

Supporting Information for the Article Entitled
Cyclopentadienyl Amidinate Ligand Directing Effects in the Enantioselective
Living Coordinative Chain Transfer Polymerization of 1,5-Hexadiene

Authored by

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Details for the synthesis of (***R*_C, *R*_{Hf}**)-**2** and (***S*_C, *S*_{Hf}**)-**3**, details for polymerization and isolation of poly(methylene-1,3-cyclopentane). Supporting NMR spectra, DSC traces, GPC traces, optical rotation, and crystallographic data for (***R*_C, *R*_{Hf}**)-**2** and (***S*_C, *S*_{Hf}**)-**3**.

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Experimental Details

General Considerations. All manipulations of air and moisture sensitive compounds were carried out under a N₂ atmosphere with standard Schlenk line or glovebox techniques. Toluene (ReagentPlus, 99%) was dried and deoxygenated by passage over activated alumina and GetterMax 135® catalyst (purchased from Research Catalyst, Inc) and collected prior to use. Chlorobenzene was dried over calcium hydride by refluxing at 130 °C for three days and distilled under N₂ prior to use. 1, 1, 2, 2-Tetrachlorethane-*d*₂(TCE-*d*₂) and chloroform-*d*₁(CDCl₃) was used as received. Toluene-*d*₈ and benzene-*d*₆(C₆D₆) was dried over Na/K alloy and isolated by vacuum transfer before use. 1,5-hexadiene, 1-hexene, and 4-methyl-1-pentene (>97%) were purchased and dried over Na/K alloy and isolated by vacuum transfer before use. [PhNMe₂H][B(C₆F₅)₄] (**B1**) was purchased and used as received. All other solvents and reagents were used as purchased unless stated otherwise. 2-methoxy-1-azacycloheptene (**4**) was prepared from previous reports.^{1,2}

Nuclear Magnetic Resonance (NMR). ¹H and ¹³C {¹H} NMR were obtained at 800 MHz and 200MHz (respectfully) was carried out with a Bruker AVII-HD 800 spectrometer fitted with a Cryo-QCI probe and TCE-*d*₂ was used as the solvent for polymer samples and heated to 110 °C, equilibrated for 30 minutes, prior to collection of spectra. Nuclear Magnetic Resonance (NMR) ¹H and ¹³C {¹H} NMR were also obtained at 400 MHz (AM400) and 100 MHz respectfully. All spectra were referenced using residual chemical shifts of the deuterated solvents. Spectra were referenced to residual solvent peaks (¹H: 7.26 ppm for chloroform-*d*₁, 7.16 for benzene-*d*₆, 6.0 ppm for 1,1,2,2 tetrachloroethane-*d*₂, and 6.97 for toluene-*d*₈; ¹³C: 77.23 ppm for chloroform-*d*₁, 78.8 ppm for 1,1,2,2-tetrachloroethane-*d*₂, 128.06 ppm for benzene-*d*₆, and 137.48 for toluene-*d*₈).

Gel Permeation Chromatography (GPC) of polymers was performed on a Viscotek GPCMax equipped with an Agilent Technologies PLgel MIXED-D column in a column oven and differential refractometer (Viscotek TDA 302) maintained at 40°C. Tetrahydrofuran (HPLC

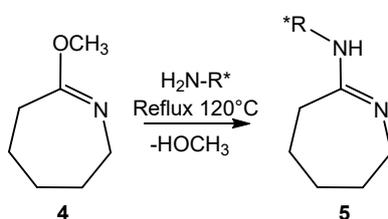
Grade) was used as the eluent with a flow rate of 1 mL/min. Polymer weight was verified relative to polystyrene standards. For room temperature GPC sample preparation, 8 mg of dry polymer sample was dissolved in 2 mL of THF (HPLC Grade).

Differential Scanning Calorimetry (DSC). DSC performed on a TA Instruments Q1000 in T-Zero aluminum sample pans using a heat/cool/heat cycle with a heating rate of 20 °C/min and a cooling rate of 5 °C/min. Data presented is for the second heat cycle.

Optical Rotation. Optical rotation was measured with JASCO P-1010 Polarimeter equipped with 589 nm sodium lamp and non-thermal jacketed cell. Solutions were made at fixed concentrations with technical toluene.

Synthesis

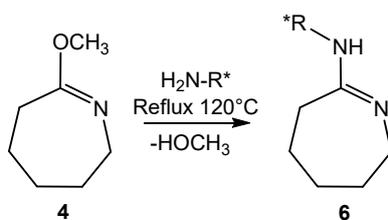
(-)-N-(R)-3-methyl-2-butylcaprolactam (5).



5: R* = (R)-3-methyl,2-butyl (71.6%)

To a 25-mL three-neck round bottom flask was added (*R*)-(-)-2-amino-3-methylbutane (0.69 g, 7.9 mmol) and 2-Methoxy-1-azacycloheptene (**4**) (1.0 g, 7.9 mmol) at ambient temperature. The reaction mixture was heated to 120°C and refluxed for 24 hours. Methanol formed was removed *in vacuo*. The crude product was isolated as an off-white crystalline material that was used without further purification in the next step. Yield of **5**: 1.03g (71.6% yield), For **5**: ¹H-NMR (400 MHz, chloroform-*d*₁, 25°C): δ (ppm) 3.64 (1H, *s*, NH), 3.34 (2H, *s*, (CH)₂), 2.23 (2H, *s*, N(CH₂)CH₂), 1.66 (4H, *m*, C(CH₂)₂), 1.53 (4H, *m*, CH₂(CH₂)₂CH₂), 1.00 (3H, *d*, J = 6.52 Hz, CH(CH₃)), 0.87 (6H, *dd*, J = 5.32 Hz CH(CH₃)₂). ¹³C{¹H} (100 MHz, chloroform-*d*₁, 25°C): δ (ppm) 50.72 (NCH₂), 34.23 (NHCH), 30.90 (CH₂(CH₂)₂), 29.42 (CH₂(CH₂)₂), 25.06 ((CH₃)₂CH), 19.34 ((CH₃)CH), 18.28 (CH(CH₃)CH₃), 17.32 (CH(CH₃)CH₃). [α]_D (°) = -43.87, [5] = 11.04 mM. See Fig. S1 and S2 for ¹H and ¹³C{¹H} NMR spectra, respectively, for **5**.

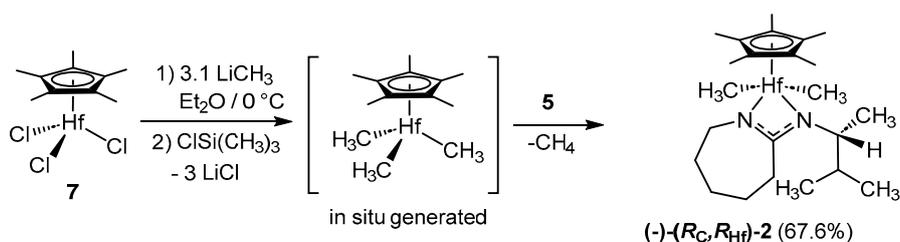
(+)-N-(S)-2-phenylpropylcaprolactam (6).



6: R* = (S)-2-phenylpropyl (92.3%)

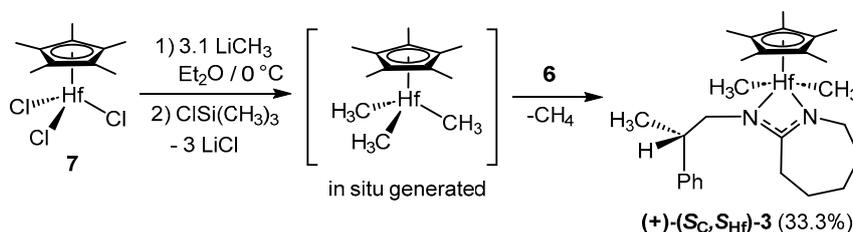
To a 25-mL three-neck round bottom flask was added (*S*)-(+)- β -methylphenethylamine (0.5 g, 3.7 mmol) and 2-Methoxy-1-azacycloheptene (**4**) (0.47 g, 3.7 mmol) at ambient temperature. The reaction was warmed to 120°C and refluxed for 24 hours. Methanol formed was removed *in vacuo* to provide the crude product as a brown oil that was used without further purification in the next step. Yield of **6**: 786 mg (92.3 % yield). For $^1\text{H-NMR}$ (400 MHz, chloroform-*d*₁, 25°C): δ (ppm) 7.33-7.18 (5H, m, Ar), 3.31 (2H, *d*, $J = 5.34$ Hz, NCH_2), 3.13 (1H, m, $\text{NH}(\text{CH}_2)$), 2.99 (1H, *q*, $J = 7.14$ Hz, $\text{CH}_3(\text{CH})$), 2.18 (2H, *s*, $\text{C}(\text{CH}_2)$), 1.67 (2H, m, $(\text{CH}_2)_2(\text{CH}_2)\text{CH}_2$), 1.51 (4H, m, $(\text{CH}_2)_2$), 1.29 (3H, *d*, $J = 6.92$ Hz, $\text{CH}(\text{CH}_3)$). $^{13}\text{C}\{^1\text{H}\}$ (100 MHz, chloroform-*d*₁, 25°C): δ (ppm): 164.08 (NCNH), 145.3 (C_6H_5), 128.5-126.3 (C_6H_5), 49.7 (NCH_2), 47.7 ($\text{C}(\text{CH}_2)$), 39.9 ($\text{CH}(\text{CH}_3)$), 34.3 ($\text{NH}(\text{CH}_2)$), 30.9 ($\text{CH}_2(\text{CH}_2)\text{CH}_2$), 29.6 ($\text{CH}_2(\text{CH}_2)\text{CH}_2$), 24.9 ($\text{CH}_3(\text{CH})$), 20.0 ($\text{CH}_2(\text{CH}_2)\text{CH}_2$). $[\alpha]_{\text{D}}^\circ = 48.11$, $[\eta] = 8.73$ mM. See Fig. S3 and S4 for ^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra, respectively, for **6**.

Cp*Hf[(N-(-)-(R)-3-methyl-2-butyl)(N^{imcap}N)](Me)₂ ((*R*_C, *R*_{Hf})-2**).**



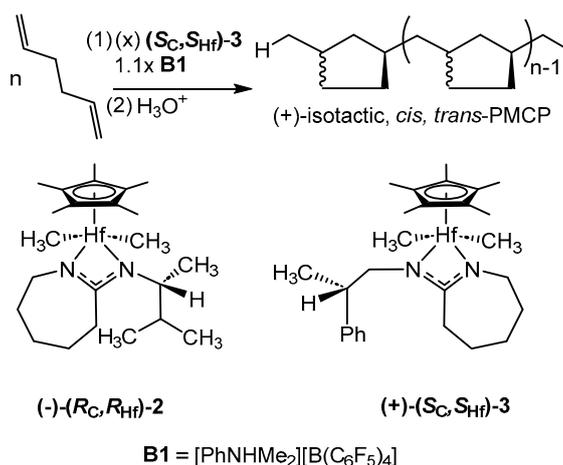
To a stirring solution of Cp^*HfCl_3 (1.0 g, 2.38 mmol) in Et_2O (50 mL) was added MeLi (1.23 M, 6.2 mL, 7.6 mmol) at -70°C . The reaction was stirred for 3 hours after warming to 0°C , where a color change was observed from magenta to white. TMSCl (0.3 mL, 2.38 mmol) was added to the mixture at -30°C , and stirred for an additional 30 minutes. **5** (0.55 g, 2.38 mmol) was added and mixture was stirred for 2 hours after equilibrating to 0°C . Solvent was removed *in vacuo*, and the white solids were extracted through a celite frit with 50 mL pentane. The resulting filtrate was concentrated upon which white crystalline solids remained. Crude crystalline solids were dissolved in minimal pentane and recrystallized after 16 hours in -30°C . White crystalline material was separated and residual solvent removed *in vacuo*. Yield: 0.845 g (67.6 % yield). For (*R*_C, *R*_{Hf})-**2**: $^1\text{H NMR}$ (400 Hz, C_6D_6 , 25°C): δ (ppm) 3.024 (1H, m, $\text{CH}_3(\text{CH})\text{N}$), 2.97 (2H, m, NCH_2CH_2), 2.03 (15H, *s*, $\text{C}_5(\text{CH}_3)_5$), 1.37 (4H, m, $(\text{CH}_2)_2$), 1.25 (4H, m, $(\text{CH}_2)_2$), 1.11-1.10 (3H, *d*, $J = 6.66$ Hz, NCHCH_3), 0.99 (3H, *d*, $J = 6.67$ Hz, CH_3CH), 0.84 (3H, *d*, $J = 6.54$ Hz, CH_3), 0.29-0.01 (6H, *d*, $J = 11.75$ Hz, $(\text{CH}_3)_2$). $^{13}\text{C}\{^1\text{H}\}$ (100 MHz, CDCl_3 , 25°C): δ (ppm) 164.1 (NCN), 145.3 (C_6H_5), 128.5-126.4 (C_6H_5), 49.7 (NCH_2), 45.9 ($\text{NC}(\text{CH}_2)$), 39.9 ($\text{CH}(\text{CH}_3)$), 34.3 ($\text{NH}(\text{CH}_2)$), 29.6 ($(\text{CH}_2)_3\text{CH}_2$), 24.9 ($\text{CH}_3(\text{CH})\text{C}_6\text{H}_5$), 20.0 ($(\text{CH}_2)_2\text{CH}_2$), 11.5 ($\text{C}(\text{CH}_3)_5$). $[\alpha]_{\text{D}}^\circ = -29.90$, $[(\text{R}_{\text{C}}, \text{R}_{\text{Hf}})\text{-2}] = 3.81$ mM. See Fig. S5 and S6 for ^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra, respectively, for **2**.

Cp*Hf[(N-(+)-(S)-2-phenylpropyl)(N^{imcap}N)](Me)₂ ((S_C, S_{Hf})-3).



To a stirring solution of Cp*HfCl₃ (1.0 g, 2.38 mmol) in Et₂O (50 mL) was added MeLi (1.23 M, 6.2 mL, 7.6 mmol) at -70°C. The reaction was stirred for 3 hours after warming to 0°C, where a color change was observed from magenta to white. TMSCl (0.3 mL, 2.38 mmol) was added to the mixture at -30°C, and stirred for an additional 30 minutes. **6** (0.43 g, 2.38 mmol) was added as a solid and mixture was stirred for 2 hours after equilibrating to 0°C. Solvent was removed in vacuo, and the white solids were extracted through a celite frit with 50 mL pentane. The resulting filtrate was concentrated upon which white crystalline solids remained. Crude crystalline solids were dissolved in minimal pentane and recrystallized after 16 hours in -30°C. Crystalline material was separated and residual solvent removed *in vacuo*. Yield: 0.47 g (33.3 % yield). For (S_C, S_{Hf})-3: ¹H NMR (400 MHz, C₆D₆, 25°C): δ (ppm) 7.165 (5H, m, Ar), 3.30 (2H, *dq*, J = 9.03 Hz, NCH₂), 2.07 (2H, *s*, NCH₂), 2.85 (1H, *q*, J = 7.08 Hz, CH(CH₃)), 2.02 (15H, *s*, C₅(CH₃)₅), 1.82 (2H, *t*, J = 6.16 Hz, C(CH₂)), 1.32 (4H, *m*, (CH₂)₂), 1.15 (3H, *d*, J = 7.04 Hz, CH(CH₃)), 0.98 (2H, *m*, CH₂(CH₂)CH₂), 0.02 (6H, *dd*, J = 15.6 Hz, (CH₃)₂). ¹³C{¹H} (100 MHz, C₆D₆, 25°C): δ (ppm) 179.4 (NCN), 146.02 (C₆H₅), 117.7 (C(CH₃)), 54.6 (NCH₂), 50.6 (M(CH₃)₂), 47.0 ((CH₂)CH), 42.01 (CH(CH₃)), 30.55-18.56 (N(CH₂)₅), 10.7 (C(CH₃)₅). [α]_D (°) = 30.44, [(R_C, R_{Hf})-3] = 3.49 mM. See Fig. S7 and S8 for ¹H and ¹³C{¹H} NMR spectra, respectively.

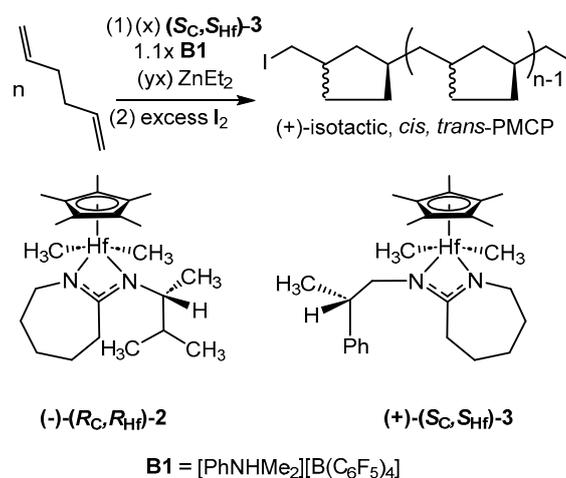
General procedure for LCP of 1,5-hexadiene.



In a 50-mL round bottom flask, to a stirring solution of chlorobenzene (3 mL) and 1,5-hexadiene (657.12 mg, 8.0 mmol) chilled to 0°C was added a yellow mixture of [PhNHMe₂H][B(C₆F₅)₄]

(35.25 mg, 0.044 mmol), (R_C, R_{Hf})-**2** or (S_C, S_{Hf})-**3** (0.04 mmol), and chlorobenzene (1 mL). The reaction was stirred for 20 hours at 0°C before adding to 40 mL acidic methanol (10% HCl). Product was concentrated then dissolved toluene and passed through silica column. Solvent was removed *in vacuo* and the white crystalline product was characterized by GPC, DSC and NMR. GPC and DSC values are provided in Table 1.

General procedure LCCTP of 1,5-hexadiene.



In a 50-mL round bottom flask, to a stirring solution of toluene (4 mL), ZnEt₂ (24.72 mg, 0.2 mmol), and 1,5-hexadiene (1.0 g, 12.17 mmol) chilled to 0°C was added a yellow mixture of [PhNHMe₂H][B(C₆F₅)₄] (35.25 mg, 0.044 mmol), (R_C, R_{Hf})-**2** or (S_C, S_{Hf})-**3** (0.04 mmol), and chlorobenzene (1 mL). The reaction was stirred for 20 hours at 0°C before quenching with excess I₂ (200 mg). Solvent removed *in vacuo* and brown oily material was dissolved in toluene and passed through silica column. Solvent was removed *in vacuo* and isolated brown oily material was characterized by GPC and NMR. GPC values are provided in Table 1

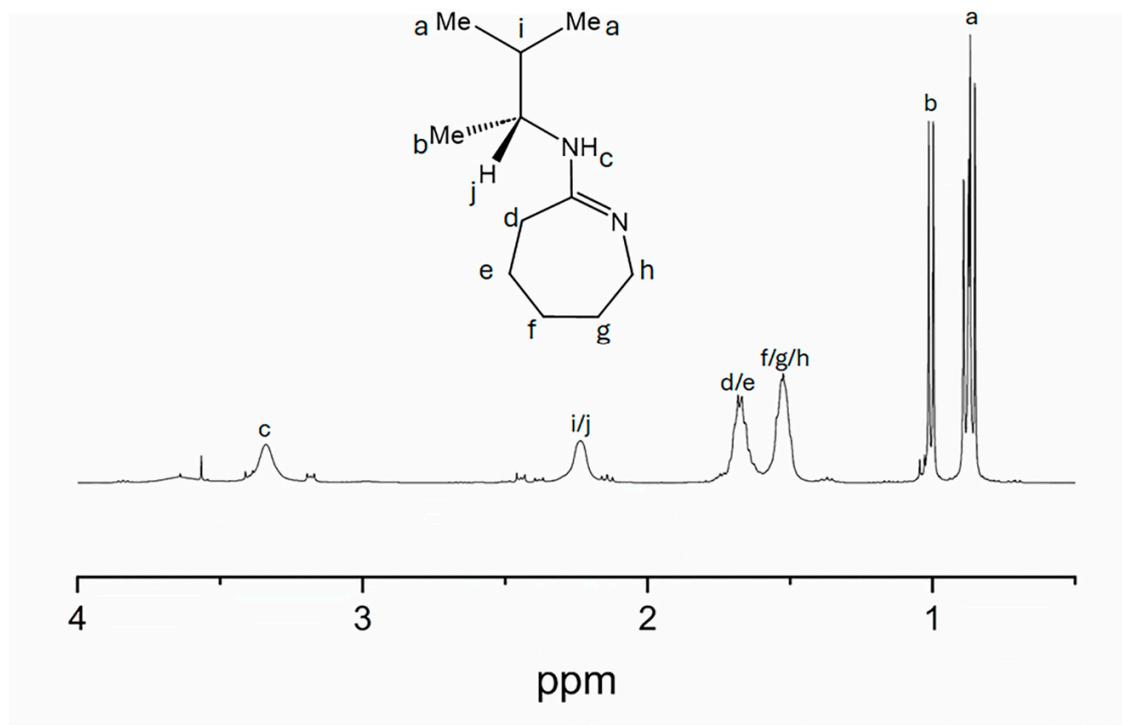
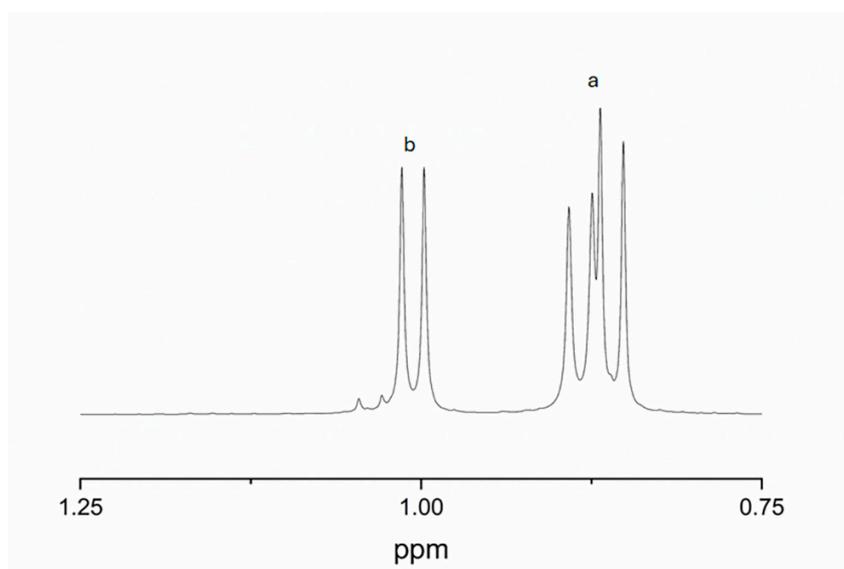
NMR Spectra

Figure S1. Partial ¹H NMR (400 MHz, CDCl₃, 25°C) spectrum of **5**.

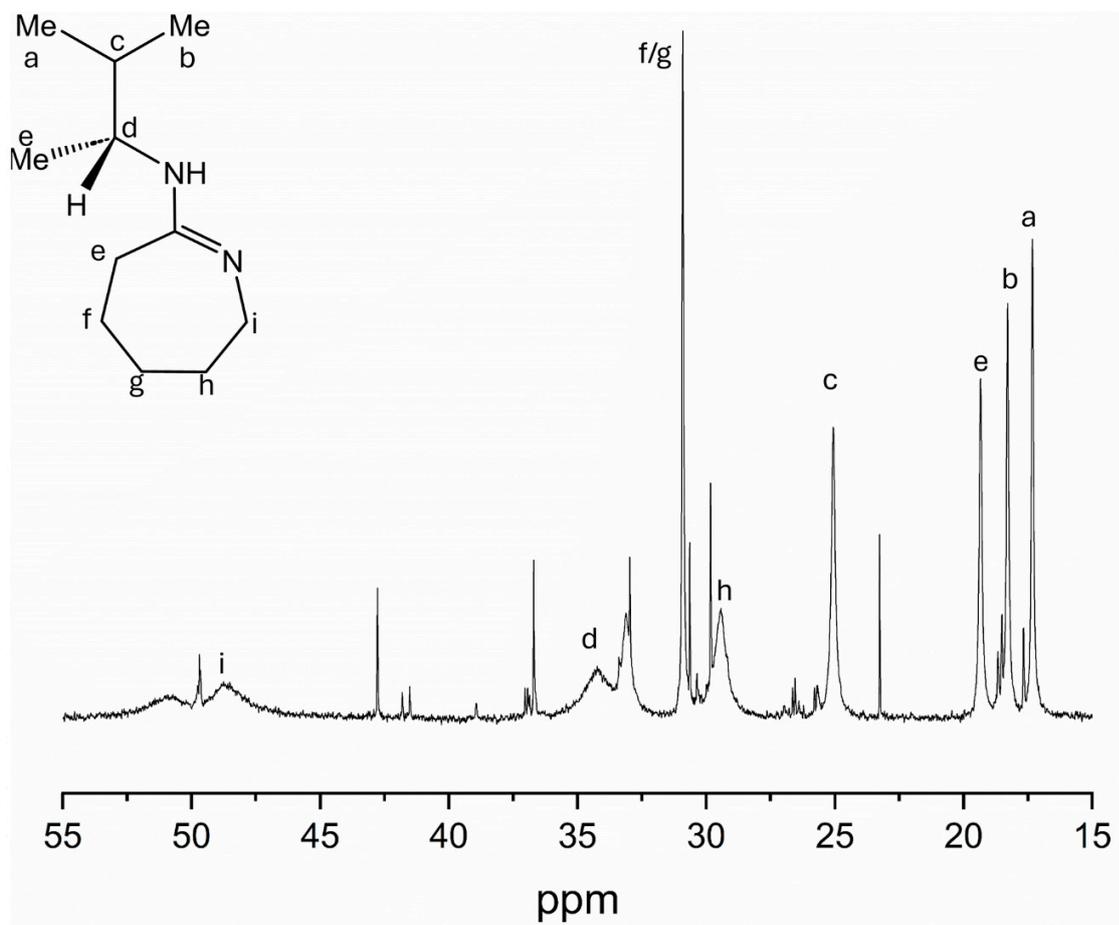


Figure S2. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3 , 25°C) spectrum of **5**.

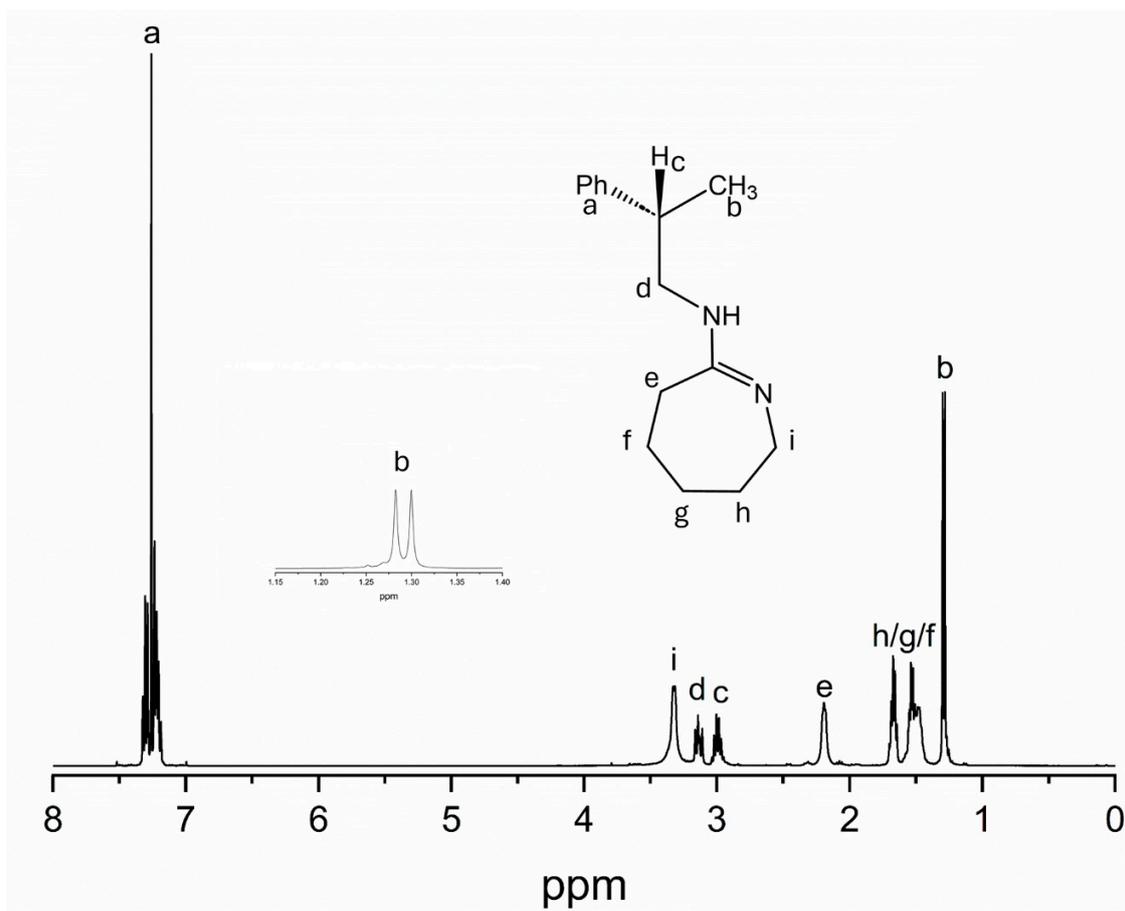


Figure S3. ^1H NMR (400 MHz, CDCl_3 , 25°C) spectrum of **6**.

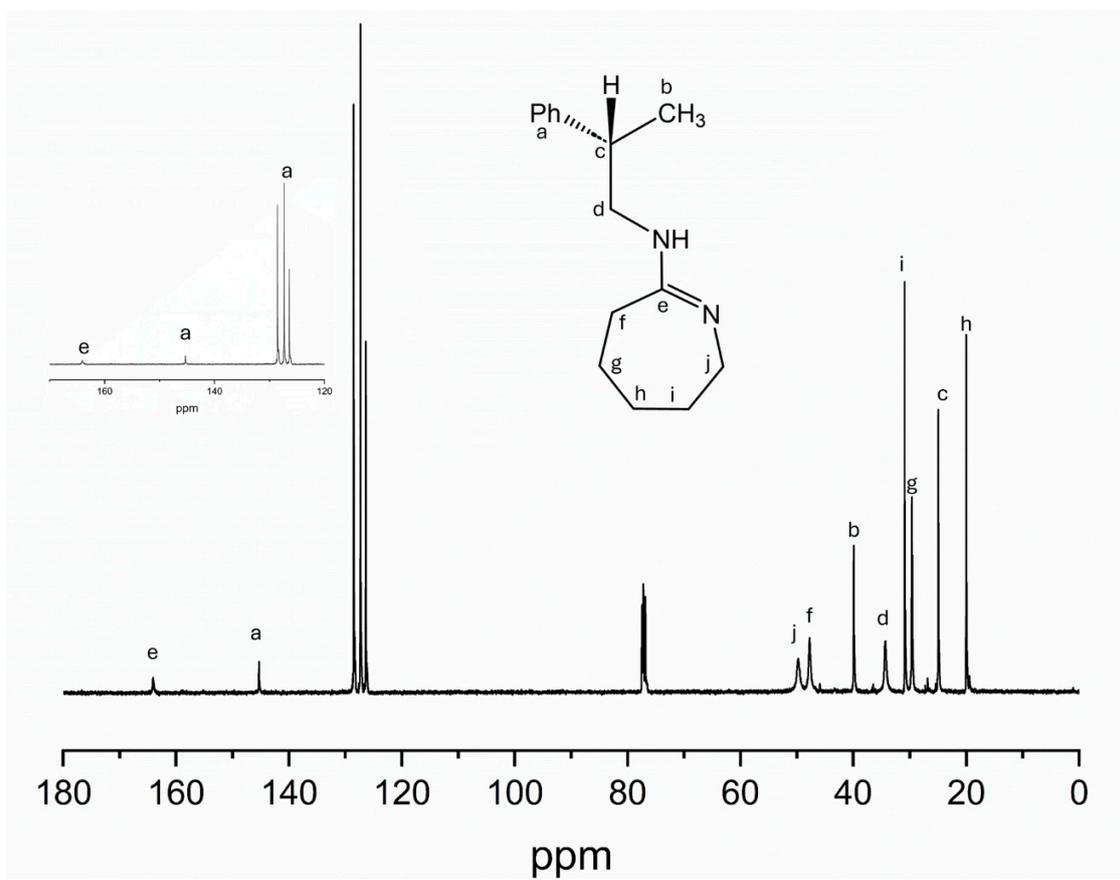


Figure S4. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3 , 25°C) spectrum of **6**.

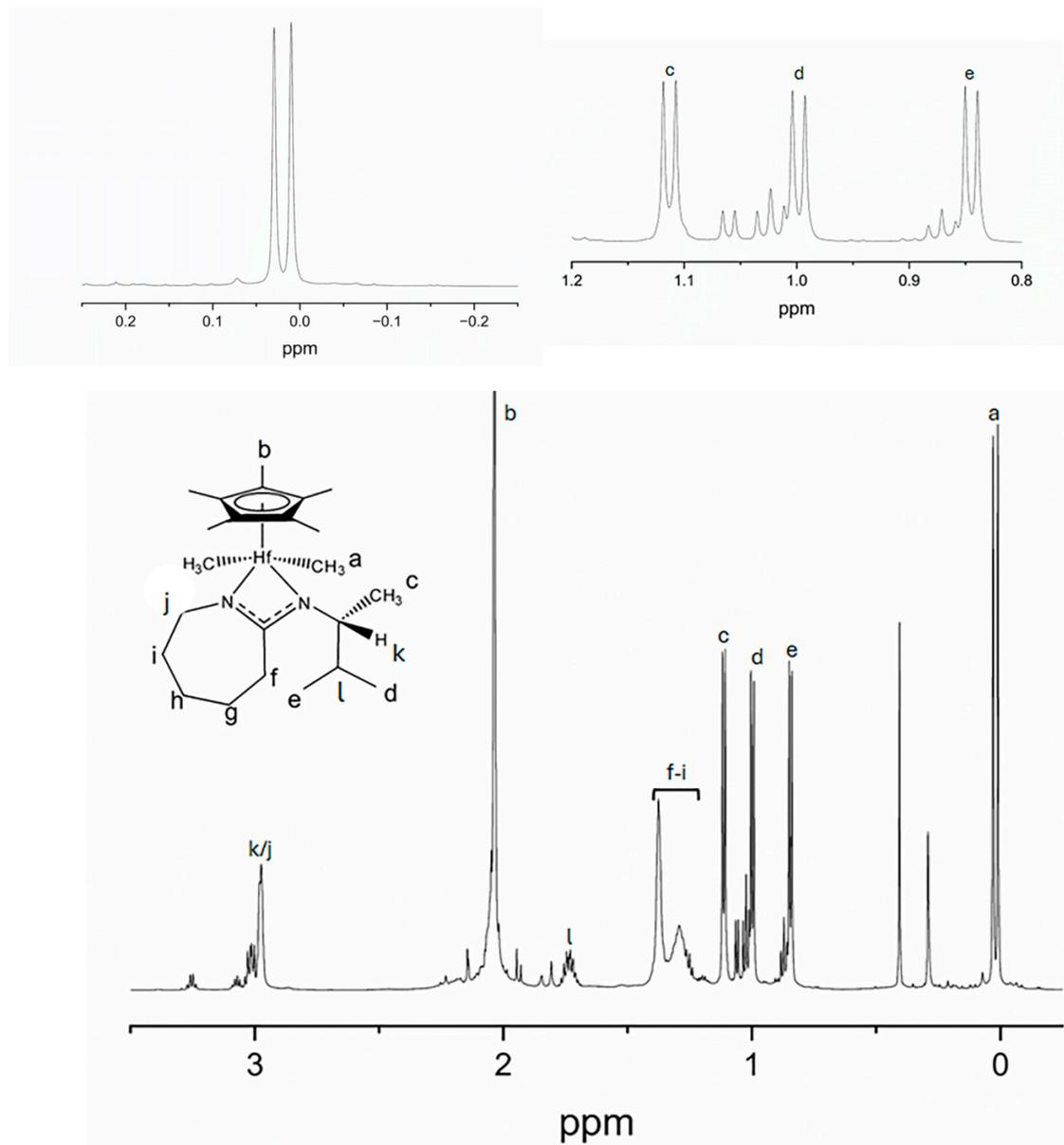


Figure S5. Partial ^1H NMR (400 MHz, C_6D_6 , 25°C) spectrum of $(R_C, R_{\text{Hf}})\text{-2}$.

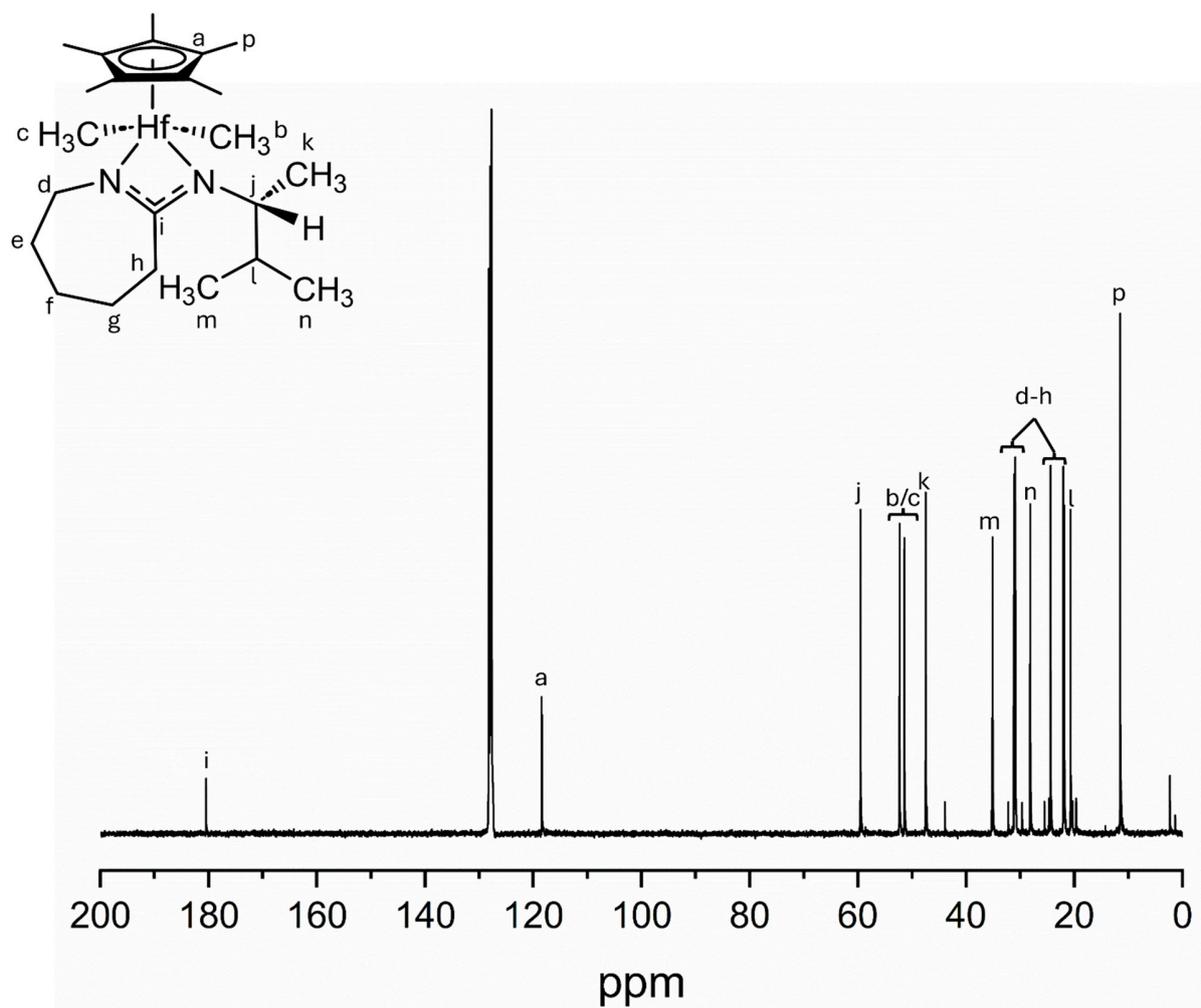


Figure S6. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, C_6D_6 , 25°C) spectrum of $(R_C, R_{\text{Hf}})\text{-2}$.

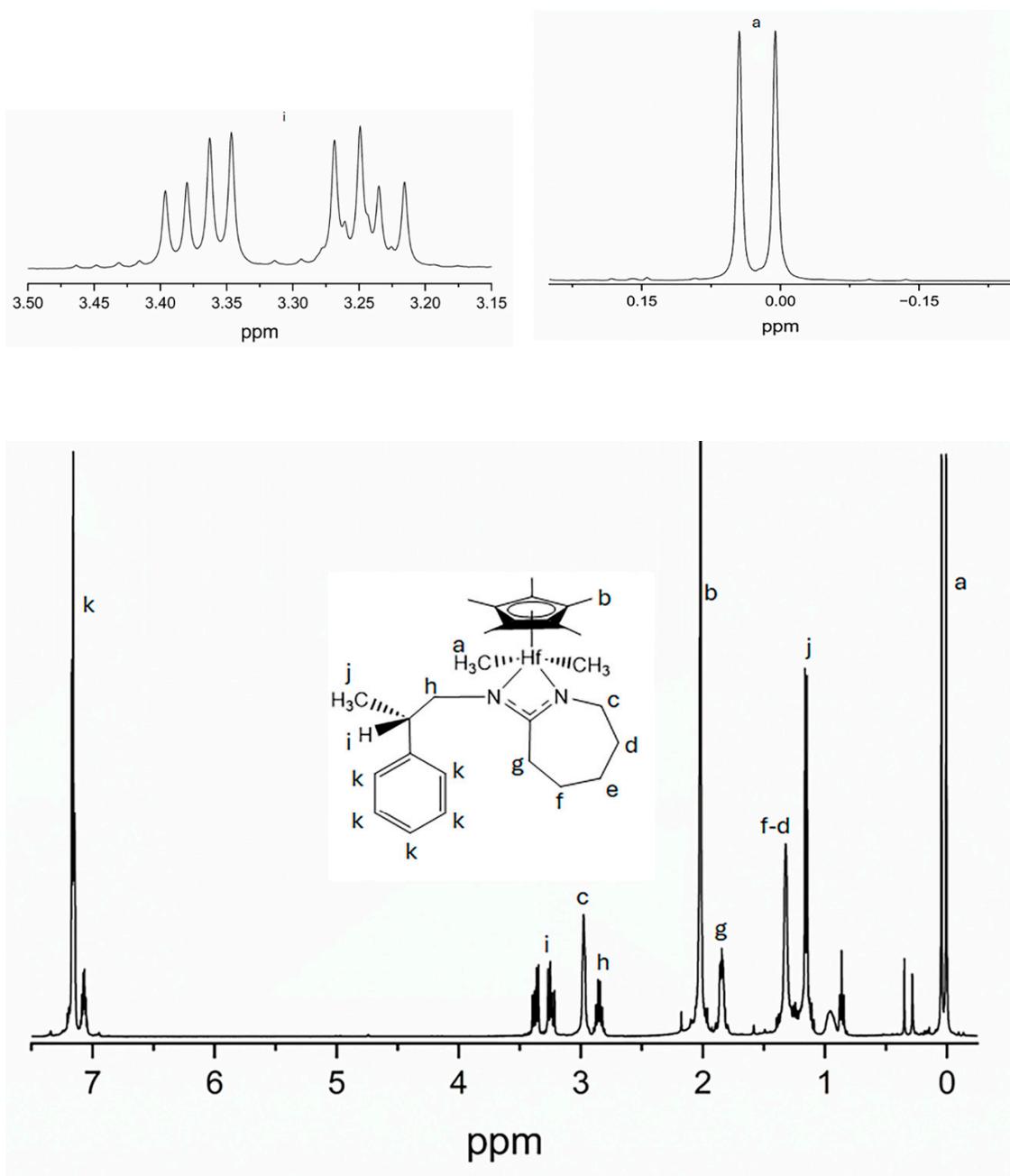


Figure S7. Partial ^1H NMR (400 MHz, C_6D_6 , 25°C) spectrum of (S_C, S_{Hf}) -3.

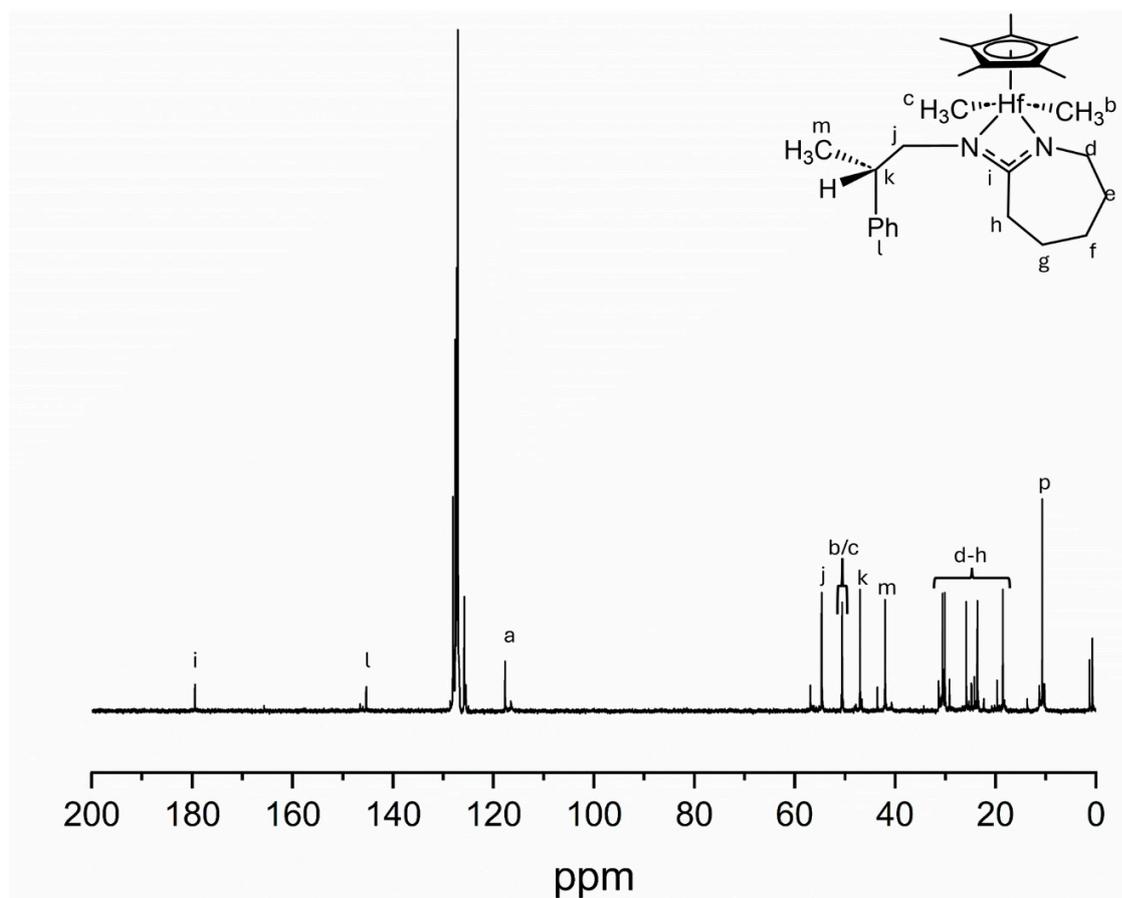


Figure S8. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, C_6D_6 , 25°C) spectrum of $(S_C, S_{Hf})\text{-3}$.

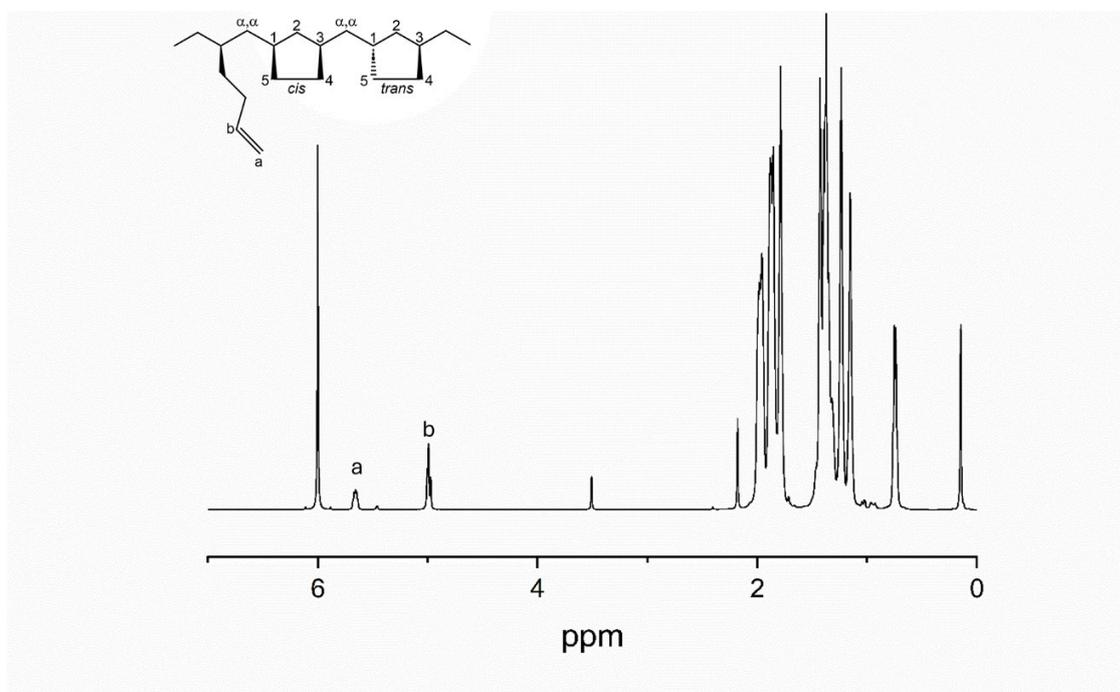


Figure S9. ¹H NMR (800 MHz, TCE-*d*₂, 110°C) spectrum of PMCP Run 1.

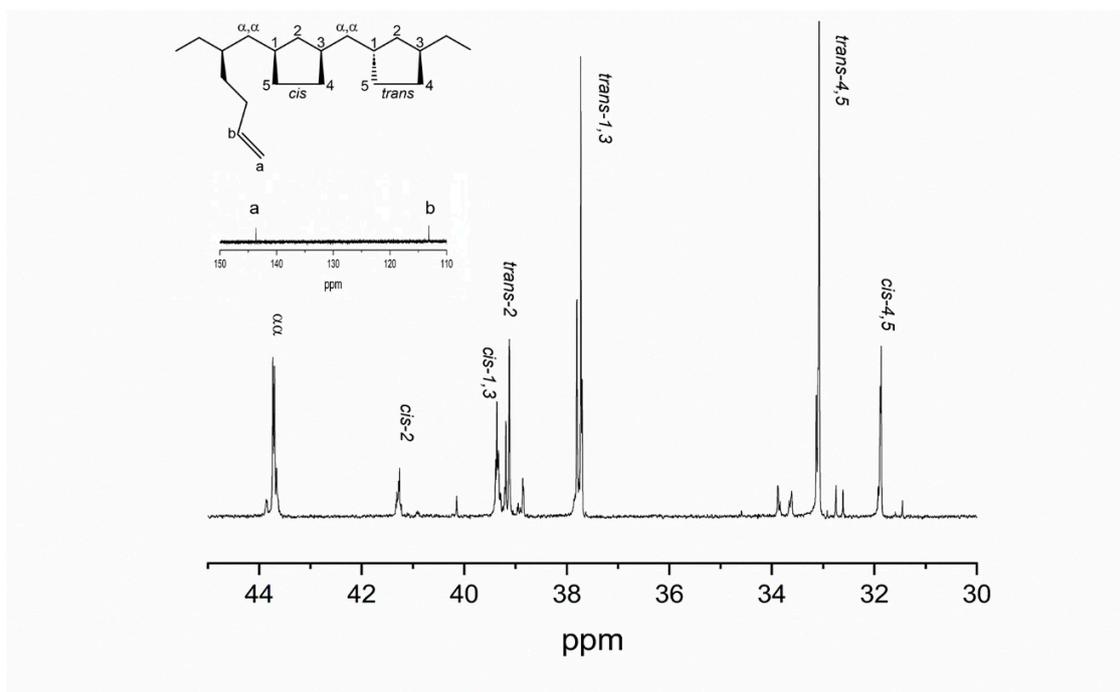


Figure S10. Partial $^{13}\text{C}\{^1\text{H}\}$ NMR (200 MHz, $\text{TCE-}d_2$, 110°C) spectrum of PMCP Run 1.

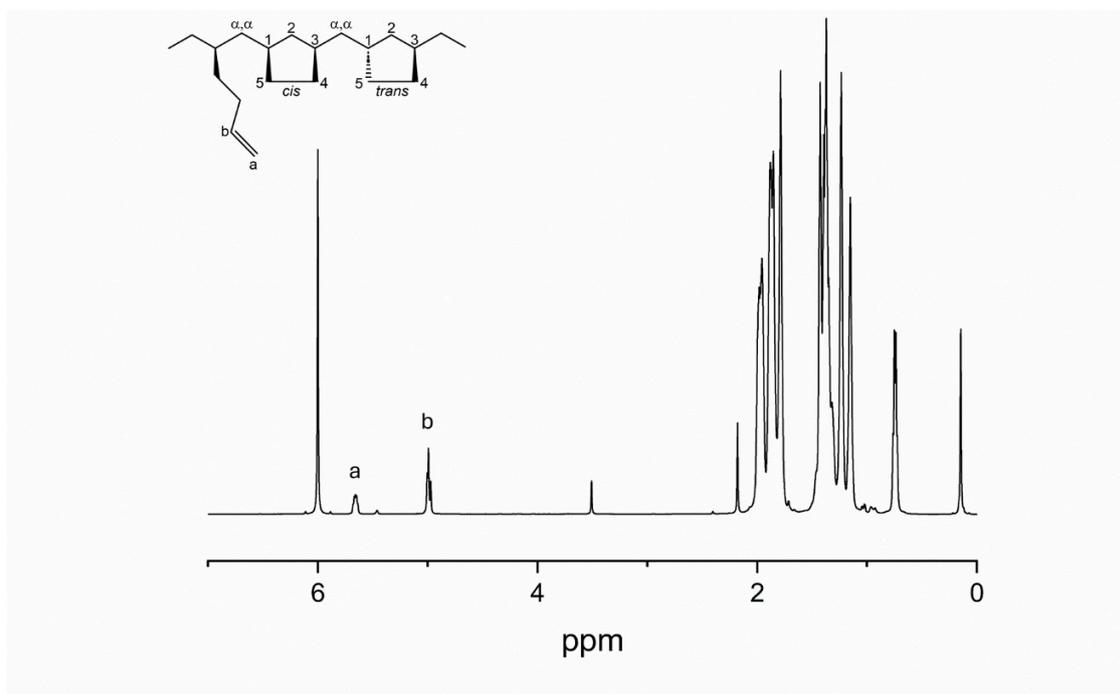


Figure S11. ^1H NMR (800 MHz, $\text{TCE-}d_2$, 110°C) spectrum of PMCP Run 2.

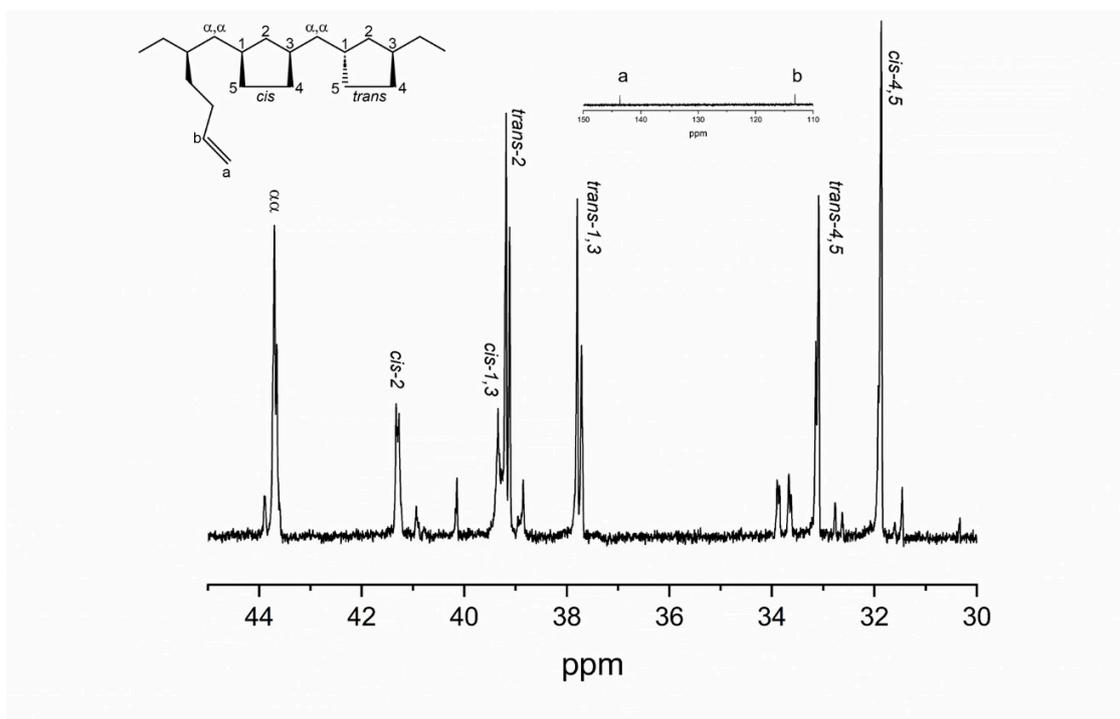


Figure S12. Partial $^{13}\text{C}\{^1\text{H}\}$ NMR (200 MHz, $\text{TCE-}d_2$, 110°C) spectrum of PMCP Run 2.

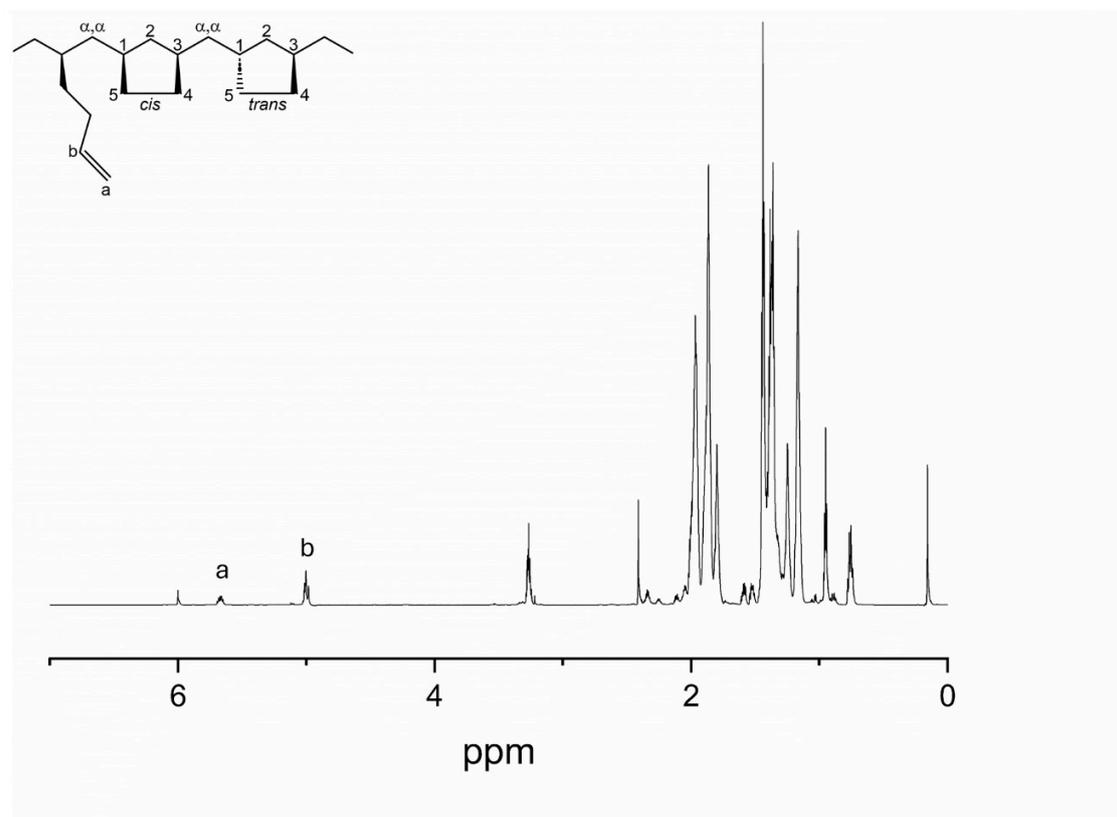


Figure S13. ^1H NMR (800 MHz, $\text{TCE-}d_2$, 110°C) spectrum of PMCP Run 3.

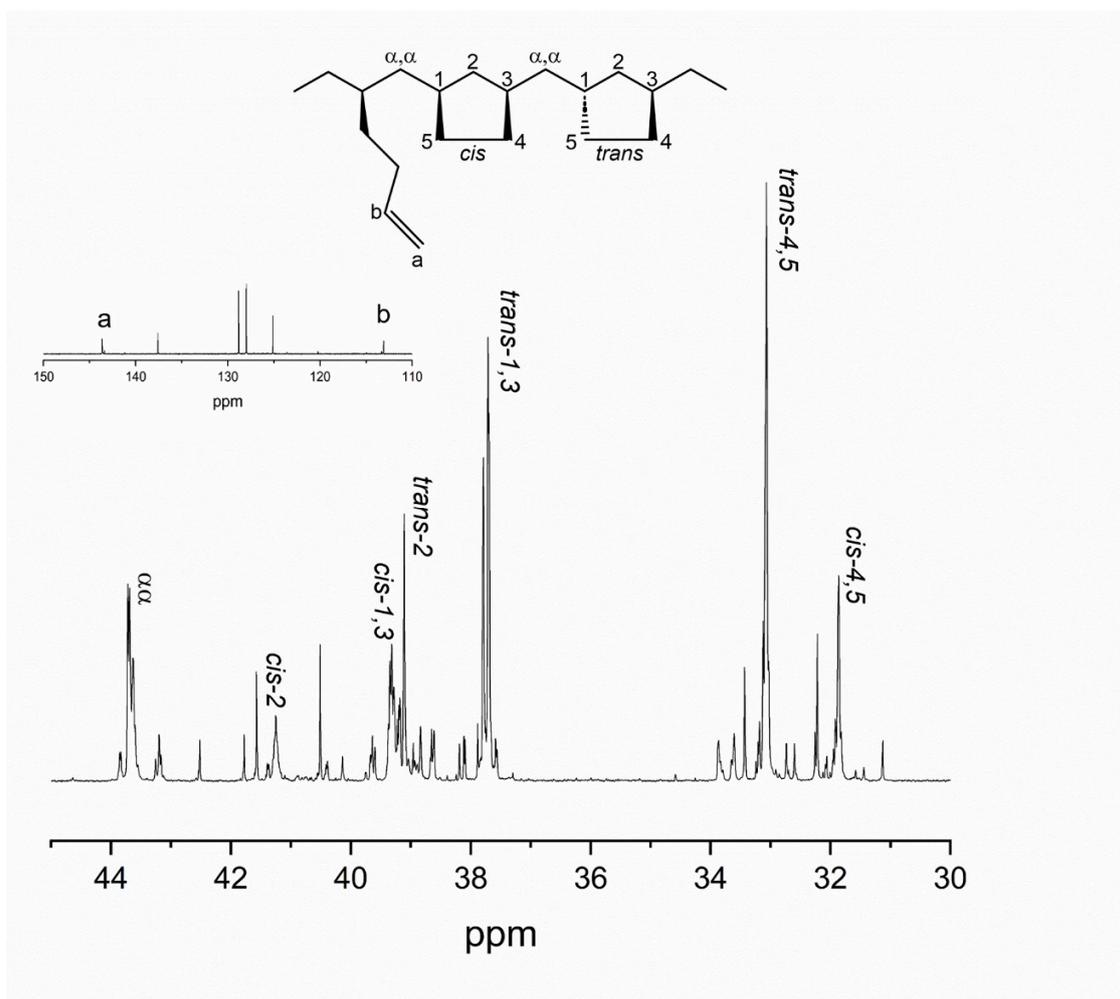


Figure S14. Partial $^{13}\text{C}\{^1\text{H}\}$ NMR (200 MHz, TCE $-d_2$, 110°C) spectrum of PMCP **Run 3**.

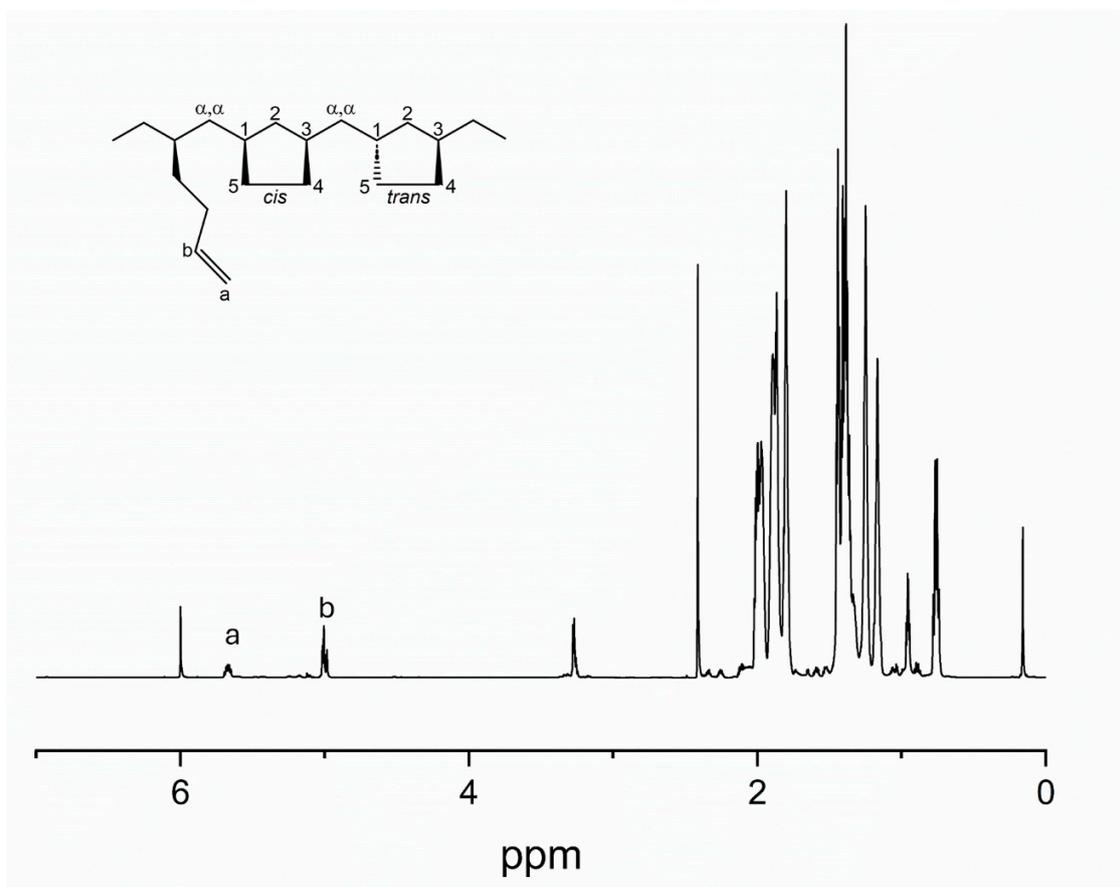


Figure S15. ¹H NMR (800 MHz, TCE -*d*₂, 110°C) spectrum of PMCP **Run 4**.

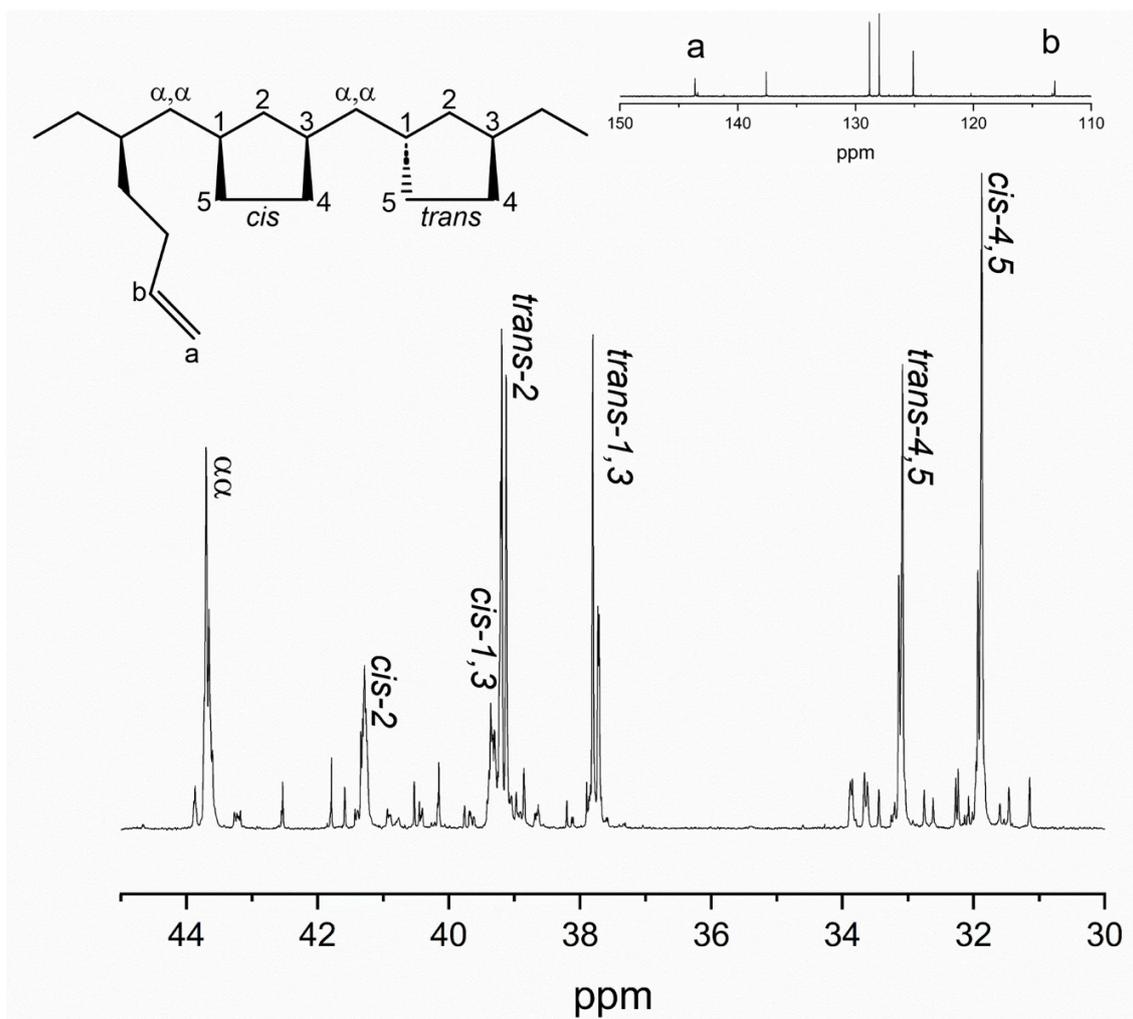
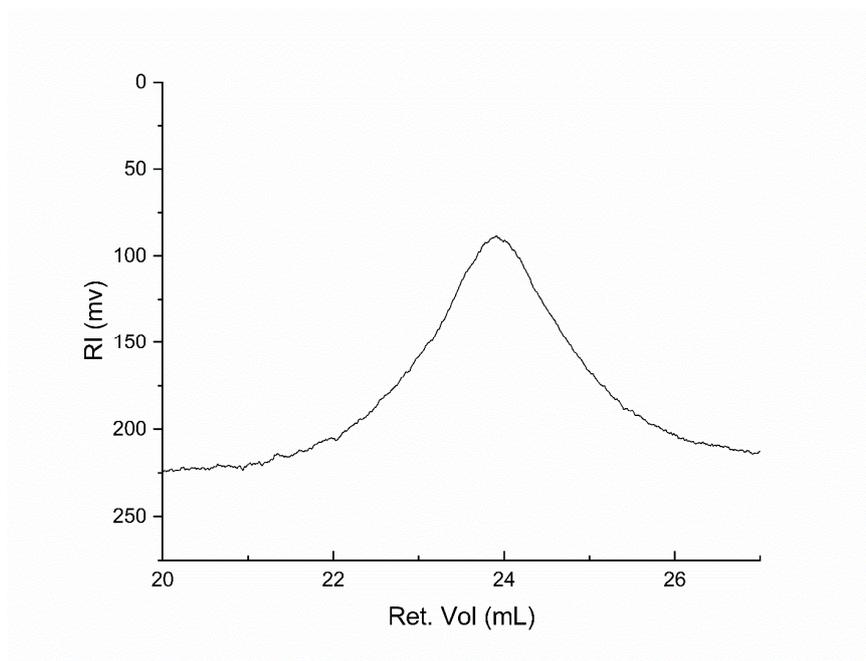
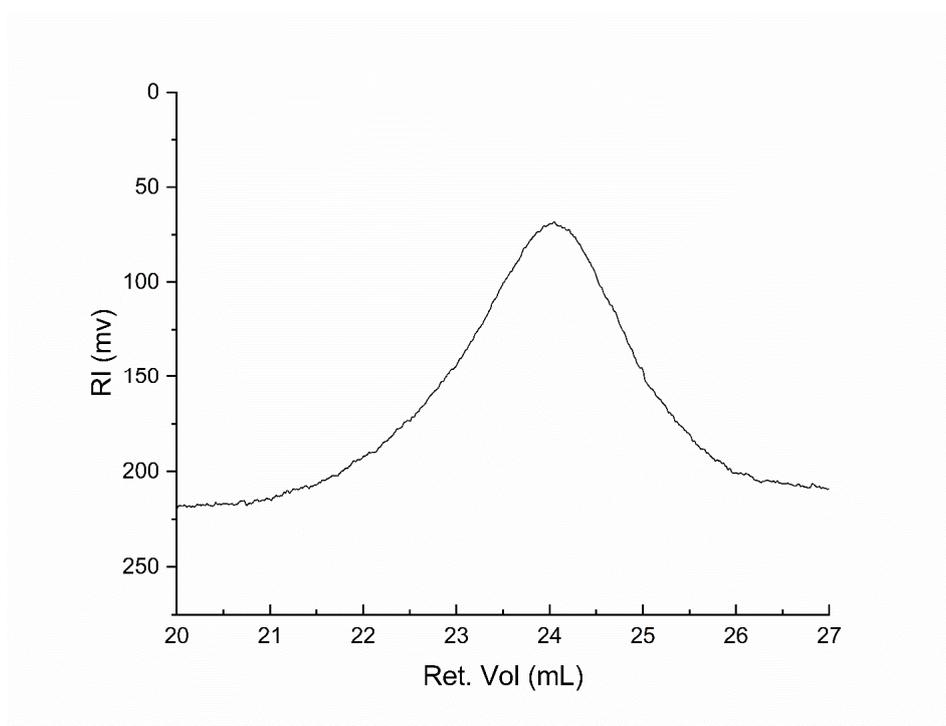


Figure S16. Partial $^{13}\text{C}\{^1\text{H}\}$ NMR (200 MHz, TCE $-d_2$, 110°C) spectrum of PMCP **Run 4**.

GPC Traces**Figure S17.** GPC Trace of PMCP from **Run 1**.**Figure S18.** GPC Trace of PMCP from **Run 2**.

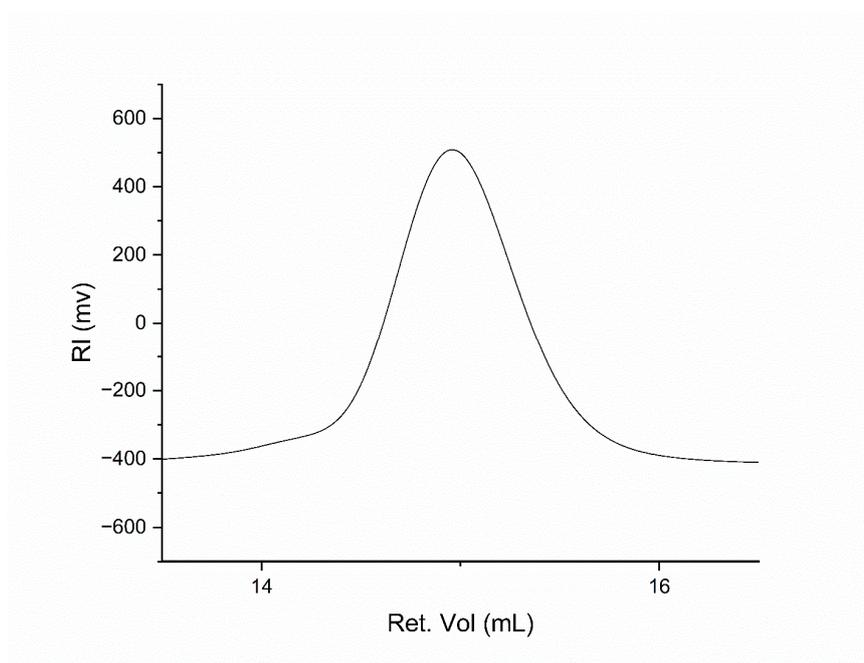


Figure S19. GPC Trace of I-PMCP from **Run 3.**

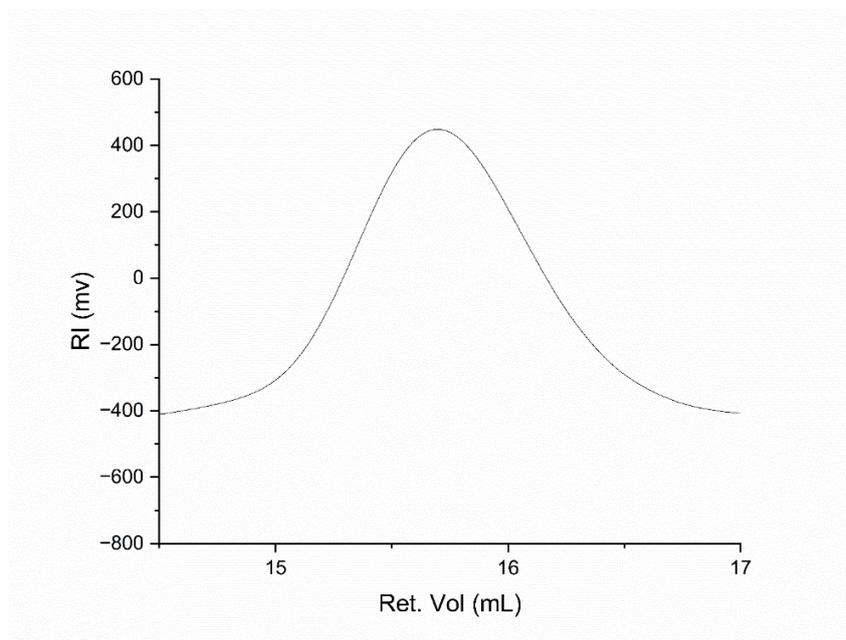


Figure S20. GPC Trace of I-PMCP from **Run 4.**

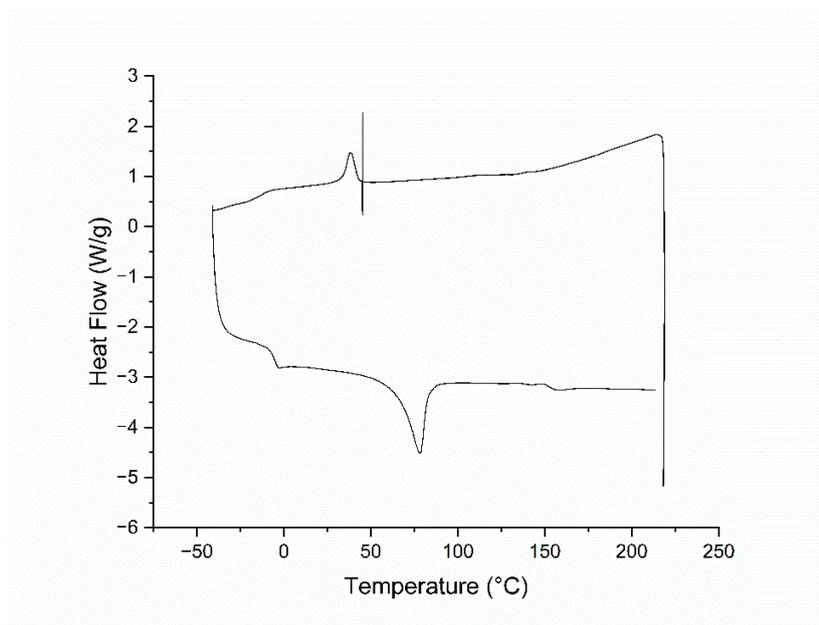
DSC Traces

Figure S21. DSC trace of PMCP **Run 1** (exo up). Heat/cool/heat cycle with heating ramp of 20 °C/min from -40 to 220°C cooling ramp of 5 °C/min from 220 to -40 °C.

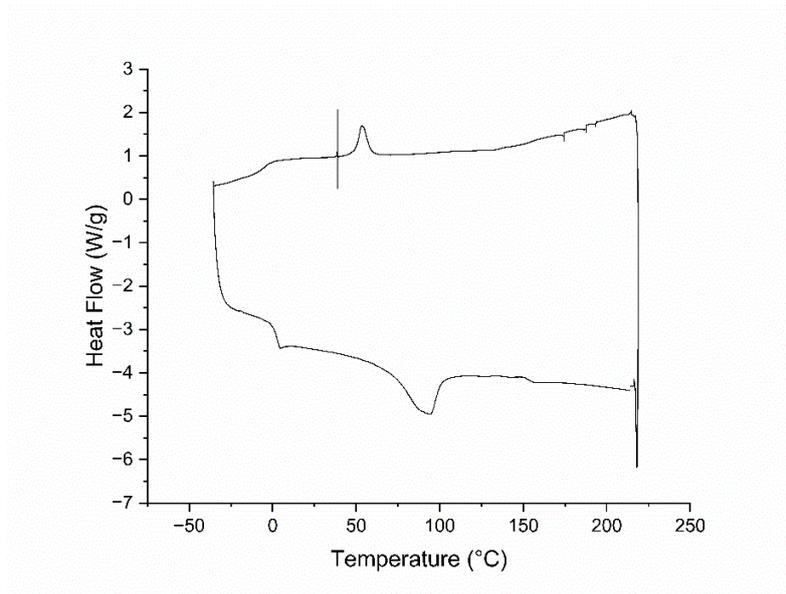


Figure S22. DSC trace of PMCP **Run 2** (exo up). Heat/cool/heat cycle with heating ramp of 20 °C/min from -40 to 220°C cooling ramp of 5 °C/min from 220 to -40 °C.

Crystallographic Information: A suitable single crystals of $C_{27}H_{42}N_2Hf$ was selected and measured on a Bruker D8Venture w/ PhotonIII diffractometer [1]. The crystal was kept at 295(2) K during data collection. The integral intensity were correct for absorption using SADABS software [2] using multi-scan method. Resulting minimum and maximum transmission are 0.275 and 0.431 respectively. The structure was solved with the ShelXT (Sheldrick, 2015a) [3] program and refined with the ShelXL (Sheldrick, 2015c) program using least-square minimisation [4]. Number of restraints used = 0.

Crystal Data for $C_{27}H_{42}N_2Hf$ ($M=573.11$ g/mol): orthorhombic, space group $P2_12_12_1$ (no. 19), $a = 8.3516(8)\text{\AA}$, $b = 16.8771(15)\text{\AA}$, $c = 18.5296(17)\text{\AA}$, $V = 2611.8(4)\text{\AA}^3$, $Z = 4$, $T = 295(2)\text{K}$, $\mu(\text{MoK}\alpha) = 4.009\text{ mm}^{-1}$, $D_{\text{calc}} = 1.458\text{ g/cm}^3$, 351440 reflections measured ($4.828 \leq 2\theta \leq 60.15$), 7660 unique ($R_{\text{int}} = 0.1021$, $R_{\text{sig}} = 0.0210$) which were used in all calculations. The final R_1 was 0.0178 ($I > 2\sigma(I)$) and wR_2 was 0.0372 (all data).

Refinement details: Integrated as crystal with 2 components tilted by 5 deg. to each other. Only peaks containing major component were included in the refinement with equivalent reflections merged except Friedel pairs.

Table S1 Crystal data and structure refinement for (S_C, S_{Hf})-3.

| | |
|---------------------|---------------------|
| Identification code | UM4208 |
| Empirical formula | $C_{27}H_{42}N_2Hf$ |
| Formula weight | 573.11 |
| Temperature/K | 295(2) |
| Crystal system | orthorhombic |
| Space group | $P2_12_12_1$ |
| $a/\text{\AA}$ | 8.3516(8) |
| $b/\text{\AA}$ | 16.8771(15) |
| $c/\text{\AA}$ | 18.5296(17) |
| $\alpha/^\circ$ | 90 |
| $\beta/^\circ$ | 90 |
| $\gamma/^\circ$ | 90 |

| | |
|---|---|
| Volume/Å ³ | 2611.8(4) |
| Z | 4 |
| $\rho_{\text{calc}}/\text{cm}^3$ | 1.458 |
| μ/mm^{-1} | 4.009 |
| F(000) | 1160.0 |
| Crystal size/mm ³ | 0.51 × 0.27 × 0.21 |
| Radiation | MoK α ($\lambda = 0.71073$) |
| 2 Θ range for data collection/° | 4.828 to 60.15 |
| Index ranges | -11 ≤ h ≤ 11, -23 ≤ k ≤ 23, -25 ≤ l ≤ 26 |
| Reflections collected | 351440 |
| Independent reflections | 7660 [R _{int} = 0.1021, R _{sigma} = 0.0210] |
| Data/restraints/parameters | 7660/0/280 |
| Goodness-of-fit on F ² | 1.056 |
| Final R indexes [I ≥ 2 σ (I)] | R ₁ = 0.0178, wR ₂ = 0.0368 |
| Final R indexes [all data] | R ₁ = 0.0194, wR ₂ = 0.0372 |
| Largest diff. peak/hole / e Å ⁻³ | 0.68/-0.34 |

A suitable single crystal of C₂₃H₄₂N₂Hf was selected and measured on a Bruker D8Venture w/ PhotonIII diffractometer [1]. The crystal was kept at 170(2) K during data collection. The integral intensity were correct for absorption using SADABS software [2] using multi-scan method. Resulting minimum and maximum transmission are 0.217 and 0.377 respectively. The structure was solved with the ShelXT (Sheldrick, 2015a) [3] program and refined with the ShelXL (Sheldrick, 2015c) program using least-square minimisation [4]. Number of restraints used = 0.

Crystal structure determination:

Crystal Data for C₂₃H₄₂N₂Hf ($M = 525.07$ g/mol): orthorhombic, space group P2₁2₁2₁ (no. 19), $a = 8.7328(5)$ Å, $b = 9.5639(5)$ Å, $c = 28.2086(15)$ Å, $V = 2356.0(2)$ Å³, $Z = 4$, $T = 170(2)$ K, $\mu(\text{MoK}\alpha) = 4.436$ mm⁻¹, $D_{\text{calc}} = 1.480$ g/cm³, 150483 reflections measured ($4.498 \leq 2\Theta \leq 59.998$), 6879

unique ($R_{\text{int}} = 0.0719$, $R_{\text{sig}} = 0.0187$) which were used in all calculations. The final R_1 was 0.0165 ($I > 2\sigma(I)$) and wR_2 was 0.0371 (all data).

Table S2 Crystal data and structure refinement for (R_C, R_{Hf})-2.

| | |
|---|--|
| Identification code | UM4193 |
| Empirical formula | $\text{C}_{23}\text{H}_{42}\text{N}_2\text{Hf}$ |
| Formula weight | 525.07 |
| Temperature/K | 170(2) |
| Crystal system | orthorhombic |
| Space group | $P2_12_12_1$ |
| $a/\text{\AA}$ | 8.7328(5) |
| $b/\text{\AA}$ | 9.5639(5) |
| $c/\text{\AA}$ | 28.2086(15) |
| $\alpha/^\circ$ | 90 |
| $\beta/^\circ$ | 90 |
| $\gamma/^\circ$ | 90 |
| Volume/ \AA^3 | 2356.0(2) |
| Z | 4 |
| $\rho_{\text{calc}}/\text{cm}^3$ | 1.480 |
| μ/mm^{-1} | 4.436 |
| $F(000)$ | 1064.0 |
| Crystal size/ mm^3 | $0.45 \times 0.37 \times 0.22$ |
| Radiation | $\text{MoK}\alpha$ ($\lambda = 0.71073$) |
| 2Θ range for data collection/ $^\circ$ | 4.498 to 59.998 |
| Index ranges | $-12 \leq h \leq 12, -13 \leq k \leq 13, -39 \leq l \leq 39$ |

| | |
|--|--|
| Reflections collected | 150483 |
| Independent reflections | 6879 [$R_{\text{int}} = 0.0719$, $R_{\text{sigma}} = 0.0187$] |
| Data/restraints/parameters | 6879/0/246 |
| Goodness-of-fit on F^2 | 1.150 |
| Final R indexes [$I \geq 2\sigma(I)$] | $R_1 = 0.0165$, $wR_2 = 0.0369$ |
| Final R indexes [all data] | $R_1 = 0.0175$, $wR_2 = 0.0371$ |
| Largest diff. peak/hole / $e \text{ \AA}^{-3}$ | 0.96/-0.74 |
| Flack parameter | -0.007(3) |