Supplementary Information for:

Switchable, Chiral Aluminium Catalysts for Ring Opening Polymerisations

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Experimental

General considerations

All reactions and handling were carried out using standard Schlenk line techniques or an MBRAUN UNlab Plus glove box under a nitrogen atmosphere. *D,L*-Lactide was triple sublimed before use to ensure purity. Dry solvents were obtained from MBRAUN-800 SPS and stored over 4.0 Å molecular sieves under a dry nitrogen environment. Proligands were synthesised as previously described.[1] All other starting materials were used as they were received from Alfa Aesar, Acros Organics, Fischer Scientific and Merck. All NMR spectra for complexes was collected on a Bruker AVIII 400 MHz spectrometer and a Bruker AVIIIHD 400 MHz spectrometer. All NMR spectra for polymers was collected on a Bruker AVIII 400 MHz spectrometer and a Bruker AVIIIHD 400 MHz spectrometer at Heriot-Watt university and a Bruker AVIII 800 MHz spectrometer at Edinburgh university. Mass spectrometry data for the complexes was collected on a Bruker micrOTOF spectrometer at the University of Edinburgh. The tacticity of the PLA samples was determined utilising the method described by Coates and Ovitt.[2] The Size Exclusion Chromatography analysis of the PLA samples were carried out using a Shimadzu High Performance Liquid Chromatograph fitted with a 7.5 mm internal diameter Agilent GPC column. The detector was a Shimadzu RID-20A. HPLC grade Tetrahydrofuran (Acros organics) was utilised as the eluent with a flow rate of 1 mL/min and an oven temperature of 35 °C. The measurement was calibrated against 10 polystyrene standards in the range of 162-364,000 g/mol and corrected using the Mark-Houwink parameters for PLA ($K = 0.0549$, $\alpha = 0.639$) and PS ($K = 0.0125$, $\alpha = 0.717$).[3, 4] All crystal structures were recorded on a D8 Venture, with Cu-K_α ($\lambda = 1.5418$ Å) source at 100 K cooled with a Cryostream. The structures were solved by intrinsic phasing SHELXT and refined by full-matrix least-squares on F^2 using SHELXL interfaced through Olex2.[5-7] Molecular graphics for all structures were generated using Mercury.[8] The supplementary crystallographic data can be found free of charge on the joint Cambridge Crystallographic Data Centre and Fachin-formationszentrum Karlsruhe Access Structures service. Deposition numbers: 2352910-2352912.

General synthesis for complexes 1-4

The proligand (2 mmol) was dissolved in dry toluene (8 mL) and then cooled to 0 °C. Trimethyl aluminium (1.00 mL of 2M in toluene, 2.0 mmol) was added dropwise to the reaction mixture. Once gas evolution had ceased the reaction mixture was allowed to warm slowly to room temperature. The reaction mixture was then heated to 90 °C for 6 hours. The reaction mixture was allowed to cool gradually to room temperature before being stirred for 16 hours at room temperature. The volatiles were removed via vacuum and the resulting yellow/off white solid was then dissolved in dry pentane (2 mL) and placed in a freezer overnight. The solution was filtered via canula, with the filtrate being collected. The volatiles were then removed under vacuum and the resulting white solid was collected in a glovebox.

Compound **1**

Yield = 0.78 g (67.3%). ^{1}H NMR (400 MHz, C$_6$D$_6$, 25 °C): δ 7.57 (d, 2H, J = 2.5 Hz, ArH), 6.83 (d, 2H, J = 2.5 Hz, ArH), 4.04 (d, 2H, J = 13.0 Hz, ArCH_2N), 3.76-3.66 (m, 4H), 2.94 (s, 2H, NCH_2COOEt), 1.69 (s, 18H, CCH_3), 1.33 (s, 18H, CCH_3), 0.79 (t, 3H, J = 7.1 Hz, CH$_2$CH_3), -0.36 (s, 3H, AlCH_3). ^{13}C NMR (100 MHz, C$_6$D$_6$, 25 °C): δ 165.8, 154.8, 139.0, 137.5, 124.0, 123.7, 119.9, 60.1, 55.1, 50.8, 34.3, 33.1, 30.8, 28.8, 12.7, 0.2. HRMS (EI): m/z [M]$^+$ found 579.3845, [M]$^+$ calculated 579.3863. Empirical formula: C$_{35}$H$_{54}$AlNO$_4$.

Compound **2**

Yield = 0.83 g (69.9%). ^{1}H NMR (400 MHz, C$_6$D$_6$, 25 °C): δ 7.56 (d, 1H, J = 2.5 Hz, ArH), 7.55 (d, 1H, J = 2.5 Hz, ArH), 6.77 (d, 1H, J = 2.3 Hz, ArH), 6.65 (d, 1H, J = 2.4 Hz, ArH), 3.96 (d, 1H, J = 13.0 Hz, ArCH_2N), 3.57-3.34 (m, 4H), 3.24 (d, 1H, J = 12.1 Hz, ArCH_2N), 2.93 (q, 2H, J = 7.1 Hz, OCH_2CH$_3$), 1.76 (s, 9H, CCH_3), 1,74 (s, 9H, CCH_3), 1,39 (s, 9H, CCH_3), 1.37 (s, 9H, CCH_3), 0.86 (d, 3H, J = 7.1 Hz, CHCH_3), 0.61 (t, 3H, J = 7.2 Hz, CH$_2$CH_3), -0.00 (s, 3H, AlCH_3). ^{13}C NMR (100 MHz, C$_6$D$_6$, 25 °C): δ 156.8, 156.7, 138.7, 138.7, 138.2, 138.1, 124.5, 124.5, 124.0, 120.7, 120.5, 62.5, 60.1, 57.1, 54.9, 35.1, 33.9, 33.9, 31.7, 31.7, 29.6, 13.2, 11.9, 1.0. HRMS (EI): m/z [M-H]$^+$ found 593.3969, [M-H]$^+$ calculated 592.3941. Empirical formula: C$_{36}$H$_{56}$AlNO$_4$.

Compound **3**

For compound **3**, 1 mmol of proligand L^3H_2 was used.

Yield = 0.26 g, (38.8%). ^1H NMR (400 MHz, C_6D_6, 25 °C): δ 7.60 (d, 1H, J = 2.5 Hz, ArH), 7.53 (d, 1H, J = 2.5 Hz, ArH), 6.98-6.93 (m, 3H), 6.90-6.85 (m, 2H), 6.81 (d, 1H, J = 2.1 Hz, ArH), 6.79 (d, 1H, J = 2.4 Hz, ArH), 4.33 (d, 1H, J = 13.3 Hz, ArCH_2N), 3.92 (d, 1H, J = 12.2 Hz, ArCH_2N), 3.59-3.44 (m, 4H), 3.37 (d, 1H, J = 10.1 Hz, ArCH_2N), 3.27-3.17 (m, 4H), 2.96 (dd, 1H, CHCH_2C$_5$H$_6$) 2.82 (t, 1H, J = 12.3 Hz, NCH(Bn)COOEt), 1.76 (s, 9H, CCH_3), 1.71 (s, 9H, CCH_3), 1.38 (s, 9H, CCH_3), 1.36 (s, 9H, CCH_3), 0.40 (t, 3H, J = 7.2 Hz, CH$_2$CH_3), -0.08 (s, 3H, AlCH_3). ^{13}C NMR could not be obtained due to solubility issue in deuterated solvent. HRMS (EI): m/z [M-H]$^+$ found 668.4256, [M-H]$^+$ calculated 668.4254. Empirical formula: $C_{42}H_{60}AlNO_4$.

Compound **4**

Yield = 0.85g, (71.6%). ^1H NMR (400 MHz, C_6D_6, 25 °C): δ 7.57 (d, 2H, J = 2.5 Hz, ArH), 6.70 (d, 2H, J = 2.5 Hz, ArH), 3.76 (q, 2H, J = 7.1 Hz, OCH_2CH$_3$), 3.40 (d, 2H, J = 13.3 Hz, ArCH_2N), 3.10 (s, 2H, J = 13.3 Hz, ArCH_2N) 2.84 (t, 2H, J = 7.6 Hz, NCH_2CH$_2$), 2.11 (t, 2H, J = 7.6 Hz, CH$_2$CH_2COOEt), 1.67 (s, 18H, CCH_3), 1.39 (s, 18H, CCH_3), 0.83 (t, 3H, J = 7.3 Hz, CH$_2$CH_3), -0.30 (s, 3H, AlCH_3). ^{13}C NMR (100 MHz, C_6D_6, 25 °C): δ 168.9, 154.4, 139.0, 137.7, 123.9, 123.4, 119.8, 59.7, 55.5, 48.0, 34.3, 33.2, 30.8, 28.8, 26.0, 12.8, 0.2. HRMS (EI): m/z [M-H]$^+$ found 592.3925, [M-H]$^+$ calculated 592.3941. Empirical formula: $C_{36}H_{56}AlNO_4$.

^{1}H and ^{13}C{^{1}H} NMR spectra for complexes 1-4
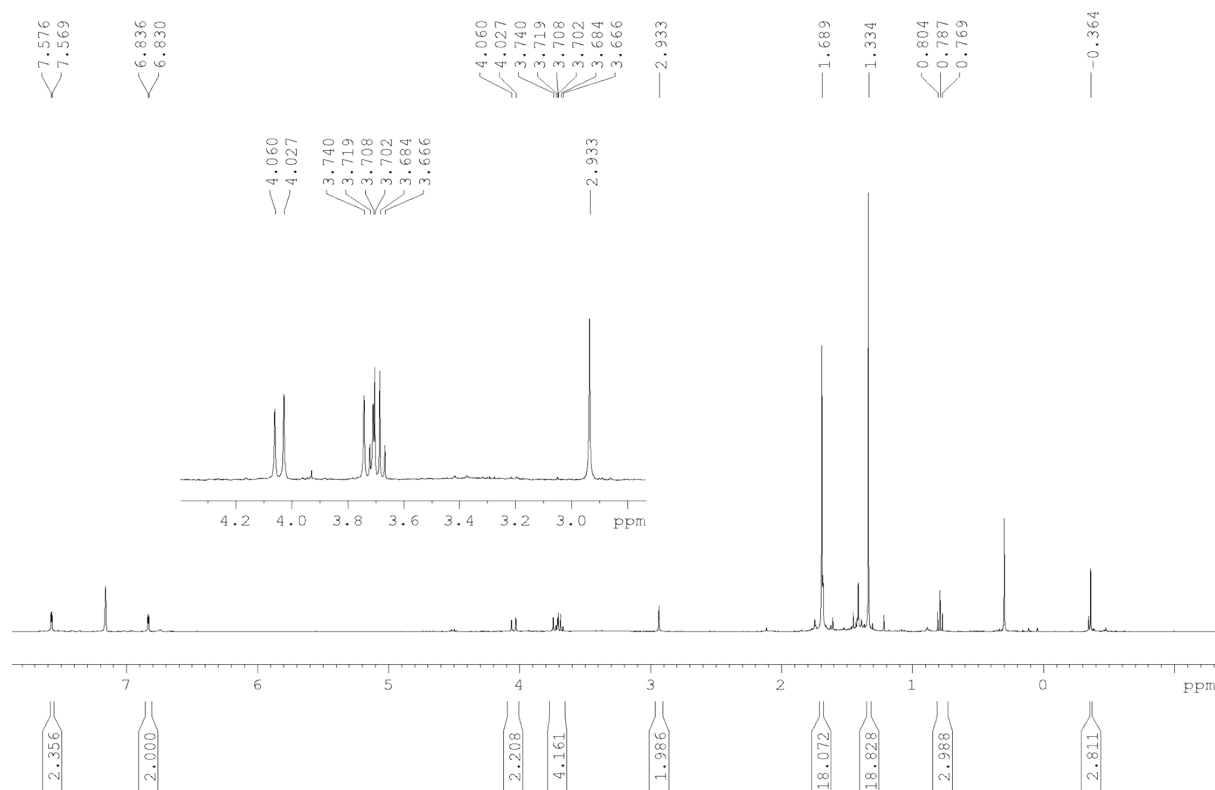
Complex **1**

Figure S1. ^{1}H NMR spectrum of complex **1** (400 MHz, C$_6$D$_6$, 25 °C).
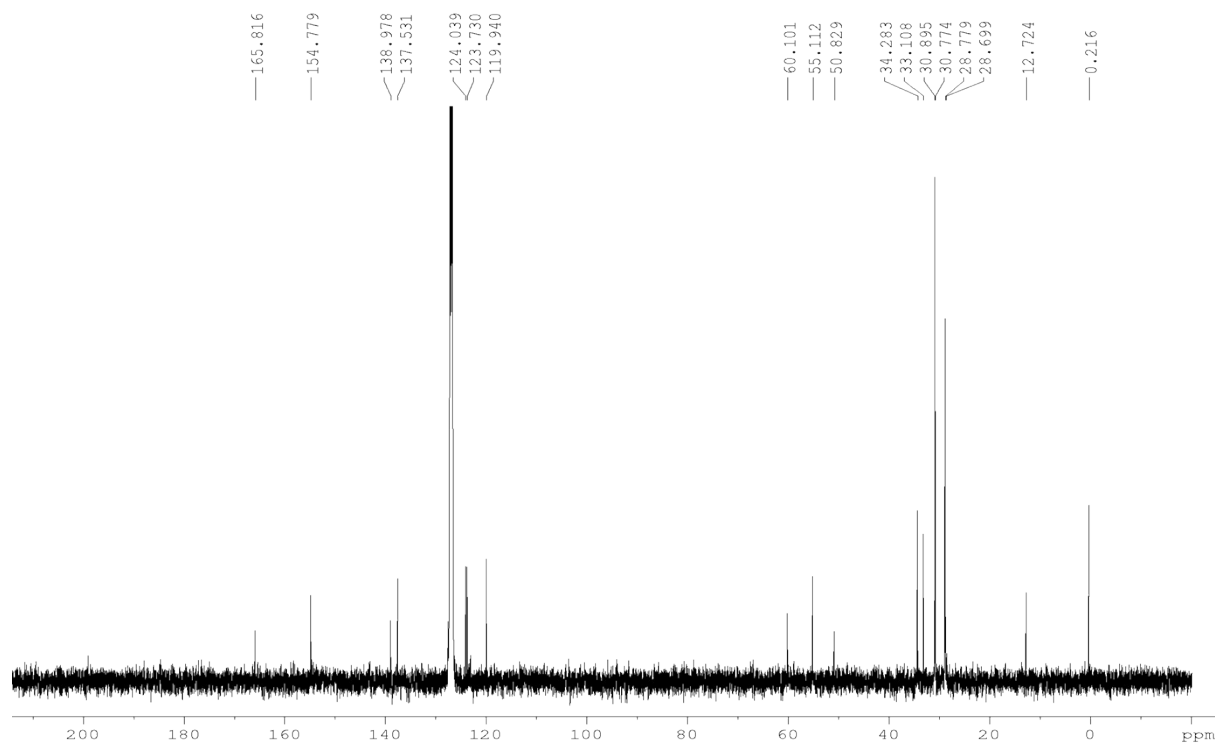
Figure S2. ^{13}C{^{1}H} NMR spectrum of complex **1** (100 MHz, C$_6$D$_6$, 25 °C)

Complex **2**
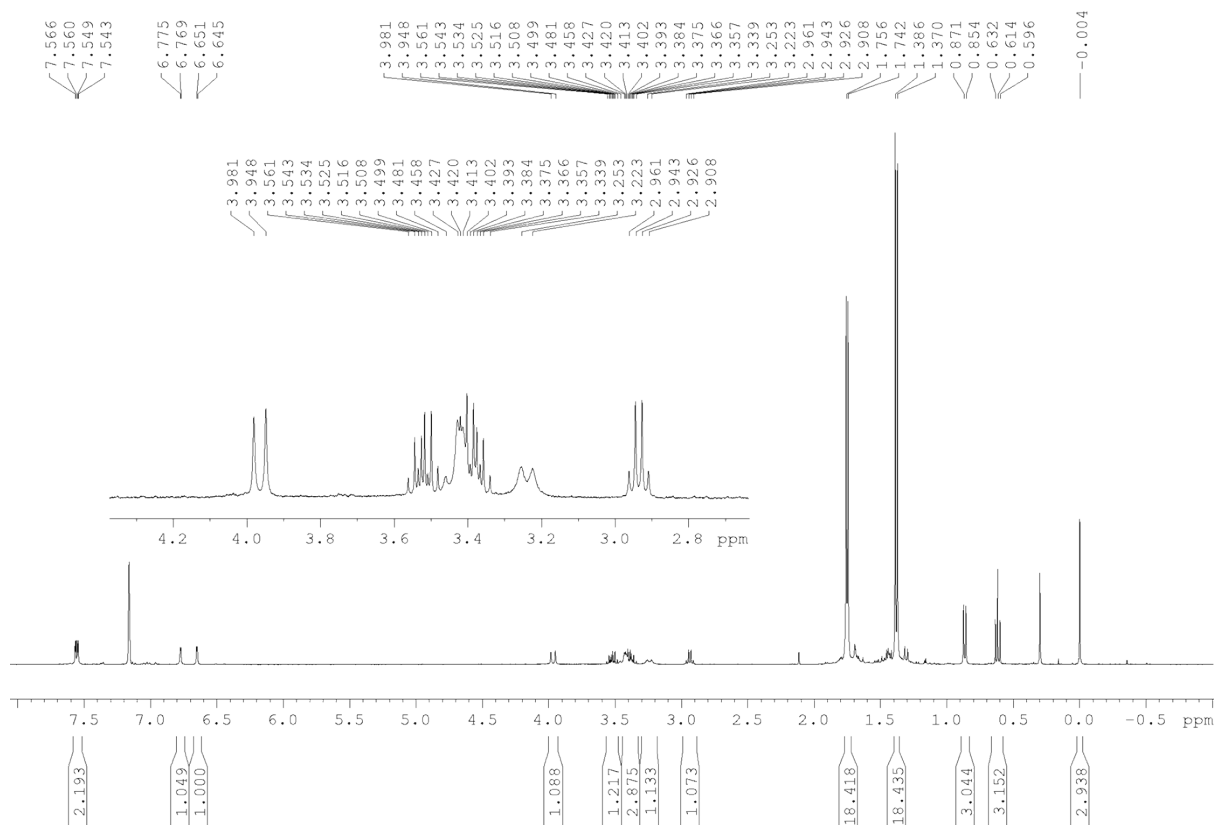
Figure S3. ^{1}H NMR spectrum of complex **2** (400 MH$_z$, C$_6$D$_6$, 25 °C).
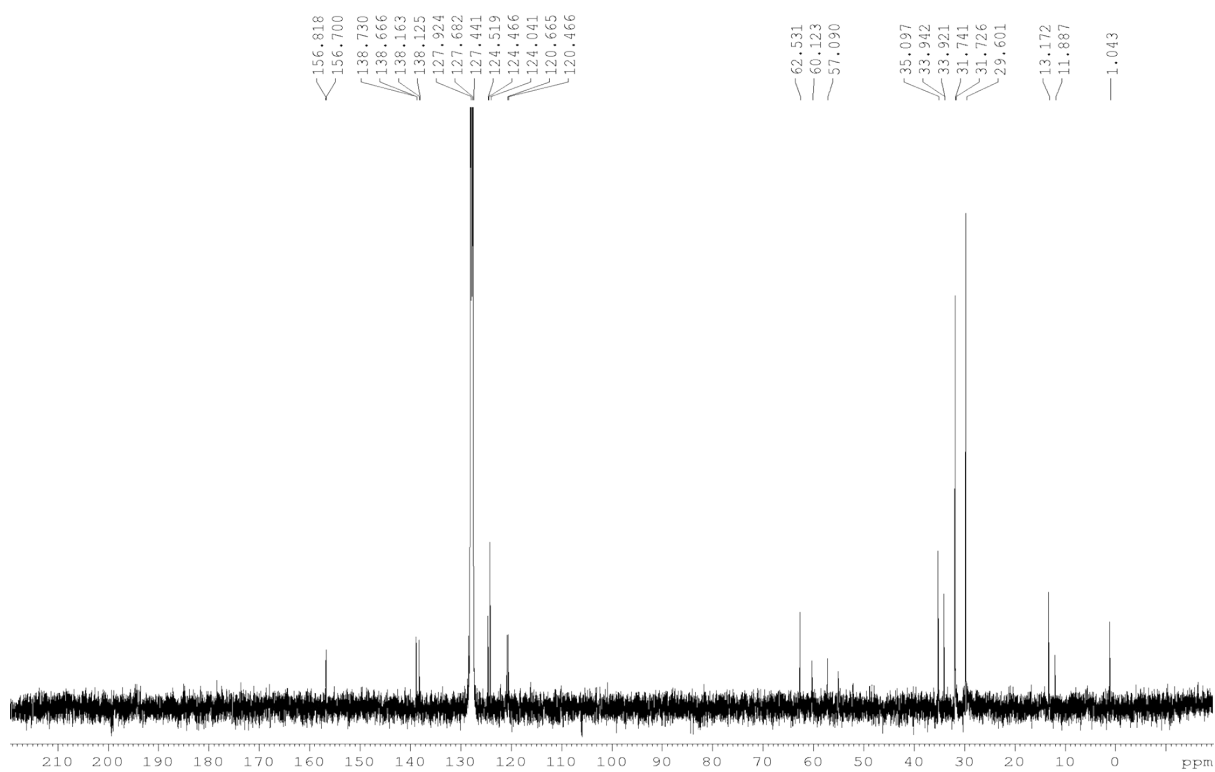
Figure S4. $^{13}C\{^{1}H\}$ NMR spectrum of complex **2** (100 MHz, C$_6$D$_6$, 25 °C).

Complex **3**
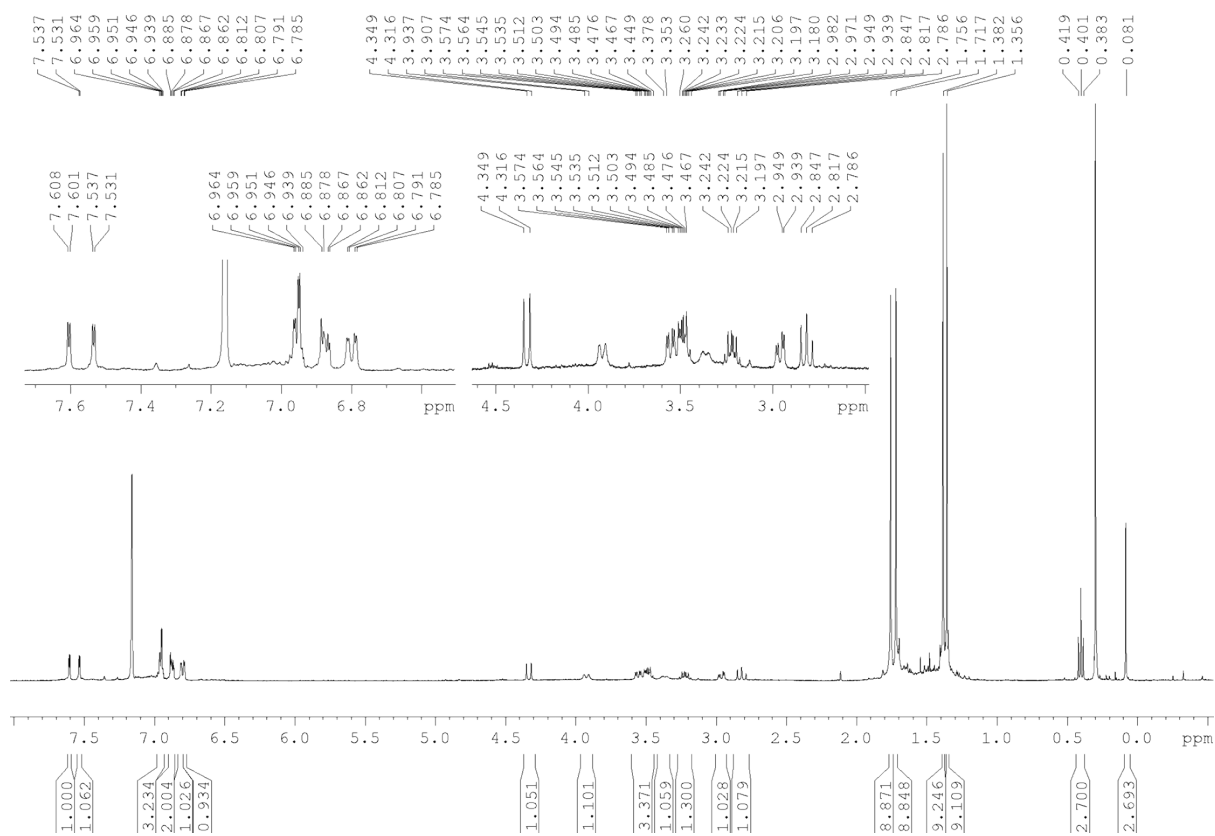
Figure S5. ^{1}H NMR spectrum of complex **3** (400 MHz, C$_6$D$_6$, 25 °C).

Complex **4**
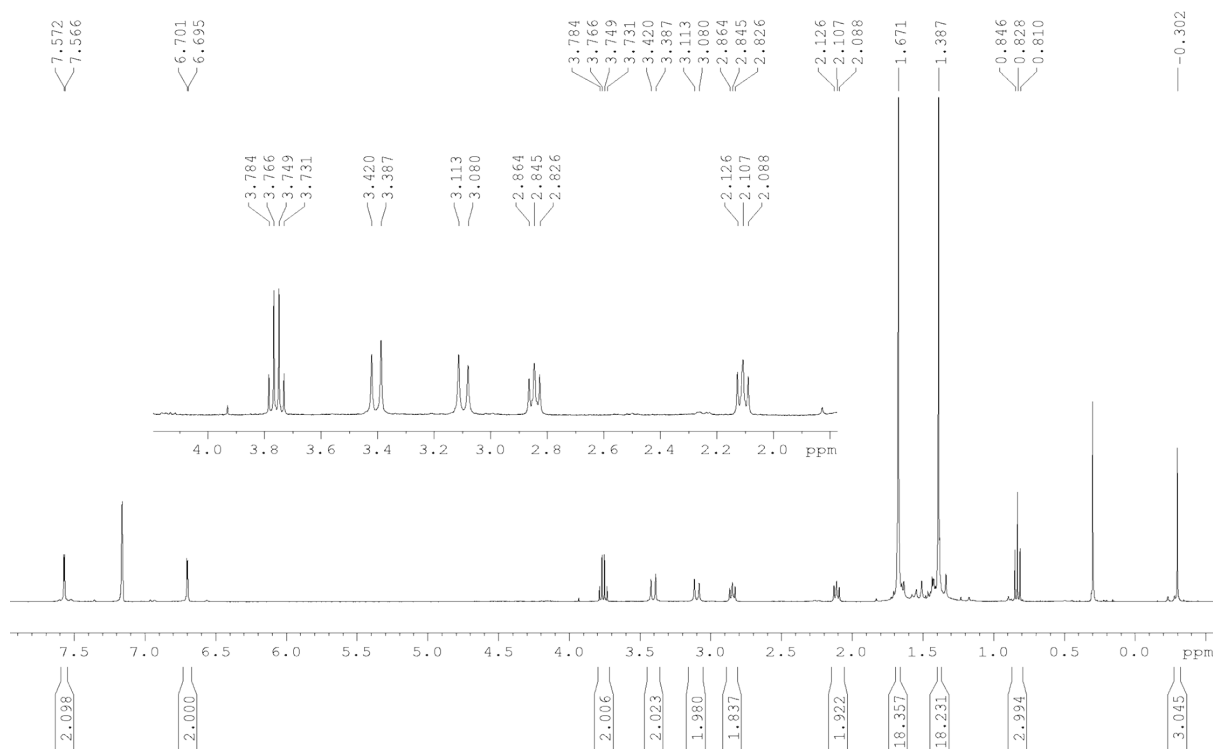
Figure S6. ^{1}H NMR spectrum of complex **4** (400 MHz, C$_6$D$_6$, 25 °C).
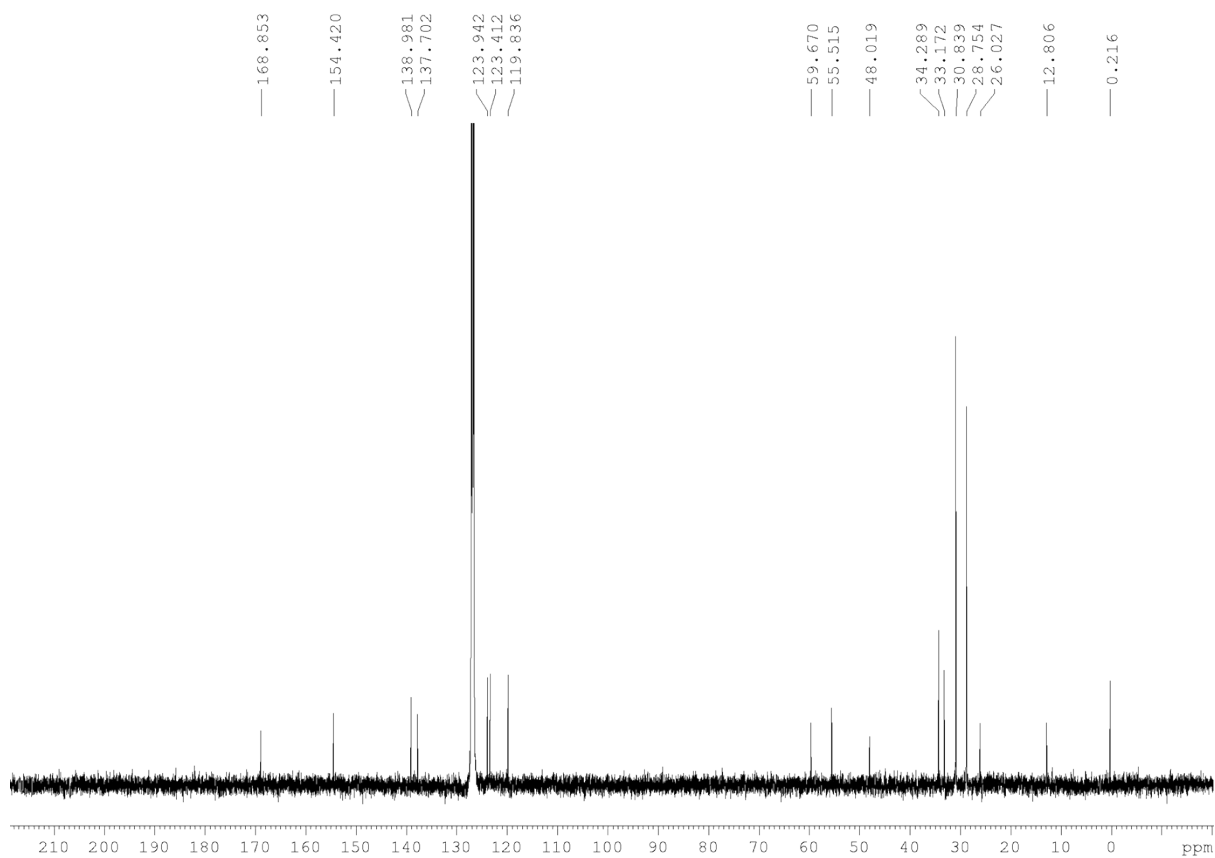
Figure S7. ^{13}C{^{1}H} NMR spectrum of complex **4** (100 MHz, C$_6$D$_6$, 25 °C).

HRMS data for complexes 1-4
Complex 1
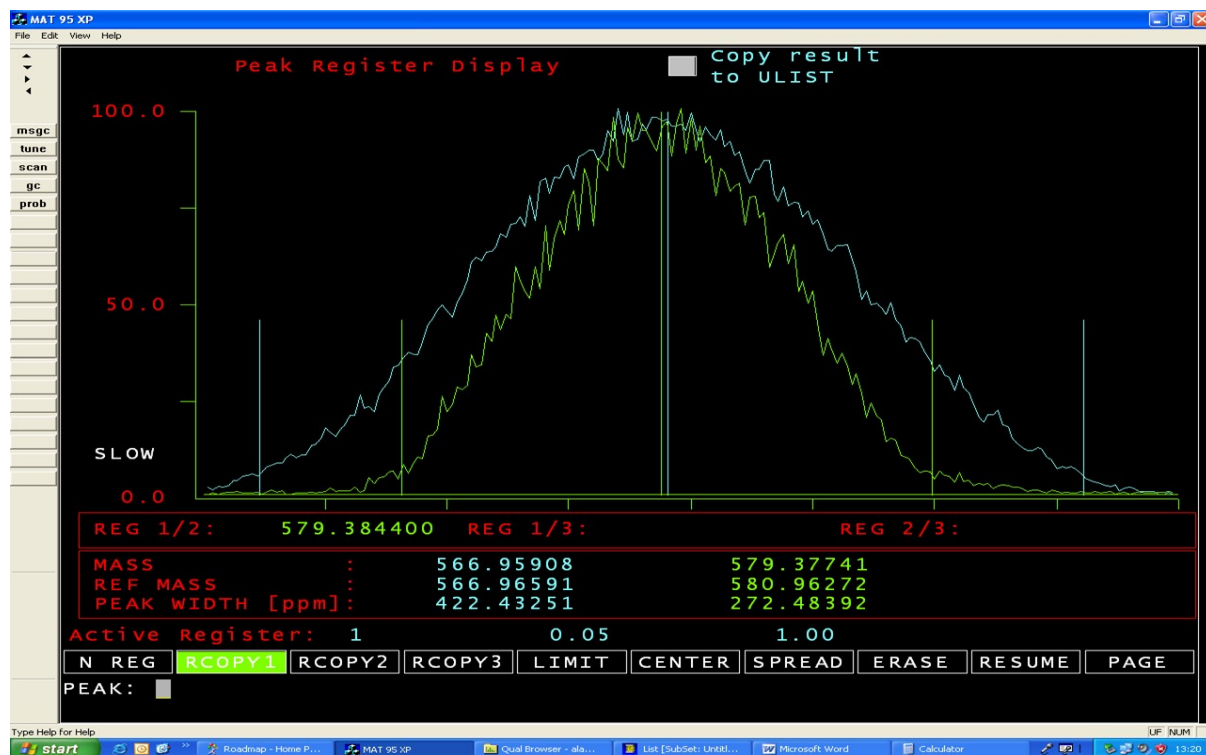

Figure S8. HRMS data for complex **1**.
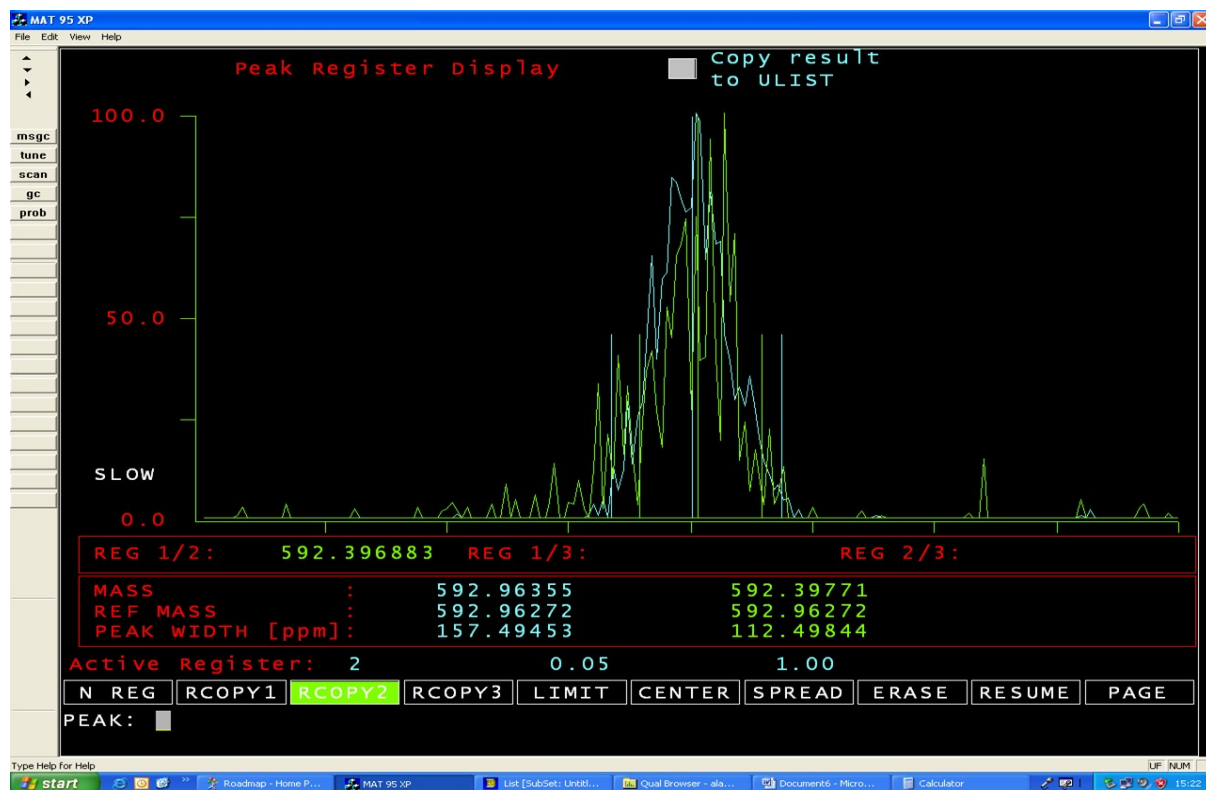
Complex 2

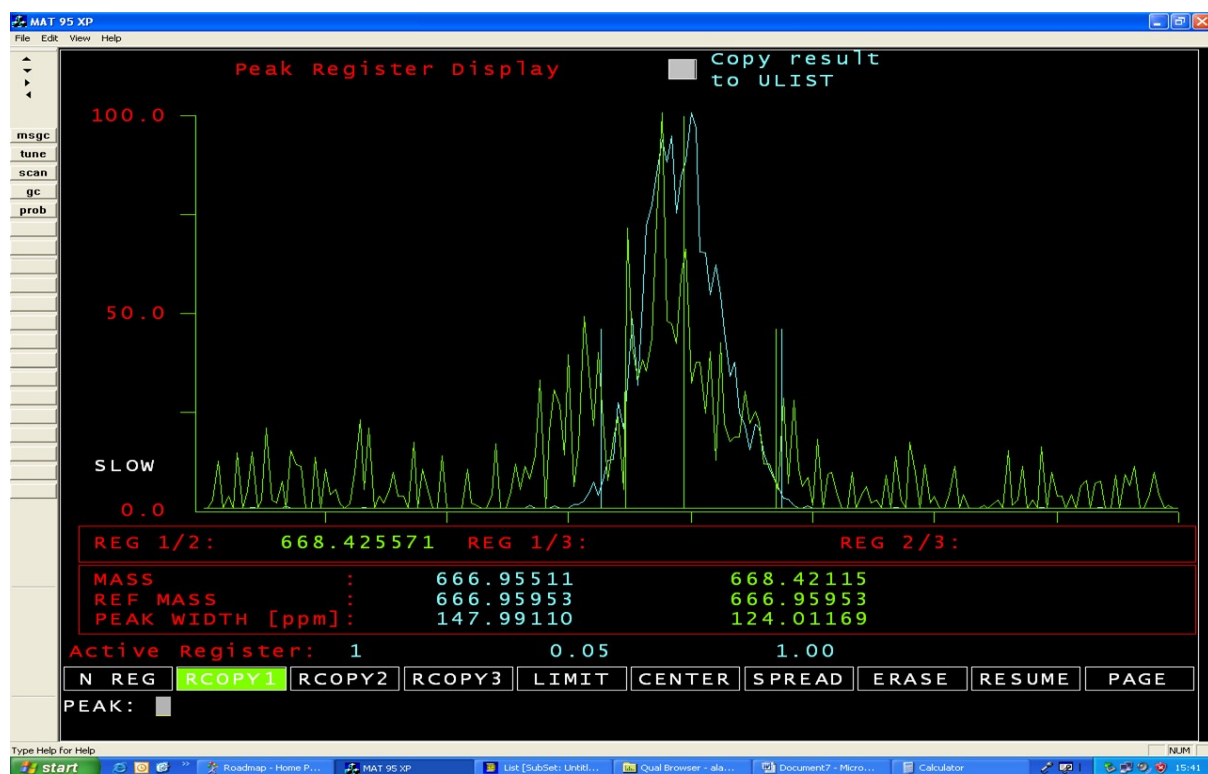**Figure S9.** HRMS data for complex **2**.

Complex 3

Figure 10. HRMS data for complex **3**.

Complex 4
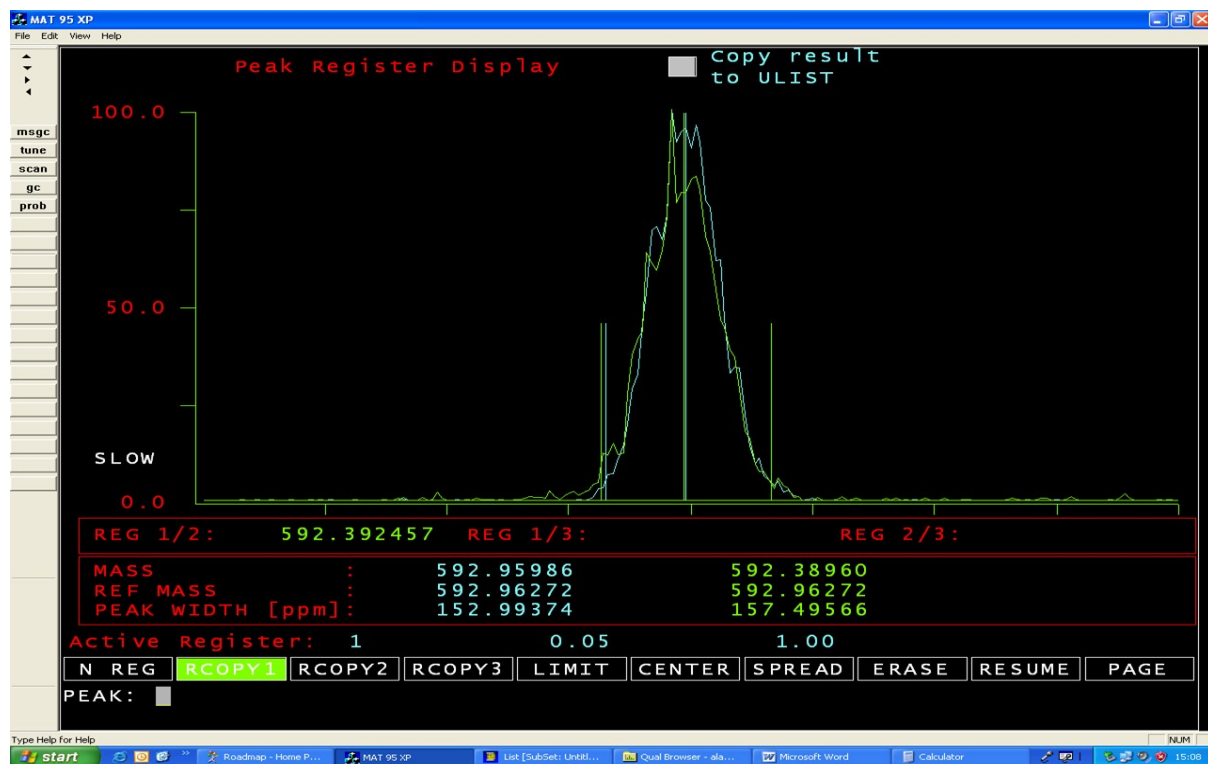
Figure S11. HRMS data for complex **4**.

Crystallographic and refinement details for complexes 5-7
Comparison of the molecular structures of 6 and 7

6

7
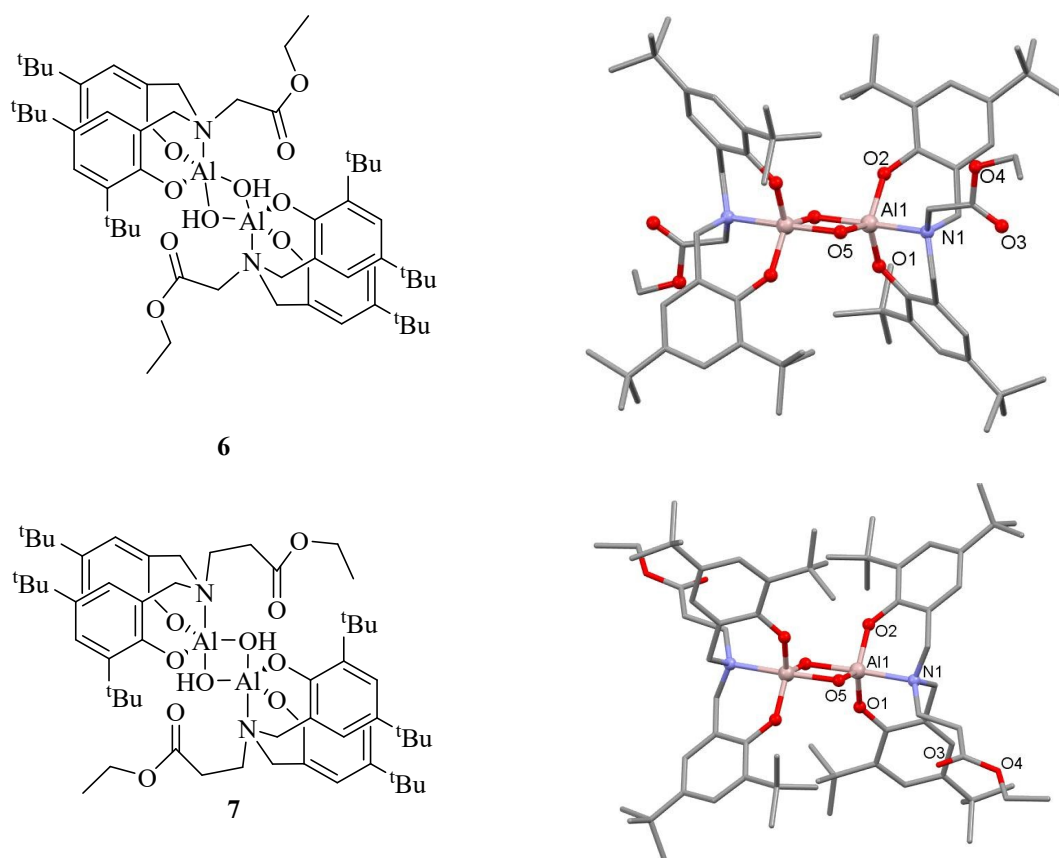
Figure S12. Top: Molecular structure of **6**. Ligand structure represented by capped sticks, Al, N, and O represented by ball and stick. Hydrogen atoms omitted for clarity. Structure grown from ASU. Selected bond lengths for 34 (Å): Al1-O1 1.744(2), Al1-O2 1.761(2), Al1-O5 1.792(2), Al1-O5' 1.879(2), Al1-N1 2.096(2), O3-C32 1.196(3), O4-C32 1.368(4). Selected bond angles(°): N1-Al1-O1 90.5(1), N1-Al1-O2 90.5(1), N1-Al1-O5 94.0(1), N1-Al1-O5' 134.2(1), Al1-O5-Al1' 101.9 (1). **Bottom:** Molecular structure of **7**. Ligand structure represented by capped sticks, Al, N, and O represented by ball and stick. Hydrogen atoms omitted for clarity. Structure grown from ASU. Selected bond lengths for 36 (Å): Al1-O1 1.744(2), Al1-O2 1.749(2), Al1-O5 1.781(2), Al1-O5' 1.895(2), Al1-N1 2.107(3), O3-C32 1.209(4), O4-C32 1.343(4). Selected bond lengths for 36 (°): O1-Al1-O2 119.9(1), O1-Al1-O5 119.3(1), O1-Al1-O5' 91.8(1), O1-Al1-N1 90.8(1).

Table S1. SCXRD data tables for complexes **5-7.**

Compound	5	6	7
Crystal growth method	Slow evaporation DCM/MeCN	Slow evaporation dry DCM/ dry MeCN	Slow evaporation dry DCM/ dry MeCN
Empirical formula	$C_{64}H_{96}Al_2N_2O_{10}$	$C_{86}H_{130}Al_2N_4O_{10}$	$C_{35}H_{52}AlNO_4$
Formula weight	1107.4	1433.9	577.75
Temperature/K	102	100	100
Crystal system	orthorhombic	monoclinic	triclinic
Space group	*Pba*2	*P*2$_1$/n	*P*-1
a/Å	22.5149(4)	10.866(5)	11.1245(7)
b/Å	34.1550(5)	31.717(15)	13.9569(8)
c/Å	8.83250(10)	13.243(6)	14.6201(9)
α/°	90	90	96.541(3)
β/°	90	110.155(18)	96.934(4)
γ/°	90	90	103.771(3)
Volume/Å^3	6792.16(17)	4284(3)	2164.5(2)
Z	4	2	4
ρ_{calc}g/cm^3	1.083	1.111	1.773
μ/mm^{-1}	0.803	0.746	1.255
F(000)	2400	1560	1256
Crystal size/mm^3	0.42 × 0.24 × 0.08	0.4 × 0.26 × 0.06	0.22 × 0.18 × 0.1
Radiation	CuKα (λ = 1.54178)	CuKα (λ = 1.54178)	CuKα (λ = 1.54178)
2Θ range for data collection/°	4.7 to 144.374	9.038 to 145.174	8.332 to 144.84
Index ranges	-27 ≤ h ≤ 27, -38 ≤ k ≤ 41, -10 ≤ l ≤ 10	-13 ≤ h ≤ 13, -38 ≤ k ≤ 39, -16 ≤ l ≤ 16	-13 ≤ h ≤ 13, -17 ≤ k ≤ 17, -18 ≤ l ≤ 17
Reflections collected	60639	65176	45098
Independent reflections	13113 [R_{int} = 0.0894, R_{sigma} = 0.0598]	8385 [R_{int} = 0.0690, R_{sigma} = 0.0411]	8467 [R_{int} = 0.0626, R_{sigma} = 0.0436]
Data/restraints/ parameters	13113/37/742	8385/29/522	8467/2/227
Goodness-of-fit on F^2	1.141	1.057	1.16
Final R indexes [I>=2σ (I)]	R_1 = 0.0931, wR_2 = 0.2582	R_1 = 0.0674, wR_2 = 0.2012	R_1 = 0.1044, wR_2 = 0.2897
Final R indexes [all data]	R_1 = 0.1104, wR_2 = 0.2791	R_1 = 0.0735, wR_2 = 0.2067	R_1 = 0.1198, wR_2 = 0.3066
Largest diff. peak/hole / e Å^{-3}	0.89/-0.35	0.86/-0.60	0.99/-1.10
Flack parameter	-0.01(7)	-	-

Polymerisation conditions

ROP of *rac*-lactide general conditions

Conditions including temperature, time and loading are stated in the tables; below is a generalised method. The catalyst loading was done per metal centres for each complex. Polymerisation were carried out in a Teflon sealed vial, placed into a custom aluminium heating.

Solution Phase

The catalyst ([M] = 0.01 M) and triply sublimed *rac*-lactide (0.288 g, 2 mmol) were placed in a vial with a Teflon seal inside a glovebox and dissolved in dry toluene (1mL). Anhydrous benzyl alcohol (1μL, 0.01 mmol) was added dropwise (if required), and the vial was sealed. The vial was removed from the glovebox and heated to 130 °C while stirring overnight. The reaction vessel was allowed to cool slightly, and an aliquot was taken to determine conversion via ^{1}H NMR spectroscopy. Ice cold acidified methanol was then added to the vial, the precipitated was collected and dried under vacuum. Samples were then analysed homonuclear decoupled ^{1}H NMR spectroscopy to determine the tacticity and GPC to determine the chain length and dispersity of the polymer formed.

Melt conditions

The catalyst ([M] = 0.01 M), and triply sublimed *rac*-lactide (0.432 g, 3 mmol) were place in a vial with a Teflon seal, in a glovebox. Anhydrous benzyl alcohol (1 μL, 0.01 mmol) was then added dropwise (if required) and the vial sealed and removed from the glove box. The vial was then heated to 130 °C once the monomer had melted, stirring and timing began. After the desired was time was over, the reaction vessel was allowed to cool to room temperature. The resulting white solid was dissolved in chloroform. An aliquot was then taken to determine the conversion via ^{1}H NMR spectroscopy. The volatiles were then removed via vacuum and the resulting in the formation of white solid. The solid was dissolved in toluene (1 mL), ice cold acidified methanol was added dropwise. The resulting white precipitated was collected and dried under vacuum. Samples were then analysed by homonuclear decoupled ^{1}H NMR spectroscopy to determine the tacticity and GPC to determine the chain length and dispersity of the polymer formed.

The activity of complexes **1-4** as catalysts for the ROP of *rac*-lactide under melt conditions were investigated by loading the complexes into Teflon sealed vials and heating to 130 °C (**Table S2**). The complexes all performed well reaching >80% conversion after six hours, with **2** reaching 94% in four hours (**Table S2**, entry 4). It is worth noting that the under the melt polymerisation conditions the lactide monomer was observed to sublime out of the reaction mixture, which in turn limits the potential conversion. The tacticity of the polymer produced was investigated and it was found that the highest observed isotactic bias was again using

complex **2** (**Table S2**, entry 6). Overall, the catalysts produced mostly atactic PLA with no significant enrichment. The dispersity values of the polymers produced were found to be excellent with values less than 1.10 recorded for all catalysts. The molecular weights again were found to be in good agreement with the calculated values for most cases where benzyl alcohol was not added (**Table S2** entries 8 and 10), indicating that complexes **3** and **4** have good control over the length of polymer formed. The lower chain lengths when benzyl alcohol is added is likely due to early termination of the polymer chains.

Table S2. Melt condition polymerisation data for complexes **1-4**

Entry	Cat.	Time (h)	Conversion (%)[b]	P_i^c	$M_{n(calc)}$ (g/mol)[d]	$M_{n(obs)}$ (g/mol)[e]	$Đ^e$
1[a]	1	4	63	0.57	13600	7700	1.06
2[a]	1	6	84	0.56	18200	10100	1.08
3	1	6	81	0.58	35100	-	-
4[a]	2	4	94	0.55	20300	-	-
5[a]	2	6	95	0.52	20600	10700	1.08
6	2	6	85	0.61	37000	8600	1.08
7[a]	3	6	84	0.50	18200	4508	1.08
8	3	4	76	0.51	32800	31100	1.08
9	3	6	79	0.51	34200	-	-
10	4	4	76	-	32700	26100	1.10
11[a]	4	6	90	0.55	19500	5329	1.07

Conditions [Al]:300[LA], [Al]= 0.01 M, 130 °C. [a] [M]:2[BnOH]. [b] Calculated from ^1H NMR spectrum, analysis of the integration of the lactide and poly(lactic acid) resonances in the methylene region. [c] Determined using homonuclear decoupled ^1H NMR spectroscopy. [d] Calculated as $M_{n(calc)} = ([LA]/[M]) \times conversion \times Mw_{LA}$ or $M_{n(calc)} = ([LA]/[M]/[BnOH]) \times conversion \times Mw_{LA}$ for reactions initiating with BnOH. [e] Determined from GPC trace at 35 °C in THF, using polystyrene standards.

ROCoP of CHO and CO₂

Cyclohexene oxide (CHO) was dried overnight over CaH_2, then distilled by means of vacuum distillation. Polymerisation reactions were performed in a Parr autoclave which was equipped with an injection inlet, allowing the autoclave to be set up outside the glovebox, and a magnetic stirrer bar. In the glovebox a Schlenk vessel was charged with a stirrer bar, the required amount of catalyst and PPNCl to generate poly carbonate, or without to produce poly ether. Then using a Schlenk line, 2 mL (2000

equivalents) cyclohexene oxide was injected into the Schlenk, and the solution was stirred until all powder had gone into solution. This solution was then injected through an injection port under a constant stream of nitrogen into a 450 mL stainless steel autoclave. This system was sealed, and the autoclave was charged with the required bar of CO_2. The autoclave was then placed into a tightly fitted aluminium heating block fitted with thermocouple. After heating for the required time, the CO_2 was expelled from the autoclave via an exhaust pipe. A crude sample of the polymer was dissolved in $CDCl_3$ and analysed on a 400 MHz NMR spectrometer. Methanol or acidified methanol was added to the polymer solution to precipitate the polymer. The polymer was filtered, and solvent was removed under high vacuum (2.0×10^{-3} mbar).

Autoclave reactor for the ROCoP of CHO and CO₂

Figure S13. Stainless steel autoclave set-up for ROCoP of CHO/CO₂, adapted for use with Schlenk line.

Table S3. The results of how time affected the conversion with complex **4**. [3.5]: [3.5]: [2000], cat: PPNCl: CHO.

| Entry	Complex	Time (hours)	Pressure (bar)	Conv. (%)^a	% CO$_3$^{b}	TON/TOF^c
1	4	2	20	25	92	147/74
2	4	4	20	34	95	200/50
3	4	8	20	43	93	253/32
4	4	15	20	55	97	324/22
5	4	24	20	56	97	329/14
6	4	48	20	60	96	353/7

Results of polymerisations carried out in neat cyclohexene oxide (2 mL), in presence of initiator (PPNCl), ratio 3.5[Al]: 3.5[PPNCl] :2000 [CHO], [cat.]: [initiator]: [monomer]. Conditions 75°C.
^{a} *Calculated from ^1H NMR spectrum.* ^{b} *% CO$_3$ includes cyclic carbonate and poly carbonate.*
^{c} *Calculated as conversion x (monomer/cat ratio) /time, or TON / time.*

**Table S4.** The impact of using toluene as the solvent on the RoCOP of CO$_2$ and CHO.

Entry	% CO$_3$	Conversion (%)	M_w/M_n (g/mol)	PDI
1*	66	37	4531/ 2909	1.56
2**	94	51	2803/ 1949	1.40

Values obtained using GPC equipped with a triple detection. *2.2 mL toluene; ** no solvent added (75°C, 24 hours, 20 bar CO$_2$, **4**).
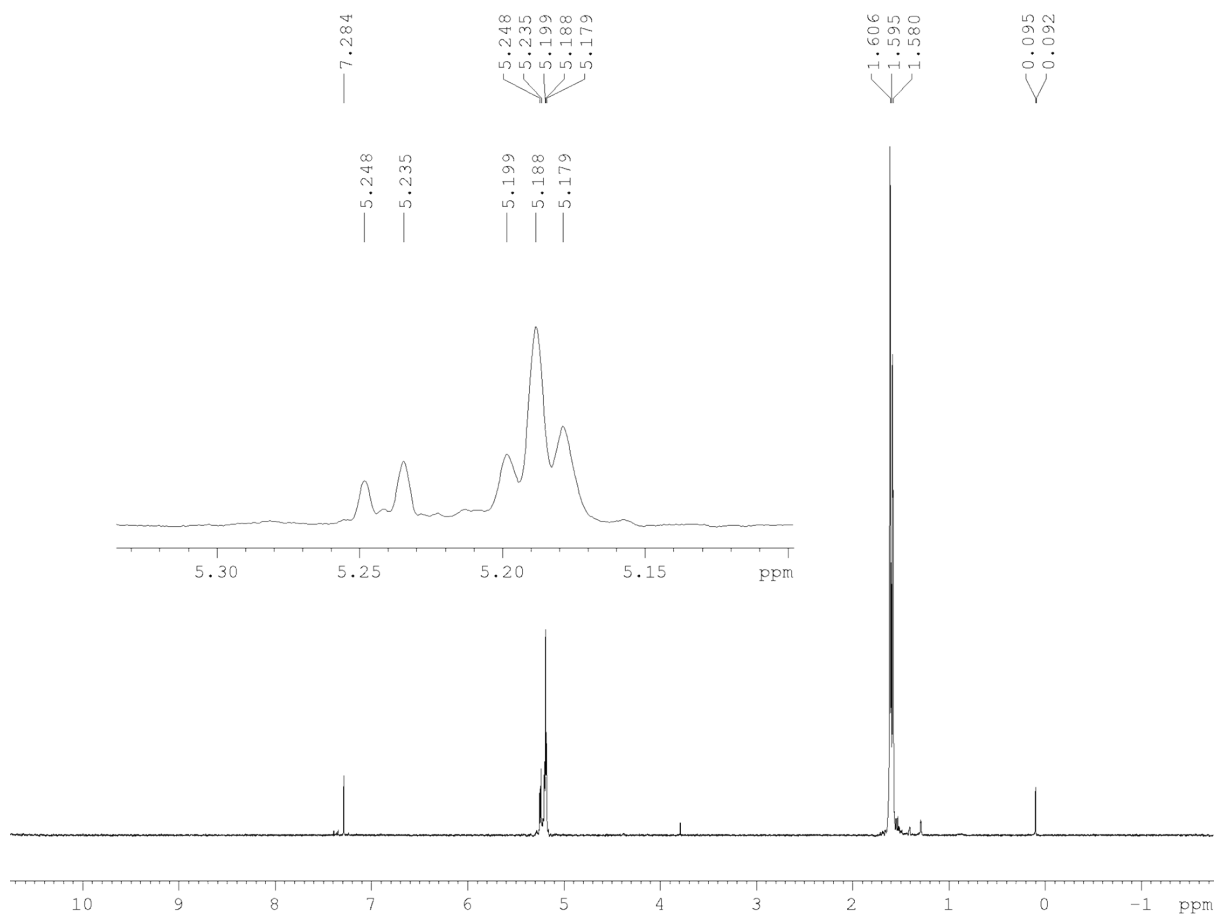
Polymer analysis

Sample polymer NMR spectra
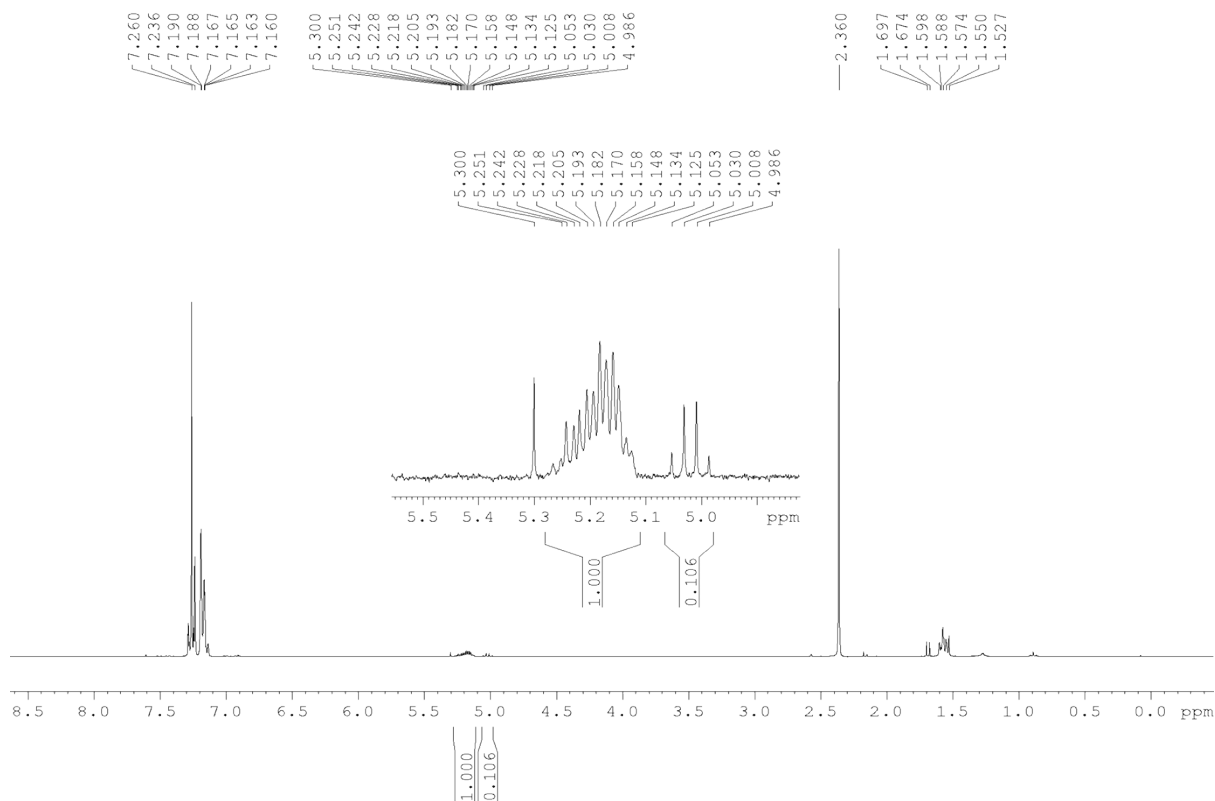
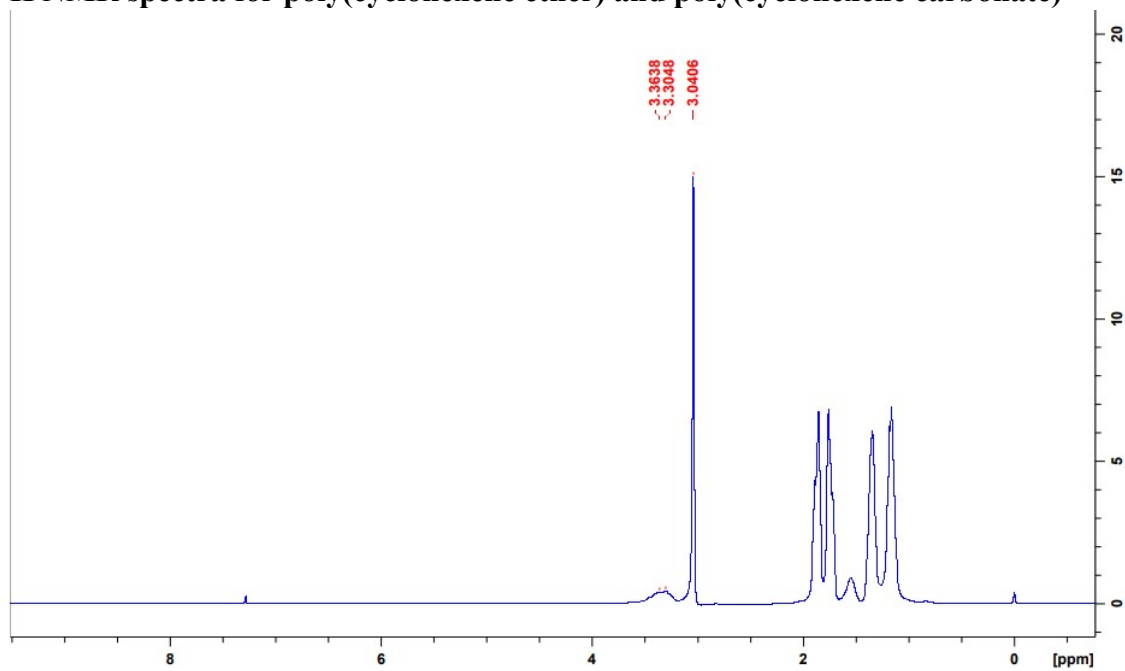**Figure S14**. ^{1}H NMR spectrum of crude poly(lactic acid) polymerisation result (300 MHz, CDCl$_3$, 25 °C).
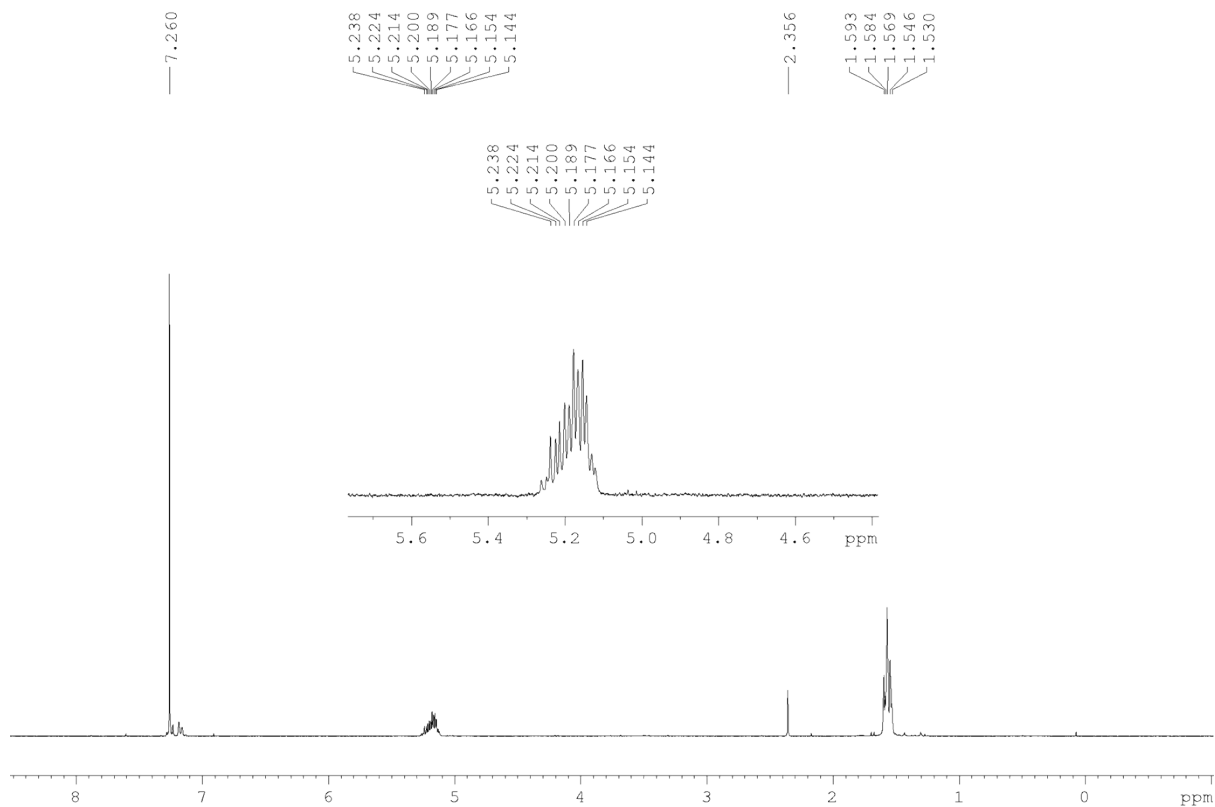
Figure S15. Sample ^{1}H NMR spectrum of purified poly(lactic acid) (300 MHz, CDCl$_3$, 25 °C).
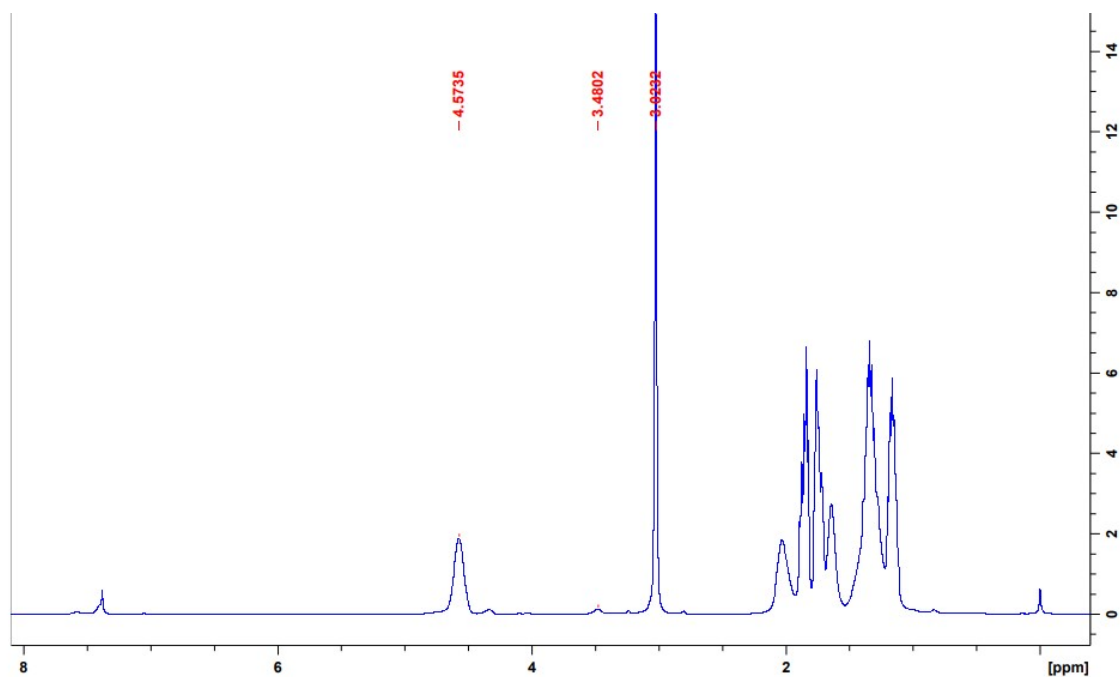
Figure S16. Sample homonuclear decoupled ^{1}H NMR spectrum of poly(lactic acid) (800 MHz, CDCl$_3$, 25 °C).

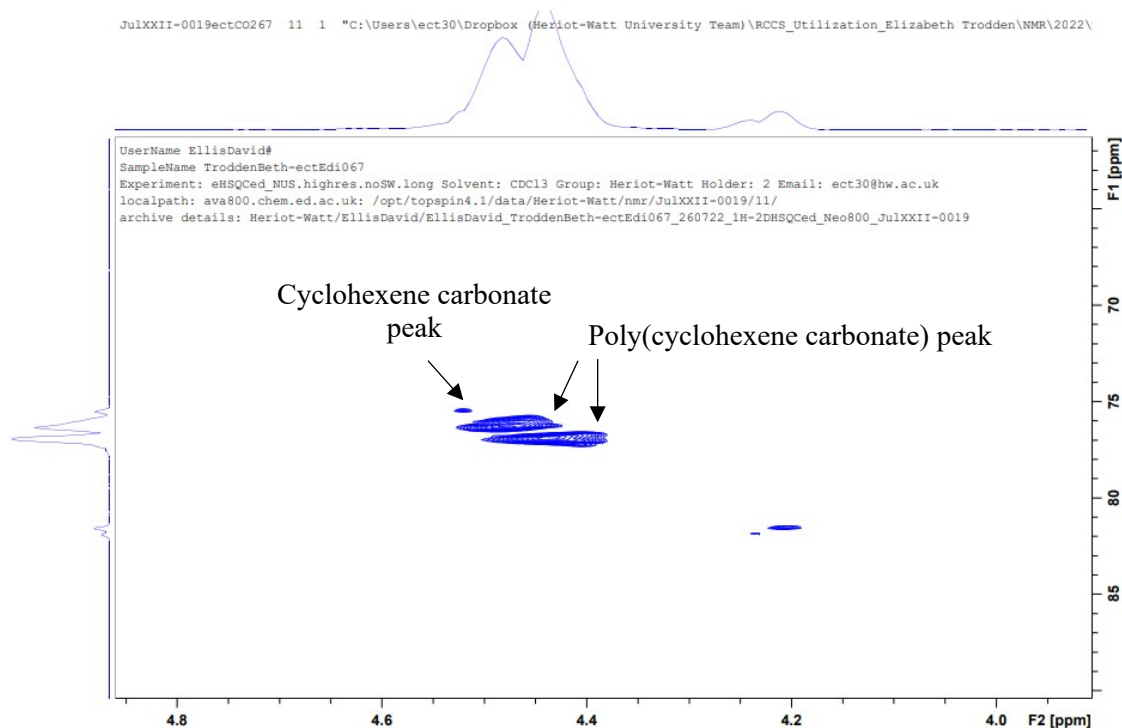**¹H NMR spectra for poly(cyclohexene ether) and poly(cyclohexene carbonate)**

**Figure S17.** *¹H NMR spectrum. Conversion of CHO to polyether.*

Figure S18. *¹H NMR spectrum. Conversion of CHO/CO₂ to polycarbonate and cyclic carbonate.*

HSQC experiment with poly(cyclohexene carbonate)

Figure S19. Expanded view of the 100 °C polymer sample run on an 800 MHz spectrometer (800 MHz, CDCl$_3$, 25 °C). Data collected with assistance of Dr. Juraj Bella. Polymeric and monomeric carbonate species are identified where the broad peak at 4.57 ppm (S18) is found to consist of overlapping signals corresponding to *trans*-cyclo hexene oxide and poly(cyclohexene ether).

¹³C{¹H} NMR of poly(cyclohexene carbonate)
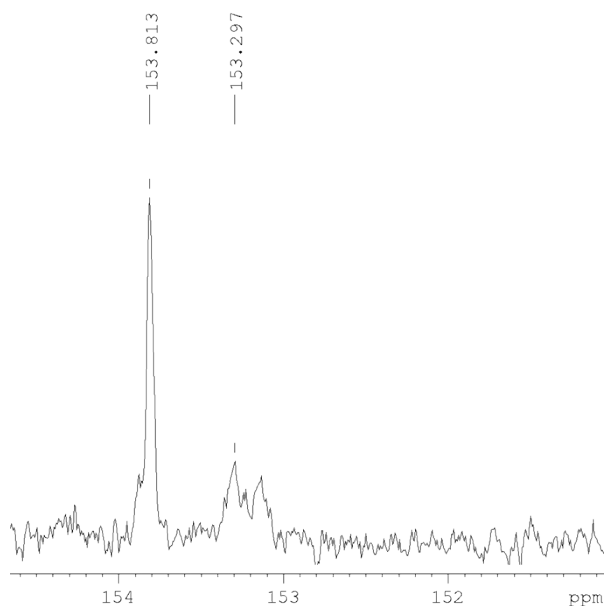
Figure S20. Carbonyl region of the ¹³C NMR spectrum of poly(cyclohexene carbonate). Signals at 153.81 ppm and 153.29 ppm have been assigned as the isotactic and syndiotactic regions, respectively, based upon the findings of Losio and Boggioni.[9]

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