Supporting Information to

Chiral Diboranes as Catalysts for the Stereoselective Organopolymerization of Epoxides

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Experimental
Materials and Synthesis

Propylene oxide (PO, TCI Chemicals, > 99.0 %), butylene oxide (BO, TCI Chemicals, > 99.0 %), styrene oxide (SO, TCI Chemicals, >98%) and allyl glycidyl ether (AGE, Sigma-Aldrich, > 99.0 %) were stirred over CaH₂ overnight. After distillation under nitrogen, the monomers PO, BO, SO and AGE were degassed twice by the freeze-pump-thaw method and then stored inside the glove box (LabMaster, MBraun, Germany, freezer at -36 °C). Toluene, THF and DCM used in polymerizations were taken from a solvent purification system (MBraun, Germany) and kept over molecular sieves (3 Å) inside the glove box. Allyl bromide (Sigma-Aldrich, > 99.0 %), (R)-(±)-1,1´-bi(2-naphtol) (Sigma-Aldrich, > 99.0 %), 1,4-benzenedimethanol (TCI Chemicals, > 99.0 %) and 1,1´-bi-2-naphtol (TCI Chemicals, > 99.0 %) were used as received. 9-Borabicyclo[3.3.1]nonane (Sigma Aldrich, 0.5 M solution in THF), 1,8-diazobicyclo[5.4.0]undec-7-ene (DBU, Sigma-Aldrich) and the phosphazene base (P₂-tBu, Sigma-Aldrich, 2.0 M solution in THF) were used as received and stored in the glove box. The NHO was synthesized as described earlier and stored inside the glove box at -36 °C.[1,2] The commercial macroinitiator samples PCL10K (polycaprolactone with $M_n = 10 000$ g/mol, Sigma Aldrich), PEG8K (polyethylene glycol with $M_n = 8 000$ g/mol, abcr) and PLA20K (polylactide with $M_n = 20 000$ g/mol, Sigma Aldrich) were used as received. PCL7K[1] and PCL2K[3] were prepared according to published procedures. PCL11K-diol was prepared in the same manner as PCL2K but BDM (1,4-benzol-dimethanol) was used as an initiator. For the diborane (1-4) synthesis, see below.
Procedural Information

Synthesis of diallyl species

General Procedure: In an oven-dried Schlenk flask, (R)-BINOL ((R)-(+)1,1′-bi-2-naphthol) and allyl bromide were dissolved in acetonitrile. Then, $K_2CO_3$ was added and the reaction mixture was stirred at room temperature for 72 h. The reaction was monitored by GC-MS. After filtration and salt removal, the solvent and excess amount of allyl bromide were removed in vacuo to yield the colorless product.

Diallyl 1

First, (R)-BINOL ((R)-(+)1,1′-bi-2-naphthol) (5.00 g, 17.5 mmol) and allyl bromide (3.63 mL, 42.0 mmol, 2.4 equiv.) were dissolved in 30 mL acetonitrile, then $K_2CO_3$ (16.08 g, 0.116 mol) was added and the reaction mixture was stirred at room temperature for 72 h. The reaction was monitored by GC-MS. Via filtration, the potassium salts were removed. The solvent and excess amount of allyl bromide were removed under reduced pressure. Yield: 2.91 g (7.94 mmol, 45 %). $^1$H NMR (400 MHz, CDCl$_3$) $\delta = 7.85-7.95$ (dd, 4H), 7.14-7.41 (m, 8H), 5.70-5.80 (m, 2H), 4.97-5.04 (m, 4H), 4.51-4.53 (m, 4H) ppm.
**Diallyl (S)-1’**

First, (S)-BINOL ((S)-(−)-1,1’-bi-2-naphtol) (5.00 g, 17.5 mmol) and allyl bromide (3.63 mL, 42.0 mmol, 2.4 equiv.) were dissolved in 30 mL acetonitrile, then K₂CO₃ (16.08 g, 0.116 mol) was added and the reaction mixture was stirred at room temperature for 72 h. The reaction was monitored by GC-MS. Via filtration, the potassium salts were removed. The solvent and excess amount of allyl bromide were removed under reduced pressure. Yield: 2.81 g (7.67 mmol, 43 %). ¹H NMR (400 MHz, CDCl₃) δ = 7.84–7.95 (dd, 4H), 7.13–7.41 (m, 8H), 5.68–5.81 (m, 2H), 4.96–5.04 (m, 4H), 4.50–4.53 (m, 4H) ppm.

**Diallyl rac-1’**

First, BINOL 1,1’-bi-2-naphtol (5.00 g, 17.5 mmol) and allyl bromide (3.63 mL, 42.0 mmol, 2.4 equiv.) were dissolved in 30 mL acetonitrile, then K₂CO₃ (16.08 g, 0.116 mol) was added and the reaction mixture was stirred at room temperature for 72 h. The reaction was monitored by GC-MS. Via filtration, the potassium salts were removed. The solvent and excess amount of allyl bromide were removed under reduced pressure. Yield: 2.78 g (7.58 mmol, 43 %). ¹H NMR (400 MHz, CDCl₃) δ = 7.69–7.79 (dd, 4H), 7.25 (d, 2H), 7.13–7.18 (dt, 2H), 7.01–7.06 (dt, 2H), 6.94 (d, 2H), 5.54–5.63 (m, 2H), 4.81–4.87 (m, 4H), 4.35–4.37 (m, 4H) ppm.

**Diallyl 2’**

First, (R)-3,3’-diphenyl-[1,1’-binaphthalene]-2,2’-diol (1.00 g, 2.28 mmol) and allyl bromide (0.51 mL, 5.50 mmol, 2.4 equiv.) were dissolved in 4 mL acetonitrile, then K₂CO₃ (2.09 g, 0.015 mol) was added and the reaction mixture was stirred at room temperature for 72 h. The reaction was monitored by GC-MS. Via filtration the potassium salts were removed. The solvent and excess amount of allyl bromide were removed in vacuo. Yield: 0.500 g (0.964 mmol, 42 %). ¹H NMR (400 MHz, CDCl₃) δ = 7.95 (s, 2H), 7.91 (d, 2H), 7.75 (dd, 4H), 7.37–7.47 (m, 8H), 7.25 (dd, 4H), 5.23–5.33 (m, 2H), 4.53–4.67 (m, 4H), 3.93–3.98 (m, 2H), 3.69–3.74 (m, 2H) ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 128.5, 128.4, 127.1, 32.2, 31.8 22.1 ppm.
**Diallyl 3’**

First, 2,2’-biphenol (5.00 g, 26.85 mmol) and allyl bromide (6.10 mL, 64.44 mmol, 2.4 equiv.) were dissolved in 40 mL acetonitrile, then K₂CO₃ (24.00 g, 0.173 mol) was added and the reaction mixture was stirred at room temperature for 72 h. The reaction was monitored by GC-MS. Via filtration the potassium salts were removed. The solvent and excess amount of allyl bromide were removed in vacuo. Yield: 4.01 g (15.10 mmol, 56%). ¹H NMR (400 MHz, CDCl₃) δ = 7.26 (dd, 4H), 6.98 (dt, 2H), 6.91 (d, 2H), 5.84-5.93 (m, 2H), 5.08-5.21 (m, 4H), 4.67 (q, 4H) ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 156.1, 133.6, 133.6, 131.5, 128.4, 128.4, 120.5, 116.4, 112.3, 68.9 ppm.

**Diallyl 4’**

1,4-Butanediol (1.00 g, 11.09 mmol) and allyl bromide (2.30 mL, 26.61 mmol, 2.4 equiv.) were dissolved in 25 mL acetonitrile, then K₂CO₃ (8.10 g, 0.05 mol) was added and the reaction mixture was stirred at room temperature for 72 h. The reaction was monitored by GC-MS. Via filtration, the potassium salts were removed. Solvent and excess amount of allyl bromide were removed under reduced pressure. Yield: 0.400 g (2.35 mmol, 21%). ¹H NMR (400 MHz, CDCl₃) δ = 5.86-5.95 (m, 2H), 5.23-5.29 (m, 2H), 5.14-5.18 (m, 2H), 3.96 (dt, 4H), 3.43-3.46 (m, 4H), 1.64-1.67 (m, 4H) ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 135.0, 134.9, 116.7, 71.8, 70.1, 26.5 ppm.

**Synthesis of Diborane Catalysts**

**Diboranes**

General Procedure: To an oven-dried Schlenk flask the corresponding diallyl-species was added under nitrogen flow. The suitable amount of 9-borabicyclo[3.3.1]nonane in THF (0.5 M) was added via cannula/septum. The reaction mixture was stirred at 60 °C overnight (condenser). After cooling to room temperature, the mixture was concentrated in vacuo to afford the crude product which was transferred into the glove box. After recrystallization from pentane (-36°C), the diboranes were obtained as white solids.
Diborane 1

The general procedure was employed, using diallyl-species 1′ (0.400 g, 1.09 mmol, 1 equiv.) and 9-borabicyclo[3.3.1]nonane in THF (0.5 M) (4.4 mL, 3.16 mmol, 2.9 equiv.). The reaction mixture was stirred at 60 °C overnight. After purification, enantiopure diborane 1 was obtained as a white solid. Crystals suitable for structure analysis were received from recrystallization in pentane at -36°C in the glove box. Yield: 0.612 g (1.00 mmol, 92 %). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta = 7.82-7.92 (dd, 4H), 7.41-7.43 (d, 2H), 7.18-7.31 (m, 6H), 3.87-4.0 (m, 4H)\) ppm. \(^1\)C NMR (101 MHz, CDCl\(_3\)) \(\delta = 154.8, 134.4, 129.4, 129.2, 127.9, 126.2, 123.5, 120.9, 116.3, 116.3, 72.3, 33.1, 30.9, 24.6, 23.2\) ppm. \(^1\)B NMR (100 MHz, CDCl\(_3\)) \(\delta = 90.64\) ppm. HRMS (ESI): \(m/z\) calc. for C\(_{42}\)H\(_{52}\)B\(_2\)O\(_2\) = 610.42, found C\(_{42}\)H\(_{52}\)B\(_2\)O\(_2\)Na\(^+\): 633.36.

Figure S2. \(^1\)H NMR analysis (CDCl\(_3\), 400 MHz) of compound 1.
Diborane (S)-1

The general procedure was employed, using diallyl-species (S)-1' (0.600 g, 1.63 mmol, 1 equiv.) and 9-borabicyclo[3.3.1]nonane in THF (0.5 M) (7.5 mL, 5.38 mmol, 2.9 equiv.). The reaction mixture was stirred at 60 °C overnight. After purification, diborane rac-1 was obtained as a white solid. Yield: 0.890 g (1.45 mmol, 89 %). ^1H NMR (400 MHz, CDCl₃) δ = 7.90-8.00 (dd, 4H), 7.48-7.50 (d, 2H), 7.26-7.39 (m, 2H), 7.26-7.27 (m, 4H), 3.94-4.08 (m, 4H) ppm. ^13C NMR (101 MHz, CDCl₃) δ = 154.7, 154.3, 134.3, 129.1, 127.8, 126.2, 125.5, 123.4, 120.8, 115.9, 72.1, 33.0, 26.7, 25.7, 24.5, 23.1 ppm.

Figure S3. ^13C NMR analysis (CDCl₃, 101 MHz) of compound 1.
Figure S4. $^1$H NMR analysis (CDCl$_3$, 400 MHz) of compound (S)-1.

Diborane rac-1

The general procedure was employed, using diallyl-species rac-1' (0.400 g, 1.09 mmol, 1 equiv.) and 9-borabicyclo[3.3.1]nonane in THF (0.5 M) (4.4 mL, 3.16 mmol, 2.9 equiv.). The reaction mixture was stirred at 60 °C overnight. After purification, diborane rac-1 was obtained as a white solid. Yield: 0.572 g (0.93 mmol, 86 %). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.82-7.93 (dd, 4H), 7.41-7.43 (d, 2H), 7.18-7.30 (m, 6H), 3.87-4.01 (m, 4H) ppm. $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ = 154.7, 134.3, 129.3, 129.0, 127.8, 126.0, 125.6, 123.4, 120.8, 116.1, 72.1, 33.0, 30.8, 24.5, 23.1 ppm. HRMS (ESI): m/z calc. for C$_{42}$H$_{52}$B$_2$O$_2$ = 610.42, found C$_{42}$H$_{52}$B$_2$O$_2$Na$: 633.35.
Figure S5. $^1$H NMR analysis (CDCl$_3$, 400 MHz) of compound rac-1.

Figure S6. $^{13}$C NMR analysis (CDCl$_3$, 101 MHz) of compound rac-1.
Diborane 2

The general procedure was employed, using diallyl-species 2’ (0.400 g, 0.771 mmol, 1 equiv.) and 9-borabicyclo[3.3.1]nonane in THF (0.5 M) (3 mL, 2.18 mmol, 2.9 equiv.). The reaction mixture was stirred at 60 °C overnight. After purification, enantiopure diborane 2 was obtained as a white solid. Yield: 0.490 g (6.42 mmol, 90%). 1H NMR (400 MHz, CDCl3) δ = 7.96 (s, 2H), 7.91 (d, 2H), 7.79 (dd, 4H), 7.38-7.48 (m, 8H), 7.27 (dd, 4H), 3.48 (q, 2H), 3.17 (q, 2H) ppm. 13C NMR (101 MHz, CDCl3) δ = 152.8, 138.2, 134.6, 132.8, 129.6, 129.0, 128.4, 127.0, 126.9, 126.0, 125.3, 124.9, 123.6, 31.8, 29.5, 24.6, 23.7, 22.0 ppm. 11B NMR (100 MHz, CDCl3) δ = 58.53 ppm. HRMS (ESI): m/z calc. for C₅₂H₆₀B₂O₂ = 762.48, found C₅₂H₆₀B₂O₂Na⁺: 785.46.

Figure S7. 1H NMR analysis (CDCl3, 400 MHz) of compound 2.
Figure S8. $^{13}$C NMR analysis (CDCl$_3$, 101 MHz) of compound 2.

Diborane 3

The general procedure was employed, using dialyl-species 3’ (1.00 g, 3.75 mmol, 1 equiv.) and 9-borabicyclo[3.3.1]nonane in THF (0.5 M) (15 mL, 10.88 mmol, 2.9 equiv.). The reaction mixture was stirred at 60 °C overnight. After purification, diborane 3 was obtained as a white solid. Yield: 1.53 g (2.99 mmol, 80 %). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.25-7.29 (m, 4H), 6.94-6.98 (m, 4H), 3.88 (t, 4H) ppm. $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ = 156.8, 131.8, 128.5, 128.4, 120.1, 112.5, 33.2, 24.5, 23.3 ppm. $^{11}$B NMR (100 MHz, CDCl$_3$) $\delta$ = 58.59 ppm. HRMS (ESI): m/z calc. for C$_{32}$H$_{48}$B$_2$O$_2$ = 510.38, found C$_{32}$H$_{49}$B$_2$O$_2$+: 511.39.
Figure S9. $^1$H NMR analysis (CDCl$_3$, 400 MHz) of compound 3.

Figure S10. $^{13}$C NMR analysis (CDCl$_3$, 101 MHz) of compound 3.
Diborane 4

The general procedure was employed, using diallyl-species 4' (0.180 g, 1.05 mmol, 1 equiv.) and 9-borabicyclo[3.3.1]nonane in THF (0.5 M) (4.3 mL, 3.06 mmol, 2.9 equiv.). The reaction mixture was stirred at 60 °C overnight. After aliphatic diborane 4 was obtained as a white solid. Yield: 0.387 g (0.933 mmol, 89 %). ¹H NMR (400 MHz, CDCl₃) δ = 3.48-3.51 (m, 4H), 3.42 (t, 4H), 1.76-1.87 (m, 18H), 1.62-1.72 (m, 14H), 1.22-1.30 (m, 8H) ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 73.2, 71.0, 33.6, 33.4, 26.3, 25.1, 23.5, 23.3 ppm. HRMS (ESI): m/z calc. for C₂₆H₄₈B₂O₂ = 414.38, found C₂₆H₄₉B₂O₂⁺: 415.26.

![Diagram of compound 4]

Figure S11. ¹H NMR analysis (CDCl₃, 400 MHz) of compound 4.
General Polymerization Procedures

All polymerizations were assembled inside the glove box. First the diborane (0.017 mmol, 2 equiv.) was dissolved in PO (0.987 g, 17.04 mmol, 2000 equiv.). Subsequently, the initiator (BnOH, 0.0425 mmol, 5 equiv.) and the corresponding organobase (0.0085 mmol, 1 equiv.) was added to result in a total molar ratio of organobase/diborane/initiator/PO = 1:2:5:2000. The reaction was quenched by evaporation of the monomer. The molecular weight of the polyether was determined via GPC analysis (CHCl₃) and ¹H NMR; the number of average PO-repeating units (x) was likewise calculated from ¹H-NMR (CDCl₃) using the PO(-CH₂)- (δ = 3.38-3.42 ppm) and Ph-CH₂-OR signal (δ = 4.55 ppm).

General Procedure for Kinetic Analyses

The polymerizations were assembled inside the glove box. First the diborane (0.017 mmol, 2 equiv.) was dissolved in (R)- or (S)-PO (0.493 g, 8.52 mmol, 1000 equiv.) and THF (4.28 mL). Subsequently, the initiator (BnOH, 0.0425 mmol, 5 equiv.) and the corresponding organobase (0.0085 mmol, 1 equiv.) was added to result in a total molar ratio of organobase/diborane/initiator/PO = 1:2:5:1000. After the polymerization was started, aliquots of
20 µL were collected via pipette (Eppendorf) in specific time intervals and quenched with wet CDCl₃. The molecular weight of the polyether was determined via GPC analysis (CHCl₃) and ¹H NMR; the number of average PO-repeating units (x) was likewise calculated from ¹H-NMR (CDCl₃) using the PO(-CH₂)- (δ = 3.38-3.42 ppm) and Ph-CH₂-OR signal (δ = 4.55 ppm).

**General Procedure for Block Copolymer Preparation**

Polymerizations were assembled inside the glovebox. First the diborane 1 (0.017 mmol, 2 equiv.) was dissolved in PO (0.493 g, 8.52 mmol, 1000 equiv.) and THF (4.28 mL). Subsequently, the macroinitiator (PCL₂⁻11K/PEO8K/PLA₂₀K, 0.021 mmol, 2.5 equiv.) and the NHO (0.0085 mmol, 1 equiv.) was added to result in a total molar ratio of NHO/initiator/PO = 1:2:5:1000. The reaction was quenched by evaporation of the monomer. The molecular weight and copolymer formation of the polyether was investigated via GPC analysis (CHCl₃), ¹H NMR and ¹H DOSY NMR analysis; the number of average PO repeat units (x) was calculated from ¹H-NMR (CDCl₃) using the PO(-CH₂)- (δ = 3.38-3.42 ppm) signal and a characteristic signal of the macroinitiator repeat units (PCL: δ = 4.17 ppm, PEO 8K: δ = 3.64 ppm, PLA: δ = 5.16 ppm).
Characterization and Analysis

*NMR Spectroscopy*

The $^1$H, $^{13}$C and $^{11}$B NMR spectra were recorded at room temperature on a *Bruker Avance III* 400 spectrometer ($^1$H at 400 MHz and $^{13}$C at 101 MHz). The chemical shifts are being reported relative to reference peaks of the applied deuterated solvent (CDCl$_3$: $\delta = 7.26$ ppm for $^1$H and $\delta = 77.16$ ppm for $^{13}$C).

*Gel Permeation Chromatography*

Gel permeation chromatography (GPC) was used to determine relative molar masses and molar mass distribution ($D_M$) of the polymer samples. GPC was measured at 30 °C in chloroform on an *Agilent Technologies* 1200 Infinity Series instrument. The instrument was calibrated against polystyrene standards (800 g/mol – $2\cdot10^6$ g/mol). The setup consists of three consecutive SDS PSS (8 mm x 300 mm) columns and a RI detector *Agilent 1200 Series* G1362. A flow rate of 1.0 mL min$^{-1}$ and a sample concentration of 2 mg/ml was applied. The injection volume was 100 μL.

*Differential Scanning Calorimetry*

A DSC 4000 instrument (*Perkin Elmer*) was employed. For the measurements, approximately 2 mg of the sample was placed in a 50 μL sample container and heated from 0°C to 120°C at scan rates of 5-20 K/min with a nitrogen flow of 20 mL/min. All thermograms were analyzed using the second heating/cooling cycle.

*MALDI-ToF*

MALDI-ToF (matrix-assisted laser desorption ionization-time of flight) mass spectrometry measurements were conducted on a *Bruker Autoflex III* (337 nm, reflector mode). The samples were prepared by mixing a matrix solution (2,5-dihydroxybenzoic acid, 5 mg/mL in THF), PPO solution (7 mg/mL in THF) and sodium trifluoromethanesulfonate solution as cationization agent (0.1 M in 90% acetone/water = 9:1) with ratio of 2:1:2. For calibration, a polystyrene standard was employed.

*Calculation of Polymer Tacticity via NMR Spectroscopy*

$^{13}$C NMR analysis can be used to conveniently determine tacticity of various aliphatic polyethers. For PPO and PBO, the well separated $m$- ($\delta = 73.5$ ppm/72.4 ppm) and r-diad ($\delta = 73.0$ ppm/71.5 ppm) signals can be directly used for evaluation. For AGE, the deconvolution of the signal at $\delta = 78.9$ ppm and analysis of the triad structure can be used to calculate the $m$-value, accordingly. Details for all three polyethers can be found in the cited literature.$^{[4]}$
Polymerizations

Polymerization Kinetics

Figure S13. Correlation of molar mass (determined by GPC/CHCl₃) vs. conversion for PO polymerization (molar ratio of NHO/1/BnOH/PO = 1:2:5:500, [M]₀ = 2.0 mol/L, THF, T = 25 °C).

Figure S14. Conversion vs. time using 1 and (R)- and (S)-PO, respectively, under identical conditions (molar ratio of NHO/1/BnOH/PO = 1:2:5:2000, bulk, T = 25 °C).

Figure S15. Conversion vs. time using rac-1 and (R)- and (S)-PO, respectively, under identical conditions (molar ratio of NHO/rac-1/BnOH/PO = 1:2:5:2000, [M]₀ = 2.0 mol/L, THF, T = 25 °C).

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Figure S16. Conversion vs. time using (S)-1 and (R)- and (S)-PO, respectively, under identical conditions (molar ratio of NHO/(S)-1/BnOH/PO = 1:2:5:2000, bulk, T = 25 °C).

Figure S17. Conversion vs. time using diboranes 1 ((R)-configuration)) or (S)-1 and (S)-BO, respectively, under identical conditions (molar ratio of NHO/(S)-1/BnOH/(S)-BO = 1:2:2.5:1000, [M]₀ = 2.0 mol/L, THF, T = 25 °C).
### Table S5. Data set of PO polymerizations using NHO, P$_2$-tBu and DBU in combination with various diboranes and BnOH (I) as initiator.

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<th>Cat (C)</th>
<th>Organo- base (B)</th>
<th>B/C/I/PO</th>
<th>solvent</th>
<th>T [°C]</th>
<th>t [h]</th>
<th>$x^2$ ($D_m)^b$</th>
<th>$M_n\text{calc},^c$ [g/mol]</th>
<th>$M_n\text{GPC},^b$ [g/mol]</th>
<th>$m^d$ [%]</th>
<th>Conv.$^e$ [%]</th>
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<td>-36</td>
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<td>301 (1.16)</td>
<td>24000</td>
<td>24000</td>
<td>68</td>
<td>75</td>
</tr>
<tr>
<td>9</td>
<td>1</td>
<td>DBU</td>
<td>1:2:5:2000</td>
<td>bulk</td>
<td>25</td>
<td>24</td>
<td>106 (1.18)</td>
<td>6000</td>
<td>8000</td>
<td>62</td>
<td>27</td>
</tr>
<tr>
<td>10</td>
<td>1</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>toluene [2M]</td>
<td>25</td>
<td>3.0</td>
<td>247 (1.13)</td>
<td>21000</td>
<td>24000</td>
<td>75</td>
<td>62</td>
</tr>
<tr>
<td>11</td>
<td>1</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>toluene [2M]</td>
<td>-36</td>
<td>24</td>
<td>321 (1.14)</td>
<td>25000</td>
<td>26000</td>
<td>82</td>
<td>80</td>
</tr>
<tr>
<td>12</td>
<td>1</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>toluene [2M]</td>
<td>-36</td>
<td>24</td>
<td>2130 (1.18)</td>
<td>123000</td>
<td>117000</td>
<td>79</td>
<td>63</td>
</tr>
<tr>
<td>13</td>
<td>1</td>
<td>NHO</td>
<td>1:1:1:000</td>
<td>bulk</td>
<td>25</td>
<td>32</td>
<td>48 (1.10)</td>
<td>3000</td>
<td>5000</td>
<td>77</td>
<td>5</td>
</tr>
<tr>
<td>14</td>
<td>1</td>
<td>NHO</td>
<td>1:1:1:000</td>
<td>bulk</td>
<td>25</td>
<td>4.4</td>
<td>266 (1.11)</td>
<td>16000</td>
<td>19000</td>
<td>72</td>
<td>27</td>
</tr>
<tr>
<td>15</td>
<td>rac-1</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>bulk</td>
<td>25</td>
<td>20</td>
<td>256 (1.32)</td>
<td>19000</td>
<td>21000</td>
<td>59</td>
<td>64</td>
</tr>
<tr>
<td>16</td>
<td>rac-1</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>THF [2M]</td>
<td>-36</td>
<td>4.8</td>
<td>138 (1.20)</td>
<td>28000</td>
<td>23000</td>
<td>75</td>
<td>35</td>
</tr>
<tr>
<td>17</td>
<td>rac-1</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>bulk</td>
<td>-36</td>
<td>4.8</td>
<td>279 (1.22)</td>
<td>20000</td>
<td>21000</td>
<td>63</td>
<td>70</td>
</tr>
<tr>
<td>18</td>
<td>rac-1</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>DCM [2M]</td>
<td>-36</td>
<td>72</td>
<td>44 (1.12)</td>
<td>3000</td>
<td>3000</td>
<td>70</td>
<td>11</td>
</tr>
<tr>
<td>19</td>
<td>rac-1</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>pentane [2M]</td>
<td>-36</td>
<td>72</td>
<td>315 (1.10)</td>
<td>19000</td>
<td>23000</td>
<td>63</td>
<td>79</td>
</tr>
<tr>
<td>20</td>
<td>rac-1</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>toluene [2M]</td>
<td>-36</td>
<td>72</td>
<td>235 (1.13)</td>
<td>14000</td>
<td>18000</td>
<td>80</td>
<td>59</td>
</tr>
<tr>
<td>21</td>
<td>2</td>
<td>NHO</td>
<td>1:4:5:1000</td>
<td>bulk</td>
<td>-36</td>
<td>24</td>
<td>88 (1.17)</td>
<td>5000</td>
<td>6000</td>
<td>74</td>
<td>22</td>
</tr>
<tr>
<td>22</td>
<td>2</td>
<td>NHO</td>
<td>1:4:5:10000</td>
<td>bulk</td>
<td>-36</td>
<td>24</td>
<td>134 (1.11)</td>
<td>8000</td>
<td>10000</td>
<td>80</td>
<td>7</td>
</tr>
<tr>
<td>23</td>
<td>2</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>THF [4M]</td>
<td>25</td>
<td>24</td>
<td>280 (1.25)</td>
<td>16000</td>
<td>13000</td>
<td>78</td>
<td>70</td>
</tr>
<tr>
<td>24</td>
<td>2</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>THF [2M]</td>
<td>25</td>
<td>8.0</td>
<td>65 (1.14)</td>
<td>4000</td>
<td>5000</td>
<td>88</td>
<td>16</td>
</tr>
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<td>25</td>
<td>2</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>THF [2M]</td>
<td>50</td>
<td>2.4</td>
<td>67 (1.15)</td>
<td>4000</td>
<td>4000</td>
<td>73</td>
<td>17</td>
</tr>
<tr>
<td>26</td>
<td>2</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>DCM [4M]</td>
<td>25</td>
<td>5.6</td>
<td>65 (1.17)</td>
<td>4000</td>
<td>4000</td>
<td>65</td>
<td>16</td>
</tr>
<tr>
<td>27</td>
<td>2</td>
<td>NHO</td>
<td>1:2:5:3000</td>
<td>DCM [2M]</td>
<td>25</td>
<td>12.0</td>
<td>50 (1.16)</td>
<td>3000</td>
<td>4000</td>
<td>75</td>
<td>13</td>
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<tr>
<td>28</td>
<td>2</td>
<td>P$_2$-tBu</td>
<td>1:4:5:2000</td>
<td>bulk</td>
<td>25</td>
<td>2</td>
<td>253 (1.3)</td>
<td>15000</td>
<td>14000</td>
<td>70</td>
<td>63</td>
</tr>
<tr>
<td>29</td>
<td>2</td>
<td>P$_2$-tBu</td>
<td>1:4:5:2000</td>
<td>bulk</td>
<td>-36</td>
<td>4</td>
<td>67 (1.16)</td>
<td>4000</td>
<td>5000</td>
<td>75</td>
<td>17</td>
</tr>
<tr>
<td>30</td>
<td>2</td>
<td>DBU</td>
<td>1:4:5:2000</td>
<td>bulk</td>
<td>25</td>
<td>7.2</td>
<td>98 (1.23)</td>
<td>6000</td>
<td>5000</td>
<td>68</td>
<td>24</td>
</tr>
<tr>
<td>31</td>
<td>3</td>
<td>NHO</td>
<td>1:4:5:2000</td>
<td>bulk</td>
<td>25</td>
<td>5 min</td>
<td>266 (1.21)</td>
<td>15000</td>
<td>14000</td>
<td>58</td>
<td>66</td>
</tr>
<tr>
<td>32</td>
<td>3</td>
<td>P$_2$-tBu</td>
<td>1:4:5:2000</td>
<td>bulk</td>
<td>-36</td>
<td>1 min</td>
<td>303 (1.18)</td>
<td>18000</td>
<td>18000</td>
<td>57</td>
<td>75</td>
</tr>
<tr>
<td>33</td>
<td>3</td>
<td>DBU</td>
<td>1:4:5:2000</td>
<td>bulk</td>
<td>25</td>
<td>30 min</td>
<td>260 (1.06)</td>
<td>15000</td>
<td>15000</td>
<td>56</td>
<td>65</td>
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<td>34</td>
<td>3</td>
<td>DBU</td>
<td>1:4:5:2000</td>
<td>bulk</td>
<td>-36</td>
<td>24</td>
<td>281 (1.26)</td>
<td>16000</td>
<td>19000</td>
<td>57</td>
<td>70</td>
</tr>
<tr>
<td>35</td>
<td>4</td>
<td>DBU</td>
<td>1:4:5:2000</td>
<td>bulk</td>
<td>25</td>
<td>24</td>
<td>207 (1.18)</td>
<td>12000</td>
<td>12000</td>
<td>57</td>
<td>52</td>
</tr>
<tr>
<td>36</td>
<td>4</td>
<td>DBU</td>
<td>1:4:5:5000</td>
<td>bulk</td>
<td>-36</td>
<td>72</td>
<td>853 (1.22)</td>
<td>49000</td>
<td>43000</td>
<td>56</td>
<td>85</td>
</tr>
</tbody>
</table>

---

a) Average number of PO repeat units determined via $^1$H NMR spectroscopy (CDCl$_3$). b) determined via GPC analysis (CHCl$_3$). c) determined via $^1$H NMR (CDCl$_3$). d) diad placement determined via $^1$H NMR spectroscopy.
### Polymerization of 1-Butylene oxide (BO)

**Table S2.** Data set of BO polymerization using diborane (C)/NHO in combination with BnOH (I) as initiator.

<table>
<thead>
<tr>
<th>#</th>
<th>Cat (C)</th>
<th>NHO/C/I/BO (molar)</th>
<th>solvent</th>
<th>[BO]</th>
<th>T [°C]</th>
<th>t [h]</th>
<th>x&lt;sup&gt;a&lt;/sup&gt; (D&lt;sub&gt;M&lt;/sub&gt;)&lt;sup&gt;b&lt;/sup&gt; [g/mol]</th>
<th>M&lt;sub&gt;calc&lt;/sub&gt;&lt;sup&gt;c&lt;/sup&gt; [g/mol]</th>
<th>M&lt;sub&gt;GPC&lt;/sub&gt;&lt;sup&gt;b&lt;/sup&gt; [g/mol]</th>
<th>M&lt;sub&gt;d&lt;/sub&gt; [%]</th>
<th>Conv. [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1:2.5:3:0000</td>
<td>toluene [2M]</td>
<td>-36</td>
<td>19 d</td>
<td>142 (1.36)</td>
<td>10300</td>
<td>10300</td>
<td>85</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>1:2.5:3:0000</td>
<td>toluene [2M]</td>
<td>-36</td>
<td>10 d</td>
<td>59 (1.13)</td>
<td>4300</td>
<td>4200</td>
<td>84</td>
<td>15</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1:2.5:3:0000</td>
<td>toluene [2M]</td>
<td>25</td>
<td>3 d</td>
<td>109 (1.16)</td>
<td>8000</td>
<td>7900</td>
<td>75</td>
<td>27</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>1:2.5:3:0000</td>
<td>bulk</td>
<td>25</td>
<td>24</td>
<td>42 (1.17)</td>
<td>3100</td>
<td>3000</td>
<td>75</td>
<td>14</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>2</td>
<td>1:2.5:3:0000</td>
<td>THF [2M]</td>
<td>25</td>
<td>3 d</td>
<td>44 (1.07)</td>
<td>3300</td>
<td>3100</td>
<td>72</td>
<td>15</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>2</td>
<td>1:2.5:3:0000</td>
<td>bulk</td>
<td>-36</td>
<td>48</td>
<td>86 (1.16)</td>
<td>6300</td>
<td>6400</td>
<td>85</td>
<td>20</td>
<td></td>
</tr>
</tbody>
</table>

*a* Average number of PO repeat units determined via 1H NMR spectroscopy (CDCl<sub>3</sub>). *b* determined via GPC analysis (CHCl<sub>3</sub>). *c* diad placement determined via 1H NMR spectroscopy.  

### Polymerization of Allyl Glycidyl Ether (AGE)

**Table S3.** Data set of AGE polymerization using diborane (C)/NHO in combination with BnOH (I) as initiator.

<table>
<thead>
<tr>
<th>#</th>
<th>Cat (C)</th>
<th>NHO/C/I/AGE (molar)</th>
<th>solvent</th>
<th>[AGE]</th>
<th>T [°C]</th>
<th>t [h]</th>
<th>x&lt;sup&gt;a&lt;/sup&gt; (D&lt;sub&gt;M&lt;/sub&gt;)&lt;sup&gt;b&lt;/sup&gt; [g/mol]</th>
<th>M&lt;sub&gt;calc&lt;/sub&gt;&lt;sup&gt;c&lt;/sup&gt; [g/mol]</th>
<th>M&lt;sub&gt;GPC&lt;/sub&gt;&lt;sup&gt;b&lt;/sup&gt; [g/mol]</th>
<th>M&lt;sub&gt;d&lt;/sub&gt; [%]</th>
<th>Conv. [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1:2.5:3:0000</td>
<td>toluene [2M]</td>
<td>-36</td>
<td>14 d</td>
<td>114 (1.33)</td>
<td>13100</td>
<td>12700</td>
<td>62</td>
<td>53</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>1:2.5:3:0000</td>
<td>bulk</td>
<td>-36</td>
<td>24</td>
<td>272 (1.15)</td>
<td>31100</td>
<td>31300</td>
<td>78</td>
<td>70</td>
<td></td>
</tr>
</tbody>
</table>

*a* Average number of PO repeat units determined via 1H NMR spectroscopy (CDCl<sub>3</sub>). *b* determined via GPC analysis (CHCl<sub>3</sub>). *c* diad placement determined via 1H NMR spectroscopy.  

### Preparation of Block Copolymers

**Table S4.** Preparation of block copolymers using different macroinitiators (I) and rac-PO as monomer in the presence of 1/NHO.

<table>
<thead>
<tr>
<th>#</th>
<th>Initiator</th>
<th>NHO/I/PO (molar)</th>
<th>solvent</th>
<th>T [°C]</th>
<th>t [h]</th>
<th>x&lt;sup&gt;a&lt;/sup&gt; (D&lt;sub&gt;M&lt;/sub&gt;)&lt;sup&gt;b&lt;/sup&gt; [g/mol]</th>
<th>M&lt;sub&gt;calc&lt;/sub&gt;&lt;sup&gt;c&lt;/sup&gt; [g/mol]</th>
<th>M&lt;sub&gt;GPC&lt;/sub&gt;&lt;sup&gt;b&lt;/sup&gt; [g/mol]</th>
<th>M&lt;sub&gt;d&lt;/sub&gt; [%]</th>
<th>Conv. [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>PCL1K</td>
<td>1:2.5:3:0000</td>
<td>toluene [2M]</td>
<td>25</td>
<td>48</td>
<td>211 (1.24)</td>
<td>38000</td>
<td>80</td>
<td>53</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>PCL7K</td>
<td>1:2.5:3:0000</td>
<td>toluene [2M]</td>
<td>25</td>
<td>20</td>
<td>150 (1.20)</td>
<td>12000</td>
<td>76</td>
<td>38</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>PCL2K</td>
<td>1:2.5:3:0000</td>
<td>toluene [2M]</td>
<td>25</td>
<td>4</td>
<td>53 (1.20)</td>
<td>6000</td>
<td>77</td>
<td>13</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>PCL1K-diol</td>
<td>1:2.5:3:0000</td>
<td>toluene [2M]</td>
<td>25</td>
<td>24</td>
<td>124 (1.16)</td>
<td>13000</td>
<td>76</td>
<td>31</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>PEO8K</td>
<td>1:2.5:2:0000</td>
<td>bulk</td>
<td>25</td>
<td>2.5</td>
<td>780 (1.11)</td>
<td>84000</td>
<td>67</td>
<td>97</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>PLA2K</td>
<td>1:2.5:3:0000</td>
<td>THF [2M]</td>
<td>25</td>
<td>7</td>
<td>106 (1.23)</td>
<td>47000</td>
<td>68</td>
<td>27</td>
<td></td>
</tr>
</tbody>
</table>

*a* Average number of PO repeat units determined via 1H NMR spectroscopy (CDCl<sub>3</sub>). *b* determined via GPC analysis (CHCl<sub>3</sub>). *c* diad placement determined via 1H NMR analysis. *d* determined via 1H NMR spectroscopy (CDCl<sub>3</sub)}.  

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S19
Representative Examples for Stereoanalysis via $^{13}$C NMR

**Figure S18.** $^{13}$C NMR analysis (CDCl$_3$, 300 K) with diad/triad sequence assignment of PPO. Polymer resulting from application of NHO/t after 3 h at -85°C.
NHO/8/BnOH/PO = 1:2:5:2000 (t = 2 h, bulk, -36°C)

DBU/2/BnOH/PO = 1:4:5:2000 (t = 4 d, bulk, -36°C)

DBU/2/BnOH/PO = 1:4:5:10000 (t = 7 d, bulk, -36°C)

**Figure S19.** Examples of $^{13}$C NMR analysis (CDCl$_3$, 300 K) with diad sequence analysis of PPO prepared under different conditions.
**Figure S20.** Example for $^{13}$C NMR analysis (CDCl$_3$, 300 K) with diad sequence analysis of PBO resulting from application NHO/1 after 72 h at room temperature.

**Figure S21.** Example for $^{13}$C NMR spectroscopy (CDCl$_3$, 300 K) with triad sequence analysis of PAGE resulting from application NHO/1 after 14 d at -36 °C.
Figure S22. $^1$H NMR analysis (CDCl$_3$, 400 MHz) of PO in the presence of diborane 3 (middle); the separate compounds are shown top and bottom.
DSC Analysis

**Figure S23.** DSC investigation (5K/min, second cycle) of PPO with $m = 88\%$ (semicrystalline).

**Figure S24.** DSC investigation (5K/min, second cycle) of PPO with $m = 65\%$ (amorphous).
Figure S25. DSC investigation (5K/min, second cycle) of PBO with $m = 75\%$ (amorphous).

Figure S26. DSC investigation (5K/min, second cycle) of PAGE with $m = 62\%$ (amorphous).
Figure S27. TGA curves of PPO recorded between 45 °C and 600 °C applying a heating rate of 10 K/min.

'H-DOSY NMR Analysis

Figure S28. 'H-DOSY NMR analysis of PCL-b-(it)-PPO. Conditions: NHO/1/PCL2K/PO = 1:2:2.5:1000, [PO]₀ = 2.0 mol/L, toluene, T = 25 °C, see Table S4, entry 3).
Figure S29. 'H-DOSY NMR analysis of PCL-b-(it)-PPO. Conditions: NHO/1/PCL7K/PO = 1:2:2:5:1000, [PO]0 = 2.0 mol/L, toluene, T = 25 °C, see Table S4, entry 2).

Figure S30. 'H-DOSY NMR analysis of PCL-b-(it)-PPO. Conditions: NHO/1/PCL10K/PO = 1:2:2:5:1000, [PO]0 = 2.0 mol/L, Toluene, T = 25 °C, see Table S4, entry 1.
Figure S31. $^1$H-DOSY NMR analysis of (it)-PPO-$b$-PEO-$b$-(it)-PPO. Conditions: NHO/PEO8K/PO = 1:2:5:2000, bulk, $T = 25$ °C, see Table S4, entry 4.

Figure S32. $^1$H-DOSY NMR spectroscopy of PLA-$b$-(it)-PPO. Conditions: NHO/PLA/PO = 1:2:5:3000, [PO]$_0$ = 2.0 mol/L, THF, $T = 25$ °C, see Table S4, entry 5.
GPC traces, PPO and Copolymers

**Figure S33.** GPC trace received from PCL10K (starting material) and PCL10K stirred in the presence of diborane 1/NHO (THF, no PO) for 24 h at RT.

**Figure S34.** GPC trace received from PCL10K (macroinitiator) and after polymerization of PO (PCL-b-(it)-PPO).
**Figure S35.** GPC trace received from PEO8K (macroinitiator) and after polymerization of PO ((it)-PPO-\(b\)-PEO-\(b\)-\((it)\)-PPO).

**Figure S36.** GPC trace received from PLA20K (macroinitiator) and after polymerization of PO (PLA-\(b\)-(it)-PPO)
Figure S37. GPC traces received from polymerization of (S)-PO using 1/NHO. Conditions: NHO/1/8nOH/(S)-PO = 1:2:5:2000 [M]₀ = 2.0 mol/L, THF, room temperature.
Crystal Structure Analysis, Tabular Data for Diborane 1

![Diborane 1 structure](image)

**Figure S38.** Single crystal X-ray structure of diborane 1.

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<td>Completeness to theta =</td>
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Absorption correction           Semi-empirical from equivalents
Max. and min. transmission      0.7528 and 0.6606
Refinement method              Full-matrix least-squares on F²
Data / restraints / parameters  5877 / 13 / 415
Goodness-of-fit on F²           1.041
Final R indices [I>2sigma(I)]   R1 = 0.0484, wR2 = 0.1187
R indices (all data)            R1 = 0.0680, wR2 = 0.1278
Absolute structure parameter    0.04(21)
Extinction coefficient          n/a
Largest diff. peak and hole     0.364 and -0.222 e.A⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (A² x 10⁻³) for buch350.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

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Table 3. Bond lengths [Å] and angles [deg] for buch350.

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Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (\(A^2 \times 10^3\)) for buch350.
The anisotropic displacement factor exponent takes the form:
-2 \(\pi^2\) \([h^2 a^*^2 U_{11} + ... + 2 h k a^* b^* U_{12}]\)

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&B(2)-C(35)-C(42)-C(41) & \quad 52.0(7)
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References


