

***Supporting Information for:***

**Preparation and Properties of Two-Leg Ladder Polymers Based on Polydiacetylenes**

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## 1. General Procedure.

All chemicals were purchased from Kanto Chemical Co. Ltd. or Sigma Aldrich Co. Ltd. and used without further purification. Gel permeation chromatography (GPC) was performed on a JAI LC-918 equipped with JAIGEL -1H and -2H columns.

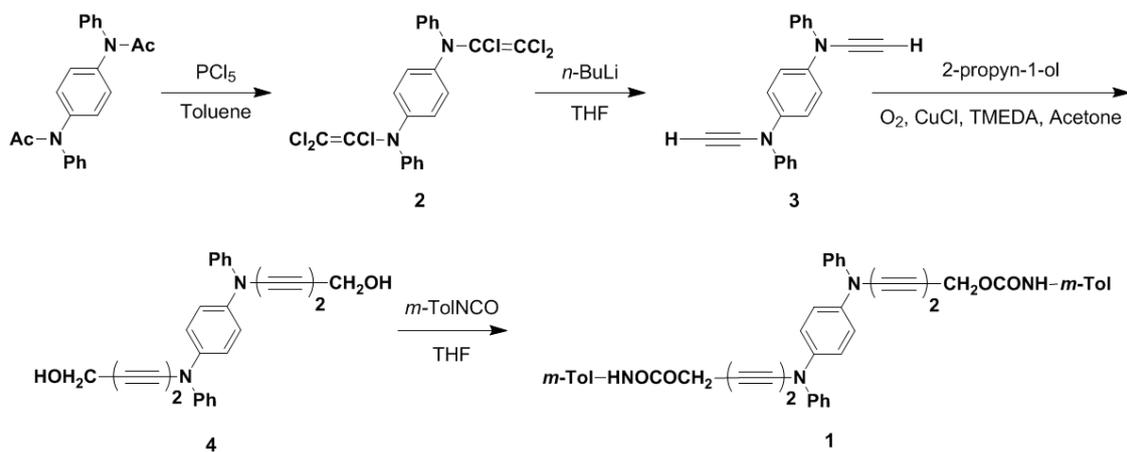
$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a JEOL JNM-ECA-400 spectrometer in deuterated solvents (chloroform-*d* or acetone-*d*<sub>6</sub>) with tetramethylsilane as an internal standard.  $^1\text{H}$  NMR data are reported as follows: chemical shift in ppm downfield from tetramethylsilane ( $\delta$  scale), multiplicity (s = singlet, d = doublet, t = triplet, dd = double doublet, tt = triple triplet and br. = broad), coupling constant (Hz) and integration.  $^{13}\text{C}$  NMR chemical shifts are reported in ppm downfield from tetramethylsilane ( $\delta$  scale). All  $^{13}\text{C}$  NMR spectra were obtained with complete proton decoupling.

IR spectra were recorded on a JASCO FT/IR-420 spectrometer by using a KBr pellet. UV-Vis absorption spectrum was measured on a HITACHI U-2010 spectrometer. UV-Vis-NIR spectrum in solid state was measured on a SHIMADZU UV-3100PC spectrometer with an ISR-3100 integrating sphere attachment. Elemental analysis was recorded on a J-SCEINCE LAB MICRO CORDER JM10.

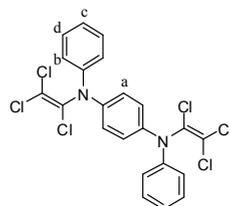
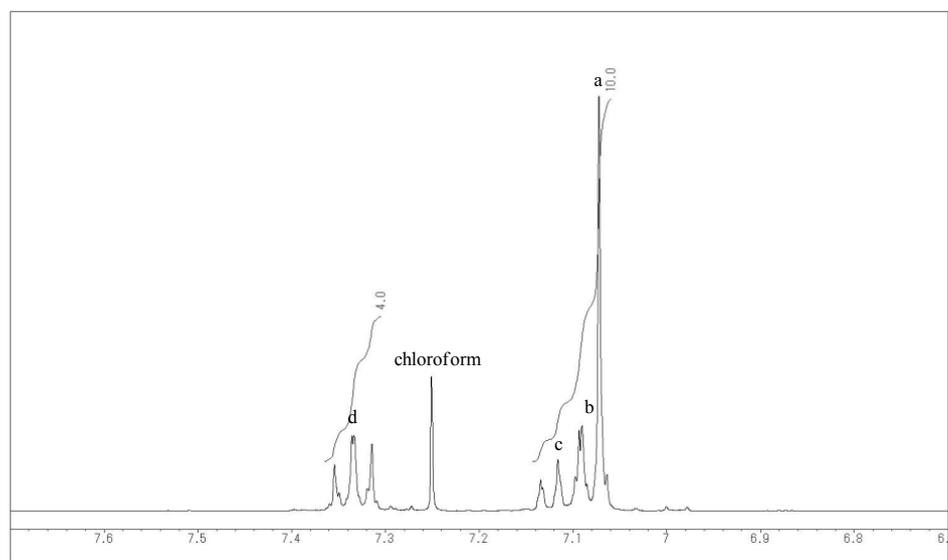
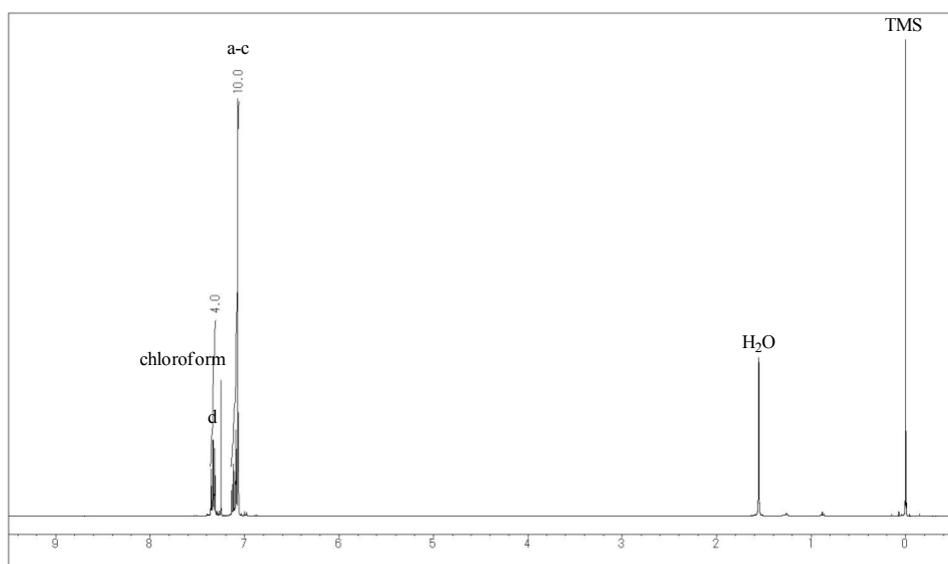
X-ray crystallographic data were obtained by a RIGAKU Saturn 724+ CCD device with a monochromatic Mo  $K\alpha$  radiation. Powder X-ray diffraction (XRD) was recorded on a RIGAKU MiniFlex II diffractometer.

Polymerization of monomer **1** was performed in an electric oven. The powdered monomer was sealed into glass tubes with exchange gas of argon. The tubes were annealed at 100 °C for 400 hours. The conductivity of the doped polymer was measured by 2-probe method, where the polymer in a pellet sealed with excess amounts of iodine was placed in an electric oven.

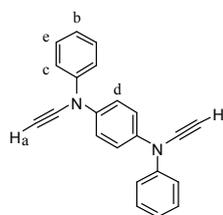
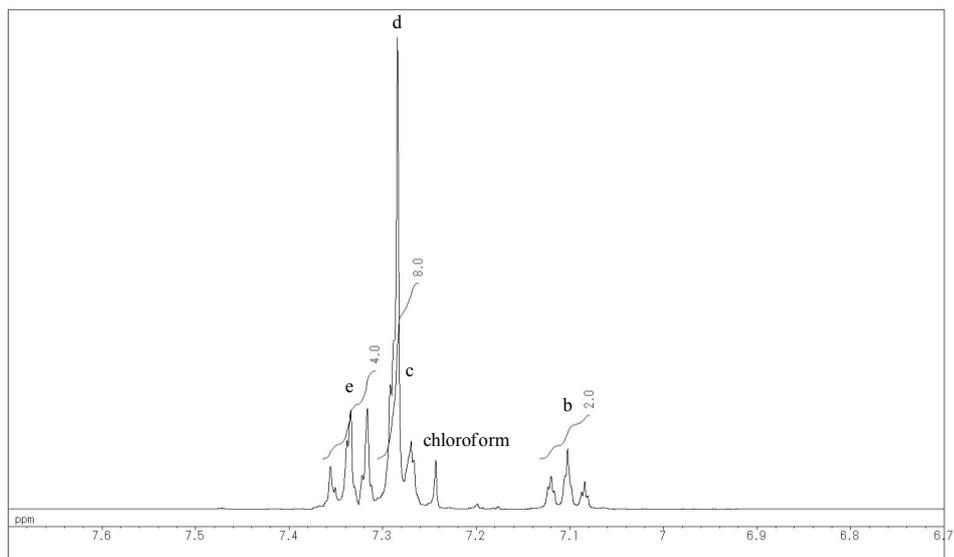
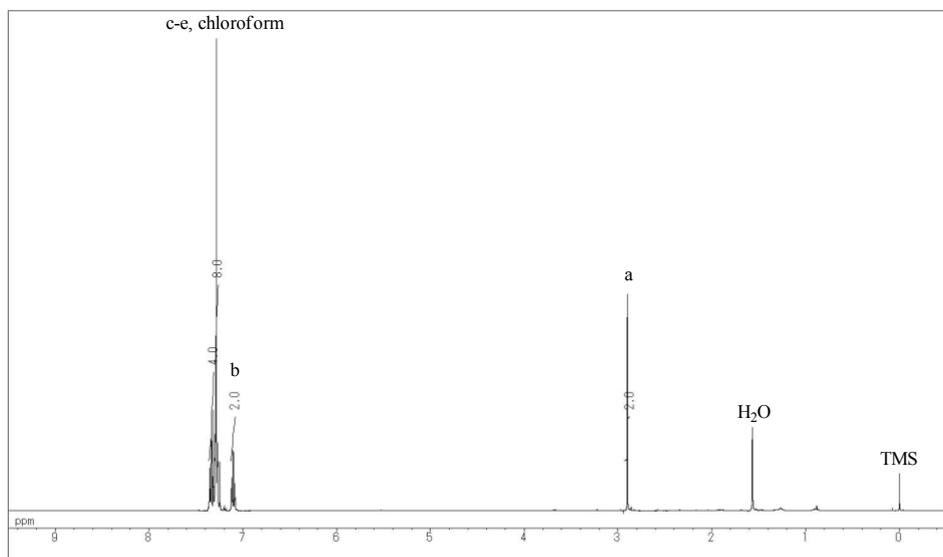
## 2. Materials.



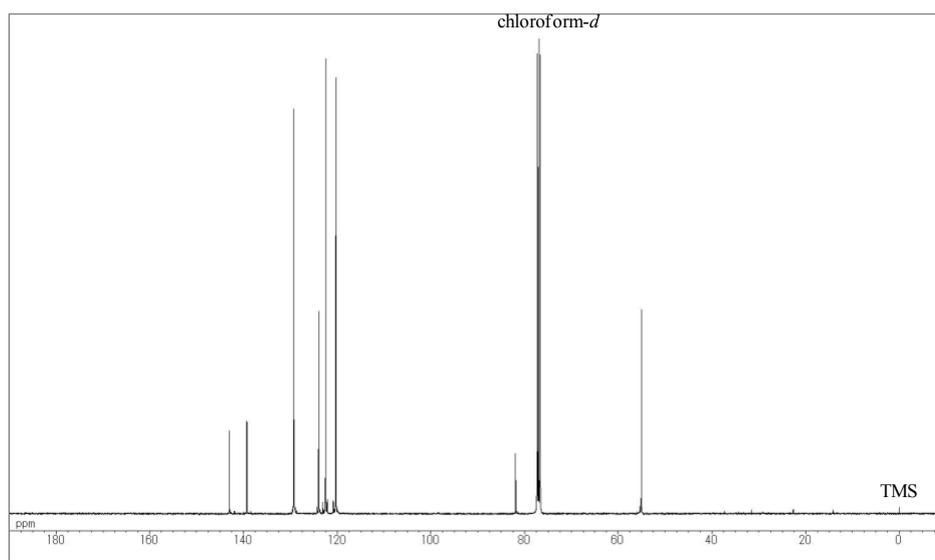
**Figure S1.** Preparation of monomer 1.



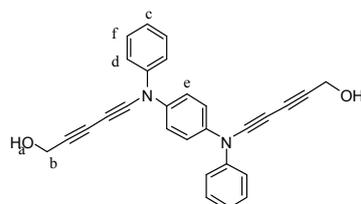
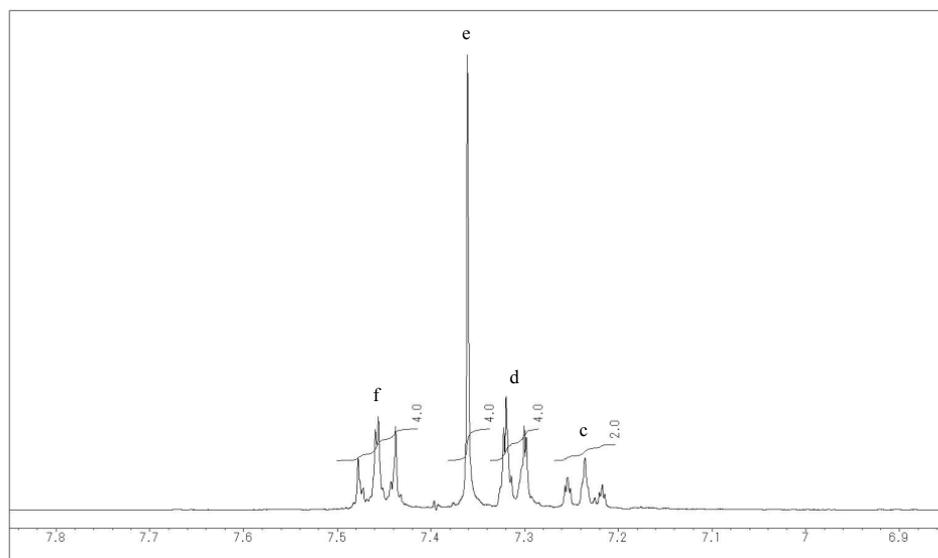
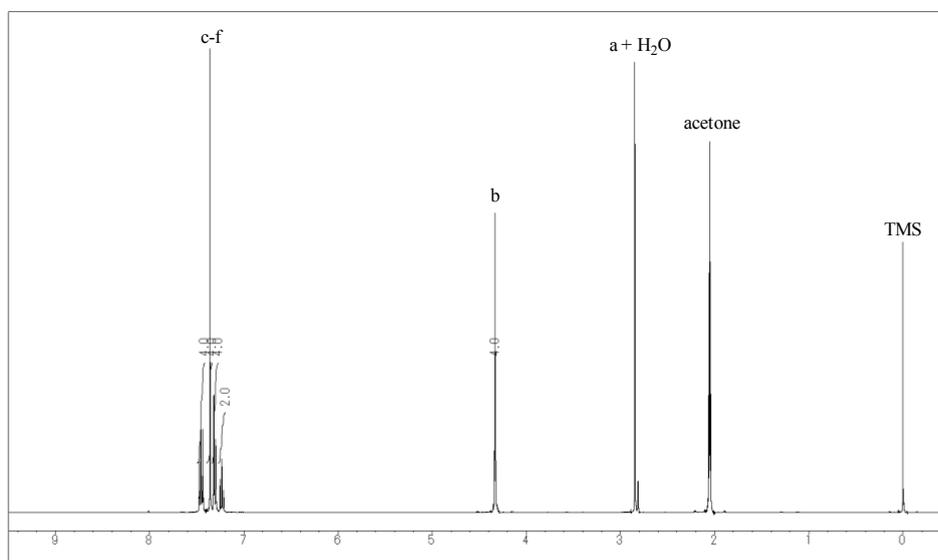
**Figure S2.**  $^1\text{H}$  NMR spectra of **2**.



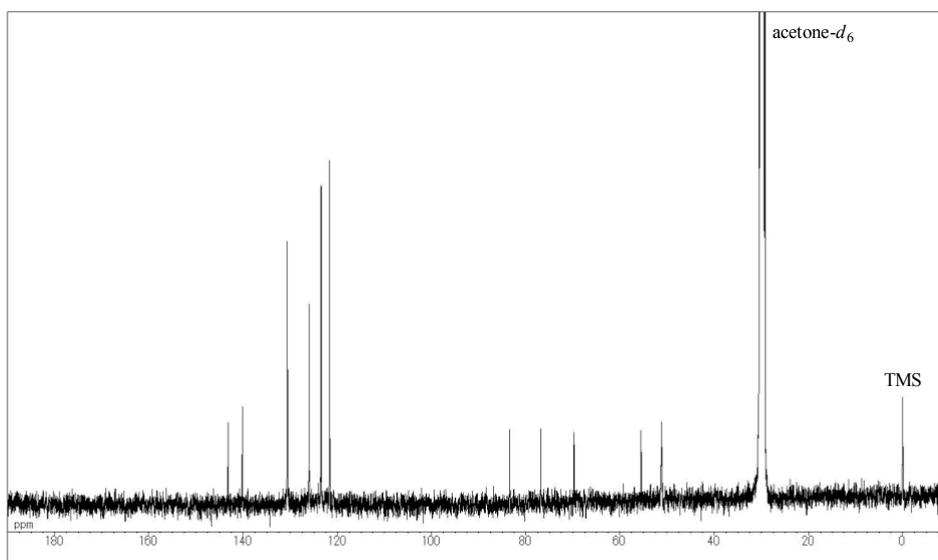
**Figure S3.**  $^1\text{H}$  NMR spectra of **3**.



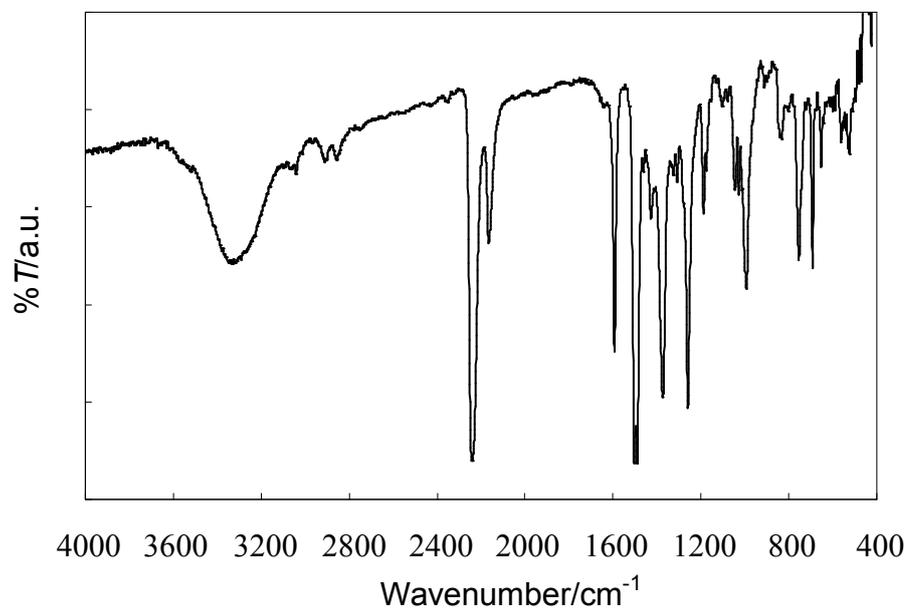
**Figure S4.**  $^{13}\text{C}$  NMR spectrum of **3**.



**Figure S5.**  $^1\text{H}$  NMR spectra of **4**.

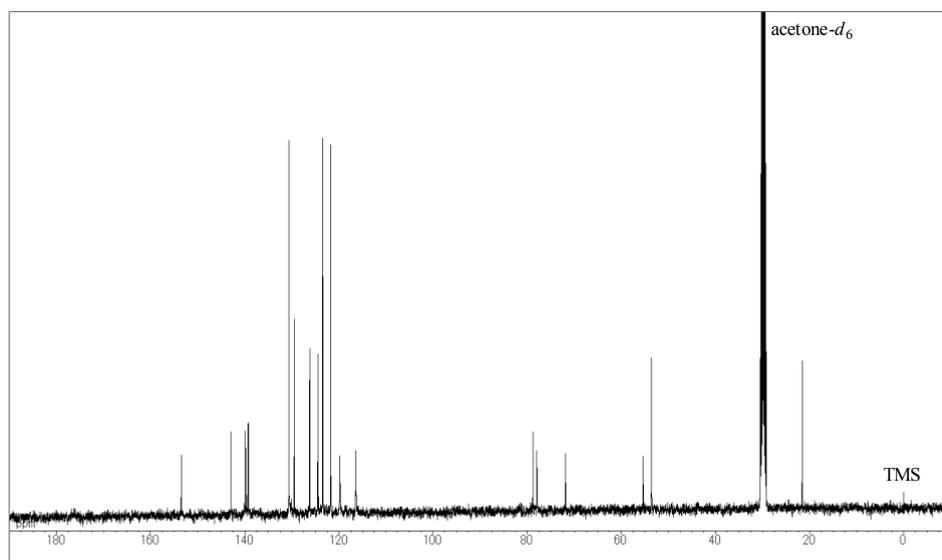


**Figure S6.**  $^{13}\text{C}$  NMR spectrum of **4**.

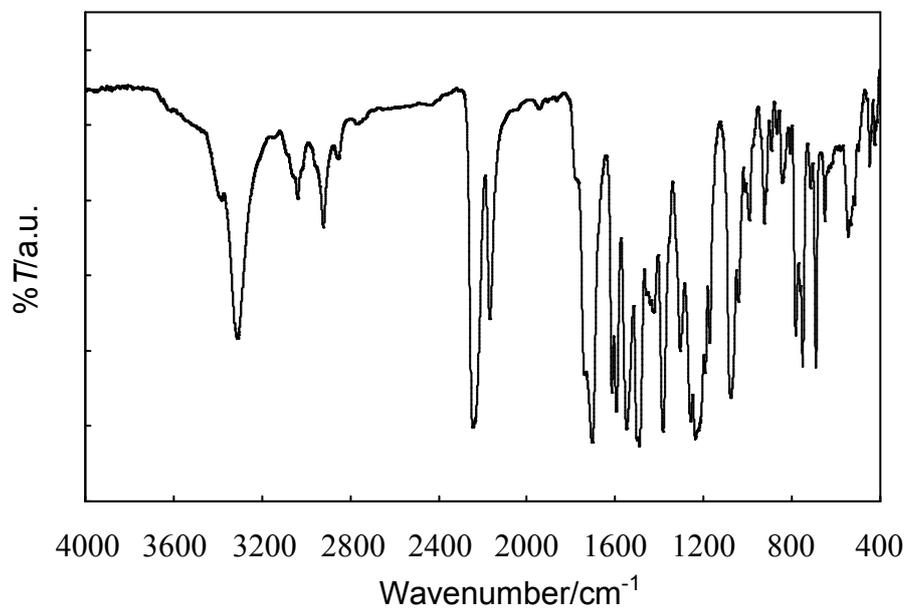


**Figure S7.** IR spectrum of **4**.



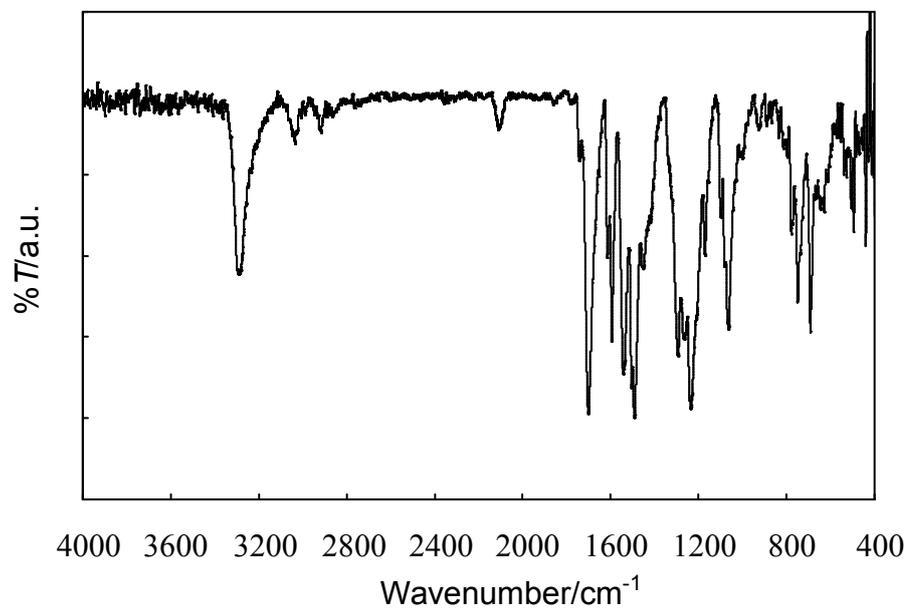


**Figure S9.**  $^{13}\text{C}$  NMR spectrum of monomer **1**.



**Figure S10.** IR spectrum of monomer **1**.

### 3. Characterization of Polymer 1.



**Figure S11.** IR spectrum of polymer 1.