

## Supporting Information

### Mechanical Generation of Isocyanate by Mechanically Induced Retro [2+2] Cycloaddition of a 1,2-Diazetidione Mechanophore

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## I. General procedures

### Materials

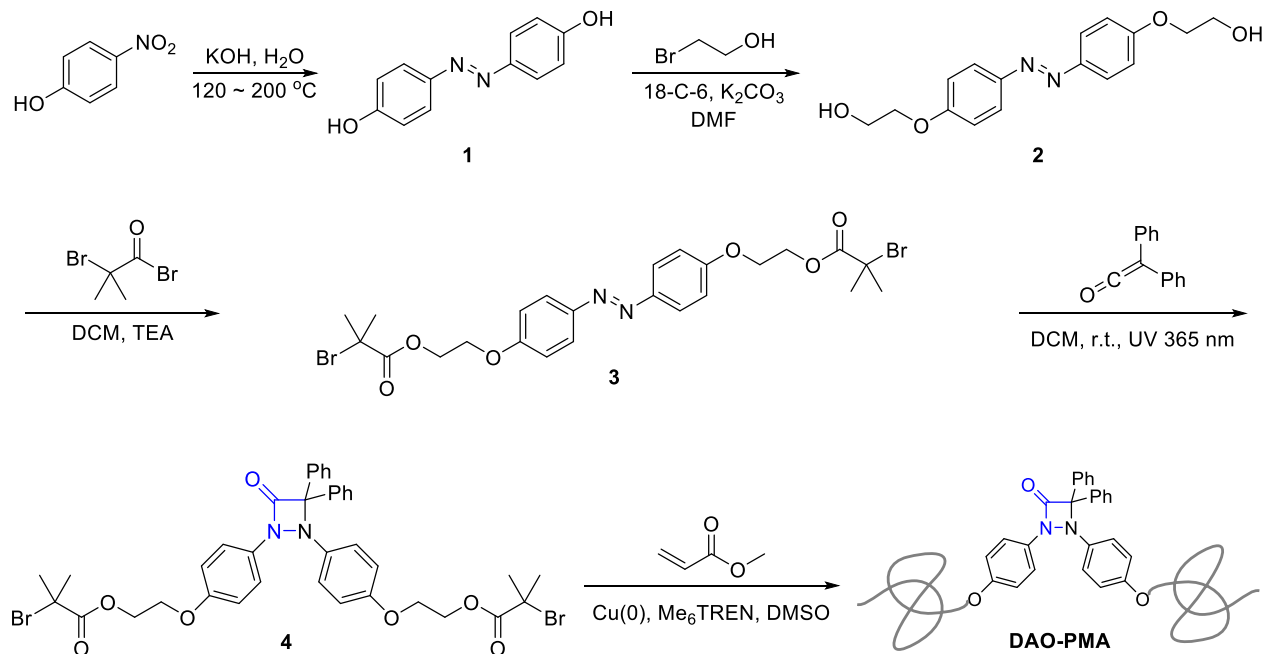
Lab general solvents (dichloromethane, chloroform, hexane, ethyl acetate, acetone, toluene, tetrahydrofuran, methanol, DMSO) were purchased from VWR or Sigma Aldrich. 4-nitrophenol, 2-bromo-ethanol, triethyl amine, 2-Bromo-isobutyrate bromide, diphenylacetyl chloride, Me<sub>6</sub>TREN, methyl acrylate, benzophenone, AlCl<sub>3</sub>, 4-Methoxyaniline, 9-(Methylaminomethyl)anthracene, DBTDL, 4-Methoxyphenyl isocyanate, KOH and K<sub>2</sub>CO<sub>3</sub> were purchased from Sigma Aldrich, Alfa Aesar or TCI and used without further purification. Flash chromatography was performed on CombiFlash®200 auto-column system from Teledyne ISCO.

### Characterizations

<sup>1</sup>H NMR spectra were collected on a 400 MHz Varian INOVA spectrometer and <sup>13</sup>C NMR spectra were collected on a 500 MHz Varian UNITY spectrometer. Chemical shifts are provided in *ppm* ( $\delta$ ) and referenced to the residual <sup>1</sup>H peak at 7.26 ppm or <sup>13</sup>C peak at 77.16 ppm in CDCl<sub>3</sub>, or to the residual <sup>1</sup>H peak at 2.50 ppm or <sup>13</sup>C peak at 39.52 ppm in DMSO-*d*<sub>6</sub>. <sup>1</sup>H shifts are reported as chemical shift, multiplicity, coupling constant if applicable, and relative integral. Multiplicities are reported as: singlet (s), doublet (d), doublet of doublets (dd), doublet of triplets (dt), doublet of doublet of doublets (ddd), doublet of doublet of triplets (ddt), triplet (t), triplet of doublets (td), quartet (q), multiplet (m), or broad (br). Coupling constants (J) are reported in Hertz. High-resolution mass spectra were collected on an Agilent LCMS-TOF-DART at Duke University's Mass Spectrometry Facility. Gel permeation chromatography (GPC) was performed on two Agilent PLgel mixed-C columns (10<sup>5</sup> Å, 7.5x300 mm, 5  $\mu$ m, part number PL1110-6500) using THF (stabilized with 100 ppm BHT) as the eluent. Molecular weights were calculated using a Wyatt Dawn EOS multi-angle light scattering (MALS) detector and Wyatt Optilab DSP Interferometric Refractometer (RI). The refractive index increment (dn/dc) values were determined by online calculation based on injections of known concentration and mass. UV-vis spectra were collected on a Varian Cary 500 UV-Vis Spectrophotometer.

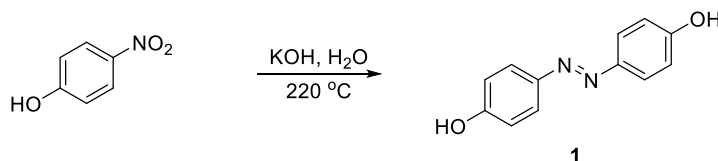
## II. Synthesis

### 1. Synthetic scheme



### 2. Synthetic detail

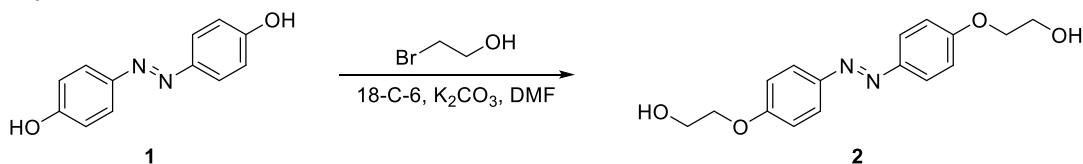
#### 1) Synthesis of 4,4'-bis(hydroxy)-azobenzene **1**



The 4,4'-bis(hydroxy)-azobenzene **1** was synthesized according to previous procedure<sup>1</sup>.

4-nitrophenol (10 g, 72 mmol) and KOH (50 g, 893 mmol) were weighted in a 250 mL round bottom flask. 12 mL DI water was then added to the mixture. The flask was then heated to 120 °C, the mixture turned orange but still cannot be stirred. After 1 h, the temperature was raised to 200 °C, the mixture slowly turned dark red and started to melt. The viscous mixture was then vigorously stirred until a lot of foam evolved. The mixture was then cooled down to room temperature. The solid was then dissolved with DI water and acidified to pH equals to 3 with HCl. Then obtained dark red aqueous solution was extracted with 3×100 mL ether. Combined ether was dried with MgSO<sub>4</sub> and further removed. The residual was recrystallized with 140 mL 50% EtOH/H<sub>2</sub>O to give pure 4,4'-bis(hydroxy)-azobenzene **1** as dark red solid (3.547 g, 46.1%). The <sup>1</sup>H NMR data is in agreement with previous reported<sup>1</sup>.

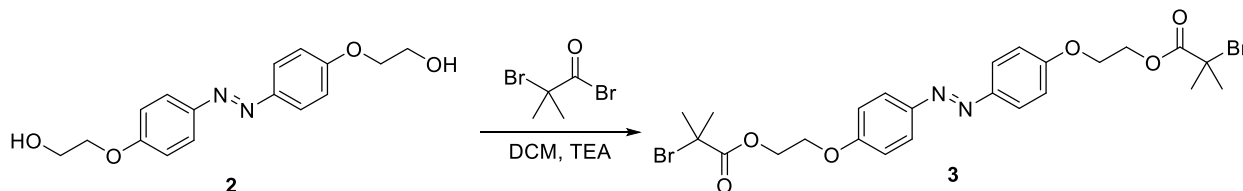
#### 2) Synthesis of azobenzene diol **2**



The azobenzene diol **2** was synthesized according to previous procedure<sup>2</sup>.

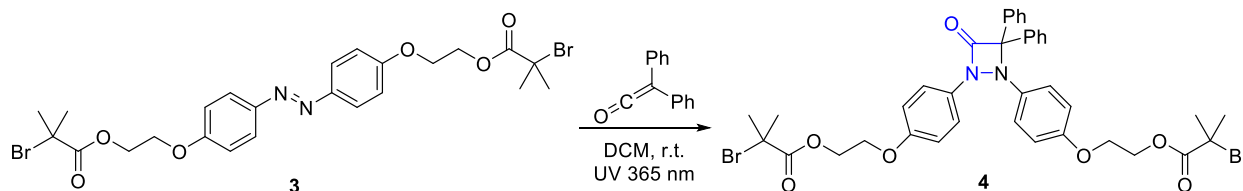
Compound **1** (1.0 g, 4.67 mmol), 2-bromo-ethanol (1.46 g, 11.68 mmol) and 18-Crown-6 (62 mg, 0.23 mmol) were dissolved in 10 mL DMF. Then,  $K_2CO_3$  (2.58 g, 18.68 mmol) was added to the solution. The mixture was then heated at 100 °C for 2 days. The mixture was cooled down and poured into 100 mL DI water. The yellow precipitation was filtered and recrystallized in EtOH to give pure azobenzene diol **2** as yellow solid (0.74 g, 52.4%). The  $^1H$  NMR data is in agreement with previous reported<sup>2</sup>.

### 3) Synthesis of azobenzene initiator **3**



Azobenzene diol **2** (650 mg, 2.15 mmol, 1.0eq.) and triethyl amine (0.9 mL, 6.5 mmol) was dissolved in 25 mL DCM. The solution was cooled with ice bath. 2-Bromo-isobutyrate bromide (638  $\mu$ L, 5.2 mmol) was then added dropwise. The reaction was slowly warmed to room temperature and allow to stir for overnight. After the reaction completed, solvent was removed. Residual was further purified by flash chromatography (0% to 25 % EtOAc/hexane gradient eluent). Pure azobenzene initiator **3** was obtained as a yellow solid (853 mg, 66.0%).  $^1H$  NMR ( $CDCl_3$ , 400MHz)  $\delta$ : 7.88 (d,  $J = 8.97$  Hz, 4H), 7.02 (d,  $J = 8.99$ , 4H), 4.56 (t, 4H), 4.31 (t, 4H), 1.95 (s, 12H).  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$ : 171.74, 160.55, 147.43, 124.51, 115.00, 65.93 64.16, 55.57, 30.82. HRMS-ESI ( $m/z$ ):  $[M + H]^+$  calculated for  $C_{24}H_{29}Br_2N_2O_6$ , 601.31145; found 601.03690.

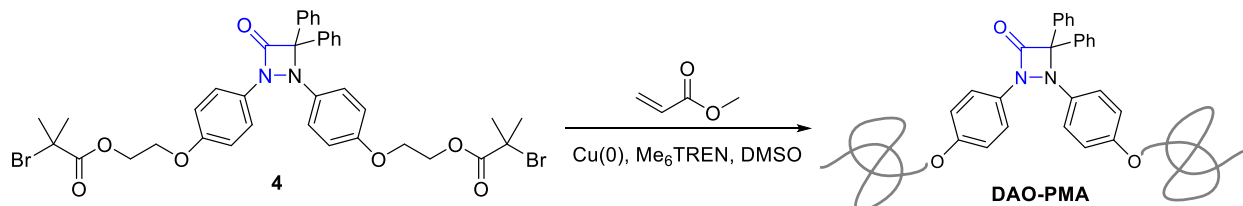
### 4) Synthesis of 1, 2-diazetidinone (DAO) mechanophore containing initiator **4**



The DAO mechanophore was synthesized using in situ generated diphenyl ketene according to previous procedure<sup>3</sup>.

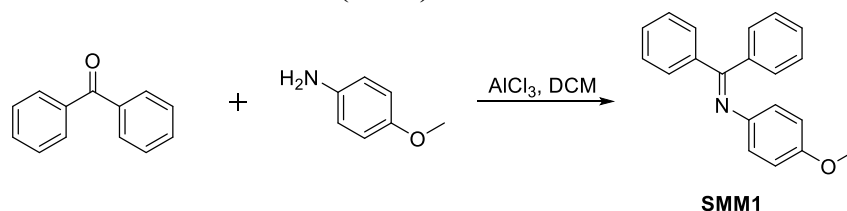
Compound **3** (400 mg, 0.66 mmol) was dissolved in 5 mL DCM. The solution was irradiated with a UV 365 nm LED light. Diphenylacetyl chloride (184 mg, 0.8 mmol) was then added the solution and stirred to dissolve. Dried triethyl amine (120  $\mu$ L, 0.86 mmol) was then added to the solution. The solution was kept under irradiation with UV 365 nm light. The reaction was monitored by TLC. After the reaction completed, the mixture was purified by flash chromatography (0% to 25 % EtOAc/hexane gradient eluent). The DAO mechanophore containing initiator **4** was obtained as a light yellow solid (383 mg, 72.4%).  $^1H$  NMR ( $CDCl_3$ , 400MHz)  $\delta$ : 7.50~6.95 (m & br, 12 H), 6.85 (q, 4H), 6.58 (d, 2H), 4.48 (t, 2H), 4.40 (t, 2H), 4.17 (t, 2H), 4.04 (t, 2H), 1.91 (s, 6H), 1.90 (s, 6H).  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$ : 171.61, 165.64, 155.47, 155.41, 138.77, 131.99, 130.08, 128.73, 128.60, 128.32, 122.34, 117.92, 115.33, 114.75, 94.04, 66.08, 65.85, 64.17, 64.08, 55.18, 30.74. HRMS-ESI ( $m/z$ ):  $[M + H]^+$  calculated for  $C_{38}H_{39}Br_2N_2O_7$ , 795.54445; found 795.11041.

### 5) Synthesis of DAO mechanophore centered PMA polymer (DAO-PMA)

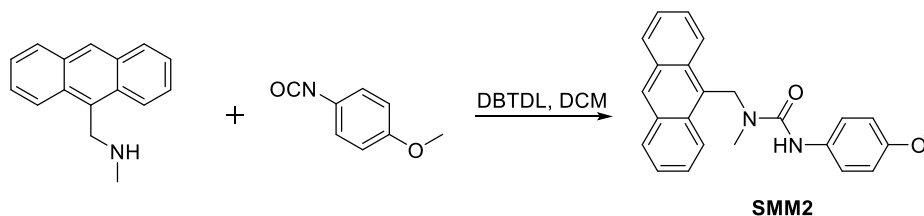


Mechanophore initiator **4** (20 mg, 0.025 mmol), Me<sub>6</sub>TREN (1.3 μL, 0.005 mmol) and methyl acrylate (3.76 mL, 41.5 mmol) monomer were weighted in a 50 mL Schlenk flask. 3.76 mL DMOS was then added and stirred to mix completely. A ~2.0 cm copper wire was firstly polished with sand paper and added to the solution. The flask was then capped and subjected to freeze-pump-thaw cycle to remove dissolved oxygen. The polymerization was proceeded for 3 h. The polymerization was terminated by connecting the Schlenk flask to air. The viscous solution was diluted with DCM and precipitated form methanol. The polymer was redissolved in DCM and precipitated from methanol. This procedure was repeated twice. The polymer was further dried under high vacuum. Obtained polymer was characterized by GPC ( $M_n = 98.0$  kDa,  $M_w = 110.5$  kDa, PDI = 1.128).

## 6) Synthesis of small molecule model (SMM)



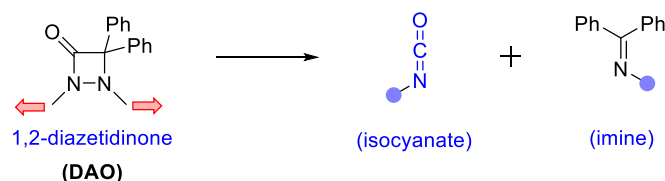
To a solution of benzophenone (243 mg, 1.33 mmol) in 12 DCM, added AlCl<sub>3</sub> (302 mg, 2.26 mmol). 4-Methoxyaniline (246 μL, 2.14 mmol) and triethyl amine (954 μL, 6.84 mmol) was added to a 20 mL scintillation vial and dissolved with 3 mL DCM. The solution was then added dropwise to the previous mixture. The reaction was allowed to stir at room temperature for 5 h. After the reaction completed, it was quenched with 20 mL DI water and extracted with 3×10 mL DCM. DCM was combined and dried with MgSO<sub>4</sub>. Pure product was obtained as light oil (332 mg, 86.7%) after column chromatography (0 ~25% EtOAc/Hexane gradient eluent). <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400MHz) δ: 7.80~7.28 (m, 8H), 7.12 (m, 2H), 6.71 (d, *J* = 8.8 Hz, 2H), 6.62 (d, *J* = 8.8 Hz, 2H), 3.64 (s, 3H). <sup>13</sup>C NMR (125 MHz, DMSO-*d*<sub>3</sub>) δ: 166.93, 155.29, 143.98, 139.14, 136.99, 136.20, 132.63, 130.65, 129.59, 128.85, 128.60, 128.54, 128.41, 128.25, 128.14, 121.96, 113.65, 54.96. HRMS-ESI (*m/z*): [M + H]<sup>+</sup> calculated for C<sub>20</sub>H<sub>18</sub>NO, 288.36945; found 288.13860.



9-(Methylaminomethyl)anthracene (MAMA) (221 mg, 1 mmol) was dissolved in 4 mL DCM. A drop of DBTDL catalyst was added to the solution. Then 4-Methoxyphenyl isocyanate (129 μL, 1 mmol) was added. The reaction was stirred at room temperature for 0.5 h, precipitated formed. The reaction was further stirred for 0.5 h. The mixture was then added dropwise to a stirring hexane. The white solid was filtered and dried under vacuum (360 mg, 97.3%). <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400MHz) δ: 8.65 (s, 1H), 8.54 (d, 2H), 8.28 (s, 1H), 8.13 (d, 2H), 7.56 (m, 4H), 7.45 (d, 2H), 6.88 (d, 2H), 5.58 (s, 2H), 3.73 (s, 3H), 2.60 (s, 3H). <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>, 125MHz) δ: 155.92, 154.60, 133.62, 131.02, 130.86, 129.13, 127.82, 126.49, 125.18, 124.36, 121.98, 113.56, 55.17, 41.78, 32.15. HRMS-ESI (*m/z*): [M + H]<sup>+</sup> calculated for C<sub>24</sub>H<sub>23</sub>N<sub>2</sub>O<sub>2</sub>, 371.45945; found 371.17562.

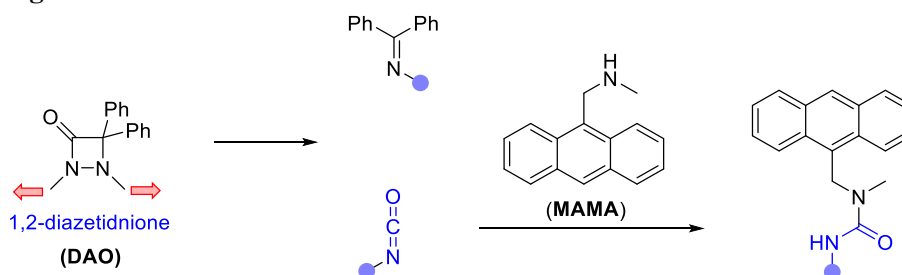
### III. Sonication experiment

#### 1. Sonication of DAO-PMA for GPC analysis



A solution of 32 mg **DAO-PMA** polymer in 16 mL dry THF ( $c = 2$  mg/mL) was transferred into a Suslick cell. The solution was purged with  $N_2$  for 10 min and then cooled with ice bath. Ultrasound impulse was applied (1s on, 1s off) at 30% amplitude. An aliquot of 0.2 mL sample were took out for GPC analysis at time points: 0, 3, 6, 10, 15, 20, 30, 45, 60 min. After sonication, the remained solution was condensed and precipitated from MeOH. The obtained polymer was dried and analyzed from  $^1H$  NMR and UV-vis.

#### 2. Labeling of DAO-PMA under sonication



A solution of 32 mg **DAO-PMA** polymer in 16 mL dry THF ( $c = 2$  mg/mL) was transferred into a Suslick cell. Then, 9-(Methylaminomethyl)anthracene (MAMA) (71 mg, 1000 eq.) and DBTDL (1  $\mu$ L) was added to the solution. The solution was purged with  $N_2$  for 10 min and then cooled with ice bath. Ultrasound impulse was applied (1s on, 1s off) at 30% amplitude for 60 min. After sonication, the solution was condensed and precipitated from MeOH. The polymer was then redissolved in 0.2 mL DCM and precipitated from MeOH. This procedure was repeated 4 times. The polymer was dried under vacuum and subjected to GPC, UV-vis and  $^1H$  NMR analysis.

#### IV. Determination of mechanically activated product

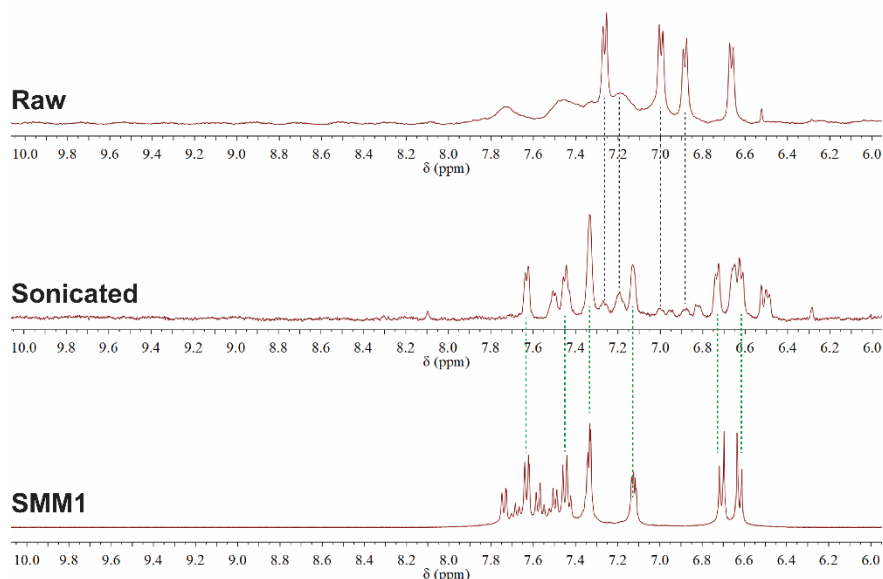


Figure S1. <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) overlay of raw and sonicated DAO-PMA polymer and compound SMM1.

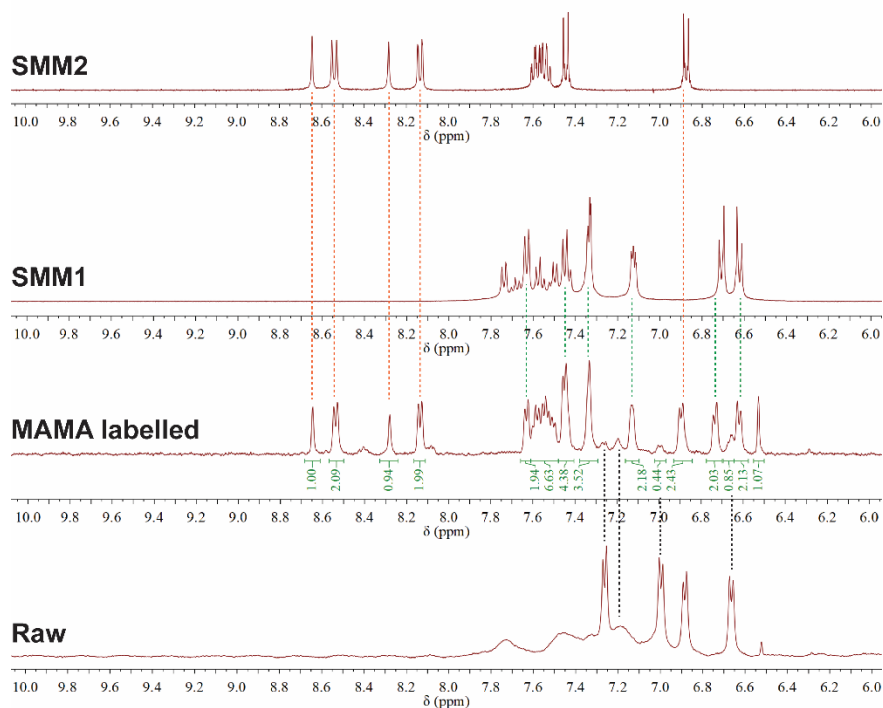
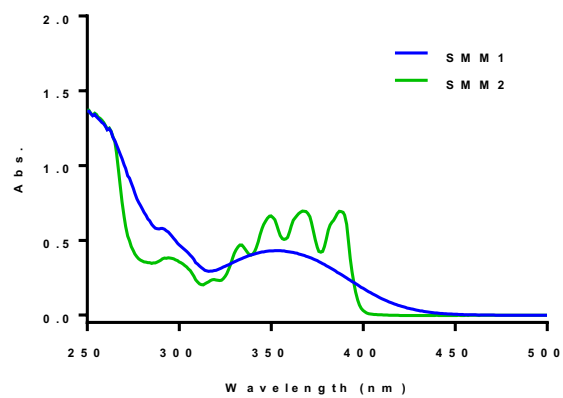


Figure S2. <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) overlay of raw and MAMA labelled DAO-PMA polymer, compound SMM1 and SMM2. The activation percentage was estimated from remaining DAO peak and formed imine:  $2.03/(0.44+2.03)*100\% = 82\%$ ; the ratio of formed imine to trapped MAMA is near  $2.03:2.09 \sim 1$ ; therefore, the isocyanate reacted quantitatively with MAMA.

## V. UV-vis spectra

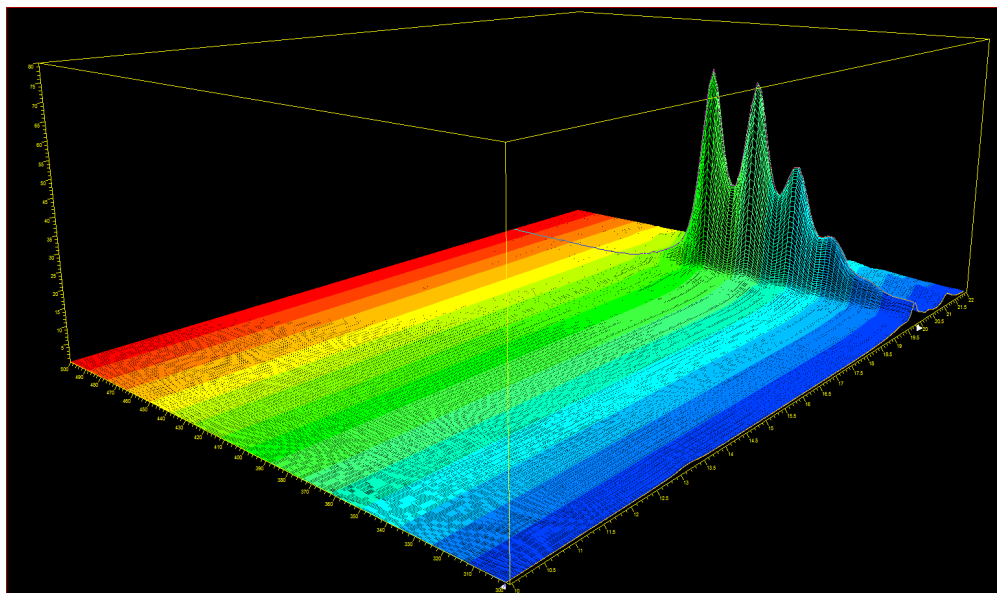


**Figure S3.** UV-vis spectra of SMM1 and SMM2 in THF.

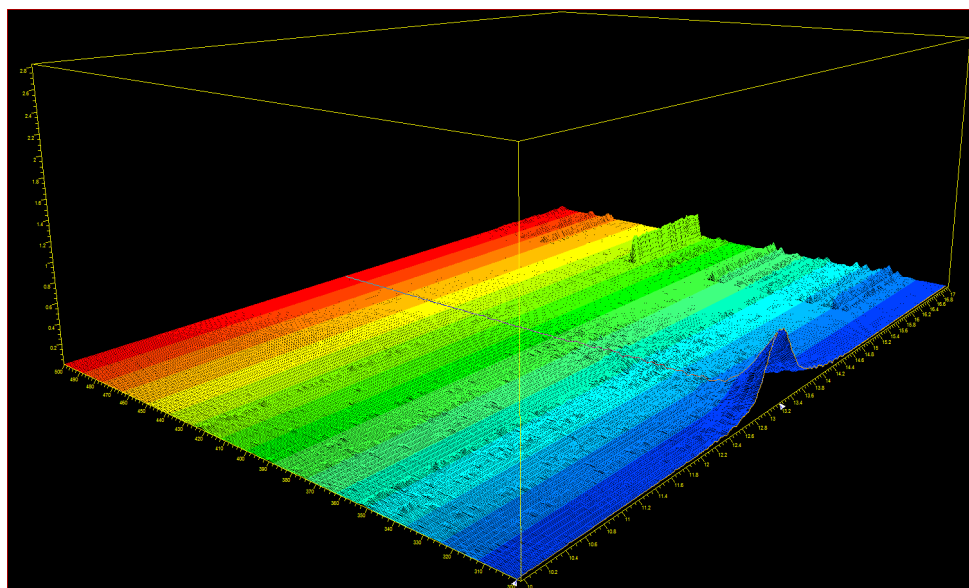


## VI. Control experiment for MAMA labelling

DAO-PMA polymer was mixed with 1000 eq. MAMA and 5  $\mu$ L DBTDL catalyst for 60 min, the mixture was then subjected to GPC analysis.



**Figure S4.** 3D UV-vis signal of DAO-PMA, MAMA and DBTDL after 60 min physical mixing. The strong UV-vis signal appeared at retention time of 19.5~20.5 min that corresponds to excess small molecule MAMA.



**Figure S5.** Zoom-in view of Figure S4 shows 12~14 min polymer retention time region presents 3D UV-vis signal appeared only at the range of 300~320 nm, which is consistent with UV-vis absorption of raw polymer.

## VII. Determination of mechanophore activation rate under sonication

### 1. Polymer degradation kinetics

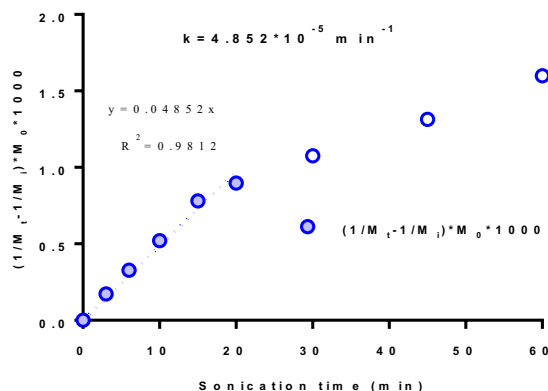
The molecular degradation rate was calculated from the following equation (1):

$$\frac{1}{M_t} - \frac{1}{M_i} = \frac{kt}{M_0}$$

Where  $M_t$  is the number average molecular weight at time  $t$ ,  $M_i$  stands for the initial number average molecular weight,  $M_0$  represents for the molecular weight of repeating unit and  $k$  refers to the degradation rate. Table 1 provides a representative number average molecular weight of sonicated polymer at time  $t$ . The corresponding polymer degradation rate was determined.

**Table 1.** A representative data set of molecular degradation during sonication

Sonication time / min	$M_n$ / kDa	$M_w$ / kDa	PDI	$(1/M_t - 1/M_i) * M_0 * 1000$	Rate / $\text{min}^{-1}$
0	98.0	110.5	1.128	0.0426	
3	78.6	93.4	1.189	0.2592	
6	69.4	84.1	1.211	0.4042	
10	61.8	74.1	1.199	0.5566	
15	55.4	65.8	1.187	0.7174	$5.152 \times 10^{-5}$
20	48.9	59.0	1.207	0.9237	
30	41.6	51.1	1.228	1.2323	
45	36.3	44.9	1.236	1.5342	
60	34.7	41.6	1.200	1.6434	

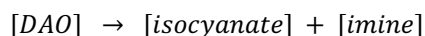


**Figure S6.** Analyzing the molecular degradation rate using equation (1). The linear fitting was applied on first 15 min sonication that lies in the first scission cycle. The obtained molecular degradation rate is  $4.852 \times 10^{-5} \text{ min}^{-1}$ .

As shown in Figure S6, the whole data set presents a poor linearity. Therefore, the data points from the first 20 min sonication were used for linear fitting since the molecular weight reduced from 98.0 kDa to 48.9 kDa that lies in the first scission cycle.

## 2. Determination of mechanophore activation rate from UV 356 nm signal

The increase at UV 365 nm signal from GPC trace corresponds to the generation of imine during the sonication. The mechanical activation of DAO mechanophore can be treated as a first order reaction:



$$-\frac{d[DAO]}{dt} = k[DAO]$$

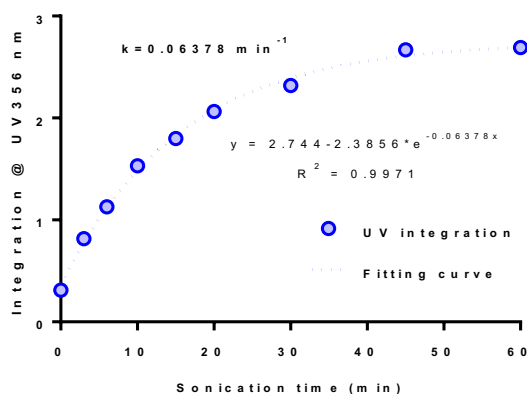
$$\ln[DAO]_t - \ln[DAO]_0 = kt$$

Since,  $[DAO]_0 = [DAO]_t + [imine]$ :

$$\ln \frac{[DAO]_0 - [imine]}{[DAO]_0} = kt$$

The concentration of generated imine species is proportional to the UV 356 nm absorption. Hence, the activation rate can be obtained from integration of UV 356 nm signal using the following equation (2):

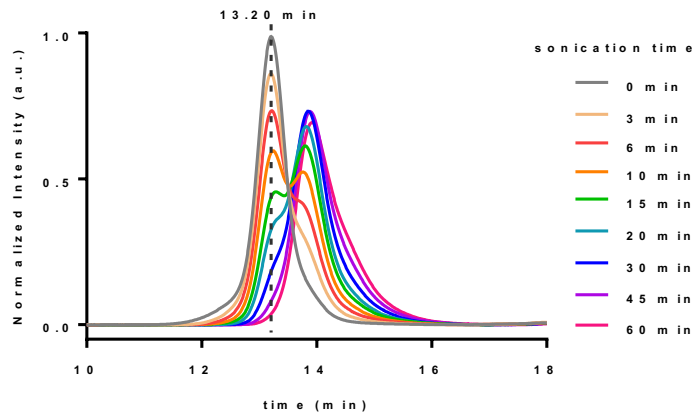
$$[imine] = [DAO]_0 - [DAO]_0 e^{-kt}$$



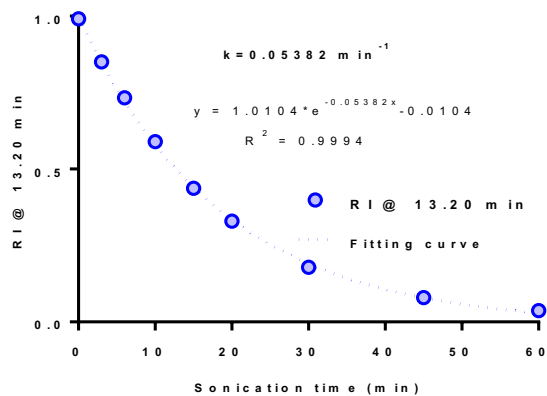
**Figure S7.** Mechanophore activation rate was obtained using integration of UV 356 nm signal from GPC traces. The data was fitted with one-phase association.

## 3. Determination of mechanophore activation rate from RI signal before deconvolution

Given the narrow PDI of prepared DAO-PMA polymer (PDI = 1.13), the activated polymer that reduces to near half molecular weight can be resolved from our two column GPC. Hence, the activation rate of DAO mechanophore can be determined using the method reported by Boydston group. The RI intensity at 13.20 min was applied to calculate the activation rate.



**Figure S8.** GPC RI signal of parent polymer at 13.20 min retention time decreased when subjected to sonication.



**Figure S9.** Mechanophore activation rate was obtained by fitting the evolution of RI signal at 13.20 min retention time.

#### 4. Determination of mechanophore activation rate from RI signal after deconvolution

In order to verify that the activation rate of mechanophore in our narrow disperse polymer can be correctly valued from the evolution of RI intensity at initial peak, we performed the deconvolution of GPC traces to analyze the activation rate by using microsoft excel solver according to previous reported method<sup>4-5</sup>.

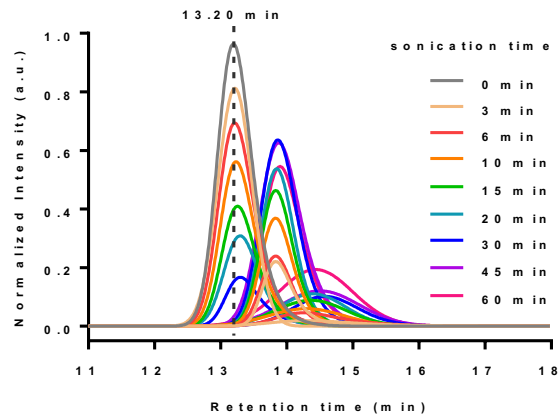


Figure S10. Deconvolution of GPC RI traces using Microsoft excel solver.

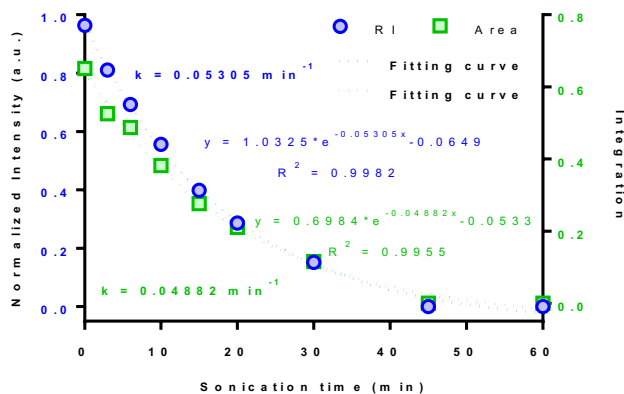


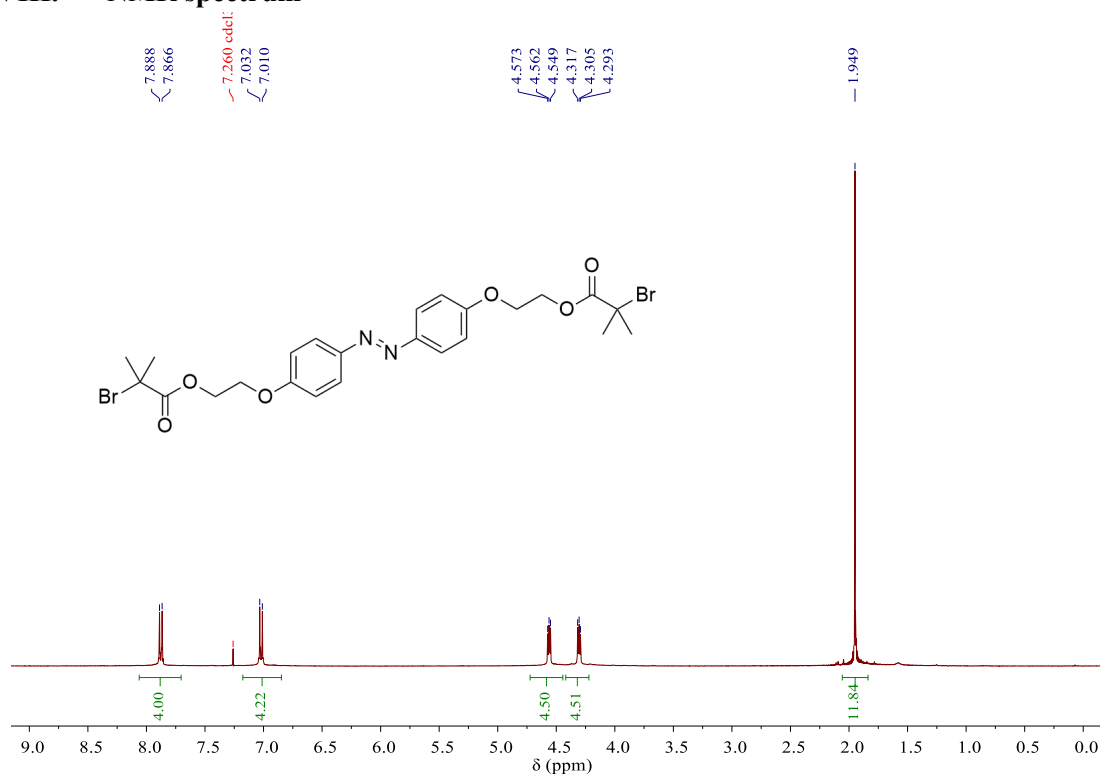
Figure S11. Mechanophore activation rate was evaluated from deconvolution of GPC RI traces. Fitting the RI signal of parent polymer at 13.20 min retention time with one-phase decay (Blue circle); fitting the RI signal integration of parent polymer with one-phase decay (Green square).

## 5. Comparison of activation rate evaluation

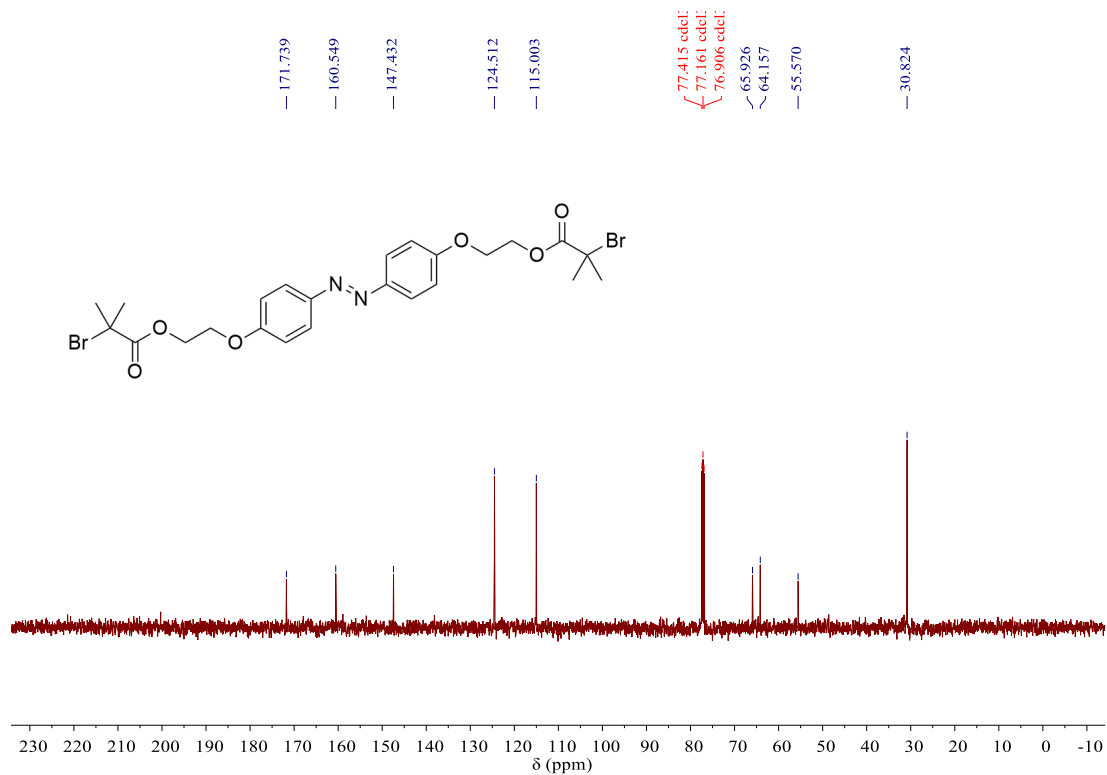
Table 2 Summary of obtained activation rate from different reported method.

Method	Activation rate (min <sup>-1</sup> )					
	Trial 1	Trial 2	Trial 3	Average	SD.	
Cassasa's theory	4.85×10 <sup>-5</sup>	4.64×10 <sup>-5</sup>	4.35×10 <sup>-5</sup>	4.61×10 <sup>-5</sup>	2.52×10 <sup>-6</sup>	
UV 356 nm integration	6.38×10 <sup>-2</sup>	6.95×10 <sup>-2</sup>	7.03×10 <sup>-2</sup>	6.79×10 <sup>-2</sup>	3.55×10 <sup>-3</sup>	
RI @ 13.20 min	5.38×10 <sup>-2</sup>	5.78×10 <sup>-2</sup>	5.77×10 <sup>-2</sup>	5.64×10 <sup>-2</sup>	2.27×10 <sup>-3</sup>	
Deconvolution	RI@13.20min	5.31×10 <sup>-2</sup>	5.55×10 <sup>-2</sup>	5.47×10 <sup>-2</sup>	5.44×10 <sup>-2</sup>	1.24×10 <sup>-3</sup>
	Area	4.88×10 <sup>-2</sup>	5.02×10 <sup>-2</sup>	5.00×10 <sup>-2</sup>	4.97×10 <sup>-2</sup>	7.36×10 <sup>-4</sup>

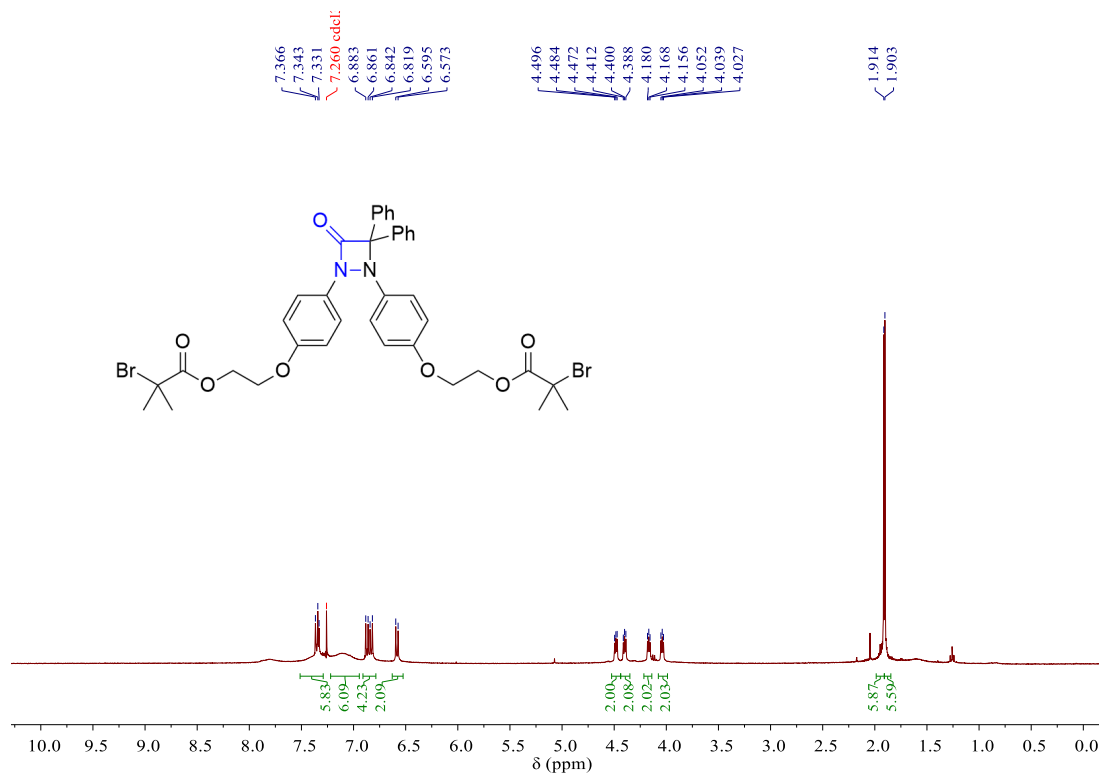
### VIII. NMR spectrum



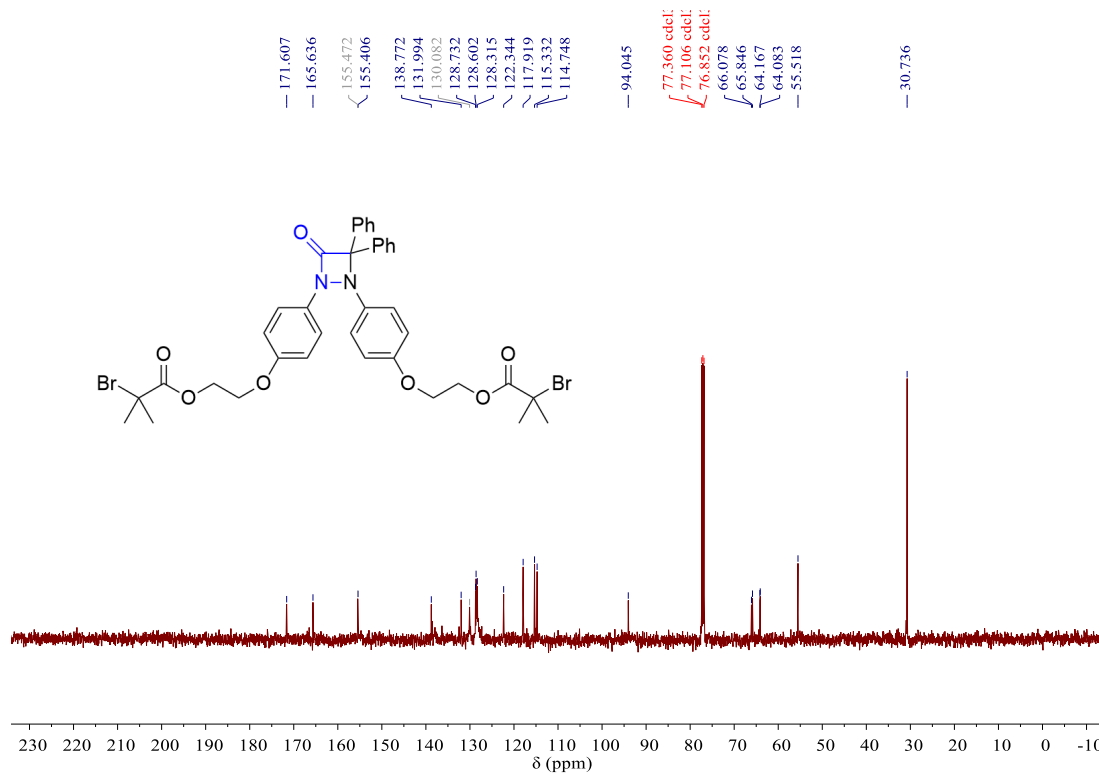
<sup>1</sup>H NMR of azobenzene initiator 3 (400 MHz, CDCl<sub>3</sub>).



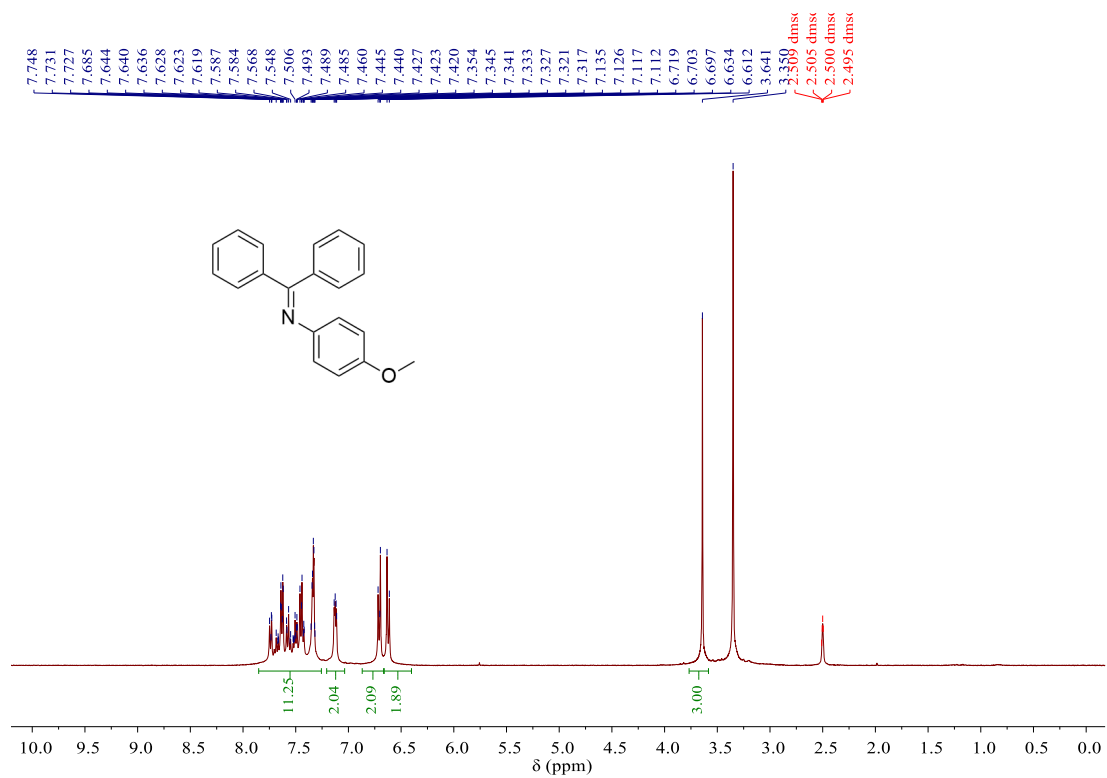
<sup>13</sup>C NMR of azobenzene initiator 3 (125 MHz, CDCl<sub>3</sub>).



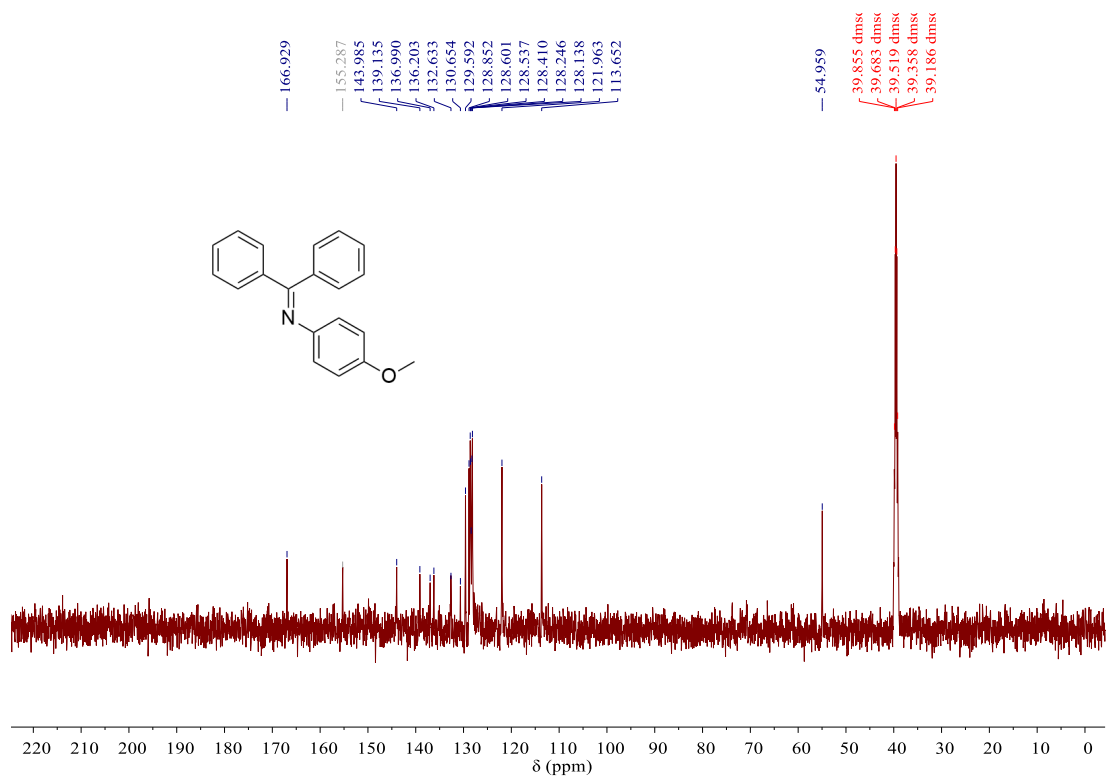
$^1\text{H NMR}$  of DAO mechaphore containing initiator 4 (400 MHz,  $\text{CDCl}_3$ ).



$^{13}\text{C NMR}$  of DAO mechaphore containing initiator 4 (125 MHz,  $\text{CDCl}_3$ ).

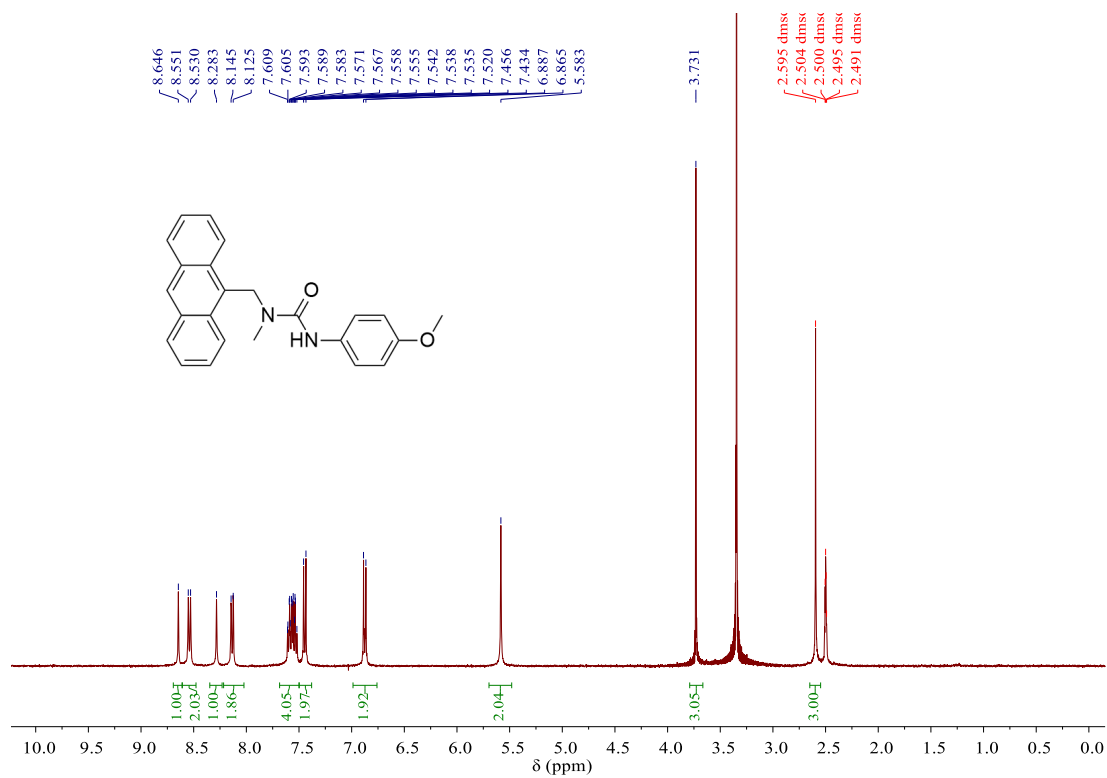


<sup>1</sup>H NMR of compound **SMM1** (400 MHz, DMSO-d<sub>6</sub>).

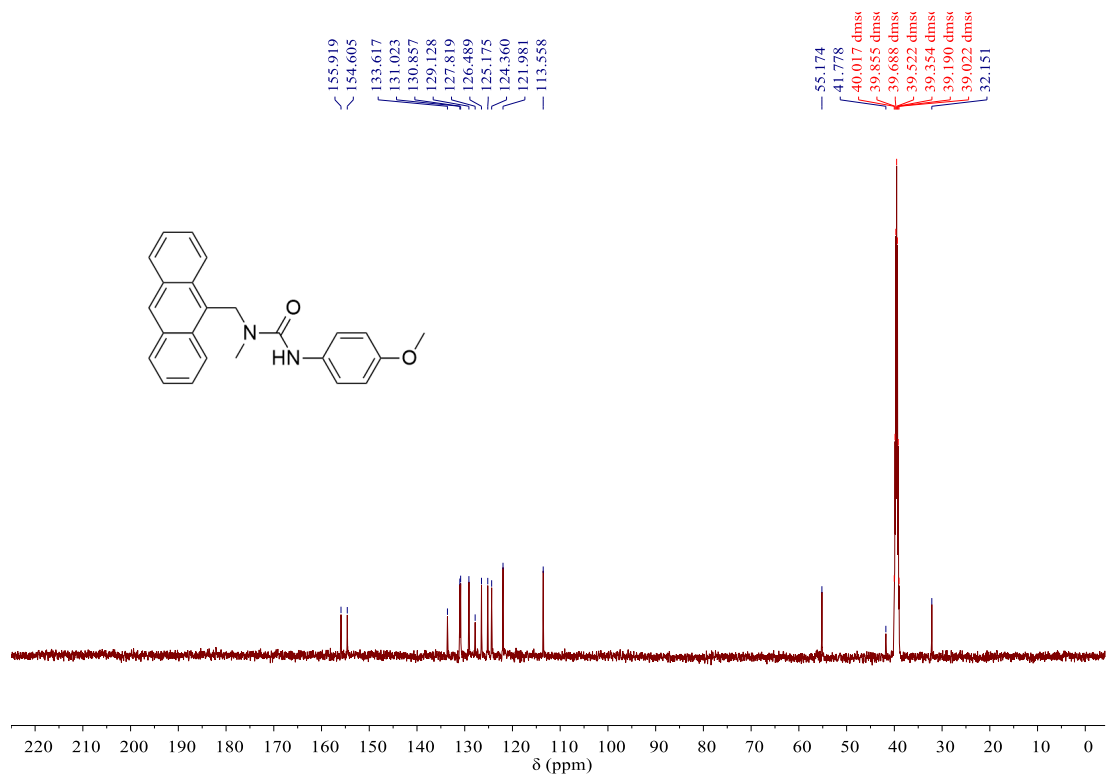


<sup>13</sup>C NMR of compound **SMM1** (125 MHz, DMSO-d<sub>6</sub>).





<sup>1</sup>H NMR of compound **SMM2** (400 MHz, CDCl<sub>3</sub>).



<sup>13</sup>C NMR of compound **SMM2** (125 MHz, DMSO-d<sub>6</sub>).

## IX. Reference

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