scheme S2. Synthetic route of POSS-NDI-*D*-Phe-POM

(2b): 1 (1.0 g, 0.89 mmol) and *D*-phenylalanine (441 mg, 2.67 mmol) were dissolved in 100 mL DMF, DIPEA (465 μL, 4.84 mmol) was added. The reaction mixture was stirred for 12 h at 90 °C. After cooling to room temperature, the mixture was concentrated under reduced pressure and purified by gel column chromatography to obtain **2b** (yield: 0.94 g, 83%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.67 (dd, J = 21.7, 7.5 Hz, 4H), 7.17 – 6.99 (m, 5H), 6.05 (dd, J = 10.3, 5.4 Hz, 1H), 4.16 (t, J = 7.2 Hz, 2H), 3.73 (dd, J = 14.3, 5.5 Hz, 1H), 3.55 (dd, J = 14.1, 10.5 Hz, 1H), 1.90 – 1.76 (m, 9H), 0.93 (t, J = 6.3 Hz, 42H), 0.74 – 0.66 (m, 2H), 0.59 (t, J = 5.8 Hz, 14H) (Figure S24).

(3b): 2b (110 mg, 0.086 mmol), Tris (32 mg, 0.26 mmol) and EEDQ (64 mg, 0.26 mmol) were dissolved in 10 mL DMF and 10 mL $CHCl_3$, and the reaction mixture was stirred for 12 h at 100 °C. After cooling to room temperature, the mixture was concentrated under reduced pressure and purified by gel column chromatography to obtain **3b** (yield: 94 mg, 80%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.70 (dd, J = 21.3, 7.5 Hz, 4H), 7.20 – 7.03 (m, 5H), 6.09 (t, J = 7.7 Hz, 1H), 4.18 (t, J = 7.2 Hz, 2H), 3.75 (dd, J = 37.7, 12.0 Hz, 6H), 3.67 – 3.62 (m, 1H), 3.55 – 3.48 (m, 1H), 1.89 – 1.78 (m, 9H), 0.94 (t, J = 5.8 Hz, 42H), 0.74 – 0.69 (m, 2H), 0.59 (t, J = 5.8 Hz, 14H) (Figure S25).

(4b): 3b (63 mg, 0.045 mmol) and Wells-Dawson POM (205 mg, 0.038 mmol) were dissolved in 5 mL DMF, the reaction mixture was stirred for 7 days at 80 °C. After cooling to room temperature, the mixture was concentrated under reduced pressure. The resulting residue was dissolved in 5 mL acetonitrile, and was added dropwise into stirred diethylether (50 mL) to precipitate the product and remove the excess 3b, this procedure was repeated twice. The resulting solid was washed with 20 mL diethylether and was dried under vacuum at 45 °C for 12 h to obtain **4b** (yield: 85 mg, 36 %). ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.66 – 8.56 (m, 1H), 7.41 (br, 1H), 7.12 – 6.94 (m, 5H), 5.80 – 5.76 (m, 1H), 5.57 (dd, J = 18.2, 11.9 Hz, 6H), 4.07 – 4.00 (m, 2H), 3.61 – 3.58 (m, 2H), 3.24 – 3.10 (m, 48H), 1.85 – 1.74 (m, 9H), 1.64 – 1.47 (m, 48H), 1.40 – 1.27 (m, 48H), 1.02 – 0.86 (m, 114H), 0.72 – 0.64 (m, 2H), 0.59 (t, J = 5.8 Hz, 14H) (Figure S26). ^{13}C NMR (101 MHz, DMSO-d_6): δ 137.97, 130.41, 130.17, 129.00, 127.88, 126.56, 126.14, 97.14, 85.94, 68.28, 65.66, 57.51, 54.61, 34.81, 34.10, 33.13, 27.40, 25.28, 23.40, 23.08, 21.92, 21.79, 19.19, 13.50, 9.05 (Figure S27). MS (MALDI-TOF, m/z): $[M–TBA+2H]^+$: Calcd: 6502.75 Da, Found: 6502.67 Da. $[M+H]^+$: Calcd: 6744.14 Da, Found: 6744.19 Da. $[M+Na+K–H]^+$: Calcd: 6804.23 Da, Found: 6804.67 Da. $[M+TBA]^+$: Calcd: 6985.61 Da, Found: 6985.43 Da. $[M+2TBA–H]^+$: Calcd: 7227.08 Da, Found: 7226.76 Da (Figure S28).



Scheme S3. Synthetic route of POSS-NDI-*Ala*-POM

2c: 1 (1.0 g, 0.89 mmol) and β-alanine (238 mg, 2.67 mmol) were dissolved in 100 mL DMF, DIPEA (465 μL, 4.84 mmol) was added. The reaction mixture was stirred for 12 h at 90 °C. After cooling to room temperature, the mixture was concentrated under reduced pressure and purified by gel column chromatography to obtain **2c** (yield: 0.91 g, 86%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.85 – 8.71 (m, 4H), 4.55 (t, J = 7.5 Hz, 2H), 4.24 – 4.13 (m, 2H), 2.86 (t, J = 7.0 Hz, 2H), 1.91 – 1.78 (m, 9 H), 0.94 (t, J = 5.6 Hz, 42 H), 0.76 – 0.67 (m, 2H), 0.59 (t, J = 5.8 Hz, 14H) (Figure S29).

3c: 2c (120 mg, 0.1 mmol), Tris (36 mg, 0.3 mmol) and EEDQ (74 mg, 0.3 mmol) were dissolved in 10 mL DMF and 10 mL $CHCl_3$, and the reaction mixture was stirred for 12 h at 100 °C. After cooling to room temperature, the mixture was concentrated under reduced pressure and purified by gel column chromatography to give **3c** (yield: 130 mg, 90 %). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.81 – 8.73 (m, 4H), 6.74 (br, 1H), 4.57 (t, J = 7.44 Hz, 2H), 4.23 – 4.16 (m, 2H), 3.70 (d, J = 5.5 Hz, 6H), 2.78 – 2.70 (m, 2H), 1.92 – 1.77 (m, 9H), 0.94 (dd, J = 6.5, 3.6 Hz, 42H), 0.76 – 0.68 (m, 2H), 0.59 (dd, J = 8.4, 4.4 Hz, 14H) (Figure S30).

4c: 3c (100 mg, 0.077 mmol) and Wells-Dawson POM (348 mg, 0.064 mmol) were dissolved in 5 mL DMF, the reaction mixture was stirred for 7 days at 80 °C. After cooling to room temperature, the mixture was concentrated under reduced pressure. The

resulting residue was dissolved in 5 mL acetonitrile, and was added dropwise into stirred diethylether (50 mL) to precipitate the product and remove the excess 3c, this procedure was repeated twice. The resulting solid was washed with 20 mL diethylether and dried under vacuum at 45 °C for 12 h to give **4c** (yield: 125 mg, 34 %). ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.70 – 8.67 (m, 4H), 7.67 (br, 1H), 5.48 (d, J = 6.9 Hz, 6H), 4.33 – 4.30 (m, 2H), 4.07 – 4.05 (m, 2H), 3.22-3.10 (m, 48H), 2.90 – 2.88 (m, 2H), 1.85 – 1.73 (m, 9H), 1.65 – 1.50 (m, 48H), 1.32 (dd, J = 14.5, 7.5 Hz, 48H), 1.00 – 0.87 (m, 114H), 0.71 – 0.64 (m, 2H), 0.57 (d, J = 6.6 Hz, 14H) (Figure S31). ^{13}C NMR (101 MHz, DMSO-d_6): δ 130.45, 130.33, 128.73, 128.45, 126.91, 126.45, 85.83, 57.50, 54.61, 34.62, 25.30, 25.26, 23.43, 23.40, 23.07, 21.92, 21.79, 19.20, 13.51, 9.07 (Figure S32). MS (MALDI-TOF, m/z): Calculated for $[M–TBA+2H]^+$: 6426.60 Da, found: 6426.58 Da. Calculated for $[M+H]^+$: 6668.07 Da, found: 6668.17 Da. Calculated for $[M+Na+K-H]^+$: 6728.16 Da, found: 6728.08 Da. Calculated for $[M+TBA]^+$: 6909.54 Da, found: 6909.89 Da. Calculated for $[M+TBA+2K–2H]^+$: 6985.74 Da, found: 6985.73 Da. Calculated for $[M+2TBA–2K–Na+2H]^+$: 7052.82 Da, found: 7052.70 Da. Calculated for $[M+2TBA–H]^+$: 7151.01 Da, found: 7151.30 Da (Figure S33).

S4. Self-Assembly Process

Method 1. Helical ribbons and tubes: An acetone solution at a concentration of 1.0 mg/mL was first prepared. The self-assembly of the heterocluster is implemented by adding 800 μL *n*-hexane into 200 μL acetone solution, and then annealing the suspension with a concentration of 0.2 mg/mL in a 50 °C oven for 7 days.

Method 2. Colloidal onions: A mixture of acetone and deionized water with a ratio of 1/2 (v/v) and the acetone solution of the heteroclusters with a concentration of 10.0 mg/mL were preheated to 50 °C, respectively. Then the hot mixture was slowly added into the hot acetone solutions until the acetone/water ratio is equal to 2/3 (v/v) and the concentration of 1.0 mg/mL. Finally, the suspension was annealed at 50 °C for 3 days.

Method 3. Cubosomes： According to the same mothed mentioned above, a 0.2 mg/mL solution in a 3/2 (v/v) acetone/*n*-decane mixture was prepared. Then, the self-assembly of the heterocluster is implemented by slow evaporation of acetone at 25 °C for about 12 h.

Method 4. Hexosomes: 460 μL water was injected to 140 μL acetonitrile of the heteroclusters via a syringe pump at 10 μL/min under mild stirring. The mixture became turbid during the injection period. After the injection process was complete, the dispersion was stirred for 30 minutes.

S5. Sample Preparations

The TEM samples were prepared by dropping a 10 μL turbid solution after self-assembly on carbon film-coated copper grids. The suspension was allowed to sit on the TEM grid for 30 seconds, and then the residual liquid was wiped off with filter paper. Finally, the samples were dried under vacuum for about 1 h.

The suspensions were used directly as samples of the UV-vis and CD tests.

The turbid solution was centrifuged, and the residue was dried under vacuum for 2 h to give the yellow power, which was used as samples of the SEM and SAXS characterizations.

S6. Supporting Table

Table S1. Peak positions of UV-vis and CD spectra and solvents used.

Samples	UV-vis (nm)	CD (nm)	Solvents (v/v)
POSS-NDI-*L*-Phe-POM	345.7, 364.1, 383.6		acetone
POSS-NDI-*D*-Phe-POM	346.3, 364.4, 383.9		acetone
POSS-NDI-*Ala*-POM	343.2, 361.3, 381.1		acetone
POSS-NDI-*L*-Phe-POM	342.8, 359.4, 379.4		acetonitrile
POSS-NDI-*D*-Phe-POM	342.0, 359.4, 379.2		acetonitrile
POSS-NDI-*Ala*-POM	341.7, 361.1, 380.7		acetonitrile
L-Tubes	351.4, 367.0, 386.2	369.7, 383.9	acetone/*n*-decane (2/8)
D-Tubes	351.7, 366.1, 385.9	368.9, 384.7	acetone/*n*-decane (2/8)
Ala-Tubes	352.7, 364.5, 385.1		acetone/*n*-decane (2/8)
L-Onions	350.4, 368.8, 388.2	369.5, 390.6	acetone/water (2/3)
D-Onions	351.8, 369.0, 388.0	367.1, 389.7	acetone/water (2/3)
Ala-Onions	348.1, 364.6, 382.2		acetone/water (2/3)
L-Cubosomes	346.4, 365.6, 385.6	369.2, 384.8	*n*-decane
D-Cubosomes	345.9, 366.2, 386.0	368.3, 383.6	*n*-decane
Ala-Cubosomes	344.9, 363.5, 383.1		*n*-decane
L-Hexosomes	345.0, 365.0, 383.8	366.5, 386.1	acetonitrile/water (14/46)
D-Hexosomes	345.8, 364.6, 383.6	367.0, 386.9	acetonitrile/water (14/46)
*Al*a-Hexosomes	343.6, 364.2, 382.8		acetonitrile/water (14/46)

S7. Supporting Figures

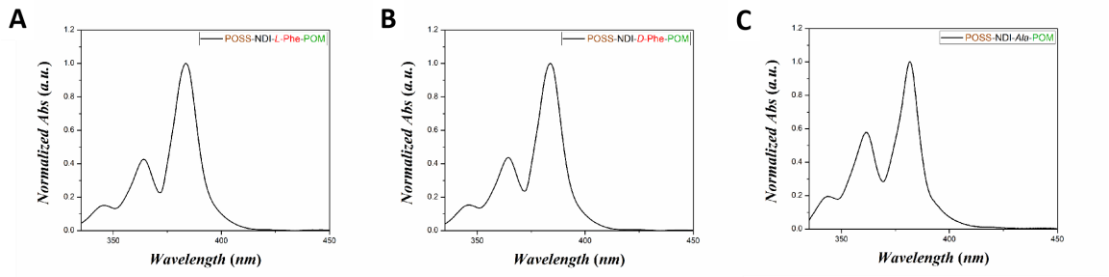


Figure S1. UV-vis absorption spectra of (A) POSS-NDI-*L*-Phe-POM, (B) POSS-NDI-*D*-Phe-POM, (C) POSS-NDI-*Ala*-POM (c = 0.1 mg/mL in acetone).

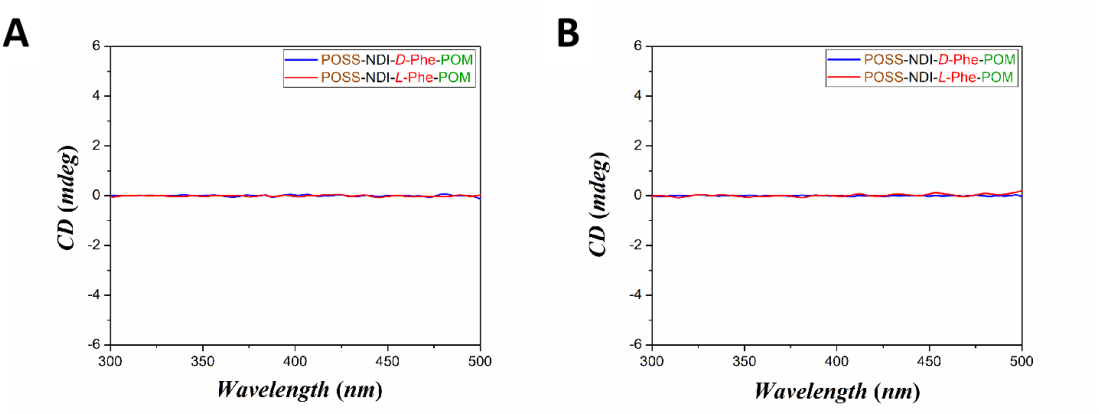


Figure S2. CD spectra of the (A) acetone (c = 0.1 mg/mL) and (B) acetonitrile (c = 0.1 mg/mL) solutions of the chiral heteroclusters.

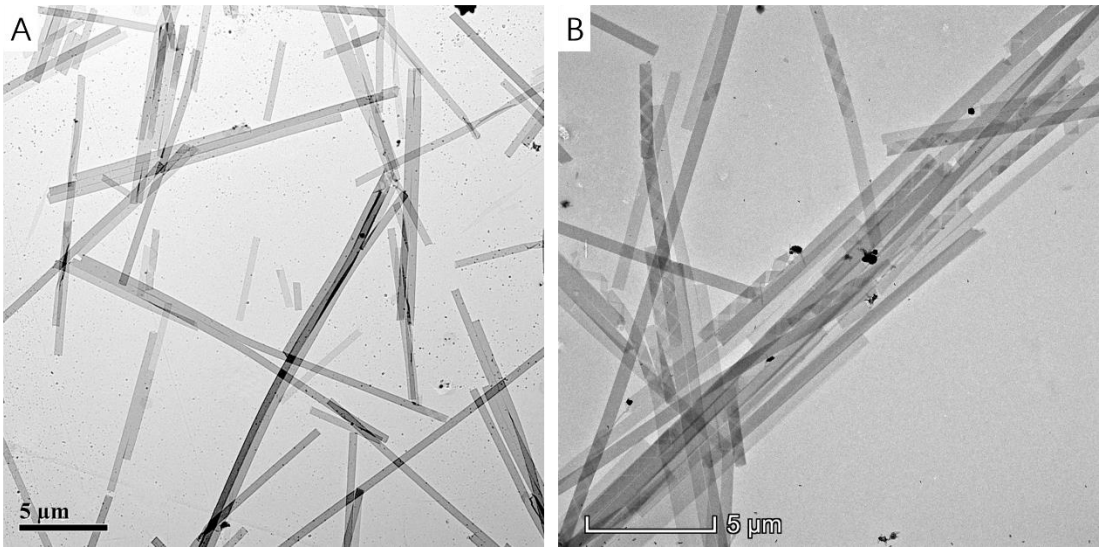
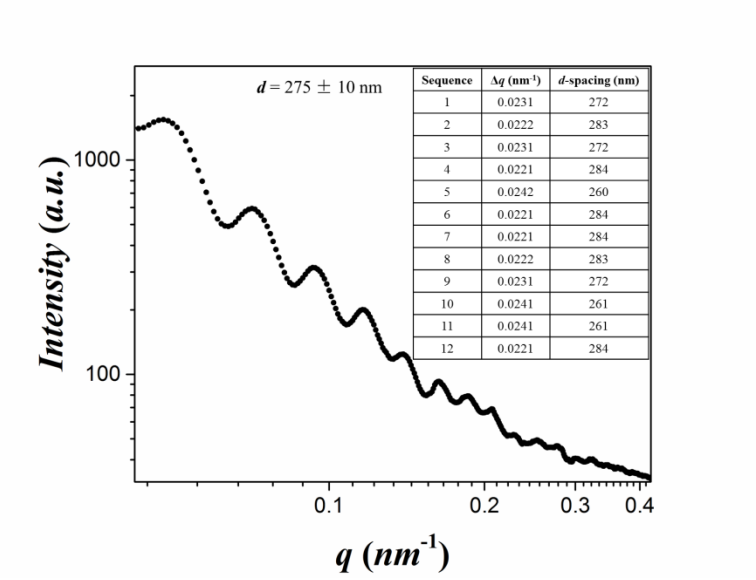


Figure S3. TEM images of (A) *L*-NDI- and (B) *Ala*-NDI-helical ribbons and tubes.



Sequence	Δq (nm⁻¹)	d-spacing (nm)
1	0.0231	272
2	0.0222	283
3	0.0231	272
4	0.0221	284
5	0.0242	260
6	0.0221	284
7	0.0221	284
8	0.0222	283
9	0.0231	272
10	0.0241	261
11	0.0241	261
12	0.0221	284

Figure S4. SAXS pattern of *L*-NDI-tubes (*c* = 0.2 mg/mL in acetone/*n*-decane = 2/8, v/v).

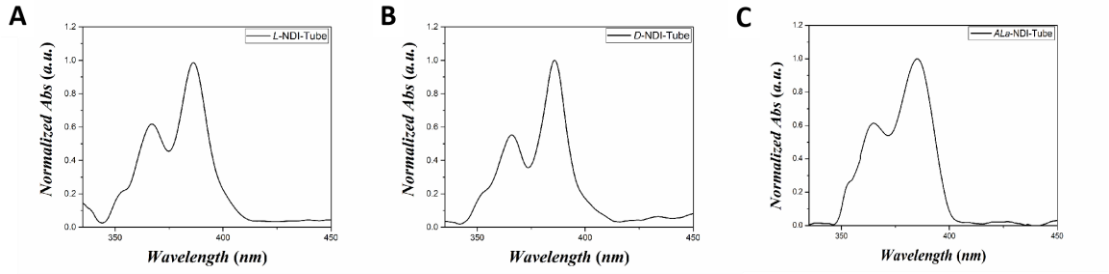


Figure S5. UV-vis absorption spectra of (A) *L*-NDI-, (B) *D*-NDI- and (C) *Ala*-NDI-helical ribbons and tubes (*c* = 0.2 mg/mL in acetone/*n*-decane = 2/8, v/v).

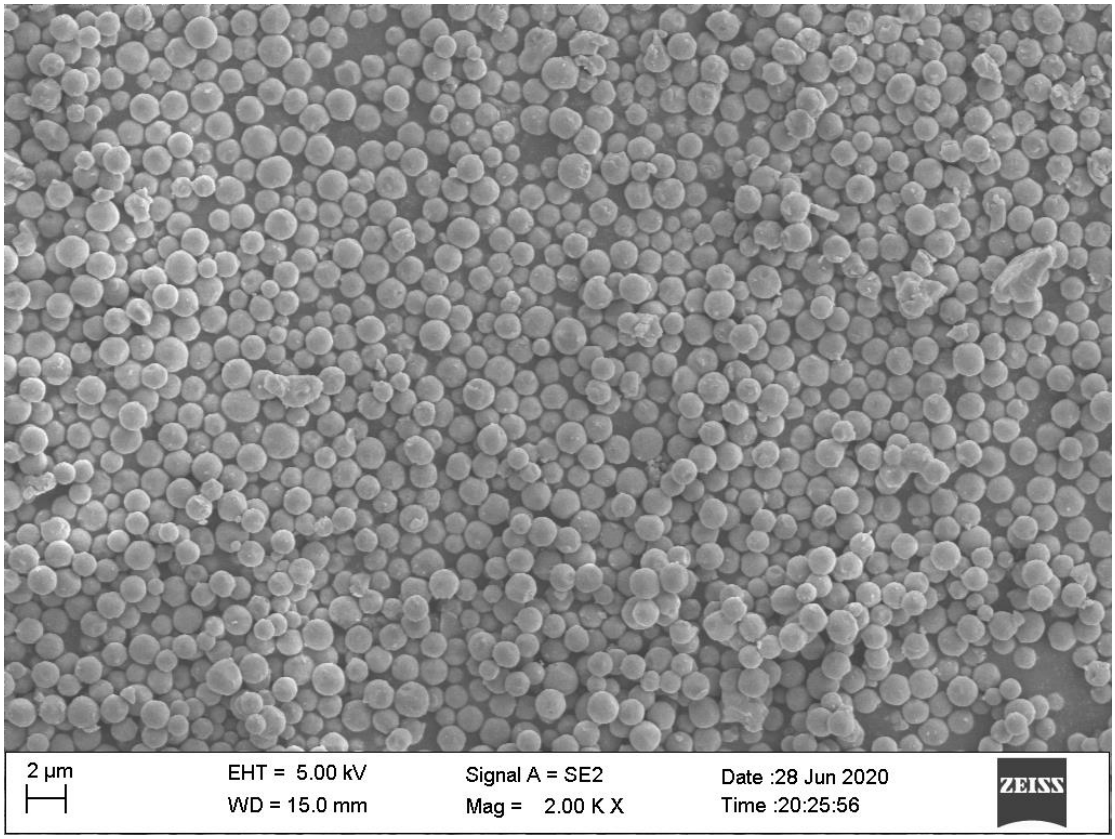


Figure S6. SEM image of *L*-NDI-cubosomes.

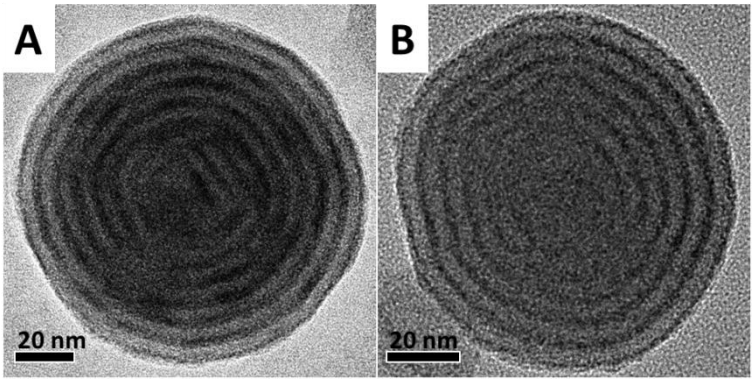


Figure S7. TEM images of (A) *D*-NDI-onions and (B) *Ala*-NDI-onions.

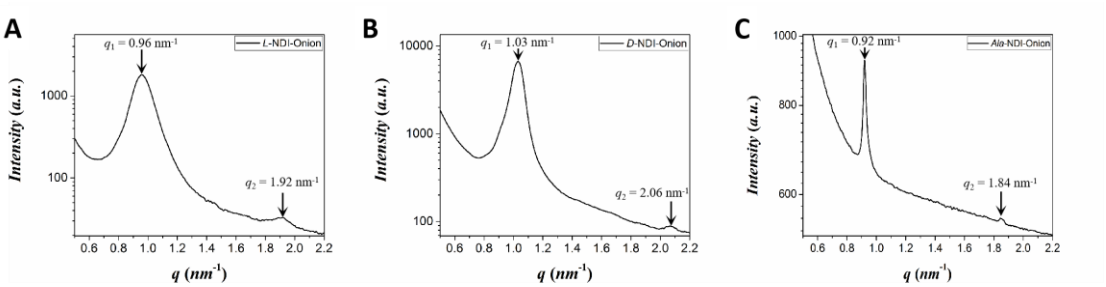


Figure S8. SAXS patterns of (A) *L*-NDI-, (B) *D*-NDI and (C) *Ala*-NDI-Onions (*c* = 1.0 mg/mL in acetone/water = 2/3, v/v).

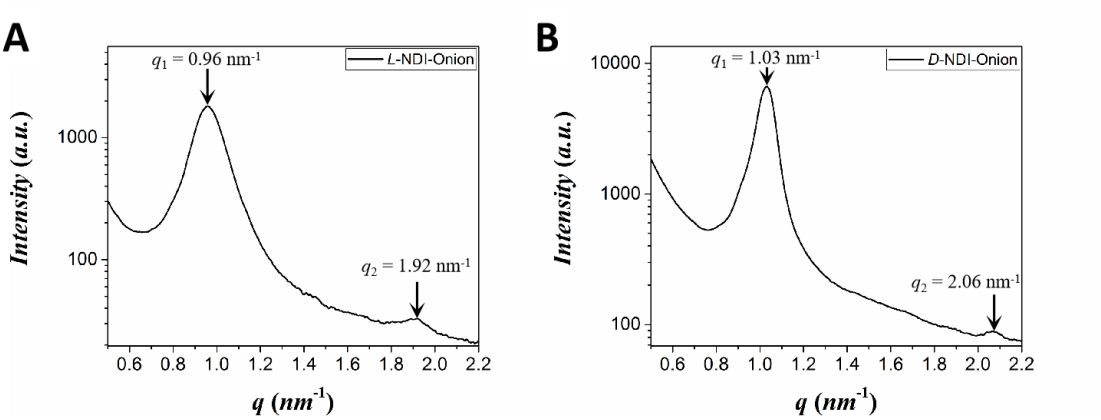


Figure S9. UV-vis absorption spectra of *L*-NDI-onions (A) and *D*-NDI-onions (B) (*c* = 1.0 mg/mL in acetone/water = 2/3, v/v).

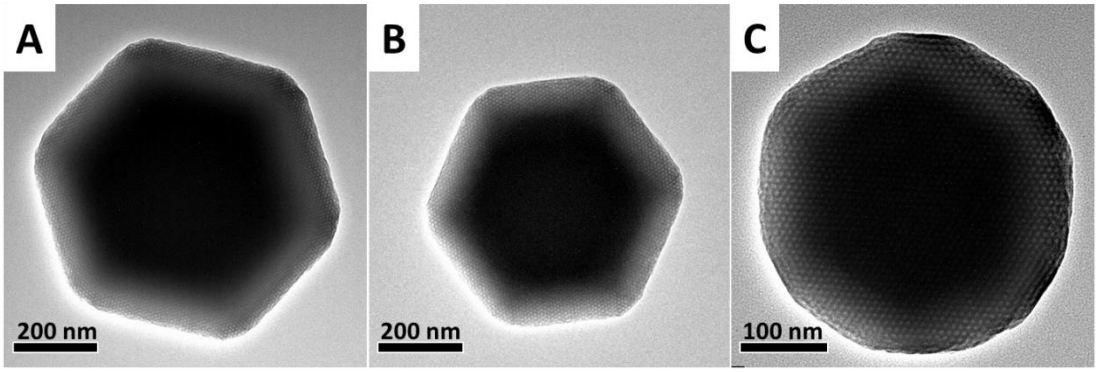


Figure S10. TEM images of cubosomes with the double diamond structure. (A) *L*-NDI-cubosomes, (B) *D*-NDI-cubosomes, and (C) *Ala*-NDI-cubosomes.

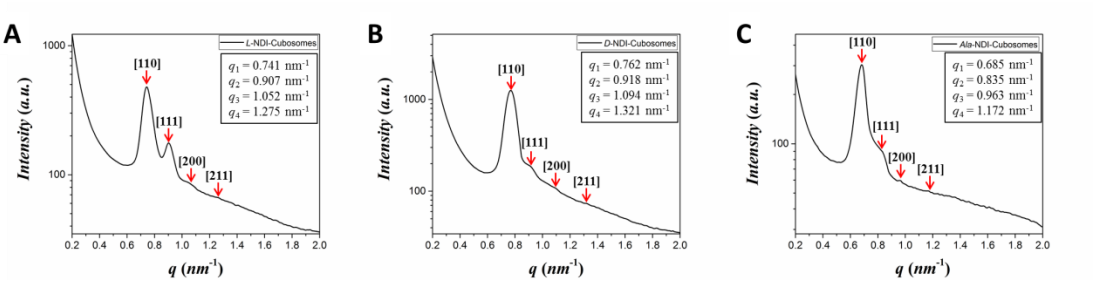


Figure S11. SAXS patterns of (A) *L*-NDI-cubosomes, (B) *D*-NDI-cubosomes and (C) *Ala*-NDI-cubosomes (*c* = 0.5 mg/mL in *n*-decane).

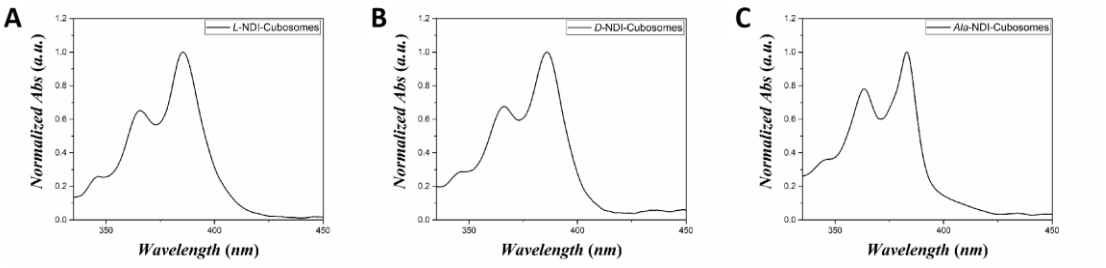


Figure S12. UV-vis absorption spectra of (A) *L*-NDI-, (B) *D*-NDI- and (C) *Ala*-NDI-Cubosomes (*c* = 0.5 mg/mL in *n*-decane).

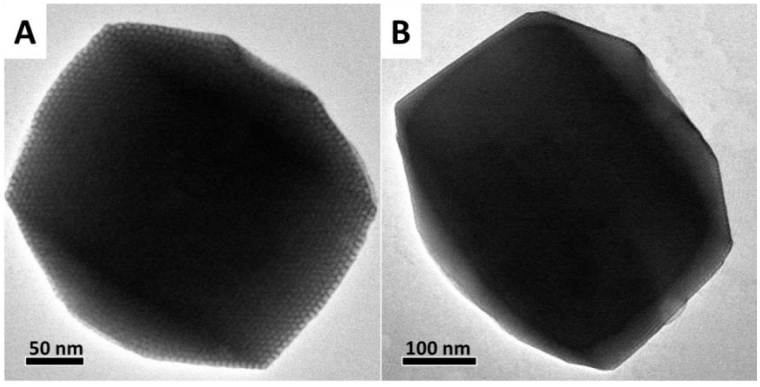


Figure S13. TEM images of (A) *D*-NDI-hexosomes and (B) *Ala*-NDI-hexosomes.

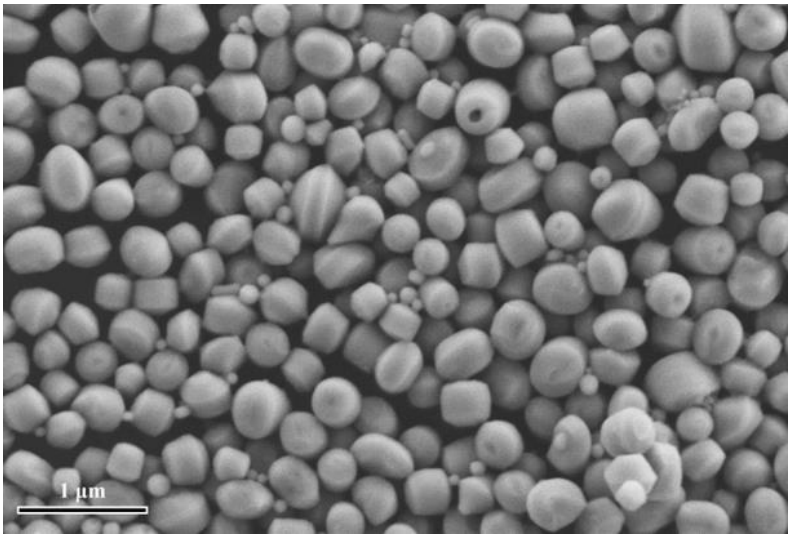


Figure S14. SEM images of hexosomes.

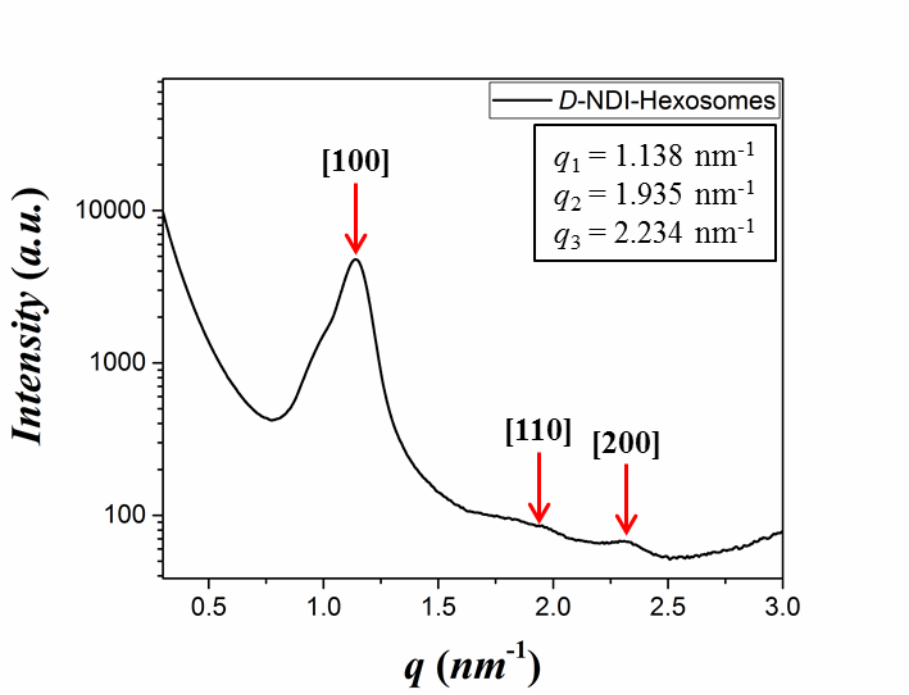


Figure S15. SAXS patterns of *D*-NDI-Hexosomes (*c* = 0.47 mg/mL in acetonitrile/water = 14/46, v/v).

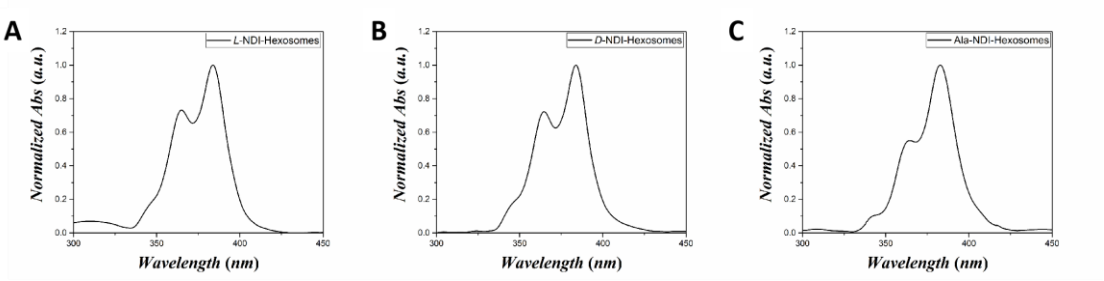


Figure S16. UV-vis absorption spectra of (A) *L*-NDI-, (B) *D*-NDI- and (C) *Ala*-NDI-Hexosomes (c = 0.47 mg/mL in acetonitrile/water = 14/46, v/v).

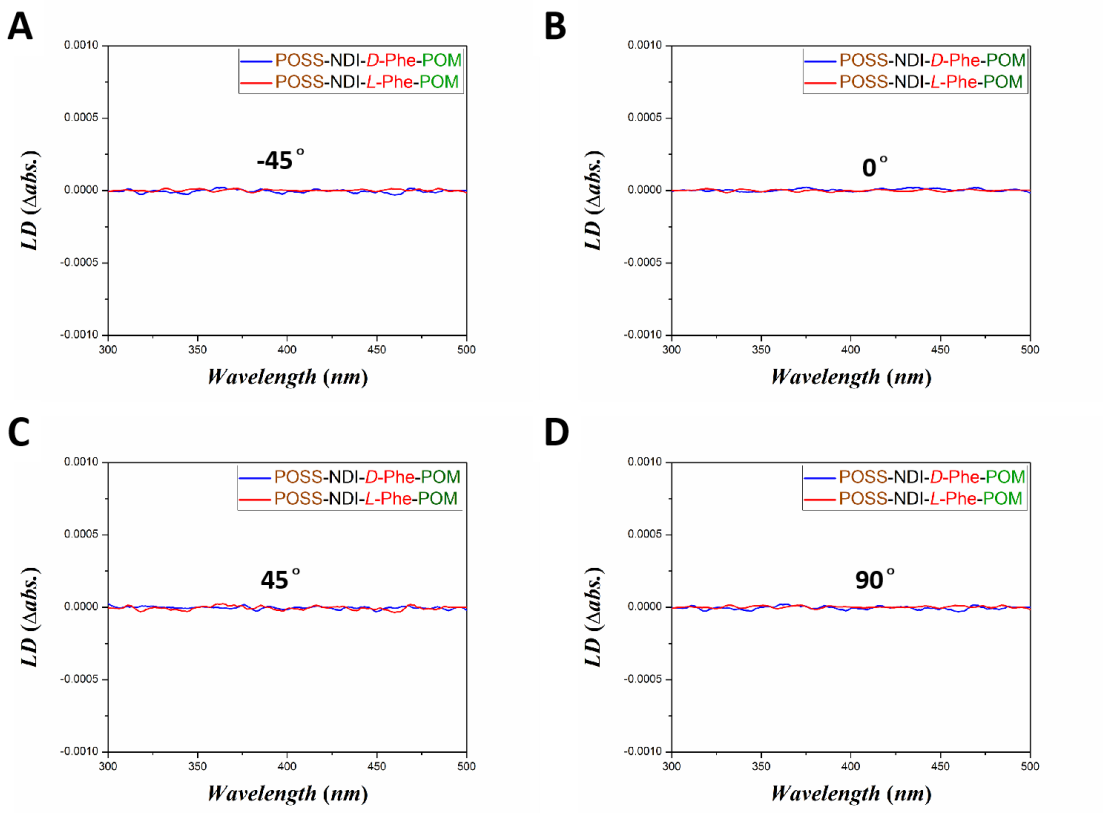


Figure S17. LD spectra of the chiral cubosomes monitored under various angles, (A) −45°, (B) 0°, (C) 45° and (D) 90° (c = 0.5 mg/mL in *n*-decane).

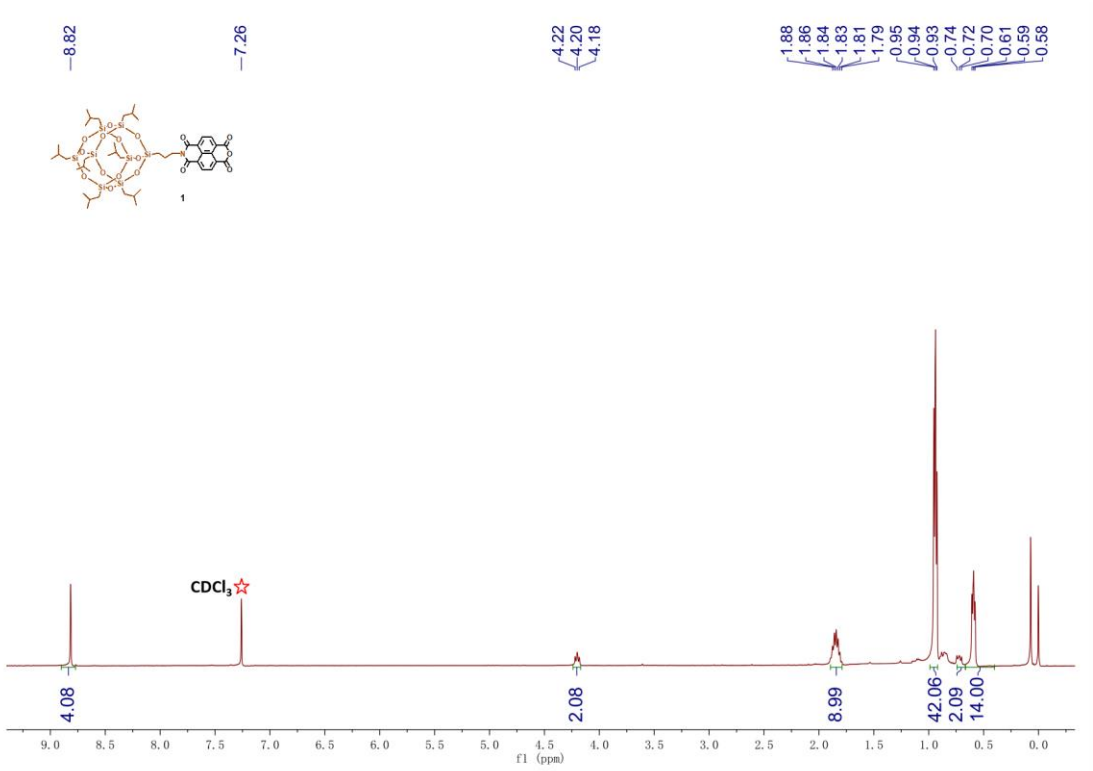


Figure S18. ^{1}H NMR spectrum of **1**.

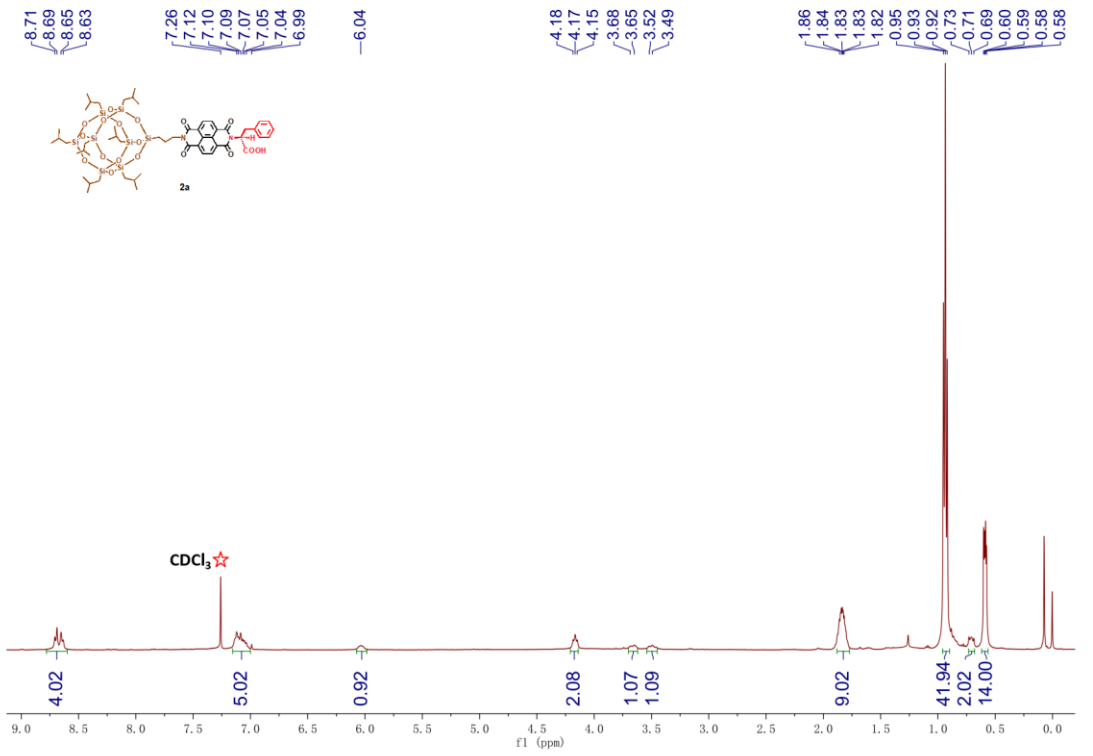


Figure S19. ^{1}H NMR spectrum of **2a**.

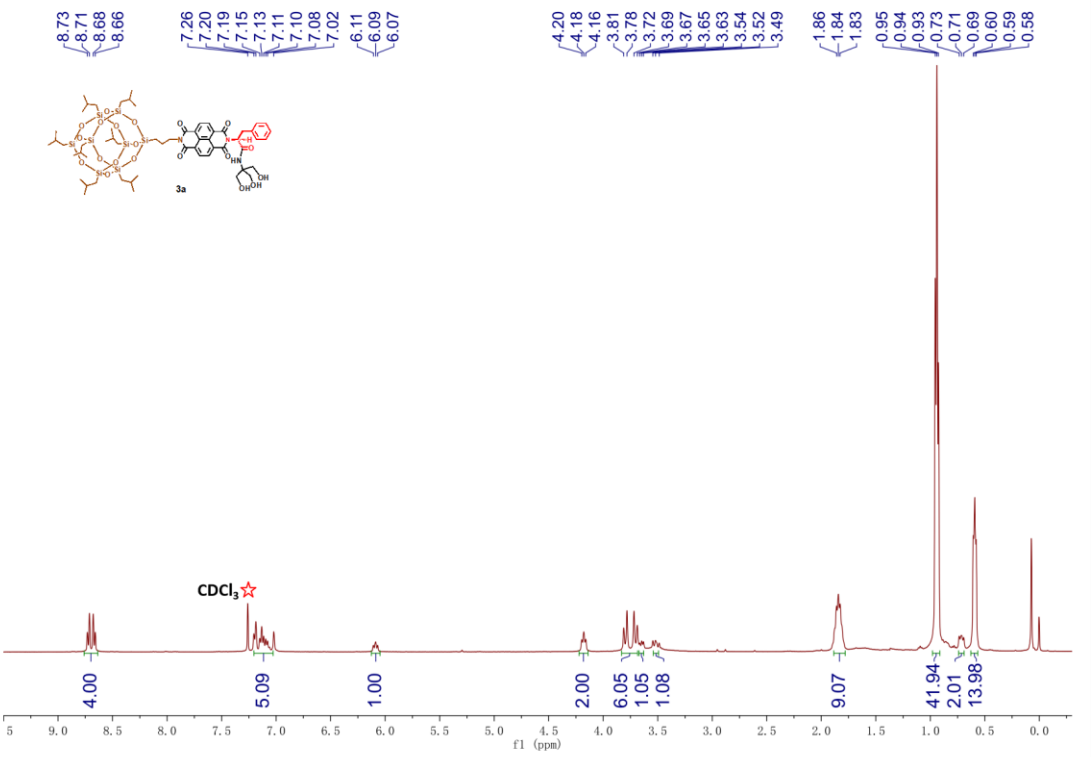


Figure S20. ^{1}H NMR spectrum of **3a**.

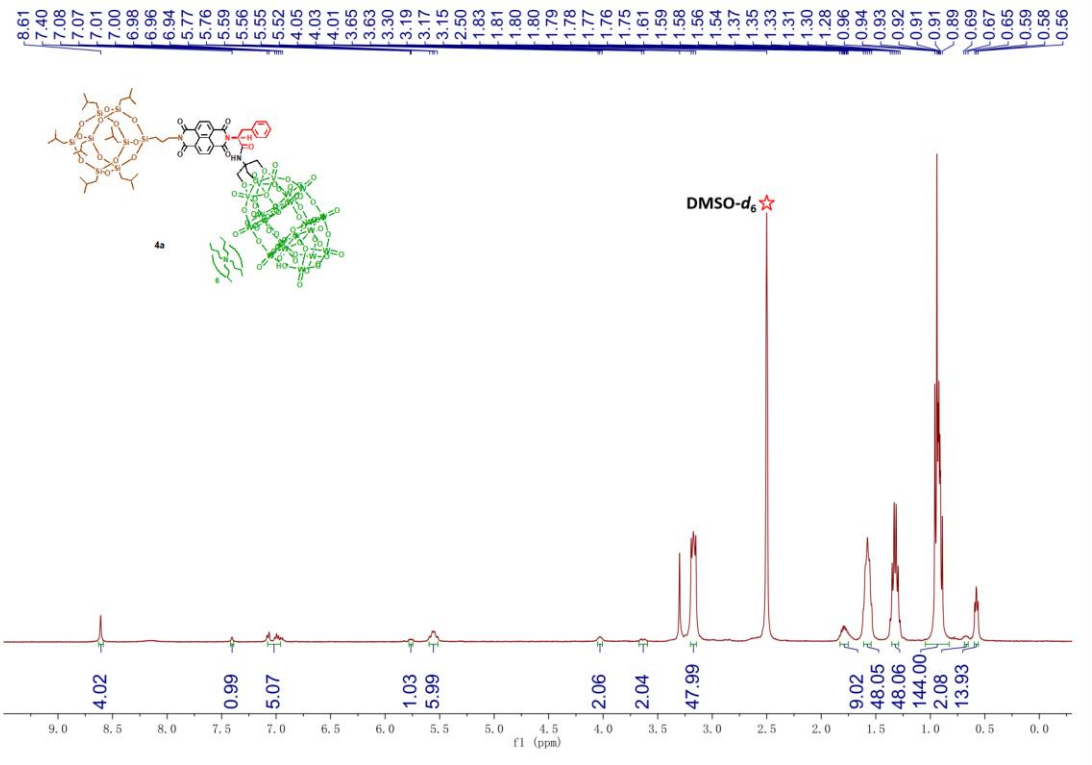


Figure S21. ^{1}H NMR spectrum of **4a**.

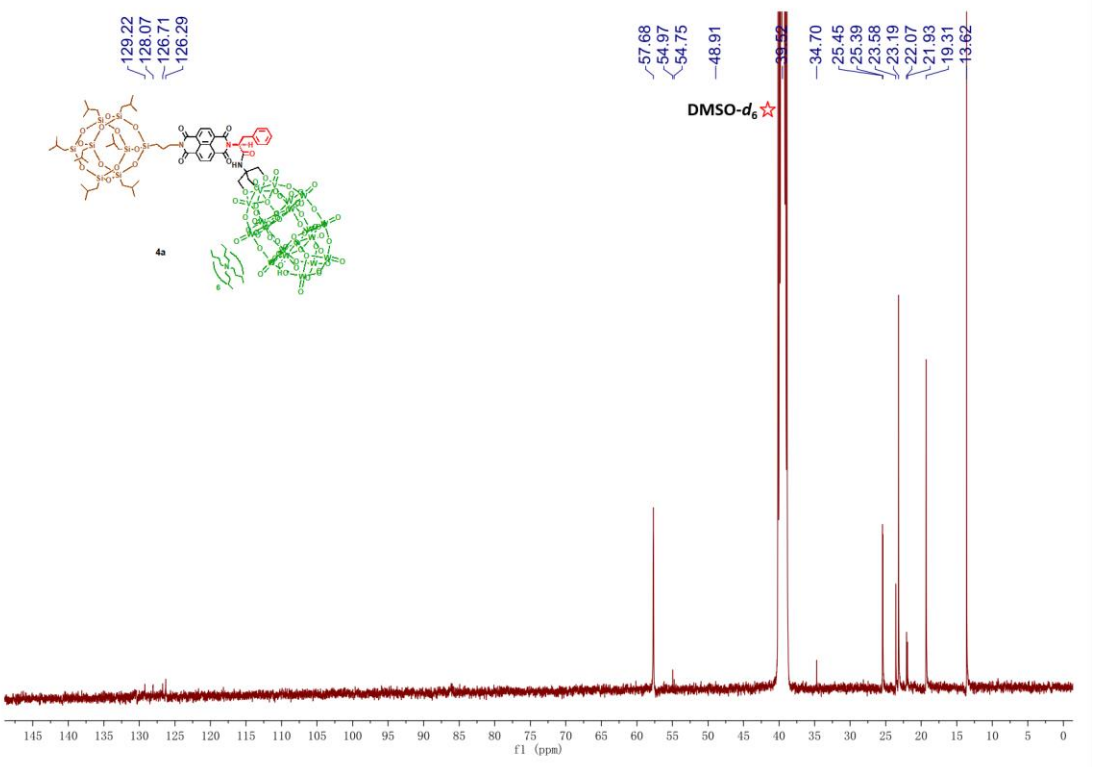


Figure S22. ^{13}C NMR spectrum of **4a**.

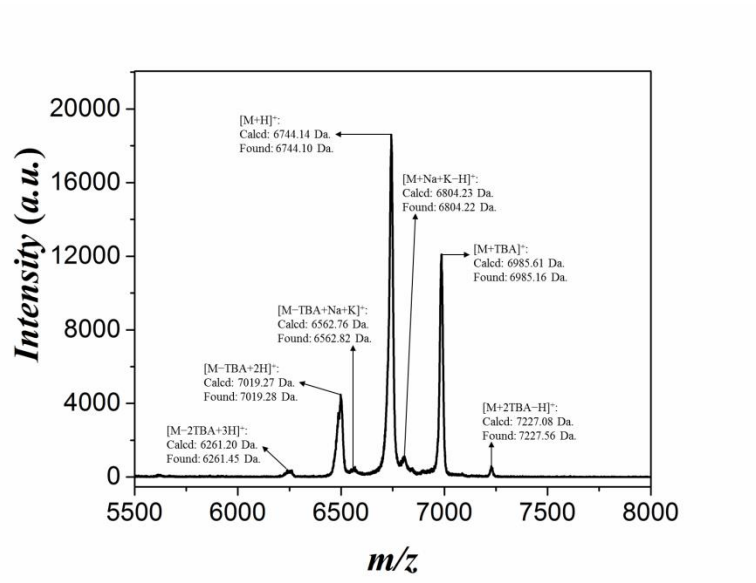


Figure S23. MALDI-TOF mass spectrum of POSS-NDI-*L*-Phe-POM (**4a**).

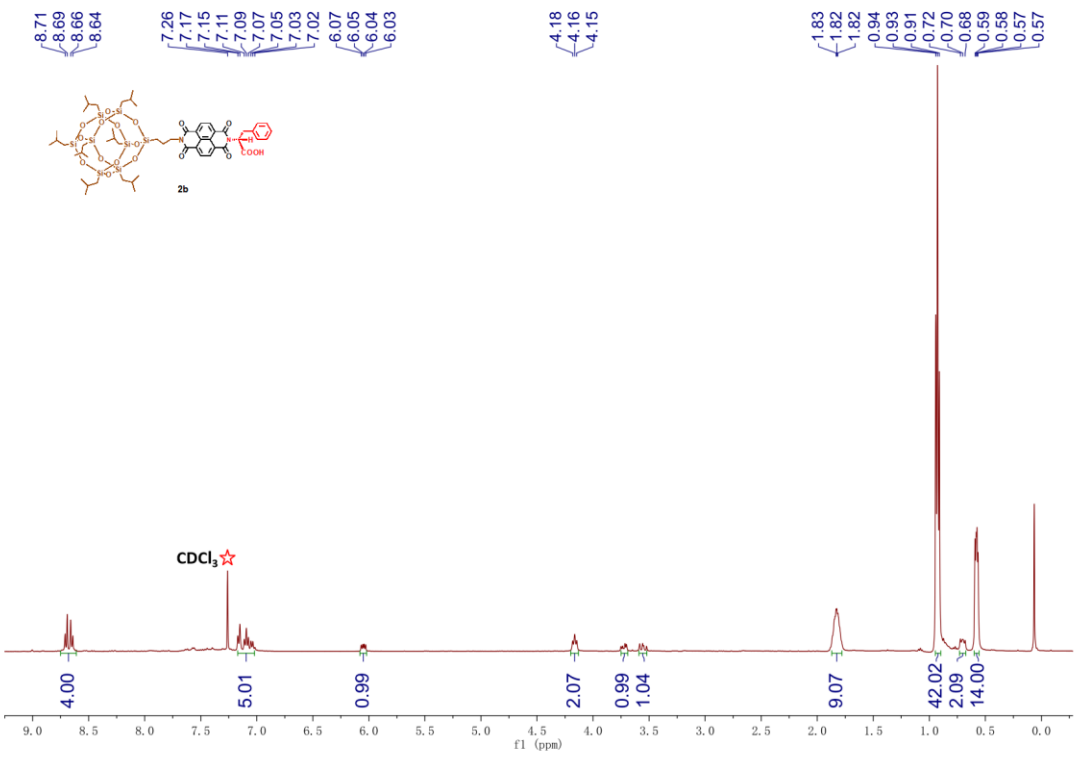


Figure S24. ^{1}H NMR spectrum of **2b**.

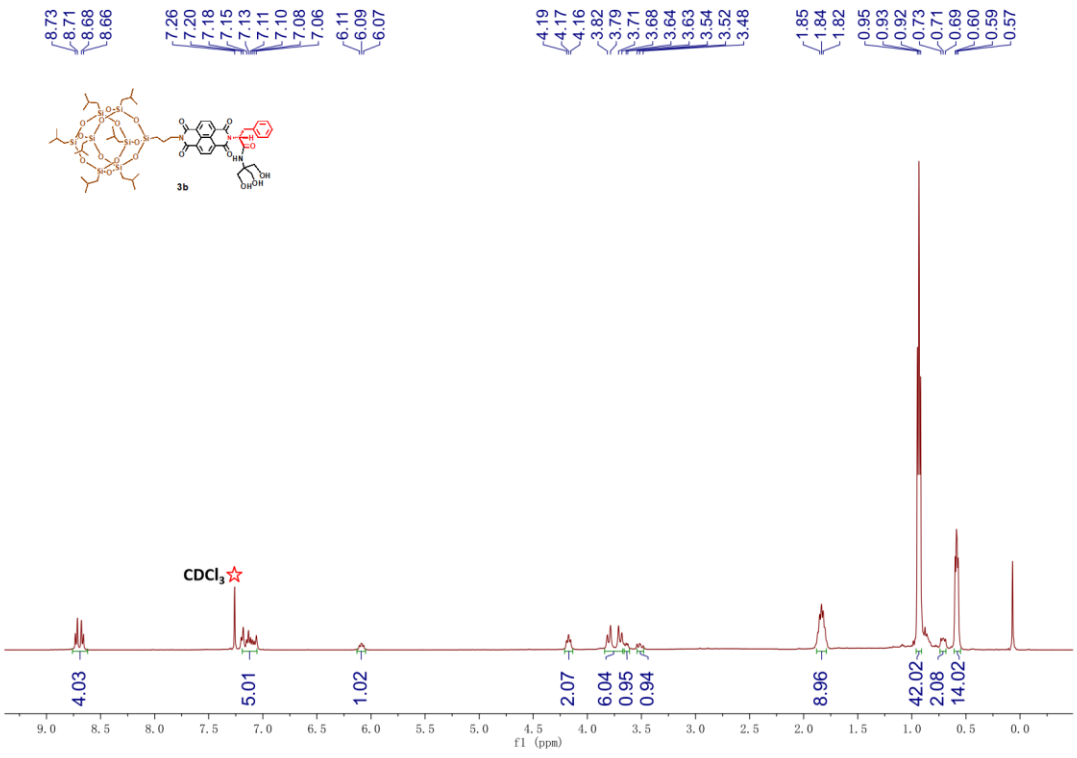


Figure S25. ^{1}H NMR spectrum of **3b**.

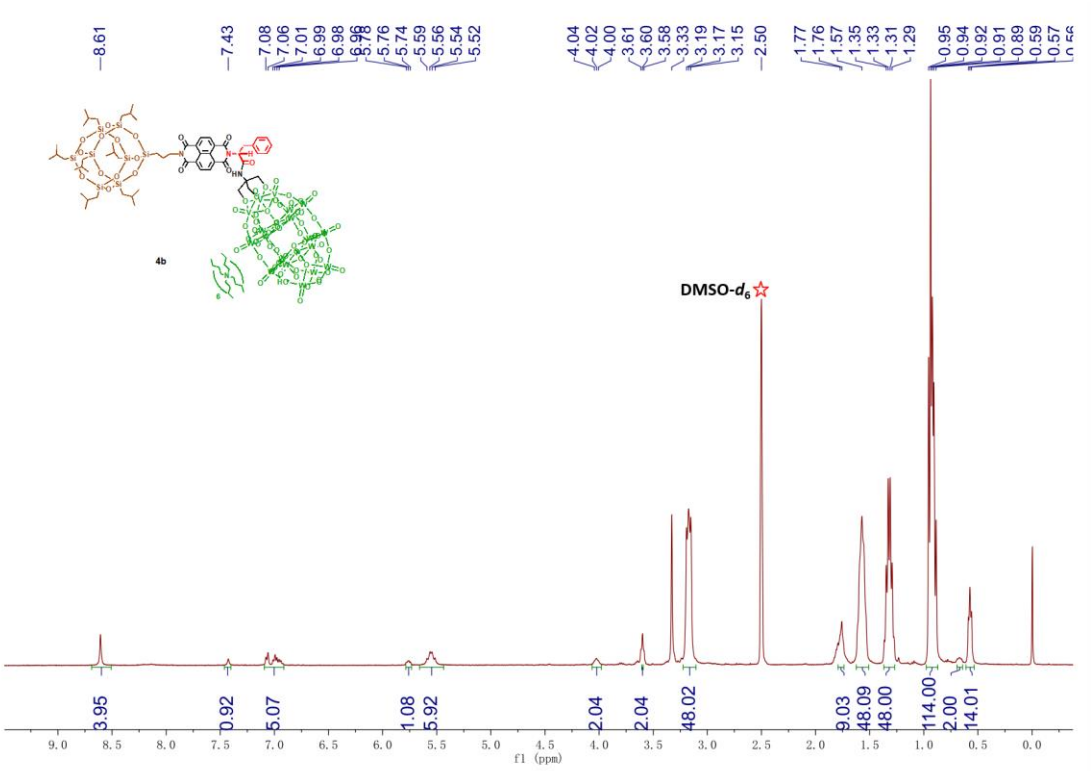


Figure S26. ^{1}H NMR spectrum of **4b**.

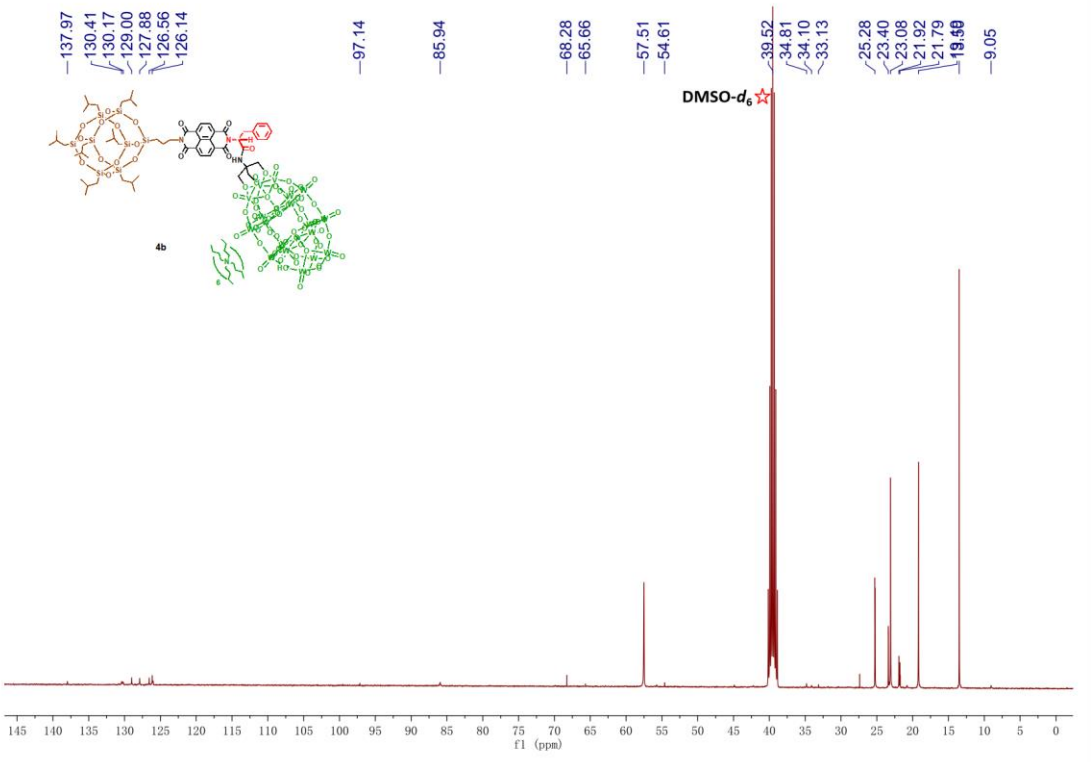


Figure S27. ^{13}C NMR spectrum of **4b**.

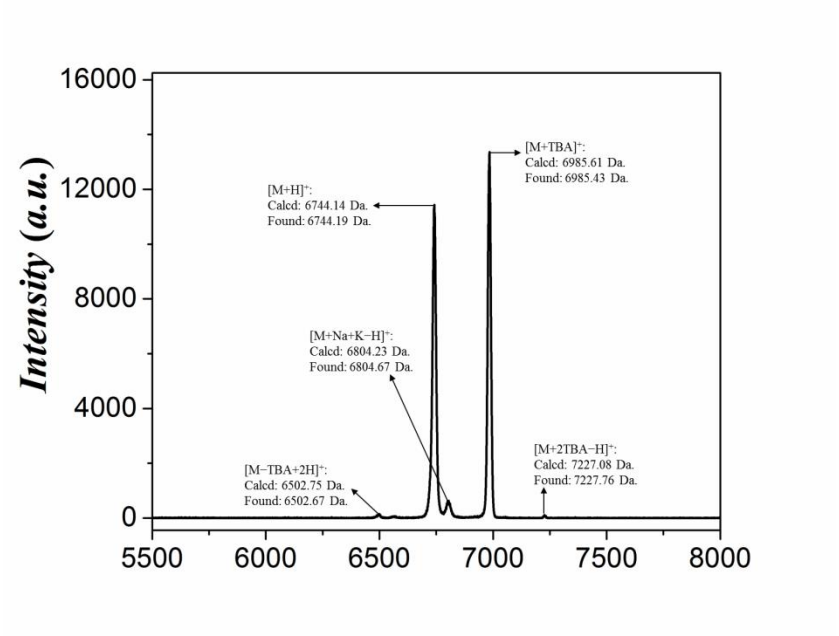


Figure S28. MALDI-TOF mass spectrum of POSS-NDI-*D*-Phe-POM (**4b**).

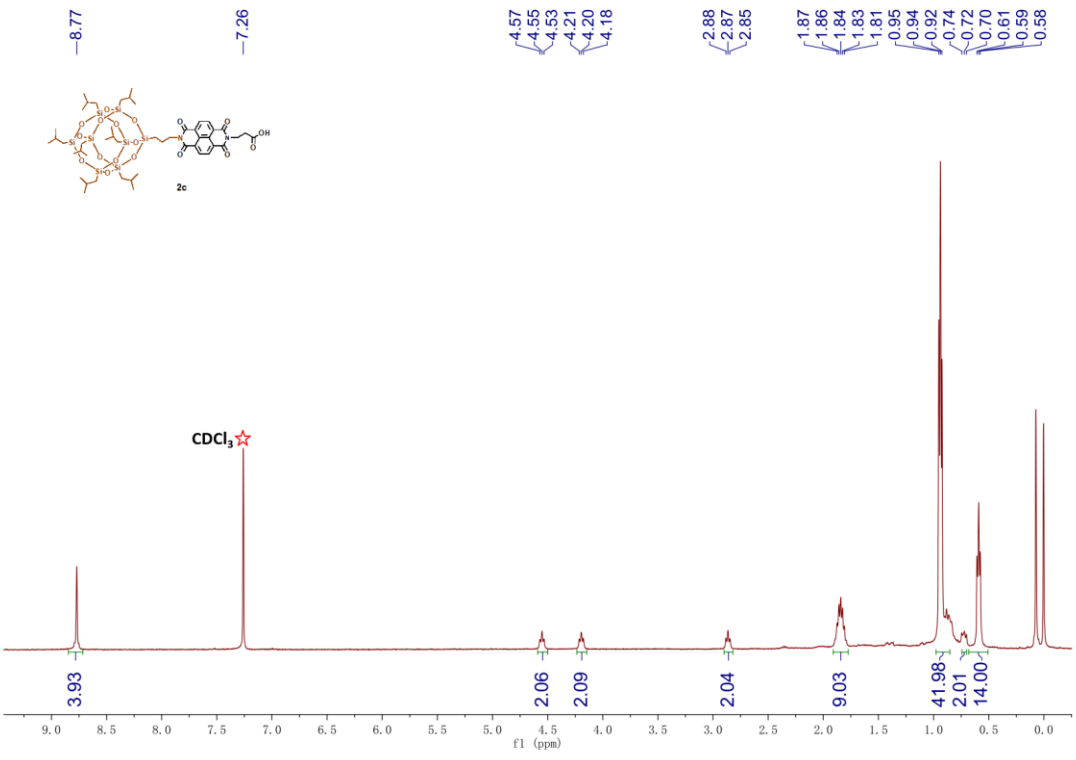


Figure S29. ^{1}H NMR spectrum of **2c**.

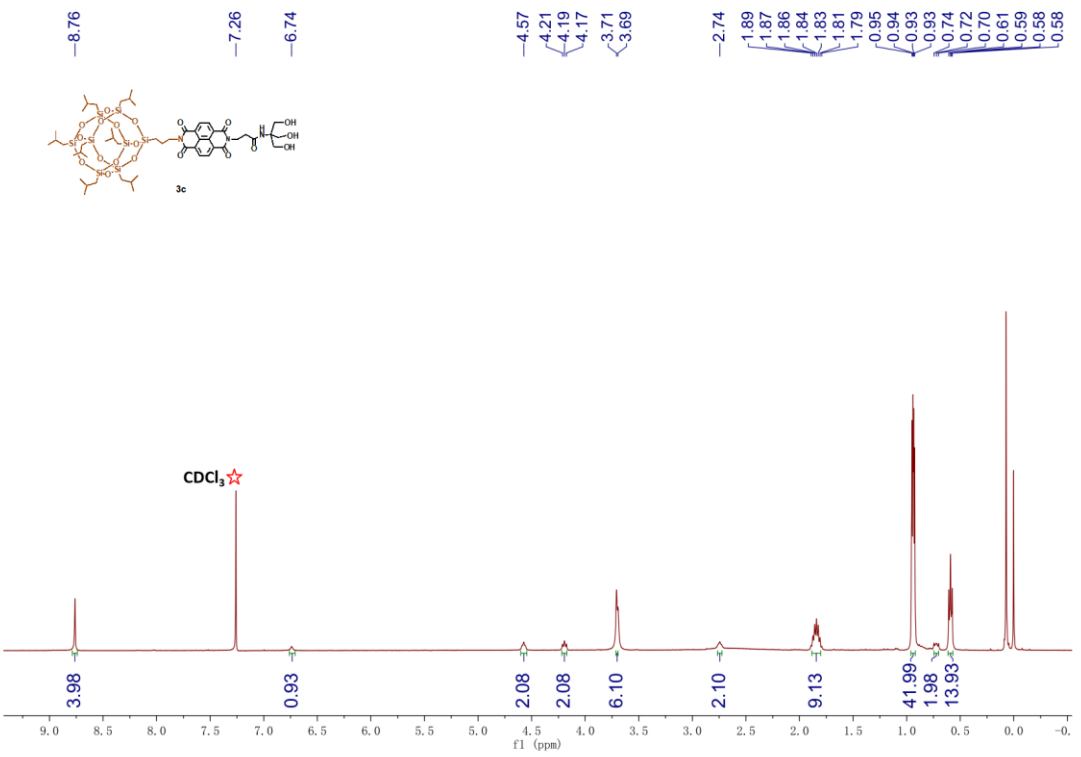


Figure S30. ^{1}H NMR spectrum of **3c**.

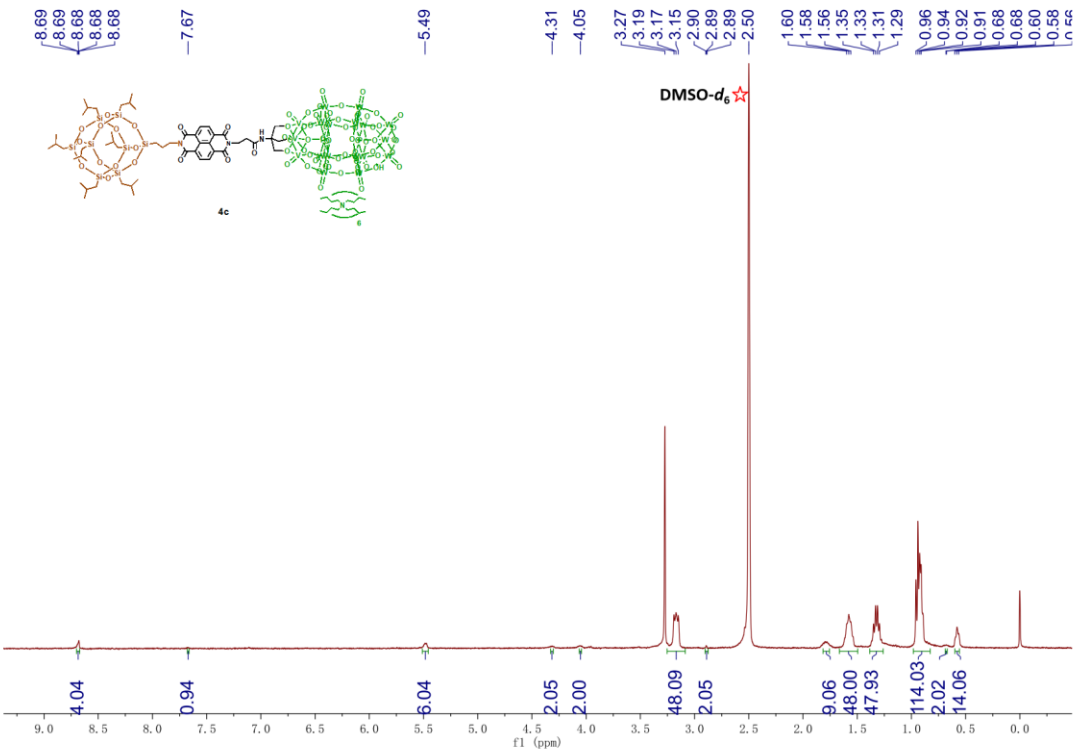


Figure S31. ^{1}H NMR spectrum of **4c**.

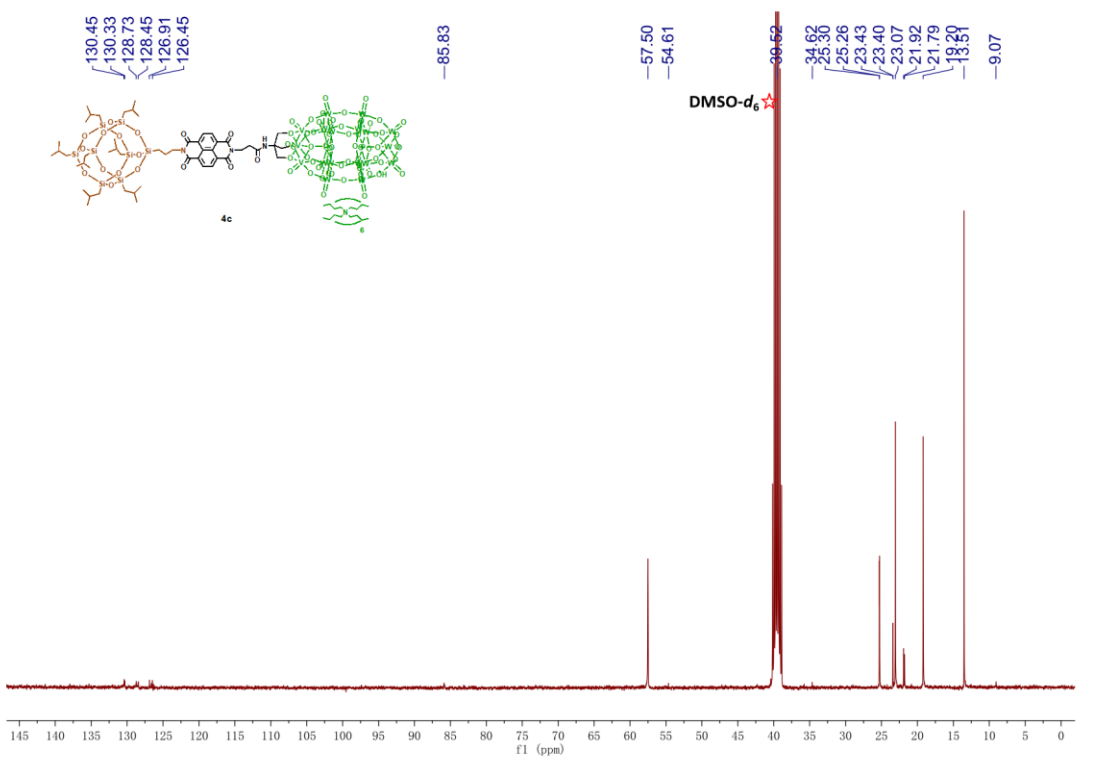


Figure S32. ^{13}C NMR spectrum of **4c**.

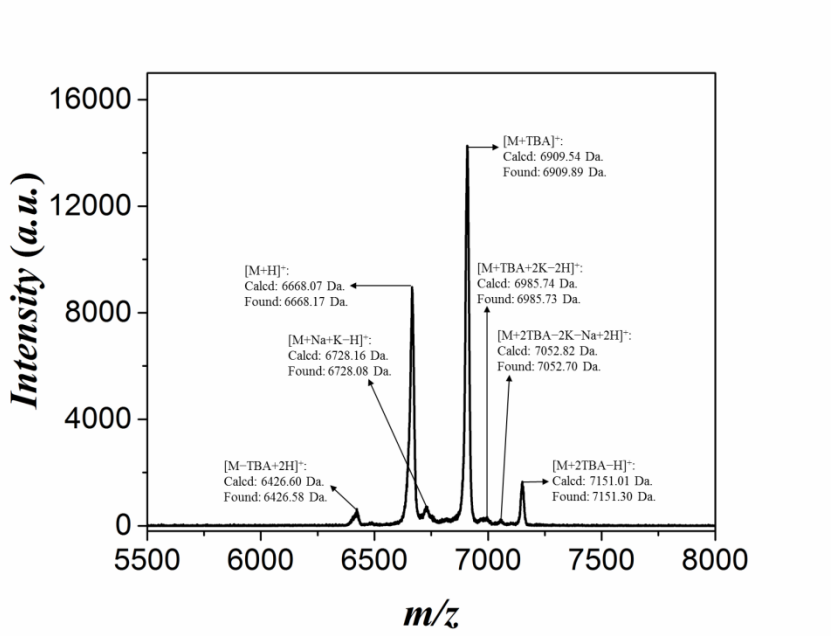


Figure S33. MALDI-TOF mass spectrum of POSS-NDI-*Ala*-POM (**4c**).

S8. References

1. G. Fide, B. Rapko, R. J. Saxton, P. J. Domaille, *J. Am. Chem. Soc.* **1986**, *108*, 2947–2960.