Electronic Supplementary Material (ESI) for Reaction Chemistry & Engineering.

Supporting Information

Turning CO/CO_2-containing industrial process gas into valuable building blocks for the polyurethane industry

Martin R. Machat*[a,b], Jakob Marbach[a], Hannah Schumacher[a], Suresh Raju[b], Markus Lansing[b], Lena C. Over[b], Liv Adler[a], Jens Langanke[a], Aurel Wolf[a], Walter Leitner*[b,c] and Christoph Gürtler*[a]

[a] *Covestro Deutschland AG, Catalysis and Technology Incubation, Kaiser-Wilhelm-Allee 60, 51368 Leverkusen, Germany. E-mail: martin.machat@covestro.com; christoph.guertler@covestro.com*

[b] *RWTH-Aachen University, CAT Catalytic Center, Worringer Weg 2, 52074 Aachen, Germany. E-mail: leitner@itmc.rwth-aachen.de*

[c] *Max-Planck-Institut für Chemische Energiekonversion, Stiftstraße 34-36, 45470 Mülheim an der Ruhr, Germany.*

Contents

1 Experimental Section

1.1 Materials

The following chemicals were used as received if not stated otherwise: benzyldimethylamine (Sigma Aldrich), $Co_2(CO)_8$ moistened with hexane (hexane 1-10%, Sigma Aldrich), Desmodur 44V20L (Covestro), Desmorapid 1792 (Covestro), Desmorapid 726B (Covestro), diethylene glycol (Sigma Aldrich), DMC catalyst (dried and grinded double metal cyanide catalyst, synthesized according to WO0180994, example 6), ethylene glycol (Sigma Aldrich), ethylene oxide (GHC), Pc-AlCl (Sigma Aldrich), α,ω-polypropylene glycol (average MW: 435 g/mol, f = 2, Covestro), propylene oxide (Sigma Aldrich), reaction gases (CO_2 (4.5), CO (4.7), CO_2/H_2 (5.02% H_2), Linde and N_2 (4.6), Air Liquide), sodium hydroxide pellets (Honeywell, ground before use), Stepanpol PS 2352 (Stepan), succinic anhydride (Polynt/abcr), Tegostab B8421 (Evonik), THF (anhydrous, Sigma Aldrich), tris(1-chloro-2-propyl)phosphate (Lanxess). $Na[Co(CO)_4]$ was prepared according to literature[1] and stored at -25 °C for prolonged time.

1.2 Chemical Procedures

1.2.1 CO_2 Conversion

1.2.1.1 Copolymerization of CO_2 and propylene oxide in batch-mode (reference reaction with pure CO_2)

A 2.0 L stainless steel reactor was charged with α,ω-polypropylene glycol (average MW: 435 g/mol, f = 2, 50.0 g, 0.116 mol, 1.00 eq.) and DMC catalyst (0.058 g, 500 ppm). The reactor was closed and heated to 130 °C under reduced pressure and constant N_2 flow. The reactor was pressurized with 25 bar of CO_2. After an activation sequence with propylene oxide (2 × 5.0 g) at 130 °C, propylene oxide (55.7 g; total PO amount: 65.7 g, 1.13 mol, 9.75 eq.) was continuously added over the course of 180 min at 115 °C, while the pressure was kept constant by additional dosing of CO_2. After stirring for 60 min at 115 °C, the reactor was cooled down, released of pressure and purged with N_2. Volatile components were removed under vacuum to obtain the product as a colorless wax (98% PO-conversion (average over three repeating experiments)).

1.2.1.2 Copolymerization of CO_2 and propylene oxide in batch-mode (reaction with mixture of CO_2 and CO/N_2 (1:1))

A 2.0 L stainless steel reactor was charged with α,ω-polypropylene glycol (average MW: 435 g/mol, f = 2, 50.0 g, 0.116 mol, 1.00 eq.) and DMC catalyst (0.058 g, 500 ppm). The reactor was closed and heated to 130 °C under reduced pressure and constant N_2 flow. The reactor was pressurized with 25 bar of CO_2 before the second gas component (CO or N_2) was added up to a total pressure of 50 bar. After an activation sequence with propylene oxide (2 × 5.0 g) at 130 °C, propylene oxide (55.7 g; total PO-amount: 65.7 g, 1.13 mol, 9.75 eq.) was continuously added over the course of 180 min at 115 °C, while the pressure was kept constant by additional dosing of CO_2. After stirring for 60 min at 115 °C, the reactor was cooled down, released of pressure and purged with N_2. Volatile components were removed under vacuum to obtain the product as a colorless wax.

1.2.1.3 *Copolymerization of CO_2 and propylene oxide in batch-mode (reaction with mixture of CO_2 and H_2 (5%))*

A 2.0 L stainless steel reactor was charged with α, ω-polypropylene glycol (average MW: 435 g/mol, f = 2, 50.0 g, 0.116 mol, 1.00 eq.) and DMC catalyst (0.058 g, 500 ppm). The reactor was closed and heated to 130 °C under reduced pressure and constant N_2 flow. The reactor was pressurized with 50 bar of a CO_2/H_2-mixture (5% H_2). After an activation sequence with propylene oxide (2 × 5.0 g) at 130 °C, propylene oxide (55.7 g; total PO amount: 65.7 g, 1.13 mol, 9.75 eq.) was continuously added over the course of 180 min at 115 °C, while the pressure was kept constant by additional dosing of the CO_2/H_2-mixture (5% H_2). After stirring for 60 min at 115 °C, the reactor was cooled down, released of pressure and purged with N_2. Volatile components were removed under vacuum to obtain the product as a colorless wax (99% PO-conversion (average over three repeating experiments)).

1.2.1.4 *Copolymerization of CO_2 and propylene oxide in continuous mode (reference reaction with pure CO_2)*

A 2.0 L stainless steel reactor was charged with α, ω-polypropylene glycol (average MW: 435 g/mol, f = 2, 150.0 g, 0.345 mol, 1.00 eq.) and DMC catalyst (0.208 g, 300 ppm). The reactor was closed and heated to 115 °C under reduced pressure and constant N_2 flow. The reactor was pressurized with 3 bar of N_2. After an activation sequence with propylene oxide (2 × 15.0 g) at 115 °C, the reactor was pressurized with 12.5 bar of CO_2. Propylene oxide (514 g; total PO-amount: 544 g, 9.37 mol, 27.1 eq.) was continuously added over the course of 180 min at 115 °C while the pressure was kept constant by additional dosing of CO_2. The temperature was reduced to 107 °C (1 °C/min). After stirring for 60 min at 107 °C, the reactor was cooled down, released of pressure and purged with N_2. Volatile components were removed under vacuum to obtain the product as a colorless wax.

1.2.1.5 *Copolymerization of CO_2 and propylene oxide in continuous mode (exemplary reaction with mixture of CO_2 and N_2 (1:1) and CO_2 volume flow of 100 mL/min)*

A 2.0 L stainless steel reactor was charged with α, ω-polypropylene glycol (average MW: 435 g/mol, f = 2, 150.0 g, 0.345 mol, 1.00 eq.) and DMC catalyst (0.208 g, 300 ppm). The reactor was closed and heated to 115 °C under reduced pressure and constant N_2 flow. The reactor was pressurized with 3 bar of N_2. After an activation sequence with propylene oxide (2 × 15.0 g) at 115 °C, the reactor was pressurized with 12.5 bar of N_2 and 12.5 bar of CO_2. Propylene oxide (514 g; total PO-amount: 544 g, 9.37 mol, 27.1 eq.) was continuously added over the course of 180 min at 115 °C while continuously dosing N_2 and CO_2 with a volume flow of 100 mL/min each. The pressure was kept constant at 25 bar while the temperature was reduced to 107 °C (1 °C/min). After stirring for 60 min at 107 °C, the reactor was cooled down, released of pressure and purged with N_2. Volatile components were removed under vacuum to obtain the product as a colorless wax.

Table SI 1: CO_2 incorporation and conversion results for the performed copolymerization procedures of CO_2 and propylene oxide.

Entry	p (CO_2) [bar]	p (N_2) [bar]	Flow rate (CO_2) [mL/min]	Flow rate (N_2) [mL/min]	CO_2 incorporation [wt.-%]	CO_2 conversion [%]
1 (ref.)	12.5	-	-	-	7.62	100
2	12.5	12.5	100	100	5.38	102
3	12.5	25.0	100	200	5.06	94
4	12.5	37.5	100	300	5.16	94
5	12.5	12.5	200	200	6.34	63
6	12.5	25.0	200	400	5.83	58
7	12.5	37.5	200	600	5.75	58
8	12.5	12.5	300	300	6.84	46
9	12.5	25.0	300	600	6.34	43

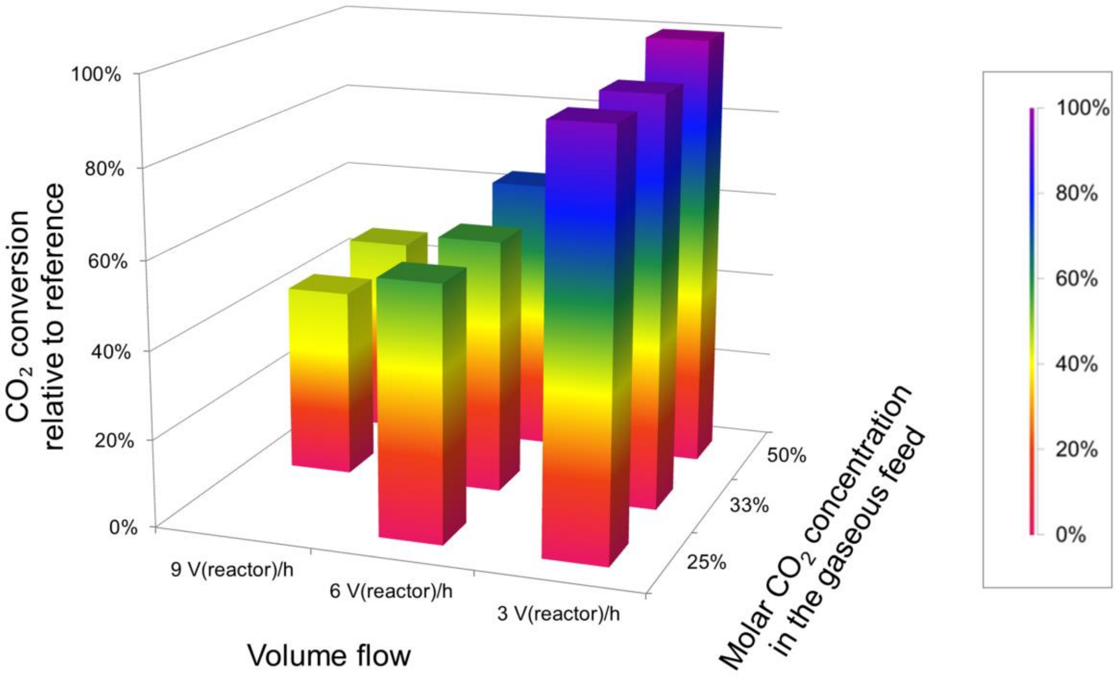

Figure SI 1: Impact of CO_2 concentration in the gas feed (due to addition of varying amounts of N_2) and CO_2 volume flow (N_2 volume flow adjusted accordingly) on the CO_2 conversion normalized referring to the experiment of pure CO_2 for the DMC-catalyzed copolymerization of CO_2 and propylene oxide determined by ^1H NMR spectroscopy.

1.2.2 CO Conversion

1.2.2.1 Small-scale synthesis of [Pc-Al][Co(CO)$_4$]

To the solid mixture of Pc-AlCl (500 mg, 0.870 mmol, 1.00 eq.) and Na[Co(CO)$_4$] (169 mg, 0.871 mmol 1.00 eq.) in a Schlenk flask, THF (30 mL) was added under light protected conditions. The mixture was stirred at room temperature for 6 h. Then the mixture was filtered through a cannula glass-fiber filter and the volatiles were removed to obtain a blue solid [Pc-Al][$Co(CO)_4$] (507 mg, 0.713 mmol, 82%). Analytical data are identical to literature.[2]

1.2.2.2 *Upscaling synthesis of [Pc-Al][$Co(CO)_4$]*

In a dry Schlenk flask, $Co_2(CO)_8$ (5.00 g, 14.6 mmol, 1.00 eq.) and dry THF (50 mL) were added under Ar atmosphere. The reactor was cooled to 0 ℃ using a cryogenic cooling setup. Under light protected conditions, NaOH (6.00 g, 150 mmol, 10.3 eq.) was added and the mixture was stirred for 6 h at 0 °C. Then the solution was decanted from the solid residue. The solid was further washed with excess THF several times and all fractions were combined.

A dry 2 L glass reactor (double-walled) fitted with a mechanical stirrer was charged with Pc-AlCl (13.1 g, 22.8 mmol, 1.56 eq.) and THF (750 mL). Then the THF solution of Na[$Co(CO)_4$] (750 mL) was added and the mixture was stirred for 3 h at room temperature. After the reaction, the solution was filtered and the solvent was removed under vacuum to obtain a blue solid (16.8 g, 86%).

1.2.2.3 *Pilot-plant production of [Pc-Al][$Co(CO)_4$]*

A dry 2 L glass reactor fitted with a reflux condenser (connected with a jacket cooling) was charged with $Co_2(CO)_8$ (50.0 g, 0.146 mol, 1.00 eq.) under inert atmosphere. Then THF (1 L) was added and cooled down to 0 ℃. The reactor was covered with aluminum foil to avoid light exposure. NaOH powder (60.0 g, 1.50 mol, 10.3 eq.) was added under Ar atmosphere in three portions under vigorous stirring. The reaction was allowed to stir for 4 h at 0 ℃ and then slowly warmed to room temperature (10 ℃ per hour). The mixture was gently stirred overnight under light-protected conditions. After allowing the solid to precipitate (1 min), the supernatant solution was collected. The solid was further mixed with additional THF (2 × 0.65 L) to collect the supernatant solution and all fractions were combined.

A dry 12.5 L reactor was charged with Pc-AlCl (151 g, 0.263 mmol, 1.80 eq.) under inert atmosphere and THF (7.55 L) was added. Then the previously collected THF solution of Na[$Co(CO)_4$] was mixed and stirred vigorously for 3 h at room temperature. The mixture was gently stirred overnight. At the end of reaction, the solution was filtered through an inline-filter (P3, 150 mm Ø) and collected in a dry 25 L distillation setup. The mixture was concentrated to 1.5 L under reduced pressure. Further complete drying of the mixture under vacuum resulted in a blue crystalline material of [Pc-Al$(THF)_2$][$Co(CO)_4$] (221 g, 0.258 mol, 98%). The yield was determined based on the starting Pc-AlCl. The quality of the catalyst was determined by FT-IR spectroscopy. The blue solid was used without additional purification for catalytic epoxide carbonylation reactions.

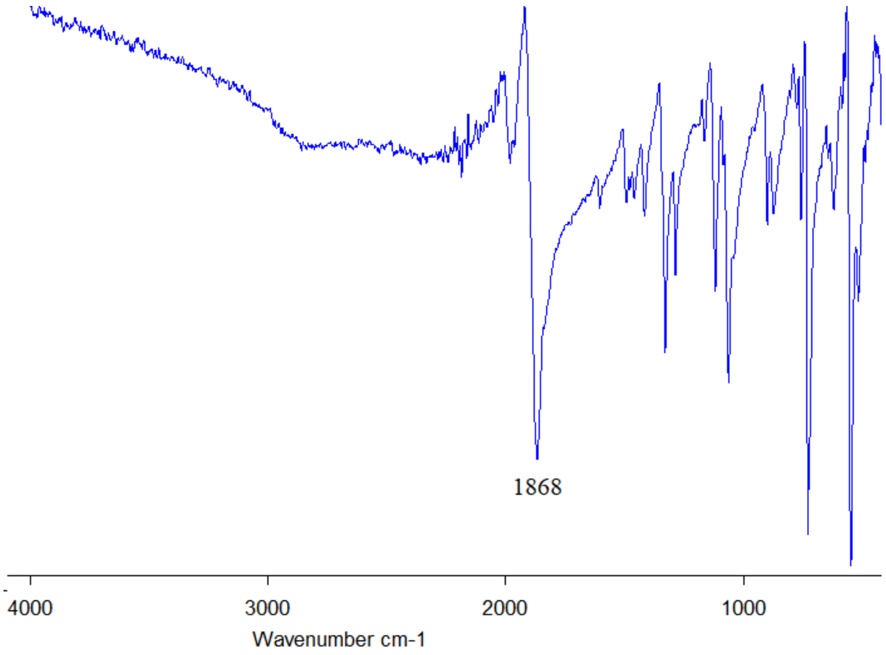

Figure SI 2: ATR FT-IR spectrum of [Pc-Al][$Co(CO)_4$] from $NaCo(CO)_4$ and Pc-AlCl on technical scale (see 1.2.2.3).

1.2.2.4 *Carbonylation of ethylene oxide to β-propiolactone and succinic anhydride using [Pc-Al][$Co(CO)_4$]*

The following scalable procedure was developed for carbonylation trials on mL – L scale. A stainless steel reactor was charged with a solution of [Pc-Al][$Co(CO)_4$] (20 mmol/L, 1.00 mol% catalyst relative to the epoxide) in THF under inert atmosphere. The reactor was heated to 60 °C and pressurized with 3 bar N_2 before CO was added up to a total pressure of 60 bar. Ethylene oxide (EO/cat =100) was added with 10 mL/min and the reaction mixture was stirred at 55 °C for 4 h while the pressure was kept constant by additional dosing of CO. The temperature was increased to 100 °C. After 2 h the reactor was cooled down, released of pressure and purged with N_2. The reaction mixture was analyzed by ^{1}H NMR spectroscopy using naphthalene as an internal standard.

^{1}H NMR (600 MHz, $CDCl_3$): $\delta = 3.00$ (s, 4 H, CH_2) ppm.

1.2.3 Polyol Synthesis

1.2.3.1 *Copolymerization of EO and succinic anhydride*

Polyol 1: A 2 L stainless steel reactor was charged with diethylene glycol (100 g, 0.942 mol, 1.00 eq.), succinic anhydride (283 g, 2.83 mol, 3.00 eq.) and benzyldimethylamine (590 mg, 1000 ppm). The reactor was closed and repeatedly filled with N_2 (3 × 25 bar N_2 $\rightarrow$ 1–2 bar N_2). Under N_2 atmosphere, the reactor was heated to 125 °C and stirred at 1200 U/min for 60 min to melt succinic anhydride. The N_2 pressure was increased to 45 bar and ethylene oxide (208 g, 4.71 mol, 4.71 eq.) was continuously added over the course of 90 min at 125 °C. After stirring for 120 min at 125 °C, the reactor was cooled down, released of pressure and purged with N_2. Volatile components were removed under vacuum yielding polyol 1 as a brown solid.

Polyol 2: Polyol 2 was synthesized according to the protocol for polyol 1 starting from diethylene glycol (217 g, 2.04 mol, 1.00 eq.), PEG-200 (108 g, 0.540 mol, 0.26 eq.), succinic anhydride (710 g, 7.10 mol, 3.45 eq.), ethylene oxide (414 g, 9.39 mol, 4.60 eq) and benzyldimethylamine (1.45 g, 3.33 mmol, 1000 ppm) and obtained as a brown solid.

Polyol 3: A 2 L stainless steel reactor was charged with diethylene glycol (110 g, 1.04 mol, 1.00 eq.) and succinic anhydride (311 g, 3.11 mol, 3.00 eq.). The reactor was closed and repeatedly filled with N_2 (3 × 25 bar N_2 → 1-2 bar N_2). Under N_2 atmosphere, the reactor was heated to 125 °C and stirred at 1200 U/min for 60 min to melt succinic anhydride. The N_2 pressure was increased to 45 bar and ethylene oxide (114 g, 2.59 mol, 2.50 eq.) was continuously added over the course of 45 min at 125 °C. Subsequently, the reaction mixture was stirred for 30 min at 125 °C. Benzyldimethylamine (974 mg, 7.20 mmol, 1500 ppm) was added before a second portion of ethylene oxide (114 g, 2.59 mol, 2.50 eq.) was continuously added over the course of 45 min at 125 °C. After stirring for 120 min at 125 °C, the reactor was cooled down, released of pressure and purged with N_2. Volatile components were removed under vacuum yielding polyol 3 as a brown liquid.

Polyol 4: Polyol 4 was synthesized accordingly starting from diethylene glycol (100 g, 0.942 mol, 1.00 eq.), PEG-200 (50 g, 0.250 mol, 0.26 eq.), succinic anhydride (328 g, 3.28 mol, 3.48 eq.), ethylene oxide (2 x 95.5 g, 4.34 mol, 4.61 eq.) and benzyldimethylamine (1.00 g, 7.40 mmol, 1500 ppm) and obtained as a brown liquid.

Polyol 5: Polyol 5 was synthesized accordingly starting from diethylene glycol (50.0 g, 0.471 mol, 1.00 eq.), CO-derived succinic anhydride (141 g, 1.41 mol, 2.99 eq.), ethylene oxide (2 x 52.0 g, 2.36 mol, 5.01 eq.) and benzyldimethylamine (443 mg, 3.28 mmol, 1500 ppm) and obtained as a brown liquid.

Polyol 6: A 2 L stainless steel reactor was charged with bis(2-hydroxyethyl) terephthalate (199 g, 0.783 mol, 0.70 eq.), glycerol (30.9 g, 0.336 mol, 0.30 eq.), succinic anhydride (218 g, 2.18 mol, 1.95 eq.) and benzyldimethylamine (920 mg, 1500 ppm). The reactor was closed and repeatedly filled with N_2 (3 × 25 bar N_2 → 1–2 bar N_2). Under N_2 atmosphere, the reactor was heated to 125 °C and stirred at 1200 U/min for 60 min to melt succinic anhydride. The N_2 pressure was increased to 45 bar and ethylene oxide (165 g, 3.75 mol, 3.35 eq.) was continuously added over the course of 90 min at 125 °C. After stirring for 120 min at 125 °C, the reactor was cooled down, released of pressure and purged with N_2. Volatile components were removed under vacuum yielding polyol 6 as a brown liquid.

1.2.3.2 Polycondensation of succinic anhydride and glycols

Polyol 7: Succinic anhydride (931 g, 9.3 mol, 1.45 eq.), ethylene glycol (399 g, 6.4 mol, 1.00 eq.) and diethylene glycol (682 g, 6.4 mol, 1.00 eq.) were placed into a 4 L four-necked round-bottom flask equipped with a heating mantle, mechanical stirrer, 50 cm packed column and a descending jacketed coil condenser. The reaction mixture was stirred under N_2 atmosphere and heated up to 220 °C. The formed water was distilled off. After the water formation subsided, 20 ppm $SnCl_2$ (28 wt% in ethylene glycol) were added and the reaction was continued at 220 °C under vacuum (200 mbar) until full conversion was reached. The product was obtained as colorless liquid.

1.2.4 Rigid Foam Formation

1.2.4.1 Rigid foam synthesis

The following formulation was used for all foaming experiments (Table SI 2). The reference polyol Stepanpol PS 2352 was completely replaced with the corresponding polyol of the corresponding experiment.

Table SI 2: Formulation for rigid foam synthesis.

Raw materials	Content in formulation [pbw]
Stepanpol PS 2352	77.6
TCPP	15.5
Tegostab B8421	2.0
Water	1.2
Desmorapid 1792	2.8
Desmorapid 726B	0.9
c/i-pentane	18.4
Desmodur 44V20L	220

All raw materials (see manuscript, Table 3 for details) of the rigid foam formulation except the polyisocyanate were weighed into a cardbox beaker, heated to 23 °C and mixed with a Pendraulik laboratory mixer. Subsequently, the polyisocyanate component (likewise heated to 23 °C) was added. The entire reaction mixture was stirred for 5 s at 4000 rpm and then poured into a wooden mould lined with paper. After 24 h sample specimens were cut out of the foam preform for further analysis.

1.3 Analytical Instruments and Procedures

1.3.1 ^{1}H NMR Spectroscopy

^{1}H NMR measurements were conducted at room temperature on a *Bruker Avance III HD- 600* spectrometer. ^{1}H NMR spectra were referenced to the residual solvent signal.

The c/l ratio was determined by ^{1}H NMR analysis using equation (1).

$$(1) \qquad c/l\ ratio\ = \frac{Area_{cyclic\ carbonate}}{\emptyset Area_{linear\ carbonate}}$$

$Area_{cyclic\ carbonate}$: Integral of area between 4.50 – 4.45 ppm in ^{1}H NMR spectrum.

$\emptyset Area_{linear\ carbonate}$: Average between the integrals of $Area_{linear\ carbonate\ 1,corrected}$ and $Area_{linear\ carbonate\ 2,corrected}$.

$$(2) \qquad Area_{linear\ carbonate\ 1,corrected} = Area_{linear\ carbonate\ 1} - Area_{cyclic\ carbonate}$$

$$(3) \qquad Area_{linear\ carbonate\ 2,corrected} = \frac{Area_{linear\ carbonate\ 2} - 3 \cdot Area_{PO}}{3}$$

$Area_{linear\ carbonate\ 1}$: Integral of area between 4.85 – 4.70 ppm in ^{1}H NMR spectrum.

$Area_{linear\ carbonate\ 2}$: Integral of area between 1.25 – 1.15 ppm in ^{1}H NMR spectrum.

$Area_{PO}$: Integral of area between 2.70 – 2.60 ppm in ^{1}H NMR spectrum.

The CO_2 conversion was determined by ^{1}H NMR analysis using equation (4).

$$(4) \qquad X_{CO_2} = 1 - \frac{Area_{PO}}{\frac{M_{target} - M_{starter}}{M_{target}} \cdot \frac{Area_{Ether}}{3} + \frac{Area_{linear\ carbonate\ 2} + 3 \cdot Area_{cyclic\ carbonate}}{3}}$$

$Area_{Ether}$: Integral of area between 1.13 – 0.97 ppm in ^{1}H NMR spectrum.

The CO_2 incorporation was determined by ^{1}H NMR analysis using equation (5).

$$(5) \qquad CO_2\ incorporation = \frac{M_{CO_2}}{M_{CO_2} + M_{PO}} \cdot \frac{\emptyset Area_{linear\ carbonate}}{\frac{M_{target} - M_{starter}}{M_{target}} \cdot \frac{Area_{Ether}}{3} + \emptyset Area_{linear\ carbonate}}$$

The number of EO-repetition units (mer_{EO}) was determined by ^{1}H NMR analysis using equation (6). It is valid for starter molecules which are based on EO-repetition units.

$$(6) \qquad mer_{EO} = eq_{SA} \cdot \frac{Area_{Ether}}{Area_{Ester}} - \frac{M_{starter} - 18\ g \cdot mol^{-1}}{M_{EO}} - \frac{theor.\ Hydroxyl\ value - Acid\ value}{theor.\ Hydroxyl\ value} + 2$$

eq_{SA}: Equivalents of succinic anhydride to starter.
$Area_{Ether}$: Integral of area between 3.88 – 3.50 ppm in ^{1}H NMR spectrum.
$Area_{Ester}$: Integral of area between 4.36 – 4.18 ppm in ^{1}H NMR spectrum.

1.3.2 Gel Permeation Chromatography (GPC)

GPC measurements were conducted on an *Agilent Technology 1200 Series* device (G1311A Bin Pump, G1313A ALS, G1362A RID), detection by RID, eluent: THF (GPC grade), flow rate: 1.0 mL/min at 40 °C column temperature, columns: 2 x PSS SDV precolumn 100 Å (5 µm), 2 × PSS SDV 1000 Å (5 µm), software: PSS Win GPC UniChrom. Calibration was done using the ReadyCal-Kit Poly(styrene) low by *PSS Polymer Standards Service* in a range of Mp = 266–66000 Da.

1.3.3 ATR-FT-IR Spectroscopy

All spectra were recorded under Ar atmosphere at room temperature on a Bruker Alpha-P FT-IR spectrometer with attenuated total reflection (ATR).

1.3.4 Hydroxyl Value

Determination of the hydroxyl value was performed according to DIN 53240-1.

1.3.5 Acid Value

Determination of the acid value was performed according to DIN EN ISO 2114.

1.3.6 Viscosity

Viscosity measurements were performed on a *MCR 51* rheometer from *Anton Paar* according to DIN 53019-1 using a CP 50-1 measuring cone with a diameter of 50 mm and an angle of 1° at shear rates of 25, 100, 200 and 500 s^{-1}. Viscosities were found to be independent of the shear rate.

1.3.7 Rigid Foam Characterization

The characteristic foaming times were determined during the foaming process. The free-rise density was determined from the volume and weight of a cut-out cube-shaped specimen. The open cell content was determined on a Accupyk 1330 device. The average vertical flame spread was determined following the procedure defined in ISO 11925-2. The thermal conductivity of the foams was determined after 3 weeks of storage on a Fox 200 device according to DIN 52616 11. The compressive strength was measured parallel and perpendicular to the foaming direction on a Zwick Roell Z010 tensile testing system. For microscope analysis the foam was cut into slices of 5 mm thickness. The cut surface was dyed with black ink to enhance the contrast between the first and deeper layers. For the sample preparation a clean cut as well as a homogenous coloring is essential for a representative analysis. Images of the prepared foam slice were acquired with a digital microscope (Keyence VHX 5000) at a magnification level of 100x. Received images were analyzed with the image processing software ImageJ Fiji. As an output the equivalent circle diameter ECD (equation 7) was computed, which is a commonly used parameter in cell size analysis as it is insensitive to cell shape and orientation.

$$(7) \qquad \mathrm{ECD} = 2000 \cdot \sqrt{\frac{\text{Cell Area}}{\pi}}\ [\mu\mathrm{m}]$$

1.4 NMR Spectra of New Polyols

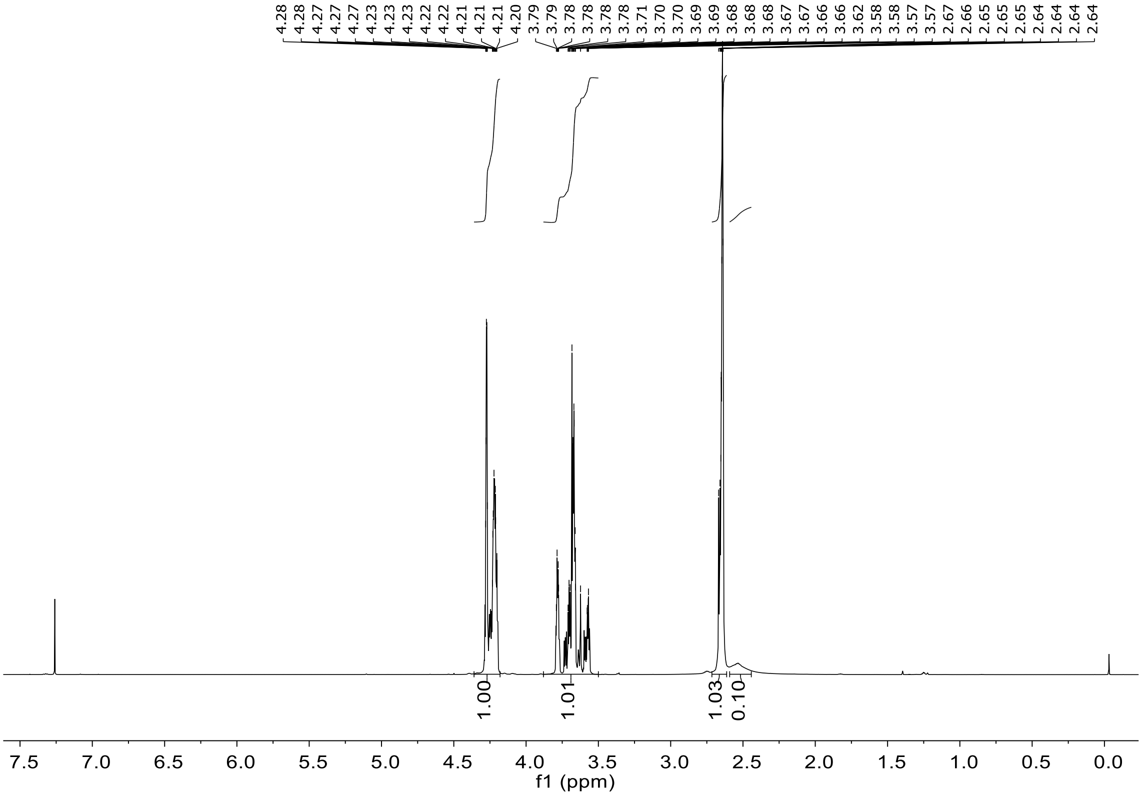

Figure SI 3: ^{1}H NMR of Polyol 1 in $CDCl_3$.

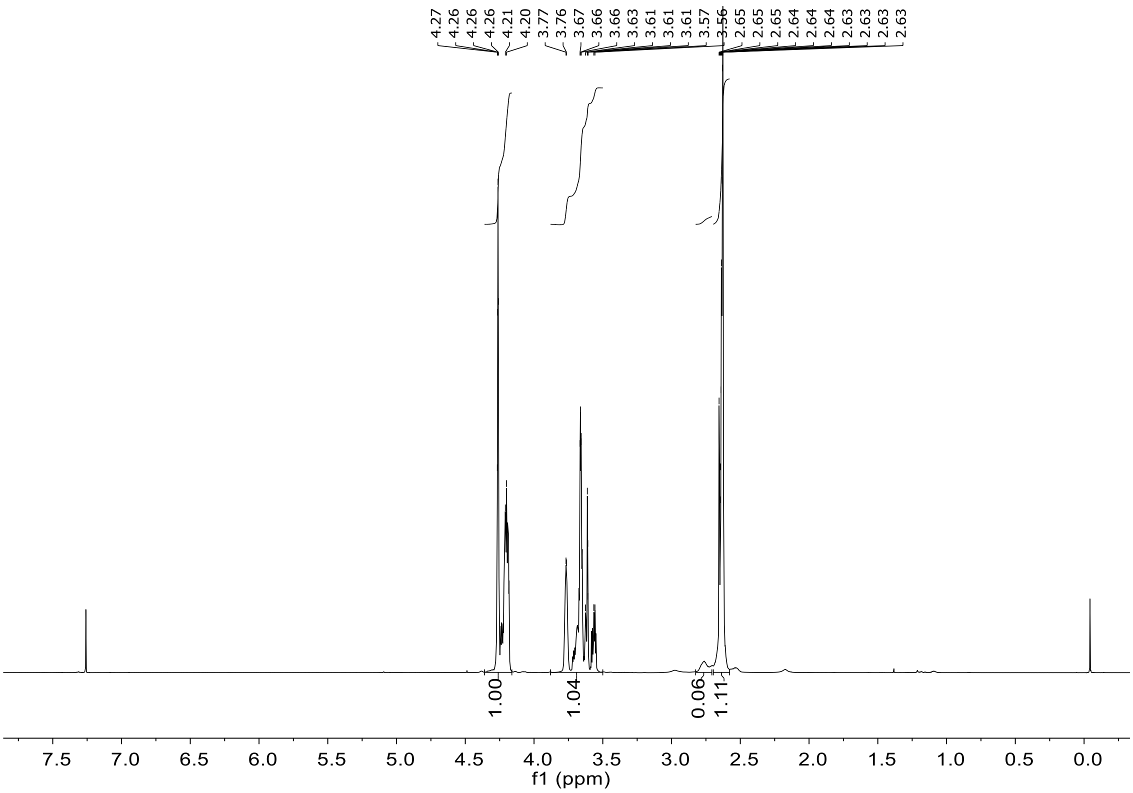

Figure SI 4: ^{1}H NMR of Polyol 2 in $CDCl_3$.

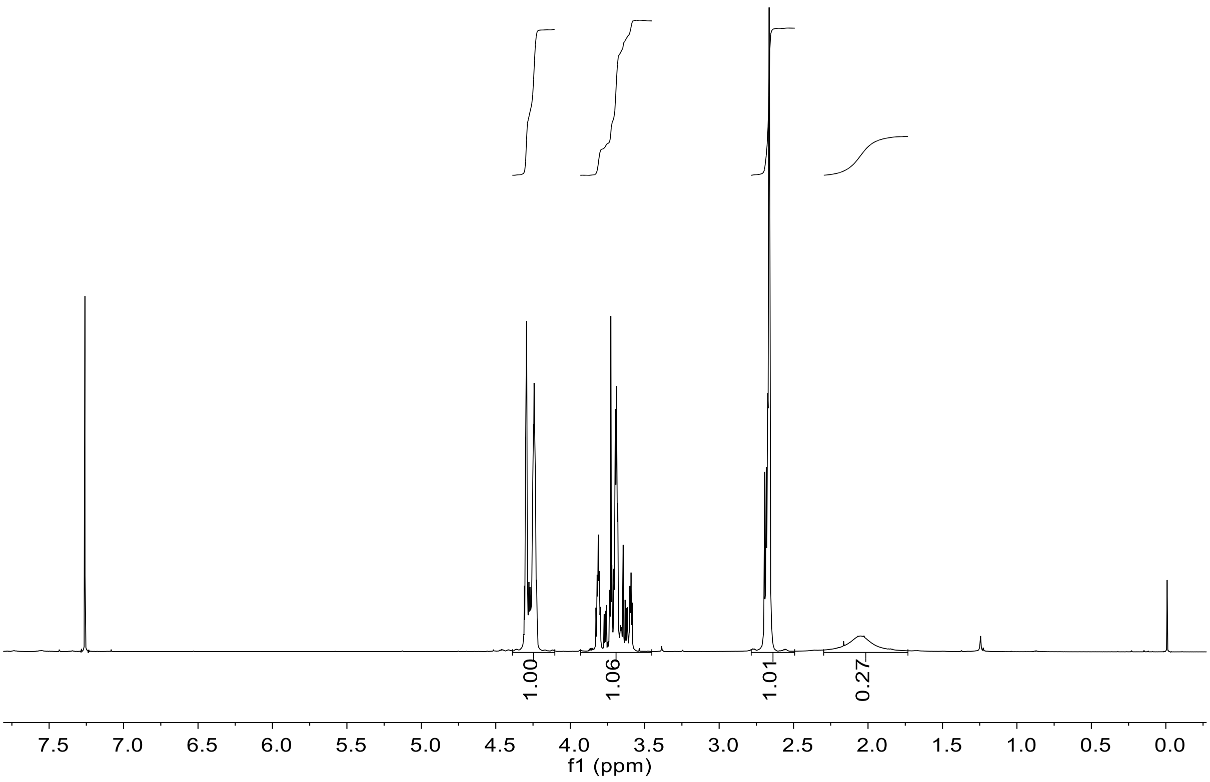

Figure SI 5: ^{1}H NMR of Polyol 3 in $CDCl_3$.

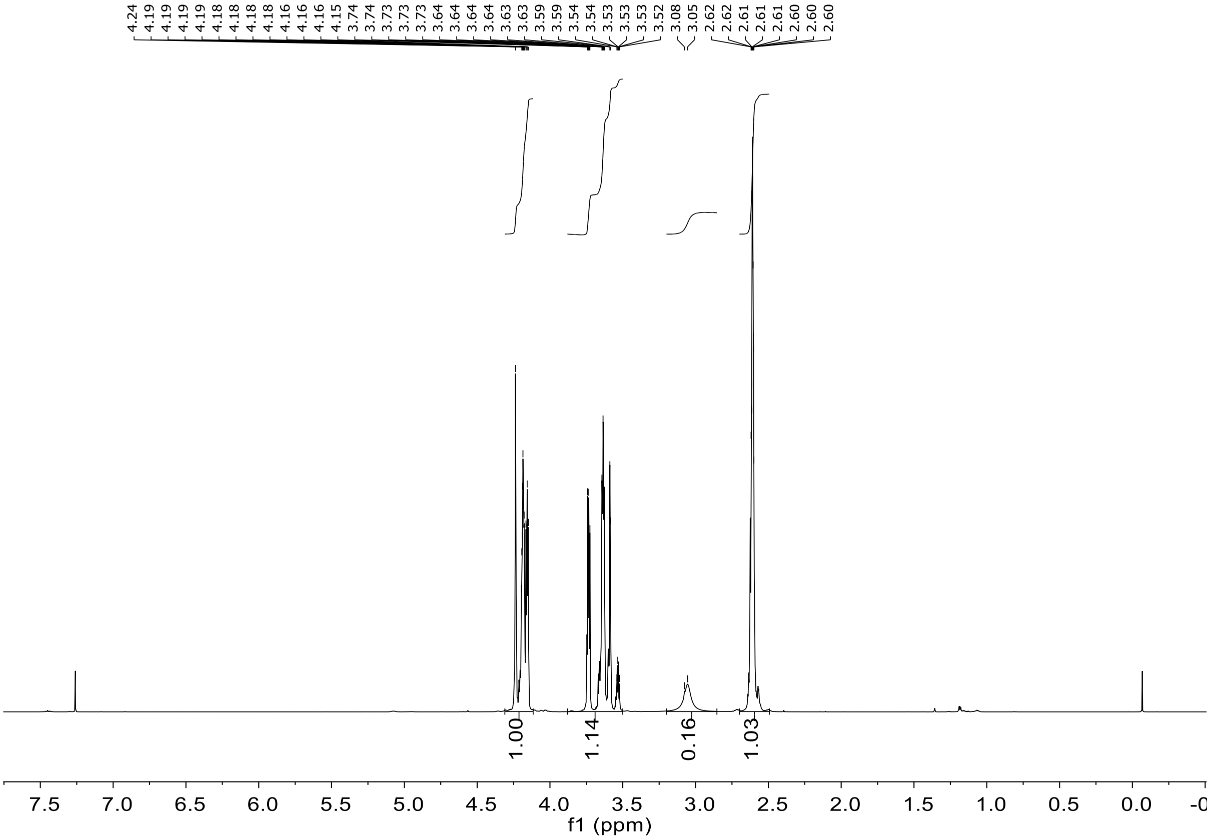

Figure SI 6: ^{1}H NMR of Polyol 4 in $CDCl_3$.

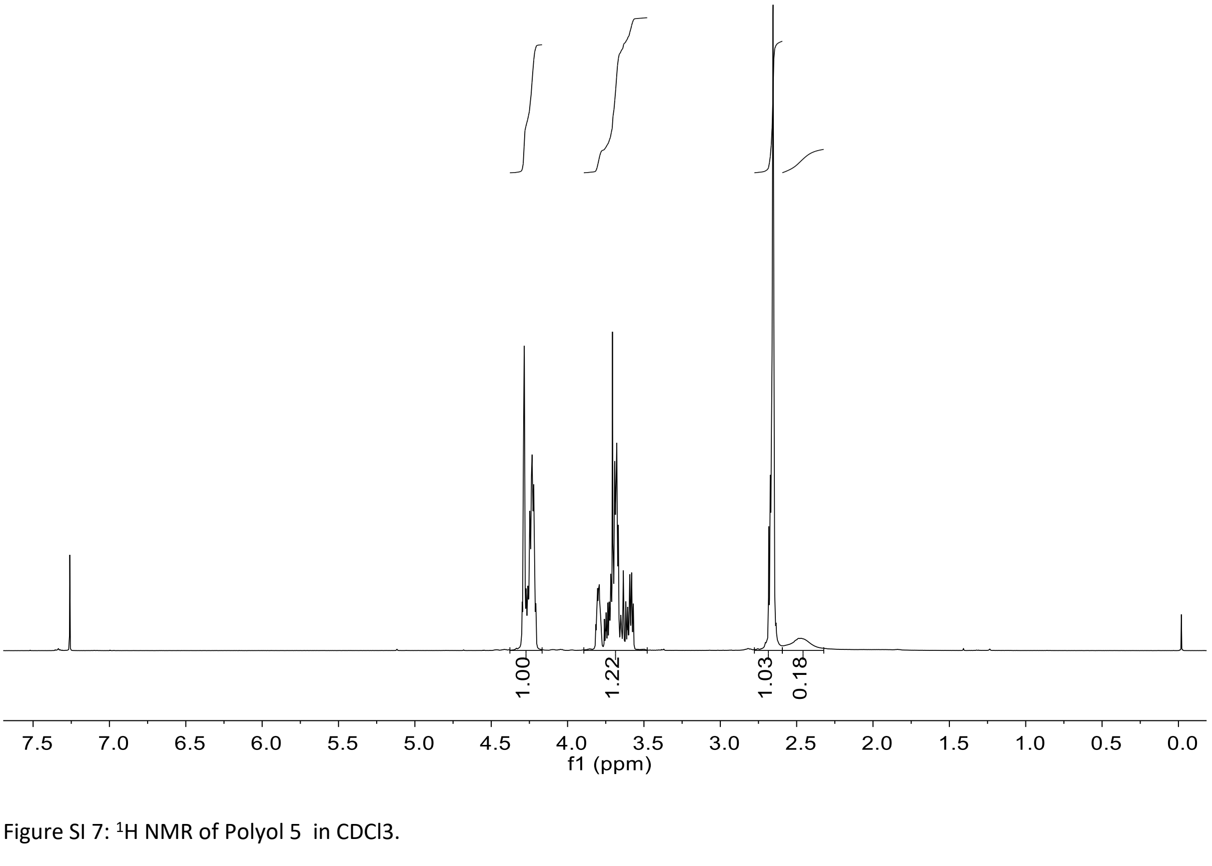

Figure SI 7: ^{1}H NMR of Polyol 5 in CDCl3.

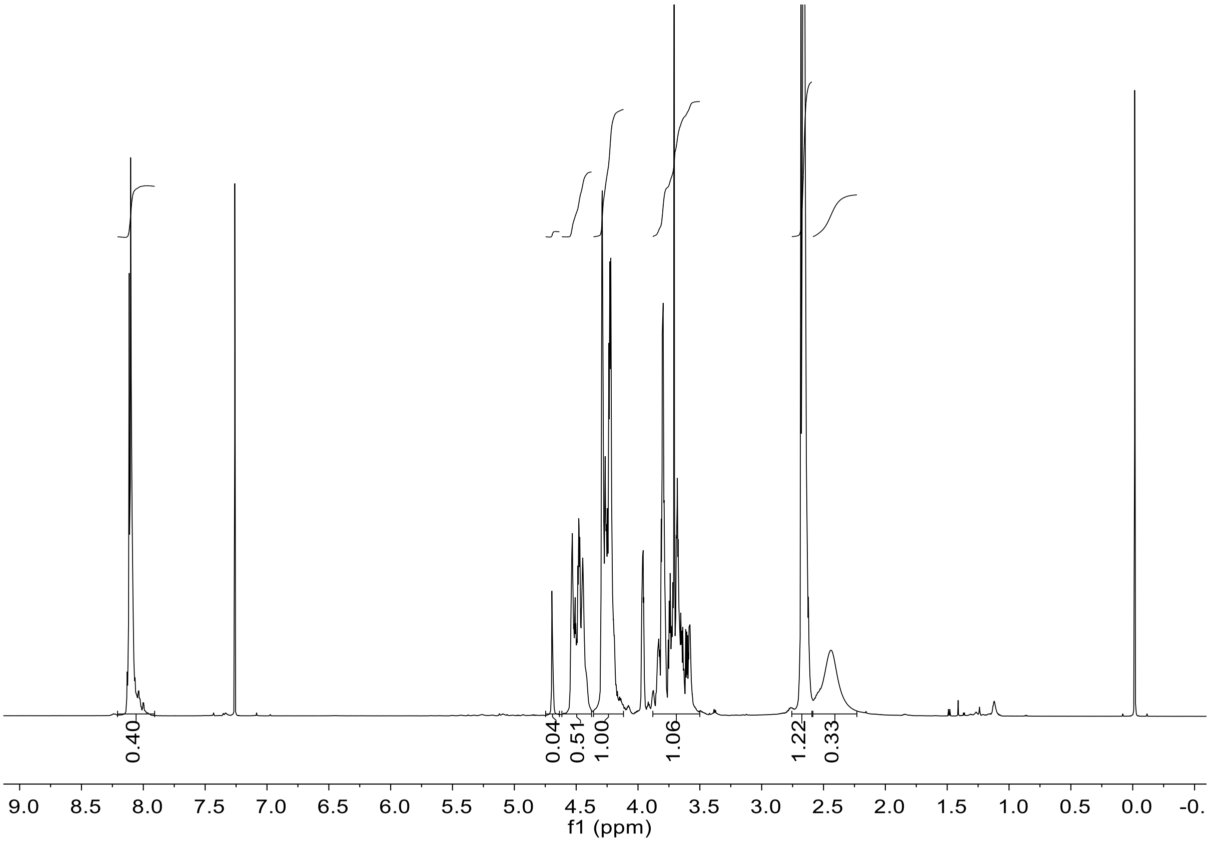

Figure SI 8: ^{1}H NMR of Polyol 6 in CDCl3.

2 References

1. W. F. Edgell and J. Lyford, *Organic Chemistry*, 1970, **9**, 1932-1933.
2. J. Jiang, S. Rajendiran and S. Yoon, *Asian Journal of Organic Chemistry*, 2019, **8**, 151-154.