

Supporting Information

Macroscopic Helices Co-Assembled from Chirality-Transferring Temperature-Responsive Carbohydrate-Based Bolaamphiphiles and 1,4-Benzenediboronic Acid

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1. General Remarks

1.1 Materials

All starting materials were obtained from commercial suppliers and were used without further purification. D-(+)-Glucose, acetic anhydride, sodium bicarbonate and 1,12-dibromododecane were obtained from VWR (Germany). D-(+)-Mannose, boron trifluoride-ethyl ether complex, iodine, sodium methylate, 7-hydroxy-4-methylcoumarin, 1,6-dibromohexane and potassium thioacetate were purchased from Sigma-Aldrich (USA). 2,2-Dimethoxy-2-phenylacetophenone (DMPA), benzene-1,4-diboronic acid (BDDBA), sodium sulfite and thioacetic acid were bought from TH. Geyer (Germany). The organic solvents including dichloromethane (DCM), ethyl acetate and hexane were obtained from TH. Geyer (Germany). Deionized water (DI water) was purified on a Millipore system.

1.2 Characterizations

FTIR spectroscopy

FTIR spectra were recorded on Alpha FTIR Spectrometer (Bruker, Germany) at room temperature. All samples were measured between 4000 and 400 cm^{-1} with a resolution of 4 cm^{-1} using Platinum ATR and accumulated 32 scans.

Electrospray mass spectrometry (ESI-MS)

The ESI-MS was recorded on Bruker Daltonik micrOTOF and maXis instruments. The samples were analyzed in positive ionization mode by direct perfusion in the ESI-MS interface (ESI capillary voltage = 4200 V, end plate offset voltage = -500 V). Samples were scanned from 50 to 1600 m/z. All systems are equipped with time-of-flight (TOF) analyzers.

Differential scanning calorimetry (DSC)

DSC scans were recorded on a NETZSCH DSC/200/F3 (NETZSCH, Germany) under a constant nitrogen flow (20 mL/min) with a heating/cooling rate of 5 K/min.

Dynamic Light Scattering Measurement (DLS)

The DLS measurements were performed on a Zetasizer Nano ZS (Malvern Instruments Ltd., UK) using 5 mW laser with the incident beam of 633 nm (He-Ne laser). For the size measurement (Z-average diameter), aqueous solutions of GCCG-12 and MCCM-12 at a concentration of 8 mg/mL DI water were filtered through 0.45 μm Millipore filters before each measurement. 1 mL of each sample was sealed inside a glass cuvette and the programmed temperature trend measurements were performed automatically from 25 $^{\circ}\text{C}$ to 90 $^{\circ}\text{C}$ and from 90 $^{\circ}\text{C}$ to 25 $^{\circ}\text{C}$, taking a measurement at every 5 $^{\circ}\text{C}$ after equilibrating for 2 min, once the measurement temperature was achieved. The average sizes from three repeated measurements were used as results.

Transmission electron microscopy (TEM)

TEM measurement was performed on a CM 12 Transmission Electron Microscope (Philips, Netherland). A droplet (total volume 7 μL) of GCCG-12/ MCCM-12 aqueous solution at a concentration of 2 mg/mL was placed onto a carbon-coated 300 mesh copper grids (Plano GmbH, Germany) that was previously glow-discharged with 40 mA electric current for 90 s to ensure hydrophilicity.

Scanning electron microscopy (SEM)

The SEM images were captured with an electron beam acceleration voltage of 5 kV on a LEO Supra-35 high-resolution field emission scanning electron microscope (Carl Zeiss AG, Germany). Before the measurement, a layer of carbon was coated on the surface of samples before SEM measurements.

Polarized light microscopy (PLM)

The PLM images were taken on Olympus BX51 with temperature control unit (CI94). All the samples were measured on glass slides with single concave and coverslip, and the temperature was controlled with a hot stage (Linkam LTS350).

Light microscopy with fluorescence

The images were recorded on ZEISS Axioplan 2 imaging microscope and the camera type of Nikon DS-Fi2. The samples were washed with DI water and put on glass slides before the observation.

Liquid-state nuclear magnetic resonance (NMR)

Nuclear magnetic resonance (NMR) spectroscopy was performed at 300 or 400 MHz (^1H -NMR) and 75 MHz (^{13}C -NMR, APT) on Bruker *Avance* III 300 and *Avance* III 400 instruments in the solvent indicated. Chemical shifts (δ) were given in parts per million (ppm) with respect to the residual solvent signal CDCl_3 containing 0.5 wt. % silver foil as stabilizer and 0.03% (v/v) TMS (^1H NMR: $\delta = 7.26$; ^{13}C NMR: $\delta = 77.00$) and D_2O (^1H NMR: $\delta = 4.79$). Peak multiplicities were reported as follows: s = singlet, d = doublet, t = triplet, dd = doublet of doublets, m = multiplet.

Solid-state MAS NMR spectroscopy

Solid-state ^{11}B magic angle spinning (MAS) NMR spectra were recorded on a Bruker AVIIIHD 600 UltraShield spectrometer with a field strength of 14.1 Tesla (corresponding to a proton Larmor frequency of 600 MHz), equipped with a 1.3 mm HXY probe. Samples were packed in 1.3 mm rotors and spun at the magic angle with 8 kHz. Temperature for data collection was set to 275 K. ^{11}B direct excitation spectra were acquired for 254 milliseconds with 100 kHz continuous wave proton decoupling at 4.6 ppm carrier frequency. ^{11}B excitation power was 35.7 kHz with a carrier frequency of 0 ppm. For sample containing brick-shaped structure formed by BDBA alone, 64 scans were accumulated with a recycle delay of 30 seconds; for samples containing helices formed by BDBA and GCCG-12 (MCCM-12), 3072 scans were accumulated with a recycle delay of 1 second. For sample (containing helices formed by BDBA and MCCM-12) after DSC 300 $^\circ\text{C}$ measurement, the recording conditions were changed to a recording time of 22

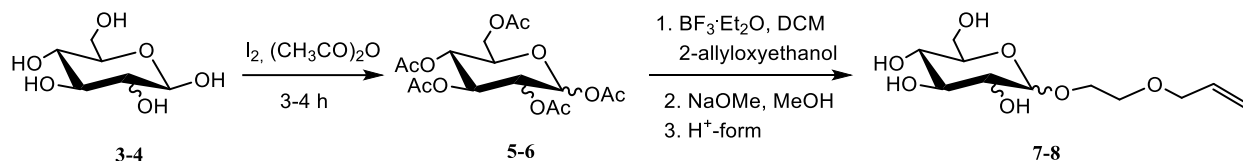
milliseconds and a recycle delay of 8 seconds, with 1024 scans; for samples (containing helices formed by GCCG-12 and BDBA, and brick-shaped structures formed by BDBA alone) after DSC 300 °C measurement, the recording conditions were changed to a recording time of 22 milliseconds and a recycle delay of 8 seconds, with 7168 scans.

¹³C MAS NMR spectra were recorded on a Bruker Ascend 600DNP spectrometer with a field strength of 14.1 Tesla, equipped with a 4 mm HXY probe. Samples were packed in 4 mm rotors and spun with 10 kHz. Temperature for data collection was set to 290 K. ¹³C detected spectra were recorded using cross polarization (CP) from ¹H. Initial ¹H excitation was 83 kHz at a carrier frequency of 4 ppm. The CP length was 3 milliseconds, with ¹H power of 57 kHz at 4 ppm carrier frequency and ¹³C power of 35 kHz at 110 ppm carrier frequency. A linear ramp from 90-100% power was applied on ¹H, making the average power during CP 54 kHz. Acquisition on ¹³C was 24.5 milliseconds with SPINAL64 heteronuclear decoupling of 83 kHz. The recycle delay was 15 seconds and 5120 scans were recorded. Spectra were processed with Bruker TopSpin 4.0.8 and the data were analyzed using OriginPro software (OriginLab, OriginPro 8.5G, USA). Exponential apodization of 100, 200 or 300 Hz was applied to all ¹¹B spectra (according to sensitivity considerations), and 200 Hz to all ¹³C spectra.

2. General Procedures

Synthesis of 2-allylethoxyl-D-monosaccharides

2-Allylethoxyl-D-monosaccharides were synthesized from commercially available D-glucose and D-mannose in 3 steps. The steps are described below (**Scheme S1**).



Scheme S1. Synthesis of 2-allylethoxyl-D-glucose (**7**) and 2-allylethoxyl-D-mannose (**8**).

General procedure for the synthesis of fully acetylated monosaccharides (**6-7**)

The fully acetylated monosaccharides were synthesized according to the literature.^[1] Catalytic amount of iodine (0.2 g) was added to a stirred mixture of acetic anhydride (30 mL) and monosaccharide (**3** glucose or **4** mannose) (5.0 g, 27.8 mmol). About 3-4 hours, the reaction mixture stirring at room temperature was completely dissolved. Then, the mixture was added to saturated Na_2SO_3 aqueous solution and saturated $NaHCO_3$ aqueous solution in ice-bath. The mixture was extracted with dichloromethane. At last, the solvent was evaporated under reduced pressure to give the pentaacetate **5** (**6**).

Penta-O-acetate-D-glucose, 5, ^1H NMR (300 MHz, CDCl_3 , δ in ppm): 2.01 (s, 3H), 2.02 (s, 3H), 2.04 (s, 3H), 2.09 (s, 3H), 2.18 (s, 3H), 4.06-4.14 (m, 2H), 4.27 (dd, 1H, $J = 3.0$ Hz, $J = 12.0$ Hz), 5.07-5.17 (m, 2H), 5.47 (t, 1H, $J = 9.0$ Hz), 6.33 (d, 1H, $J = 3.0$ Hz); ^{13}C NMR (75 MHz, CDCl_3 , δ in ppm): 20.39, 20.51, 20.61, 20.64, 20.82, 61.43, 67.87, 69.16, 69.79, 89.03, 168.69, 169.33, 169.59, 170.16, 170.56; HR-MS (ESI) m/z calcd for $\text{C}_{16}\text{H}_{22}\text{NaO}_{11}$ $[\text{M}+\text{Na}]^+$ 413.1060, found: 413.1054.

Penta-O-acetate-D-mannose, 6, ^1H NMR (300 MHz, CDCl_3 , δ in ppm): 1.99 (s, 3H), 2.04 (s, 3H), 2.08 (s, 3H), 2.16 (s, 6H), 4.02-4.11 (m, 2H), 4.24-4.30 (m, 1H), 5.25 (d, 1H, $J = 3.0$ Hz), 5.33 (t, 2H, $J = 3.0$ Hz), 6.07 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3 , δ in ppm): 20.55, 20.58, 20.63, 20.69, 20.78, 62.03, 65.46, 68.26, 68.66, 70.52, 90.52, 167.99, 169.46, 169.66, 169.91, 170.57; HR-MS (ESI) m/z calcd for $\text{C}_{16}\text{H}_{22}\text{O}_{11}$ $[\text{M}+\text{Na}]^+$ 413.1060, found: 413.1054.

General procedure for the synthesis of 2-allylethoxyl monosaccharides (7-8)

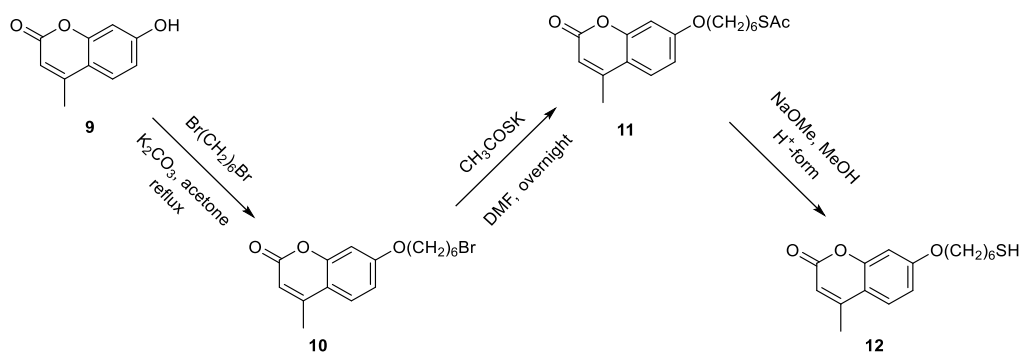
2-Allylethoxyl monosaccharides were generated via a published route with adaptation.^[2] To a solution of the fully acetylated monosaccharide **5** (**6**) (1 g, 2.5 mmol) in dichloromethane was added 2-allyloxyethanol (0.35 mL, 3.3 mmol), followed by the addition of boron trifluoride diethyl etherate (0.41 mL, 3.33 mmol) in the ice-bath under N_2 protection. Then, the mixture was stirred overnight at room temperature. After that, cold water was added and the mixture was washed with NaHCO_3 solution. After the solvent was removed under reduced pressure, an oily residue was obtained without further purification. Then, the oily residue was dissolved in 10 mL methanol, and 50 mg NaOMe were added. The mixture was stirred for overnight and then added to the ion exchange resin (H^+ -form) for separation by adjusting the pH to neutral. Finally, purification by column chromatography on silica gel led to oil compounds as product **7** (**8**).

2-Allylethoxyl-D-glucose, 7, ^1H NMR (300 MHz, D_2O , δ in ppm): 3.27-3.90 (m, 9H), 4.03-4.10 (m, 3H), 4.49 (d, 1H, $J = 9.0$ Hz), 5.25-5.39 (m, 2H), 5.90-6.03 (m, 1H); ^{13}C NMR (75 MHz, D_2O , δ in ppm): 60.36, 68.68, 69.59, 70.77, 71.75, 73.08, 75.62, 75.86, 102.22, 118.61, 133.48; HR-MS (ESI) m/z calcd for $\text{C}_{11}\text{H}_{21}\text{O}_7$ $[\text{M}+\text{H}]^+$ 265.1287, found: 265.1282.

2-Allylethoxyl-D-mannose, 8, ^1H NMR (300 MHz, D_2O , δ in ppm): 3.65-3.75 (m, 5H), 3.78-3.92 (m, 4H), 3.98 (t, 1H, $J = 3.0$ Hz), 4.11 (dd, 2H, $J = 3.0$ Hz, $J = 6.0$ Hz), 4.90 (d, 1H, $J = 3.0$ Hz), 5.28-5.40 (m, 2H), 5.91-6.04 (m, 1H); ^{13}C NMR (75 MHz, D_2O , δ in ppm): 60.89, 66.38, 66.70, 68.59, 69.96, 70.49, 71.69, 72.69, 99.90, 118.37, 133.67; HR-MS (ESI) m/z calcd for $\text{C}_{11}\text{H}_{21}\text{O}_7$ $[\text{M}+\text{H}]^+$ 265.1287, found: 265.1282.

Synthesis of 7-mercaptohexyloxy-4-methylcoumarins

7-Mercaptohexyloxy-4-methylcoumarins were synthesized from commercially available 7-hydroxy-4-methylcoumarin in 3 steps. The steps are described below (**Scheme S2**).



Scheme S2. Synthesis of 7-mercaptohexyloxy-4-methylcoumarin (**12**)

Synthesis of 7-bromoalkyloxy-4-methylcoumarins (**10**)

According to the literature,^[3] anhydrous K_2CO_3 (0.78 g, 5.68 mmol) and 1,6-dibromohexane (11.35 mmol) were added to a solution of 7-hydroxy-4-methylcoumarin (0.5 g, 2.84 mmol) in acetone (5 mL), and the mixture was refluxed for 10 hours. After cooling, the reaction mixture was filtered, and the filtrate was evaporated under reduced pressure. The residue was purified on silica gel chromatography to afford solid product **10**.

7-bromoalkyloxy-4-methylcoumarin, 10, 1H NMR (300 MHz, $CDCl_3$, δ in ppm): 1.52-1.59 (m, 4H), 1.80-1.96 (m, 4H), 2.41(d, 3H, $J = 3.0$ Hz), 3.44 (t, 2H, $J = 6.0$ Hz), 4.03 (t, 2H, $J = 6.0$ Hz), 6.14 (d, 1H, $J = 3.0$ Hz), 6.81 (d, 1H, $J = 3.0$ Hz), 6.84-6.87 (m, 1H), 7.50 (d, 1H, $J = 9.0$ Hz); ^{13}C NMR (75 MHz, $CDCl_3$, δ in ppm): 18.65, 25.16, 27.81, 28.68, 32.57, 33.45, 68.26, 101.27, 111.85, 112.64, 113.42, 125.76, 152.65, 155.08, 160.98, 162.48; HR-MS (ESI) m/z calcd for $C_{16}H_{20}BrO_3$ $[M+H]^+$ 339.0596, found: 339.0590.

Synthesis of 7-acetylmercaptohexyloxy-4-methylcoumarins (**11**)

According to previously reported substitution reaction,^[4] potassium thioacetate (4.5 mmol) was added to a solution of **10** (3 mmol) in DMF (5 mL). The mixture was stirred at room temperature for 1 day. When finished, the mixture was poured to water and extracted with ethyl acetate. The solvent was removed by rotary evaporation and the crude product was purified on silica gel chromatography to afford solid compound **11**.

7-acetylmercaptohexyloxy-4-methylcoumarin, 11, 1H NMR (300 MHz, $CDCl_3$, δ in ppm): 1.39-1.54 (m, 4H), 1.55-1.67 (m, 2H), 1.78-1.87 (m, 2H), 2.33 (s, 3H), 2.40 (s, 3H), 2.87-2.92 (m, 2H), 3.99-4.04 (m, 2H), 6.14 (d, 1H, $J = 3.0$ Hz), 6.81 (d, 1H, $J = 3.0$ Hz), 6.85 (dd, 1H, $J = 3.0$ Hz, $J = 9.0$ Hz), 7.50 (d, 1H, $J = 9.0$ Hz); ^{13}C NMR (75 MHz, $CDCl_3$, δ in ppm): 18.60, 25.46, 28.39, 28.77, 28.93, 29.29, 30.52, 68.44, 101.18, 111.87, 112.63, 113.64, 125.45, 152.06, 155.36, 160.99, 162.17, 195.91; HR-MS (ESI) m/z calcd for $C_{18}H_{23}O_4S$ $[M+H]^+$ 335.1317, found: 335.1312.

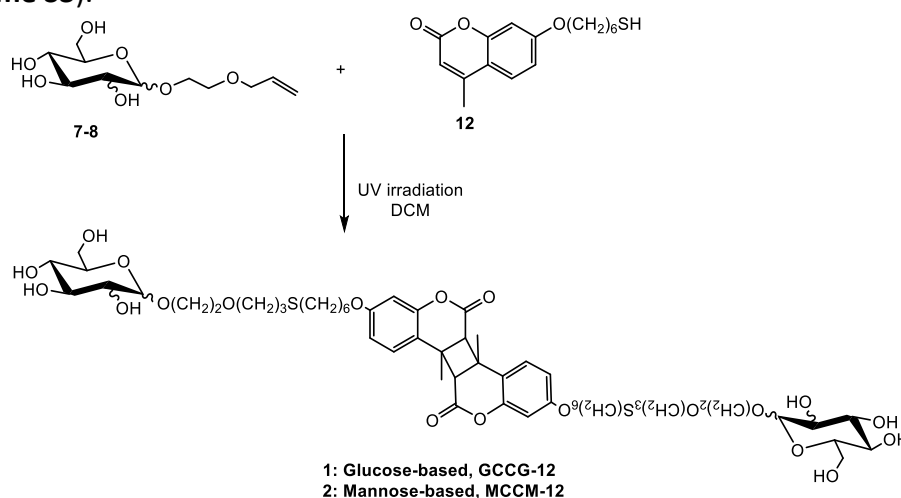
Synthesis of 7-mercaptohexyloxy-4-methylcoumarins (**12**)

Under protection with N_2 gas, NaOMe (6 mmol) was added to a solution of **11** (3 mmol) in methanol. Then, the mixture was stirred overnight and added to the ion exchange resin (H^+ -form) to adjust the pH to neutral. Finally, purification by column chromatography on silica gel led to white solid compound **12**.

7-mercaptohexyloxy-4-methylcoumarin, 12, ^1H NMR (300 MHz, CDCl_3 , δ in ppm): 1.29-1.34 (m, 1H), 1.43-1.46 (m, 4H), 1.58-1.67 (m, 2H), 1.74-1.83 (m, 2H), 2.35 (s, 3H), 2.48-2.55 (m, 2H), 3.95-4.00 (m, 2H), 6.07 (s, 1H), 6.74 (d, 1H, $J = 3.0$ Hz), 6.81 (dd, 1H, $J = 3.0$ Hz, $J = 9.0$ Hz), 7.45 (d, 1H, $J = 9.0$ Hz); ^{13}C NMR (75 MHz, CDCl_3 , δ in ppm): 18.50, 24.36, 25.33, 27.87, 28.72, 33.68, 68.25, 101.17, 111.62, 112.40, 113.26, 125.36, 152.43, 155.09, 161.09, 161.97; HR-MS (ESI) m/z calcd for $\text{C}_{16}\text{H}_{21}\text{O}_3\text{S}$ $[\text{M}+\text{H}]^+$ 293.1211, found: 293.1206.

Synthesis of sugar-based bolaamphiphiles

Sugar-based bolaamphiphiles were synthesized from synthesized intermediate compounds 2-allylethoxyl-D-monosaccharides (**7-8**) and 7-mercaptohexyloxy-4-methylcoumarins (**12**) using thiol-ene click reaction and photo-dimerization in one-pot. The synthesis route is described below (**Scheme S3**).



Scheme S3. Synthesis of glucose-based **1** and mannose-based **2** bolaamphiphiles.

General procedure: 2-allylethoxyl-D-monosaccharide **7** (**8**) (4.5 mmol) and 7-mercaptohexyloxy-4-methylcoumarin **12** (3 mmol) were dissolved in 5 mL dichloromethane. The solution was purged with argon gas for 3 min and then exposed to UV light (wavelength from 300-400 nm, 8 W) for several days. When finished, the solvent was removed on rotary evaporator and the crude product was purified on silica gel chromatography to afford solid compound **1** (**2**).

Glucose-based bolaamphiphile, 1, ^1H NMR (300 MHz, CDCl_3 , δ in ppm): 1.41-1.91 (m, 28H), 2.50-2.61 (m, 8H), 3.31-3.44 (m, 5H), 3.56-3.86 (m, 24H), 3.98-4.10 (m, 4H), 4.37 (s, 1H), 4.91-5.37 (m, 4H), 6.01 (d, 2H, $J = 3.0$ Hz), 6.63 (d, 2H, $J = 9.0$ Hz), 7.07 (d, 2H, $J = 9.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3 , δ in ppm): 25.69, 28.64, 28.70, 28.95, 29.30, 29.49, 31.43, 32.04, 41.14, 55.24, 61.28, 68.16, 68.68, 69.35, 69.76, 69.89, 73.29, 75.80, 76.27, 102.08, 102.97, 112.33, 113.70, 127.31, 150.24, 159.68, 164.83; HR-MS (ESI) m/z calcd for $\text{C}_{54}\text{H}_{80}\text{NaO}_{20}\text{S}_2$ $[\text{M}+\text{Na}]^+$ 1135.4582, found: 1135.4577.

Mannose-based bolaamphiphile, 2, ^1H NMR (300 MHz, CDCl_3 , δ in ppm): 1.41-1.49 (m, 10H), 1.59-1.72 (m, 14H), 1.83-1.87 (m, 4H), 2.50-2.60 (m, 8H), 2.87 (s, 2H), 3.41 (s, 2H), 3.53-3.62 (m,

12H), 3.72-4.02 (m, 18H), 4.87-5.31 (m, 4H), 6.02 (d, 2H, $J = 3.0$ Hz), 6.63 (dd, 2H, $J = 3.0$ Hz, $J = 9.0$ Hz), 7.07 (d, 2H, $J = 9.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3 , δ in ppm): 25.68, 28.67, 28.70, 28.94, 29.49, 29.51, 31.48, 32.03, 41.12, 55.23, 60.84, 66.21, 66.62, 68.15, 69.81, 70.66, 71.27, 72.11, 72.37, 102.11, 112.31, 113.67, 127.33, 134.52, 150.23, 159.68, 164.81; HR-MS (ESI) m/z calcd for $\text{C}_{54}\text{H}_{80}\text{NaO}_{20}\text{S}_2$ $[\text{M}+\text{Na}]^+$ 1135.4582, found: 1135.4577.

3. Temperature-responsive reversible clear-turbid-transition of CHO-Bolas in solutions of diverse pH values

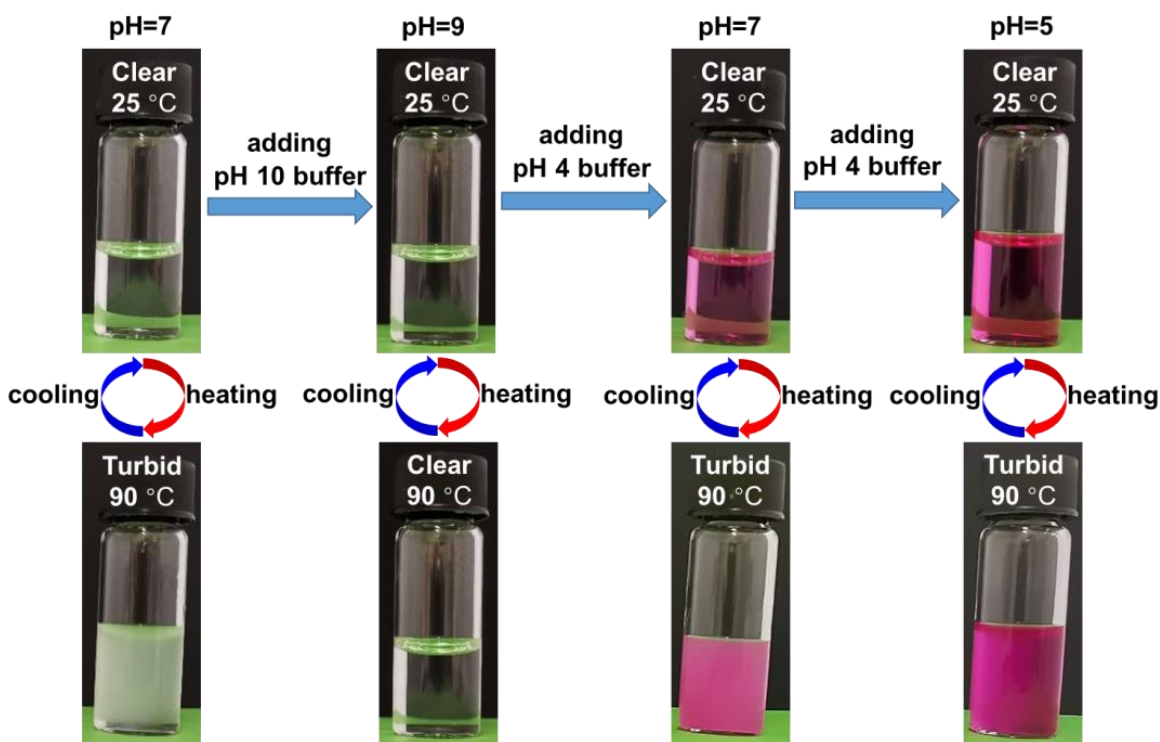


Figure S1. Photo images showing the temperature-responsive reversible clear-turbid cycles of aqueous solutions of CHO-Bolas at diverse pH values. The pink color is due to the color of the commercial pH 4 buffer.

4. Assembly Experiments

All assembly experiments were performed in 5-fold diluted buffer solution by mixing 0.1 mL pH 10 buffer solution with 0.4 mL DI water. In a 4 mL capped bottle, the mixture compounds were dispersed in 0.5 mL diluted buffer solution using a 1:3 mole ratio of GCCG-12/MCCM-12 (10 mg) to BDDBA (4.5 mg), and then sonicated for 10 minutes. A control experiment was conducted by

only dispersing 4.5 mg BDBA in 0.5 mL diluted buffer solution and following sonication at room temperature for 10 minutes. Then, all three capped bottles were placed in pre-heated oil bath at 80 °C until clear solutions were obtained. The solutions were slowly cooled to room temperature and the aggregates were generated after 3-4 hours. Afterwards, the assembled aggregates were purified by washing three times with DI water to remove buffer solution, followed by filtration through 0.45 μm membrane (PVDF hydrophilic, Carl-Roth, Germany).

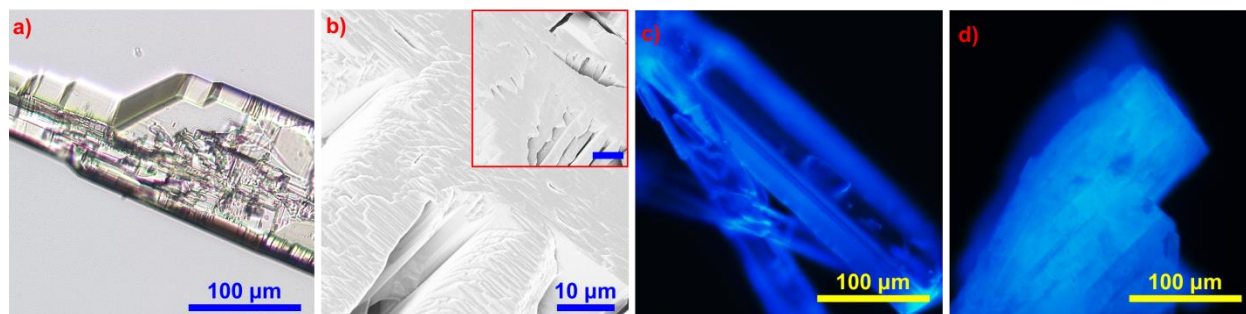


Figure S2. The brick-shaped structures self-assembled by BDBA alone: a) Optical light microscopy image; b) SEM image. The inset shows more typical stack structure with the scale bar of 50 μm ; c) Fluorescence microscopy image of as-prepared sample; d) Fluorescence microscopy image after the treatment at 300 °C in nitrogen gas.

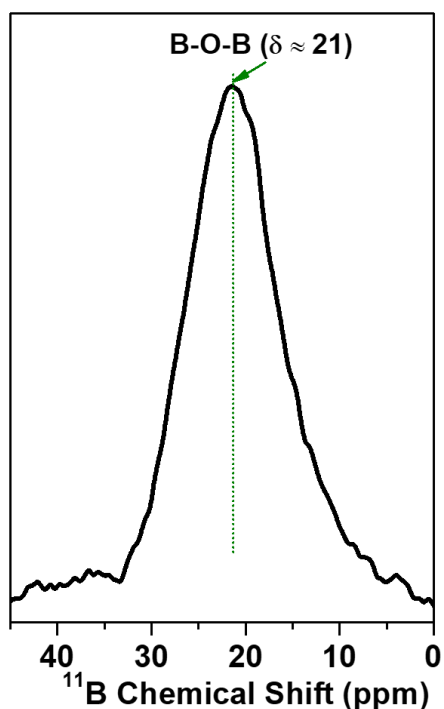


Figure S3. Solid-state ^{11}B NMR spectra of brick-shaped structure formed by BDBA alone after the treatment at 300 °C in nitrogen gas.

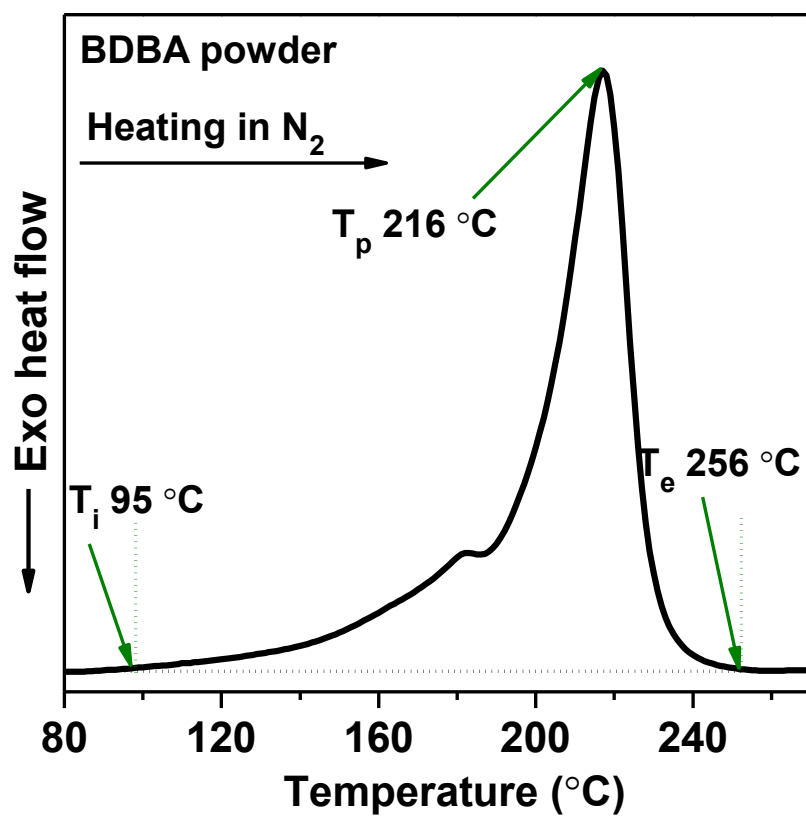
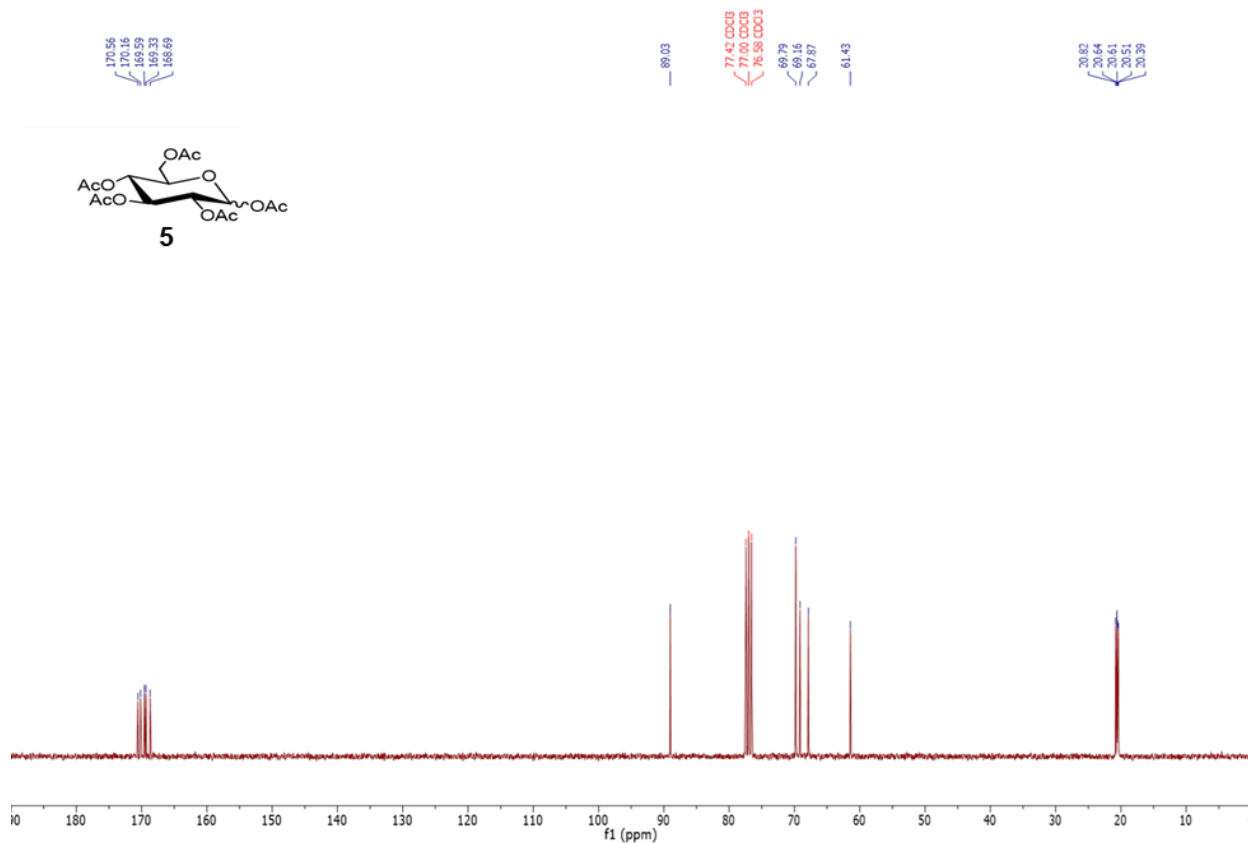
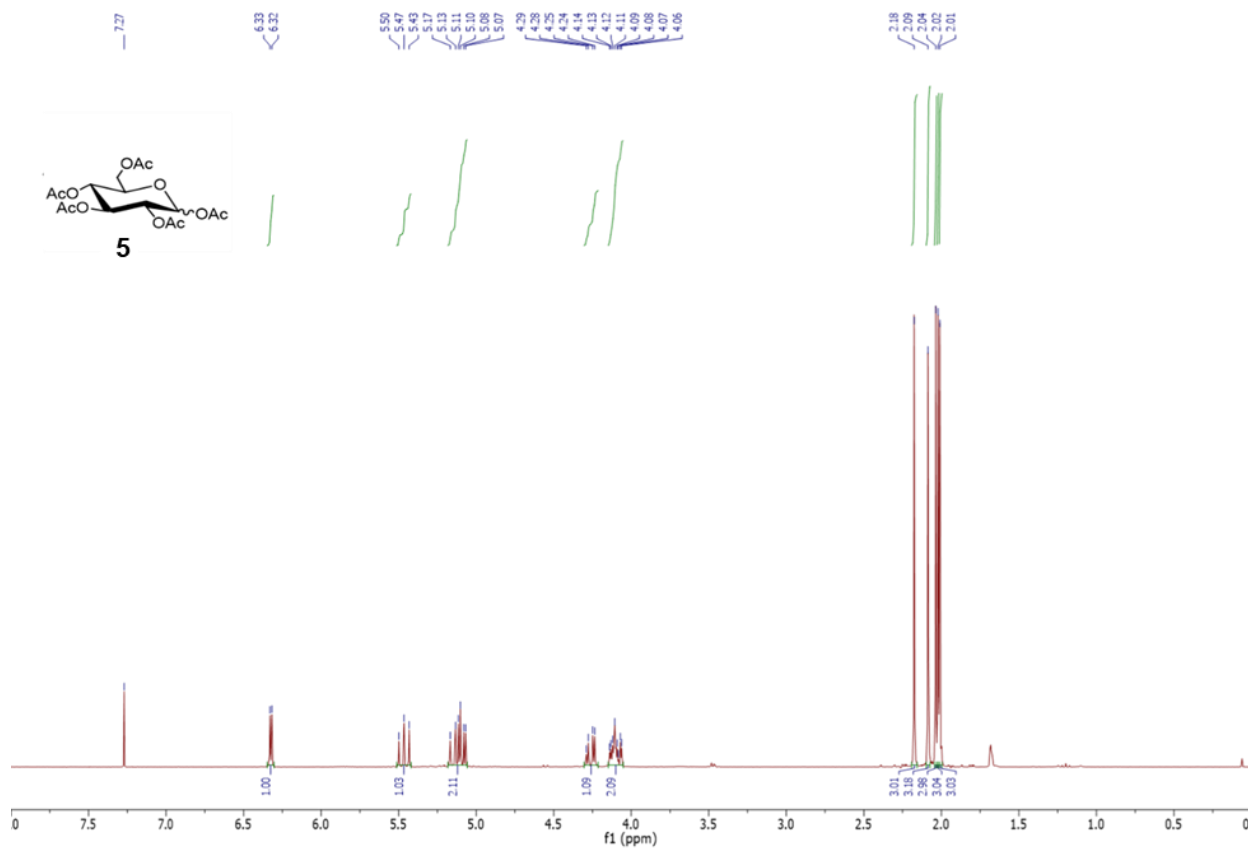
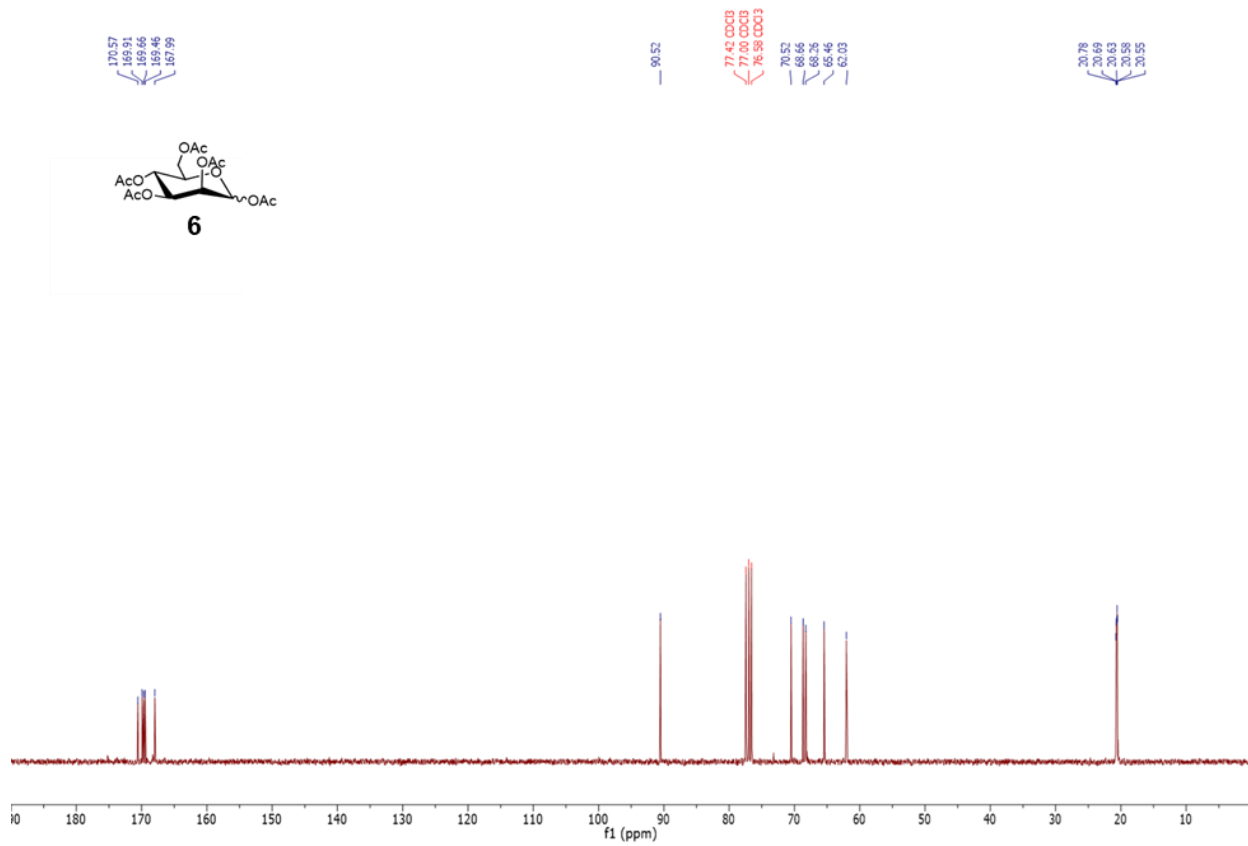
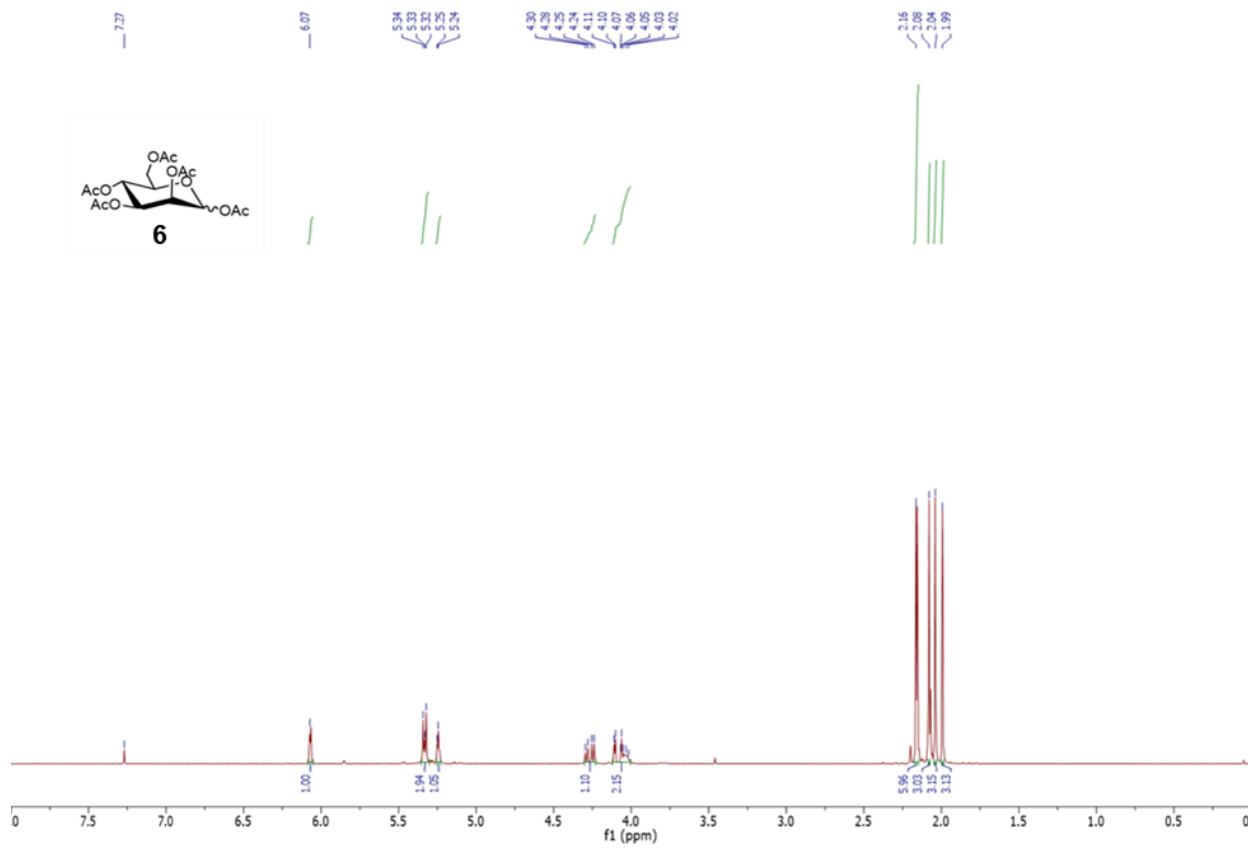
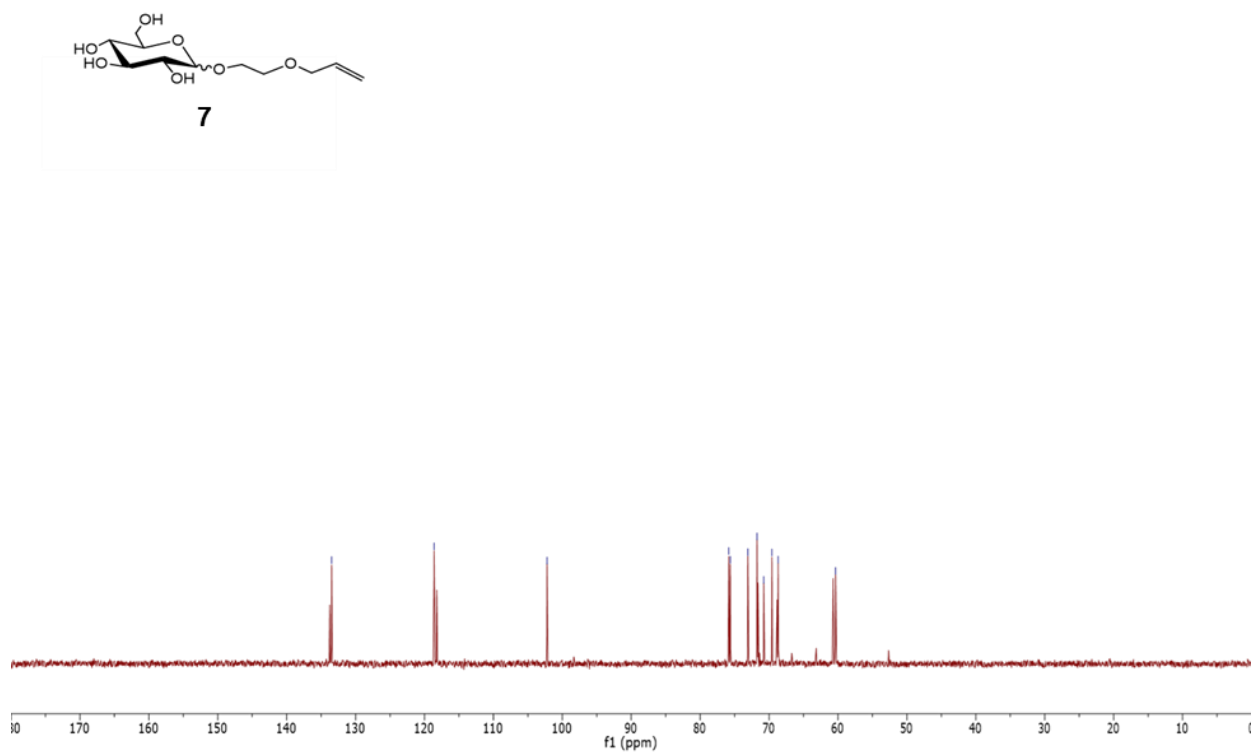
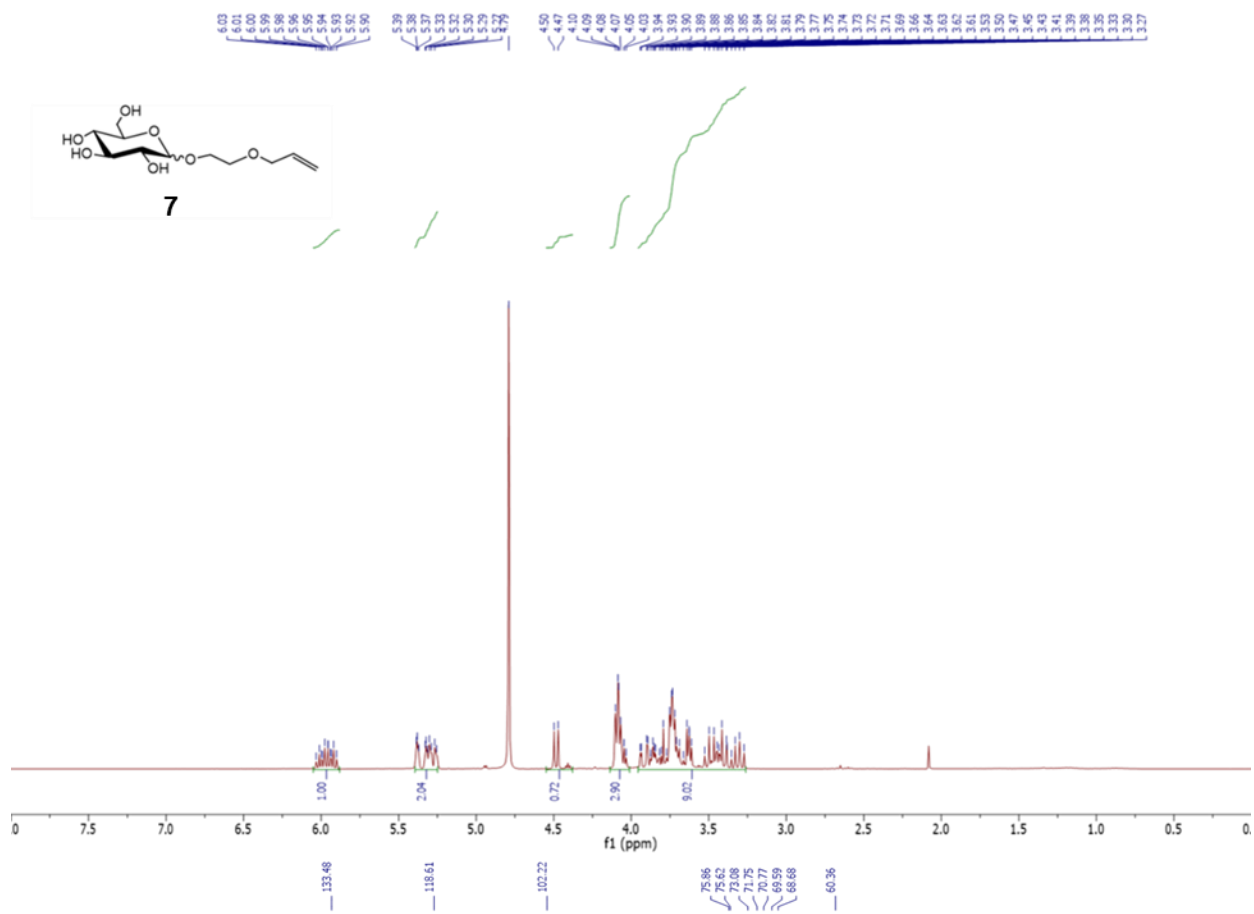


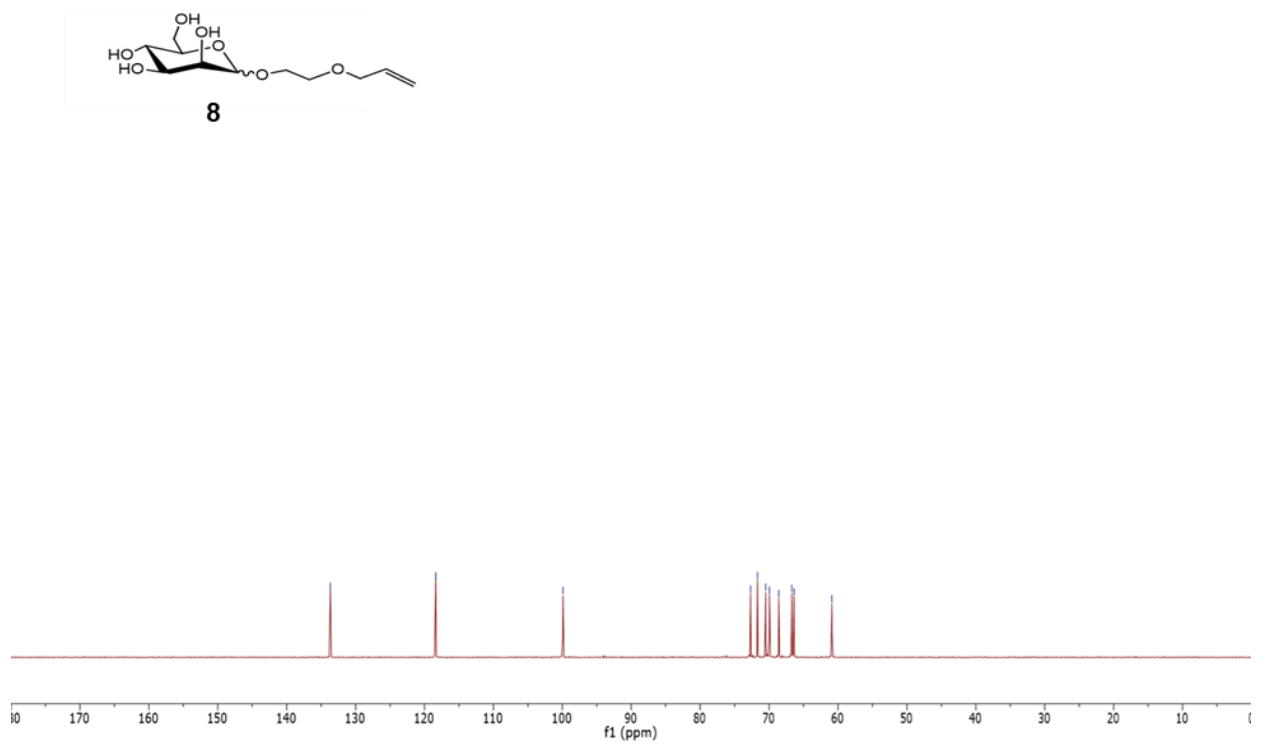
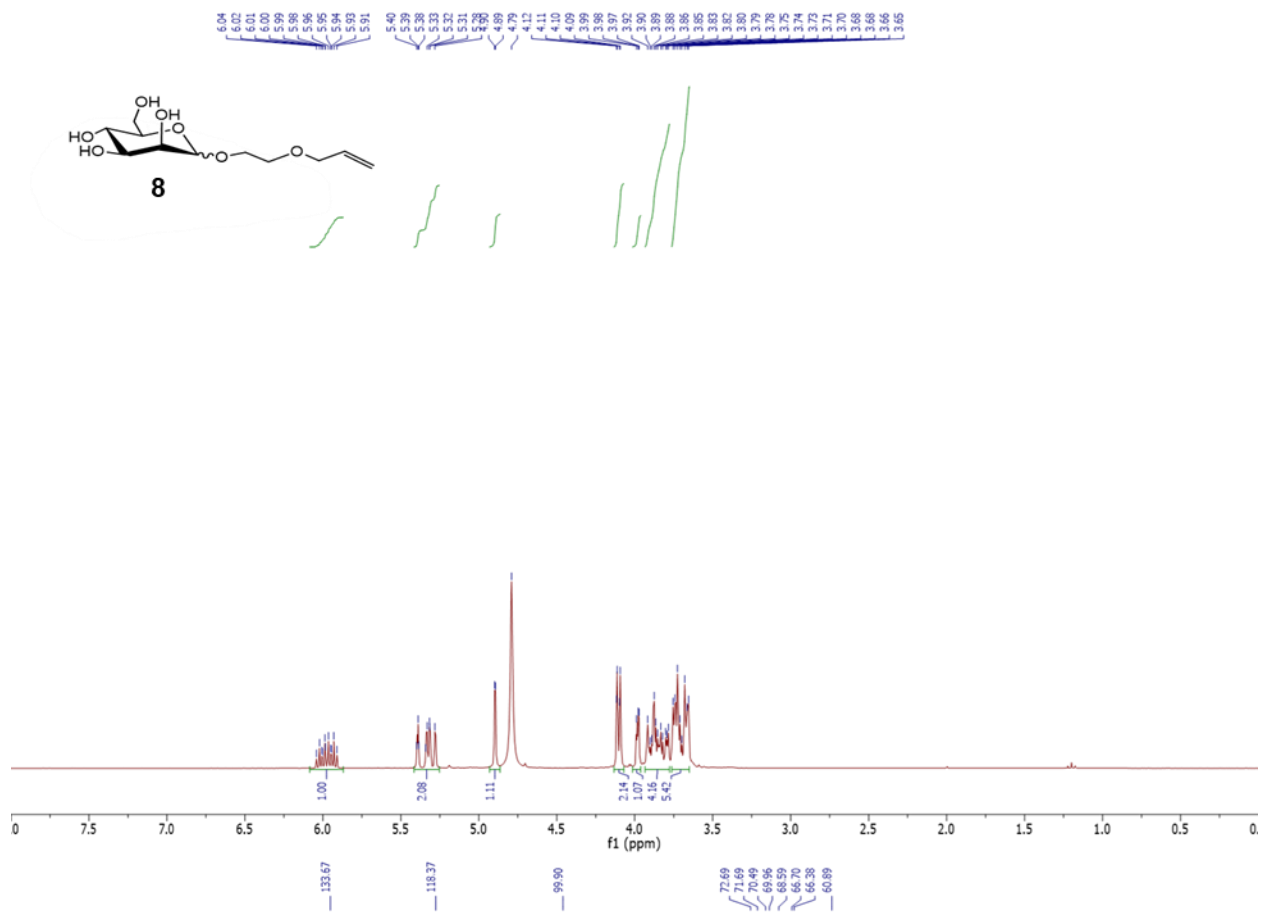
Figure S4. DSC curves of BDBA powder brought from company heated from 20 °C to 300 °C at a heating rate of 5 K/min under a nitrogen atmosphere.

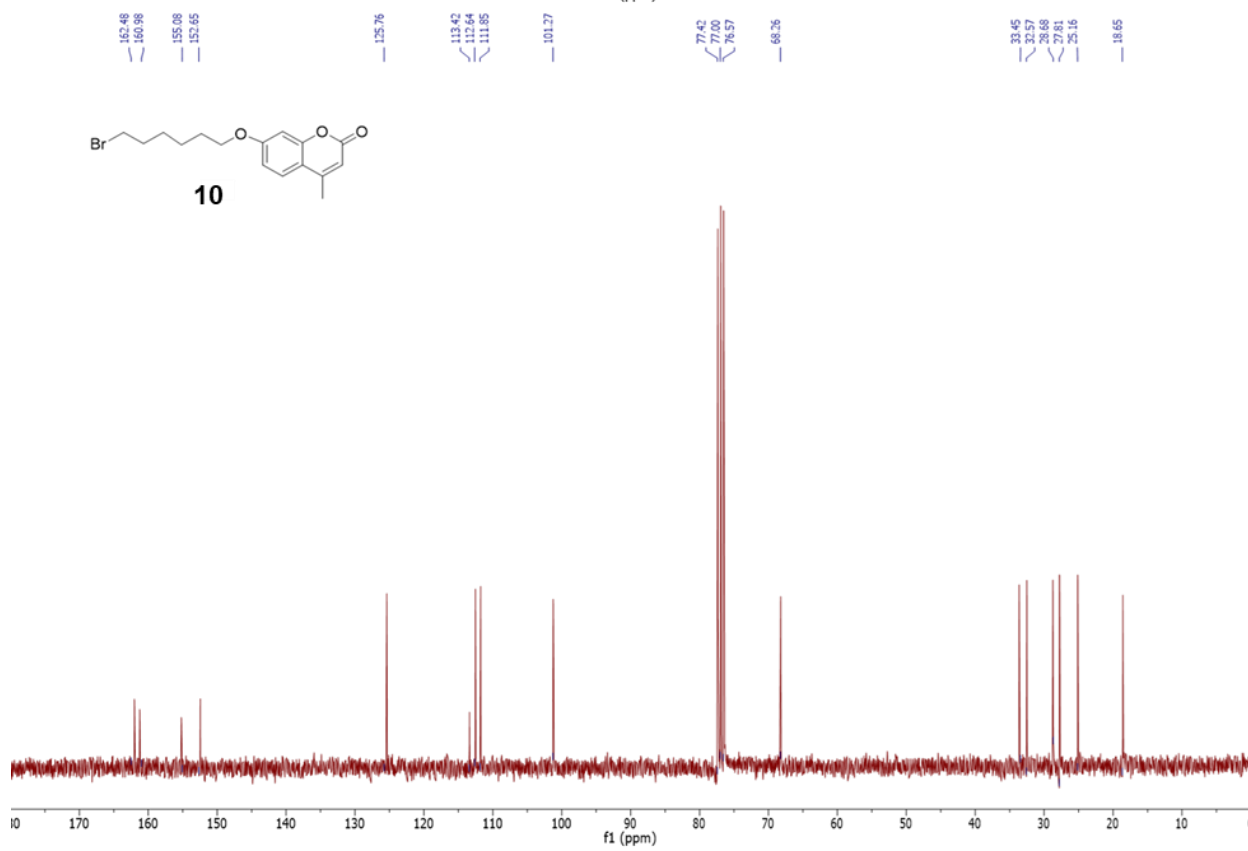
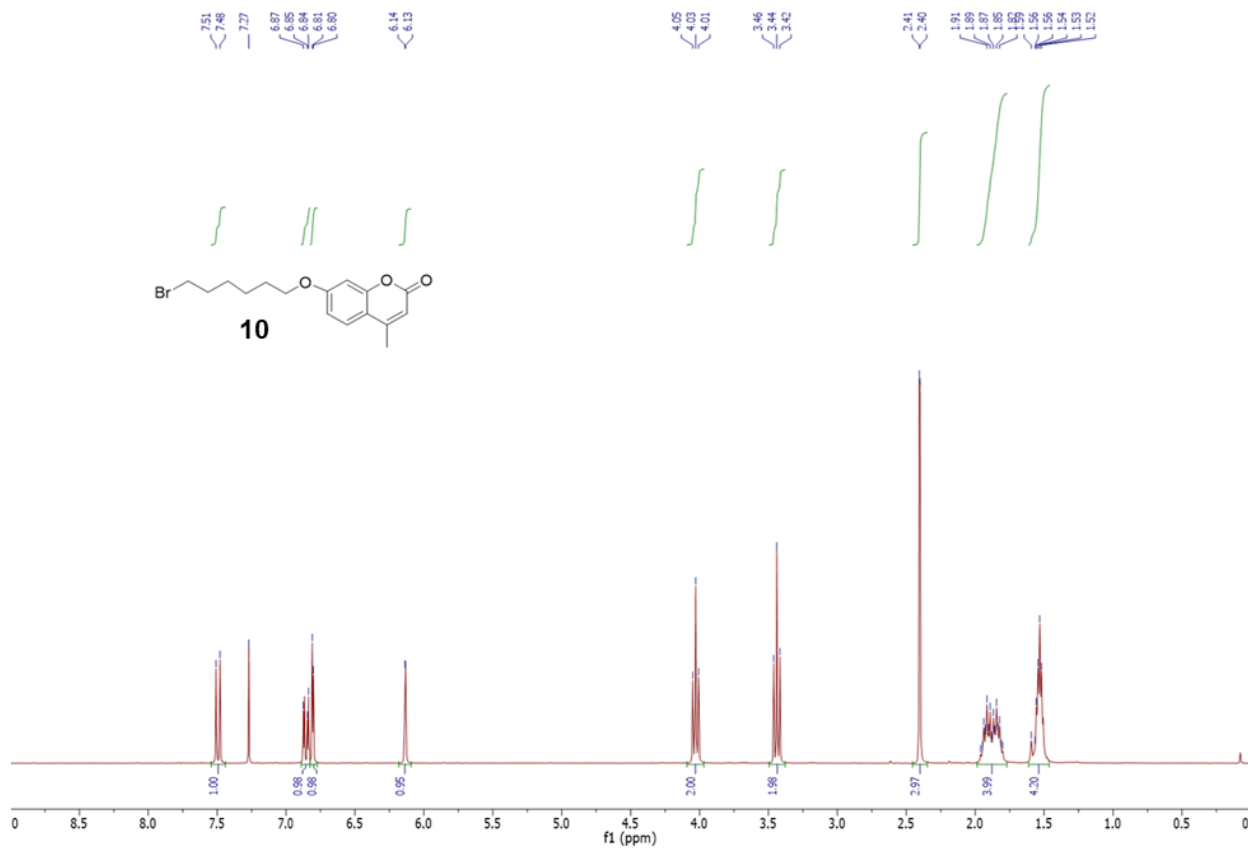
5. ¹H NMR and ¹³C NMR Spectra

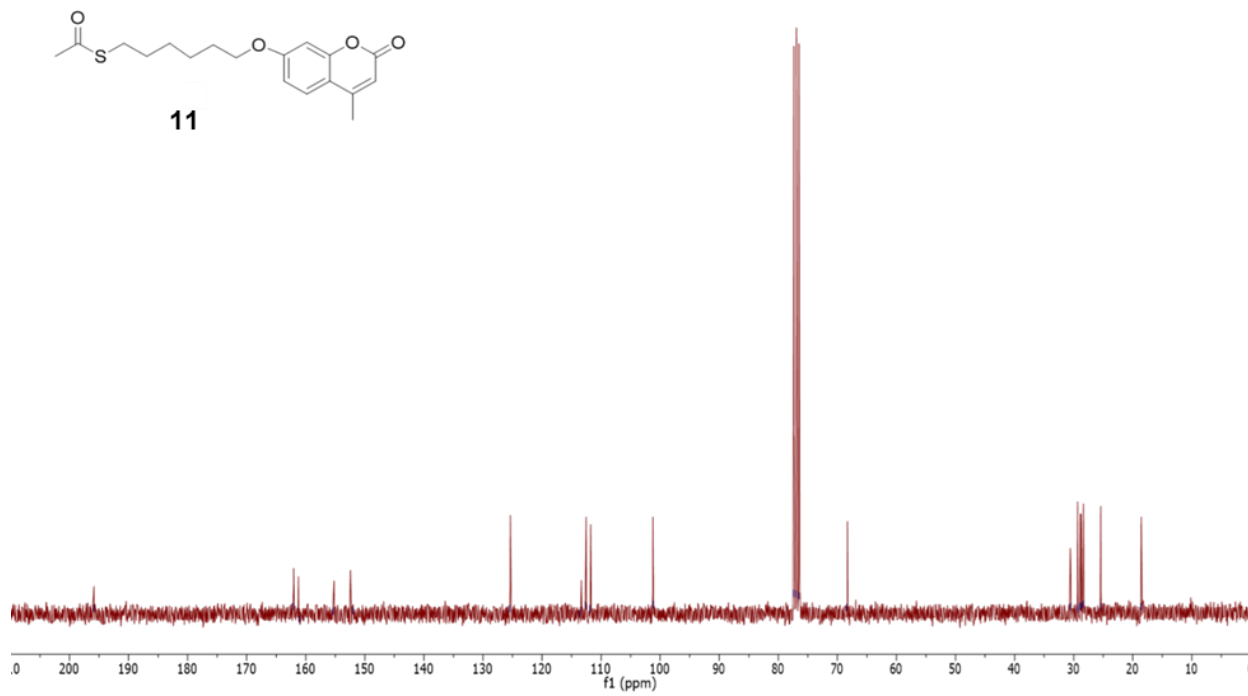
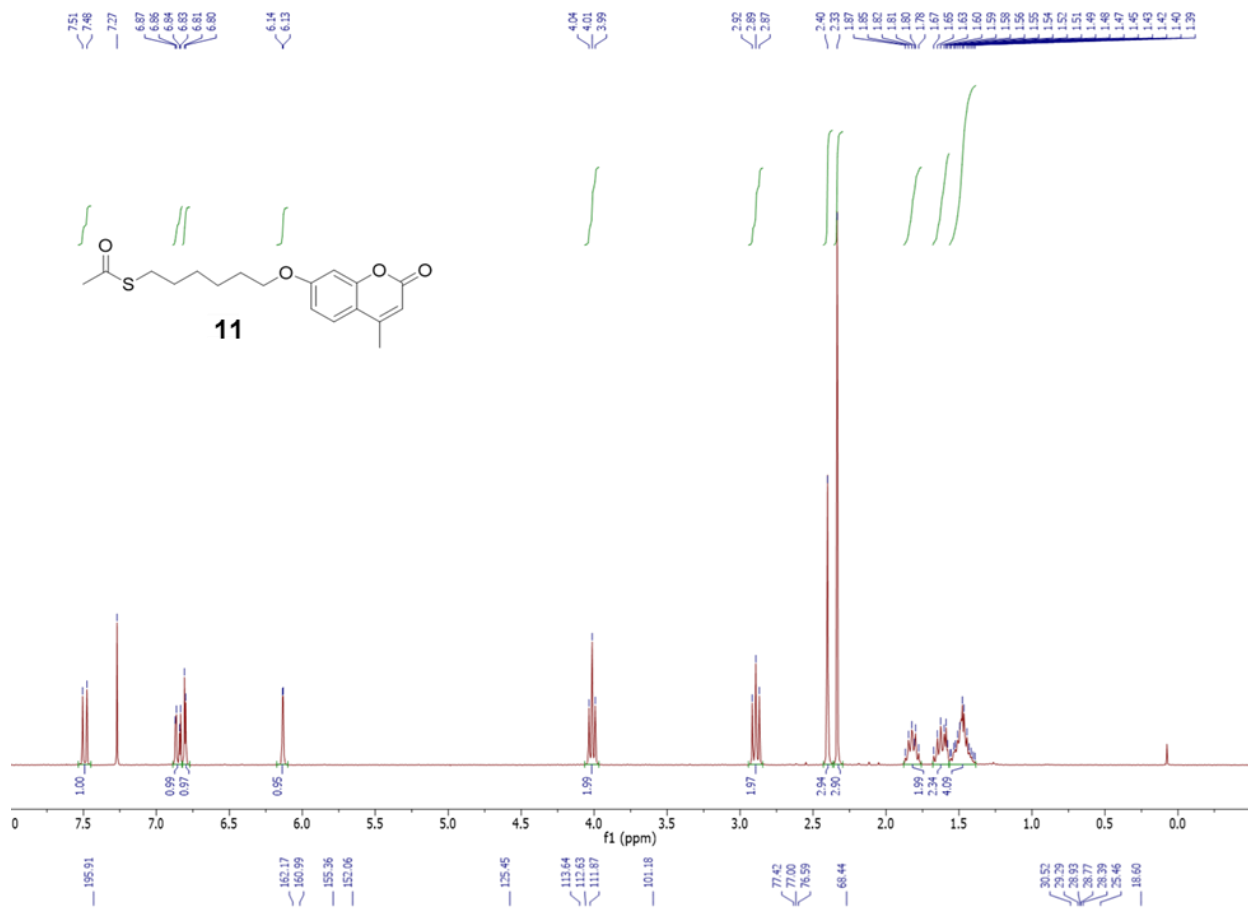


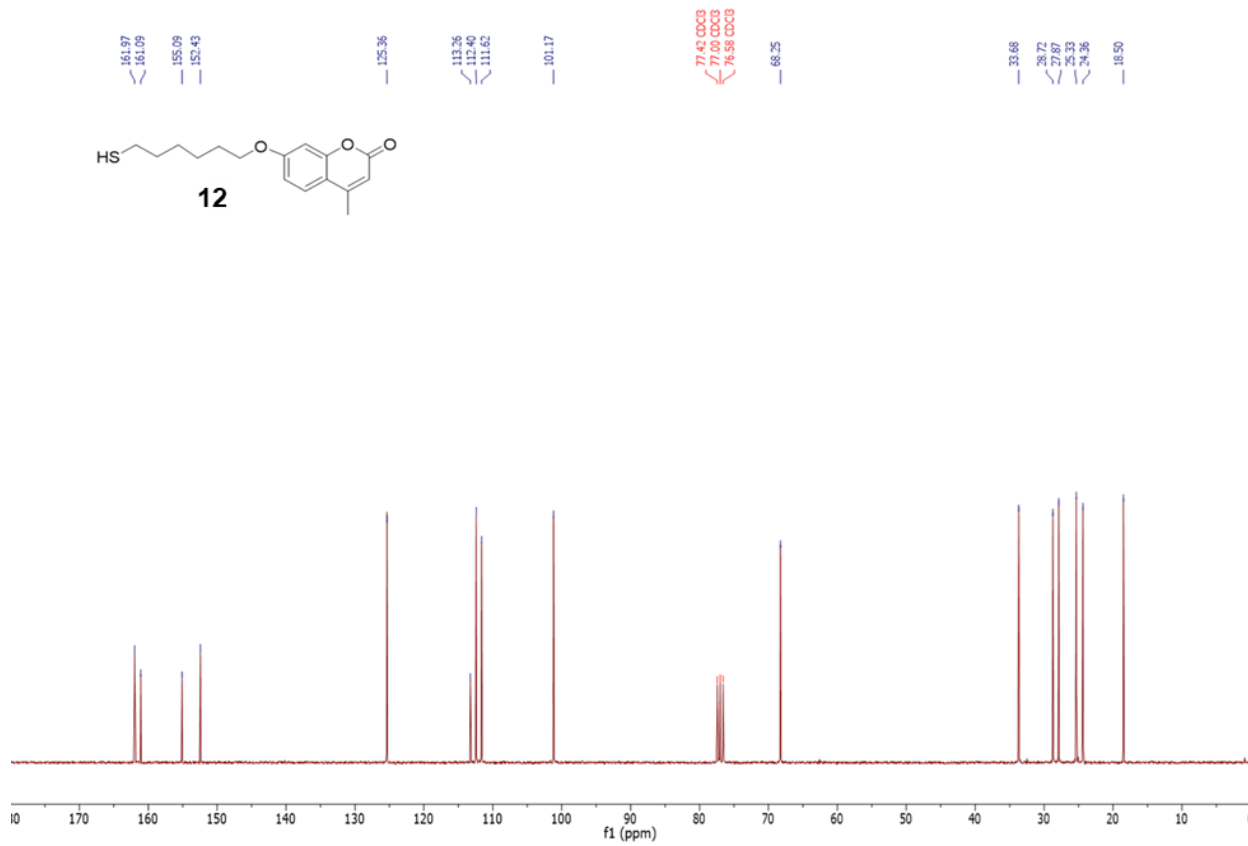
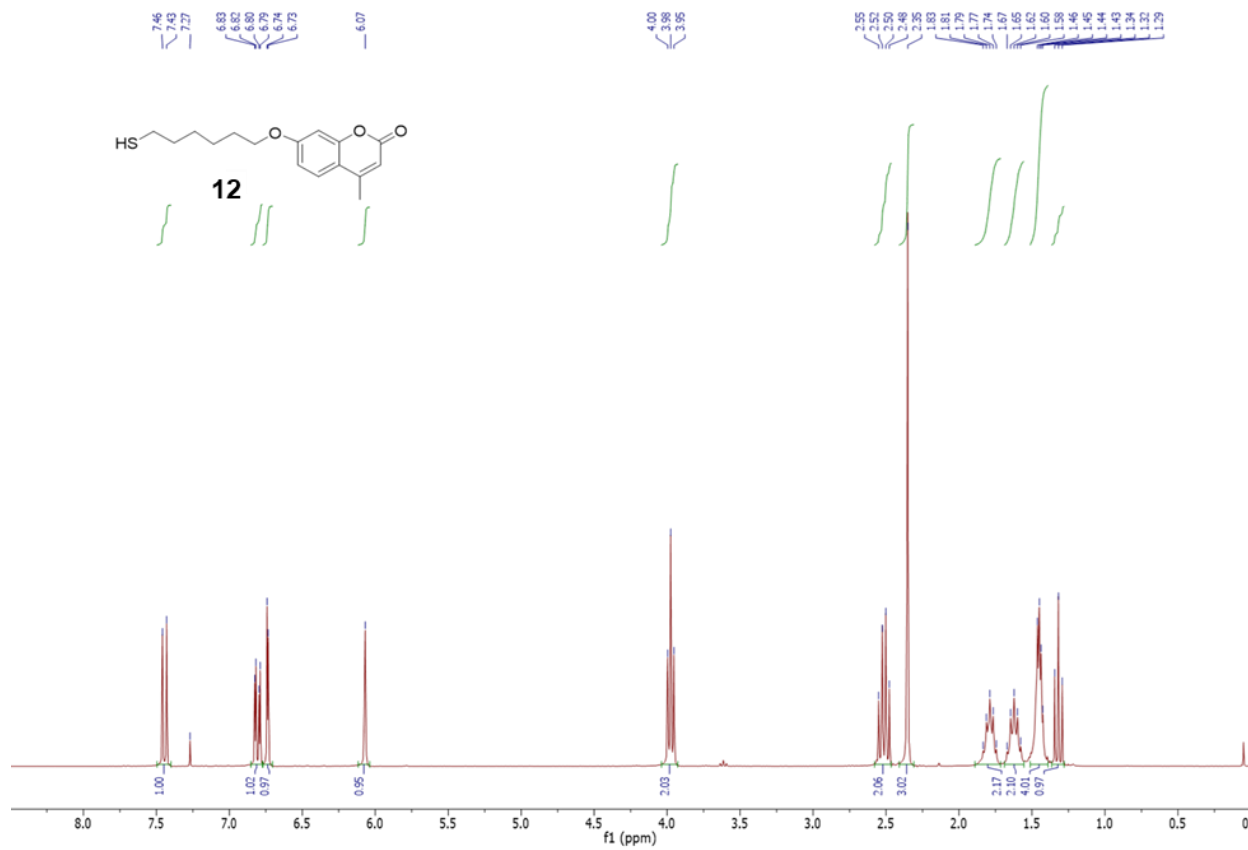


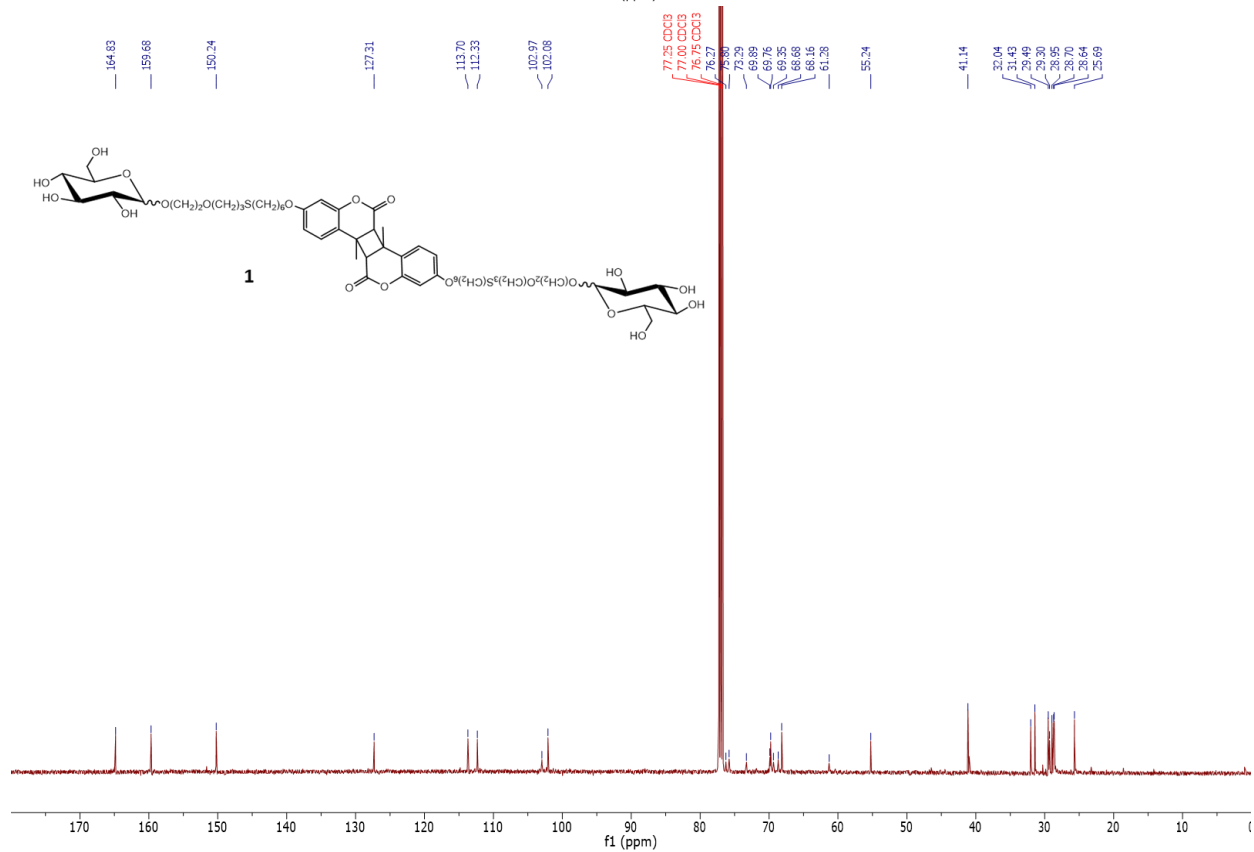
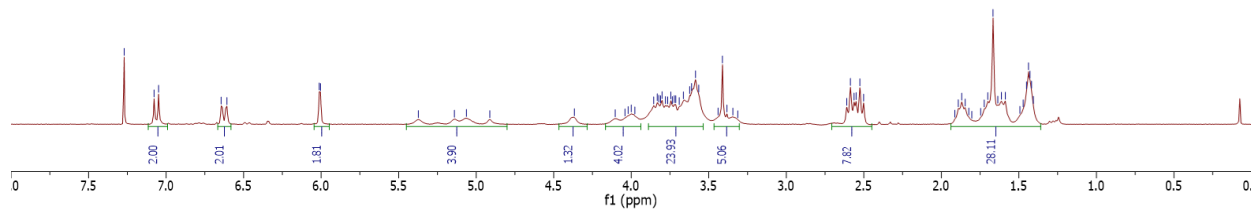
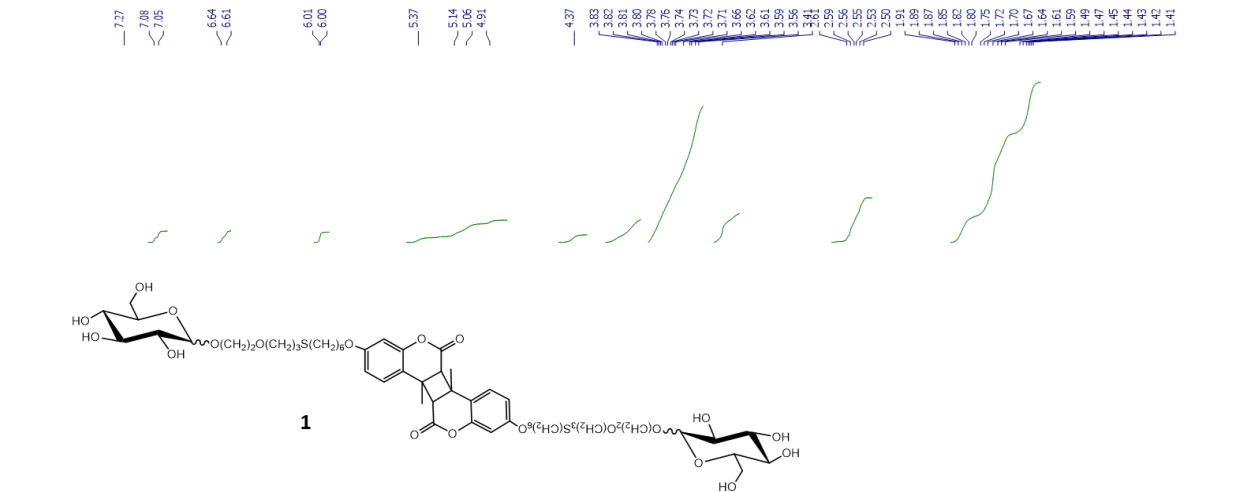


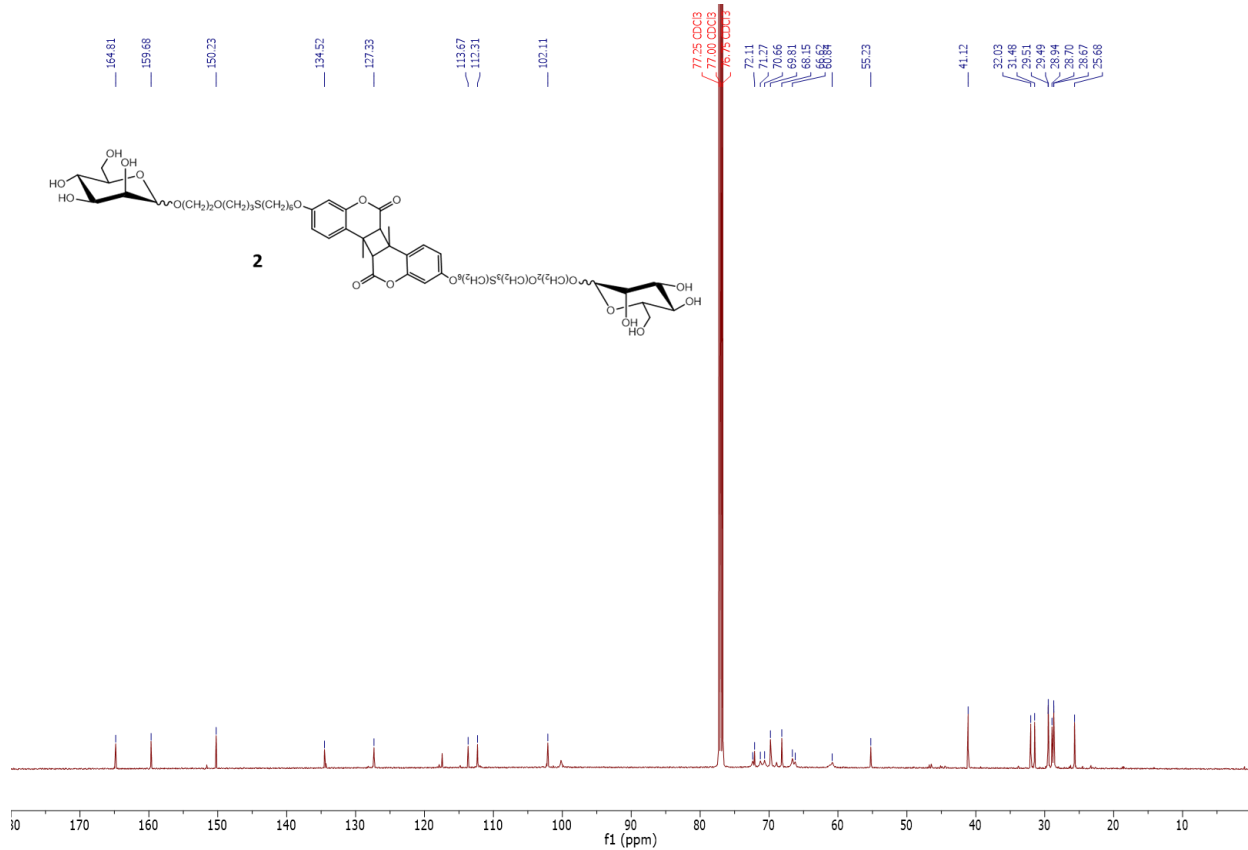
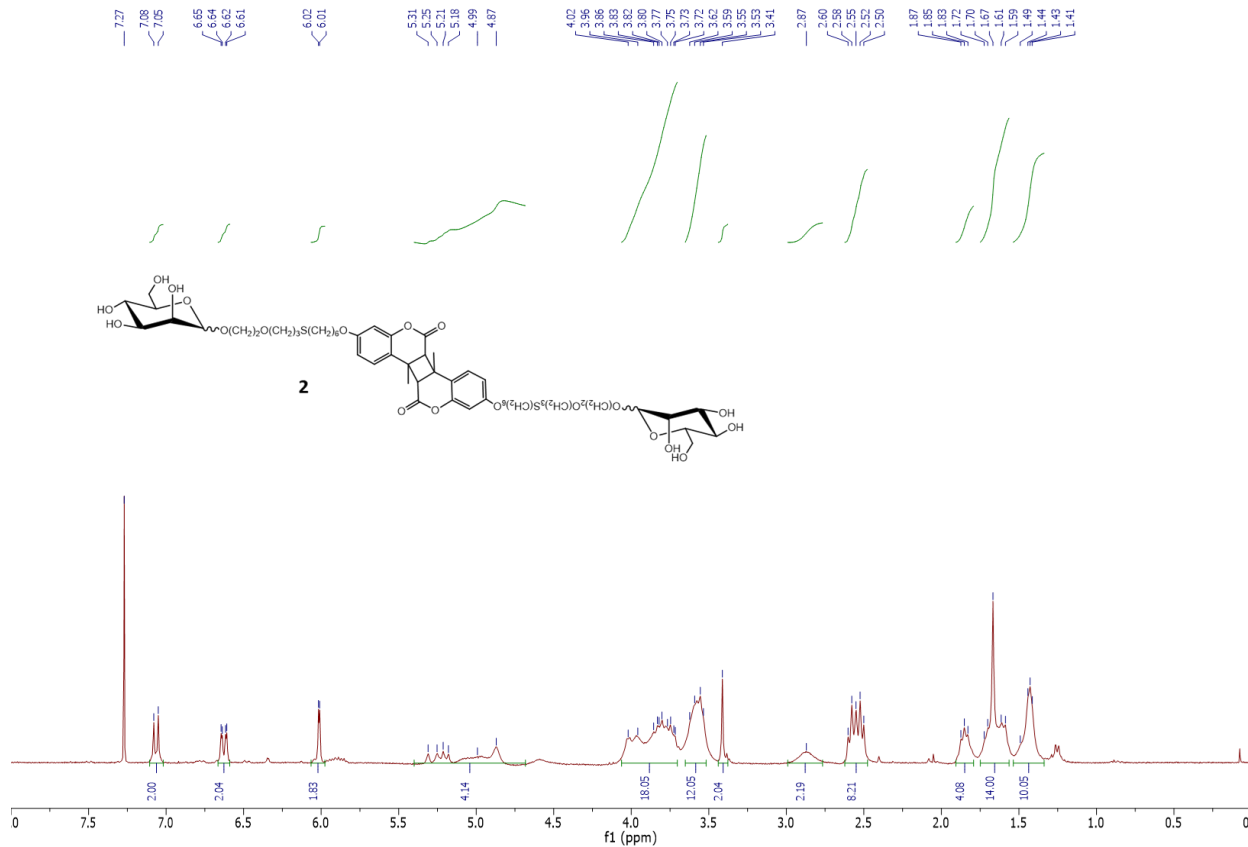












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