Electronic Supplementary Information

For

Ortho-Vanillin Derived Al(III) and Co(III) Catalyst Systems for Switchable Catalysis using ε-Decalactone, Phthalic Anhydride and Cyclohexene Oxide

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Contents

General Procedures

Materials

Triethyl aluminium (93%) and cobalt (II) acetate tetrahydrate were purchased from Sigma-Aldrich. Both were used as received. Solvents used for air-sensitive synthesis were collected from a solvent purification system (SPS), degassed with freeze-pump-thaw cycles and stored over 3 Å molecular sieves under an inert atmosphere.

Cyclohexene oxide (Acros Organics) was purified according to the method reported by Greiner *et al*.[1] Sodium hydride was added to cyclohexene oxide under an inert atmosphere, followed by addition of methyl iodide (the sodium hydride acts as both a drying agent, and deprotonates any residual diols present in the epoxide, which are subsequently methylated upon methyl iodide addition). The cyclohexene oxide was subsequently isolated *via* fractional distillation under reduced pressure. Purification of phthalic anhydride (Sigma Aldrich) was achieved though stirring in dry toluene. The supernatant was filtered, and the toluene subsequently removed *in vacuo*. The resultant white powder recrystallised from hot (60 °C) chloroform and subsequently sublimed under vacuum at 80 °C. ε-Decalactone (Sigma Aldrich) was dried over CaH_2, followed by fractional distillation at 130 °C under reduced pressure. 1,2-Cyclohexanediol (Sigma Aldrich) was recrystallised from dry ethyl acetate. All materials were stored under a nitrogen atmosphere, in a glovebox. The proligand, $L_{van}H_2$, was synthesised through the Schiff base condensation reaction of *o*-vanillin with 2,2-dimethyl-1,3-propanediamine.[2]

Methods

NMR Spectroscopy: ^{1}H NMR, ^{27}Al and ^{31}P{^{1}H} spectra were obtained using a Bruker Avance III HD 400 NMR spectrometer. ^{13}C{^{1}H} NMR were obtained using a Bruker AV III HD 500 MHz NMR spectrometer.

GPC analysis: Carried out using a Shimadzu LC-20AD instrument, equipped with a Refractive Index (RI) detector and two PSS SDV 5 μm linear M columns. The eluent used was HPLC-grade THF, heated to 30 °C, and with a flow rate of 1.0 mL min^{-1}.

MALDI-ToF spectrometry: Carried out on a Bruker Autoflex Speed MALDI-ToF. Samples were prepared by preparing a solution of polymer (10 mg mL^{-1} in THF), dithranol (10 mg mL^{-1} in THF) and KTFA (10 mg mL^{-1} in THF) in a 1:4:1 ratio, which was subsequently spotted onto a plate and allowed to completely dry before analysis.

Elemental analysis was carried out by Mr Stephen Boyer at London Metropolitan University.

DSC: Analysis of triblock copolymers was carried out using a DSC3+ (Mettler-Toledo, Ltd). The triblock polyesters were heated to 200 °C for 5 minutes to remove thermal history, before heating and cooling from -80 to +200 °C at a rate of 10 °C min^{-1}. Each sample was run for three heating-cooling cycles. Glass transition temperatures (T_g) were recorded from the midpoint of the transition during the third heating curve.

X-Ray Crystallography: Crystallographic data were collected, and structures solved by Dr Andreas Phanopoulos, Dr Ryan Kerr, and Dr Christopher Durr. Air sensitive crystalline samples were isolated in a nitrogen filled glovebox and immersed in fluorinated oil. Crystalline samples were mounted on a MiTeGen Micromount, and cooled to 150 K with dry nitrogen using an Oxford Cryosystem Cryostream.[3] Data was collected using an Oxford Diffraction Supernova

diffractometer using Cu K_α (λ = 1.5417 Å) or Mo K_α (λ = 0.7107 Å) radiation. The resulting reflection data was processed with CrysAlis Pro.[4] The crystal structure of complex **1** was solved using SHELXT and structure refinement was conducted with SHELXL-14 within the WinGX software package.[5,6] The crystal structures of complexes **2** and **3** were solved using the SHELXT program and least-square refined using the SHELXL program within the Olex2 system suite.[7-9]

Synthesis of Catalysts

L_{van}AlEt (1)

Under an inert atmosphere, $AlEt_3$ (0.17 g, 1.5 mmol) was added dropwise to a stirring solution of $L_{van}H_2$ (0.57 g, 1.5 mmol) in anhydrous toluene (10 mL) at -30 °C. The reaction mixture was allowed to warm to room temperature, and then stirred for 1 hour. A yellow powder precipitated from the reaction mixture, which was isolated *via* centrifuge, washed with toluene (3 x 10 mL) and hexane (1 x 10 mL) and subsequently dried under vacuum to give **1** as a yellow powder (0.39 g, 1.02 mmol, 68%). X-ray Diffraction quality crystals were grown from a saturated solution of **1** in THF, layered with hexane.

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K); δ 8.07 (2H, s, H^e), 6.92 (2H, dd, *J* = 7.7 , 1.6 Hz, H^b), 6.80 (2H, dd, *J* = 7.7, 1.6 Hz, H^d), 6.60 (2H, t, *J* = 7.7 Hz, H^c), 3.96 (2H, d, *J* = 12.0 Hz, H^f), 3.85 (6H, s, H^a) 3.22 (2H, d, *J* = 12.0 Hz, H^g), 1.14 (3H, s, H^h), 0.95 (3H, s, H^i), 0.88 (3H, t, *J* = 8.0 Hz), -0.01 (2H, q, *J* = 8.0 Hz) ppm. **^{13}C NMR** ($CDCl_3$, 126 MHz, 298 K); δ 169.0 (C^h), 157.7 (C^g), 151.8 (C^b), 124.8 (C^e), 119.0 (C^f), 117.9 (C^c), 115.6 (C^d), 71.0 (C^i), 56.9 (C^a), 36.1 (C^j), 26.0 (C^k), 23.20 (C^l), 9.97 (C^n), 1.2 (C^m) ppm. **^{27}Al NMR** ($CDCl_3$, 104 MHz, 298 K): 5.8 ppm. **Anal. Calc.** ($C_{23}H_{29}AlN_2O_4$); C, 65.08; H, 6.89; N, 6.60. Found: C, 64.95; H, 6.69; N, 6.56.

L_{van}Co(OAc) (2)

Under an inert atmosphere, $[Co(OAc)_2.4H_2O]$ (0.45 g, 1.8 mmol) was added to a stirred solution of $L_{van}H_2$ (0.67 g, 1.8 mmol) in anhydrous THF (10 mL) at 25 °C, resulting in a rapid colour change from yellow to brown. The solution was stirred under an inert atmosphere for 2 hours before being exposed to air and stirred at room temperature for 48 hours, resulting in precipitation of a green powder. The precipitate was isolated *via* centrifuge and was subsequently washed sequentially with THF (3 x 10 mL) and hexane (3 x 10 mL) before being dried under vacuum to give **2** as a green powder (0.54 g, 1.26 mmol, 70%). X-ray Diffraction quality crystals were grown from a saturated solution of **1** in DCM, layered with hexane.

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K); δ 7.52f (1H, s, H^e), 7.18 (1H, s, $H^{e'}$), 6.92-6.87f (3H, m, H^d, $H^{d'}$, H^b), 6.56-6.55 (2H, m, H^c and $H^{b'}$), 6.47 (1H, m, $H^{c'}$), 4.63 (1H, d, ^2J = 10.5 Hz, H^f), 3.88 (s, 3H, H^a), 3.56 (s, 3H, $H^{a'}$), 3.40 (1H, d, ^2J = 10.5 Hz, H^g), 3.28 (1H, d, ^2J = 13.0 Hz $H^{f'}$), 2.87 (1H, d, ^2J = 13.0 Hz, $H^{g'}$), 1.85 (3H, s, H^j), 1.17 (3H, s, H^h), 0.92 (3H, s, H^i) ppm. **^{13}C NMR** ($CDCl_3$, 126 MHz, 298 K); δ 193.5 (C^m), 168.4 (C^h), 166.8 ($C^{h'}$), 160.4 (C^f), 157.9 ($C^{f'}$), 155.3 (C^b), 154.4 ($C^{b'}$), 125.8 (C^e), 124.7 (C^g), 124.3 ($C^{e'}$), 122.2 ($C^{g'}$), 117.7 (C^d), 117.1 ($C^{d'}$), 115.7 (C^c), 114.9 ($C^{c'}$), 70.5 ($C^{i+i'}$), 57.4 (C^a), 56.7 ($C^{a'}$), 35.4 (C^j), 25.9 (C^k), 23.8 (C^n), 23.3 (C^l) ppm. **Anal. Calc.** ($C_{23}H_{29}AlN_2O_4$); C, 56.79; H, 5.60; N, 5.76. Found: C, 56.40; H, 5.85; N, 5.59.

L_{van}AlOAc (3)

Under an inert atmosphere, glacial AcOH (0.01 mL, 0.19 mmol) was added to a stirred solution of **1** (80 mg, 0.19 mmol) in anhydrous THF (10 mL) at 25 °C. The reaction mixture was stirred for 2 hours, resulting in the precipitation of a yellow powder. The precipitate was isolated *via* centrifuge, before being dried under vacuum to yield **3** as a pale yellow powder (70 mg, 0.15 mmol, 79%). X-ray Diffraction quality crystals were grown from a saturated solution of **1** in $CHCl_3$, layered with hexane.

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K); δ 8.10 (2H, s, H^e), 6.92 (2H, dd, *J* = 7.7, 1.6 Hz, H^b), 6.87, (2H, dd, *J* = 7.7, 1.6 Hz, H^d), 6.62 (2H, t, *J* = 7.7 Hz, H^c), 3.87 (2H, d, *J* = 11.9 Hz, H^f), 3.80 (6H, s, H^a), 3.28 (2H, d, *J* = 11.9 Hz, H^g), 2.03 (3H, s, H^j), 1.14 (3H, s, H^h), 0.93 (3H, s, H^i) ppm. **^{13}C NMR** ($CDCl_3$, 126 MHz, 298 K); δ 181.1 (C^m), 169.4 (C^h), 158.1 (C^g), 152.0 (C^b), 125.3 (C^e), 119.6 (C^f), 118.0 (C^c), 115.8 (C^d), 69.8 (C^i), 56.8 (C^a), 36.8 (C^j), 25.9 (C^l), 24.7 (C^k), 20.5 (C^n) ppm. **^{27}Al NMR** ($CDCl_3$, 104 MHz, 298 K): 14.8 ppm. **Anal. Calc.** ($C_{23}H_{29}AlN_2O_4$); C, 60.79 H, 5.99; N, 6.16. Found: C, 60.77; H, 6.12; N, 6.05.

Crystallographic Data for Complexes 1, 2 and 3

Table S1. Selected bond lengths and angles for complex **1**.

Bond	Bond Length (Å)	Bond	Bond Angle (°)
N (1) – Al (1)	2.083(2)	N (1) – Al (1) – N (2)	84.47(10)
N (2) – Al (1)	2.020(3)	N (1) – Al (1) – O (2)	87.92(10)
O (2) – Al (1)	1.801(2)	N (1) – Al (1) – O (3)	167.80(10)
O (3) – Al (1)	1.849(2)	N (1) – Al (1) – C (22)	91.45(12)
C (22) – Al (1)	1.983(3)	N (2) – Al (1) – O (2)	119.25(11)
		N (2) – Al (1) – O (3)	86.91(10)
		N (2) – Al (1) – C (22)	119.89(13)
		O (2) – Al (1) – O (3)	88.78(9)
		O (2) – Al (1) – C (22)	120.48(14)
		O (3) – Al (1) – C (22)	100.33(12)

Table S2. Selected bond lengths and angles for complex **2**.

Bond	Bond Length (Å)	Bond	Bond Angle (°)
N (1) – Co (1)	1.9119(13)	N (1) – Co (1) – N (2)	93.09(6)
N (2) – Co (1)	1.9159(14)	N (1) – Co (1) – O (2)	92.84(5)
O (2) – Co (1)	1.8904(12)	N (1) – Co (1) – O (3)	95.12(5)
O (3) – Co (1)	1.8812(12)	N (1) – Co (1) – O (5)	163.39(5)
O (5) – Co (1)	1.9849(11)	N (1) – Co (1) – O (6)	96.80(5)
O (6) – Co (1)	1.9529(11)	N (2) – Co (1) – O (2)	173.83(5)
		N (2) – Co (1) – O (3)	90.14(5)
		N (2) – Co (1) – O (5)	89.43(5)
		N (2) – Co (1) – O (6)	91.26(5)
		O (2) – Co (1) – O (3)	87.65(5)
		O (2) – Co (1) – O (5)	85.37(5)
		O (2) – Co (1) – O (6)	89.72(5)
		O (3) – Co (1) – O (5)	101.29(5)
		O (3) – Co (1) – O (6)	167.90(5)
		O (5) – Co (1) – O (6)	66.71(5)
		O (5) – C (22) – O (6)	116.65(14)

Table S3. Selected bond lengths and angles for complex **3**.

Bond	Bond Length (Å)	Bond	Bond Angle (°)
N (1) – Al (1)	1.9977(9)	N (1) – Al (1) – N (2)	89.93(4)
N (2) – Al (1)	2.0127(9)	N (1) – Al (1) – O (2)	90.81(3)
O (2) – Al (1)	1.8240(8)	N (1) – Al (1) – O (3)	103.42(4)
O (3) – Al (1)	1.8162(8)	N (1) – Al (1) – O (5)	157.05(4)
O (5) – Al (1)	1.9935(8)	N (1) – Al (1) – O (6)	91.35(3)
O (6) – Al (1)	1.9910(8)	N (2) – Al (1) – O (2)	178.63(4)
		N (2) – Al (1) – O (3)	87.67(4)
		N (2) – Al (1) – O (5)	89.94(4)
		N (2) – Al (1) – O (6)	88.35(4)
		O (2) – Al (1) – O (3)	91.03(4)
		O (2) – Al (1) – O (5)	89.83(3)
		O (2) – Al (1) – O (6)	92.79(3)

Table S4. Summary of Crystallographic Refinement Data for complexes **1**, **2** and **3**.

	Complex **1**	Complex **2**	Complex **3**
Local Code	062ckw17	027ap19	008rwfk20
Chemical Formula	$C_{23}H_{29}AlN_2O_4$	$C_{23}H_{27}CoN_2O_6$	$C_{23}H_{27}AlN_2O_6$
Formula Weight	424.46	486.39	454.44
Temperature (K)	150(2)	150.00(16)	150.01(15)
Space Group	Orthorhombic, $P2_12_12_1$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
a (Å)	7.86900(10)	8.99960(10)	8.99580(10)
b (Å)	15.7511(2)	12.8364(2)	12.88180(10)
c (Å)	17.6113(3)	19.0472(3)	19.2304(2)
α (°)	90	90	90
β (°)	90	96.7450(10)	96.6150(10)
γ (°)	90	90	90
V (Å^3)	2182.84(5)	2185.15(5)	2213.62(4)
Z	4	4	4
D_{calcd} (Mg/m^3)	1.292	1.478	1.364
Crystal Size (mm)	0.17*0.17*0.24	0.19*0.22*0.39	0.11*0.26*0.29
Theta range for data collection	3.765 to 75.942	4.1230 to 76.0760	4.0910 to 76.0720
μ (mm^{-1})	1.08	6.53	1.17
Reflections collected	9972	12050	23912
Unique reflections	4237	4529	4605
Data completeness to [θ]	74.33	74.33	74.33
R_{int}	0.034	0.031	0.028
Final R1 [I > 2σ(I)]	0.031	0.034	0.036
Final wR2 [I > 2σ(I)]	0.0957	0.0807	0.0812
Goodness-of-fit on F^2	1.039	1.072	1.070
R1 values (all data)	0.0391	0.0375	0.0344

wR2 values (all data)	0.0980	0.0830	0.083

Representative Polymerization

Inside a nitrogen filled glovebox, the catalyst, co-catalyst, chain transfer agent and desired monomers were added to a dried vial equipped with a magnetic stirrer bar. The vial was sealed with a melamine-cap containing a Teflon inlay, and further sealed with first Parafilm M and then electrical insulation tape. This sealed vial was then heated to 100 °C for the time stated. Aliquoting was performed inside the glovebox by removing *ca*. 10 μL of the polymerisation mixture with a syringe. The polymerisations were quenched by exposing the reaction mixture to air, followed by removal of volatiles. A ^{1}H NMR spectrum of the crude product was measured in $CDCl_3$. GPC samples were prepared by dissolving approx. 10 mg of the crude product in 1 mL HPLC grade THF and filtered before use.

End Group Analysis by $^{31}P\{^{1}H\}$ NMR Spectroscopy

Analysis of the hydroxyl polymer end groups was carried out following a reported procedure.[10] In an NMR tube, the triblock polyester (40 mg), purified *via* precipitation from methanol, was added to of a stock solution (40 μL of a solution make from pyridine (10 mL), bisphenol A (400 mg) and $Cr(acac)_3$ (5.5 mg) and 2-chloro-4,4,5,5-tetramethyldioxaphospholane (40 mg), in $CDCl_3$ (0.5 mL). The mixture was allowed to react at room temperature for 6 hours before $^{31}P\{^{1}H\}$ NMR spectra were recorded.

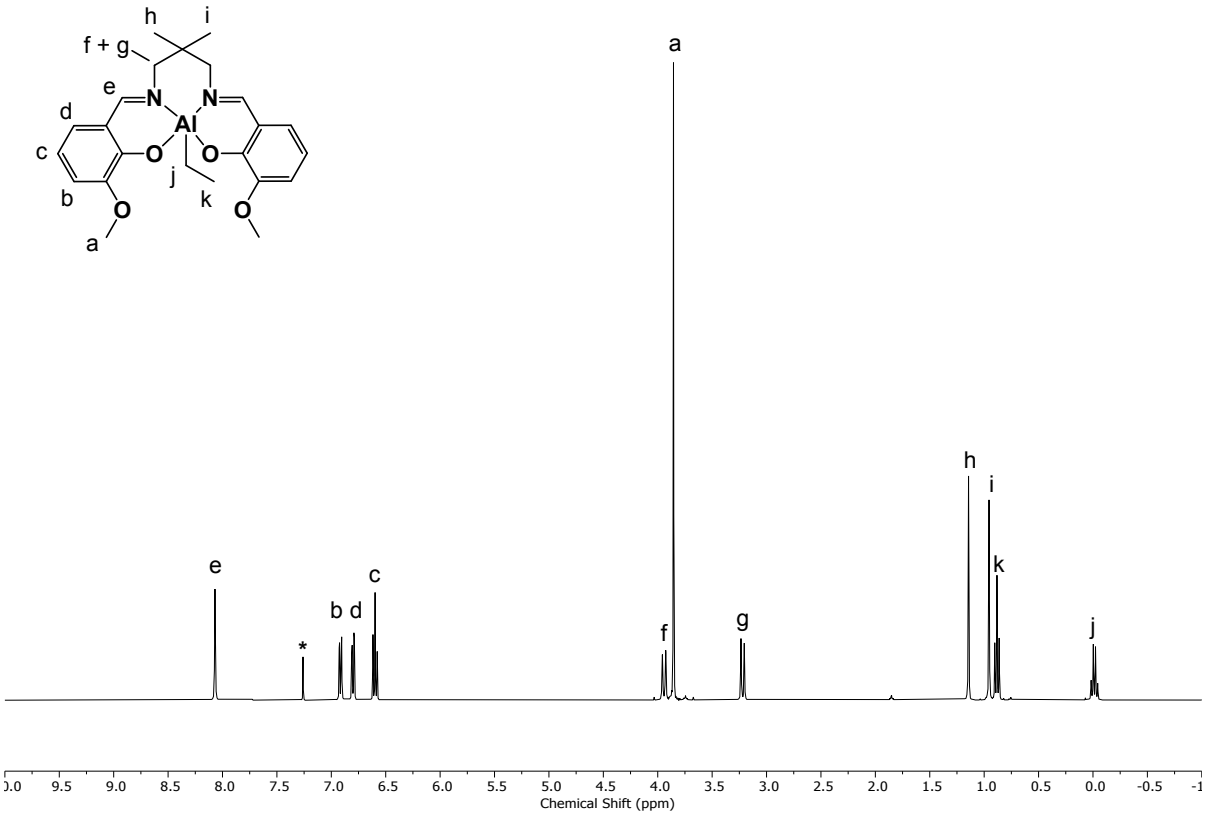

Figure S1. ^{1}H NMR Spectrum (400 MHz, 298 K, $CDCl_3$ (*)) of complex **1**.

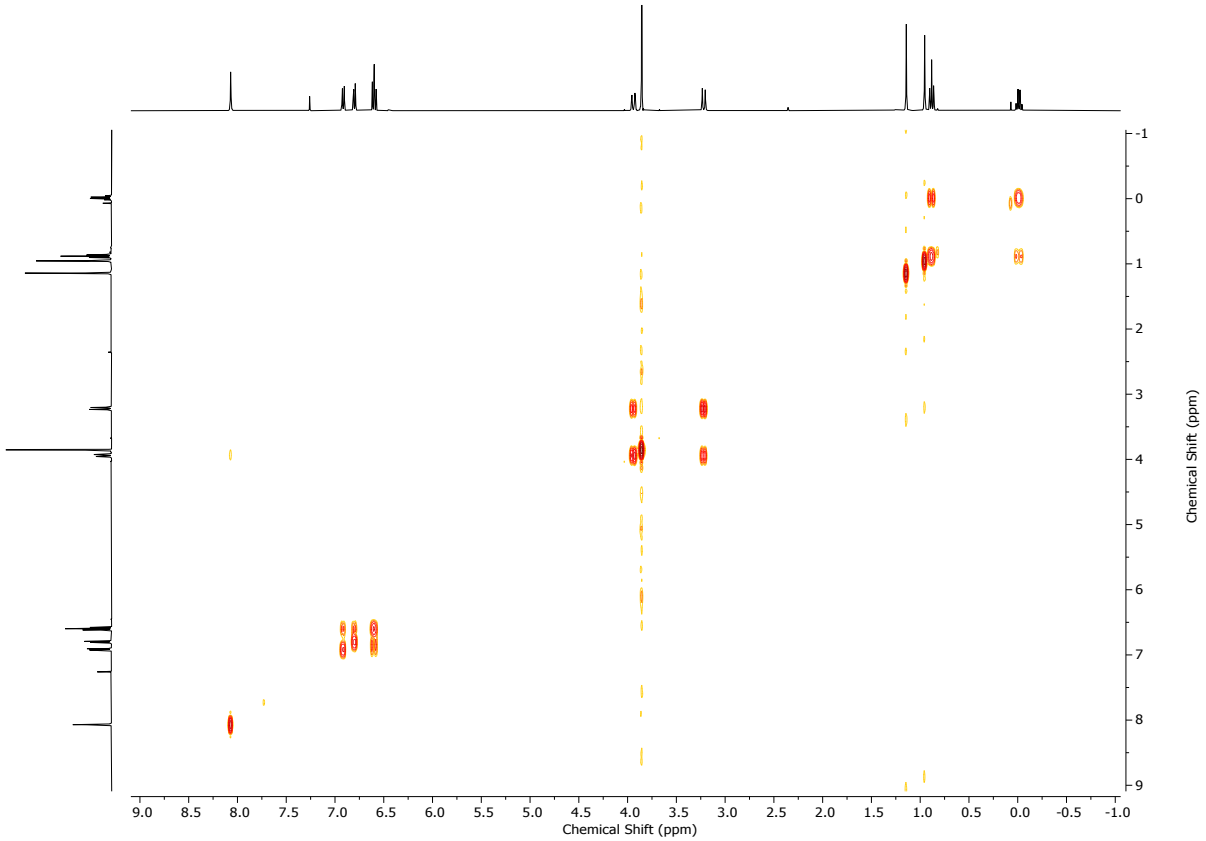

Figure S2. ^{1}H COSY NMR Spectrum (400 MHz, 298 K, $CDCl_3$) of complex **1**.

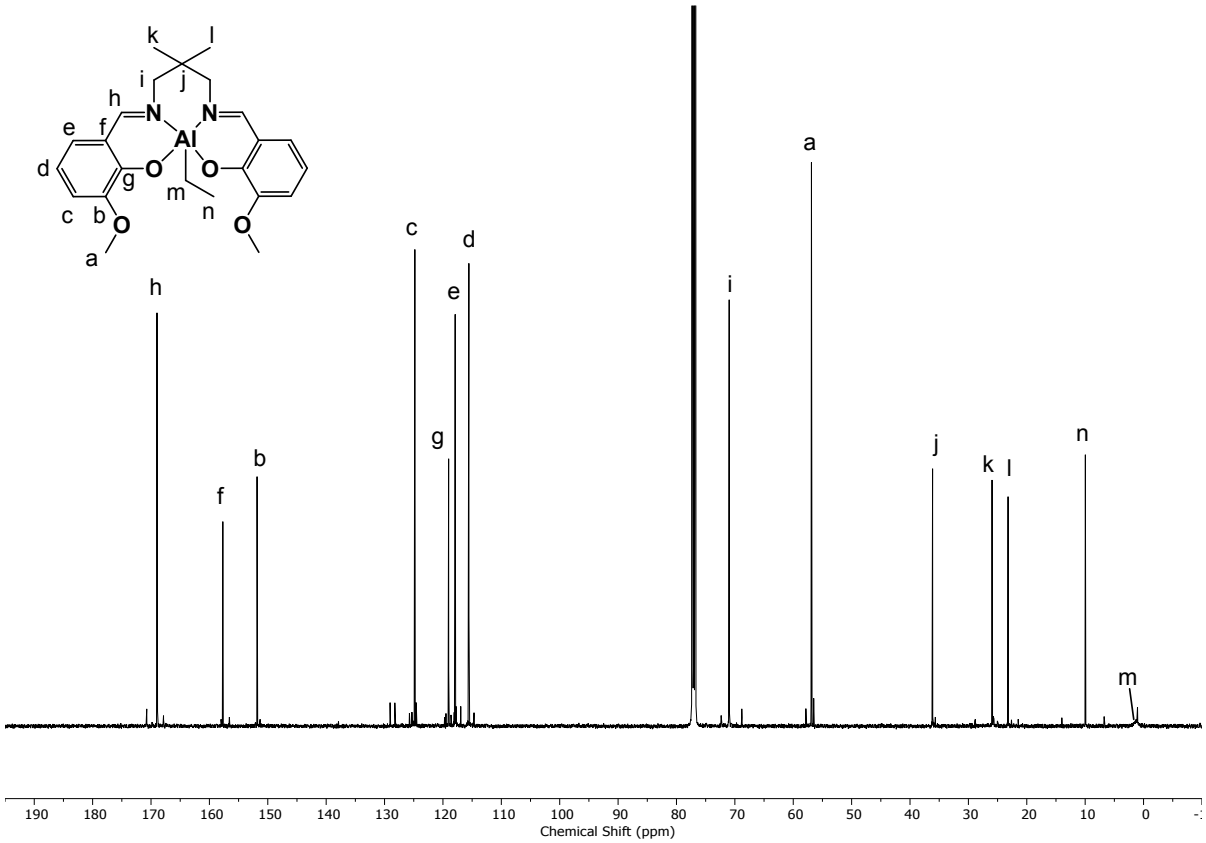

Figure S3. $^{13}C\{^{1}H\}$ NMR Spectrum (500 MHz, 298 K, $CDCl_3$(*)) of complex **1**. (*N.b.* Carbon M is broadened due to coupling to the quadrupolar Al nucleus).

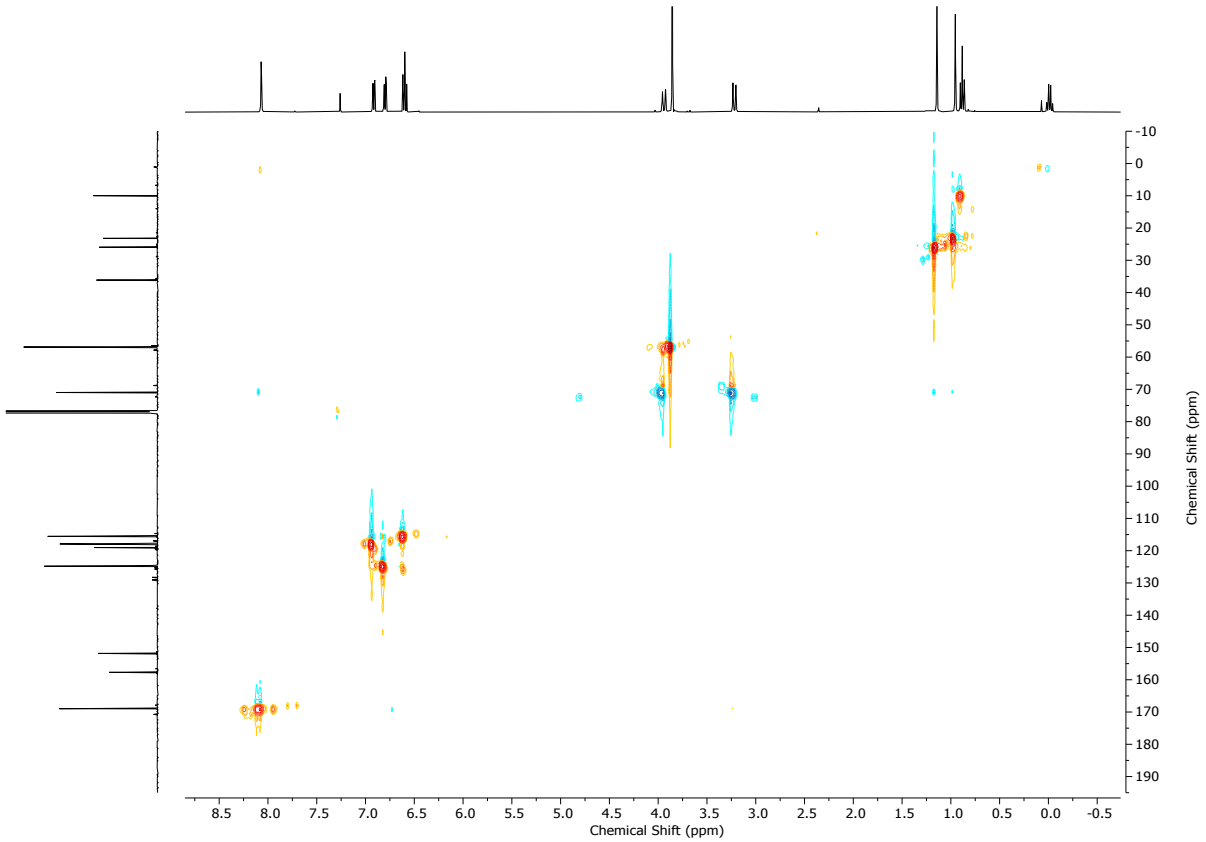

Figure S4. ^{1}H-^{13}C HSQC NMR Spectrum (298 K, $CDCl_3$) of complex **1**.

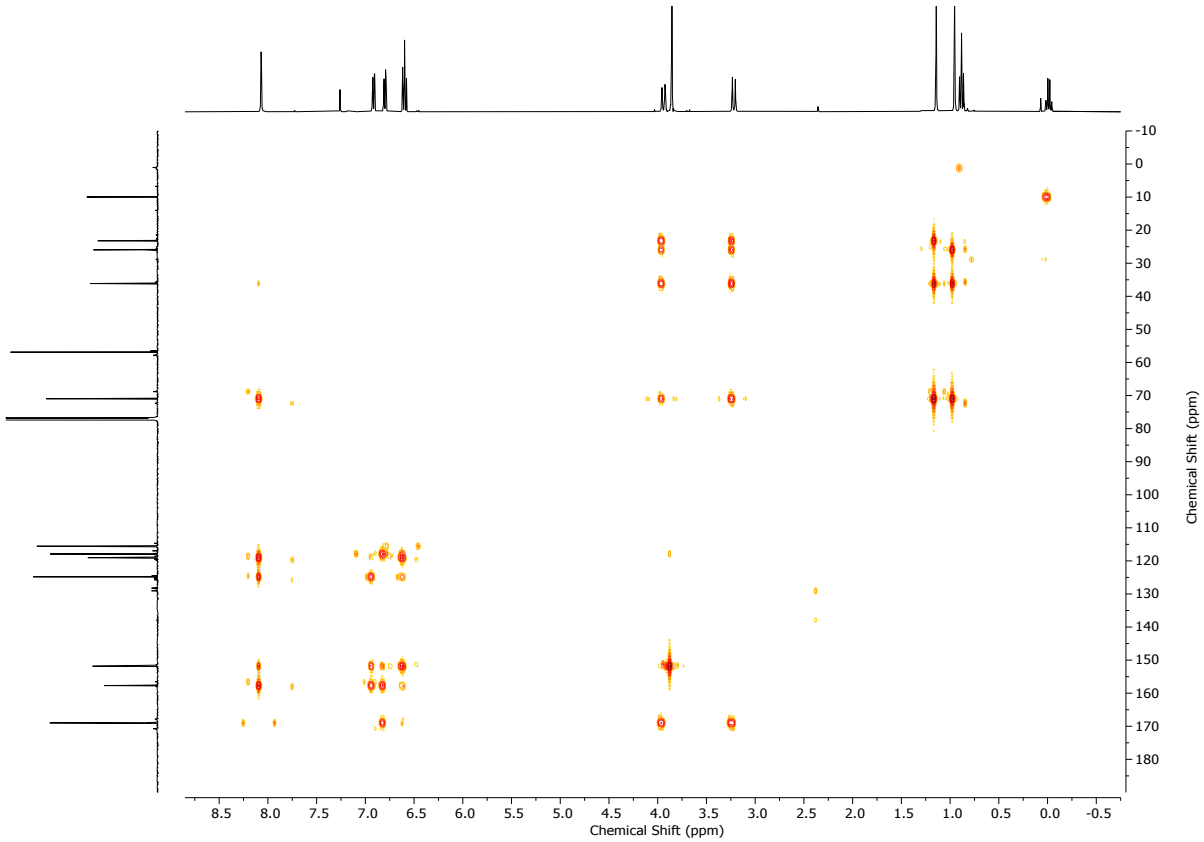

Figure S5. ^{1}H-^{13}C HMBC NMR Spectrum (298 K, $CDCl_3$) of complex **1**.

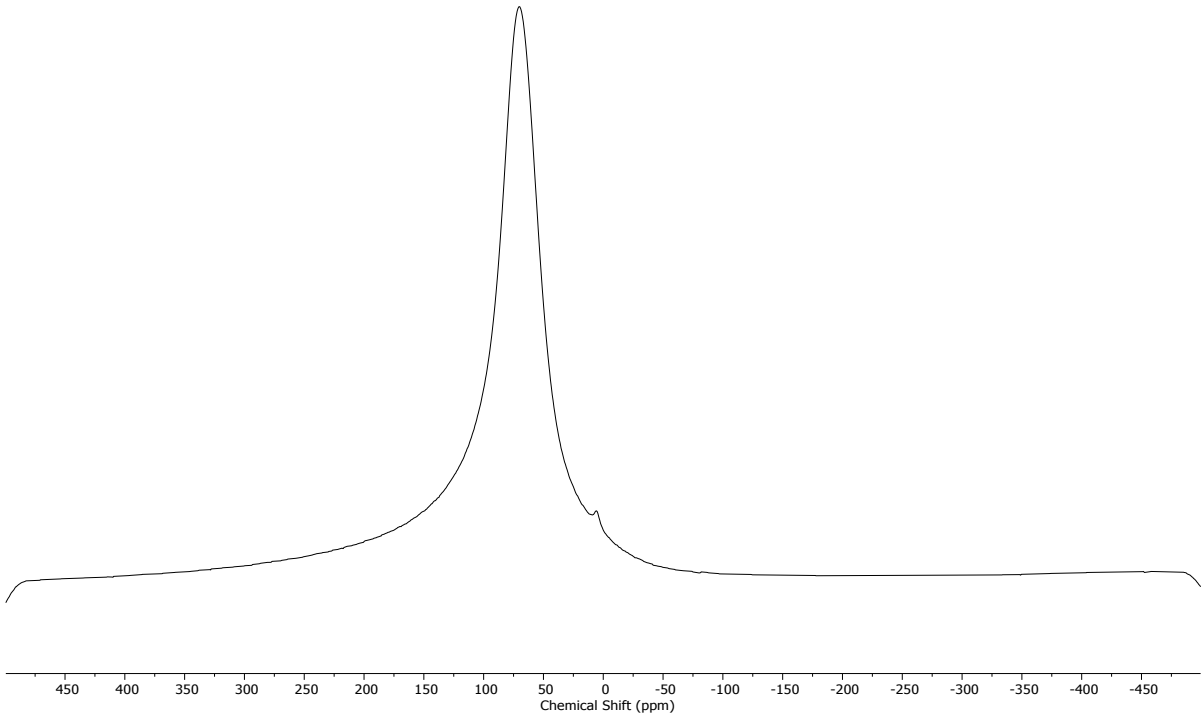

Figure S6. ^{27}Al NMR Spectrum (104 MHz, 298 K, $CDCl_3$) of complex **1**.

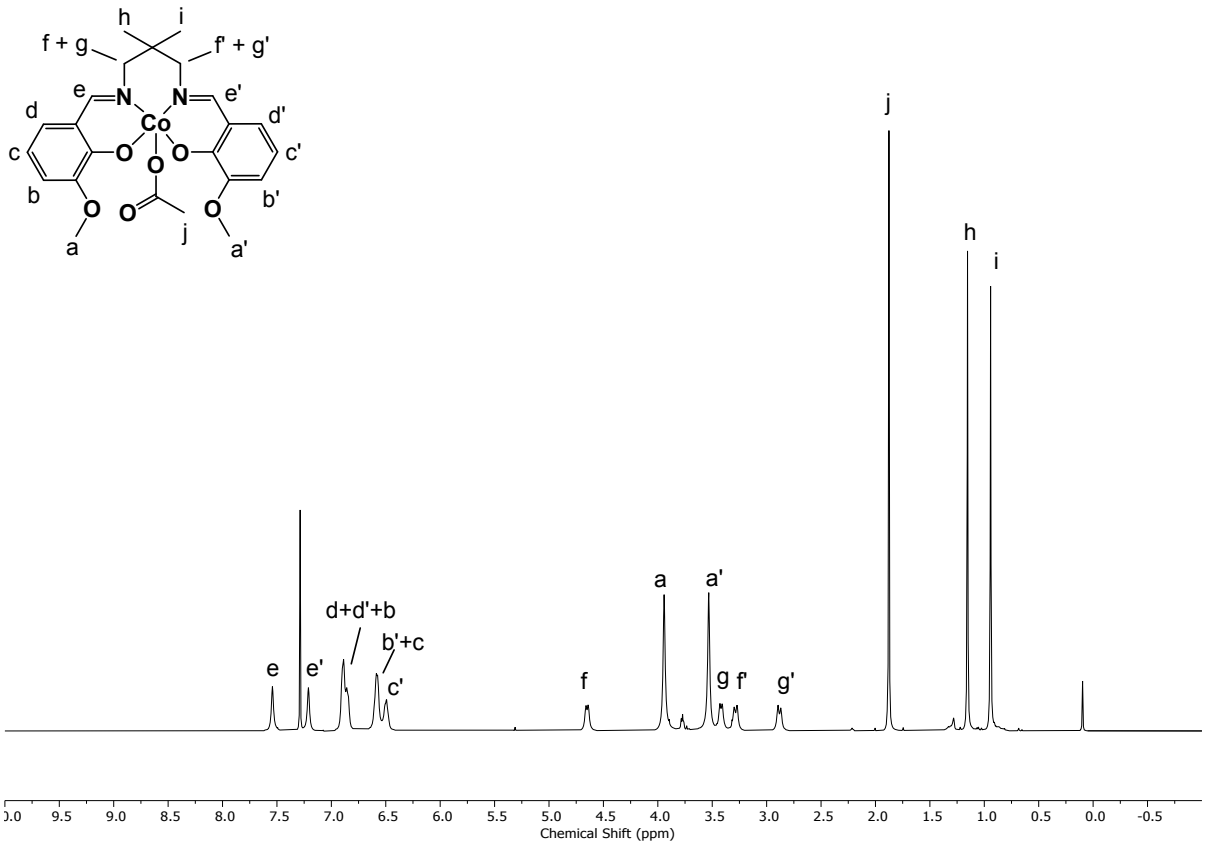

Figure S7. ^{1}H NMR Spectrum (400 MHz, 298 K, $CDCl_3$(*)) of complex **2**.

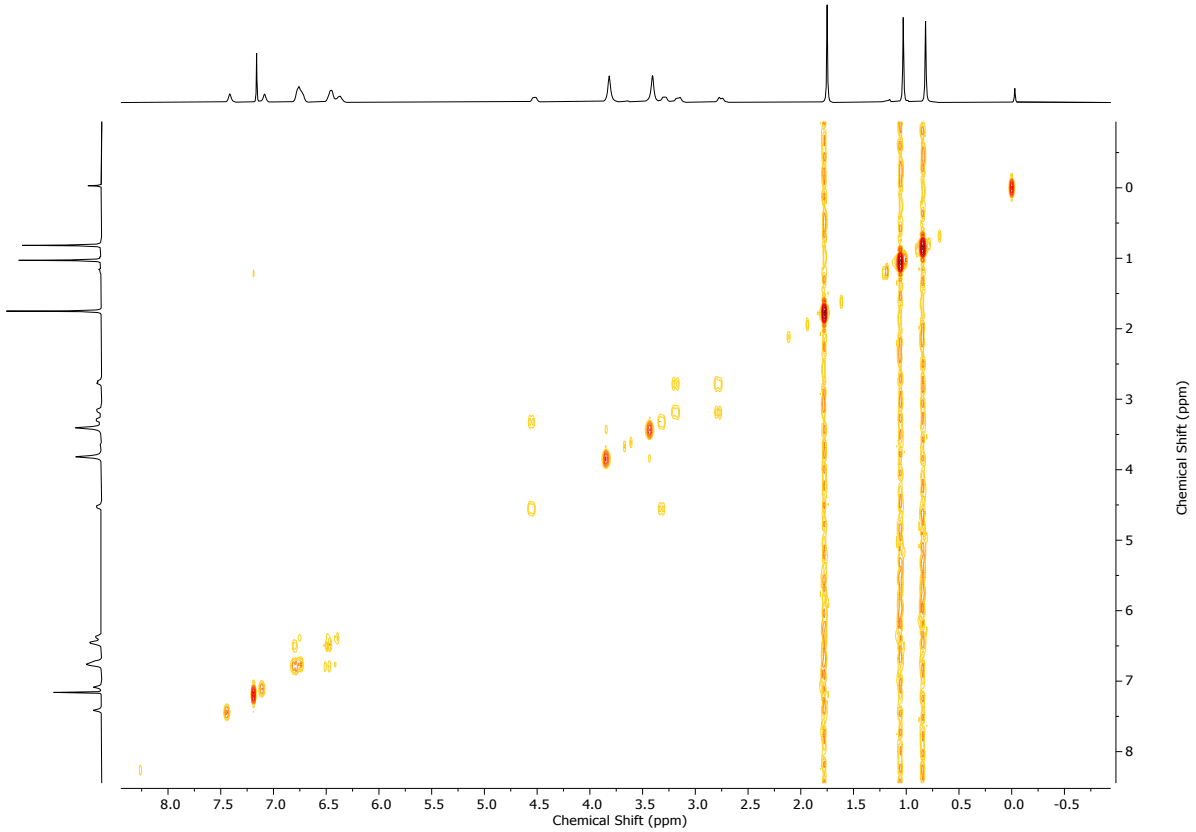

Figure S8. ^{1}H COSY NMR Spectrum (400 MHz, 298 K, $CDCl_3$) of complex **2**.

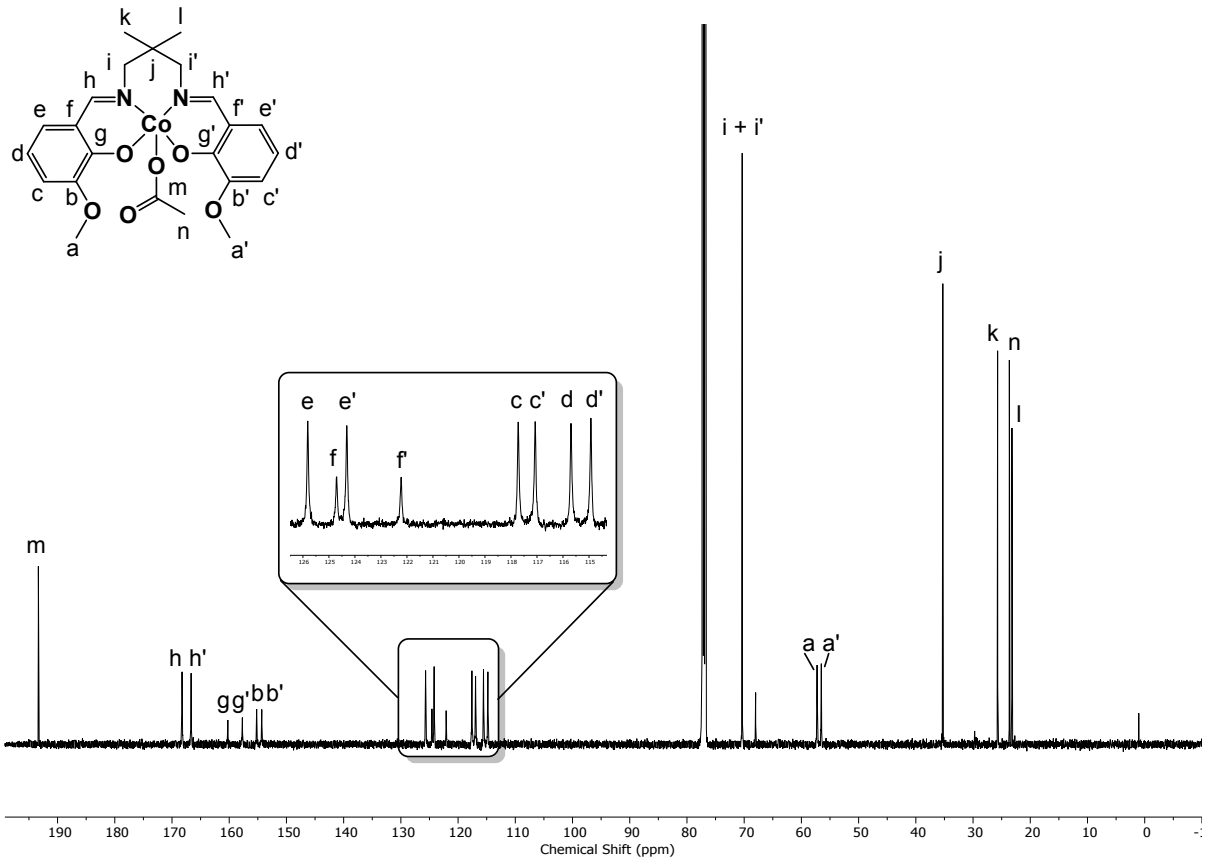

Figure S9. **$^{13}C\{^{1}H\}$ NMR** Spectrum (500 MHz, 298 K, $CDCl_3$(*)) of complex **2**.

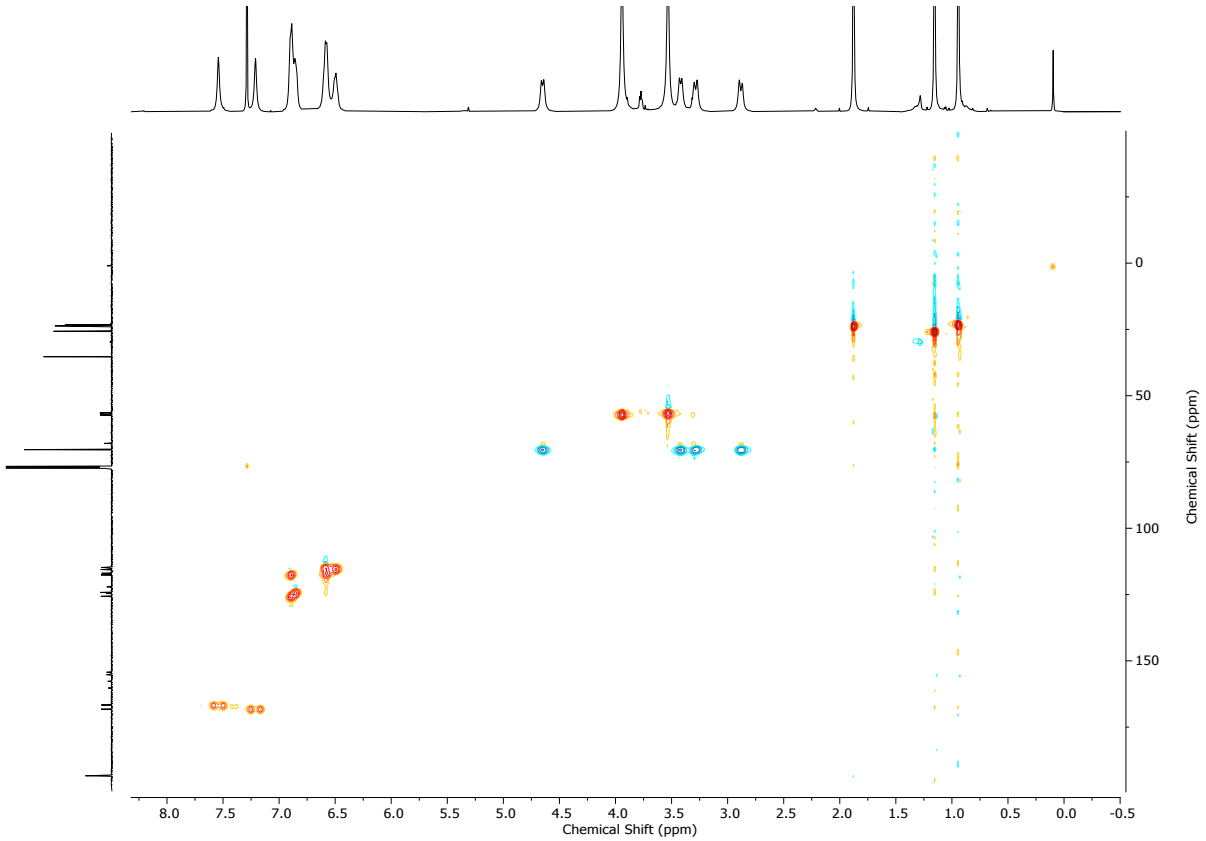

Figure S10. ^{1}H-^{13}C HSQC NMR Spectrum (298 K, CDCl3) of complex **2**.

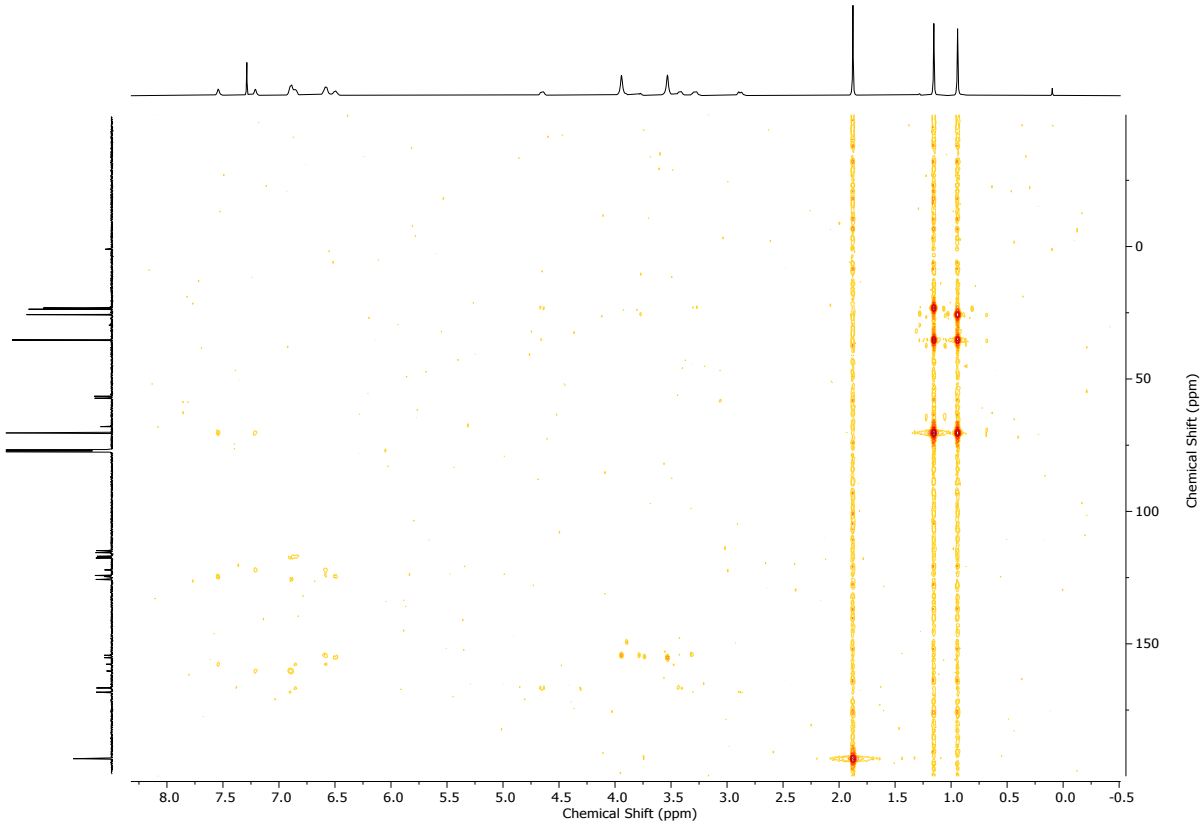

Figure S11. ^{1}H-^{13}C HMBC NMR Spectrum (298 K, $CDCl_3$) of complex **2**.

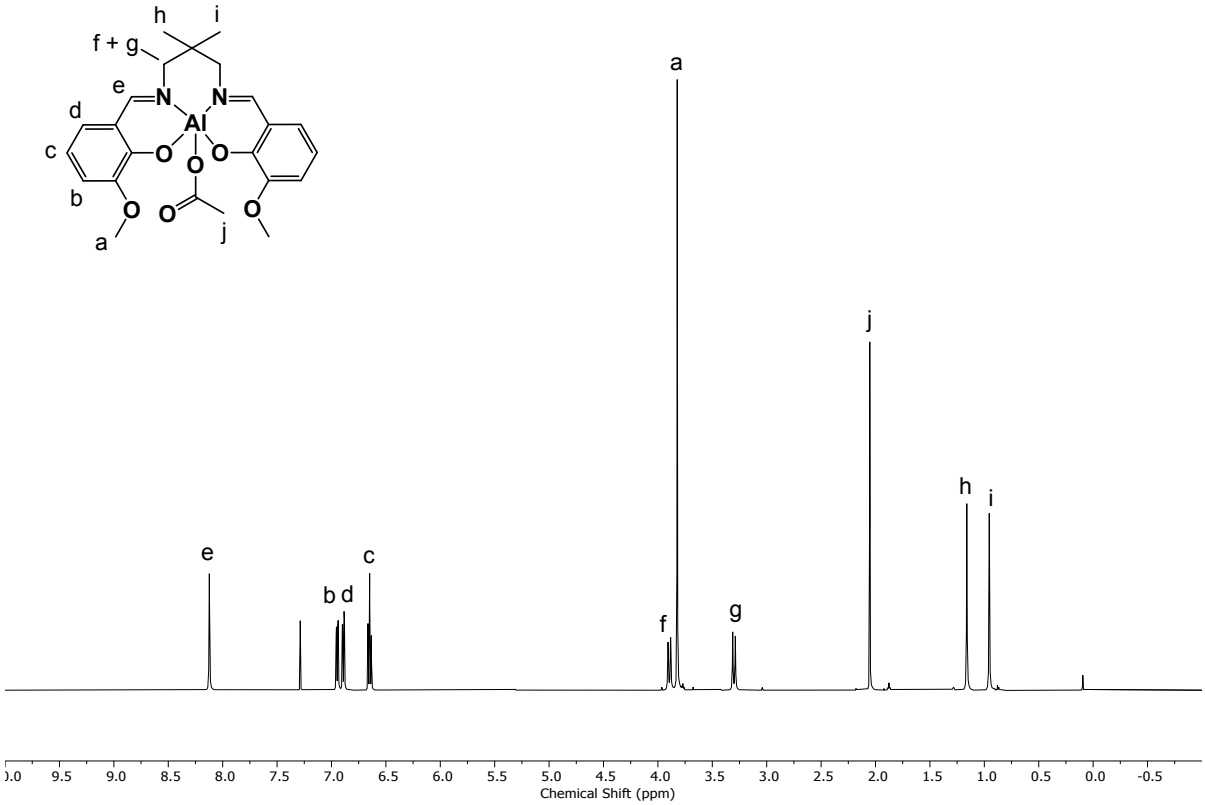

Figure S12. ^{1}H NMR Spectrum (400 MHz, 298 K, $CDCl_3$(*)) of complex **3**.

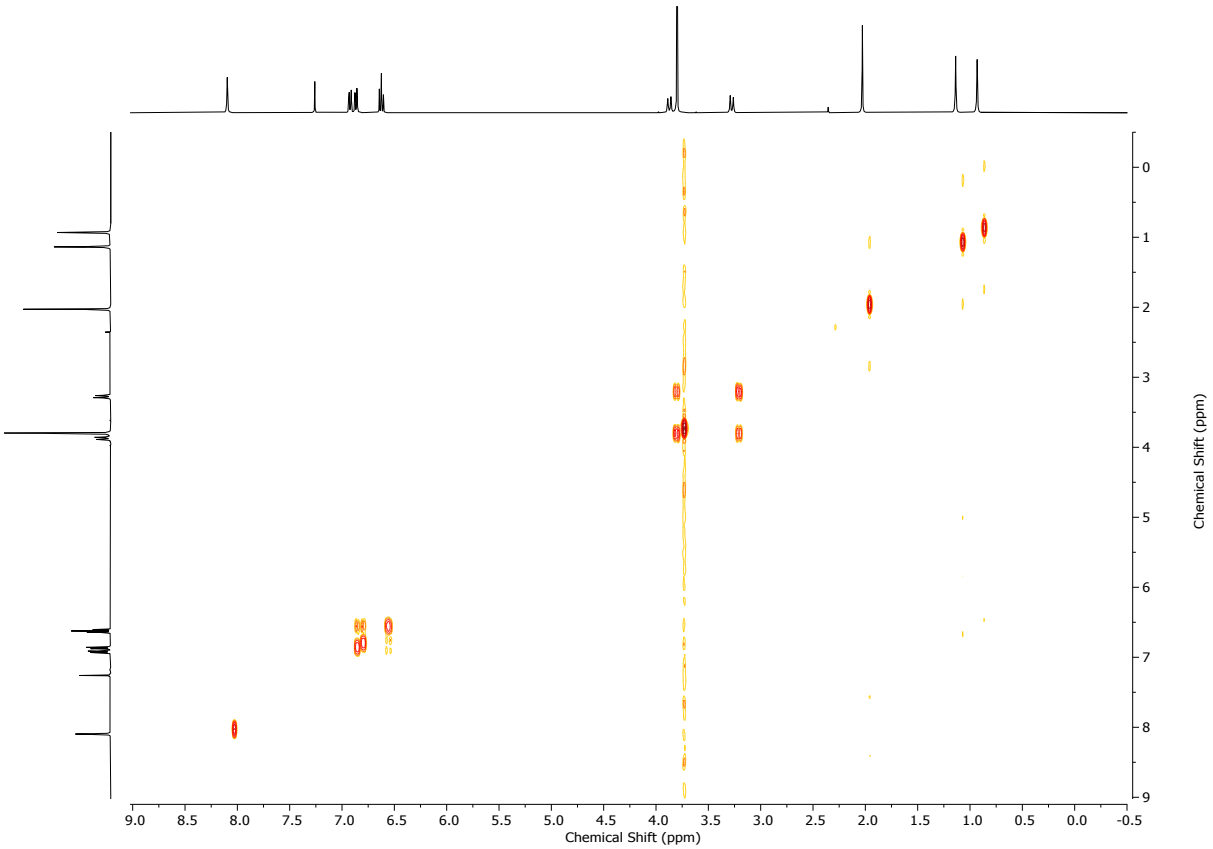

Figure S13. ^{1}H COSY NMR Spectrum (400 MHz, 298 K, $CDCl_3$) of complex **3**.

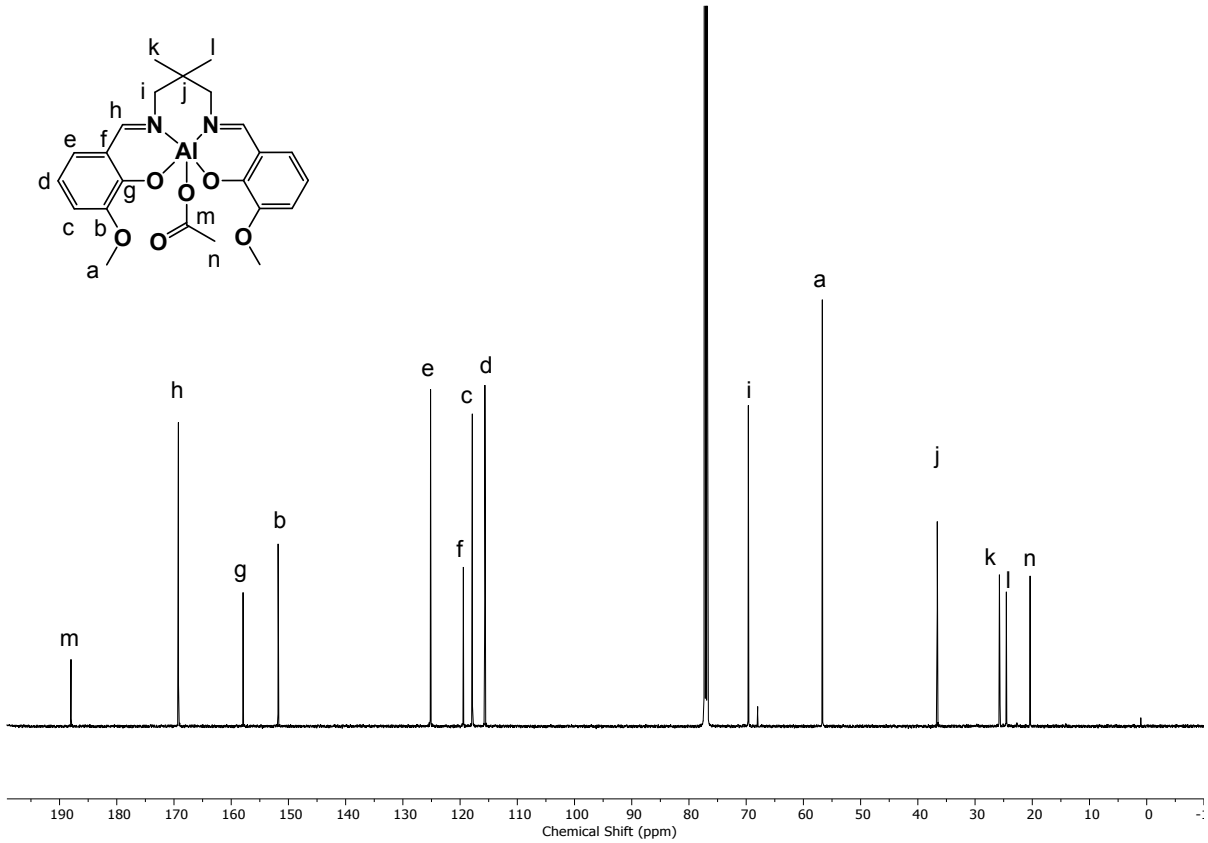

Figure S14. $^{13}C\{^{1}H\}$ NMR Spectrum (500 MHz, 298 K, $CDCl_3$) of complex **3**.

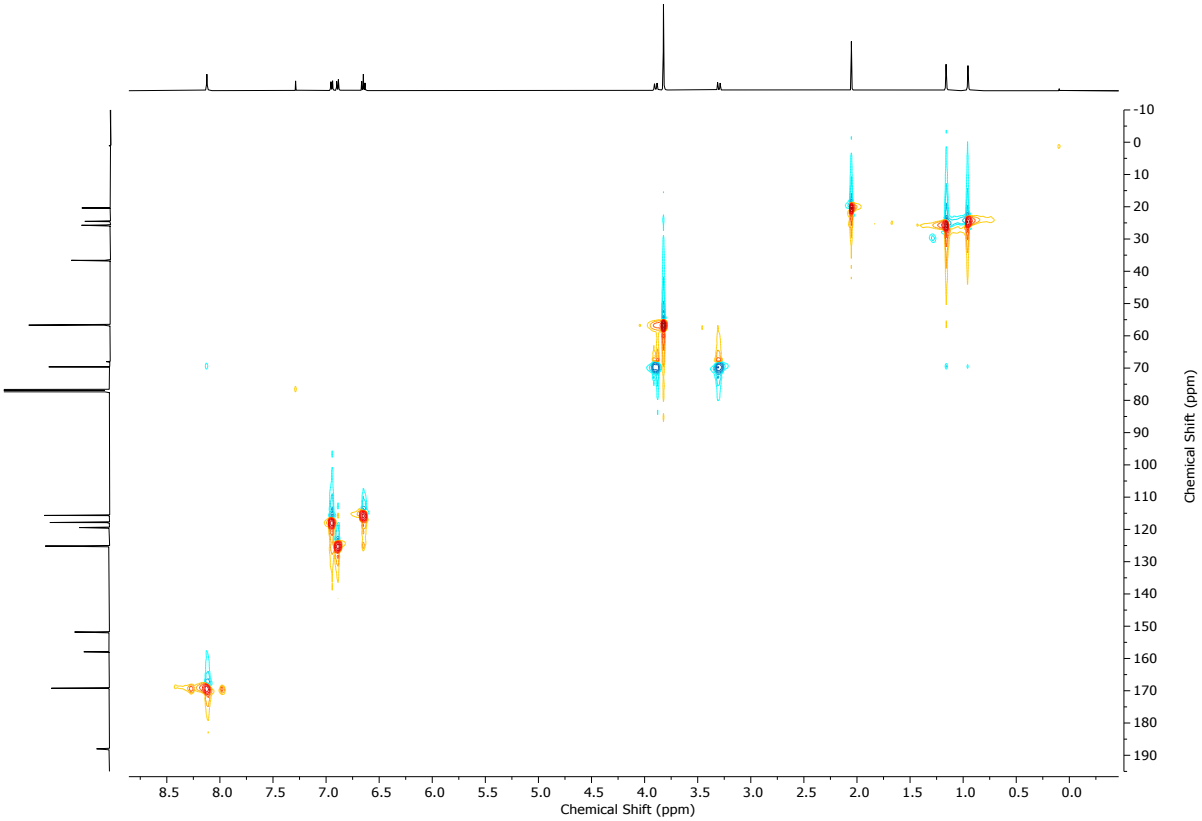

Figure S15. ^{1}H-^{13}C HSQC NMR Spectrum (298 K, $CDCl_3$) of complex **3**.

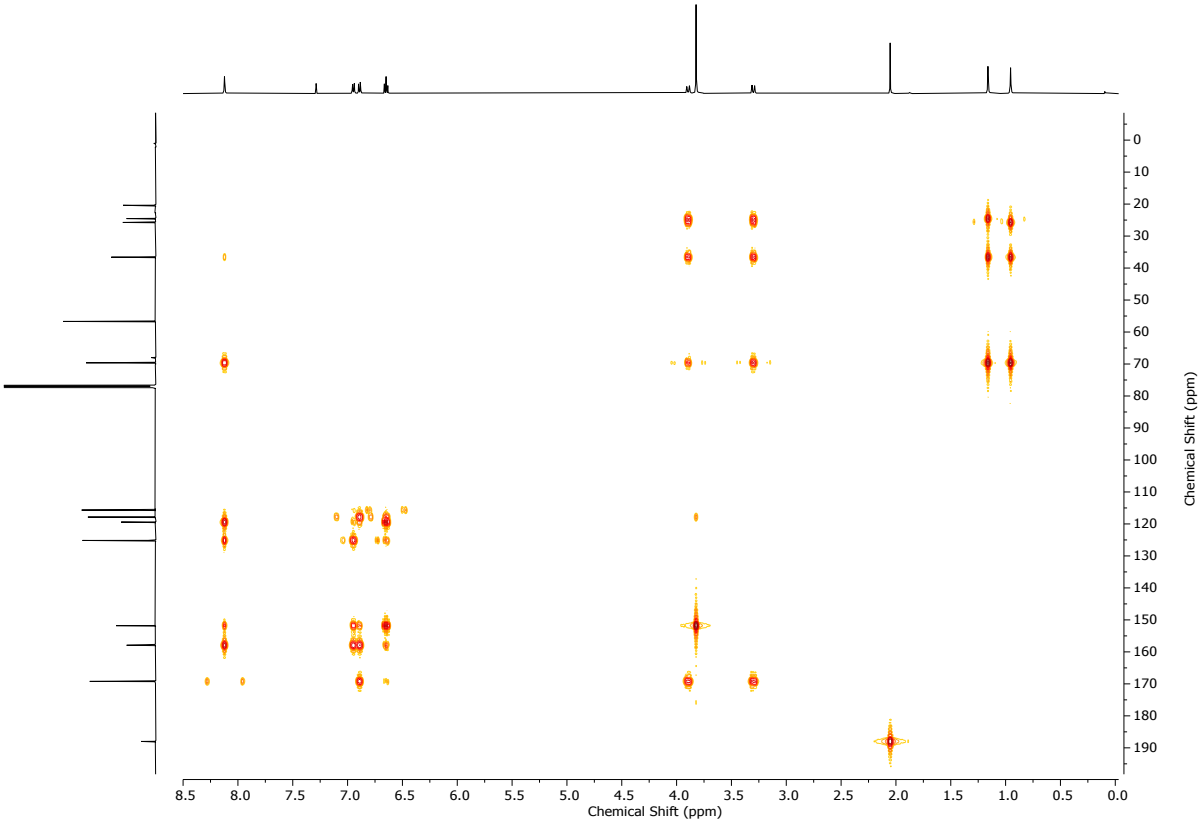

Figure S16. ^{1}H-^{13}C HMBC NMR Spectrum (298 K, $CDCl_3$) of complex **3**.

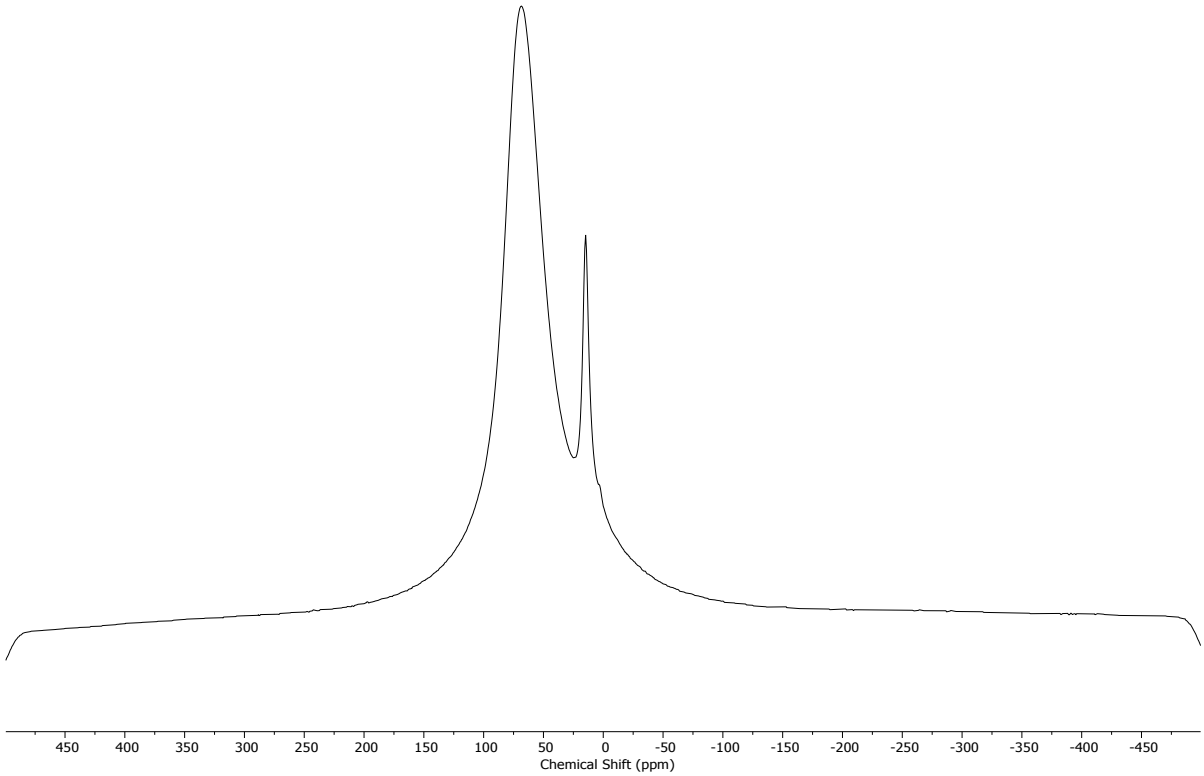

Figure S17. ^{27}Al NMR Spectrum (104 MHz, 298 K, $CDCl_3$) of complex **3**.

Table S5. Polymerisation data for the ROCOP of PA and CHO Utilising Complexes **1**, **2** and SalcyCrCl showing complete PA consumption.

Catalyst[a]	Time (m)	Conv. (%)	$M_{n,\ GPC}$ [Đ][b] (kg mol^{-1})	$M_{n,\ Theory}$[c] (kg mol^{-1})
1	15	100	7.5 [1.16]	8.2
2	15	100	6.5 [1.27]	6.2
SalcyCrCl	10	100	5.1 [1.27]	6.2

[a] Catalysis conditions: [Cat]:[PPNCl]:[CHD]:[PA]:[CHO] = 1:1:2:100:500, given, T = 100 °C. [b] Determined by GPC, in THF, using narrow dispersity polystyrene standards. [c] Theoretical M_n values. Determined through (TON*M_n(Repeat Unit))/ (Number of equivalents of CHD + Number of equivalents of PPNCl (+ Number of equivalents of catalyst when **2** and SalcyCrCl are utilised)).

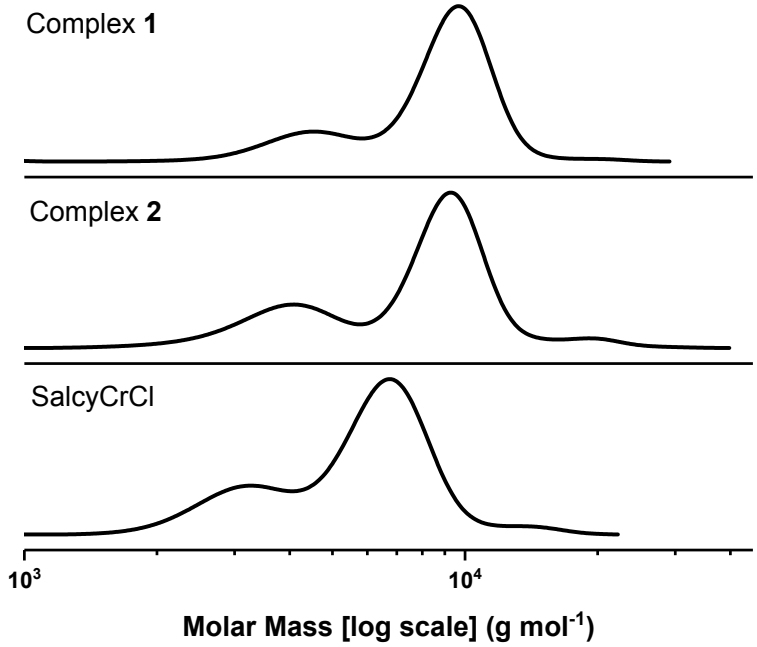

Figure S18. GPC traces for PCHPE synthesised by complexes **1**, **2**, and [SalcyCrCl], corresponding to Table S1, Entries 1, 2, and 3, respectively.

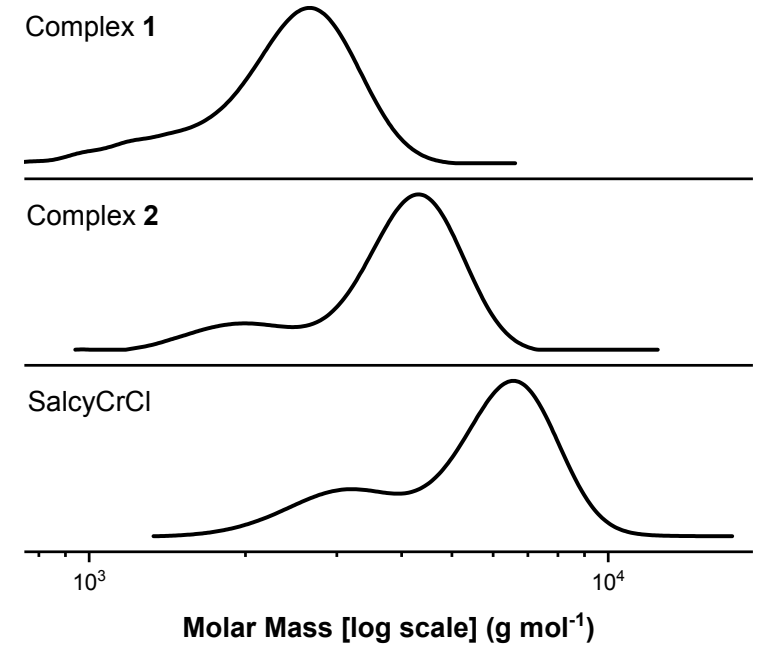

Figure S19. GPC traces for PCHPE synthesised by complexes 1, 2, and [SalcyCrCl], corresponding to Table 1, Entries 1, 2, and 3, respectively.

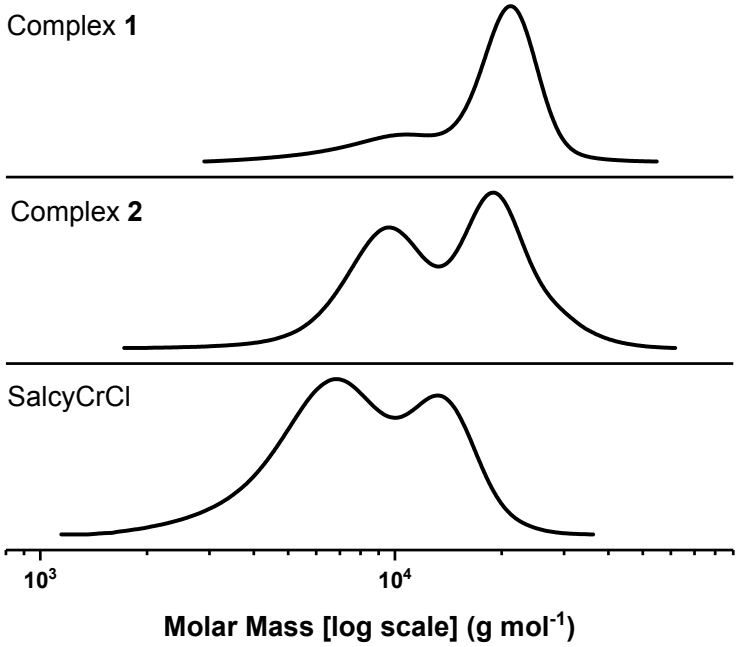

Figure S20. GPC traces for PCHPE synthesised by complexes **1**, **2**, and [SalcyCrCl], corresponding to Table 1, Entries 4, 5, and 6, respectively.

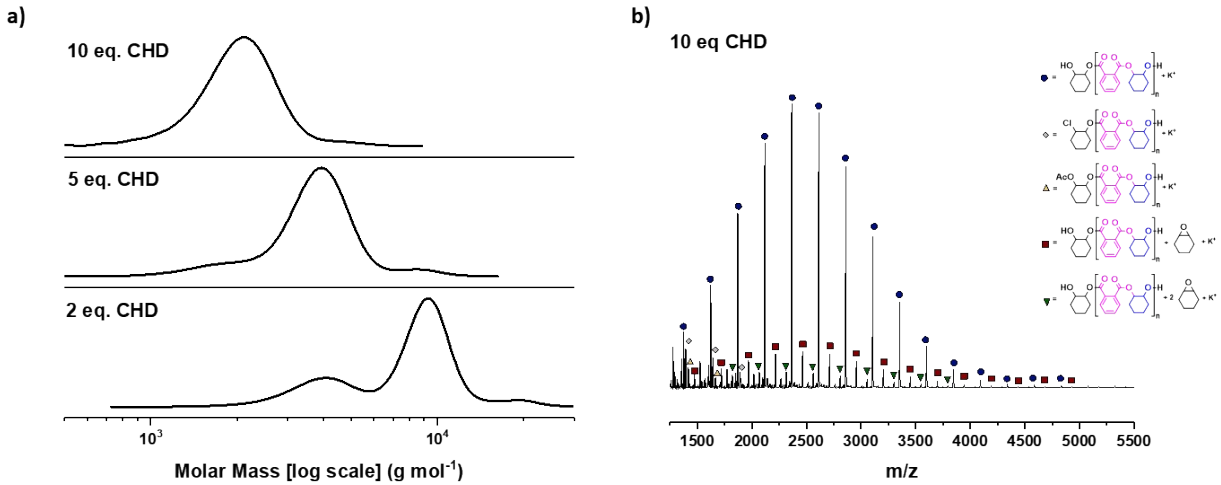

Figure S21. a) GPC traces for PCHPE synthesised with complex **2**, showing that 10 equivalents are necessary to yield monomodal molecular weight distributions. b) MALDI-ToF spectrum of PCHPE synthesised with 10 equivalents of CHD.

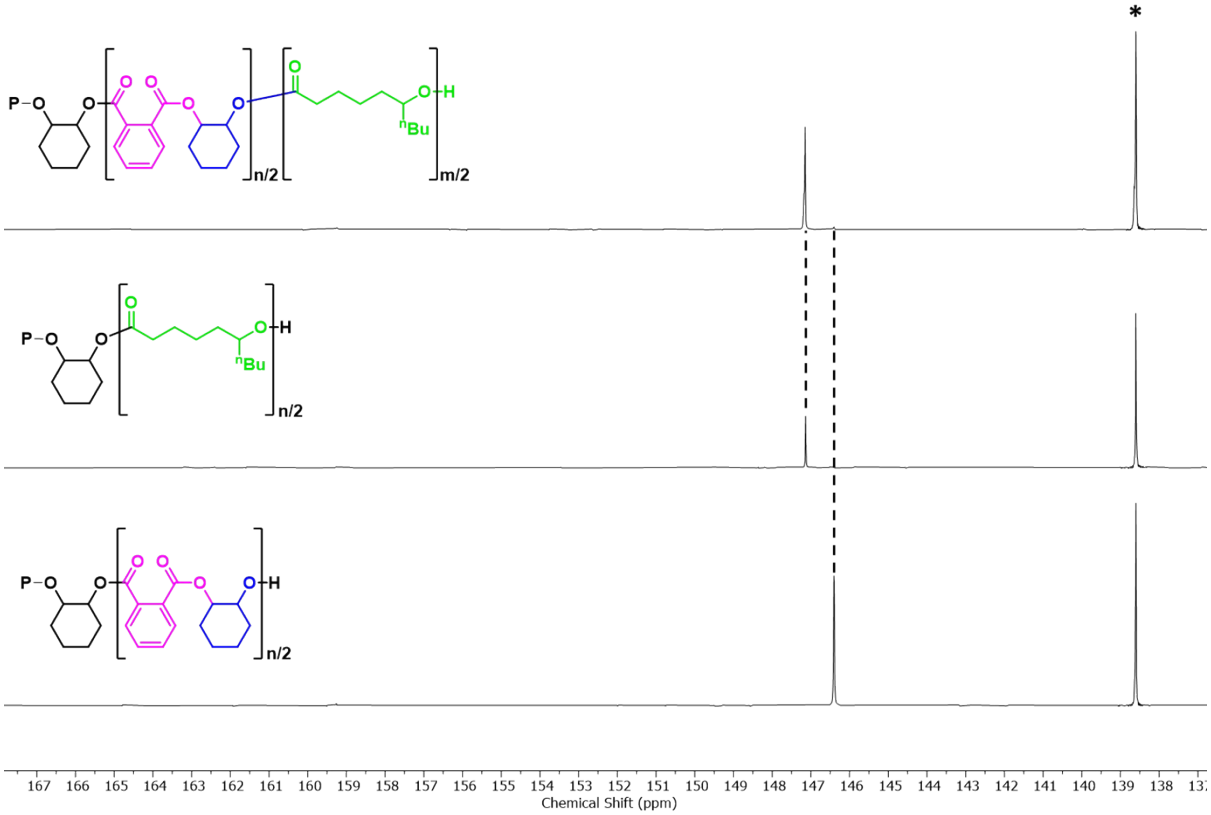

Figure S22. $^{31}P\{^{1}H\}$ NMR Spectrum (298 K, 162 MHz, $CDCl_3$) of PDL-*b*-PCHPE-*b*-PDL (Table 2, Entry 1), and the corresponding spectra for PDL and PCHPE homopolymers.

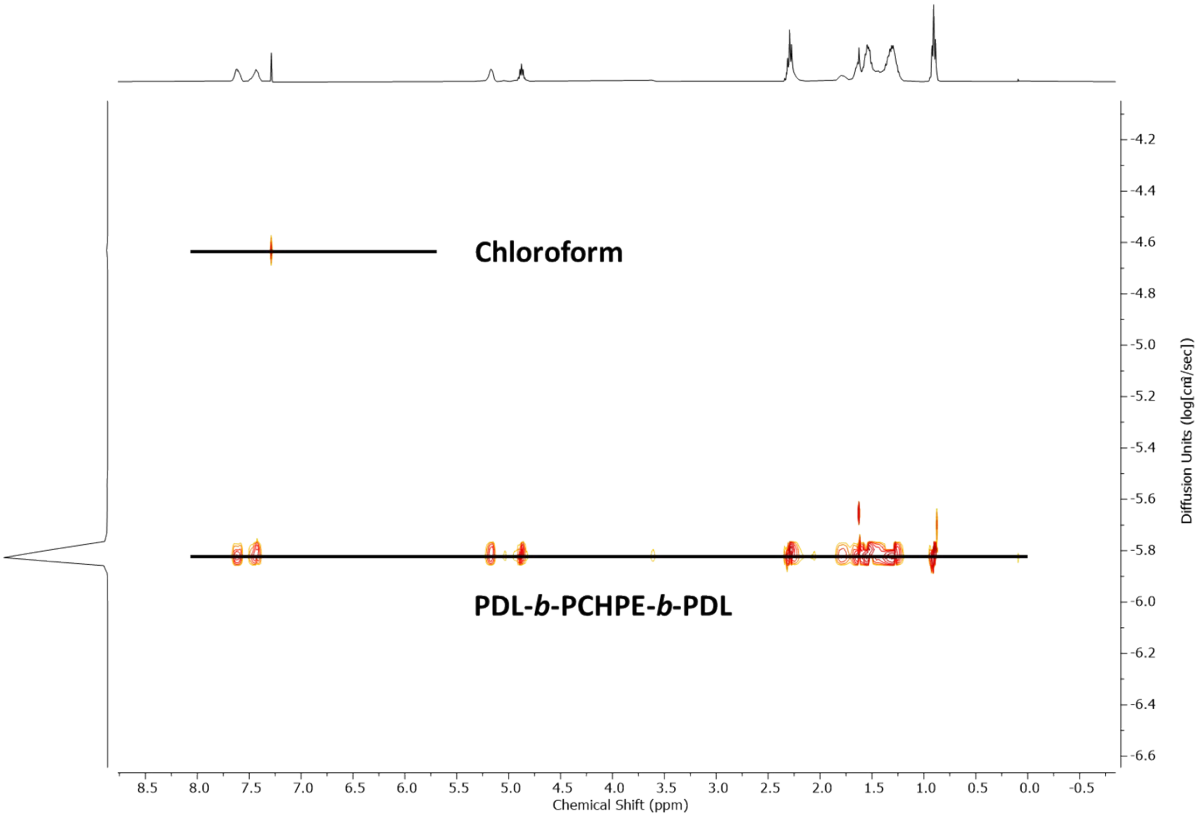

Figure S23. DOSY NMR Spectrum (298 K, $CDCl_3$) of PDL-b-PCHPE-b-PDL (Table 2, Entry 1) showing a single diffusion coefficient, consistent with formation of a triblock copolymer. Previous reports have shown that DOSY NMR spectra of a 50:50 wt% blend of PDL and PCHPE homopolymers show two separate diffusion coefficients.[11]

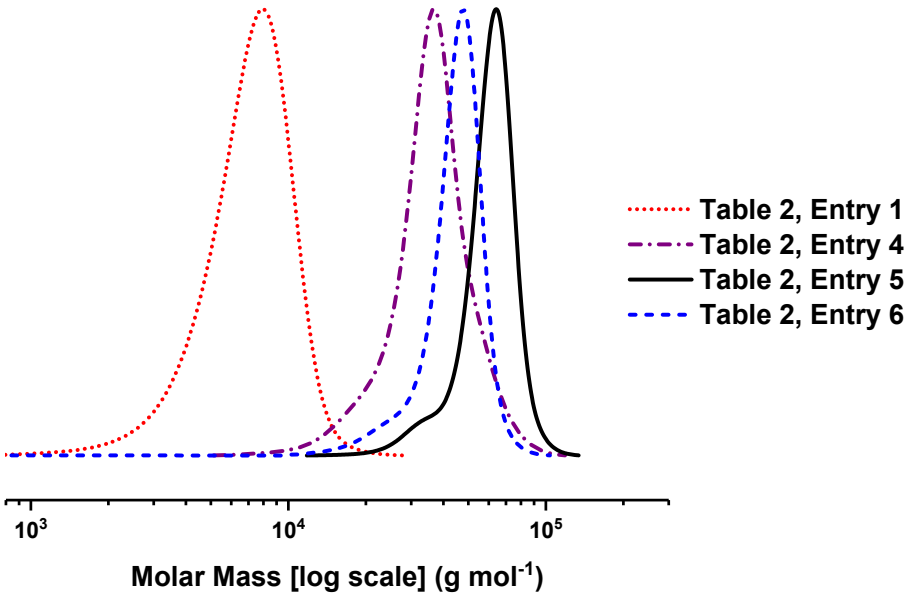

Figure S24. GPC traces for triblock copolymers synthesised with complex **1**, at various catalyst loadings (Entries 1, 4 and 5) and equivalents of CTA (Entry 6).

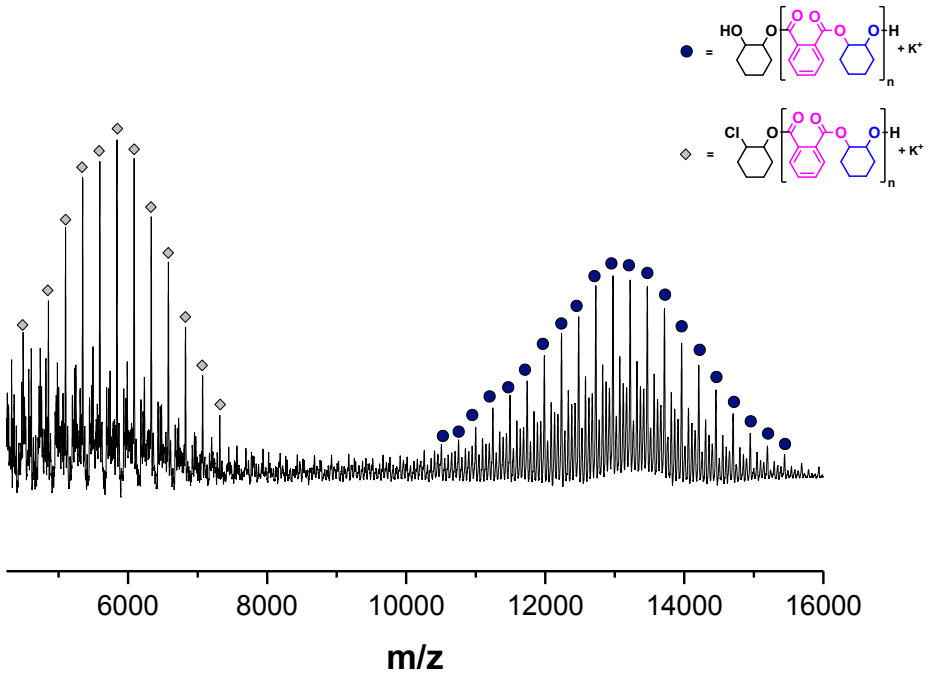

Figure S25. MALDI-ToF spectrum of PCHPE synthesised during switchable catalysis (Table 2, Entry 5) before initiation of DL ROP, showing the low weight shoulder to be due to initiation from chloride anions.

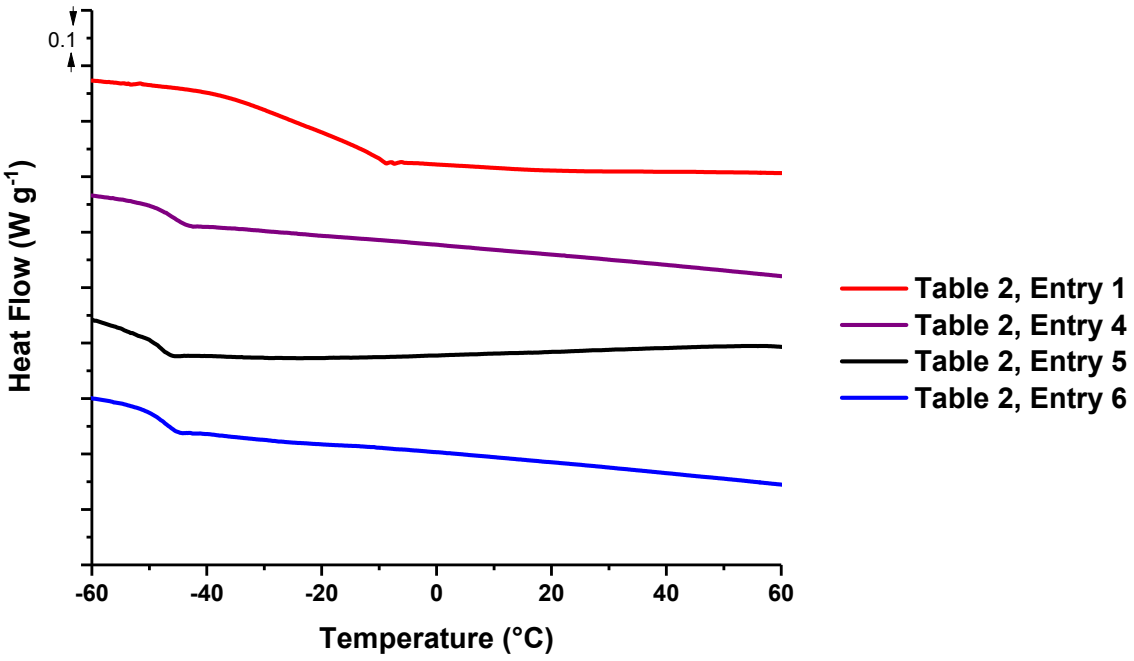

Figure S26. DSC heating curves for triblock synthesised with complex **1**, showing the T_g for the PDL block.

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