Supporting Information

Catalytic Cyclization and Competitive Deactivation with Ru($P^R_2N^{R'}_2$) Complexes

James M. Stubbs, John-Paul J. Bow, Richard J. Hazlehurst, and Johanna M. Blacquiere*

Department of Chemistry
University of Western Ontario
London, Ontario, Canada, N6A 5B7

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I General Procedures, Materials and Instrumentation.

All reactions were manipulated under N_2 using standard Schlenk or glovebox techniques. All glassware was oven dried prior to use. Triphenylphosphine oxide (99%) was obtained from Alfa Aesar. Bis(diphenylphosphino)propane (dppp; 98%), pyrene (98%), 2-ethynylbenzyl alcohol (99%), and 4-pentyn-1-ol (97%) were obtained from Sigma-Aldrich. Thallium hexafluorophosphate (97%) was obtained from Strem. Chloroform-*d* (99.8%) and acetone-d_6 (99.9%) were obtained from Cambridge Isotope Laboratories. $[Ru(Cp)(MeCN)_3]PF_6$,[1] $P^{Ph}_2N^{Bn}_2$,[2] $[Ru(Cp)(P^{tBu}_2N^{Bn}_2)(NCMe)]PF_6$,[3] $Ru(Cp)(P^{tBu}_2N^{Bn}_2)Cl$[4] and 2-ethynyl-5-methoxybenzyl alcohol[5] were synthesized following literature procedures. $P^{tBu}_2N^{Bn}_2$ was used as gifted. Dry and degassed solvents were obtained from an Innovative Technology 400-5 Solvent Purification System and stored over 4 Å molecular sieves (Fluka and activated at 150 ˚C for 12 h) under N_2 unless otherwise noted. Acetone was dried with Cs_2CO_3 and degassed by bubbling with N_2. Chloroform-*d* was dried with 4 Å molecular sieves and degassed by bubbling with N_2. All other chemicals were used as received.

All NMR spectra were recorded on either an Inova 600 MHz or Mercury 400 MHz instrument. ^1H and ^{13}C spectra acquired were referenced internally against the residual solvent signal to TMS at 0 ppm. ^{31}P spectra were referenced externally to 85% phosphoric acid at 0.00 ppm. Infrared spectra were collected on solid samples using a PerkinElmer UATR TWO FTIR spectrometer. Elemental analysis was performed by Laboratoire d'Analyse Élémentaire de l'Université de Montréal. MALDI-TOF mass spectra were collected using an AB Sciex 5800 TOF/TOF mass spectrometer using pyrene as the matrix in a 20:1 molar ratio with the sample. The instrument is equipped with a 349 nm OptiBeam On-Axis laser. The laser pulse rate was 400 Hz and data were collected in reflectron positive mode.

Reflectron mode was externally calibrated at 50 ppm mass tolerance. Each mass spectrum was collected as a sum of 500 shots.

[Ru(Cp)($P^{Ph}_2N^{Bn}_2$)(NCCH$_3$)]PF$_6$ (1b). [RuCp(NCMe)$_3$]PF$_6$ (461 mg, 1.06 mmol, 1 equiv.) and $P^{Ph}_2N^{Bn}_2$ (511 mg, 1.06 mmol, 1 equiv.) were combined in a 100 mL Schlenk flask with acetonitrile (5 mL) and heated at 70°C for 4 h. The ligand solubilizes on heating causing the solution to turn yellow. After cooling to room temperature, the solvent was removed under vacuum to afford a yellow air-sensitive powder. Yield: 841 mg (95%). ^{1}H (600 MHz, $CDCl_3$): δ 7.64-58 (m, Ph-*H*, 4H), 7.53-7.46 (m, Ph-*H*, 6H), 7.38-7.17 (m, Ph-*H*, 10H), 4.71 (s, Cp-*H*, 5H), 3.81 (s, PhC*H*$_2$N, 2H), 3.66 (s, PhC*H*$_2$N, 2H), 3.22-3.05 (m, PC*H*$_2$N, 4H), 2.98-2.88 (m, PC*H*$_2$N, 2H), 2.81-2.73 (m, PC*H*$_2$N, 2H), 2.26 (s, RuNCC*H*$_3$, 3H). ^{31}P{^{1}H} (243 MHz, $CDCl_3$): δ 38.4 (s, Ru*P*), –144.2 (sept, $^1J_{P\text{-}F}$ = 714 Hz, *P*F$_6$). ^{13}C{^{1}H} (151.5 MHz, $CDCl_3$): δ 137.0 (s, CH_2*C*-Ar), 136.5 (s, CH_2*C*-Ar), 134.3 (dd, $^1J_{C\text{-}P}$ = 21.4 Hz, $^3J_{C\text{-}P}$ = 21.4 Hz, P*C*-Ar), 131.6-128.2 and 128.0 (*C*-Ar), 128.1 (s, RuN*C*CH$_3$), 81.7 (s, *Cp*), 65.5 (s, N*C*H$_2$Ph), 52.5 (dd, $^1J_{C\text{-}P}$ = 18.1 Hz, $^3J_{C\text{-}P}$ = 18.1 Hz, N*C*H$_2$PPh), 51.7 (dd, $^1J_{C\text{-}P}$ = 18.1 Hz, $^3J_{C\text{-}P}$ = 18.1 Hz, N*C*H$_2$PPh), 4.2 (s, *C*H$_3$CNRu). Anal. Calc. for $C_{37}H_{40}F_6N_3P_3Ru$•0.25 hexanes: C, 54.00; H, 5.12; N, 4.91. Found: C, 54.34; H, 4.93; N, 4.76. MALDI MS (pyrene matrix): Calc. m/z 649.1 [RuCp($P^{Ph}_2N^{Bn}_2$)]$^+$, Obs. m/z 649.2.

RuCp(dppp)(NCCH$_3$)]PF$_6$ (3). [RuCp(NCMe)$_3$]PF$_6$ (81 mg, 0.19 mmol, 1 equiv.) and dppp (77 mg, 0.19 mmol, 1 equiv.) were combined in a pre-weighed vial in the glovebox with acetonitrile (5 mL) and stirred for 4 h at RT causing the solution to turn yellow from orange. The solvent was removed under vacuum to give a pure yellow solid. Yield: 145 mg (96%). ^{1}H (600 MHz, $CDCl_3$): δ 7.48-7.40 (m, Ph-*H*, 12H), 7.25 (dd, $^3J_{Hg\text{-}P}$ = 7.2 Hz, $^3J_{Hg\text{-}Hh}$ = 7.2 Hz, Ph-*H*, 4H), 7.12 (m, Ph-*H*, 4H), 4.60 (s, *H*-Cp, 5H), 2.62 (m, P-C*H*H′, 2H), 2.45 (m, CH$_2$-C*H*H′, 1H), 2.36 (s, C*H*$_3$CN, 3H), 2.30 (m, P-CH*H′*, 2H), 1.71 (m, P-CH*H′*, 1H). ^{31}P{^{1}H} (242.9 MHz, $CDCl_3$): δ 37.4 (s, *P*-C), -144.4 (sept, $^1J_{P\text{-}F}$ = 714 Hz, *P*F$_6$). ^{13}C{^{1}H} (150.9 MHz, $CDCl_3$): δ 138.6 (m, P-*C*$_{Ar}$), 137.2 (m, P-*C*$_{Ar}$), 132.5 (d, $^3J_{C\text{-}P}$ = 5.7 Hz, *m*-*C*$_{Ar}$), 132.5 (d, $^3J_{C\text{-}P}$ = 5.7 Hz, *m*-*C*$_{Ar}$), 131.7 (d, $^3J_{C\text{-}P}$ = 5.3 Hz, *m*-*C*$_{Ar}$), 131.7 (d, $^3J_{C\text{-}P}$ = 5.3 Hz, *m*-*C*$_{Ar}$), 130.2 (s, *p*-*C*$_{Ar}$), 130.1 (s, *p*-*C*$_{Ar}$), 129.0 (d, $^2J_{C\text{-}P}$ = 5.0 Hz, *o*-C$_{Ar}$), 129.0 (d, $^2J_{C\text{-}P}$ = 5.0 Hz, *o*-C$_{Ar}$), 129.0 (s, *C*N), 128.6 (d, $^2J_{C\text{-}P}$ = 5.0 Hz, *o*-*C*$_{Ar}$), 82.8 (s, *Cp*), 26.9 (dd, $^1J_{C\text{-}P}$ = 15.3 Hz, $^3J_{C\text{-}P}$ = 15.3 Hz, *P*-*C*H$_2$), 20.8 (s, CH$_2$-*C*H$_2$), 4.4 (s, *C*H$_3$). Anal. Calc. for $C_{34}H_{34}F_6NP_3Ru$•0.08 dppp: C, 54.45; H, 4.56; N, 1.76. Found: C, 54.84; H, 4.60; N, 1.43. MALDI MS (pyrene matrix): Calc. m/z 579.0 [RuCp(dppp)]$^+$, Obs. m/z 579.1.

Attempted Synthesis of [Ru(Cp)($P^{tBu}_2N^{Bn}_2$)(-C=CHC$_6$H$_4$OH)]PF$_6$ (7a). [Ru(Cp)($P^{tBu}_2N^{Bn}_2$)(NCMe)]PF$_6$ (14 mg, 0.018 mmol, 1.5 mM, 1 equiv.) and 2-ethynylbenzyl alcohol (239 mg, 1.81 mmol, 150 mM, 100 equiv.) were combined in a 100 mL Schlenk with acetone (12 mL) and heated to 54 ˚C for 5 days. ^{31}P{^{1}H} NMR spectra indicate a maximum conversion of 77% of **7a** with the balance being **1a**. The reaction was cooled and the solvent was removed under vacuum to produce a brownish yellow solid. The solid was washed with hexanes (3 × 15 mL) and dried under vacuum. Analysis by ^{31}P{^{1}H} NMR spectroscopy in $CDCl_3$ revealed **7a** (45%), **1a** (11%) and three unknown species in ca. 45%. After 24 h these quantities changed to: **7a** (25%), **1a** (10%) and three unknown species in ca. 65%. This relatively fast decomposition prevents isolation of clean **7a** and precludes characterization of this species free of organic substrate/product by correlation NMR spectroscopy.

In Situ Characterization of [Ru(Cp)($P^{tBu}_2N^{Bn}_2$)(-C=CHC$_6$H$_4$OH)]PF$_6$ (7a). In a glovebox, substrate **4a** (171 mg, 1.30 mmol) and catalyst **1a** (10 mg, 0.013 mmol) were combined in a vial in acetone-d_6 (1 mL) with a stir bar. The initial concentrations of the species were: **4a** (1.30 M) and **1a** (0.013 mM). The vial was heated and stirred for 7 h at 54 ˚C. ^{31}P{^{1}H} NMR spectroscopy revealed one new signal found at 71.2 ppm in 84% yield with the balance being **1a**. ^{1}H, ^{31}P{^{1}H}, ^{1}H–^{1}H COSY, and ^{1}H–^{13}C gHMBCAD NMR spectra were collected. Diagnostic ^{1}H and ^{13}C signals are identified to support assignment as **7a**. ^{1}H (600 MHz, $(CD_3)_2CO$): δ 7.48 (Ru–C$_α$(NCH$_2$Ph)=C*H*Ar),

4.85 (Ru–C_α(NCH_2Ph)=CHAr). $^{13}C\{^1H\}$ (150.9 MHz, $(CD_3)_2CO$): δ 196.9 (Ru–C_α), 62.0 (Ru–C_α(NCH$_2$Ph)=CHAr). $^{31}P\{^1H\}$ (242.9 MHz, $(CD_3)_2CO$): δ 71.2 (s, Ru-*P*), –144.4 (sept, $^1J_{P\text{-}F}$ = 714 Hz, PF_6).

Representative Procedure for Catalytic Cyclization of 4a. In a glovebox, the following stock solutions were prepared: 2-Ethynylbenzyl alcohol **4a** (159 mg, 1.20 mmol, 0.300 M) and dimethyl terephthalate (38 mg, 0.19 mmol, 0.049 M) in acetone (4.010 mL); **1a** (6 mg, 0.007 mmol, 6 mM) in acetone (1.153 mL); **1b** (6 mg, 0.007 mmol, 6 mM) in acetone (1.118 mL); **3** (7 mg, 0.009 mmol, 6 mM) in acetone (1.503 mL). Four sets (A-D) of 5 vials (20 vials total) containing stir bars were charged with the **4a**/dimethyl terephthalate stock solution (250 μL) and additional acetone (125 μL). To each vial of set A was added the **1a** stock solution (125 μL) giving a final volume of 500 μL. To each vial of set B was added the **1b** stock solution (125 μL) giving a final volume of 500 μL. To each vial of set D, NEt_3 was added (6.28 μL). To each vial of sets C and D was added the **3** stock solution (125 μL) giving a final volume of 500 μL. The final concentrations for all vials were 0.150 M in substrate. A final vial was charged with substrate/internal standard stock solution (100 μL) for use as the time = 0 sample, required for accurate quantification of substrate and product. The vials were capped and removed from the glove box and heated to 40 °C (sets A-D) with stirring. After 0.167, 0.5, 1, 6, and 24 hours one vial from each of the sets was removed from heat, cooled, and exposed to air to quench. The solvent was then removed in vacuo; the remaining residue was dissolved in $CDCl_3$ and analyzed by ^1H NMR spectroscopy. Substrate consumption and product formation was determined relative to the internal standard (dimethyl terephthalate). The product **5a** has been previously reported[6] and ^1H NMR spectra match these reported values.

In Situ Monitoring of Ru Species During Catalysis. In a glovebox, the following stock solutions were prepared: **4a** (1.60 M, 1.13 mmol) with dimethyl terephthalate (0.388 M, 0.275 mmol) in acetone-d_6 in a vial with a septum cap; **1a** (35 mM, 0.011 mmol) with triphenylphosphine oxide (35 mM, 0.011 mmol) in acetone-d_6 in a septum capped NMR tube. A $^{31}P\{^1H\}$ NMR spectrum was acquired at time = 0. The INOVA 600 NMR spectrometer was heated to the appropriate temperature (313 K or 323 K). The substrate stock solution (0.450 mL) was injected in the septum capped NMR tube. The NMR tube was shaken and immediately placed in the instrument. ^1H and $^{31}P\{^1H\}$ NMR spectra were collected every 5 minutes for 90 minutes. The initial concentrations of the species were: **4a** (960 mM); dimethyl terephthalate (233 mM); **1a** (14 mM); $OPPh_3$ (14 mM).

Representative Procedure for Performing Cyclization of 4 with [Ru(Cp)($P^{tBu}_2N^{Bn}_2$)]PF_6. $TlPF_6$ was used in this procedure. Thallium is extremely TOXIC and due care is needed.[7] Solid waste and solution waste contaminated with thallium were placed in a separate containers marked for thallium waste. Glassware contaminated with thallium were heated in water to dissolve residual thallium salts. In a glovebox, the following stock solutions were prepared: 2-Ethynylbenzyl alcohol **4a** (226 mg, 1.70 mmol, 0.300 M) and dimethyl terephthalate (50 mg, 0.25 mmol, 0.050 M) in acetone (5.710 mL); Ru(Cp)($P^{tBu}_2N^{Bn}_2$)Cl (3.7 mg, 0.006 mmol, 3 mM) and $TlPF_6$ (4.0 mg, 0.011, 6 mM) in acetone (1.915 mL). Four sets (A-D) of 5 vials (20 vials total) containing stir bars were charged with the **4a**/dimethyl terephthalate stock solution (150 μL). To each vial in set A and B the **1a** stock solution (150 μL) was added giving a final volume of 300 μL. To each vial in set C and D was added the **1a** stock solution (15 μL) and acetone (135 μL) giving a final volume of 300 μL. The final concentrations for all vials were 0.150 M in substrate. A final vial was charged with substrate/internal standard stock solution (150 μL) for use as the time = 0 sample, required for accurate quantification of substrate and product. The vials were capped and removed immediately after catalyst stock solution was added from the glove box and heated to 25˚C (set A and C) and 40 °C (sets B and D) with stirring. After 0.167, 0.5, 1, 6, and 24 hours one vial from each of the sets was removed from heat, cooled, and exposed to air to quench. The solvent was then removed in vacuo; the remaining residue was dissolved in $CDCl_3$ and analyzed by ^1H NMR spectroscopy. The starting material and/or product was referenced internally to dimethyl terephthalate.

II NMR Spectra

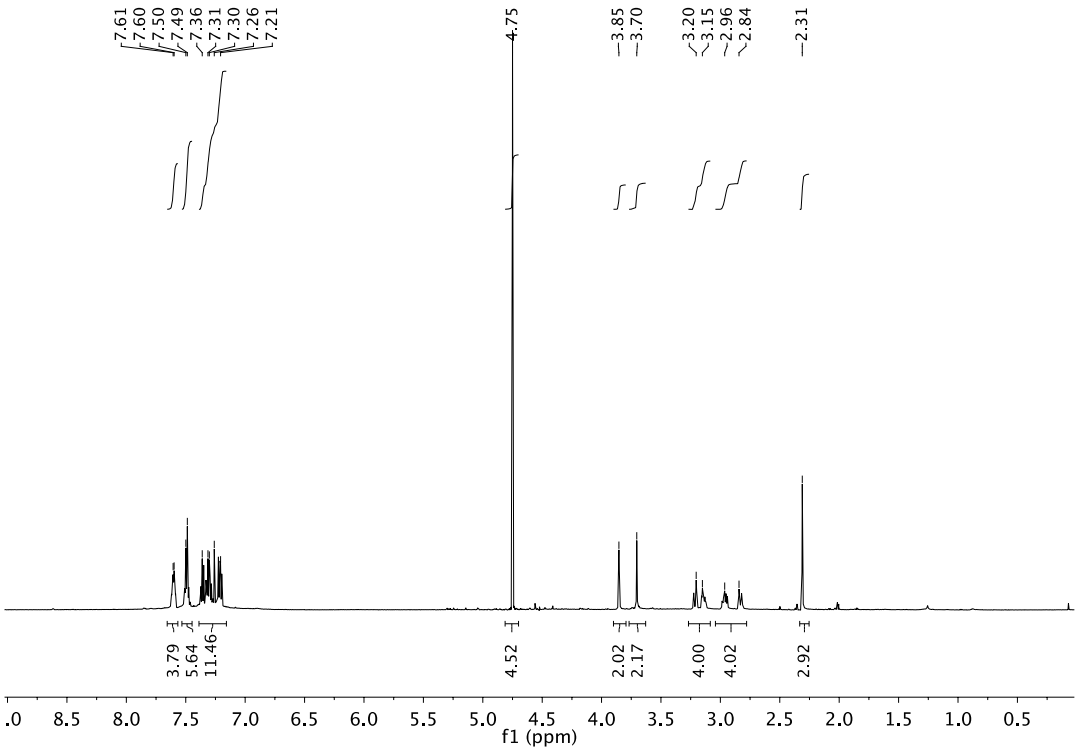

Figure S1. ^{1}H NMR spectrum of **1b** in $CDCl_3$ (600 MHz).

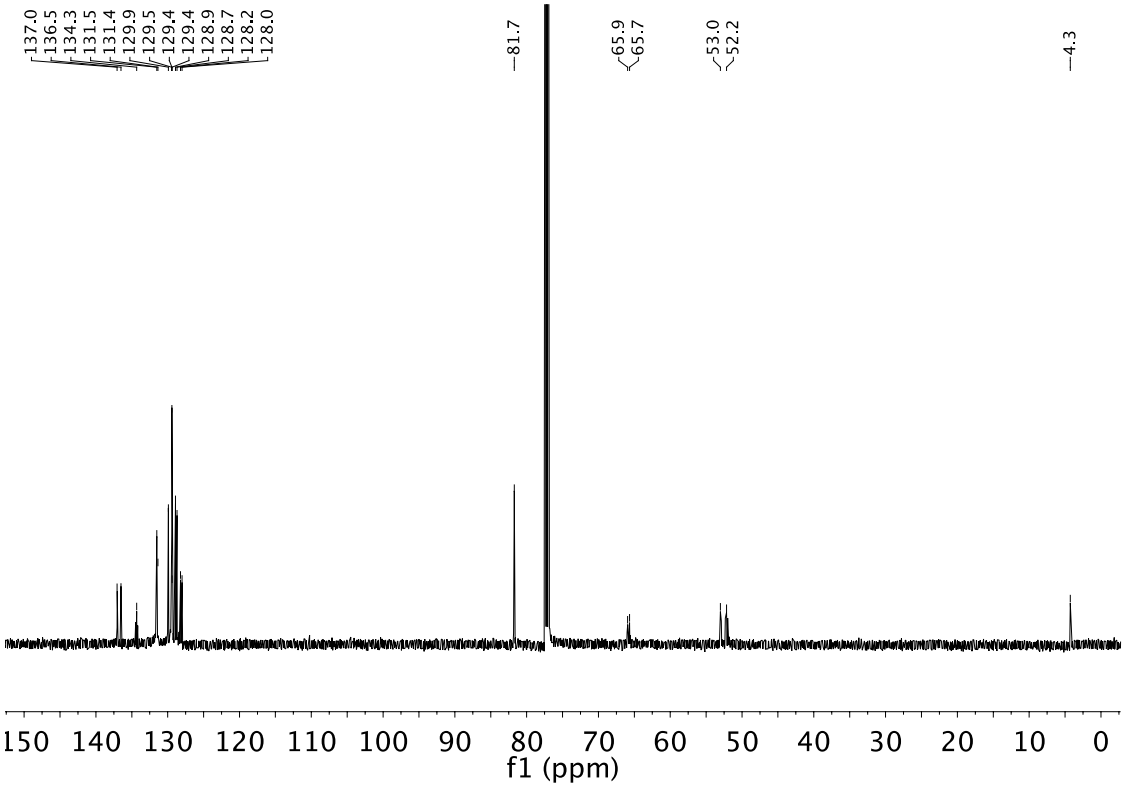

Figure S2. $^{13}C\{^{1}H\}$ NMR spectrum of **1b** in $CDCl_3$ (151 MHz).

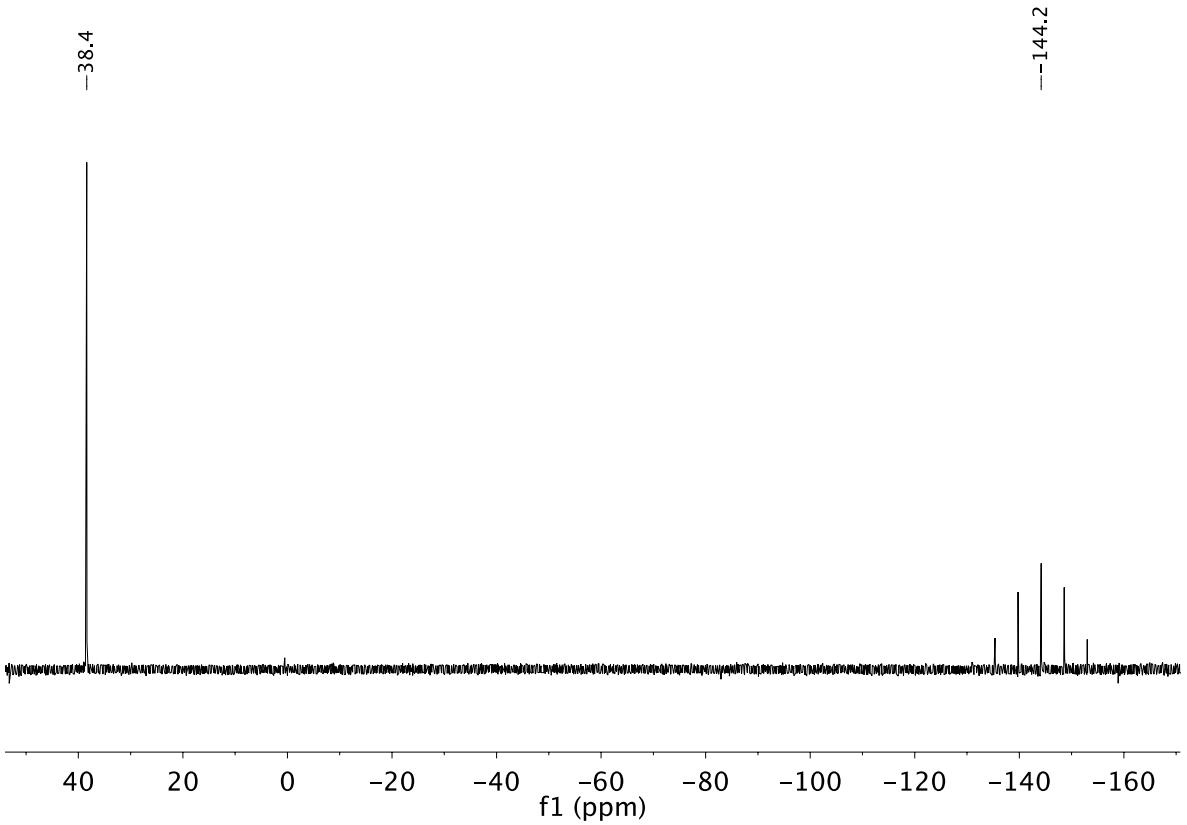

Figure S3. $^{31}P\{^{1}H\}$ NMR spectrum of **1b** in $CDCl_3$ (243 MHz).

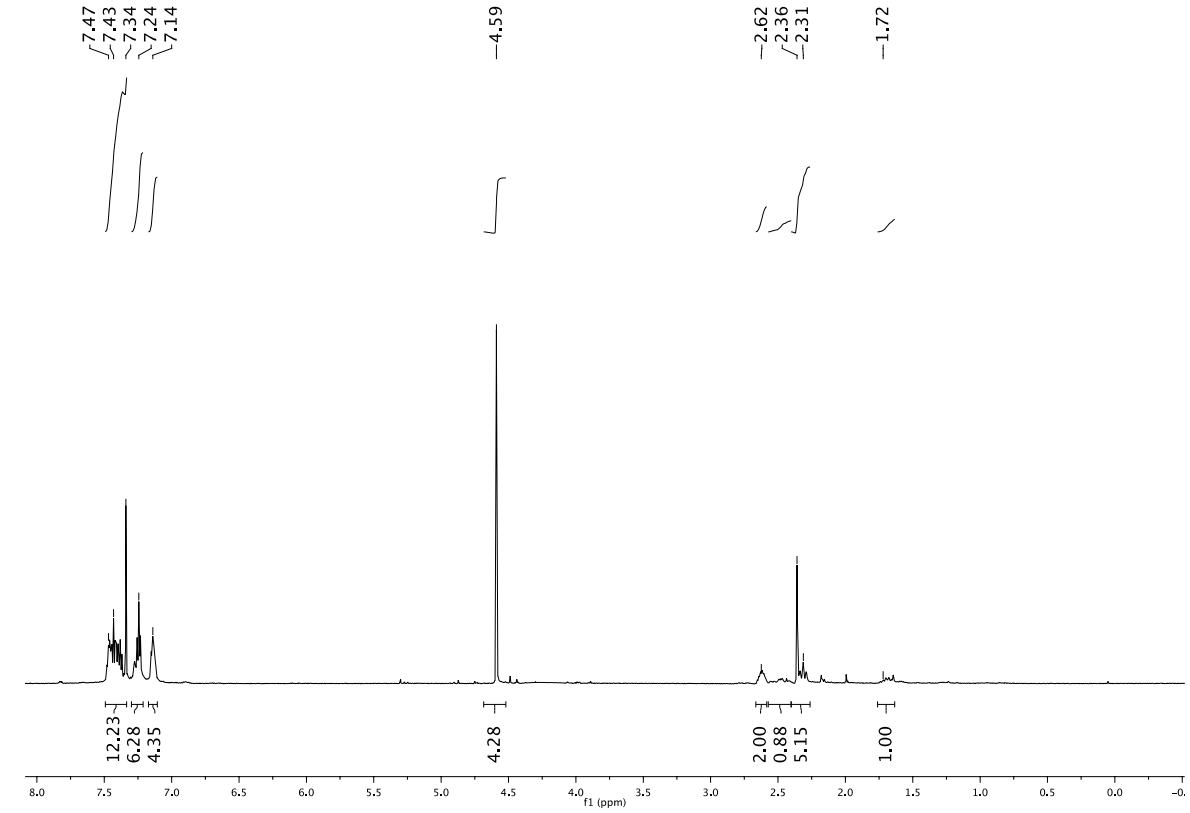

Figure S4. ^{1}H NMR spectrum of **3** in $CDCl_3$ (600 MHz)

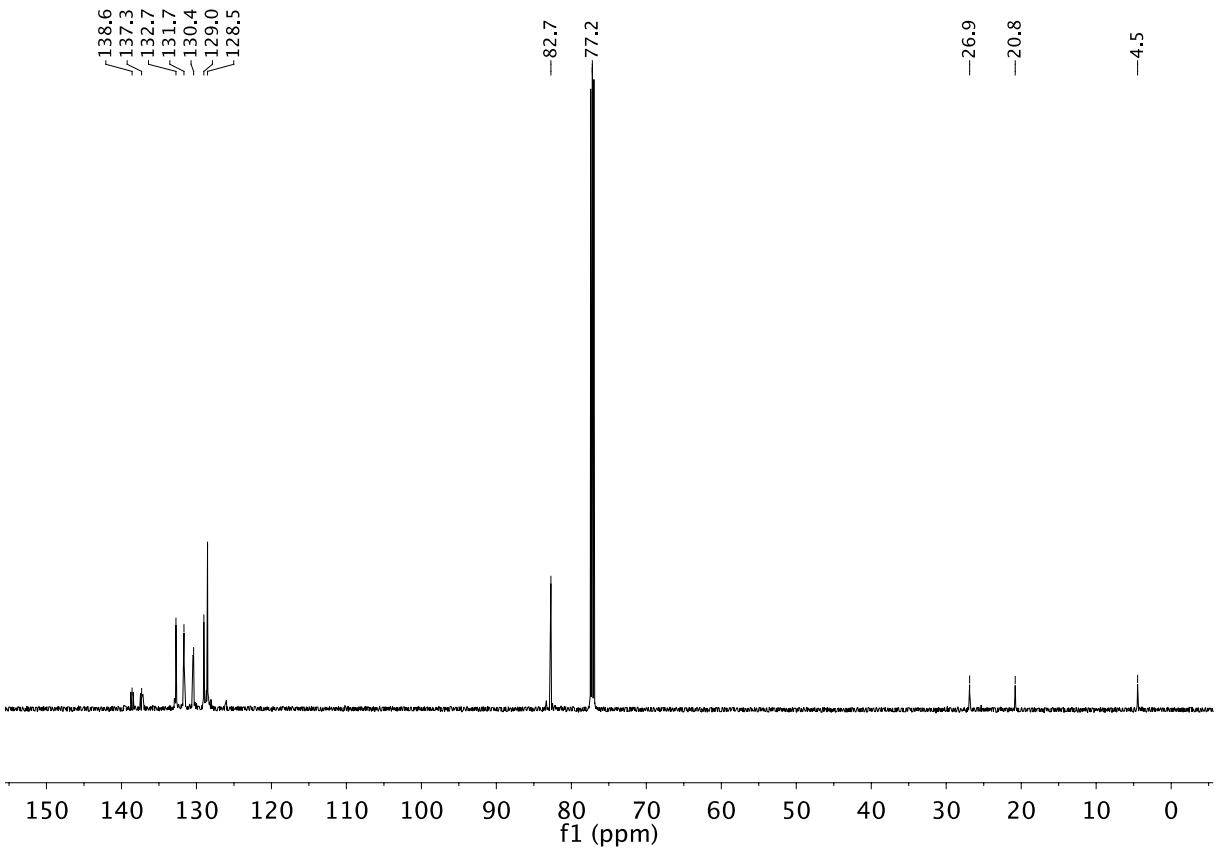

Figure S5. $^{13}C\{^{1}H\}$ NMR spectrum of **3** in $CDCl_3$ (151 MHz).

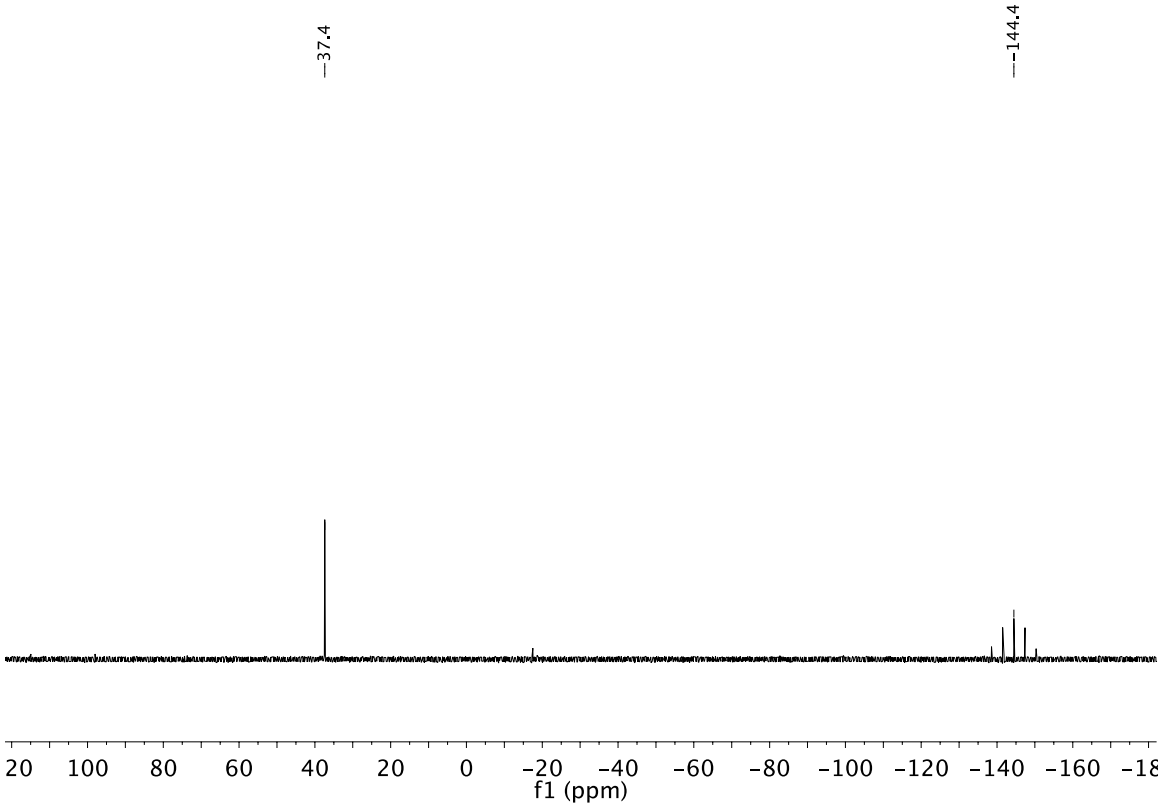

Figure S6. $^{31}P\{^{1}H\}$ NMR spectrum of **3** in $CDCl_3$ (243 MHz).

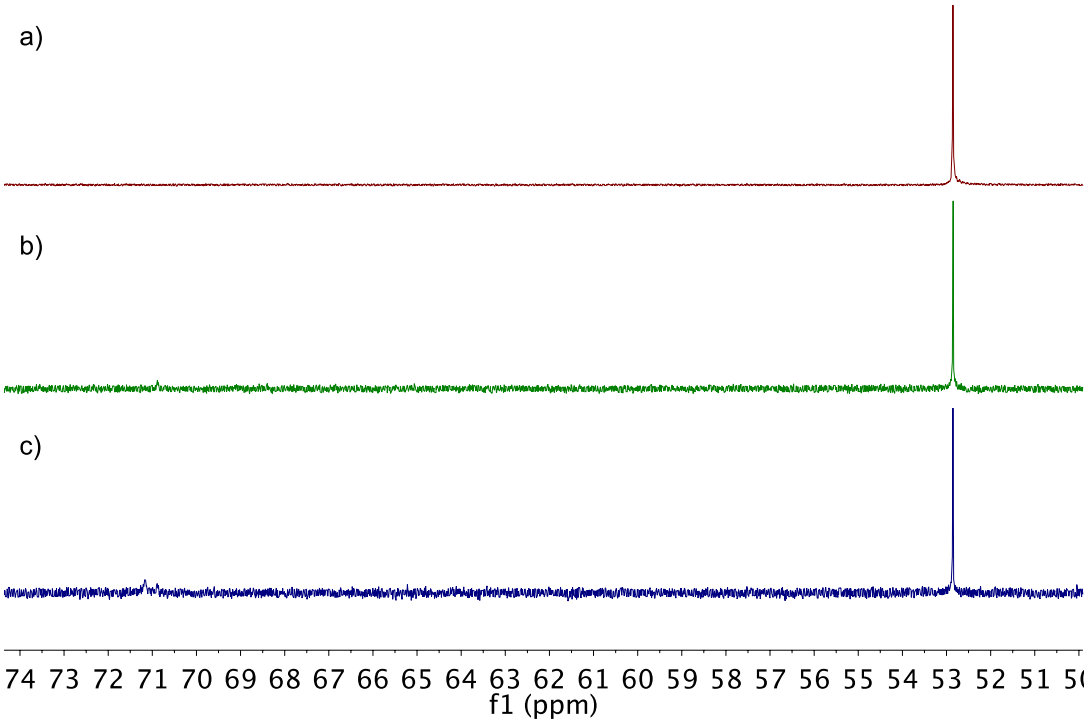

Figure S7. $^{31}P\{^{1}H\}$ NMR stacked spectra of the catalytic reaction of 2-ethynylbenzyl alcohol (**4a**) with **1a** (1.5 mol%) at 40 ˚C at time points of: a) 0 min (before substrate addition); b) 15 minutes; c) 90 minutes.

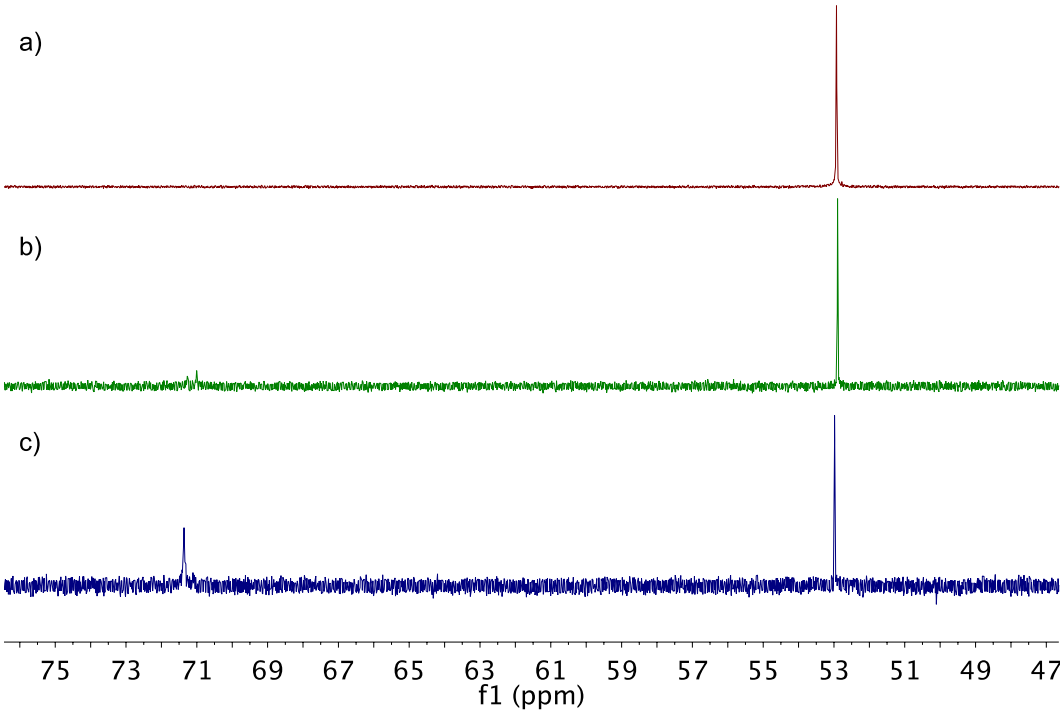

Figure S8. $^{31}P\{^{1}H\}$ NMR stacked spectra of the catalytic reaction of 2-ethynylbenzyl alcohol (**4a**) with **1a** (1.5 mol%) at 40 ˚C at time points of: a) 0 min (before substrate addition); b) 15 minutes; c) 90 minutes.

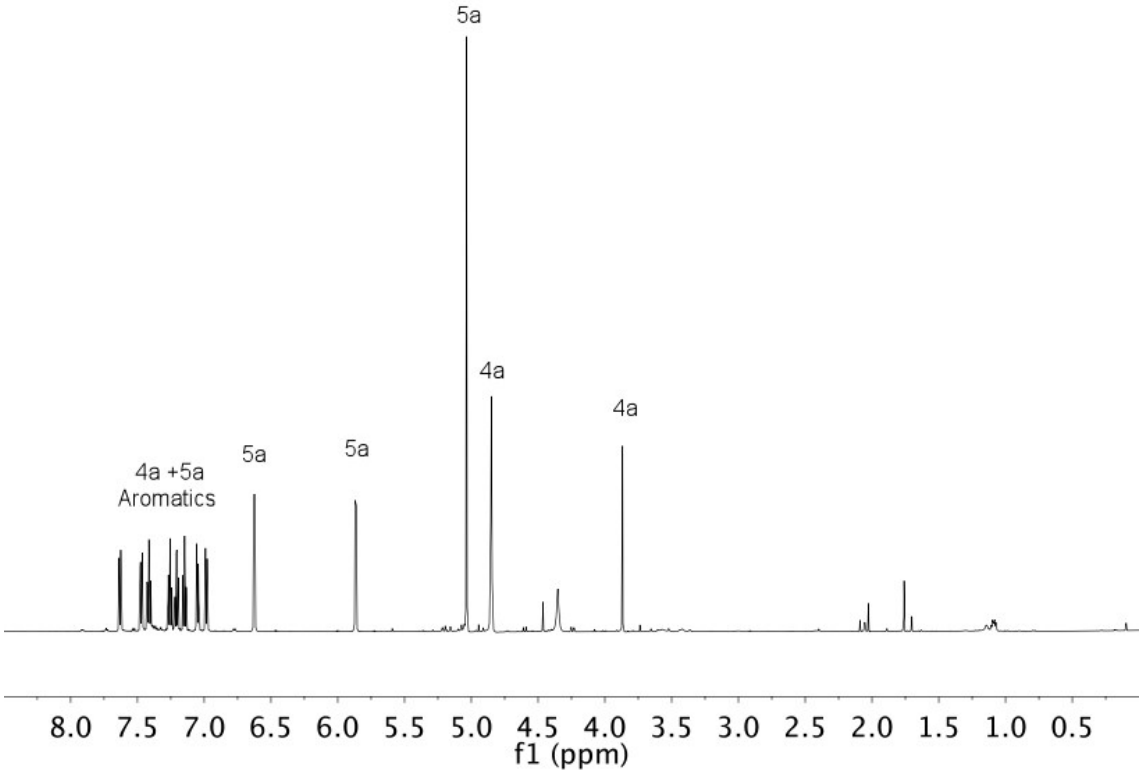

Figure S9. ^{1}H NMR spectrum of the in situ characterization of **7a** in acetone-d_6 (600 MHz), formed under catalytic conditions with 100 equiv of substrate **4a** after heating (54 ˚C) for 7h. The majority species observed is the cyclization product **5a**.

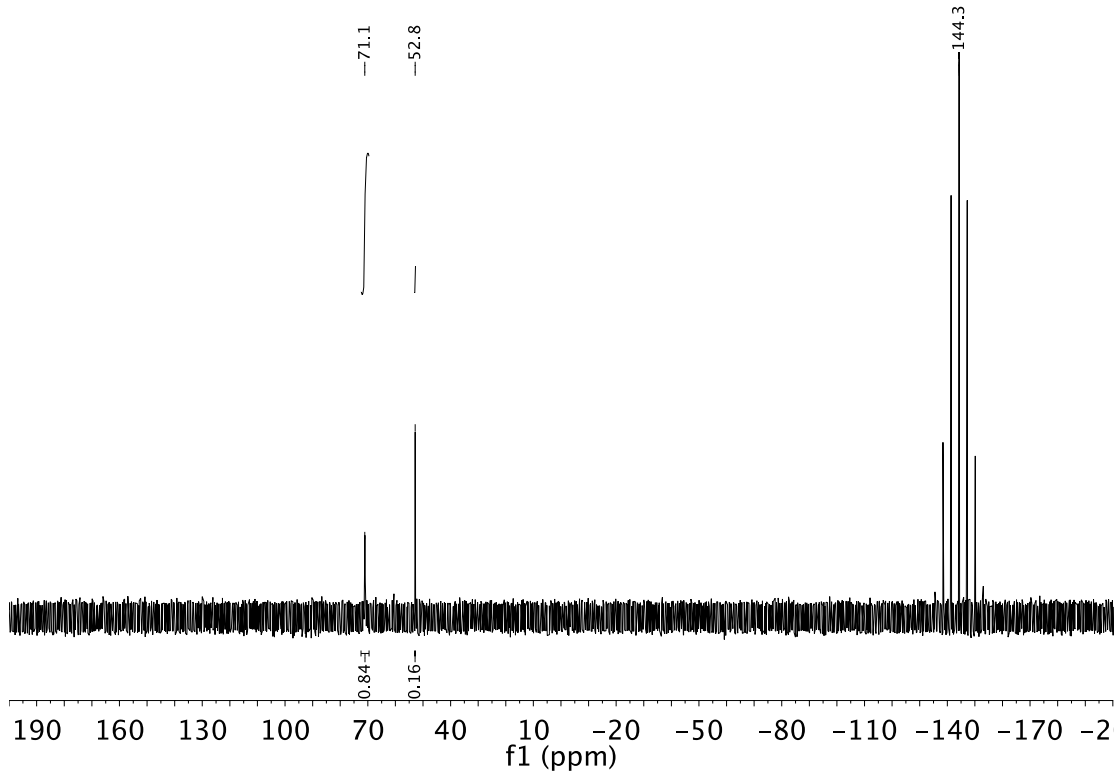

Figure S10. ^{31}P {^{1}H} NMR spectrum of the in situ characterization of **7a** in acetone-d_6 (243 MHz), formed under catalytic conditions with 100 equiv of substrate **4a** after heating (54 ˚C) for 7h. The signal for **1a** is found at 52.8 ppm.

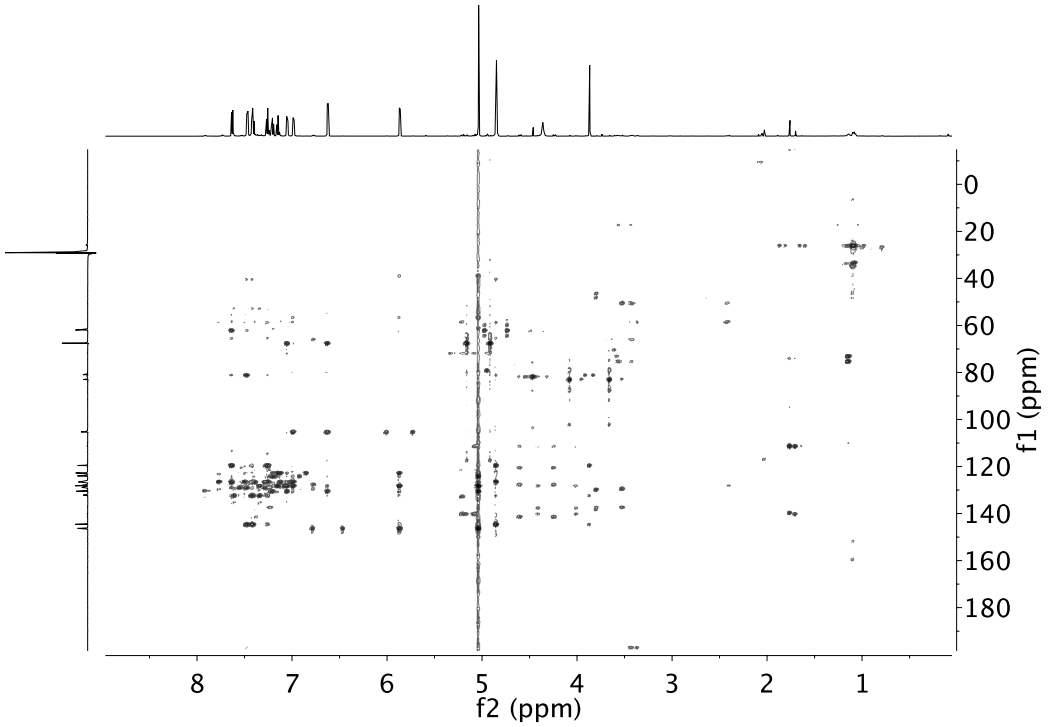

Figure S11. $^{1}H–^{13}C$ gHMBCAD NMR spectrum of the in situ characterization of **7a** in acetone-d_6, formed under catalytic conditions with 100 equiv of substrate **4a** after heating (54 ˚C) for 7h.

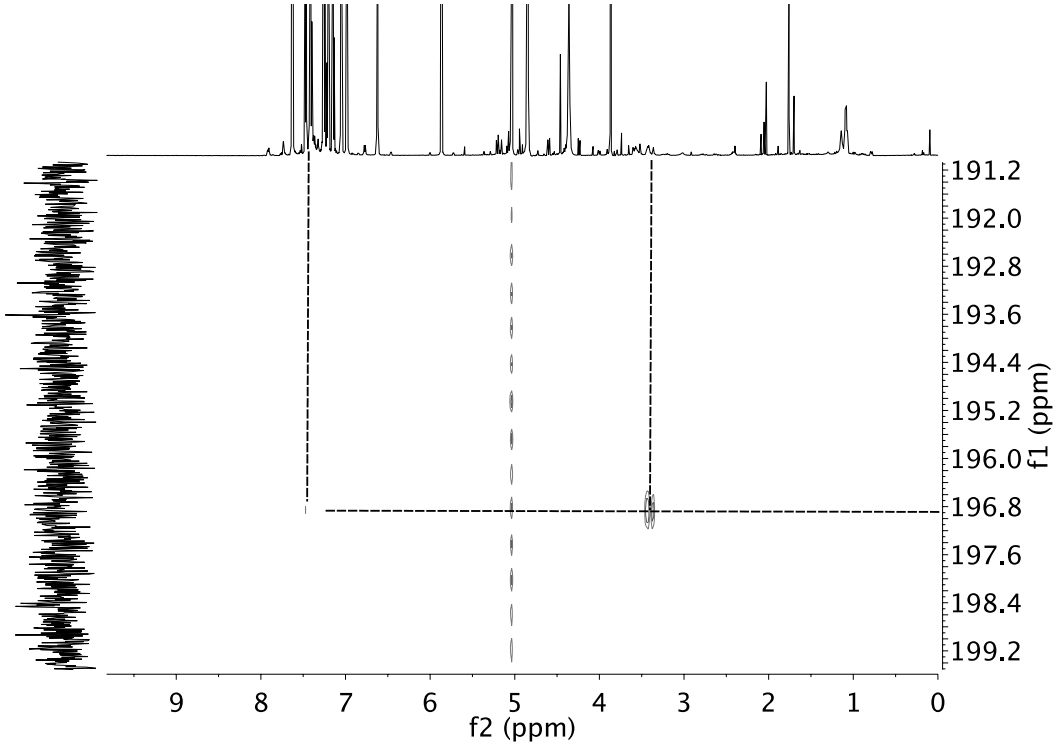

Figure S12. A zoom in on the important signals in $^{1}H–^{13}C$ gHMBCAD NMR spectrum of the in situ characterization of **7a** in acetone-d_6, formed under catalytic conditions with 100 equiv of substrate **4a** after heating (54 ˚C) for 7h. The carbon resonance is consistent with Cα of a Ru(vinyl) functionality, the ^{1}H cross peaks at 7.48 and 3.39 ppm are consistent with RuC=C*H*Ph and PCH_2N moieties, respectively of the catalyst $P^{Ph}{}_2N^{Bn}{}_2$ ligand.

III IR Spectra

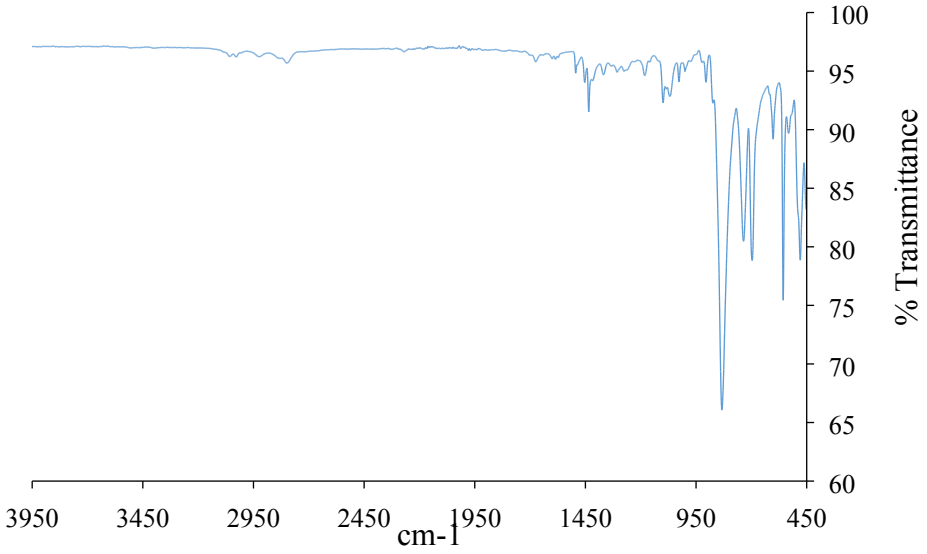

Figure S13. IR spectrum of solid **1b** collected with a PerkinElmer UATR Two FT-IR Spectrum Two

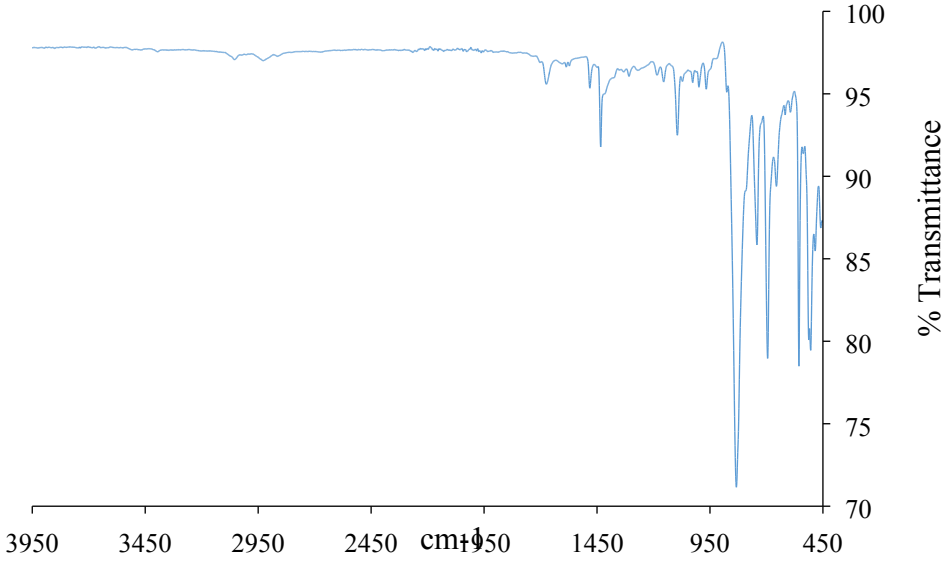

Figure S14. IR spectrum of solid **2** collected with a PerkinElmer UATR Two FT-IR Spectrum Two

IV MALDI data

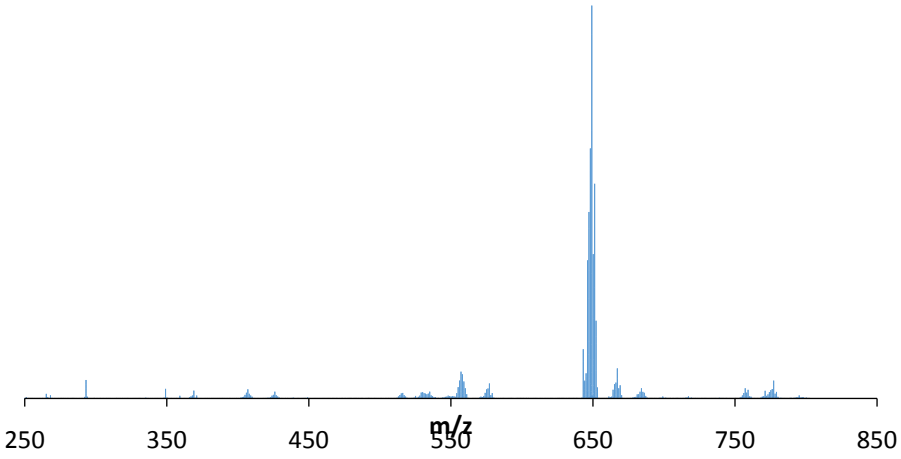

Figure S15. MALDI-TOF mass spectrum of **1b** collected with pyrene as the matrix.

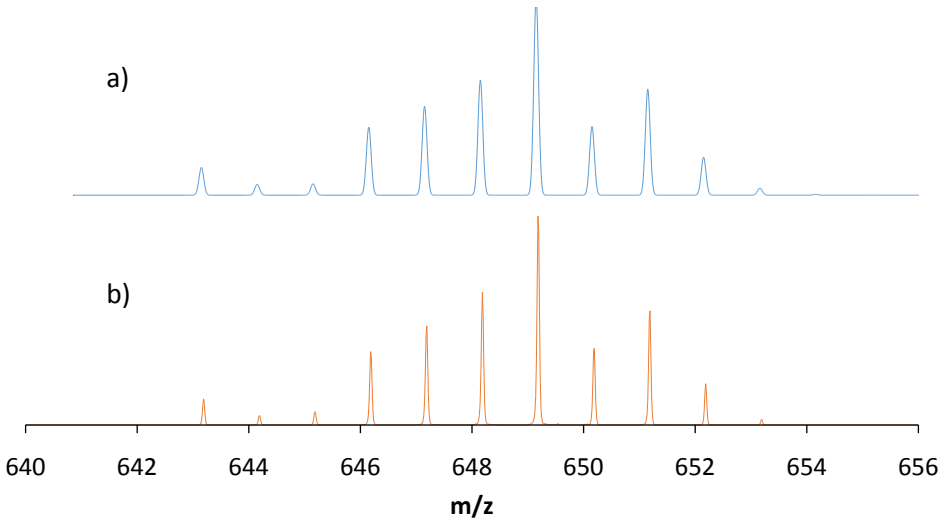

Figure S16. MALDI-TOF MS isotope patterns a) Simulated[8] for [**1b**–MeCN–PF_6]$^+$ with *m/z* = 649.1; b) expansion of the spectrum in Figure S15 to show the observed signal found at *m/z* = 649.2. Observed data were acquired with pyrene as the matrix.

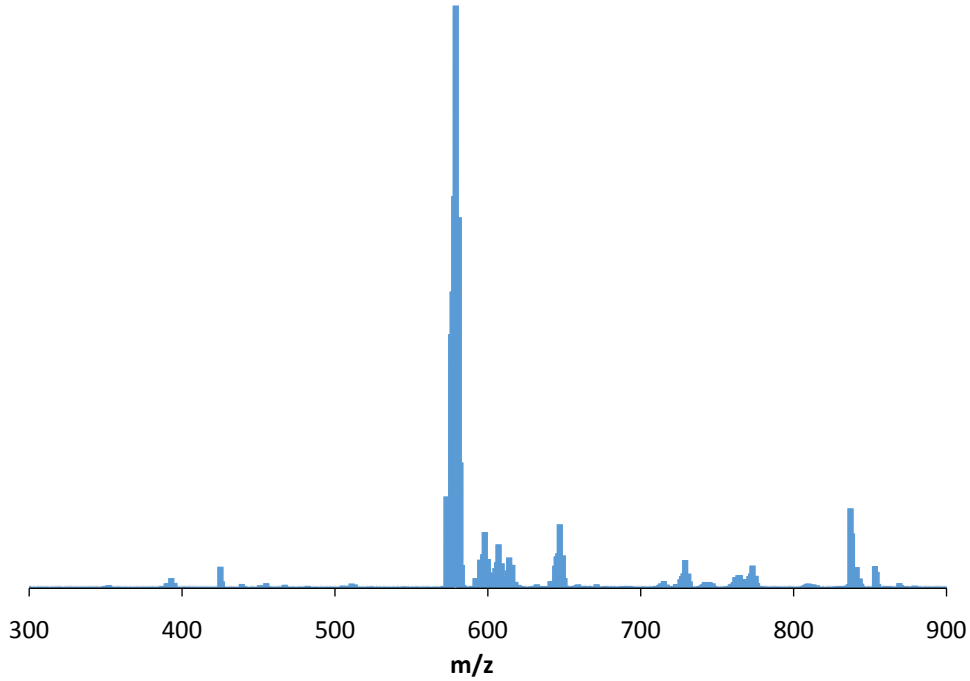

Figure S17. MALDI-TOF mass spectrum of **2** collected with pyrene as the matrix.

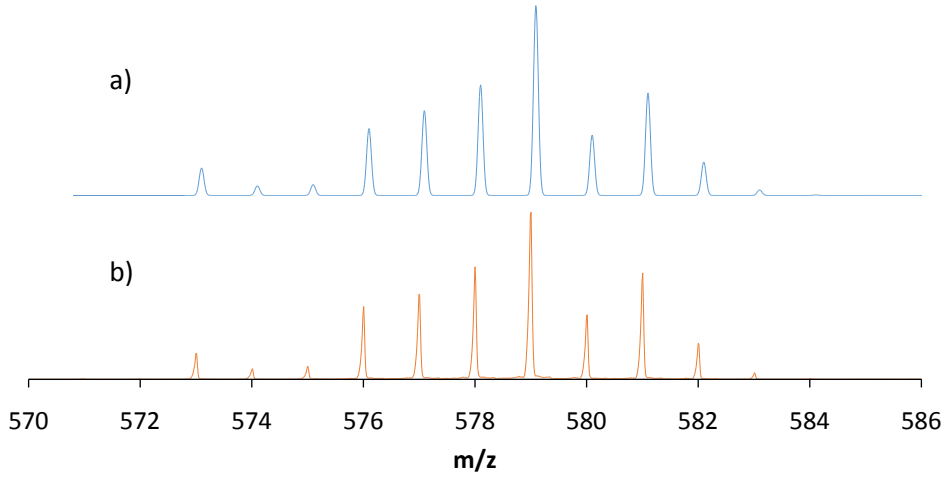

Figure S18. MALDI-TOF MS isotope patterns a) Simulated[8] for [**2**–MeCN–PF_6]$^+$ with *m/z* = 579.1; b) expansion of the spectrum in Figure S17 to show the observed signal found at *m/z* = 579.1. Observed data were acquired with pyrene as the matrix.

V Additional Catalysis Graphs

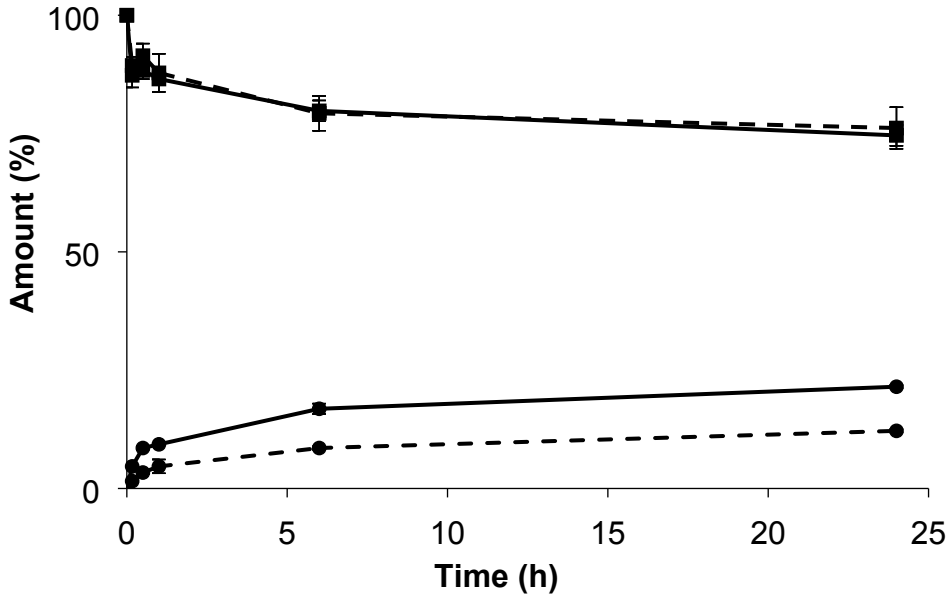

Figure S19. – Cyclization of **4b** (150 mM) by 1 mol% of catalyst **1a** (solid line) and **1b** (dashed line) at 40 ˚C monitored over 24 h. The quantities of substrate **4b** (■) and product **5b** (●) are depicted. Amounts were determined by ^{1}H NMR spectroscopy by integration of signals for **4b**/**5b** relative to an internal standard. Reactions were conducted in duplicate. Data points represent the average of the two runs and the error bars give the span of the conversion values of each data set.

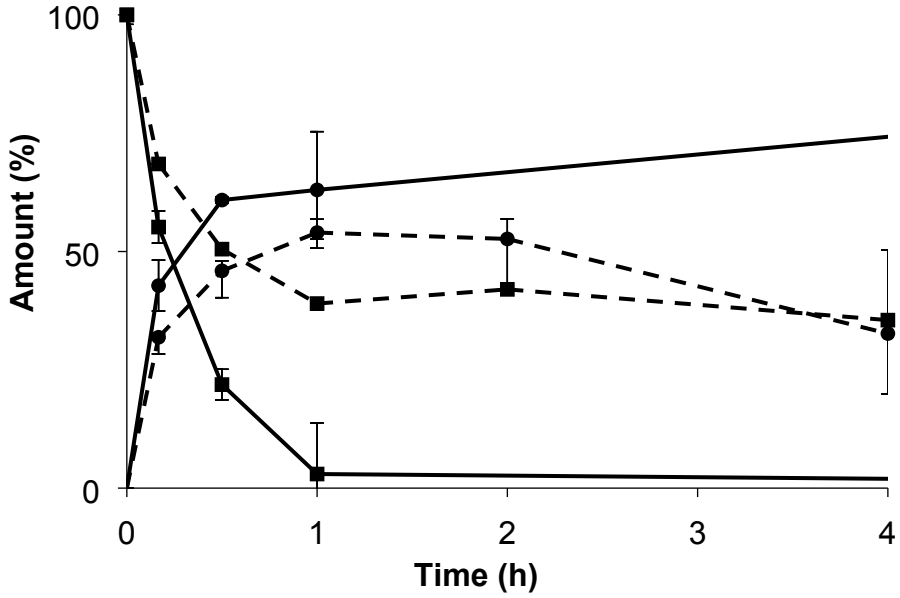

Figure S20. Cyclization of **4a** (150 mM) by 1 mol% of **1a** at 40 ˚C (solid line) and 54 ˚C (dashed line) monitored over 24 h. The quantities of substrate **4a** (■) and product **5a** (●) are depicted Amounts were determined by ^{1}H NMR spectroscopy by integration of signals for **4b**/**5b** relative to an internal standard. Reactions were conducted in duplicate. Data points represent the average of the two runs and the error bars give the span of the conversion values of each data set.

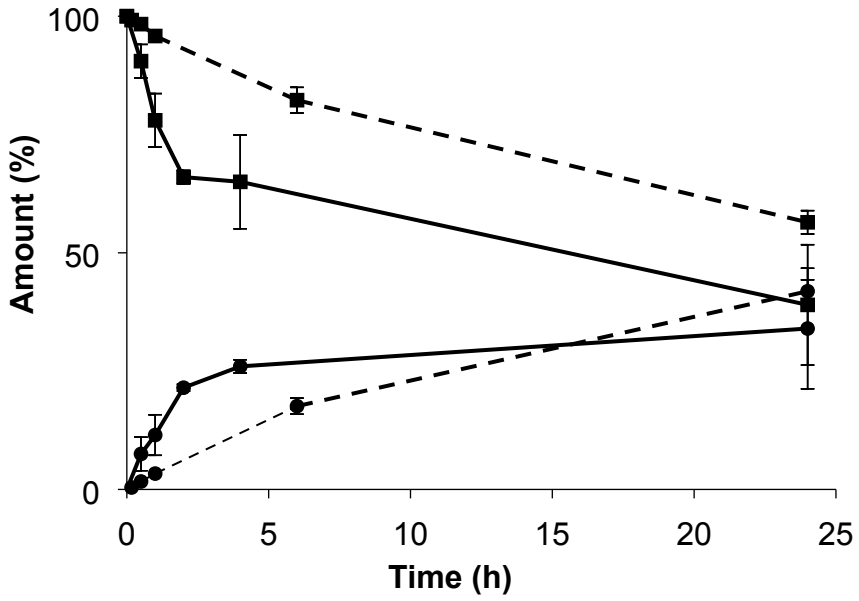

Figure S21. Cyclization of **4a** (150 mM) by 1 mol% of **1b** at 40 ˚C (solid line) and 54 ˚C (dashed line) monitored over 24 h. The quantities of substrate **4a** (■) and product **5a** (●) are depicted. Amounts were determined by ^{1}H NMR spectroscopy by integration of signals for **4b**/**5b** relative to an internal standard. Reactions were conducted in duplicate. Data points represent the average of the two runs and the error bars give the span of the conversion values of each data set.

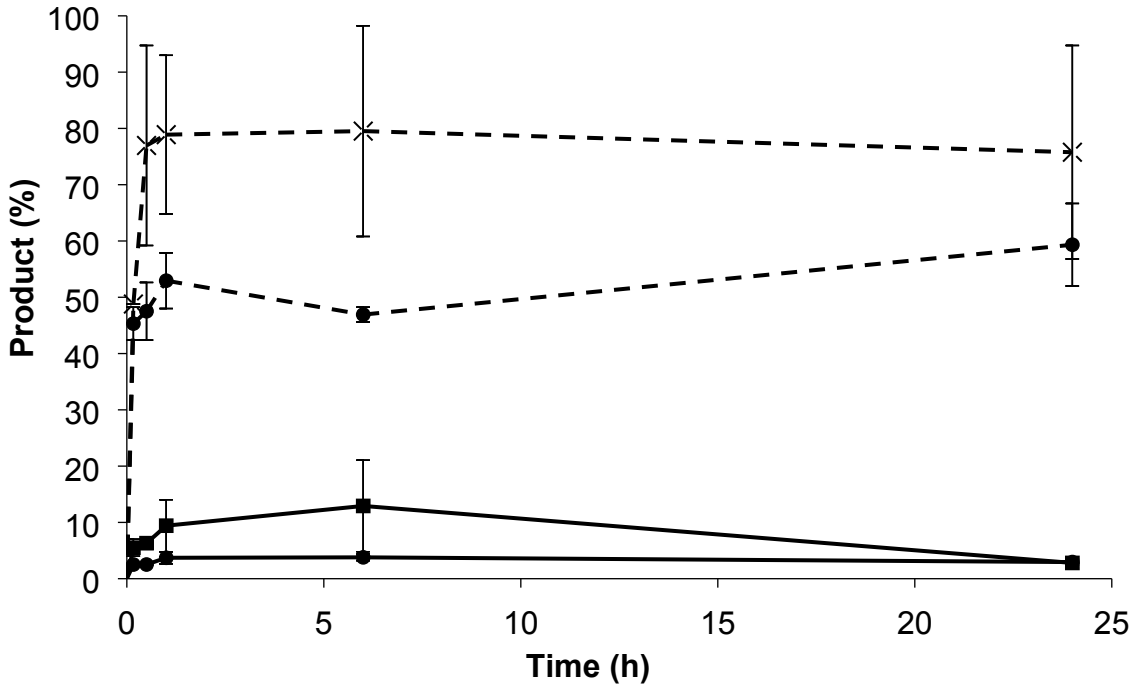

Figure S22. Cyclization of **4a** (150 mM) by 0.1 (solid line) and 1 mol% (dashed line) of precatalyst RuCl(Cp)($P^{Ph}_2N^{Bn}_2$) treated with $TlPF_6$ at 25 ˚C (●) and 40 ˚C (■) over 24 h. Amounts were determined by ^{1}H NMR spectroscopy by integration of signals for **4b**/**5b** relative to an internal standard. Reactions were conducted in duplicate. Data points represent the average of the two runs and the error bars give the span of the conversion values of each data set.

VI Crystallographic Data

Data Collection and Processing. The sample of **2** was mounted on a Mitegen polyimide micromount with a small amount of Paratone N oil. All X-ray measurements were made on a Bruker Kappa Axis Apex2 diffractometer at a temperature of 110 K. The unit cell dimensions were determined from a symmetry constrained fit of 9671 reflections with 5.6° < 2□ < 68.64°. The data collection strategy was a number of ω and φ□scans which collected data up to 72.808° (2θ). The frame integration was performed using SAINT.[9] The resulting raw data was scaled and absorption corrected using a multi-scan averaging of symmetry equivalent data using SADABS.[10]

Structure Solution and Refinement. The structure was solved by using a dual space methodology using the SHELXT program.[11] All non-hydrogen atoms were obtained from the initial solution. The hydrogen atoms were introduced at idealized positions and were allowed to ride on the parent atom. The asymmetric unit contained a region of electron density which was presumably due to disordered solvent molecule(s). However, attempts to derive a chemically sensible disorder model were unsuccessful. The SQUEEZE routine from PLATON was therefore applied to the data.[12] The structural model was fit to the data using full matrix least-squares based on F^2. The calculated structure factors included corrections for anomalous dispersion from the usual tabulation. The structure was refined using the SHELXL-2014 program from the SHELXL suite of crystallographic software.[11] Graphic plots were produced using the NRCVAX program suite.[13] Additional information and other relevant literature references can be found in the reference section of this website (http://xray.chem.uwo.ca).

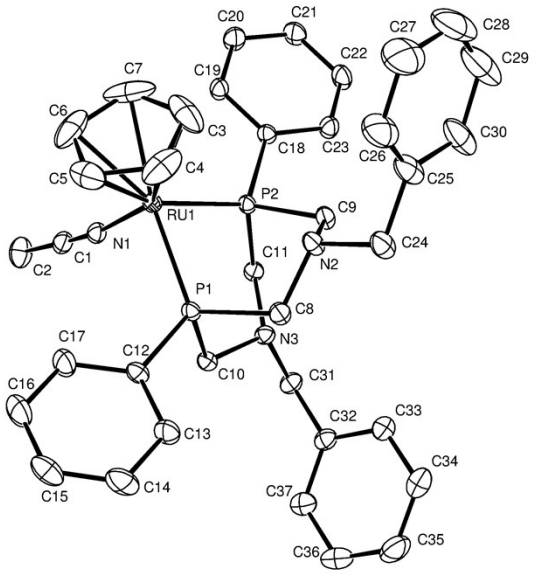

Figure S23. ORTEP drawing of **2** showing naming and numbering scheme. Ellipsoids are at the 50% probability level. Hydrogen atoms, $[PF_6]^-$ counter-ion and diethyl ether molecule of solvation were omitted for clarity.

Table S1: Summary of Crystal Data for **1b**

Formula	$C_{41}H_{50}F_6N_3OP_3Ru$ (**1b**)
CCDC Number	1418495
Formula Weight (*g/mol*)	908.82
Crystal Dimensions (*mm*)	0.288 × 0.186 × 0.090
Crystal Color and Habit	colourless prism
Crystal System	triclinic
Space Group	P -$\bar{1}$
Temperature, K	110
a, Å	10.832(3)
b, Å	13.759(3)
c, Å	15.524(5)
α,°	99.505(7)
β,°	94.478(10)
γ,°	104.276(5)
V, Å^3	2194.3(10)
Number of reflections to determine final unit cell	9671
Min and Max 2θ for cell determination, °	5.6, 68.64
Z	2
F(000)	936
□ (*g/cm*)	1.375
□, Å, (MoK□)	0.71073
□, (*cm^{-1}*)	0.526
Diffractometer Type	Bruker Kappa Axis Apex2
Scan Type(s)	Ω and ϕ scans
Max 2θ for data collection, °	72.808
Measured fraction of data	0.997
Number of reflections measured	115317
Unique reflections measured	19679
R_{merge}	0.0356
Number of reflections included in refinement	19679
Cut off Threshold Expression	I > 2σ (I)
Structure refined using	full matrix least-squares using F^2
Weighting Scheme	$w=1/[\sigma^2(Fo^2)+(0.0464P)^2+0.3723P]$ where $P=(Fo^2+2Fc^2)/3$
Number of parameters in least-squares	499
R_1	0.0393
wR_2	0.0889

R_1 (all data)	0.0564
wR_2 (all data)	0.0958
GOF	1.056
Maximum shift/error	0.004
Min & Max peak heights on final □F Map (e^-/Å)	-0.668, 0.786

Where:
$R_1 = \square(|F_o| - |F_c|) / \square F_o$
$wR_2 = [\square(w(F_o^2 - F_c^2)^2) / \square(w F_o^4)]^{½}$
$GOF = [\square(w(F_o^2 - F_c^2)^2) / (\text{No. of reflns.} - \text{No. of params.})]^{½}$

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