# Supplementary Information

# Poly(hydroxyurethane): Catalytic Applicability for the Cyclic Carbonate Synthesis from Epoxides and $CO_2$

Suguru Motokucho[†,*] and Hiroshi Morikawa[‡]

[†]Chemistry and Material Engineering Program, Nagasaki University, 1-14, Bunkyo-Machi, Nagasaki-shi 852-8521, Japan
[‡]Department of Applied Chemistry, Kanagawa Institute of Technology, 1030, Shimo-ogino, Atsugi, Kanagawa 243-0292, Japan

[*]Corresponding author: Suguru Motokucho, E-mail: motoku@nagasaki-u.ac.jp

Total: 14 pages, 10 figures, 3 tables, and 1 scheme

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## 1. Materials and methods

Poly(hydroxyurethane)[1] (PHU (HPU A-0110)) and modified PHU containing a trimethylsilyl protecting group (TMS-PHU (HPU A0110-TMS)) were supplied by Dainichiseika Color & Chemicals Mfg. Co., Ltd. (Tokyo, Japan). Phenyl glycidyl ether (PGE) and other epoxides were purchased from TCI (Tokyo, Japan). Butyric acid glycidyl ester used as a precursor of 5CC-8 was prepared as described in the literature[2]. 18-Crown-6 was purchased from FUJIFILM Wako Pure Chemical Corporation (Osaka, Japan) and used without further purification. All reagents and solvents were of reagent grade and used as received unless stated otherwise.

$^{1}$H and $^{13}$C nuclear magnetic resonance (NMR) spectra (400 MHz $^{1}$H and 100 MHz $^{13}$C) were recorded on a JNM-GX400 spectrometer (JEOL, Ltd., Japan) in $CDCl_3$ or dimethyl sulfoxide-$d_6$ (DMSO-$d_6$) at room temperature (approximately 20 °C), and chemical shifts were determined using tetramethylsilane as an internal standard. Fourier transform infrared (FT-IR) spectra were recorded on a JASCO FTIR 460 KY Plus spectrometer (Tokyo, Japan), and transmission spectra were obtained using KBr discs. The number- and weight-average molecular weights ($M_n$ and $M_w$, respectively) and polydispersity index ($Đ = M_w/M_n$) values of polymer samples were estimated by gel permeation chromatography (GPC) using a JASCO (JASCO Corp., Japan) high-performance liquid chromatography system (pump: PU-2060; reflective index detector: RI-2031; column oven: CO-4060) equipped with a polystyrene gel column (SB-804 HQ; Shodex Co. Ltd., Japan). Dimethylformamide (DMF) eluent containing 10 mmol/L LiBr was passed at a flow rate of 1.0 mL/min. The system was calibrated with polystyrene standards. Optical rotations were measured in the 50-mm length cell of a JASCO P-2200 digital polarimeter at a temperature of 21 °C and the average value was calculated from the results of ten independent measurements.

## 2. Synthesis and characterization

**Synthesis of PGE-5CC and separation of PHU**: In a typical procedure, 750 mg (5.00 mmol) PGE was added to a mixture of 277 mg (1.00 mmol) PHU and 6.6 mg (0.025 mmol) 18-crown-6 in a 200-mL autoclave. After pouring liquid $CO_2$, the autoclave was sealed and weighed on an electronic balance BPS6K01 (Asone, Japan; resolution: 0.1 g; capability: 6.000 kg) to calculate the weight of $CO_2$ in the reactor set to 39.0 g (890 mmol). Subsequently, the autoclave was heated to 120 °C for 16 h, cooled, and depressurized by venting the reactor bulb. Using an aliquot of the reaction mixture, a $CDCl_3$ solution for $^{1}$H-NMR studies was prepared by removing the insoluble PHU fraction. The conversion of PGE and its selectivity for **PGE-5CC** production were estimated using the NMR singlet signal of 18-crown-6 as an internal standard.

The reaction mixture was dissolved in tetrahydrofuran and poured into a large amount of diethyl ether to precipitate a white solid. After filtration, the obtained solid was dried under vacuum at 40 °C for 20 h to recover PHU (269 mg), which was examined by $^{1}$H-NMR, FT-IR, and GPC techniques. The filtrate was concentrated, and the crude product was purified by $SiO_2$ column chromatography (eluent: hexane/ethyl acetate = 4/1, v/v) to obtain 942 mg (4.85 mmol) **PGE-5CC** as a white solid corresponding to 97% yield.

The reaction, purification, and analysis steps were individually performed to evaluate the effects of variations in the reaction time (4−16 h) and temperature (90−150 °C) on the PGE conversion and its selectivity for **PGE-5CC** production.

**Estimation of the molar ratio of urethane groups ([urethane groups]$_0$) to PGE**: The molar amount of PHU urethane groups was estimated by the following equation:

$$[urethane\ groups]_0 = \frac{[weight\ of\ PHU\ (g)]}{[molecular\ weight\ of\ PHU\ repeating\ unit\ \ [544.64]]} \times 2 \quad \text{(S1)}$$

From the molar amounts of urethane groups and PGE, the molar ratio [urethane groups]$_0$/[PGE]$_0$ was determined.

**Reactions of substituted epoxides with $CO_2$**: Synthesis of **5CC-1** is given as an example here. First, 471 mg (5.09 mmol) epichlorohydrin was added to a mixture of 277 mg (1.00 mmol) PHU and 6.6 mg (0.025 mmol) 18-crown-6 in a 200-mL autoclave. The reaction was performed following the **PGE-5CC** synthesis procedure described above. After the reaction, the resulting crude product was purified by $SiO_2$ column chromatography (eluent: hexane/ethyl acetate = 7/1, v/v) to obtain 640 mg (4.68 mmol) **5CC-1** as a colorless oil with a 92% yield.

**Reaction of PGE with $CO_2$ using TMS-PHU catalyst**: A reaction similar to the PHU-catalyzed process was also performed in this study. 750 mg (5.00 mmol) PGE was added to a mixture of 350 mg (1.00 mmol) TMS-PHU and 6.6 mg (0.025 mmol) 18-crown-6 in a 200 mL autoclave. The autoclave filled with pressured $CO_2$ (5.2 MPa) was heated to 90 °C for 16 h. After the reaction, $^1$H-NMR measurements were performed to estimate the conversion of PGE and selectivity for **PGE-5CC** production using an aliquot of the reaction mixture. The mixture was then re-precipitated from the $CHCl_3$ solution thrice using diethyl ether three times to recover 270 mg of TMS-PHU as a white solid. The resulting diethyl ether solution was concentrated, and the crude product was purified by $SiO_2$ column chromatography (eluent: hexane/ethyl acetate = 4/1, v/v) to obtain 476 mg (2.45 mmol) **PGE-5CC** as a white solid corresponding to a 49% yield.

**Synthesis of PHU from the reaction of 5CC-12 and 1,6-hexamethylenediamine**: 1,6-hexamethylenediamine (1.16 g, 10 mmol) was added to a solution of **5CC-12** (4.28 g, 10 mmol) in DMF (20 mL) under nitrogen atmosphere. The mixture was stirred for 24 h at 100 °C. The reaction mixture was poured into 450 mL of diethyl ether resulting in the precipitate of a white solid. The precipitate was collected by filtration, washed with diethyl ether, and dried *in vacuo* at 50 °C to give PHU as a white solid (4.77 g, 87 % yield). The obtained PHU exhibited $^1$H-NMR spectrum and GPC profile very similar to that of the PHU supplied by Dainichiseika Color & Chemicals Mfg. Co., Ltd.

$M_n = 1.02\times10^4$, $M_w = 2.18\times10^4$, $Đ = 2.14$.

$^1$H-NMR spectrum and GPC profile were shown in **Figure S5** and **S6**.

**3. Estimations of PGE conversion and selectivity by $^1$H-NMR (see Figure S1)**

The conversion of PGE and selectivity for **PGE-5CC** production were estimated via the following equations:

$$\text{Conversion of PGE (\%)} = (1 - \frac{f/200}{X/24}) \times 100 \quad \text{(S2)}$$

$$\text{Selectivity of the reaction for the production of PGE-5CC (\%)} = \frac{F/200}{X/24}/(1 - \frac{f/200}{X/24}) \times 100 \quad \text{(S3)}$$

where $f$ = integral ratio of PGE methine protons (from Figure S1)

$F$ = integral ratio of **PGE-5CC** methine protons (from Figure S1)

$X$ = integral ratio of 18-crown-6 used as an internal standard.

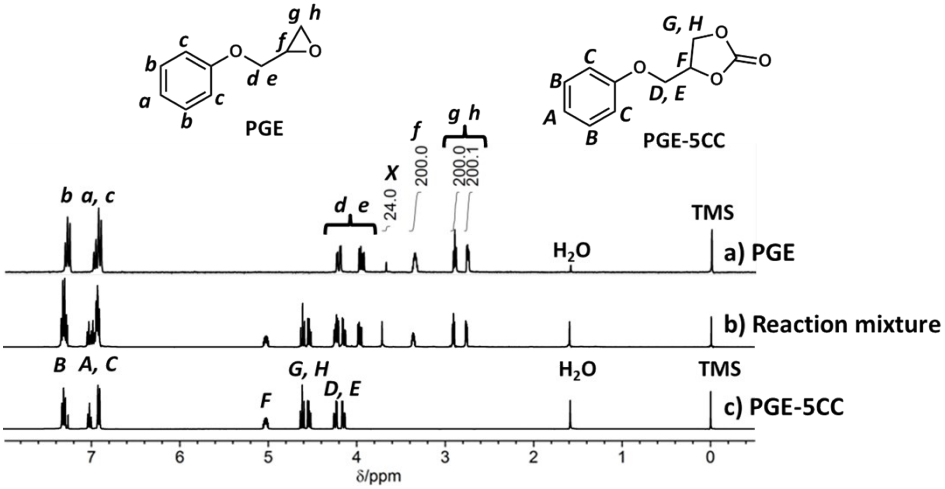



**Figure S1**. $^1$H-NMR ($CDCl_3$) spectra of a) PGE, b) the reaction mixture, and c) **PGE-5CC**. Reaction conditions: 90 °C, 16 h, and 5.2 MPa $CO_2$ pressure in the presence of PHU ([urethane groups]$_0$/[PGE]$_0$ = 0.20). Signal ***X*** is generated by 18-crown-6.

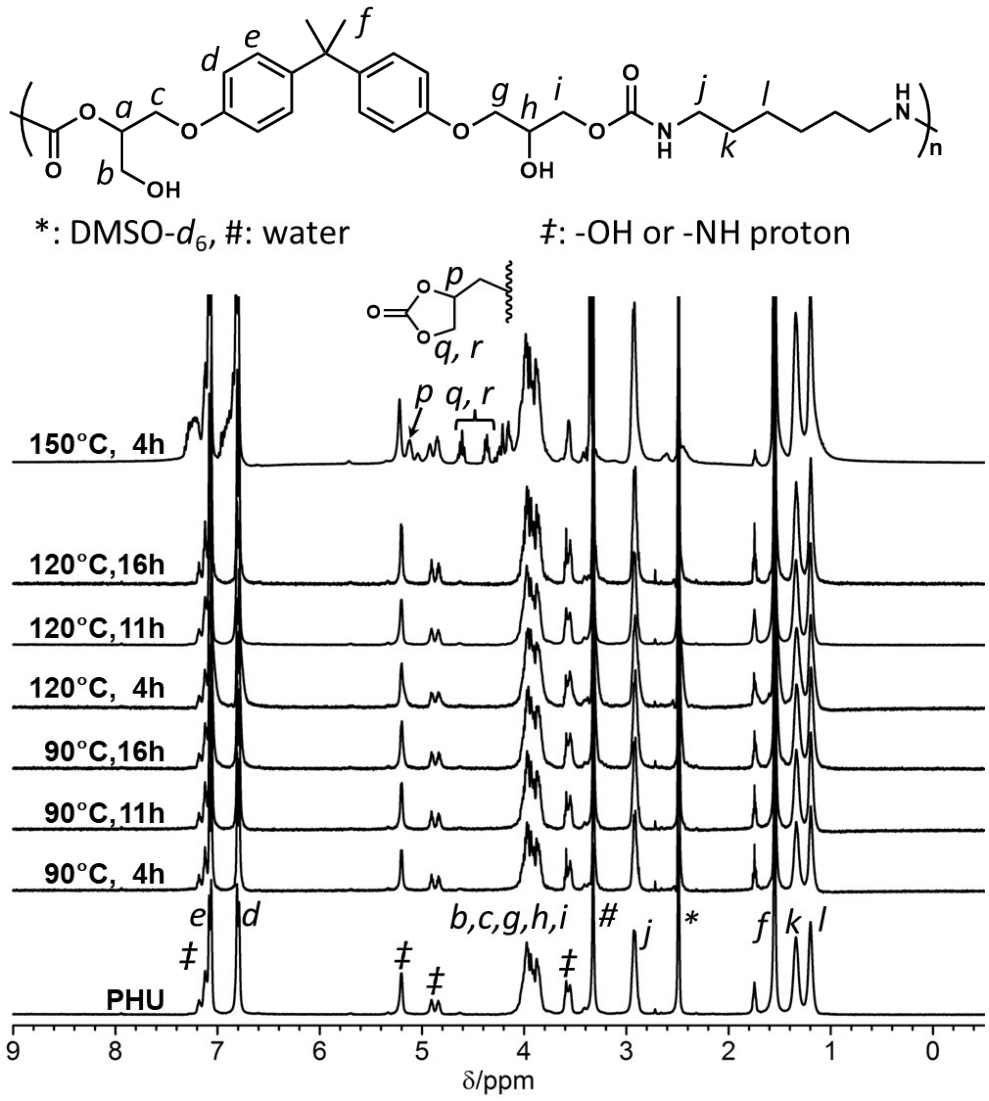



**Figure S2**. $^1$H-NMR (DMSO-$d_6$) spectra of the pristine PHU and PHU after the reaction by filtration.

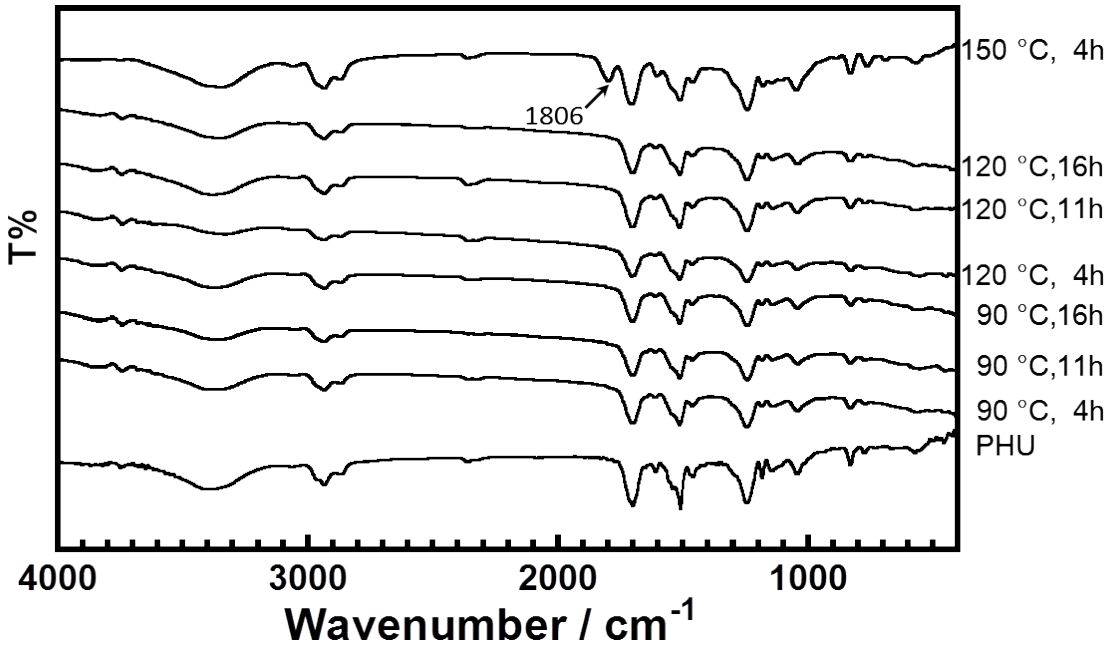



**Figure S3**. FT-IR spectra of the pristine PHU and PHU after the reaction by filtration.

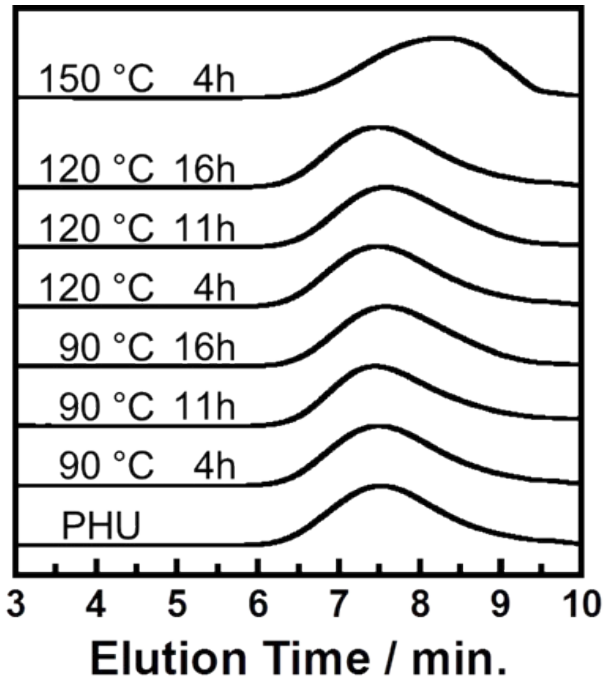



**Figure S4**. GPC profiles of the pristine and recovered PHU. DMF was used as the eluent.

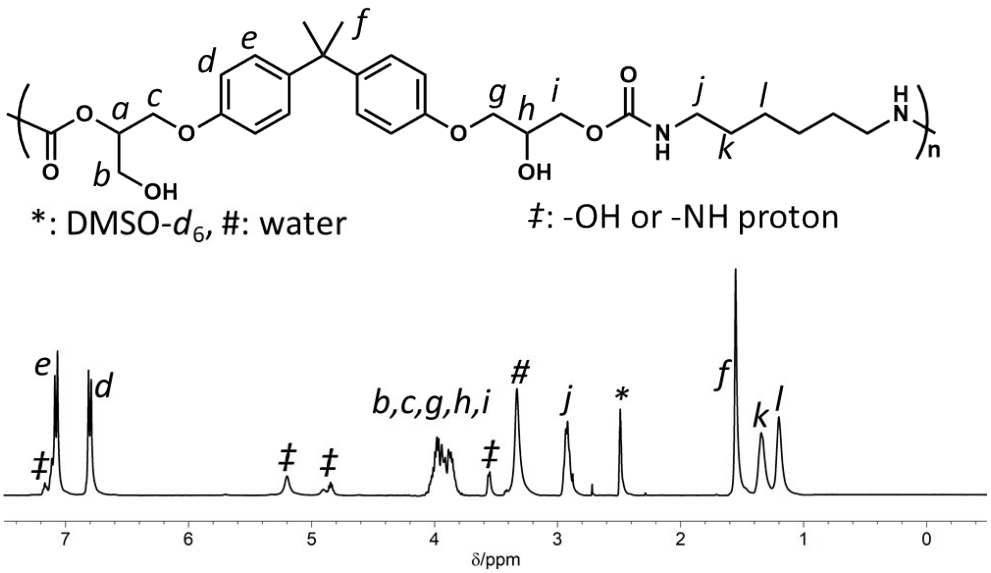



**Figure S5**. $^1$H-NMR (DMSO-$d_6$) spectrum of the PHU synthesized from **5CC-12** and 1,6-hexamethylenediamine.

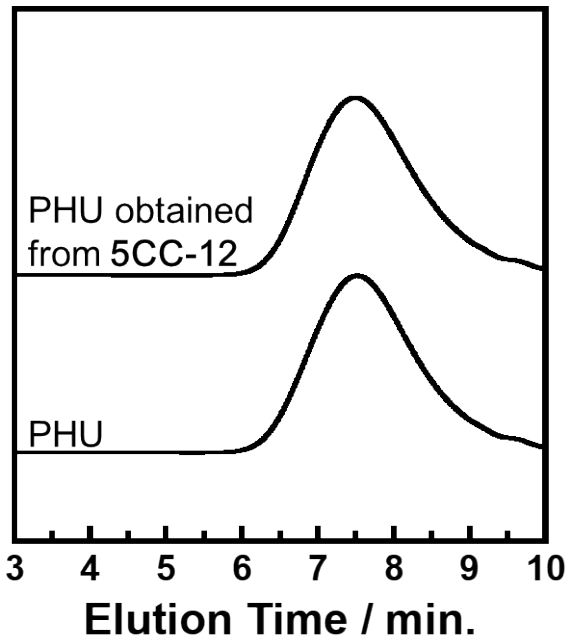



**Figure S6**. GPC profiles of the pristine PHU and PHU synthesized from **5CC-12** and 1,6-hexamethylenediamine.

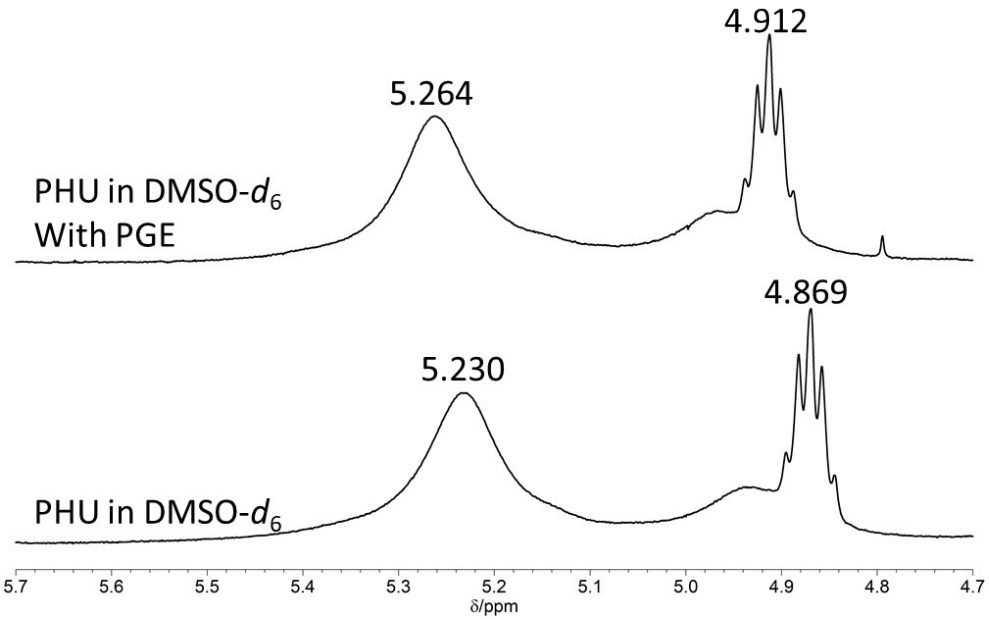



**Figure S7**. $^1$H-NMR (400 MHz) spectra of PHU (0.32 mmol of urethane moiety) and the mixture of PGE (0.64 mmol) and PHU (0.32 mmol) in 0.8 mL of DMSO-$d_6$.

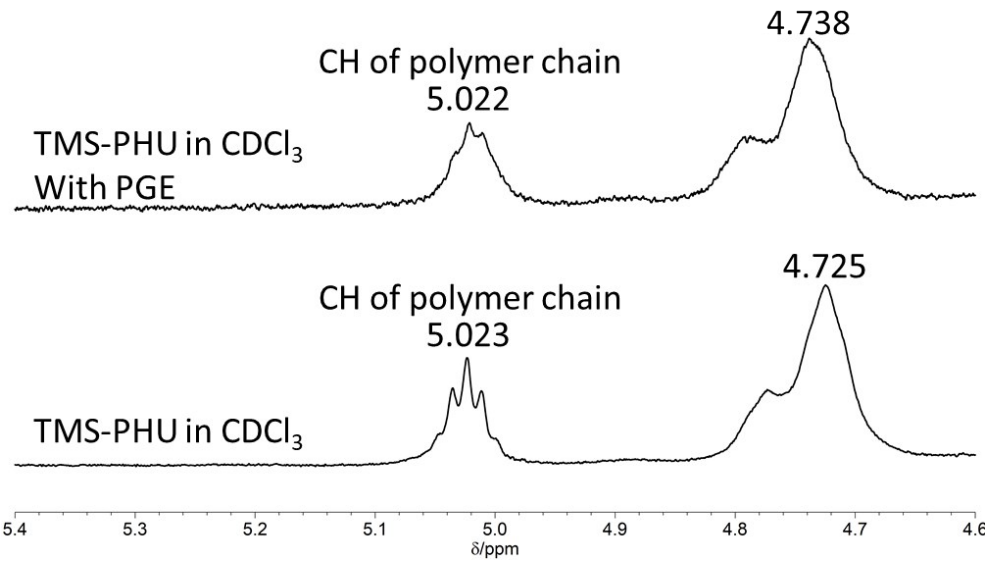



**Figure S8**. $^1$H-NMR (400 MHz) spectra of TMS-PHU (0.32 mmol of urethane moiety) and the mixture of PGE (0.64 mmol) with TMS-PHU (0.32 mmol) in $CDCl_3$.

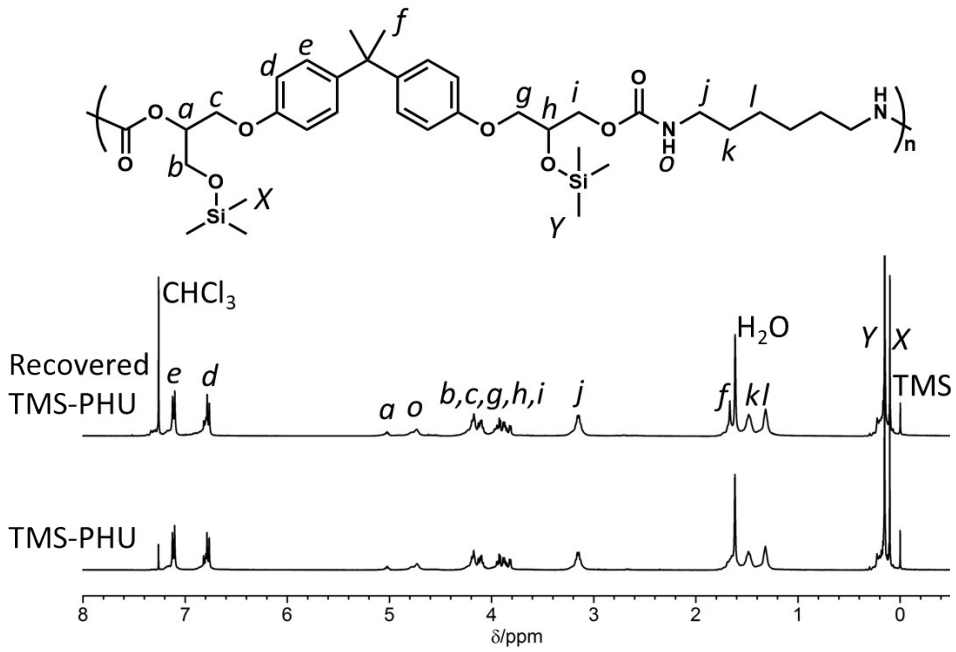



**Figure S9**. $^{1}$H-NMR ($CDCl_3$) spectra of the pristine TMS-PHU and the TMS-PHU recovered after reaction by filtration.

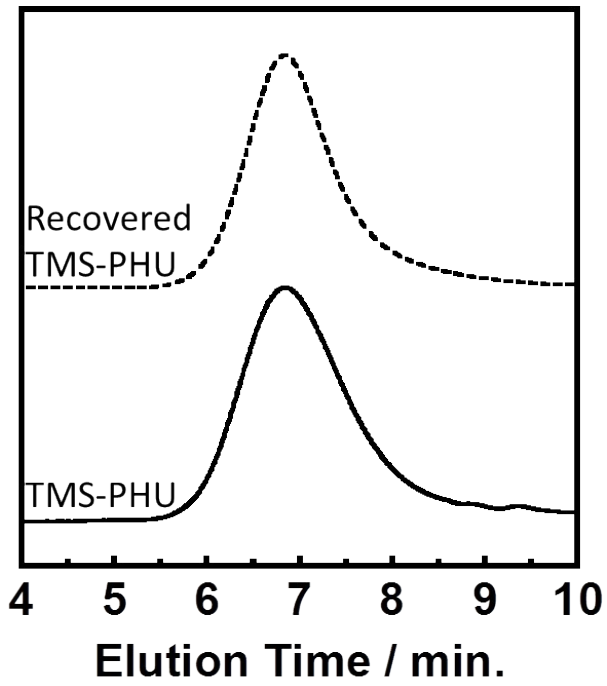



**Figure S10**. GPC profiles of the pristine and recovered TMS-PHU. DMF was used as the eluent.

**Table S1**. Weights of the added and recovered PHU.

| Entry | Temp. / °C | Time / h | PHU weight / g | Weight of recovered PHU / g |
|---|---|---|---|---|
| 4 | 90 | 4 | 0.275 | 0.267 |
| 5 | 90 | 11 | 0.274 | 0.269 |
| 6 | 90 | 16 | 0.275 | 0.263 |
| 8 | 120 | 4 | 0.277 | 0.266 |
| 9 | 120 | 11 | 0.282 | 0.273 |
| 10 | 120 | 16 | 0.277 | 0.269 |
| 11 | 150 | 4 | 0.277 | 0.220 |

The entry numbers correspond to those of Table 1 in the main file.

**Table S2**. $M_n$, $M_w$, and $Đ$ ($M_w/M_n$) values of the added and recovered PHU.

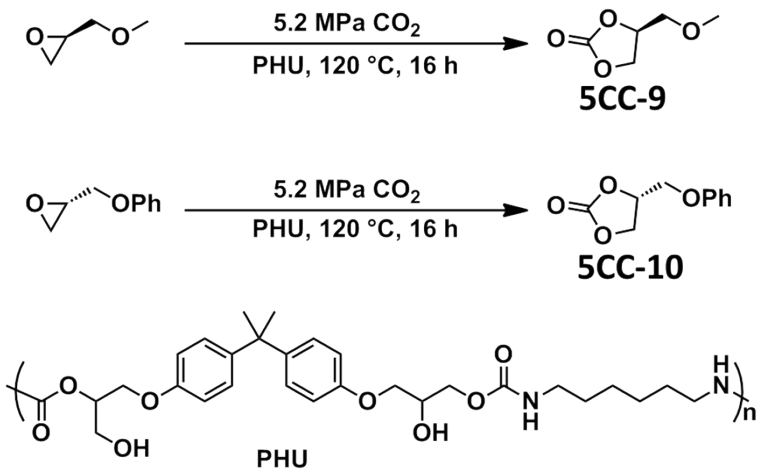
| | $M_n$ /$10^4$ | $M_w$ /$10^4$ | $Đ$ |
|---|---|---|---|
| PHU | 1.03 | 2.19 | 2.13 |
| Entry 11 (150 °C, 4 h) | 0.34 | 0.87 | 2.59 |

**$M_n$: number-average molecular weight; $M_w$: weight-average molecular weight;** $Đ$: dispersity defined as **$M_w/M_n$. The samples** are identical to those analyzed in Figure S4.

**Table S3**. $M_n$, $M_w$, and $Đ$ ($M_w/M_n$) values of the added and recovered TMS-PHU.

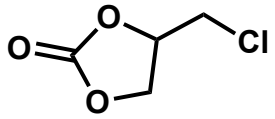
| | $M_n$ /$10^4$ | $M_w$ /$10^4$ | $Đ$ |
|---|---|---|---|
| TMS-PHU | 2.33 | 4.55 | 1.95 |
| Recovered TMS-PHU | 2.25 | 4.44 | 1.97 |

**$M_n$: number-average molecular weight; $M_w$: weight-average molecular weight;** $Đ$: dispersity defined as **$M_w/M_n$. The samples** are identical to those analyzed in Figure S6.

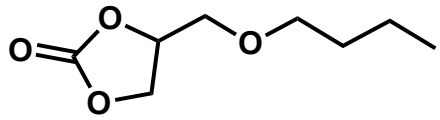



**Scheme S1**. PHU-catalyzed syntheses of 5CCs (**5CC-9** and **5CC-10**) through the reaction of optically active epoxides and $CO_2$.

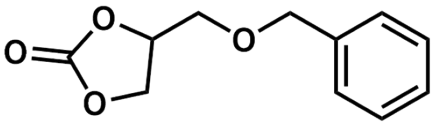
**4. Characterization of 5CCs**



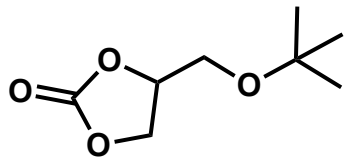
**4-(chloromethyl)-1,3-dioxolan-2-one (5CC-1)**[3, 4]: Colorless oil; $^1$H-NMR (400 MHz, $CDCl_3$): $\delta$ 5.00–4.94 (m, 1H), 4.60 (dd, $J$ = 8.4, 8.8 Hz, 1H), 4.42 (dd, $J$ = 5.6, 8.8 Hz, 1H), 3.81–3.70 (m, 2H). $^{13}$C-NMR (100 MHz, $CDCl_3$): $\delta$ 154.0, 74.2, 67.0, 43.5.



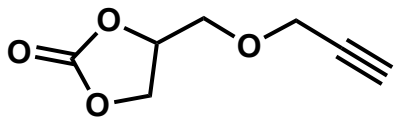
**4-(butoxymethyl)-1,3-dioxolan-2-one (5CC-2)** [5]: Colorless oil; $^1$H-NMR (400 MHz, $CDCl_3$): $\delta$ 4.84–4.81 (m, 1H), 4.53–4.48 (m, 1H), 4.39 (dd, $J$ = 6.0, 7.2 Hz, 1H), 3.70–3.66 (m, 1H), 3.62–3.58(m, 1H), 3.51(dd, $J$ = 8.4, 8.4Hz, 2H), 1.61–1.52 (m, 2H), 1.41–1.32 (m, 2H), 0.92 (t, $J$ = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$): $\delta$ 155.0, 75.1, 71.7, 69.5, 66.2, 31.4, 19.0, 13.7.



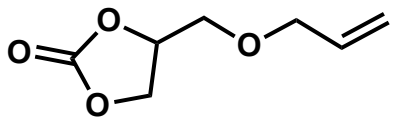
**4-((benzyloxy)methyl)-1,3-dioxolan-2-one (5CC-3)**[4, 5]: White solid; $^1$H-NMR (400 MHz, $CDCl_3$): $\delta$ 7.36−7.31 (m, 5H), 4.84–4.78 (m, 1H), 4.62 (d, $J$ = 12.0 Hz, 1H), 4.57 (d, $J$ = 12.0 Hz, 1H), 4.48 (dd, $J$ = 8.4, 8.4 Hz, 1H), 4.38 (dd, $J$ = 8.4, 6.1 Hz, 1H), 3.73–3.60 (m, 2H). $^{13}$C-NMR (100 MHz, $CDCl_3$): $\delta$ 154.9, 137.0, 128.5, 128.0, 127.7, 74.9, 73.6, 68.8, 66.2.



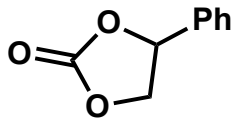
**4-(tert-butoxymethyl)-1,3-dioxolan-2-one (5CC-4)**[3]: Colorless oil; $^1$H-NMR (400 MHz, $CDCl_3$): $\delta$ 4.82–4.76 (m, 1H), 4.49 (dd, $J$ = 8.0, 8.0 Hz, 1H), 4.39 (dd, $J$ = 8.0, 8.4 Hz, 1H), 3.65–3.51 (m, 2H), 1.20 (s, 9H). $^{13}$C-NMR (100 MHz, $CDCl_3$): $\delta$155.2, 75.1, 73.7, 66.4, 61.2, 27.2.



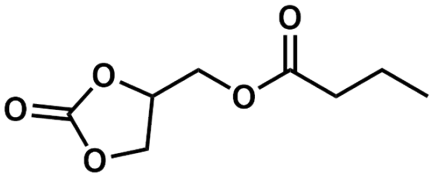
**4-((prop-2-yn-1-yloxy)methyl)-1,3-dioxolan-2-one (5CC-5)**[5]: Colorless oil; $^1$H-NMR (400 MHz, $CDCl_3$): $\delta$ 4.89–4.84 (m, 1H), 4.52 (dd, $J$ = 8.0, 8.0 Hz, 1H), 4.41 (dd, $J$ = 6.0, 8.4 Hz, 1H), 4.30–4.19 (m, 2H), 3.82–3.73 (m, 2H), 2.51 (t, $J$ = 2.3 Hz, 1H). $^{13}$C-NMR (100 MHz, $CDCl_3$): $\delta$ 154.8, 78.5, 75.6, 74.6, 68.4, 66.2, 58.8.



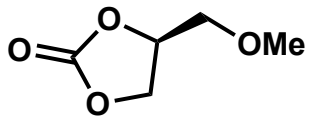
**4-((allyloxy)methyl)-1,3-dioxolan-2-one (5CC-6)**[3, 4]: Colorless oil; $^1$H-NMR (400 MHz, $CDCl_3$): $\delta$ 5.91–5.83 (m, 1H), 5.32–5.22 (m, 2H), 4.85–4.81 (m, 1H), 4.51 (dd, $J$ = 8.2, 8.2 Hz, 1H), 4.40 (dd, $J$ = 6.0, 8.4 Hz, 1H), 4.08–4.05 (m, 2H), 3.70 (dd, $J$ = 4.0, 11.2 Hz, 1H), 3.62 (dd, $J$ = 4.0, 11.2 Hz, 1H). $^{13}$C-NMR (100 MHz, $CDCl_3$): $\delta$ 154.9, 133.6, 117.9, 75.0, 72.6, 68.8, 66.2.



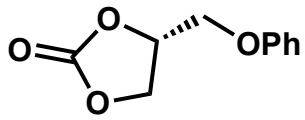
**4-Phenyl-1,3-dioxolan-2-one (5CC-7)**[3–5]: White solid; $^1$H-NMR (400 MHz, $CDCl_3$): $\delta$ 7.46–7.35 (m, 5H), 5.68 (dd, $J$ = 8.0, 8.0 Hz, 1H), 4.80 (dd, $J$ = 8.0, 8.4 Hz, 1H), 4.35 (dd, $J$ = 8.0, 8.4 Hz, 1H). $^{13}$C-NMR (100 MHz, $CDCl_3$): $\delta$ 154.8, 135.7, 129.7, 129.2, 125.8, 78.0, 71.1.



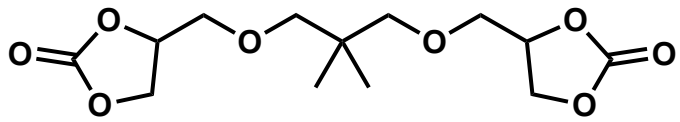
**(2-oxo-1,3-dioxolan-4-yl)methyl butyrate (5CC-8)**[6]: Colorless oil; $^1$H-NMR (400 MHz, $CDCl_3$): $\delta$ 4.99–4.94 (m, 1H), 4.58 (dd, $J$ = 8.7, 8.5 Hz, 1H), 4.40–4.25 (m, 3H), 2.36 (t, $J$ = 7.4 Hz, 2H), 1.71–1.62 (m, 2H), 0.96 (t, $J$ = 7.5 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$): $\delta$ 173.0, 154.5, 73.8, 65.9, 62.7, 35.6, 18.1, 13.4.



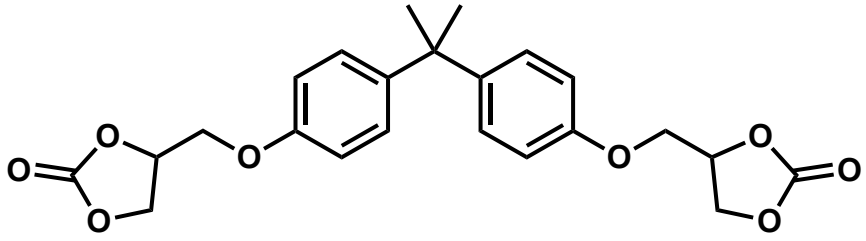
**(*S*)-4-(methoxymethyl)-1,3-dioxolan-2-one (5CC-9)**[7]: Colorless oil; $^1$H-NMR (400 MHz, $CDCl_3$) $\delta$ 4.84–4.79 (m, 1H), 4.50 (dd, $J$ = 8.0, 8.0 Hz, 1H), 4.38 (dd, $J$ = 6.0, 8.0 Hz, 1H), 3.65 (dd, $J$ = 4.0, 10.8 Hz, 1H), 3.57 (dd, $J$ = 3.6, 10.8 Hz, 1H), 3.43 (s, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$): $\delta$ 154.9, 75.0, 71.2, 65.9, 59.2. $[a]_D^{21}$ = -35.3 ($c$ = 1.0, EtOH) [lit.7 $[a]_D^{24}$ = -36.5 ($c$ = 1.0, EtOH, >98% ee (*S*))].



**(*R*)-4-(phenoxymethyl)-1,3-dioxolan-2-one (5CC-10)**[4]: White solid; $^1$H-NMR (400 MHz, $CDCl_3$) $\delta$ 7.28 (t, $J$ = 8.0 Hz, 2H), 7.00 (t, $J$ = 7.6 Hz, 1H), 6.90 (d, $J$ = 8.4 Hz, 2H), 5.03–4.98 (m, 1H), 4.58 (dd, $J$ =8.4, 8.4 Hz, 1H), 4.50 (dd, $J$ = 6.0, 8.8 Hz, 1H), 4.24 (dd, $J$ = 4.0, 10.8 Hz, 1H), 4.10 (dd, $J$ = 3.6, 10.8 Hz, 1H). $^{13}$C-NMR (100 MHz, $CDCl_3$): $\delta$ 157.7, 154.7, 129.6, 121.8, 114.5, 74.2, 66.8, 66.1.
$[a]_D^{21}$ = +18.0 ($c$ = 1.0, EtOH) [lit.4 $[a]_D^{20}$ = +18.3 ($c$ = 1.2, EtOH; 99% ee (*R*))].



**4,4'-(((2,2-dimethylpropane-1,3-diyl)bis(oxy))bis(methylene))bis(1,3-dioxolan-2-one) (5CC-11)**[1]: White solid; $^1$H-NMR (400 MHz, $CDCl_3$) $\delta$ 4.85–4.82 (m, 2H), 4.54–4.36 (m, 4H), 3.73–3.70 (m, 2H), 3.60–3.57 (m, 2H), 3.29–3.23 (m, 4H), 0.88 (s, 6H). $^{13}$C-NMR (100 MHz, $CDCl_3$, mixture of stereoisomers): $\delta$ 155.1, 76.7, 76.6, 75.2, 70.3, 70.0, 66.1, 36.2, 21.8.



**4,4'-(((propane-2,2-diylbis(4,1-phenylene))bis(oxy))bis(methylene))bis(1,3-dioxolan-2-one) (5CC-12)**[1]: White solid; $^1$H-NMR (400 MHz, $CDCl_3$): $\delta$ 7.14 (d, $J$ = 8.8 Hz, 4H), 6.81 (d, $J$ = 8.8 Hz, 4H), 5.04–4.98 (m, 2H), 4.60 (dd, $J$ = 8.5, 8.5 Hz, 2H), 4.52 (dd, $J$ = 5.9, 8.5 Hz, 2H), 4.23 (dd, $J$ = 4.3, 10.6 Hz, 2H), 4.12 (dd, $J$ = 3.5, 10.7 Hz, 2H), 1.63 (s, 6H). $^{13}$C-NMR (100 MHz, $CDCl_3$): $\delta$155.4, 154.4, 144.1, 127.7,

113.8, 73.9, 66.7, 66.0, 41.6, 30.7.

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