Supporting Information

Solvent Design for the Catalyst Recycling of Rhodium/Amine Catalysts via scCO2 Extraction in the Reductive Hydroformylation of Alpha Olefins

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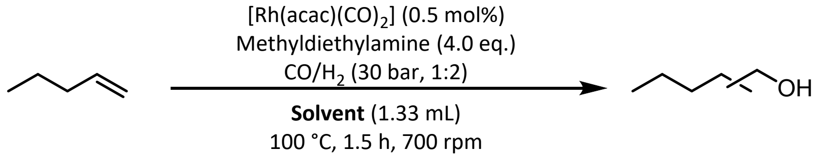
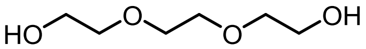
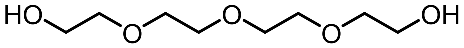
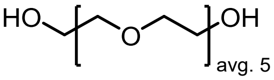
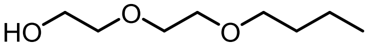
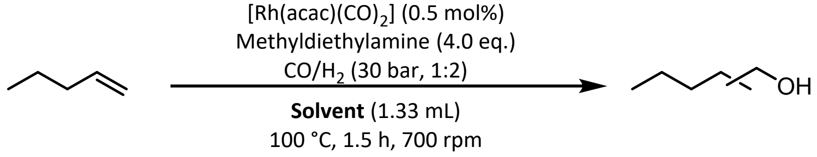
S 1 Solvent screening for the Rh/NEt_2Me catalyzed reductive hydroformylation of 1-pentene

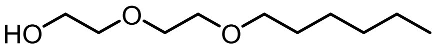
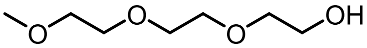
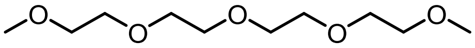
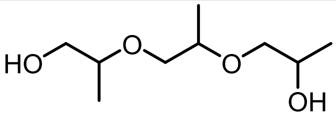
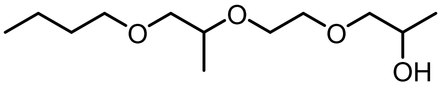
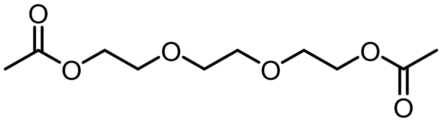
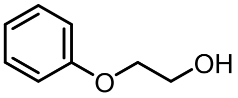
Table S 1: Reductive hydroformylation of 1-pentene catalyzed by [Rh(acac)(CO)$_2$]/NEt_2Me using different solvents. Hansen Parameters have been determined [a] by comparison with literature[1], [b] by calculation from thermodynamic data[1], [c] by group contribution methods as described by NIJENHUIS and VAN KREVELEN [2]. Hansen-Solubility-Parameters = dispersion forces (δ_D); dipole-dipole interactions (δ_P); hydrogen-bonding-contribution (δ_H)).



Product Yield [%]						**Hansen-Solubility-Parameters**		
$Y_{n\text{-Hexanol}}$	**$Y_{iso\text{-Hexanol}}$**	**$Y_{n\text{-Hexanal}}$**	**$Y_{iso\text{-Hexanal}}$**	**$Y_{Pentane}$**	**$Y_{Pentene\ Isomers}$**	**δ_D**	**δ_P**	**δ_H**
Solvent =  Triethylene glycol[a]								
46.8	16.0	1.1	1.4	0.5	11.0	16.0	12.5	18.6
Solvent =  Tetraethylene glycol[c]								
29.4	18.2	7.1	9.9	0.9	14.5	18.0	9.8	17.5
Solvent =  Polyethyleneglycole 300 (PEG_{300})								
45.7	13.1	0.0	0.0	0.0	18.6	-	-	-
Solvent =  Diethylene glycol monobutyl ether[a]								
42.4	15.3	0.0	0.0	2.2	32.3	16.0	7.0	10.6



[Rh(acac)(CO)$_2$] (0.5 mol%)
Methyldiethylamine (4.0 eq.)
CO/H$_2$ (30 bar, 1:2)
Solvent (1.33 mL)
100 °C, 1.5 h, 700 rpm

Product Yield [%]						**Hansen-Solubility-Parameters**		
$Y_{n\text{-Hexanol}}$	**$Y_{iso\text{-Hexanol}}$**	**$Y_{n\text{-Hexanal}}$**	**$Y_{iso\text{-Hexanal}}$**	**Y_{Pentane}**	**$Y_{\text{Pentene Isomers}}$**	**δ_D**	**δ_P**	**δ_H**
Solvent =  Diethylene glycol monohexyl ether[c]								
30.3	8.3	3.7	2.4	2.6	45.8	16.0	6.0	10.0
Solvent =  Triethylene glycol monomethy ether[a]								
43.0	13.9	0.0	0.0	1.6	34.1	16.2	7.6	12.5
Solvent =  Tetraethylene glycol dimethyl ether[b]								
39.3	13.7	0.0	0.0	1.0	39.0	16.6	6.2	5.8
Solvent =  Tripropyleneglycol[c]								
37.0	10.8	1.0	1.0	2.3	30.9	16.9	7.4	16.3
Solvent =  Tripropylene glycol monobutyl ether[c]								
18.5	5.7	5.1	2.9	1.1	56.6	16.3	5.2	10.8
Solvent =  Triethylene glycol diacetate[c]								
40.3	14.2	0.0	0.0	1.0	37.3	17.0	9.9	8.1
Solvent =  2-Phenoxyethanol[a]								
38.2	18.0	1.8	2.0	1.9	37.5	17.8	5.7	14.3

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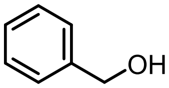
<tr><td colspan="9">Solvent = 
Benzylalcohol[a]</td></tr>
<tr><td>36.3</td><td>11.5</td><td>0.9</td><td>1.2</td><td>1.6</td><td>36.6</td><td>18.4</td><td>6.3</td><td>13.7</td></tr>
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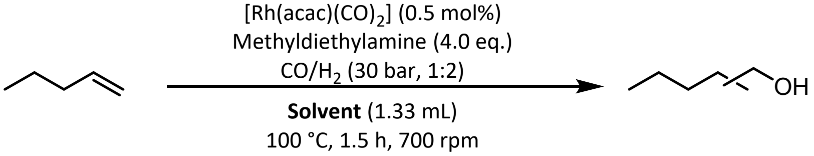

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<tr><th colspan="6">Product Yield [%]</th><th colspan="3">Hansen-Solubility-Parameters</th></tr>
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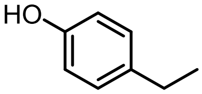
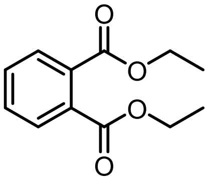
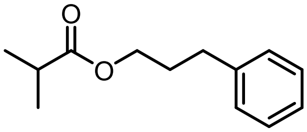
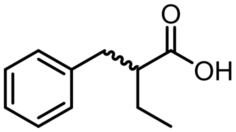
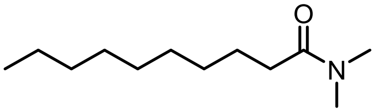
<tr><td colspan="9">Solvent = 
4-Ethylphenol[a]</td></tr>
<tr><td>25.3</td><td>8.6</td><td>0.0</td><td>1.0</td><td>1.9</td><td>49.5</td><td>19.2</td><td>5.3</td><td>12.8</td></tr>
<tr><td colspan="9">Solvent = 
Diethyl phthalate[a]</td></tr>
<tr><td>45.8</td><td>17.7</td><td>0.0</td><td>0.0</td><td>1.8</td><td>28.6</td><td>17.6</td><td>9.6</td><td>4.5</td></tr>
<tr><td colspan="9">Solvent = 
3-Phenylpropyl isobutyrat[c]</td></tr>
<tr><td>8.4</td><td>2.1</td><td>10.5</td><td>4.5</td><td>1.0</td><td>64.3</td><td>17.7</td><td>4.4</td><td>5.0</td></tr>
<tr><td colspan="9">Solvent = 
Ethylcinnamate[a]</td></tr>
<tr><td>12.2</td><td>2.6</td><td>8.8</td><td>2.7</td><td>0.8</td><td>50.3</td><td>18.4</td><td>8.2</td><td>4.1</td></tr>
<tr><td colspan="9">Solvent = 
N,N-Dimethyldecanamide[c]</td></tr>
<tr><td>45.9</td><td>14.6</td><td>2.1</td><td>2.2</td><td>1.6</td><td>27.0</td><td>15.8</td><td>4.7</td><td>5.4</td></tr>
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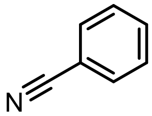
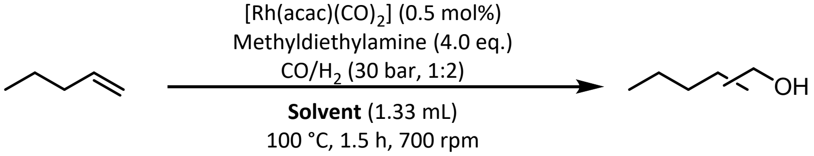
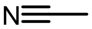
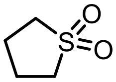
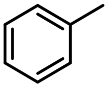
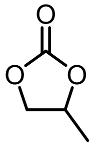
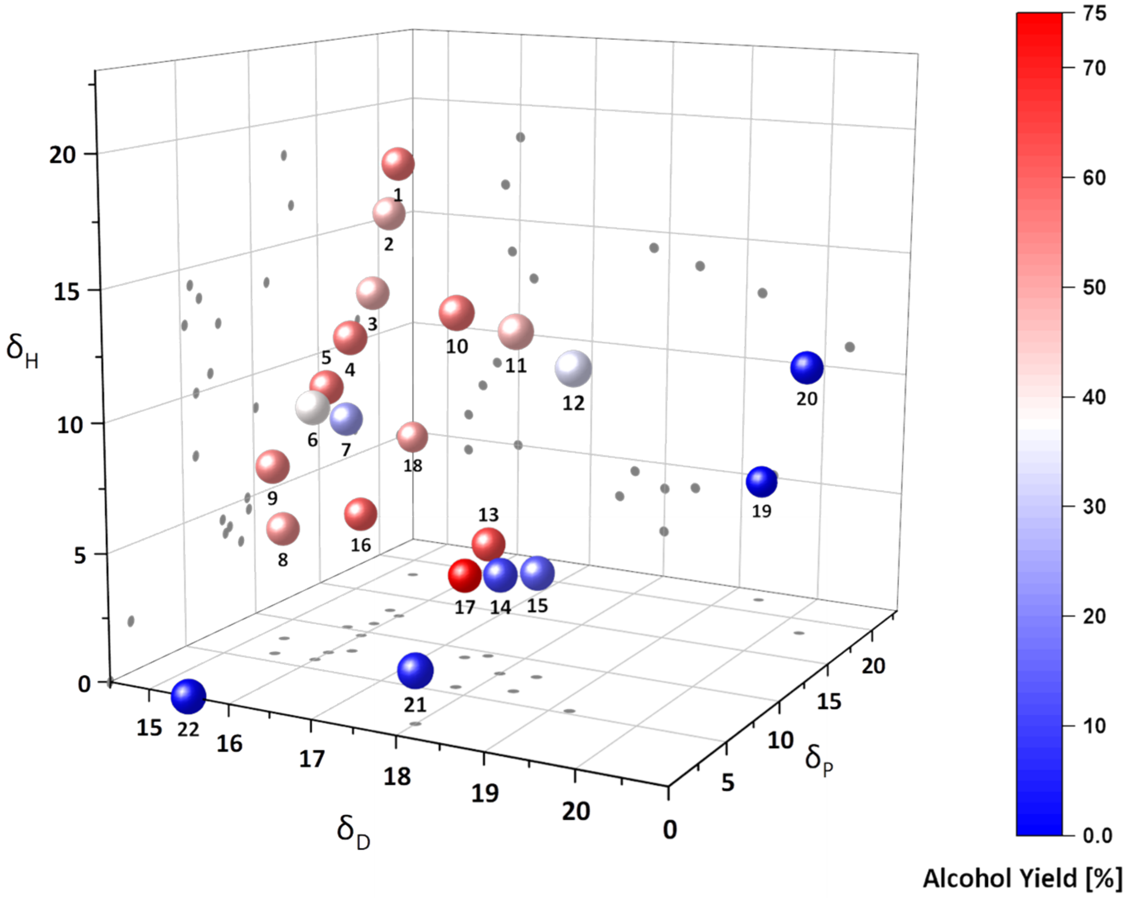
Solvent =  Benzonitrile[a]								
52.8	21.6	0.0	0.7	1.3	16.7	17.4	9.0	3.3



Product Yield [%]						Hansen-Solubility-Parameters		
$Y_{n\text{-Hexanol}}$	$Y_{iso\text{-Hexanol}}$	$Y_{n\text{-Hexanal}}$	$Y_{iso\text{-Hexanal}}$	Y_{Pentane}	$Y_{\text{Pentene Isomers}}$	δ_D	δ_P	δ_H
Solvent =  Acetonitrile[a]								
40.4	10.8	10.7	8.0	1.5	19.1	15.3	18.0	6.1
Solvent =  Sulfolan[a]								
1.2	0.00	29.4	8.9	0.4	19.6	20.3	18.2	10.9
Solvent =  Toluene[a]								
3.8	0.9	13.8	6.2	1.3	67.0	18.0	1.4	2.0
Solvent =  Octane[a]								
0.8	0.7	12.6	5.7	0.9	69.5	15.5	0.0	0.0
Solvent =  Propylene carbonate[a]								
0.4	0.0	35.0	11.1	0.0	16.9	20.0	18.0	4.1

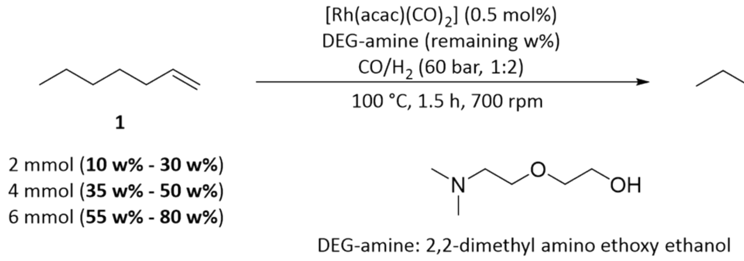


1 TEG
2 TetraEG
3 TPG
4 TEG monomethyl ether
5 DEG monobutyl ether
6 DEG monohexyl ether
7 TPG monobutyl ether
8 TetraEG dimethyl ether
9 TEG diacetate
10 2-Phenoxyethanol
11 Benzylalcohol
12 4-Ethylphenol
13 Diethylphtalate
14 3-Phenylpropyl isobutyrat
15 Ethyl cinnamate
16 Dimethyldecanamide
17 Benzonitrile
18 Acetonitrile
19 Propylene carbonate
20 Sulfolane
21 Toluene
22 Octane

DEG: Diethylene glycole
TEG: Triethylene glycole
TPG: Tripropylene glycole
TetraEG: Tetraethylene glycole

Figure S 1: 3D-Hansen-Space derived from the Hansen Parameters in Table S1. Plotting the Hansen-Solubility-Parameters in a three-dimensional space against their catalytic performance (in this case resembled by the alcohol yield in the reductive hydroformylation). The color code resembles the alcohol yield of the respective solvent in the reductive hydroformylation of 1-pentene as displayed in Table S 1.

S 2 Substrate concentration increase using 2-[2-(dimethylamino)ethoxy]ethanol (DEG-amine) as solvent/ligand:



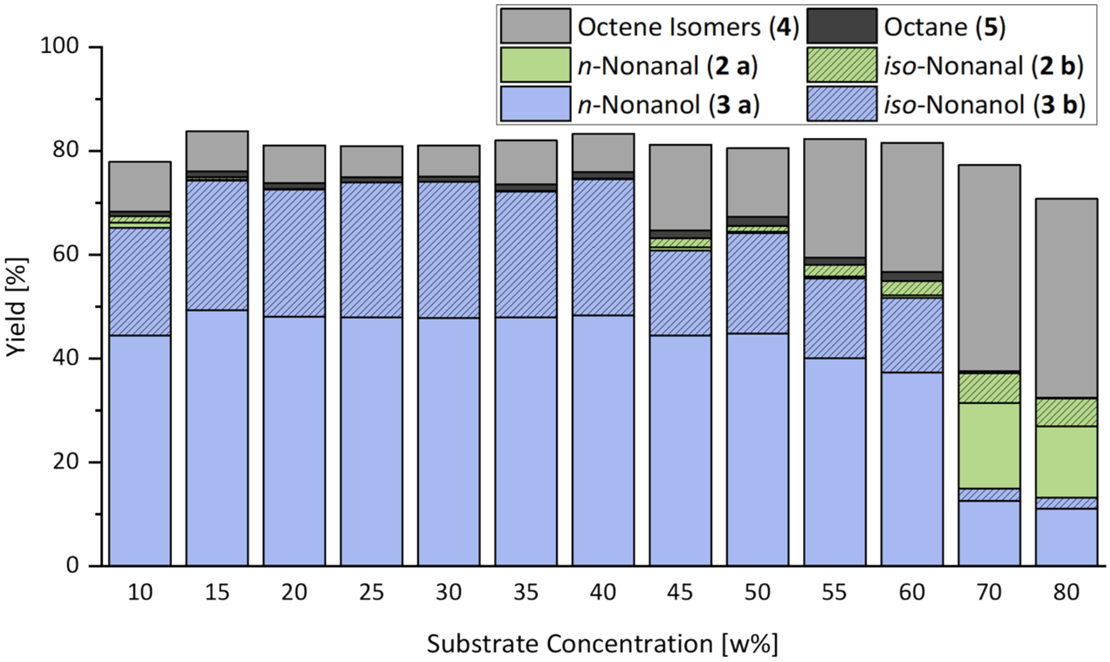
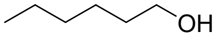
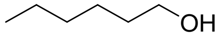
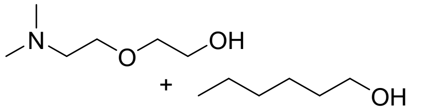
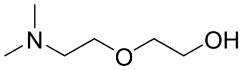
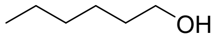


Figure S 2: Investigation on increasing the susbtrate concentration for the reductive hydroformylation of 1-octene, using 2-[2-(dimethylamino)ethoxy]ethanol (DEG-amine) as the solvent. Reaction conditions: 1-Octene (2.00 mmol (10 - 30 w%)/4.00 mmol (35 – 50 w%)/6.00 mmol (55 – 80 w%), 1.00 eq.), [Rh(acac)(CO)2] (0.5 mol%), DEG-amine (remaining w%), CO/H2 (60 bar, 1:2), 100 °C, 1.5 h, 1200 rpm.

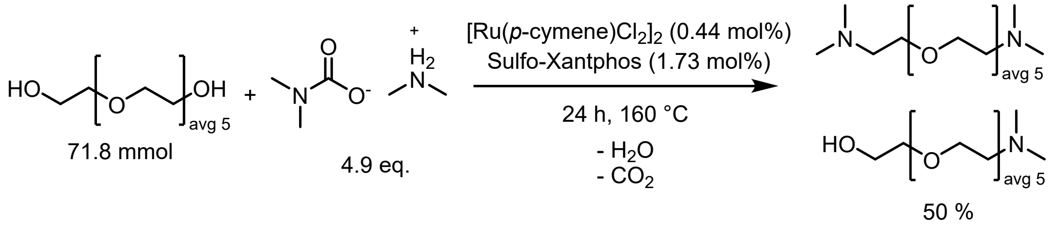
S 3 Model extraction of 1-hexanol from 2-[2-(dimethylamino)ethoxy]ethanol (DEG-amine) and polyethylene glycol 300 (PEG$_{300}$)

Table S 2: Extraction of alcohols using scCO$_2$ as extracting solvent from model solutions. For the extraction the setup described in the main script was used. Composition of the extract was determined via quantitative NMR as described in S 9.2.

No.	Initial Solution	Conditions for the Extraction	Extracted Compounds
2.1	PEG$_{200}$ +  60:40 w% (PEG$_{200}$:1-Hexanol), total mass 9.5 g	50 °C, 80 bar, 335 g *sc*CO$_2$ (2.7 g/min)	 >98 %
2.2	 60:40 w% (DEG-amin:1-Hexanol), total mass 14.5 g	50 °C, 80 bar, 280 g *sc*CO$_2$ (2.7 g/min)	 53 %  47 %

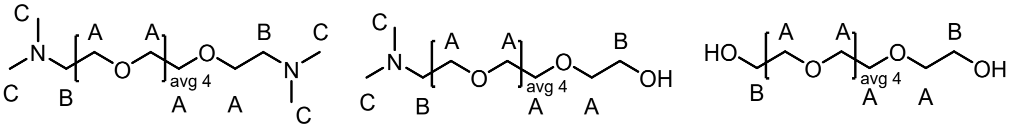
S 4 Experimental procedures:

S 4.1 Alcohol amination of PEG$_{300}$ using dimethylcarbamate (DimCarb)

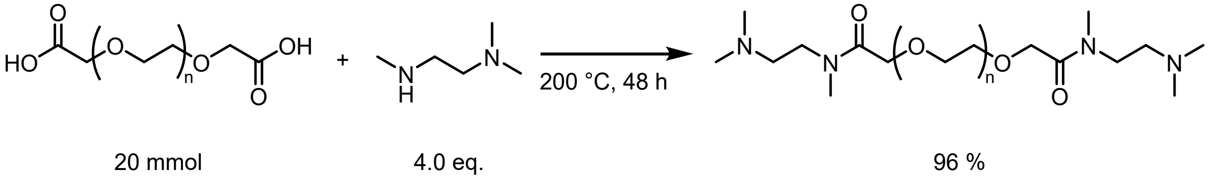


The diol (71.8 mmol, 1.0 eq.)(triethyleneglycol, 112-27-06, Acros Organics, 99%; polyethyleneglycole 300, 25322-68-3, Sigma Aldrich, no purity given), dimethylcarbamate (45 mL, 351 mmol, 4.9 eq.) (4137-10-4, Acros Organics, CO$_2$/HNMe$_2$ >2:1), [Ru(*p*-cymene)Cl$_2$]$_2$ (195.2 mg, 0.32 mmol, 0.44 mol%) (53462-29-0, Sigma Aldrich, ≥99%) and sulfoxanphos (984.3 mg, 1.73 mmol, 1.73 mol%) (215792-51-1, synthesized by Otto-von-Guericke-University, ≥95%) are added to a 200 mL stainless steel reactor with magnetic stir bar. The reactor is sealed and heated to 160 °C for 24 h at a stirring speed of 700 rpm. After the reaction the reactor is cooled to RT and the ruthenium catalyst is separated from the mixture by column chromatography over a Silca-Al$_2$O$_3$ plug (4 cm each). The column is washed with 200 mL dichloromethane (75-09-02, Carl Roth, ≥99.5%) and the obtained solution is transferred to a round bottom flask. DCM is removed under reduced pressure and the excess dimethylcarbamate is separated via distillation under argon flow (90 °C, atm). The remaining orange liquid is again purified by column chromatography over a Silca-Al$_2$O$_3$ column (6 cm each) which is rinsed with dichloromethane. dichloromethane is removed under reduced pressure. A mixture of di-, mono- and non-aminated diol is obtained as light-yellow viscous oil (11.8 g, 50%).

^{1}H NMR of a mixture of non- mono- di-aminated product (400 MHz, CDCl$_3$) δ = 3.75 – 3.53 (m, 69H, **A**), 2.50 (t, *J* = 5.7 Hz, 2H, **B**), 2.25 (s, 6H, **C**) ppm.

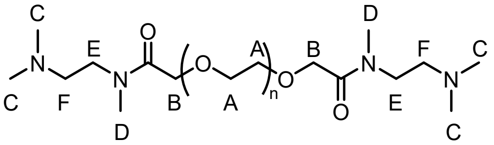


S 4.2 Amide condensation of polyethylene glycol 600 diacid (PEG_{600}-diacid) and trimethyldiaminoethane



PEG_{600} carboxylic acid (12.00 g, 10.14 ml, 20.0 mmol, 1.00 eq.) (39927-08-7, Merck Millipore, ≥95%) and trimethyldiaminoethane (8.172 g, 10,16 mL, 80.00 mmol, 4.0 eq.) (142-25-6, abcr, 98%) are added to a 200 mL stainless steel autoclave with magnetic stir bar. The solution is heated to 200 °C for 24 h at 700 rpm. Afterwards, the reactor is cooled to RT and excess amine as much as reaction water is removed under reduced pressure (100 mbar at 55 °C -> 24 mbar at 60 °C). The product is obtained as yellow viscous oil (14.74 g, 96%).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 4.21 (s, 2H, **B**), 4.18 (s, 2H, **B**), 3.76 – 3.54 (m, 43H, **A**), 3.46 (dd, *J* = 15.3, 8.4 Hz, 2H, **E**), 3.36 (t, *J* = 7.1 Hz, 2H, **E**), 3.02 – 2.87 (m, 6H, **D**), 2.42 (t, *J* = 7.0 Hz, 4H, **F**), 2.23 (s, 12H, **C**) ppm.



S 4.3 General procedure for high pressure autoclave experiments

For reductive hydroformylation experiments a high-pressure autoclave (12 mL for screening experiments, 50 mL for recycling experiments) equipped with manometer, glass inlet and magnetic stir bar is used. Into the glass inlet the respective olefin (1-pentene: 109-67-1, Alfa Aesar, 97%; 1-octene: 111-66-0, Acros Organics, ≥99%), $[Rh(CO)_2(acac)]$ (14874-82-9, abcr, 99%), the solvent and, if used, the amine ligand is weighed. The glass inlet is inserted into the autoclave reactor and afterwards sealed and flushed with 10 bar of hydrogen for at least two times. Subsequently, the reactor is pressurized up the corresponding partial pressure of hydrogen and filled to the corresponding total pressure with carbon monoxide. Afterwards, the autoclave is heated to 100 °C for 1.5 h (if not stated differently) using a heating cone on a magnetic stirring plate. After the reaction, the reactor is cooled to RT in a water bath and expanded to ambient pressure. The reaction mixture is transferred into a sample vail for GC analysis as explained in chapter 6.3.1.

S 5 Atmospheric-Pressure-Chemical-Ionization Mass-Spectrometry (APCI-MS) spectra for the aminated polyethylene glycol PEG_{300} derived from alcohol amination as described in S 4.1

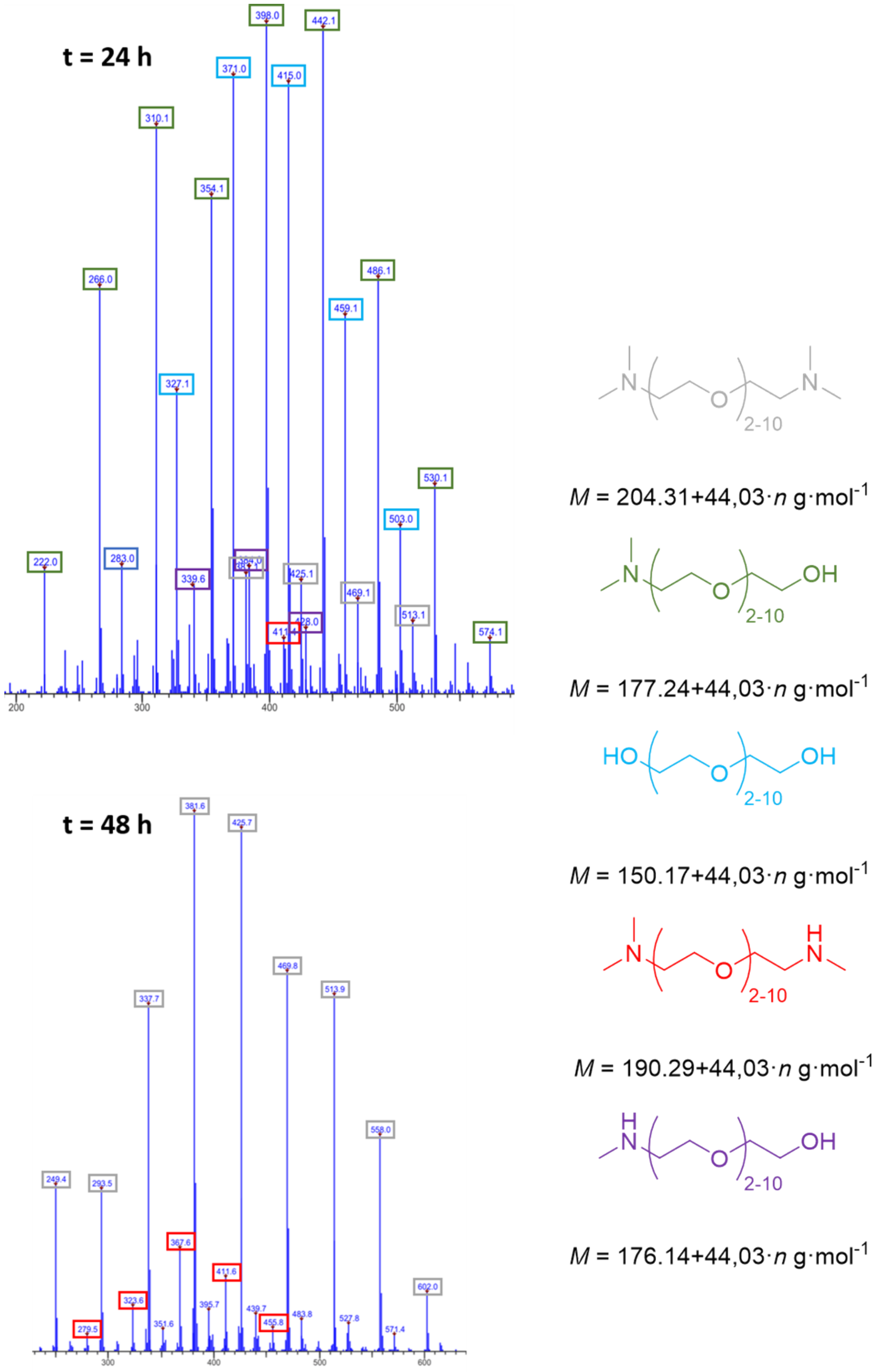
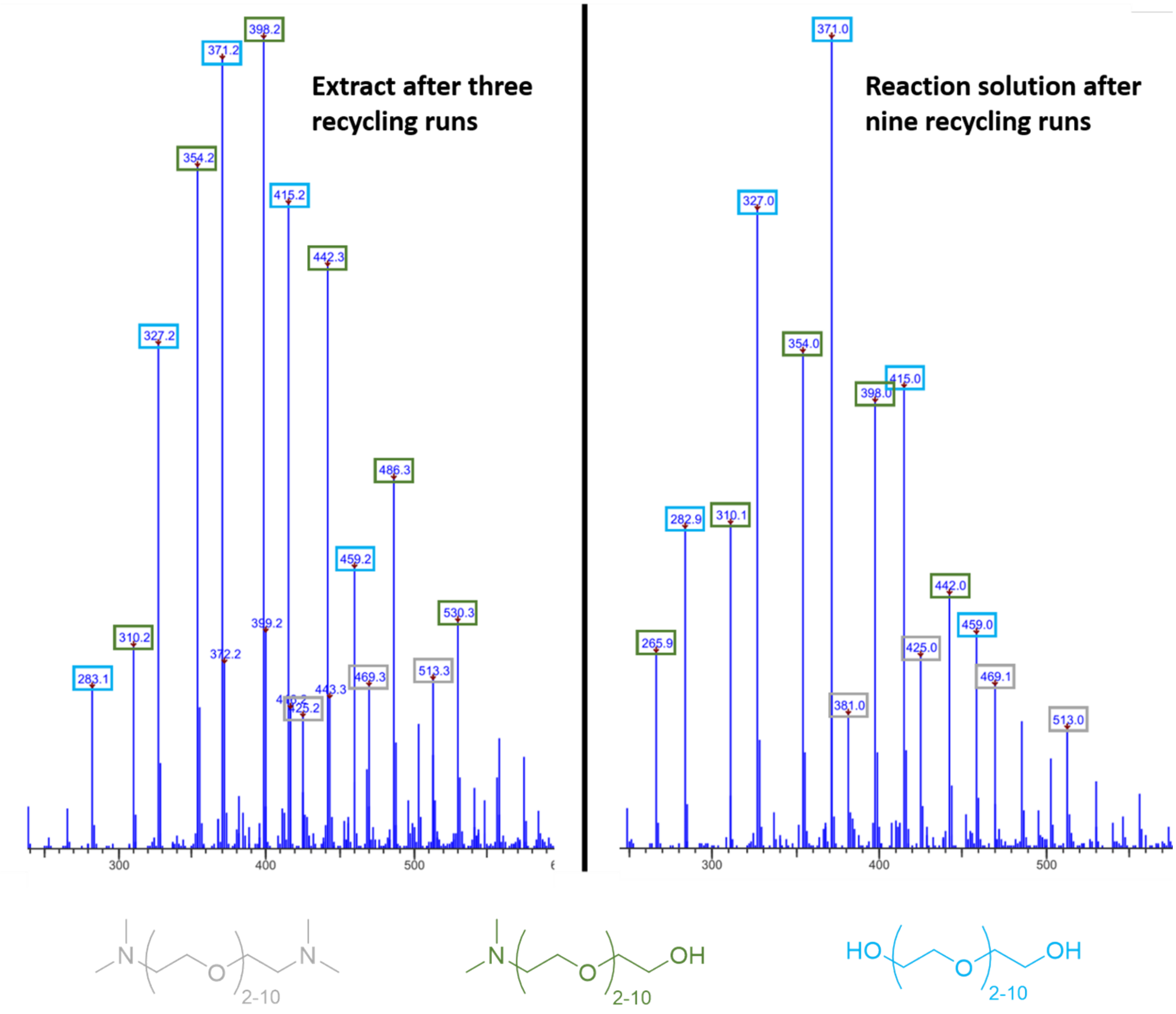


Figure S 3: APCI spectra of the purificated product mixture after 24 h (top) and 48 h (bottom) for the alcohol amination of PEG_{300} using dimethylcarbamate as amination agent (S4.1). Representative excerpts of the mass distribution for the product mixtures are shown.

S 6 Atmospheric-Pressure-Chemical-Ionization Mass-Spectrometry (APCI-MS) spectra of the aminated polyethylene glycol PEG$_{300}$ mixture during recycling experiments



$M = 204.31+44{,}03\cdot n\ \mathrm{g\cdot mol^{-1}}$ $M = 177.24+44{,}03\cdot n\ \mathrm{g\cdot mol^{-1}}$ $M = 150.17+44{,}03\cdot n\ \mathrm{g\cdot mol^{-1}}$

Figure S 4: APCI spectra of the extracted solution after the third recycling run (left) and the catalyst solution after nine recycling runs (right) measured for the recycling experiments depicted in Figure 9 in the main script.

S 7 Extraction efficiency of scCO2 for the alcohol/aldehyde product mixture during catalyst recycling experiments using PEG600-diamine as the stationary phase (main script Figure 11)

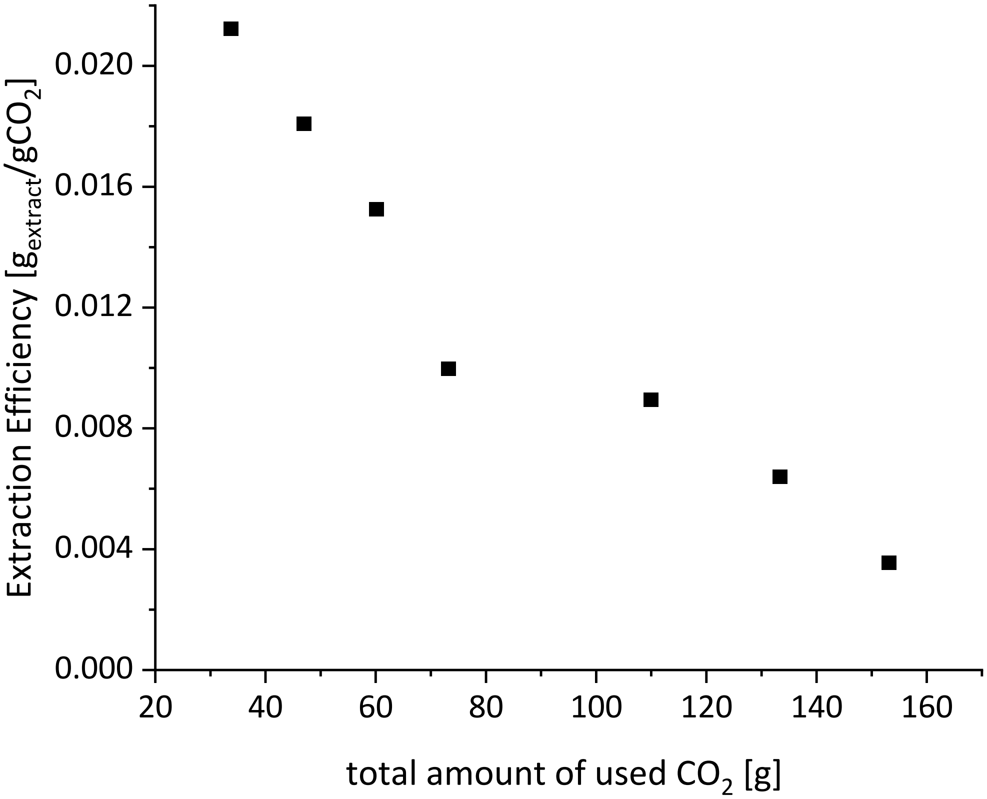


Figure S 5: Extraction efficiency with progressing CO_2 consumption during the first recycling run using PEG_{600}-diamine as the stationary catalyst phase (main script Figure 11).

S 8 Analysis of the CO_2 exhaust gas stream *via* GC-FID and GC-TCD after the extraction process

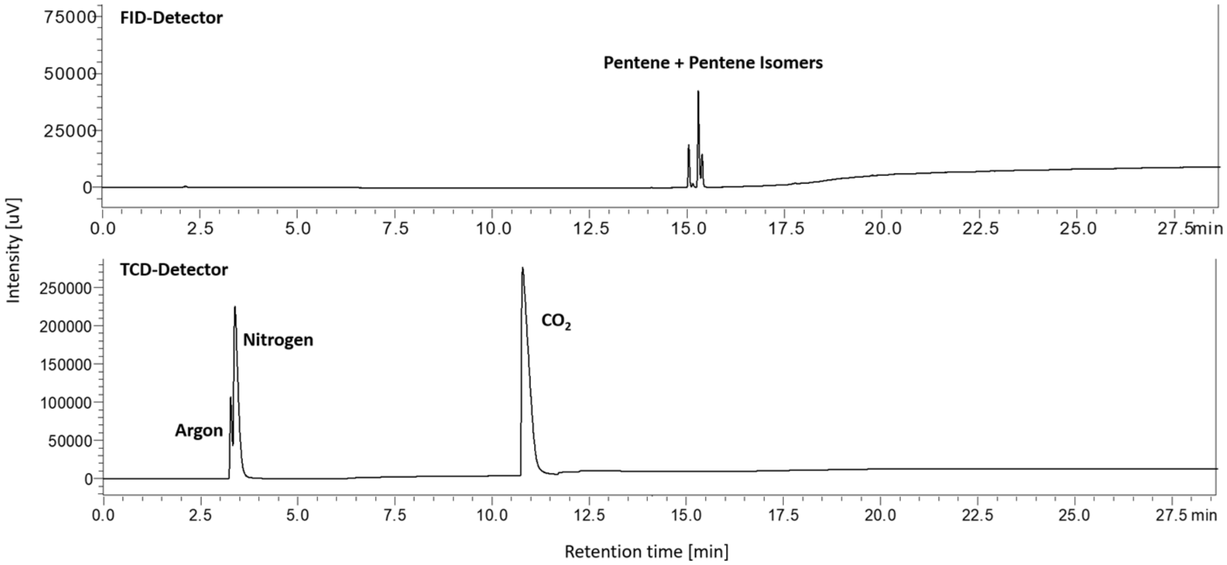


Figure S 6: Gas-Chromatographie of the exhaust gas stream after leaving the product container during the third recycling run using PEG_{600}-diamin as stationary catalyst phase (main script figure 11). In the top chromatogram a Flame-Ionization-Detector (FID) is used for analysis of hydrocarbons. In the bottom chromatogram a Thermal-Conductivity-Detector (TCD) is used for analysis of CO_2 and other gaseous molecules not visible in the FID. For sample taking a 200 mL glass cylinder equipped with two valves to allow for a gas flow and a septum for sample taking was used and connected to the exhaust line.

S 9 Analytical Procedures:

S 9.1 Gas-Chromatography with integrated Flame-Ionization-Detector (GC-FID) and Mass-Spectrometry-Detector (GC-MS)

Gas chromatographic (GC) analysis of the reaction mixture was performed to determine conversion and yields. For this a *Nexis GC-2030* gas chromatograph equipped with a FID detector, an *AOC-20sPlus* autosampler and an *AOC-20iPlus* injector by *Shimadzu* equipped with a *Rtx-1* column was used. For sample preparation, 25 mg tetradecane as internal standard (629-59-4, abcr, 99%), 200 mg of the sample and 800 mg *iso*-propanol (67-63-0, Carl Roth, ≥99.8%) were weighed into a GC vial and inserted to the GC via the auto sampler. The specifications of the used program and column are listed in Table S 3.

Table S 3: GC parameters and column specifications.

Parameter	Value
Column	30 m Rtx-1 (diameter 0,25 mm, film thickness 0,50 µm)
Carrier gas	N_2
Split-ratio	01:35
Carrier gas flow	1,47 mL/min
Detector	FID
Temperature program	hold 35 °C 12 min, 8 °C/min to 180 °C, 20 °C/min to 245 °C, hold 245 °C 5 min
Injection volume	0,2 µL

In order to quantify the results, calibrations of the pure compounds were recorded. For simplicity reasons, the same calibration factor was used for pentane and pentene isomers. Furthermore, the calibration factors of *n*- and *iso*-alcohols and *n*- and *iso*-aldehydes were assumed to be equal. The standard deviation for relevant analytes has been determined to be ≤5%.

For qualitative analysis of the individual compounds, gas chromatography with subsequent mass spectrometry (GC-MS) was used. For this, a *GCMS-QP2020* mass spectrometer with *GC-2010-Plus* gas chromatograph, *AOC-20sPlus* auto sampler and *AOC-20iPlus* injector (*Shimadzu*) have been used. Fragmentation patterns of individual compounds are listed below.

1-Octene MS (EI^+): **m/z** (rel. Int.) **39** (39%), **40** (12%), **41** (91%), **42** (60%), **43** (80%), **45** (41%), **47** (10%), **55** (100%), **56** (63%), **57** (13%), **69** (34%), **70** (90%), **71** (11%), **79** (28%), **81** (43%), **83** (30%), **84** (17%).

Octane MS (EI^+): **m/z** (rel. Int.) **39** (10%), **41** (43%), **43** (100%), **55** (13%), **56** (21%), **57** (37%), **70** (15%), **71** (25%), **84** (10%), **85** (44%).

***n*-Nonanal MS** (EI^+): **m/z** (rel. Int.) **54** (12%), **55** (45%), **56** (63%), **57** (100%), **67** (19%), **68** (26%), **69** (29%), **70** (38%), **71** (14%), **81** (26%), **82** (34%), **83** (12%), **95** (22%), **96** (21%), **98** (33%).

***iso*-Nonanal MS** (EI$^+$): **m/z** (rel. Int.) **55** (9%), **56** (4%), **57** (29%), **58** (100%), **59** (4%), **71** (15%), **84** (4%), **100** (4%).

***n*-Nonanol MS** (EI$^+$): **m/z** (rel. Int.) **39** (13%), **41** (74%), **42** (38%), **43** (72%), **55** (84%), **56** (100%), **57** (34%), **58** (26%), **68** (22%), **69** (35%), **70** (77%), **82** (12%), **83** (36%), **84** (25%).

***iso*-Nonanol MS** (EI$^+$): **m/z** (rel. Int.) **39** (12%), **41** (65%), **42** (23%), **43** (91%), **55** (41%), **56** (54%), **57** (100%), **58** (45%), **69** (37%), **70** (39%), **71** (71%), **83** (23%), **84** (29%).

1-Pentene MS (EI$^+$): **m/z** (rel. Int.) **27** (34%), **29** (37%), **40** (11%), **41** (75%), **42** (100%), **55** (90%), **57** (11%), **70** (55%).

Pentane MS (EI$^+$): **m/z** (rel. Int.) **27** (19%), **29** (17%), **39** (20%), **41** (57%), **42** (61%), **43** (100%), **57** (20%), **72** (15%).

***iso*-Hexanal MS** (EI$^+$): **m/z** (rel. Int.) **39** (14%), **41** (30%), **43** (100%), **55** (11%), **57** (17%), **58** (77%), **71** (18%), **72** (11%).

***n*-Hexanal MS** (EI$^+$): **m/z** (rel. Int.) **39** (24%), **41** (77%), **42** (15%), **43** (58%), **44** (100%), **45** (23%), **55** (19%), **56** (96%), **57** (64%), **58** (16%), **67** (13%), **71** (10%), **72** (24%), **82** (18%).

***iso*-Hexanol MS** (EI$^+$): **m/z** (rel. Int.) **39** (10%), **41** (31%), **42** (18%), **43** (100%), **55** (27%), **56** (25%), **69** (30%), **70** (32%), **71** (34%), **84** (10%).

***n*-Hexanol MS** (EI$^+$): **m/z** (rel. Int.) **39** (9%), **41** (37%), **42** (37%), **43** (53%), **55** (48%), **56** (100%), **57** (8%), **69** (35%).

S 9.2 Proton Nuclear-Magnetic-Resonance-Spectroscopy (^{1}H-NMR)

Nuclear magnetic resonance (NMR) spectroscopy was used to identify isolated substances and to quantify alcohol, aldehyde and amine amounts after the *sc*CO_2 extraction experiments. NMR spectra were recorded using an *Acend 400* spectrometer by *Bruker*. For sample preparation two drops of the sample are dissolved in deuterated chloroform (865-49-6, Sigma Aldrich, 99.8%). Chemical shifts δ in parts per million (ppm) are referenced to the signal (δ = 7.21 ppm) of the deuterated solvent.

For quantitative NMR (*q*-NMR) analysis a defined amount of mesitylene (108-67-8, abcr, 98%) as internal standard is added to the sample. Based on the number of protons of an isolated functional group which can be assigned to a substance and the internal standard, the amount of a specific substance is calculated. For the determination of the amount of amine an average value based on both signals in proximity to the amine (B and C in Figure S 7) was used. For *q*-NMR measurements relaxation delay times (D1) of 20 s were used, which allowed complete relaxation of spins. The standard deviation of the *q*-NMR measurements was determined to be ≤1.5% within the concentration range of reaction product analytes. All measurements were performed at room temperature. An example with assignment of all occurring species is shown in Figure S 7.

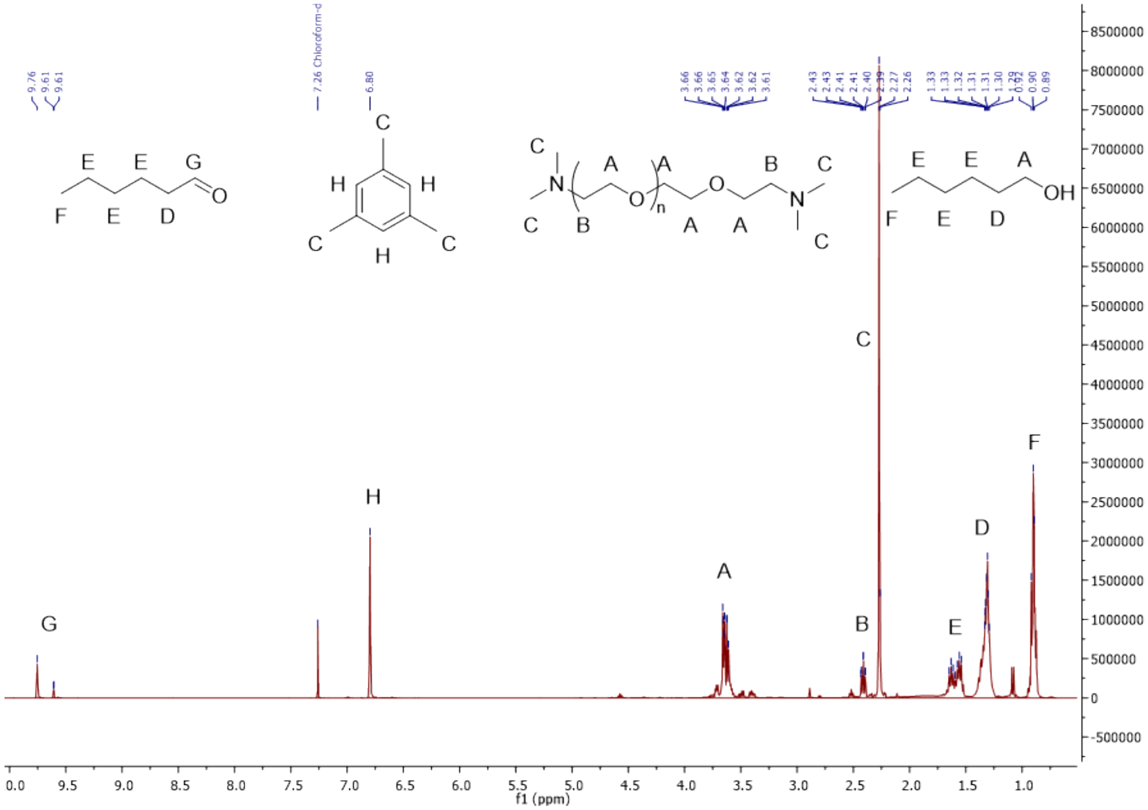


Figure S 7: Example of an QNMR spectrum of the second recycling run with assignment of all occurring species. For simplification reasons it is not distinguished between n- and iso-products

S 9.3 Inductively-Coupled-Plasma Mass-Spectrometry (ICP-MS)

Metal contents for the recycling experiments were determined by Inductively-Coupled-Plasma Mass-Spectrometry (ICP-MS). For the determination of metal contents in the recycling experiments using the PEG_{300}-diamin (main script, Figure 9) ICP-MS experiments have been measured according to the following procedure: Prior to injection, all organic material must be digested. For this, 10 mg of sample are mixed with 8 mL concentrated HNO_3 and heated in a microwave oven (CEM, Mars 6) to 200 °C (1500 W, temperature ramp of 30 minutes from RT). Afterwards the temperature is held for 15 minutes. After cooling, the sample is filled to 100 mL with distilled H_2O and injected into the ICP-MS (ICPMS-2030, Shimadzu). For each measurement, commercially available calibration standards (Carl Roth) of the target metal analyte, in the expected concentration range, are injected prior to the measurement. The metal concentration is determined by comparing the average of all measurements with the pre-measured calibration curves. Metal contents for the recycling experiments using the PEG_{600}-diamine (main script, Figure 11) have been measured with the help of the Kolbe Microanalytical Laboratory in 46047 Oberhausen, Germany. For each ICP-MS experiment the sample is injected seven times. The minimum and maximum value of each experiment are disregarded and from the remaining five injections an average value and a standard deviation from the average is given. The standard deviation in all cases was determined to be ≤1% of the total metal concentration. The absolute error derived from the analytical procedure was determined to be 0.075 ppm of the total metal concentration.

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