Electronic Supplementary Material (ESI) for Polymer Chemistry.


**Supporting Information**

# Photolabile ROMP Gels Using *ortho*-Nitrobenzyl Functionalized Crosslinkers

## 1. General Considerations

All synthetic manipulations were performed under standard air-free conditions under an atmosphere of argon gas with magnetic stirring unless otherwise mentioned. Flash chromatography was performed using silica gel (230-400 mesh) as the stationary phase. NMR spectra were acquired on a Bruker Avance III 500 or Bruker DPX-300 spectrometer. Chemical shifts are reported relative to residual protonated solvent (7.26 ppm for $CHCl_3$). All reactants and solvents were purchased from commercial suppliers and used without further purification, unless otherwise noted. Dry THF, dry DCM, and dry diethyl ether were obtained from an Innovative Technologies PureSolv 400 solvent purifier.

All solution optical spectra were acquired of samples in quartz cuvettes (NSG Precision Cells). Electronic absorbance spectra were acquired with a Varian Cary-100 instrument in double-beam mode using a solvent-containing curette for background subtraction spectra. Fluorescence emission spectra were obtained by using either a PTI Quantum Master 4 equipped with a 75 W Xe lamp, or Cary Eclipse Fluorescence Spectrophotometer equipped with a Xe pulse lamp pulsed at 80 Hz, peak power equivalent to 75 kW. Rheological data was obtained on ARES G2 rheometer.

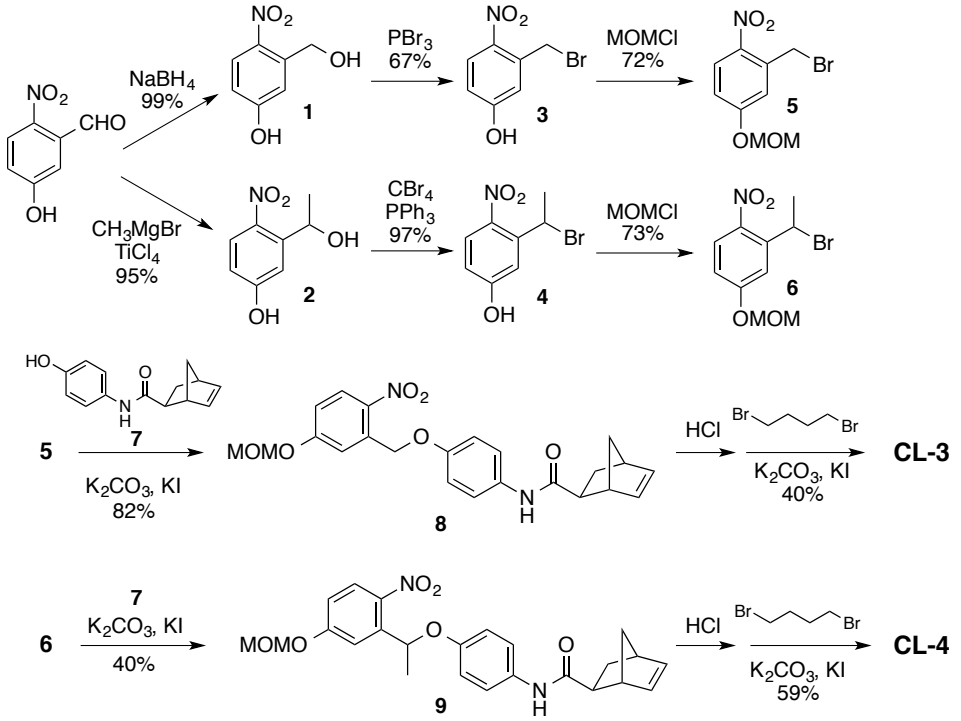
Irradiation of gels for storage modulus measurements were performed with a blak-ray B-100A high intensity UV lamp (100 Watt, 365nm, power density at sample = 9 $mW/cm^2$) at fixed positions in the light path. Irradiations of gels in all other experiments were performed with a 200W Hg/Xe lamp (Newport-Oriel) equipped with a condensing lens, recirculating water filter, manual shutter, and FSQ-BG40 blue bandpass filter (Newport Corporation) at fixed positions in the light path. The power density at the sample was 25 $mW/cm^2$.

## 2. Materials:

5-hydroxy-2-nitrobenzyldehyde, methylmagnesium bromide solution (3.0 M in diethyl ether)，N,N'-dicyclohexylcarbodiimide (DCC), chloromethyl methyl ether (MOMCl), phosphorus tribromide ($PBr_3$), sodium sulfate anhydrous, tetrabromomethane ($CBr_4$), exo-5-norbornenecarboxylic acid, 4-aminophenol, Grubbs 2$^{nd}$ generation ruthenium carbenecatalyst, potassium iodide (KI), N,N-Diisopropylethylamine (i-$Pr_2NEt$), 2-(Methylamino)ethanol, 1,4-dibromobutane and dry N,N-dimethylformamide (DMF), dry triethylamine (TEA), triphenylphosphine ($PPh_3$), methacryloyl chloride, and poly(ethylene glycol)methyl ether methacrylate (Average Mn 300) were purchased from Sigma Aldrich. 4-dimethylaminopyridine (DMAP) and sodium borohydride ($NaBH_4$) were purchased from Acros Organics. Titanium (IV) tetrachloride ($TiCl_4$) was purchased from Fluka. 4-Chloro-7-nitrobenzofurazan (NBDCl) and benzene was purchased from Alfa Aesar. Magnesium sulfate anhydrous ($MgSO_4$), sodium chloride (NaCl), hydrochloric acid (concentrated HCl), ammonium chloride ($NH_4Cl$) and potassium carbonate were purchased from Fisher Scientific. NMR solvents were purchased from Cambridge Isotope Laboratories.

## 3. Synthetic Procedures

**CL-1**, **CL-2**, **inert CL**, **M1** and **M3** were synthesized according to our previously published study on photoreactive ROMP gels.[1]




5-hydroxy-2-nitrobenzenemethanol **1**. To a 50mL three neck round bottom flask was added $NaBH_4$ (0.23g, 6.1 mmol) and methanol, stirred. A methanol solution of 2-nitro-5-hydroxybenzyldehyde (0.50 g, 3.0 mmol) was added dropwise into the flask at 0 °C. (Attention: the mixture may react violently due to the presence of the phenol group) The reaction mixture was then allowed to warm up to ambient temperature and stirred for 2 hours. The reaction was then poured into $H_2O$. The pH of the solution was adjusted to around 6 by 0.1M HCl. The solution was then extracted with diethyl ether three times, dried over $MgSO_4$, filtered, and concentrated using a rotary evaporator to yield **1** as a light brown solid with no further purification. Yield: 0.50 g (99%). $^1$H NMR (500 MHz, MeOD): 8.09 (d, J=9 Hz, 1 H), 7.26 (dd, J=1, 1.5Hz, 1H), 6.78 (dd, J=9, 3Hz, 1H), 4.94 (s, 2 H).

5-hydroxy-α-methyl-2-nitrobenzenemethanol **2**. Dry diethyl ether (100 mL) was cooled to -78 °C in a 250 mL three neck flask. $TiCl_4$ (4.7g, 0.025 mol) was added into the flask dropwise. The mixture was stirred at -78 °C and yielded yellow precipitation. 3M solution of MeMgBr in ether (8.3ml, 0.025mol) was added dropwise into the flask and the mixture turned to an orange color. The reaction was stirred for 30 minutes and the temperature of the mixture was allowed to slowly increase to around -30 °C. Then, 2-nitro-5-hydroxybenzyldehyde (1.7 g, 0.010 mol) was added into the flask very slowly. (Attention: the mixture may react violently due to the presence of the phenol group) The

reaction was stirred at around -30 °C for 3 hours, and then poured into water, extracted with ethyl acetate three times, dried over $MgSO_4$, and concentrated using a rotary evaporator to yield **2** as a yellow solid with no further purification. Yield: 1.7 g (95%). $^1$H NMR (500 MHz, MeOD): 7.95 (d, J= 9 Hz, 1 H), 7.25 (d, J=3, 1 H), 6.76 (dd, J=2.5, 9 Hz, 1 H), 5.47 (q, J=6 Hz, 1 H), 1.44 (d, J=6 Hz, 3 H).

5-hydroxy-2-nitrobenzenemethyl bromide **3**. To a 50 mL three neck round bottom flask was added **1** (0.26 g, 0.0015 mol) and 10 mL dry DMF. $PBr_3$ (1.0 g, 3.7 mmol) was added dropwise into the flask at -78 °C. The reaction was warmed to ambient temperature and stirred overnight. The reaction was poured into water, extracted by ethyl acetate 3 times, dried over $Na_2SO_4$ and concentrated using a rotary evaporator. The crude product was purified via flash chromatography using hexanes/ethyl acetate (1:1 v:v) as the eluent to yield **3** as a yellow solid. Yield: 0.24 g (67%). $^1$H NMR (500MHz, $CDCl_3$): 8.10 (d, 9 Hz, 1 H), 7.00 (d, 2.5 Hz), 6.87 (dd, J=2.5, 9 Hz, 1 H), 5.87 (s, 1 H), 4.84 (s, 2 H).

5-hydroxy-α-methyl-2-nitrobenzenemethanol **4**. A two neck flask was charged with **2** (0.33 g, 1.8 mmol), $CBr_4$ (1.0 g, 3.0 mol), $PPh_3$ (0.79 g, 3.0 mmol) and 25 mL dry THF. The reaction mixture was stirred overnight at ambient temperature. The reaction mixture was filtered, concentrated with a rotary evaporator and purified via flash chromatography using hexanes/ethyl acetate (1.5:1 v:v) as the eluent to yield **4** as a light brown solid. Yield: 0.43 g (97%). $^1$H NMR (500 MHz, MeOD): 7.94 (d, 9 Hz, 1 H), 7.25(d, J=3Hz, 1 H), 6.83 (dd, J=2.5, 9 Hz, 1 H), 5.46 (q, J=6.5 Hz, 1 H), 1.43 (d, J= 6.5 Hz, 3 H).

5-methoxymethyl-2-nitrobenzenemethyl bromide **5**. To a stirred mixture of **3** (1.0 g， 4.3 mmol) in 50 mL $CH_2Cl_2$ were added MOMCl (5 equiv) at 0 °C, then i-$Pr_2$NEt (3.0 equiv) was added. The reaction was stirred overnight at ambient temperature. After slow addition of satured $NH_4Cl$ at 0 °C, the mixture was extracted with $CH_2Cl_2$ 3 times. The combined organic layer was washed with brine, dried over $Na_2SO_4$, filtered, and concentrated in vacuo. The crude product was purified by flash column chromatography using hexanes/ethyl acetate (2.5:1 v:v) to afford **5** as a light yellow solid. Yield: 1.0 g (84%). $^1$H NMR (500MHz, $CDCl_3$): 8.14 (d, J=9.5 Hz, 1 H), 7.32 (d, J=3 Hz, 1 H), 7.09 (dd, J=2.5, 9 Hz, 1 H), 5.27 (s, 2 H), 5.01 (s, 2 H), 3.48-3.50 (m, 3 H)

5-methoxymethyl -α-methyl-2-nitrobenzenemethyl bromide **6**. To a stirred mixture of **4** (0.43g, 1.8 mmol) in 20ml $CH_2Cl_2$ were added MOMCl (5 equiv) at 0 °C, then i-$Pr_2$NEt (3 equiv) was added. The reaction was stirred overnight at ambient temperature. After slow addition of satured $NH_4Cl$ at 0 °C, the mixture was extracted with DCM 3 times. The combined organic layer was washed with brine, dried over $Na_2SO_4$, filtered, and concentrated in vacuo. The crude product was purified by flash column chromatography using hexanes/ethyl acetate (5:2 v:v) to afford **6** as a light yellow solid. Yield: 0.37 g (73%). $^1$H NMR (500 MHz, $CDCl_3$): 7.97 (d, J=9 Hz, 1 H), 7.49 (d, J=2.5 Hz, 1 H), 7.05 (dd, J=2.5, 9 Hz, 1 H), 5.88 (q, J=6.5 Hz, 1 H), 5.24-5.30 (m, 2 H), 3.50 (s, 3 H), 1.88 (d, J=6.5, 3 H).

**7**. Exo-5-norbornenecaboxylic (1.4 g, 0.010 mol), 4-aminophenol (1.3 g, 0.012 mol), DMAP (0.20 g, 0.0016 mol) and 100 mL dry THF were added into a 250 mL three neck round bottom flask. DCC (2.5g, 0.012 mol) in dry THF was added dropwise into the flask at 0 °C. The mixture was stirred overnight at ambient temperature. The reaction mixture was then concentrated *in vacuo* and redissolved in ethyl acetate, washed by HCl solution (pH around 3), brine, and water. The organic phase was dried over $Na_2SO_4$, filtered, concentrated with a rotary evaporator, and the crude product was further purified via flash chromatography using hexanes/ethyl acetate (2:1) as the eluent. Pink solid obtained was recrystallized from $CH_2Cl_2$. After filtration **7** was obtained as white crystals. Yield: 0.62 g (27%). $^1$H NMR (500 MHz, MeOD): 7.31 (q, J=2.5, 7 Hz, 2 H), 6.72 (q, J=2.5, 7 Hz, 2 H), 6.18-6.19 (m, 2 H), 2.95 (d, J=1.5 Hz, 1 H), 2.92 (s, 1 H), 2.25-2.29 (m, 1 H), 1.93-1.97 (m, 1 H), 1.76 (d, J=8.5 Hz, 1 H), 1.33-1.39 (m, 2 H).

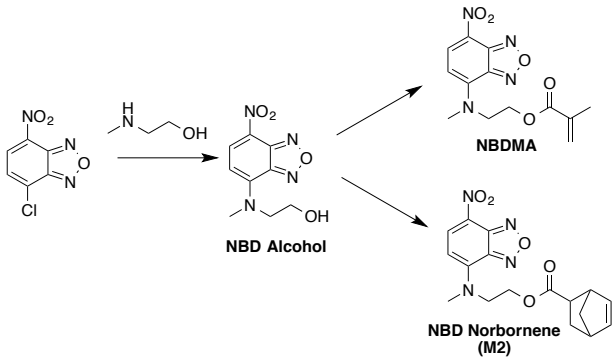
**8**. A 25 mL three neck round bottom flask was charged with **5** (0.14 g, 0.51 mmol), **7** (0.12 g, 0.52 mmol), $K_2CO_3$ (0.14 g, 1.0 mmol), KI (0.016 g, 0.10 mmol) and 2 mL dry DMF. The reaction was stirred at 40 °C overnight, diluted with brine, and extracted with $CH_2Cl_2$ 3 times. The combined organic phase was washed with water, dried over $Na_2SO_4$, filtered and concentrated *in vacuo*. The crude product was purified by flash chromatography using hexanes/ethyl acetate (2:1 v:v) as the eluent to yield **8** as a yellow solid. Yield: 0.18 g (82%). $^1$H NMR (500 MHz, $CDCl_3$): 8.23 (d, J=9 Hz, 1 H), 7.51 (d, J= 3 Hz, 1 H), 7.46 (q, J= 2, 7 Hz, 2 H), 7.11 (s, 1 H), 7.07 (q, J= 2.5, 9 Hz, 1 H), 6.97 (q, J=2.5, 7 Hz, 2 H), 6.15-6.19 (m, 2 H), 5.48 (s, 2 H), 5.25 (s, 2 H), 3.48 (s, 3 H), 3.05 (s, 1 H), 2.97 (s, 1 H), 2.11-2.17 (m, 1 H), 2.00-2.06 (m, 1 H), 1.78 (d, J=8.5 Hz, 1 H), 1.38-1.43 (m, 2 H)

**9**. A 25ml three neck round bottom flask was charged with **6** (0.11 g, 0.38 mmol), **7** (0.070 g, 0.31 mmol), $K_2CO_3$ (0.083 g, 0.60 mmol), KI (0.010 g, 0.060 mmol) and 2 mL dry DMF. The reaction was stirred at 40 °C overnight, diluted with brine, and extracted with $CH_2Cl_2$ 3 times. The combined organic phase was washed with water, dried over $Na_2SO_4$, filtered, and concentrated *in vacuo*. The crude product was purified by flash chromatography using hexanes/ethyl acetate (2:1 v:v) as the eluent to yield **9** as a yellow solid. Yield: 0.068g (82%). $^1$H NMR (300 MHz, $CDCl_3$): 8.08 (d, J=9 Hz, 1 H), 7.33 (d, J=2.7 Hz, 2 H), 7.30 (s, 1 H), 7.20 (s, 1 H), 6.99 (dd, J=2.7, 9 Hz, 1 H), 6.74 (dd, J=2.1, 6.9 Hz, 2 H), 6.12-6.16 (m, 1 H), 6.05-6.12 (m, 2H), 5.16 (s, 2H), 3.43 (s, 3 H), 2.98 (s, 1 H), 2.92 (s, 1 H), 2.06-2.12 (m, 1 H), 1.93-2.03 (m, 1 H), 1.73 (d, J= 8.1, 1 H), 1.67 (d, J=6.3 Hz, 3 H), 1.30-1.40 (m, 2 H).

**CL-3**. To a stirred solution of **8** (0.77 g, 1.8 mmol) in 12 mL THF was added 2 mL concentrated HCl. The reaction was kept at ambient temperature overnight, diluted with brine, extracted with ethyl acetate 3 times, dried over $Na_2SO_4$, filtered and concentrated *in vacuo*. The crude product was purified via flash chromatography using hexanes/ethyl acetate (5:2 v:v) to yield **PreCL-3** as a yellow solid. Yield: 0.67 g (97%). $^1$H NMR (500 MHz, MeOD): 8.16 (d, J=18 Hz, 1 H), 7.43-7.48 (m, 2 H), 7.22 (d, J=5, 1 H), 6.93-6.98 (m, 2 H), 6.83 (dd, J=5, 18 Hz, 1 H), 6.18-6.20 (m, 2 H), 5.45 (s, 1 H), 2.90-2.97 (m, 2 H),

2.25-2.31 (m, 1 H), 1.92-1.99 (m, 1 H), 1.76 (d, J=16, 1 H), 1.31-1.40 (m, 2 H).
**PreCL-3** (0.67 g, 1.8 mmol), 1,4-dibromobutane (0.18 g, 0.83 mmol), $K_2CO_3$ (0.35 g, 2.5 mmol), KI (0.020 g, 0.12 mmol) and 5 mL dry DMF were added into a 25 mL three neck round bottom flask. The reaction was stirred at 50 °C over 3 days, diluted with brine, extracted with $CH_2Cl_2$ 3 times. The combined organic phase was washed with water, dried over $Na_2SO_4$, filtered, and concentrated *in vacuo*. The crude product was purified via flash chromatography using $CH_2Cl_2$/ethyl acetate (10:1 v:v) as eluent to yield **CL-3** as a light yellow solid. Yield: 0.27 g (40%). $^1$H NMR (500 MHz, THF-$d_8$): 8.90 (s, 1 H), 8.19 (d, J=9 Hz, 1 H), 7.53-7.60 (m, 2 H), 7.37 (d, J=2.5, 1 H), 6.99 (dd, J=2.5, 9 Hz, 2 H), 6.89-6.93 (m, 2 H), 6.10-6.15 (m, 2 H), 5.45 (s, 2 H), 4.16 (s, 2 H), 3.57 (s, 2 H), 2. 92 (s, 1 H), 2.86 (s, 1 H), 2.11-2.17 (m, 1 H), 2.00-2.06 (m, 1 H), 1.97 (s, 2 H), 1.83 (d, J=8, 1 H), 1.72 (s, 2 H), 1.20-1.30 (m, 2 H). $^{13}$C NMR (500 MHz, THF-$d_8$): 173.7, 164.5, 155.0, 141.0, 139.2, 138.5, 137.0, 135.2, 128.5, 121.4, 115.7, 114.5, 113.9, 69.2, 68.3, 48.7, 46.8, 45.9, 42.7, 31.0, 26.6. HRMS calculated for $C_{46}H_{46}N_4O_{10}$ $(M + Na)^+$: 837.3106; found: 837.3097.

**CL-4**. To a stirred solution of **9** (0.060 g, 0.14 mmol) in 1 mL THF was added 0.17 mL concentrated HCl. The reaction was stirred under ambient temperature overnight, diluted with brine, extracted with ethyl acetate 3 times, dried over $Na_2SO_4$, filtered and concentrated *in vacuo*. The crude product was purified via flash chromatography using hexanes/ethyl acetate (2:1 v:v) to yield **PreCL-4** as a yellow solid. Yield: 0.056 g (96%). $^1$H NMR (500 MHz, MeOD): 8.06 (d, J=9 Hz, 1 H), 7.32-7.36 (m, 2 H), 7.09 (d, 3 Hz, 1 H), 6.78 (dd, J=3, 9 Hz, 1 H), 6.71-6.76 (m, 2 H), 6.15-6.19 (m, 2 H), 6.05 (q, J=6.5 Hz, 1 H), 2.90-2.94 (m, 2 H), 2.22-2.27 (m, 1 H), 1.89-1.95 (m, 1 H), 1.74 (s, 1 H), 1.72 (s, 1 H), 1.64 (d, J=6.5 Hz, 3 H), 1.28-1.38 (m, 2 H)
**PreCL-4** (0.056 g, 0.14 mmol), 1,4-dibromobutane (0.014 g, 0.065 mmol), $K_2CO_3$ (0.027 g, 0.20mmol), KI (0.0030 mg, 0.018 mmol) and 0.8 mL dry DMF were added into a 5 mL three neck round bottom flask. The reaction was stirred at 50 °C over 3 days, diluted with brine, and extracted with $CH_2Cl_2$ 3 times. The combined organic phase was washed with water, dried over $Na_2SO_4$, filtered, and concentrated *in vacuo*. The crude product was purified via flash chromatography using $CH_2Cl_2$/hexanes/ethyl acetate (20:2:1 v:v:v) as eluent to yield **CL-4** as a light yellow solid. Yield: 0.036 g (67%).$^1$H NMR (500 MHz, $CDCl_3$): 8.11 (dd, J=2, 9 Hz, 1 H), 7.27-7.33 (m, 2 H), 7.19-7.26 (m, 1 H), 7.15-7.18 (m, 1 H), 6.77-6.82 (m, 1 H), 6.69-6.75 (m, 2 H), 6.14-6.19 (m, 1 H), 6.06-6.13 (m, 2 H), 3.95-4.07 (m, 2 H), 2.98 (s, 1 H), 2.93 (s, 1 H), 2.06-2.13 (m, 1 H), 1.93-2.01 (m, 1 H), 1.92 (s, 2 H), 1.73 (d, J=8 Hz, 1 H), 1.66 (d, J= 6.5 Hz, 3 H), 1.31-1.40 (m, 2 H). $^{13}$C NMR (500MHz, $CDCl_3$): 173.9, 163.6, 153.9, 143.0, 140.2, 138.6, 136.0, 128.0, 121.8, 115.9, 113.6, 112.5, 71.9, 68.1, 47.4, 46.4, 45.7, 41.7, 30.7, 25.6, 23.5. HRMS calculated for $C_{48}H_{51}N_4O_{10}$ $(M+H)^+$: 843.3600; found: 843.3616.




**NBD alcohol** was synthesized following published methods.[2]

NBD-Norbornene **M2**: **NBD alcohol** (0.16 g, 0.67 mmol), exo-5-norbornene carboxylic acid (0.10 g, 0.72 mmol), DCC (0.17 g, 0.83mmol), DMAP (0.017 g, 0.14 mmol) were dissolved in 10 mL dry $CH_2Cl_2$ in a 25 mL three neck round bottom flask. The reaction was stirred at ambient temperature overnight and then diluted with $CH_2Cl_2$, washed with water 3 times, dried over $Na_2SO_4$, and concentrated *in vacuo*. The crude product was purified via flash chromatography using hexanes/ethyl acetate (3:1 v:v) as eluent, followed by recrystallization in ethanol to yield **M2** as an orange solid. Yield: 0.13 g (54%). $^1$H NMR (500 MHz, $CDCl_3$): 8.47 (d, J=9 Hz, 1 H), 6.19 (d, J=9 Hz, 1 H), 6.09-6.12 (m, 1 H), 6.01-6.04 (m, 1 H), 4.42-4.49 (m, 4 H), 3.48 (s, 3 H), 2.87 (d, J=1.5 Hz, 2 H), 2.05-2.09 (m, 1 H), 1.72-1.77 (m, 1 H), 1.34-1.38 (m, 1 H), 1.24-1.32 (m, 2 H). $^{13}$C NMR (500MHz, $CDCl_3$):175.9, 145.3, 144.7, 144.6, 138.2, 135.4, 135.1, 123.5, 101.7, 61.7, 54.2, 46.5, 46.3, 43.0, 41.9, 41.6, 30.3.

**NBDMA**: **NBD alcohol** (0.29 g, 0.0012 mol), freshly distilled methacryloyl chloride (0.20 g, 0.0019 mol) and dry pyridine (0.15 g, 0.0019 mol) were dissolved in 15 mL dry $CH_2Cl_2$ in a 50 mL three neck round bottom flask. The reaction was stirred at ambient temperature overnight and then quenched by addition of water. The mixture was washed with $CH_2Cl_2$ for 3 times, dried over $Na_2SO_4$, and concentrated *in vacuo*. The crude product was purified via flash chromatography using $CH_2Cl_2$ as eluent to yield **NBDMA** as an orange solid. Yield: 0.22 g (60%). NMR results are in consistence with previous publications.[3]

Poly (poly(ethylene glycol) methyl ether methacrylate-co-NBDMA) (**PEGNBD**). Poly(ethylene glycol) methyl ether methacrylate (0.50 g), NBDMA (0.025 g, 5% equiv by weight), AIBN (5.0 mg, 1% equiv by weight) and DMF (5 mL) were added into a 25 mL round bottom flask. The mixture was purged with argon for 20 min, and then warmed to 60 °C and stirred overnight. The cooled mixture was precipitated into copious hexane/ether mixture (1:1 v:v), centrifuged, and crude product was collected by decanting. The crude product was redissolved in 10 mL $CH_2Cl_2$, precipitated into hexane, and centrifuged. After decanting the remaining sticky liquid was dried in vacuum oven for 3 days to yield an orange gum-like liquid. Yield: 0.26 g (50%). $^1$H NMR (500 MHz, $CDCl_3$): 8.45-8.65 (broad), 6.28-6.50 (broad), 4.4-4.7 (broad), 3.9-4.4 (broad), . Mn 35367, Mw 60760, PDI 1.72.

**General conditions for gelation.**

*Organogels.*

A solution of Grubbs 2nd generation catalyst (2.4 mg) in 0.20 mL $CH_2Cl_2$ was added into a 20 mL vial charged with 0.20 mL of a DMF solution of monomers (40mg) and the crosslinker. The vial was swirled and gelation was observed in no more than 30 seconds. All gels were purified using Soxhlet extraction with $CH_2Cl_2$ over two days and dried in vacuum. In each irradiation experiment in which 2 mg of xerogel was used, the form factor of the gels was a thin sheet of thickness of approximately 1 mm.

*Trapping of water soluble polymer into the hydrogel.*

Grubbs 2nd generation catalyst (1.8 mg) was added into a 20 mL vial charged with 0.30 mL $CH_2Cl_2$ solution of **M3** (50 mg), **CL-4** (2.7 mg) and **PEGNBD** (1.0 mg). The vial was swirled and gelation was observed in about 20 minutes. All gels were purified using Soxhlet extraction with $CH_2Cl_2$ over two days and dried in vacuum.

## 4. Other Experimental Details

*Rheological measurements.*

Each set of measurements started with swelling 8.0 mg gel in benzene in a vial for at least 1 hr before measurement. All gel in the vial was transferred onto the rheometer and rheological data was measured. After one data point was obtained, all substance from the sample holder of the rheometer was collected into a quartz cuvette. Gel in the cuvette was either (1) irradiated for a certain amount of time with a blak-ray B-100A high intensity UV lamp, or (2) kept in the dark for certain amount of time in control experiments. Then all the substance in the cuvette was transferred back onto the rheometer to collect another data point. All experiments were performed at ambient temperature. Gel was immersed throughout the experiments and extra benzene was added if there was not enough benzene covering the gel.

*Photodegradation experiments monitored by visual inspection or fluorescence spectroscopy.*

Each set of experiments started with immersing about 2.0 mg of organogel in about 3 mL benzene in a quartz cuvette equipped with a small magnetic stir bar. The gel was swollen for at least 30 minutes, then the sample in the cuvette was irradiated by UV with the micro stir bar stirring at about 100 rpm. After a certain amount of time, the cuvette was left in the dark with no disturbance to allow the gel to settle to the bottom of the cuvette, after which emission spectra of the liquid portion of the sample was collected using excitation at 465nm. If further irradiation was needed, the cuvette containing the sample was irradiated again and another spectra was obtained following a similar procedure. Cuvettes were always located at the same position relative to the UV lamp. In control experiments, all conditions were the same other than that the samples were kept in the dark.

*Photorelease of guest from the hydrogel*

Each set of experiments started with immersing about 1.0 mg of hydrogel that had trapped fluorescent polymer guest in about 3 mL water in a quartz cuvette equipped with a small

magnetic stir bar. Other conditions are the same as in photodegradation experiments monitored by visual inspection or fluorescence spectroscopy.

## 5. Additional Figures

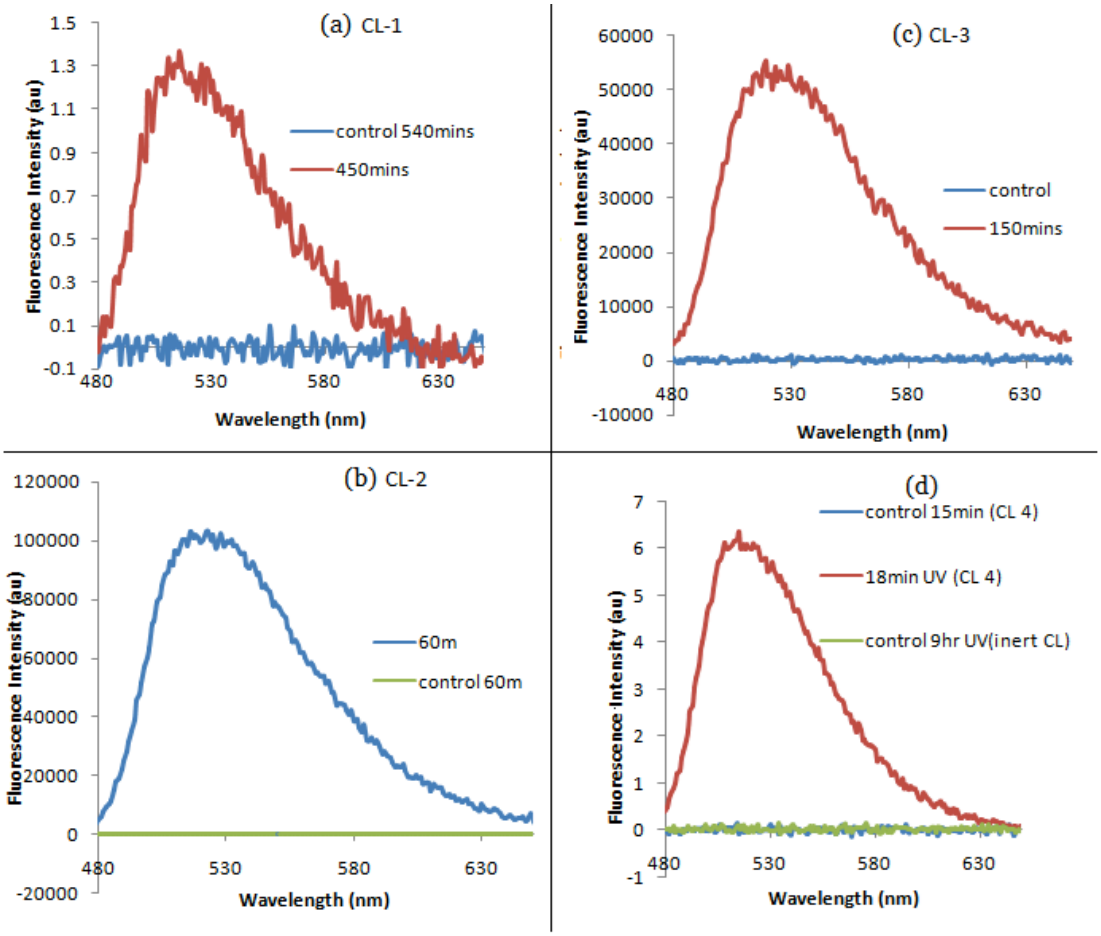
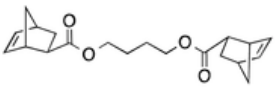
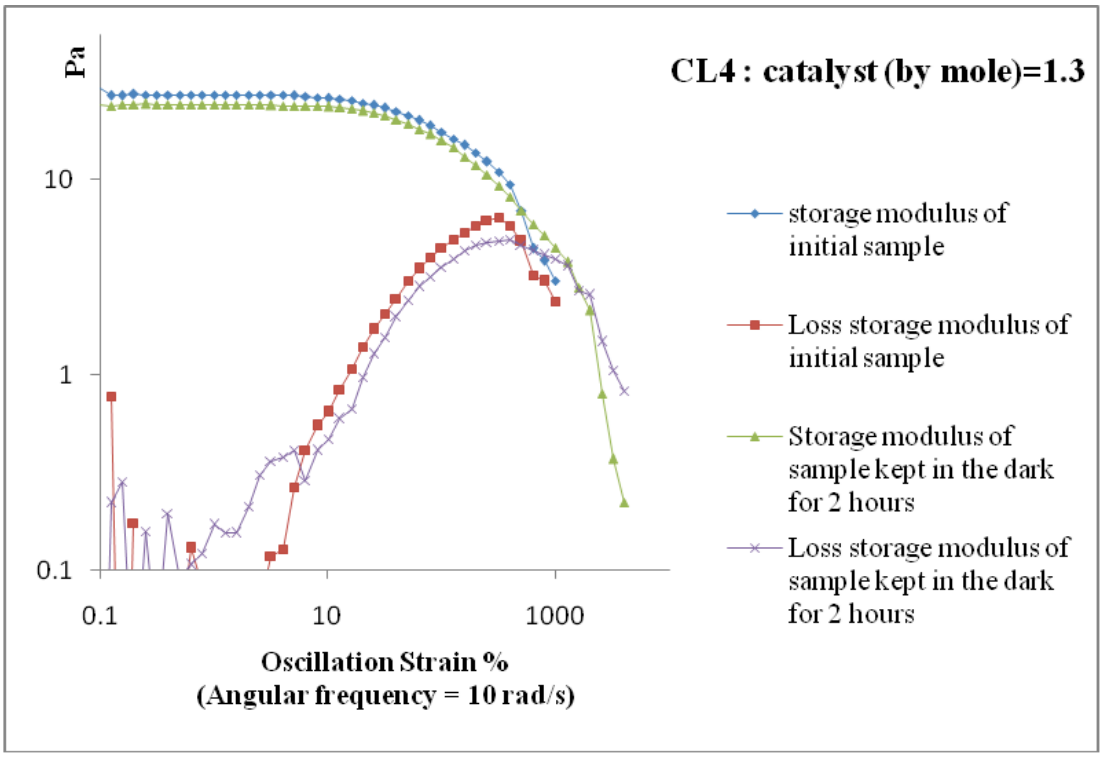



Figure S1. Control experiments of the dissolution of fluorescent polymer chains. (a) & (d) were obtained with Cary Eclipse Fluorescence Spectrophotometer. (b) & (c) were obtained with PTI Quantum Master 4 Fluorescence Spectrophotometer. (a) Red line: fluorescence emission of the photodegraded gel (with **CL-1**) solution irradiated for 450mins; blue line: fluorescence emission of the gel (with **CL-1**) sample in the dark after 540mins. (b) Blue curve: fluorescence emission of the gel (with **CL-2**) solution irradiated for 450mins; cyan curve: fluorescence emission of the gel (with **CL-2**) sample in the dark after 540mins. (c) Red curve: fluorescence emission of the gel (with **CL-3**) solution irradiated for 150mins; blue line: fluorescence emission of the gel (with **CL-1**) sample in the dark after 150mins. (d) Red curve: fluorescence emission of the gel (with **CL-4**) solution irradiated for 18mins; blue curve: fluorescence emission of the gel (with **CL-4**) solution in the dark for 15mins; cyan curve: fluorescence of the control gel (with **inert CL**) solution irradiated for 9 hrs.



**Inert CL**



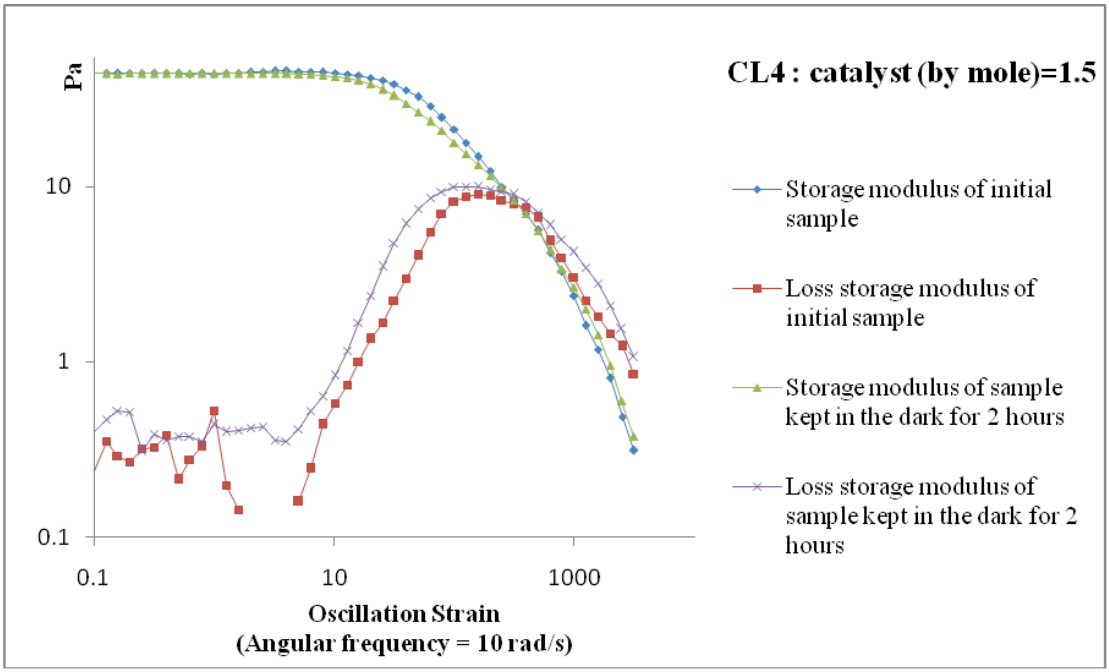




Figure S2. Rheology experiments of unirradiated gels. Dynamic storage modulus (G') and loss modulus (G'') versus strain for the gels.

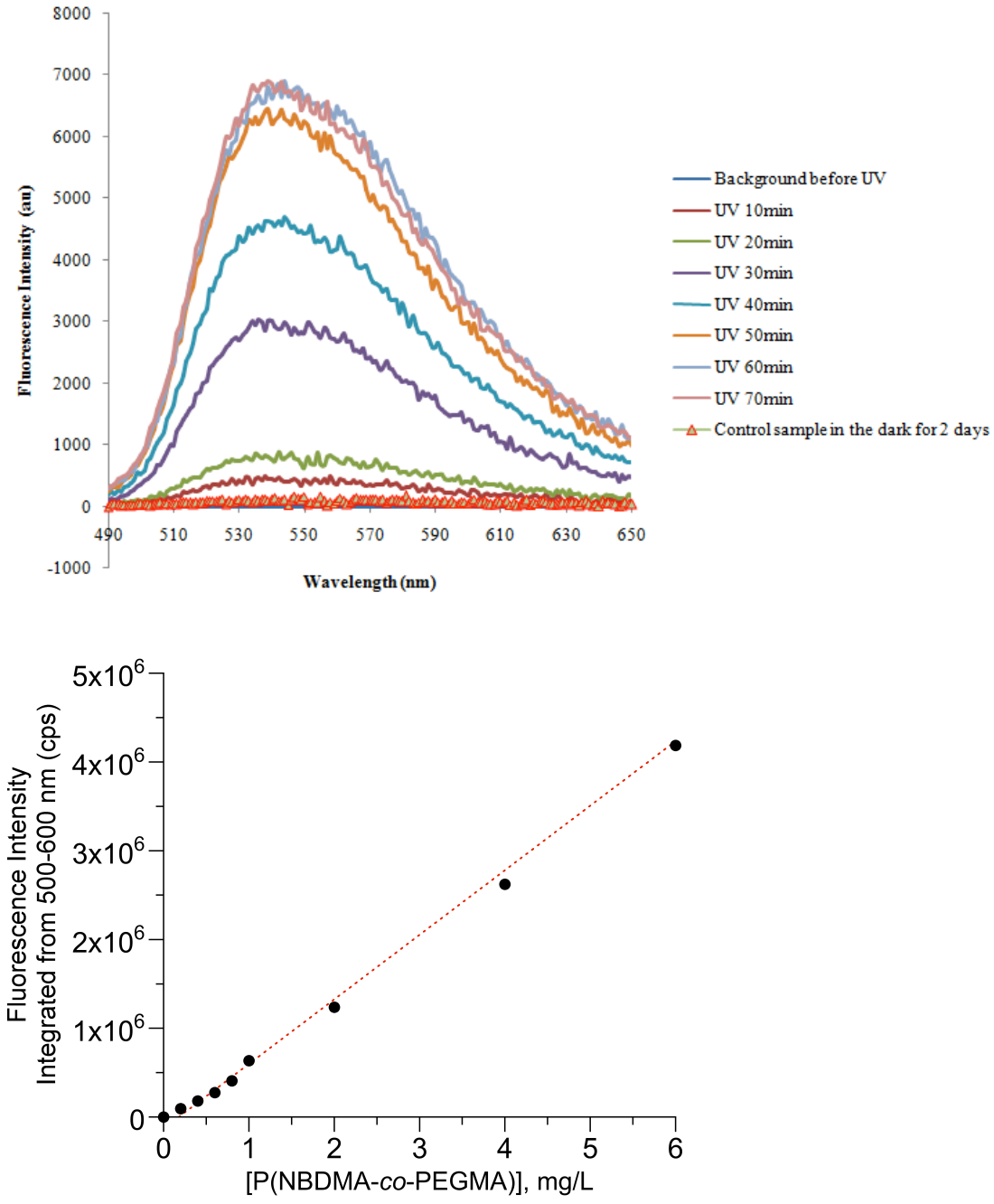



Figure S3. UV-induced release of fluorescent NBD-labeled methacrylic polymer **PEGMA** from hydrogel prepared with hydrophilic monomer **M3** and crosslinker **CL-4**. According to the linear calibration curve, the final concentration of released fluorescent polymer was 0.9 mg/L.

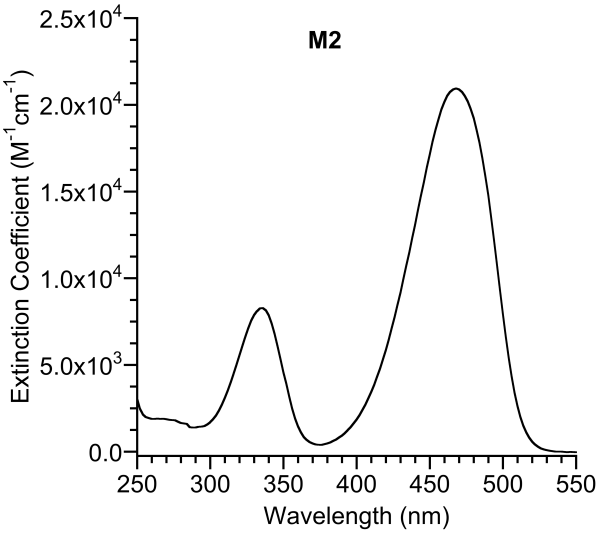
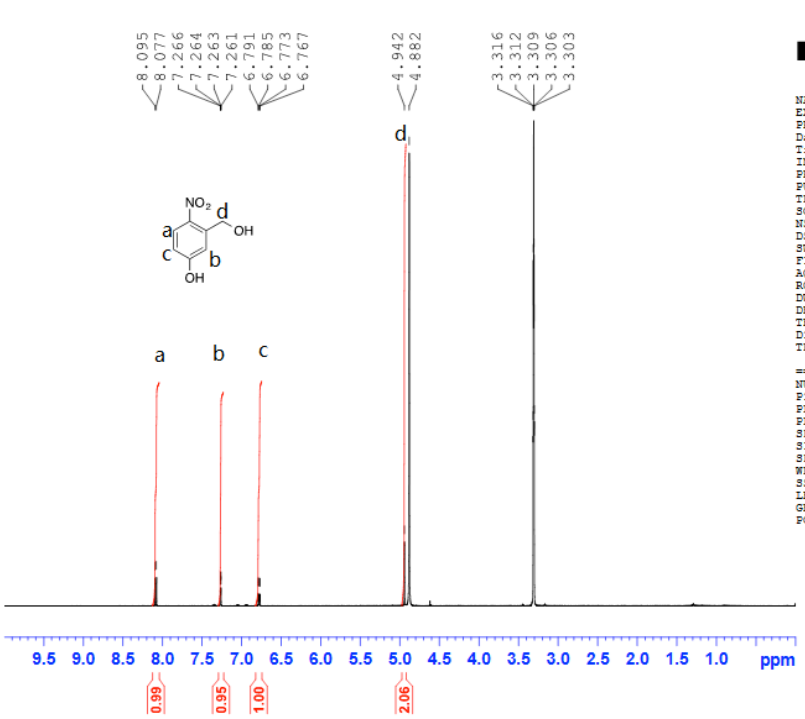



Figure S4. Absorbance spectrum of fluorescent monomer **M2**.

## 6. NMR Spectra

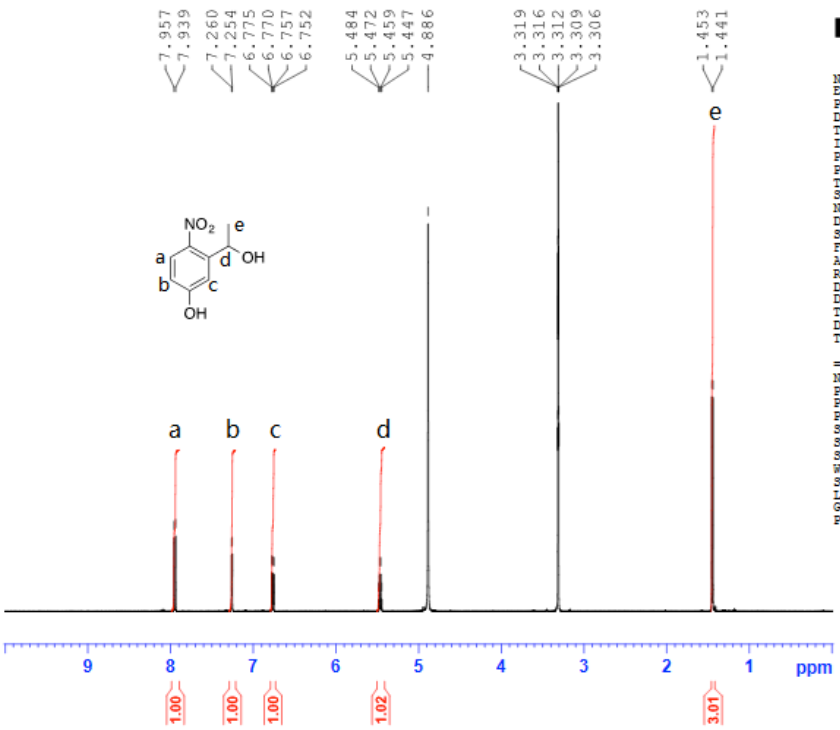
**1**




```
NAME                xhu02-003
EXPNO                       1
PROCNO                      1
Date_            20140711
Time                    14.51
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                 MeOD
NS                        32
DS                         2
SWH             10000.000 Hz
FIDRES          0.152588 Hz
AQ              3.2768500 sec
RG                       203
DW                  50.000 usec
DE                   6.50 usec
TE                   294.6 K
D1              1.50000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                   20.00 usec
PL1                   1.00 dB
PL1W         17.75783539 W
SFO1         500.1318364 MHz
SI                     65536
SF           500.1300113 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                    1.00
```

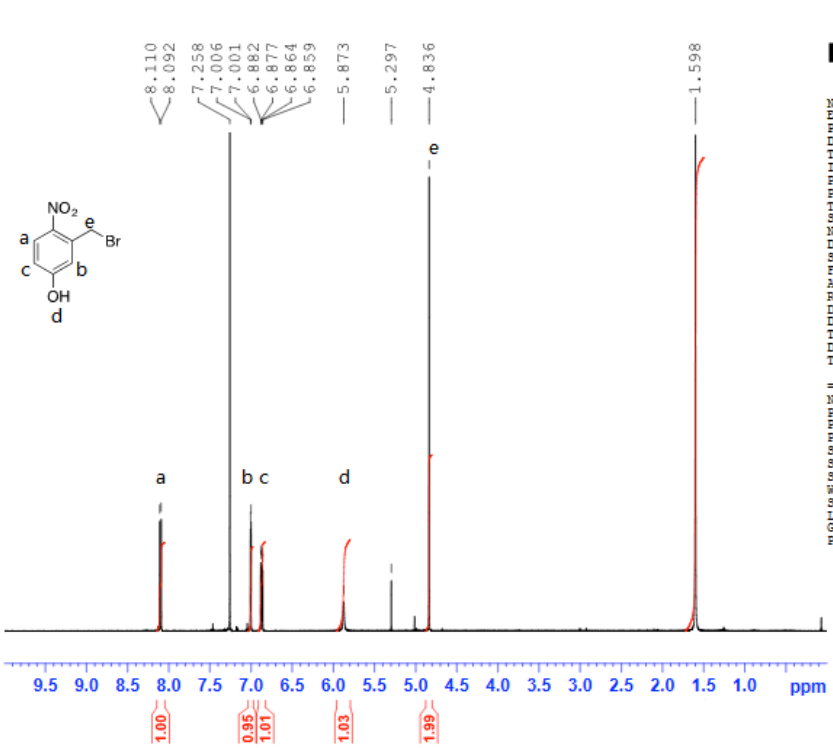
**2**




```
NAME                xhu02-004
EXPNO                       1
PROCNO                      1
Date_            20140711
Time                    14.59
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                 MeOD
NS                        32
DS                         2
SWH             10000.000 Hz
FIDRES          0.152588 Hz
AQ              3.2768500 sec
RG                       203
DW                  50.000 usec
DE                   6.50 usec
TE                   294.5 K
D1              1.50000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                   20.00 usec
PL1                   1.00 dB
PL1W         17.75783539 W
SFO1         500.1318364 MHz
SI                     65536
SF           500.1300097 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                    1.00
```

**3**

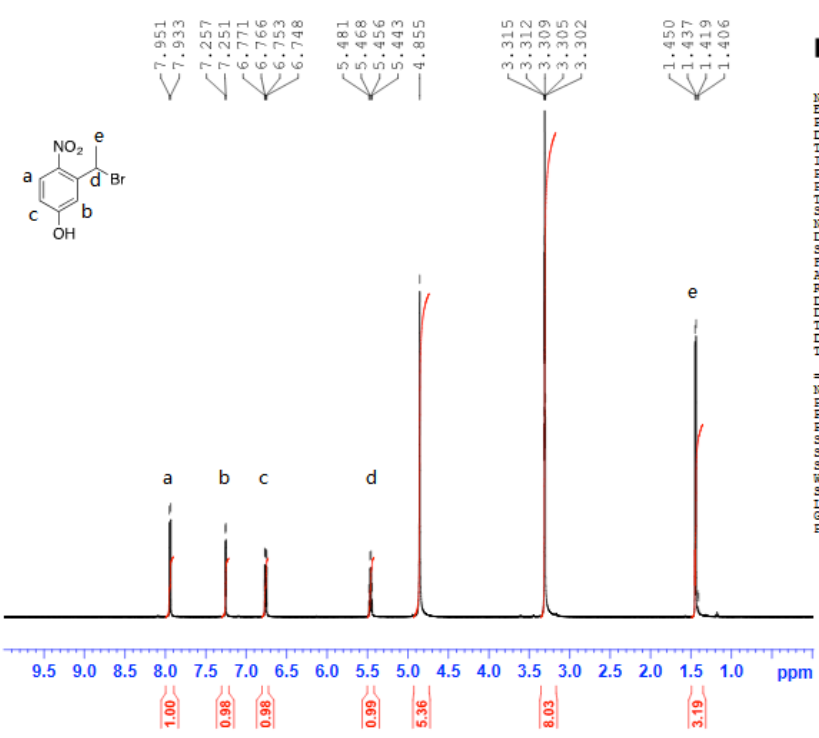



BRUKER

```
NAME                xhu02-007
EXPNO                       1
PROCNO                      1
Date_               20140714
Time                   17.22
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                10000.000 Hz
FIDRES             0.152588 Hz
AQ                 3.2768500 sec
RG                       203
DW                  50.000 usec
DE                   6.50 usec
TE                     300.0 K
D1                 2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    20.00 usec
PL1                    1.00 dB
PL1W              17.75783539 W
SFO1              500.1318364 MHz
SI                       65536
SF                500.1300143 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.00
```

**4**

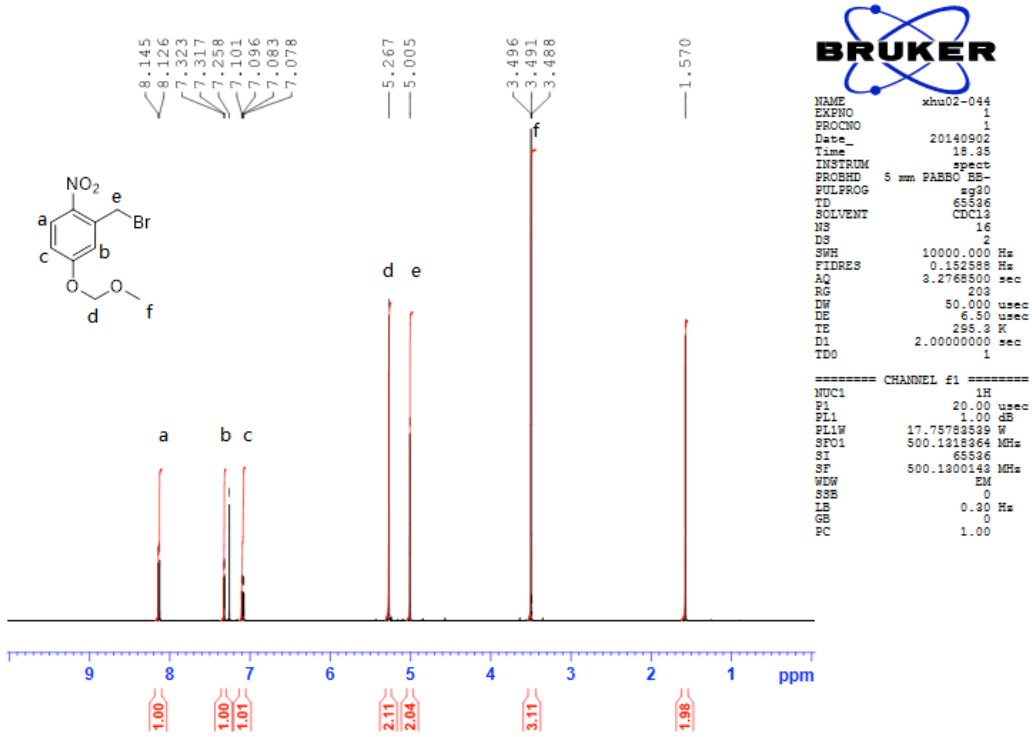



BRUKER

```
NAME                xhu02-029
EXPNO                       1
PROCNO                      1
Date_               20140819
Time                   11.14
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                MeOD
NS                        16
DS                         2
SWH                10000.000 Hz
FIDRES             0.152588 Hz
AQ                 3.2768500 sec
RG                       203
DW                  50.000 usec
DE                   6.50 usec
TE                     297.2 K
D1                 2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    20.00 usec
PL1                    1.00 dB
PL1W              17.75783539 W
SFO1              500.1318364 MHz
SI                       65536
SF                500.1300115 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.00
```

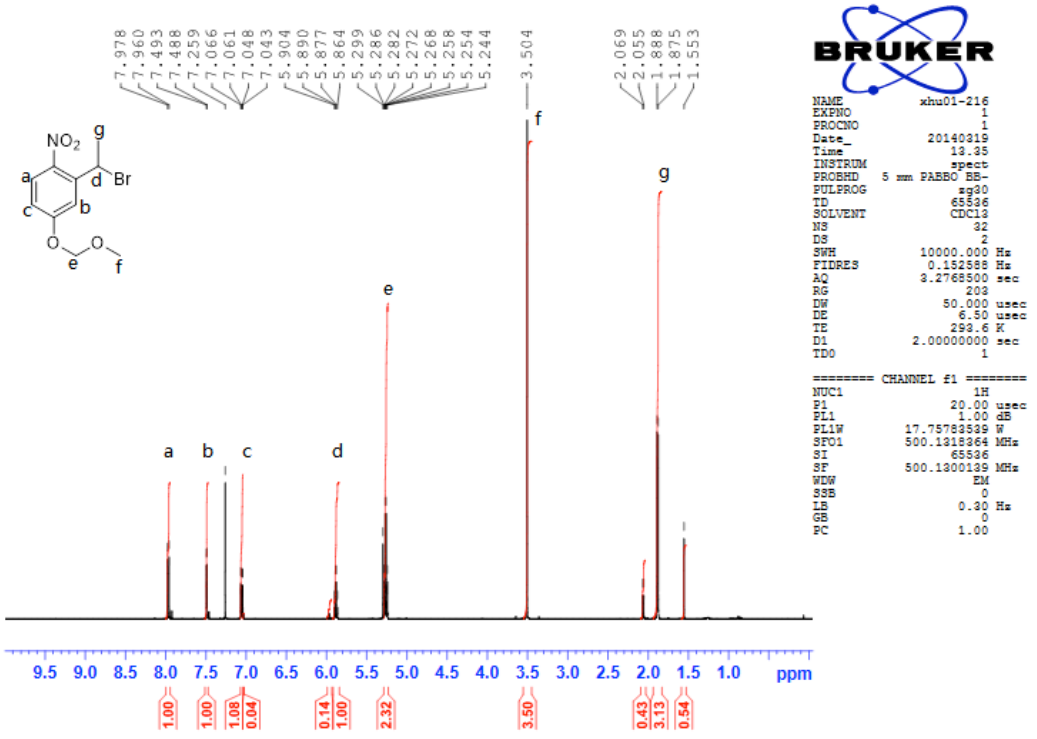
## 5




```
NAME                xhu02-044
EXPNO                       1
PROCNO                      1
Date_            20140902
Time                    18.35
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH             10000.000 Hz
FIDRES              0.152588 Hz
AQ              3.2768500 sec
RG                       203
DW                  50.000 usec
DE                    6.50 usec
TE                  295.3 K
D1              2.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                    20.00 usec
PL1                    1.00 dB
PL1W           17.75783539 W
SFO1           500.1318364 MHz
SI                     65536
SF             500.1300143 MHz
WDW                       EM
SSB                        0
LB                  0.30 Hz
GB                         0
PC                      1.00
```

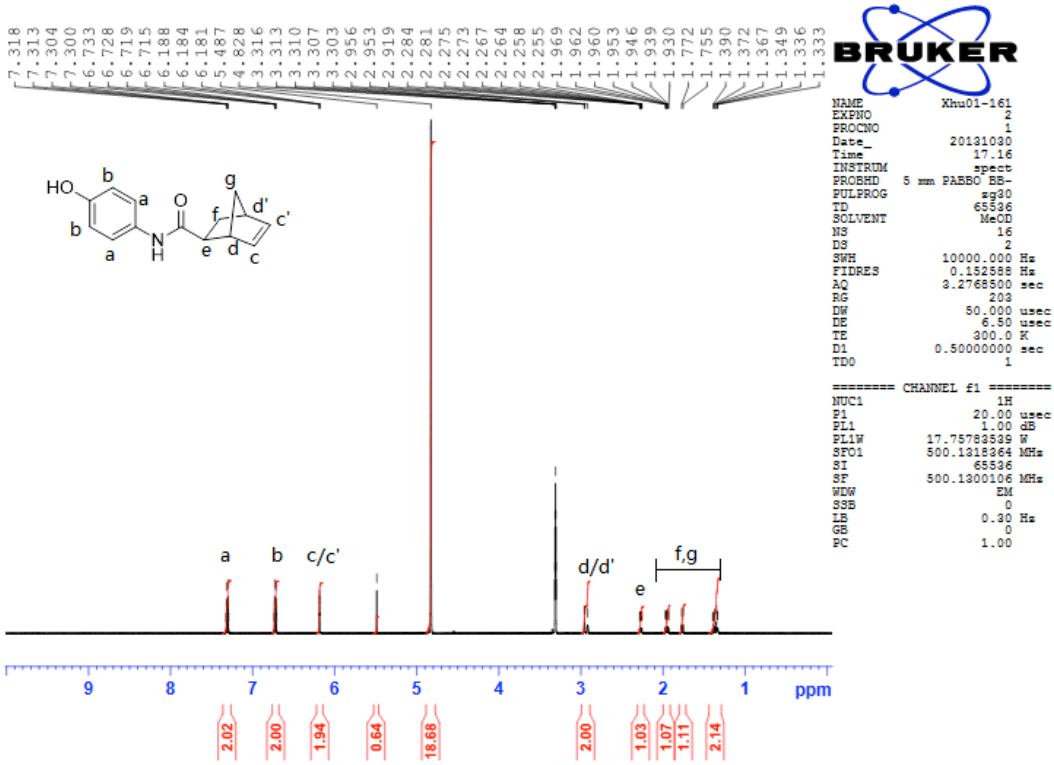
## 6




```
NAME                xhu01-216
EXPNO                       1
PROCNO                      1
Date_            20140319
Time                    13.35
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        32
DS                         2
SWH             10000.000 Hz
FIDRES              0.152588 Hz
AQ              3.2768500 sec
RG                       203
DW                  50.000 usec
DE                    6.50 usec
TE                  293.6 K
D1              2.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                    20.00 usec
PL1                    1.00 dB
PL1W           17.75783539 W
SFO1           500.1318364 MHz
SI                     65536
SF             500.1300139 MHz
WDW                       EM
SSB                        0
LB                  0.30 Hz
GB                         0
PC                      1.00
```

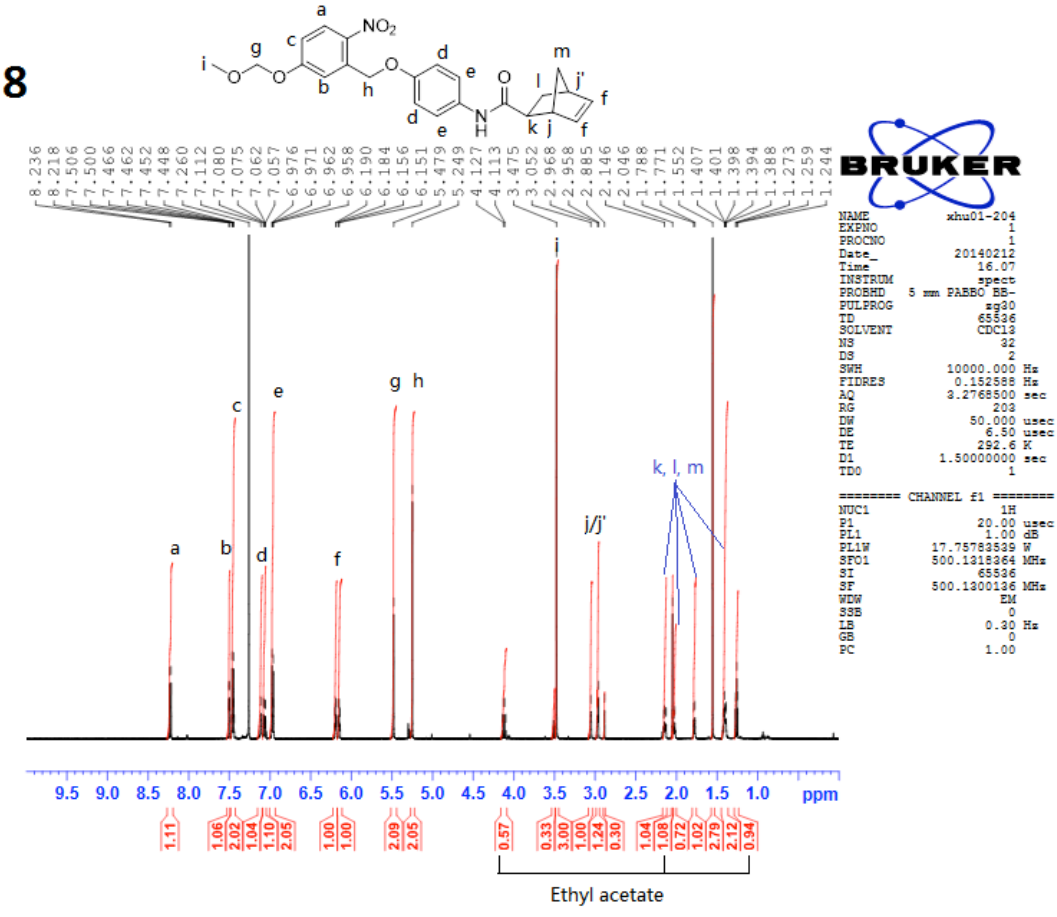
**7**




```
NAME                Xhu01-161
EXPNO                       2
PROCNO                      1
Date_              20131030
Time                   17.16
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                MeOD
NS                         16
DS                          2
SWH              10000.000 Hz
FIDRES               0.152588 Hz
AQ                3.2768500 sec
RG                        203
DW                   50.000 usec
DE                    6.50 usec
TE                    300.0 K
D1               0.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    20.00 usec
PL1                    1.00 dB
PL1W            17.75783539 W
SFO1           500.1318364 MHz
SI                     65536
SF             500.1300106 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```

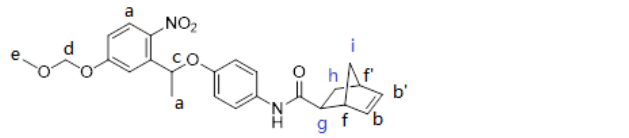
**8**

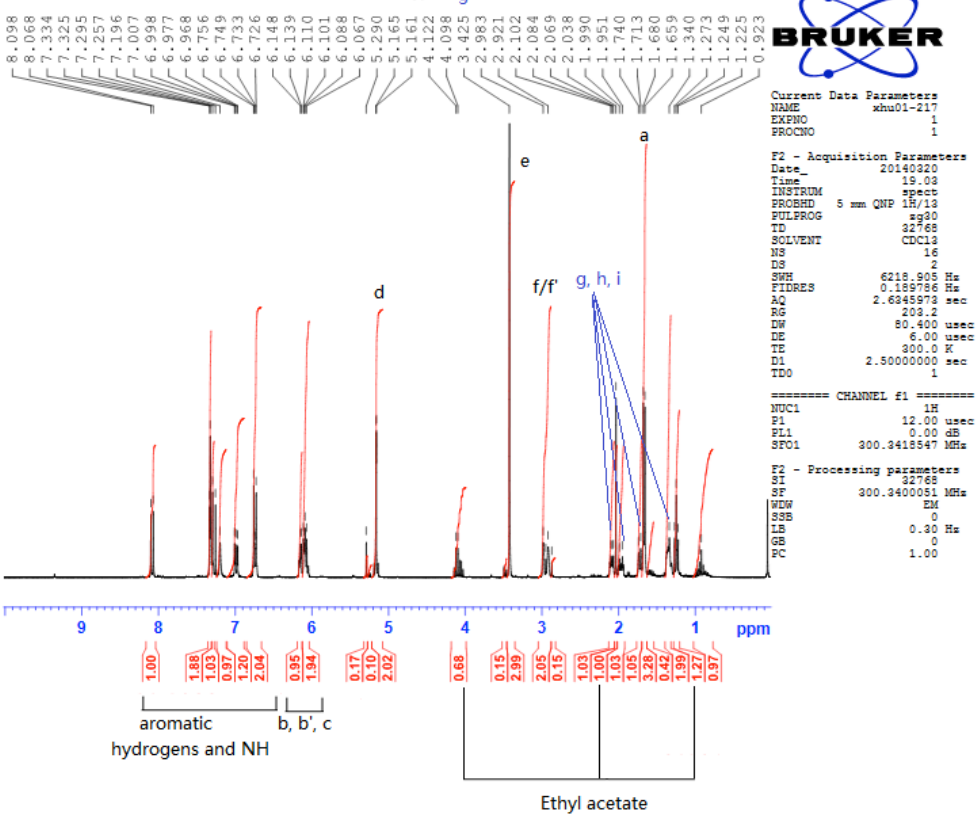



```
NAME               xhu01-204
EXPNO                       1
PROCNO                      1
Date_              20140212
Time                   16.07
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         32
DS                          2
SWH              10000.000 Hz
FIDRES               0.152588 Hz
AQ                3.2768500 sec
RG                        203
DW                   50.000 usec
DE                    6.50 usec
TE                    292.6 K
D1               1.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    20.00 usec
PL1                    1.00 dB
PL1W            17.75783539 W
SFO1           500.1318364 MHz
SI                     65536
SF             500.1300136 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```

**9**


a
NO2
e
d
c
O
i
h
f'
b'
a
O
N
H
g
f
b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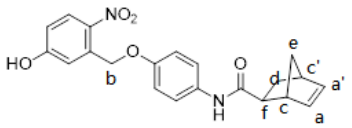
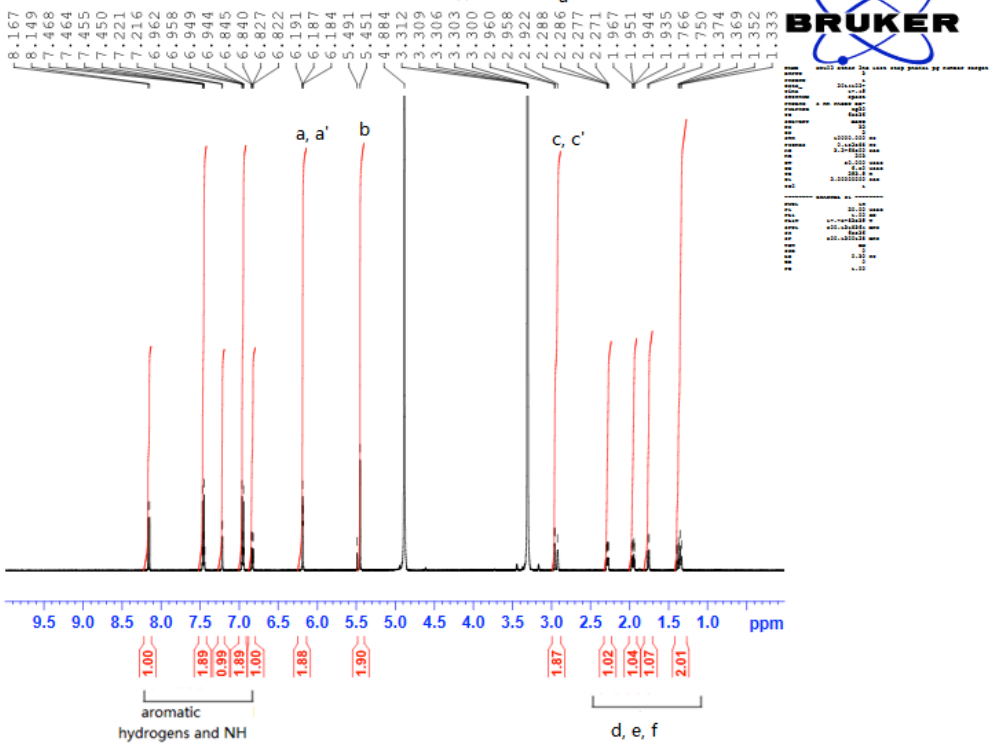
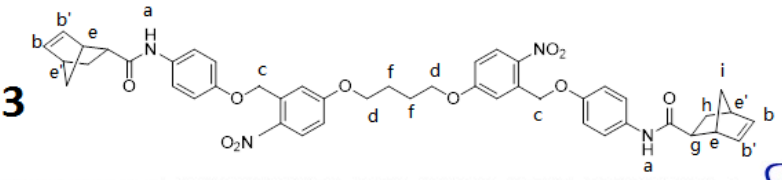
Current Data Parameters
NAME xhu01-217
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140320
Time 19.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6218.905 Hz
FIDRES 0.189786 Hz
AQ 2.6345973 sec
RG 203.2
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.50000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 300.3418547 MHz
F2 - Processing parameters
SI 32768
SF 300.3400051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
a
e
d
f/f'
g, h, i
9
8
7
6
5
4
3
2
1
ppm
1.00
1.88
1.03
0.97
1.20
2.04
0.95
1.94
0.17
0.10
2.02
0.68
0.15
2.99
2.05
0.15
1.03
1.00
1.03
1.05
3.28
0.42
1.99
1.27
0.97
aromatic
hydrogens and NH
b, b', c
Ethyl acetate

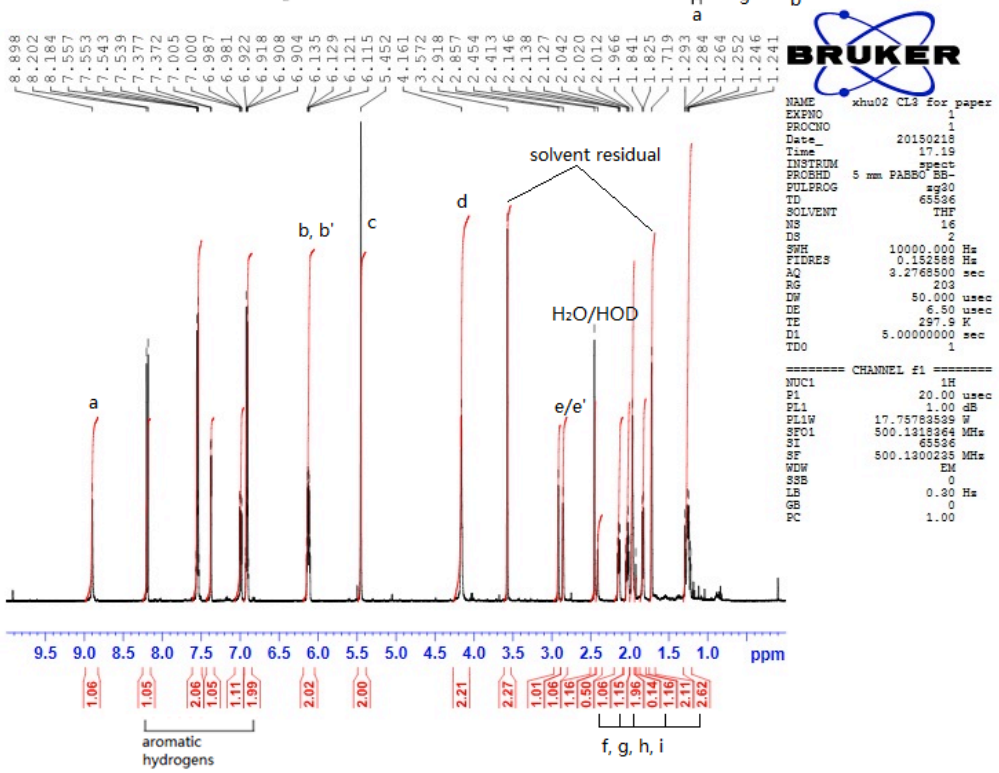
**PreCL-3**

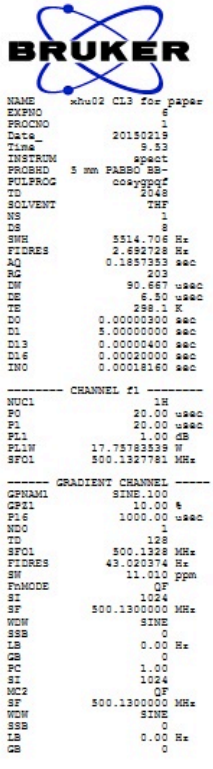

NO2
HO
b
O
e
c'
a'
d
f
c
a
O
N
H


BRUKER
a, a'
b
c, c'
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.00
1.89
0.99
1.89
1.00
1.88
1.90
1.87
1.02
1.04
1.07
2.01
aromatic
hydrogens and NH
d, e, f

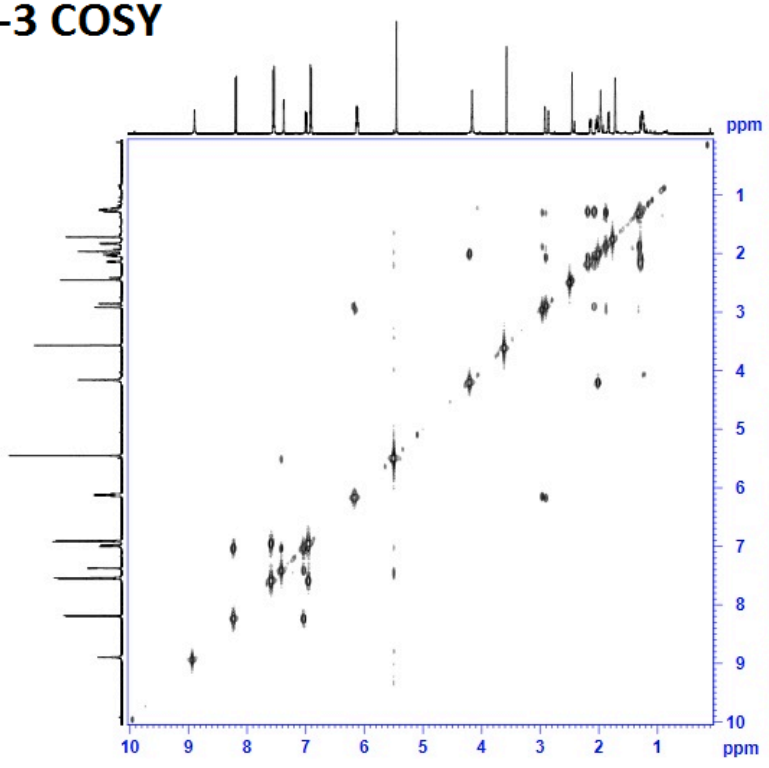
## CL-3


a
b'
e
b
e'
c
f
d
i
h
e'
b
g
e
b'
d
f
c
a
$NO_2$
$O_2N$


8.898 8.202 8.184 7.557 7.553 7.543 7.539 7.377 7.372 7.005 7.000 6.987 6.981 6.922 6.918 6.908 6.904 6.135 6.129 6.121 6.115 5.452 4.161 3.572 2.918 2.857 2.454 2.413 2.146 2.138 2.127 2.042 2.020 2.012 1.966 1.841 1.825 1.719 1.293 1.284 1.264 1.252 1.246 1.241
solvent residual
b, b'
c
d
$H_2O$/HOD
a
e/e'
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.06 1.05 2.06 1.05 1.11 1.99 2.02 2.00 2.21 2.27 1.01 1.06 1.16 0.50 1.06 1.15 1.96 0.14 1.16 2.11 2.62
aromatic hydrogens
f, g, h, i

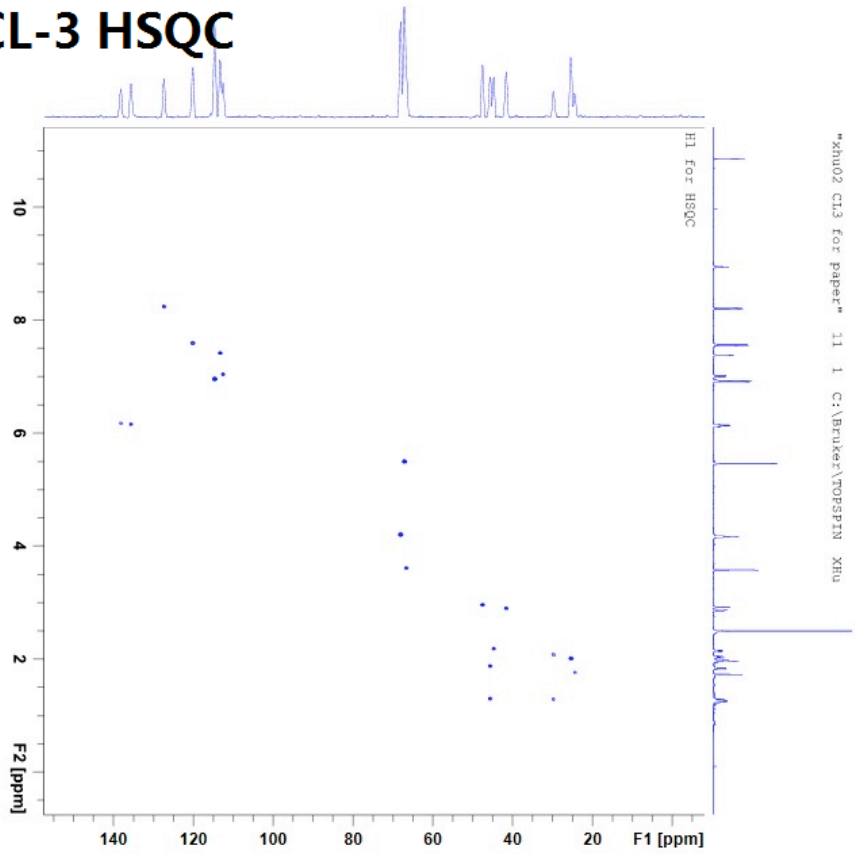

BRUKER
NAME xhu02 CL3 for paper
EXPNO 1
PROCNO 1
Date_ 20150218
Time 17.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT THF
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 297.9 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 20.00 usec
PL1 1.00 dB
PL1W 17.75783539 W
SFO1 500.1318364 MHz
SI 65536
SF 500.1300235 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

## CL-3 COSY

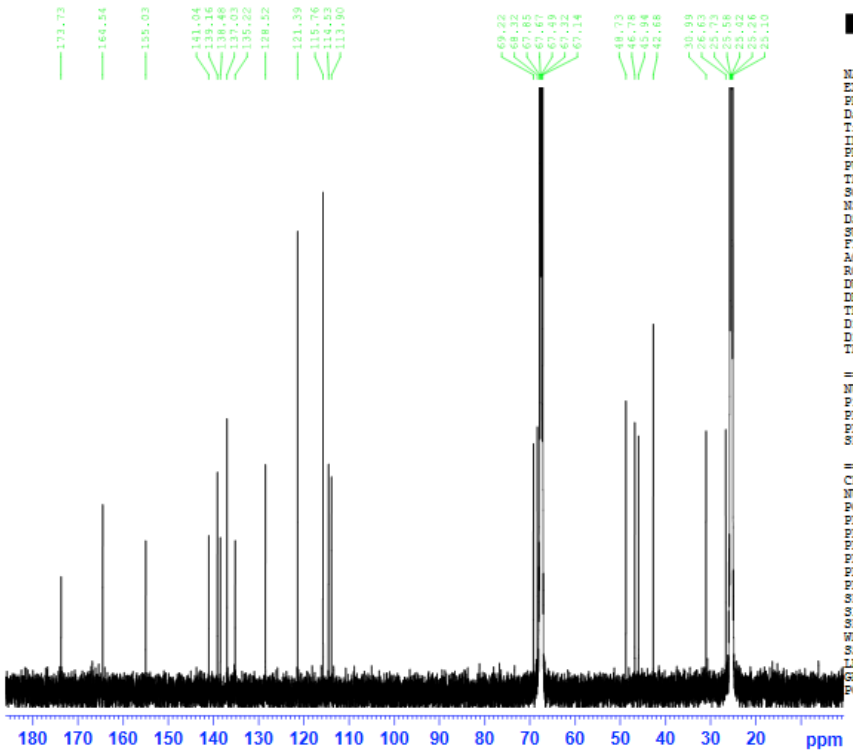

ppm
1 2 3 4 5 6 7 8 9 10
10 9 8 7 6 5 4 3 2 1 ppm


BRUKER
NAME xhu02 CL3 for paper
EXPNO 6
PROCNO 1
Date_ 20150219
Time 9.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG cosygpqf
TD 2048
SOLVENT THF
NS 1
DS 8
SWH 5514.706 Hz
FIDRES 2.692728 Hz
AQ 0.1857353 sec
RG 203
DW 90.667 usec
DE 6.50 usec
TE 298.1 K
D0 0.00000300 sec
D1 5.00000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00018160 sec
-------- CHANNEL f1 --------
NUC1 1H
P0 20.00 usec
P1 20.00 usec
PL1 1.00 dB
PL1W 17.75783539 W
SFO1 500.1327781 MHz
------ GRADIENT CHANNEL -----
GPNAM1 SINE.100
GPZ1 10.00 %
P16 1000.00 usec
NDO 1
TD 128
SFO1 500.13328 MHz
FIDRES 43.020374 Hz
SW 11.010 ppm
FnMODE QF
SI 1024
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

## CL-3 HSQC



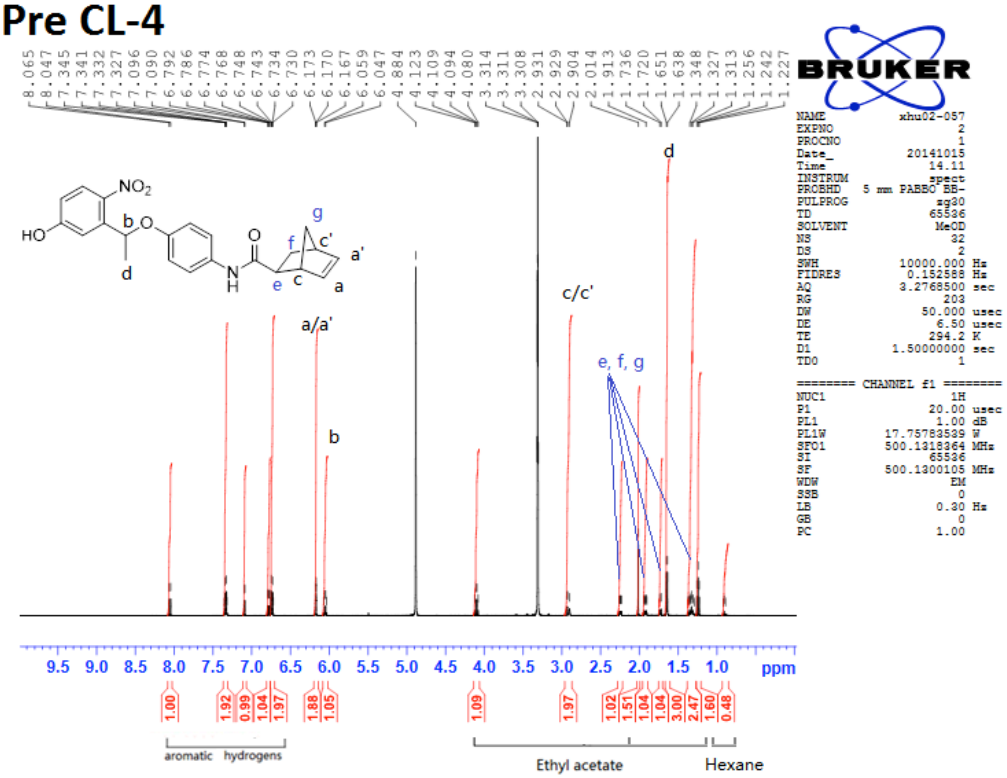

## CL-3 C13



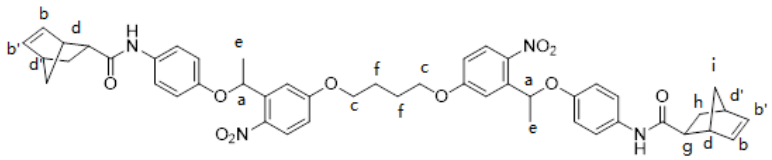

BRUKER

```
NAME          xhu02 CL3 for paper
EXPNO                           2
PROCNO                          1
Date_                    20150219
Time_                        4.50
INSTRUM                     spect
PROBHD   5 mm PABBO BB-
PULPROG                  zgpg30
TD                            65536
SOLVENT                         THF
NS                             3600
DS                                4
SWH                     29761.904 Hz
FIDRES                      0.454131 Hz
AQ                      1.1010548 sec
RG                              203
DW                           16.800 usec
DE                            6.50 usec
TE                          297.8 K
D1                     10.00000000 sec
D11                     0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          13C
P1                            9.50 usec
PL1                           0.00 dB
PL1W                 89.92553711 W
SFO1                125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                        80.00 usec
PL2                           1.00 dB
PL12                         13.04 dB
PL13                         16.80 dB
PL2W                17.75783539 W
PL12W                1.11017132 W
PL13W                0.46707872 W
SFO2                500.1320005 MHz
SI                            65536
SF                  125.7576527 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40
```

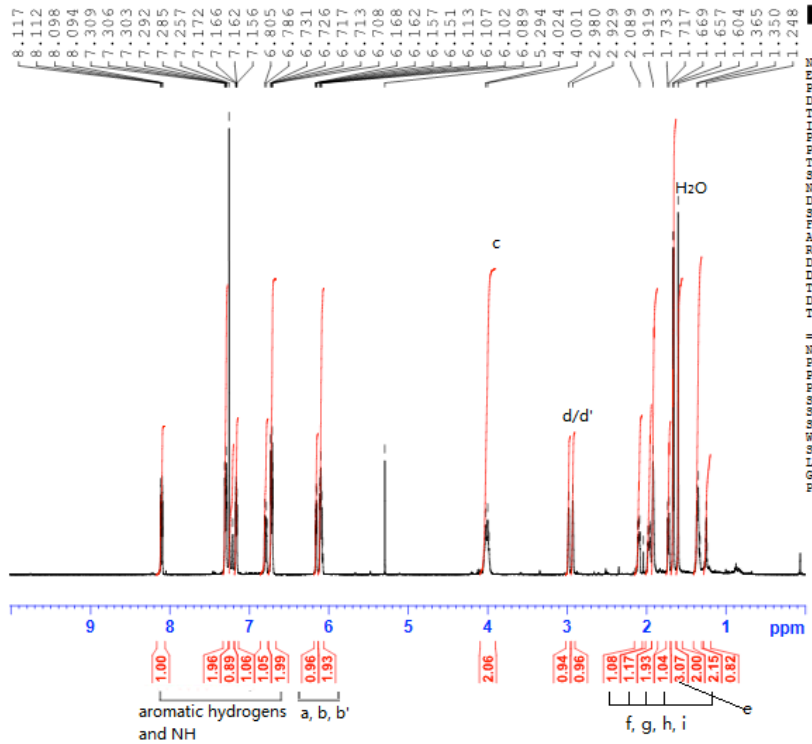
## Pre CL-4




```
NAME                xhu02-057
EXPNO                       2
PROCNO                      1
Date_              20141015
Time                    14.11
INSTRUM                 spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                 MeOD
NS                        32
DS                         2
SWH               10000.000 Hz
FIDRES            0.152588 Hz
AQ                3.2768500 sec
RG                       203
DW                  50.000 usec
DE                   6.50 usec
TE                  294.2 K
D1                1.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                   20.00 usec
PL1                   1.00 dB
PL1W             17.75783529 W
SFO1            500.1318364 MHz
SI                     65536
SF              500.1300105 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```

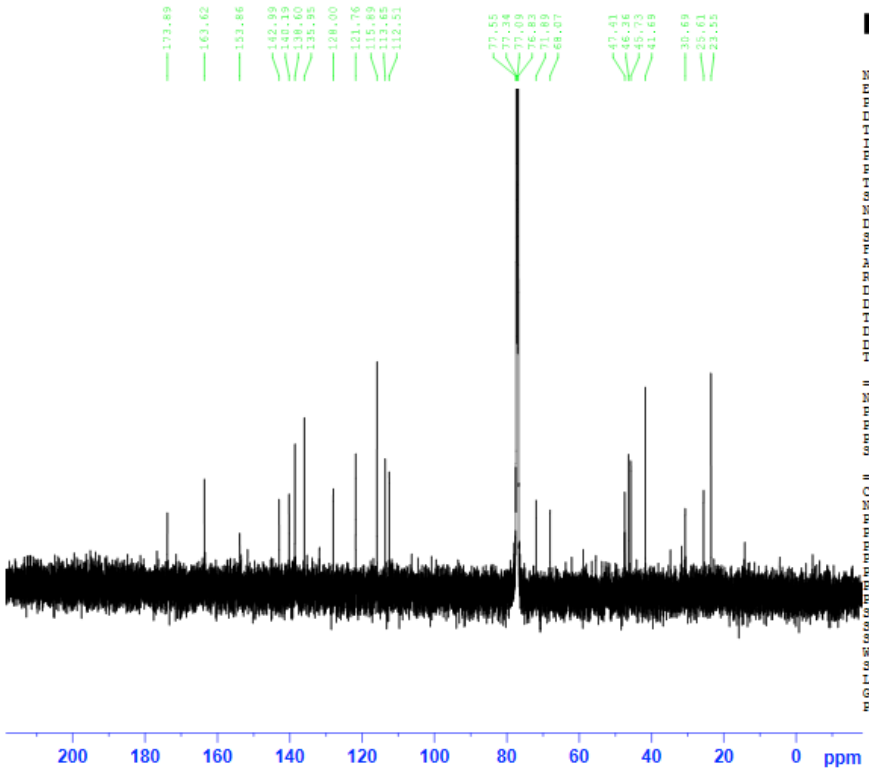



## CL-4




```
NAME                xhu02-046
EXPNO                       2
PROCNO                      1
Date_              20140829
Time                    17.06
INSTRUM                 spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                 CDCl3
NS                        16
DS                         2
SWH               10000.000 Hz
FIDRES            0.152588 Hz
AQ                3.2768500 sec
RG                       203
DW                  50.000 usec
DE                   6.50 usec
TE                  294.4 K
D1                2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                   20.00 usec
PL1                   1.00 dB
PL1W             17.75783529 W
SFO1            500.1318364 MHz
SI                     65536
SF              500.1300148 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```

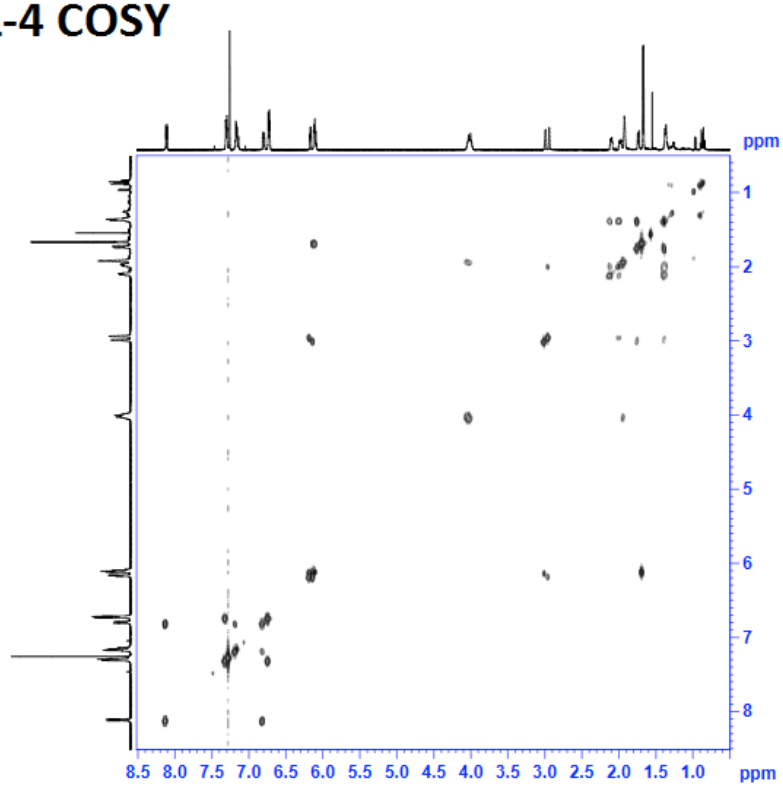
## CL-4 C13




BRUKER

```
NAME            xhu02 CL4 for paper
EXPNO                            10
PROCNO                            1
Date_                      20150223
Time                           6.51
INSTRUM                       spect
PROBHD    5 mm PABBO BB-
PULPROG                     zgpg30
TD                             65536
SOLVENT                      CDCl3
NS                              2048
DS                                 4
SWH                    29761.904 Hz
FIDRES                  0.454131 Hz
AQ                      1.1010548 sec
RG                               203
DW                         16.800 usec
DE                          6.50 usec
TE                          292.0 K
D1                     15.00000000 sec
D11                     0.03000000 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                            13C
P1                            9.50 usec
PL1                           0.00 dB
PL1W                 89.92553711 W
SFO1                 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                     waltz16
NUC2                            1H
PCPD2                       80.00 usec
PL2                           1.00 dB
PL12                         13.04 dB
PL13                         16.80 dB
PL2W                 17.75783539 W
PL12W                 1.11017132 W
PL13W                 0.46707872 W
SFO2                 500.1320005 MHz
SI                            65536
SF                   125.7577802 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40
```

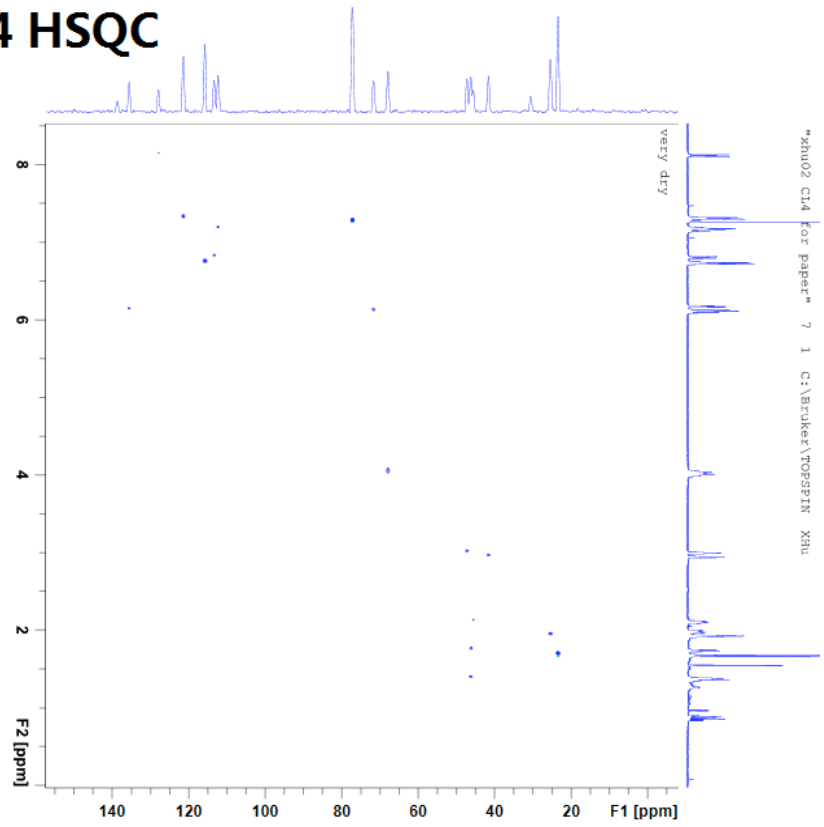
## CL-4 COSY



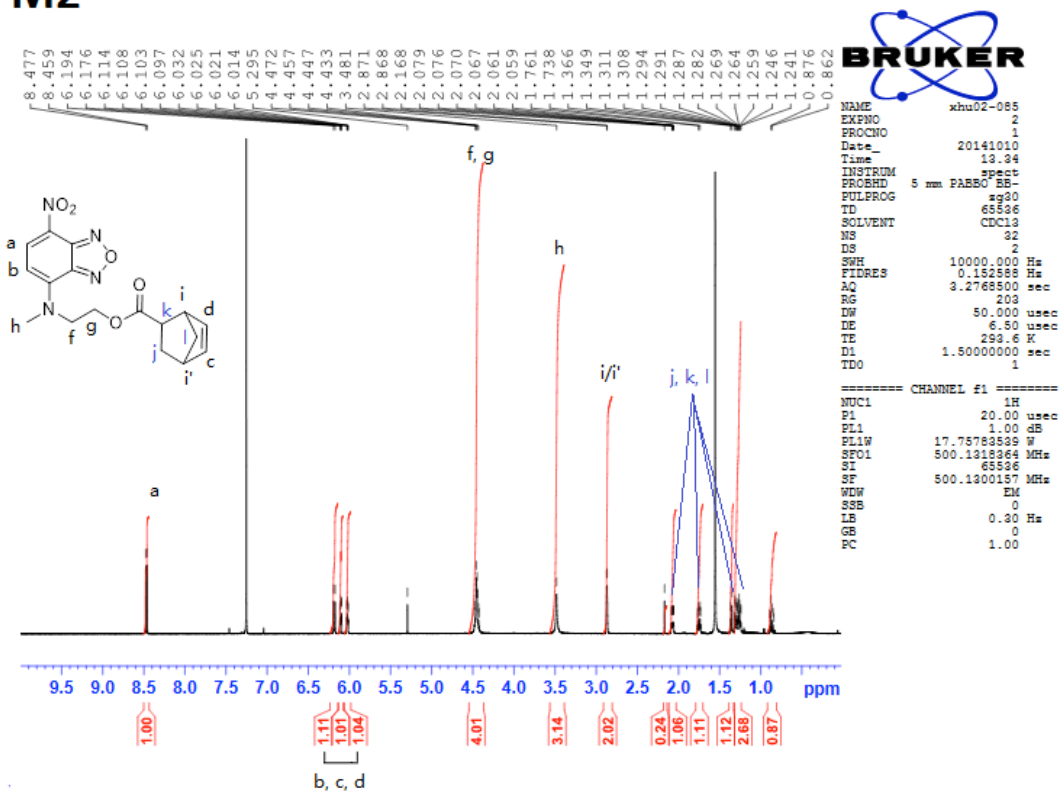

BRUKER

```
NAME            xhu02 CL4 for paper
EXPNO                             9
PROCNO                            1
Date_                      20150222
Time                          20.58
INSTRUM                       spect
PROBHD    5 mm PABBO BB-
PULPROG                   cosygpqf
TD                             2048
SOLVENT                      CDCl3
NS                                1
DS                                8
SWH                    4012.841 Hz
FIDRES                 1.959395 Hz
AQ                     0.2552308 sec
RG                               203
DW                        124.600 usec
DE                          6.50 usec
TE                          292.2 K
D0                     0.00000300 sec
D1                    10.00000000 sec
D13                    0.00000400 sec
D16                    0.00020000 sec
IN0                    0.00024960 sec

-------- CHANNEL f1 --------
NUC1                            1H
P0                           20.00 usec
P1                           20.00 usec
PL1                           1.00 dB
PL1W                 17.75783539 W
SFO1                 500.1322567 MHz

------ GRADIENT CHANNEL -----
GPNAM1                   SINE.100
GPZ1                         10.00 %
P16                        1000.00 usec
NDO                               1
TD                              128
SFO1                 500.1323 MHz
FIDRES                31.300079 Hz
SW                        8.011 ppm
FnMODE                       QF
SI                             1024
SF                   500.1300024 MHz
WDW                         SINE
SSB                              0
LB                            0.00 Hz
GB                               0
PC                            1.00
SI                             1024
MC2                          QF
SF                   500.1300015 MHz
WDW                         SINE
SSB                              0
LB                            0.00 Hz
GB                               0
```

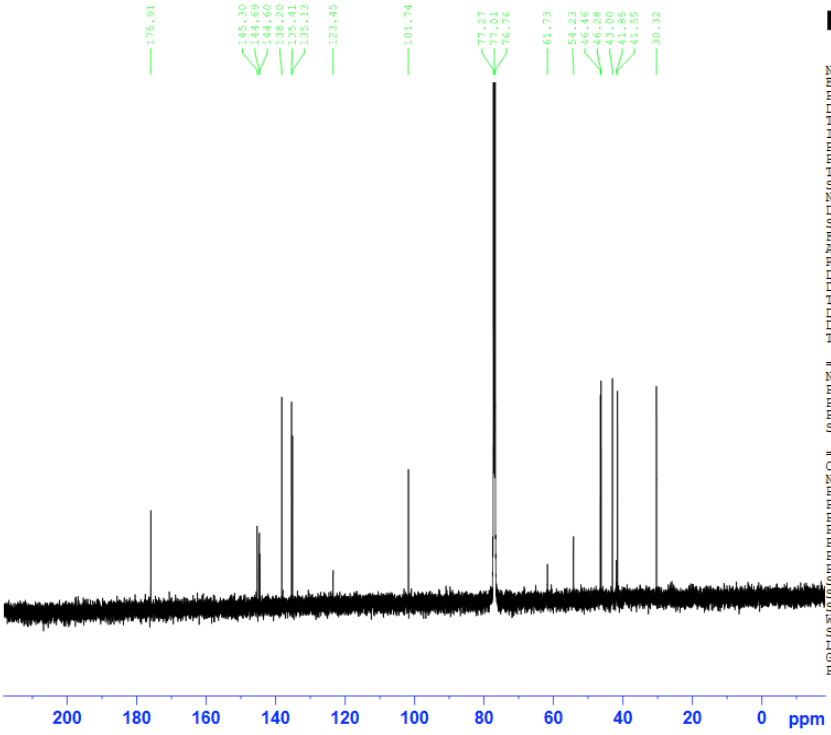
## CL-4 HSQC


F2 [ppm]
F1 [ppm]
very dry
"xhu02 CL4 for paper" 7 1 C:\Bruker\TOPSPIN XHu

## M2


BRUKER
NAME xhu02-085
EXPNO 2
PROCNO 1
Date_ 20141010
Time 13.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 293.6 K
D1 1.50000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 20.00 usec
PL1 1.00 dB
PL1W 17.75783539 W
SFO1 500.1318364 MHz
SI 65536
SF 500.1300157 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
a
b, c, d
f, g
h
i/i'
j, k, l
ppm

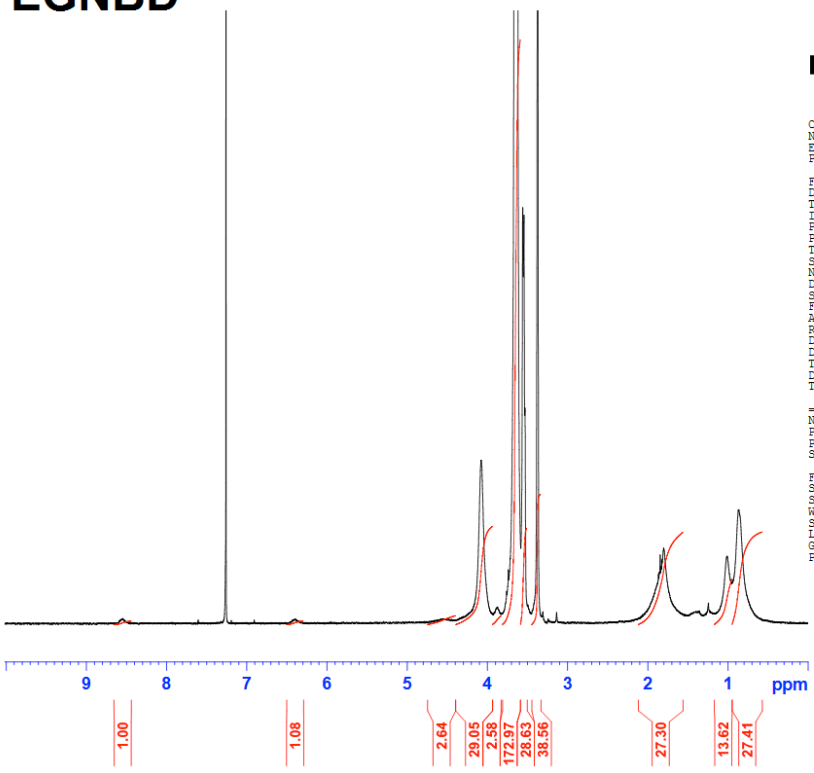
## M2




BRUKER

```
NAME                xhu02-085
EXPNO                       4
PROCNO                      1
Date_                20150403
Time_                   23.20
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        5000
DS                           4
SWH                29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010548 sec
RG                         203
DW                    16.800 usec
DE                     6.50 usec
TE                     293.1 K
D1                10.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        9.50 usec
PL1                       0.00 dB
PL1W              89.92553711 W
SFO1             125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     1.00 dB
PL12                   13.04 dB
PL13                   16.80 dB
PL2W              17.75783539 W
PL12W              1.11017132 W
PL13W              0.46707872 W
SFO2             500.1320005 MHz
SI                       65536
SF               125.7577890 MHz
WDW                         EM
SSB                          0
LB                      1.00 Hz
GB                           0
PC                        1.40
```

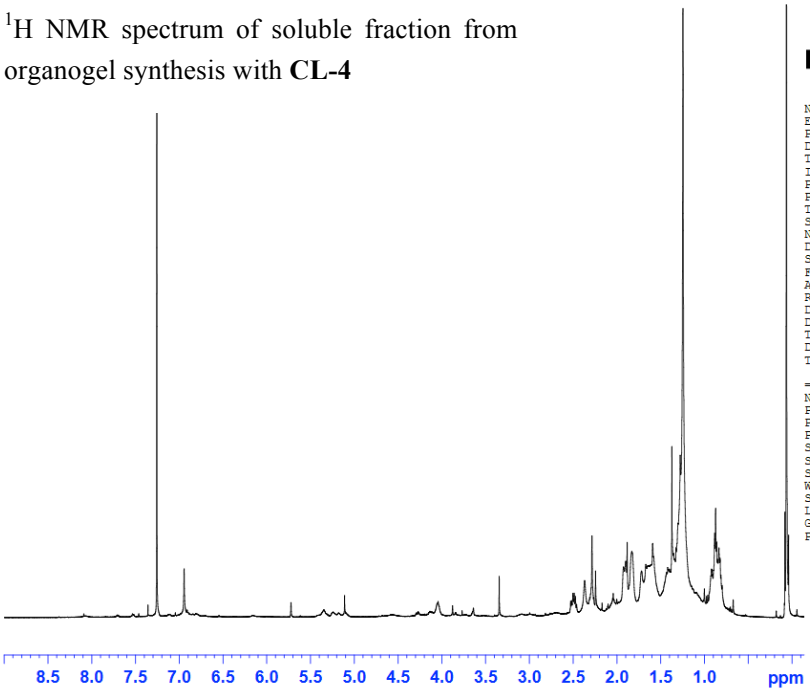
## PEGNBD



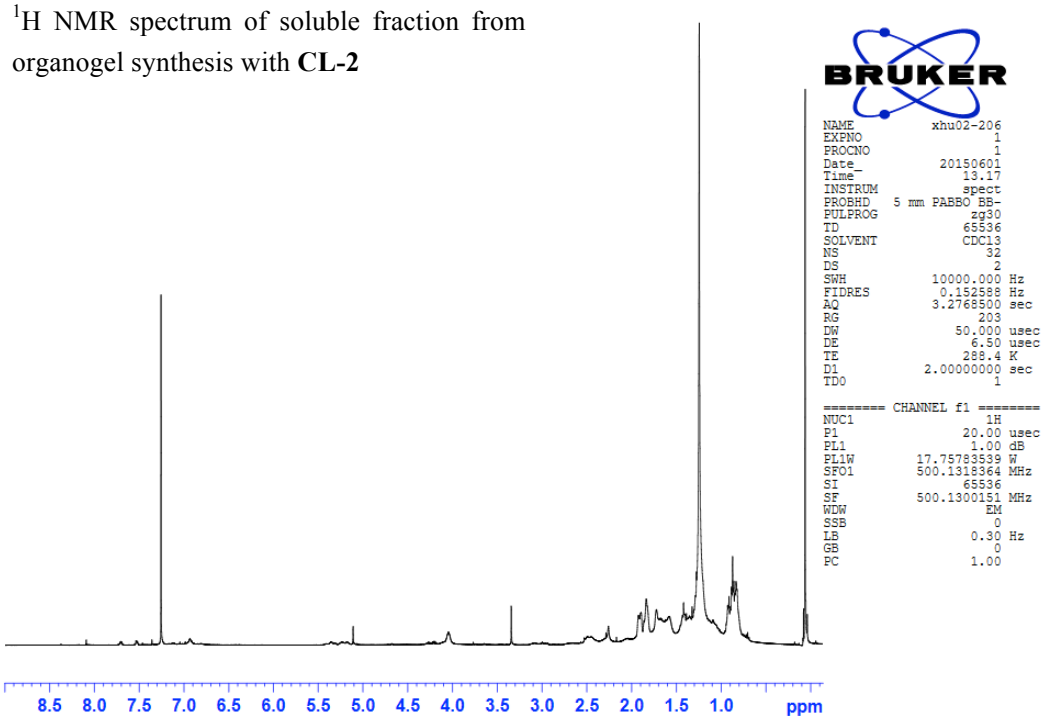

BRUKER

```
Current Data Parameters
NAME      xhu02-P PEGMA co NBDMA
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_                20150317
Time_                   17.37
INSTRUM                 spect
PROBHD    5 mm QNP 1H/13
PULPROG                 zg30
TD                      32768
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                6218.905 Hz
FIDRES             0.189786 Hz
AQ                 2.6345973 sec
RG                      322.5
DW                    80.400 usec
DE                     6.00 usec
TE                     300.0 K
D1                0.50000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                       12.00 usec
PL1                       0.00 dB
SFO1             300.3418547 MHz

F2 - Processing parameters
SI                      32768
SF               300.3400040 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                        1.00
```

$^{1}$H NMR spectrum of soluble fraction from organogel synthesis with **CL-4**




$^{1}$H NMR spectrum of soluble fraction from organogel synthesis with **CL-2**



## 7. References

(1) Gumbley, P.; Hu, X.; Lawrence, J. A.; Thomas, S. W. *Macromol. Rapid Commun.* **2013**, *34*, 1838.

(2) Katzenstein, J. M.; Janes, D. W.; Hocker, H. E.; Chandler, J. K.; Ellison, C. J. *Macromolecules* **2012**, *45*, 1544.

(3) Chen, B. Y.; Kuo, C. C.; Huang, Y. S.; Lu, S. T.; Liang, F. C.; Jiang, D. H. *Acs Appl Mater Inter* **2015**, *7*, 2797.