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Supporting Information

Efficient Solventless Dehydrogenation of Formic Acid by a CNC-Based Rhodium Catalyst

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Contents

1. Experimental details.

All the reagents (FA, HCOONa, deuterated FA compounds –HCOOD, DCOOD, DCOOH–, NEt_3, KHDMS, ^{t}BuOK, MiliQ Water) used in this work were purchased from commercial sources (Aldrich and Merck) and used without further purification. Glassware was dried at 120 °C before use. All reactions were carried out with rigorous exclusion of air using Schlenk-tube techniques and a dry box. Organic solvents (CD_3CN, HCOOH) were dried by standard procedures and distilled under argon prior to use, or obtained oxygen- and water-free from a Solvent Purification System (Innovative Technologies). All catalytic reactions were assembled in a nitrogen-filled dry box with oven-dried glassware (Man on the Moon series X102 kit micro-reactor with a total volume of 19 mL) and were stirred with Teflon-coated magnetic stirring bars. Formic acid and water were degassed prior to use.

^{1}H, ^{31}P{^{1}H} and ^{13}C{^{1}H} NMR spectra were recorded on Bruker Avance 300 (300.13, 121 and 75.48 MHz, respectively) and Bruker Avance 400 (400.16, 162.51 and 100.61 MHz, respectively) spectrometers. NMR chemical shifts (expressed in parts per million) are referenced to residual solvent peaks (^{1}H and ^{13}C). Coupling constants, *J*, are given in hertz (Hz). ^{2}H NMR experiments were measured in deuterated solvents after previous optimization of shim values in a ^{1}H experiment. Spectral assignments were achieved by combination of ^{1}H-^{1}H COSY, ^{1}H-^{1}H NOESY, ^{13}C{^{1}H}-APT and ^{1}H-^{13}C HSQC/HMBC NMR experiments.

High-resolution electrospray mass spectra (HRMS) were acquired using a MicroTOF-Q hybrid quadrupole time-of-flight spectrometer (Bruker Daltonics, Bremen, Germany). GC-MS analysis were recorded on an Agilent 5973 mass selective detector interfaced to an Agilent 6890 series gas chromatograph system, using a HP-5MS 5% phenyl methyl siloxane column (30 m x 250 mm with a 0.25 mm film thickness).

2. FA Dehydrogenation Catalysis.

The dehydrogenation of formic acid (FA) can generally be performed by direct weighting of catalyst **1** (0.016 %, 7.5 mg, 8.4 μmol) and sodium formate (5-40 %, mol) using an analytic balance placed into a drybox. The solid reagents were further transferred to a *Man on the Moon* reactor, and then pre-heated to 353 K. Then, under an argon atmosphere, 2 mL of neat formic acid is introduced in the reactor *via* syringe. Instantaneously, in the resulting yellow solution starts to afford a high amount of gas bubbles and generated pressure are monitored by a manometer connected to the reactor. Catalytic reaction is then left under stirring until complete dehydrogenation of the solvent.

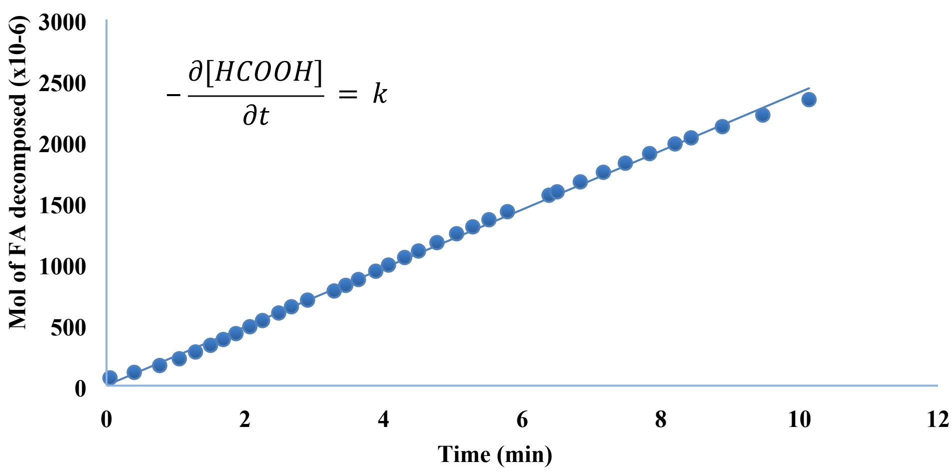
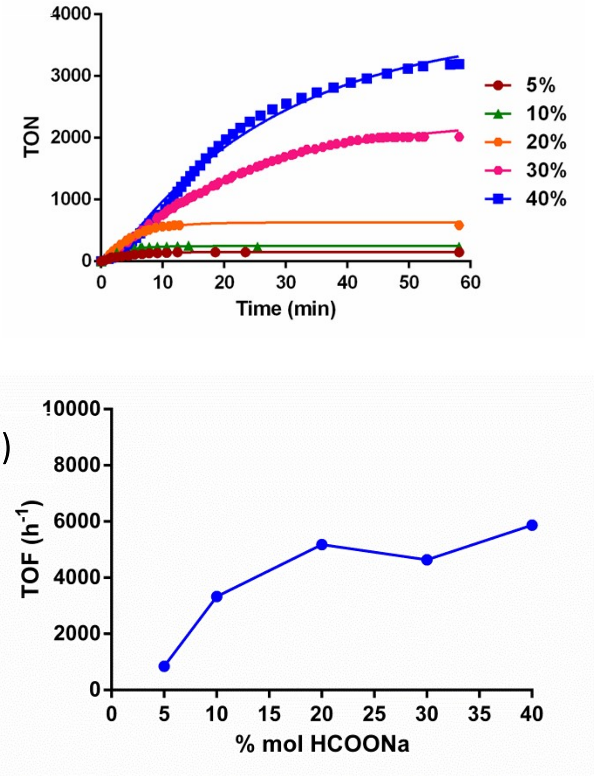


Figure. S1. Plot of moles of FA (lineal section) decomposed in CO_2 and molecular hydrogen with catalyst **1** (0.016 % mol) and HCOONa (30%).

Pressure measurements were carried out using a manometer connected to the reactor (Man on the moon X-102-A04), and we use the initial rates method in order to compare different catalytic parameters (% of base or water addition) and to calculate kinetic isotopic effects.

A)



B)

Figure S2. A) Plot of TON numbers *vs* time under different base conditions. **B)** Graphic of TOF values (h^{-1}) *vs* HCOONa at different loadings.

3. Water effect studies.

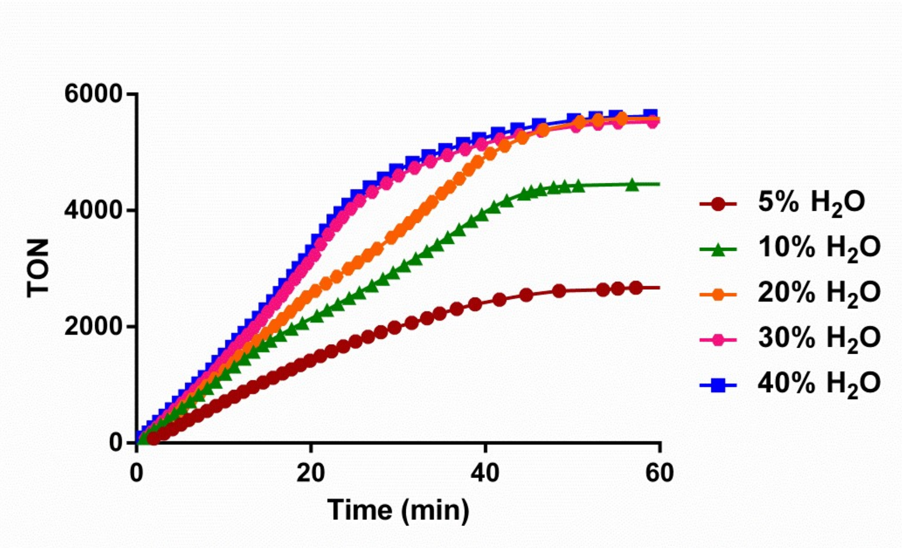


Figure S3. Plot of TON numbers *vs* time under different water concentrations. 5-10-20-30-40 % of MiliQ and deoxygenated water were introduced to the reactor *via* microsyringe through the reactor´s septum. Then, 2 mL of neat FA was added to the mixture, heated at 80 ºC, and the pressure variation was monitored *via* a pressure transducer.

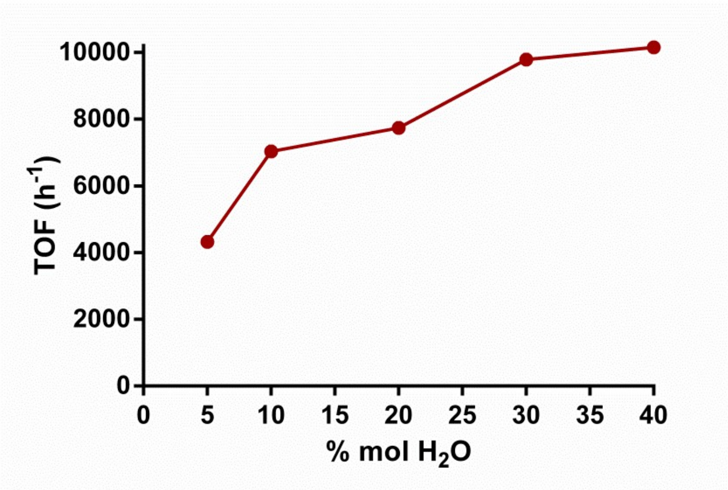


Figure S4. Plot of maximum TOF (h^{-1}) values *vs* mol % of water in neat FA.

4. IR analyses.

IR spectrum of the mixture of gases obtained from catalysis.

The mixture of gases obtained from the reaction catalyzed by **1** in neat FA was studied by IR spectroscopy, showing an excellent selectivity towards the dehydrogenation reaction to H_2 and CO_2, since no traces CO were detected (the CO bands should appear between 2100 and 2200 cm^{-1}).

For that purpose, a Teflon balloon was connected to a 50 mL Schlenk tube, which was charged with rhodium catalyst **1** (30 mg, 0.037 mmol) and sodium formate (1.081 g, 15.9 mmol). To the mixture, neat FA (2 mL) was added and then the mixture was heated at 353 K. The gas produced was collected after 30 min in the balloon, and then it was measured by gas IR spectroscopy. The gas sample was fitted with a vent line connected to a three-way valve, which allows for direct sampling of the gaseous products for IR analysis. The measurement was performed in a Thermo Nicolet Nexus 5700 FTIR spectrometer using as measurement chamber Thermo Smart collector equipped with a gold-plated high pressure, catalytic cell with ZnSe window. The measurement parameters were as follows: Range: 4000-600 cm^{-1}; Bandwidth: 4 cm^{-1}; scan accumulation: 500.

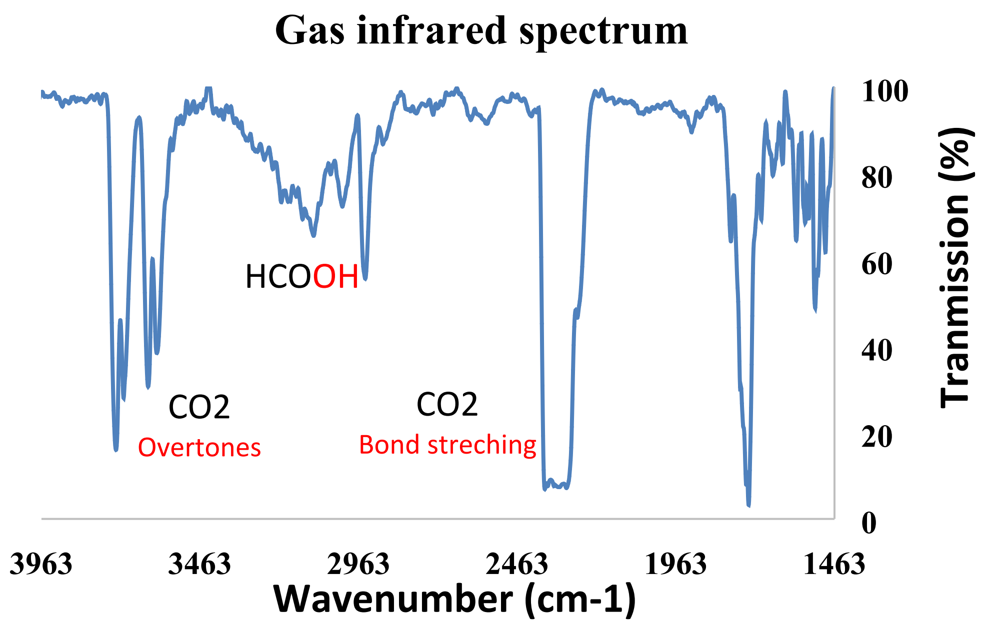


Figure S5. IR spectrum of the gaseous mixture obtained after reaction of neat FA with 40 mol% of HCOONa and 0.016 mol % **1**.

GC Analyses.

A Teflon balloon was connected to a 50 mL Schlenk that was charged with rhodium catalyst **1** (30 mg, 0.037 mmol) and sodium formate (1.081g, 15.9 mmol). The mixture was dissolved in neat FA (2 mL) and then heated at 353 K. The affording gas was collected after 30 min in the balloon and then introduced in the GC-MS prechamber. GC–MS analysis was performed using Agilent 6890N Gas Chromatograph coupled to Agilent 5973 MS detector. The column was HP PLOT Molesieve 5 Å (30 m x 0.32 mm, 25 μm). GC–MS data were obtained using the following conditions: carrier gas helium (He 99.9995 %); flow rate 3 mL/min; the injector temperature was 150 ºC in a pulsed splitless mode (60 psi, 1 min); volume: 50 μL. As detector was used electron impact-MS (70 eV) in a selected mode: SIM (*m/z* = 28). The oven temperature program was: 50 ºC (10 min), –25 ºC/min, –225 ºC (0 min), –30 ºC/min and –300 ºC (3 min). The results are expressed as GC peak areas percent ± RSD (%).

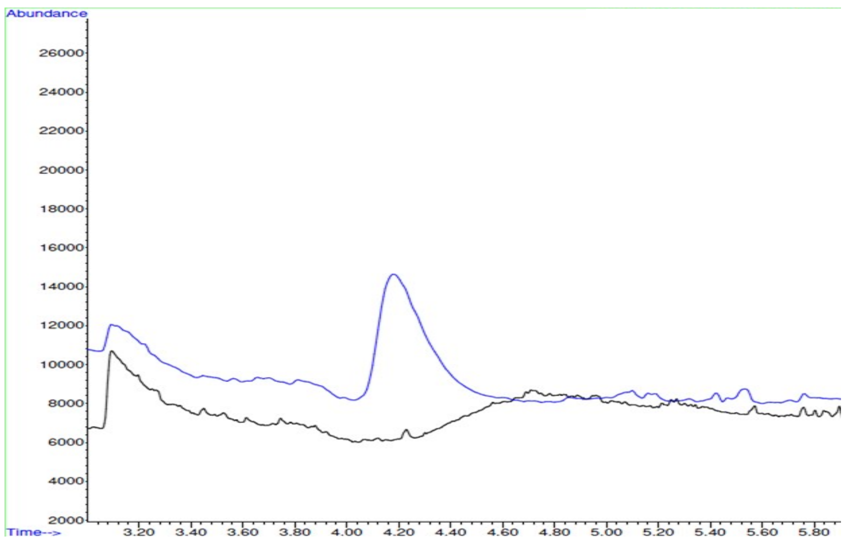


Figure S6. GC-MS of the gaseous products obtained after catalysis under optimized conditions (neat FA, 40 mol% HCOONa, 0.016 mol % **1**) in black (below) and a 40 ppm mixture of CO in air in blue (top); CO peak at 4.20 s.

5. NMR spectra.

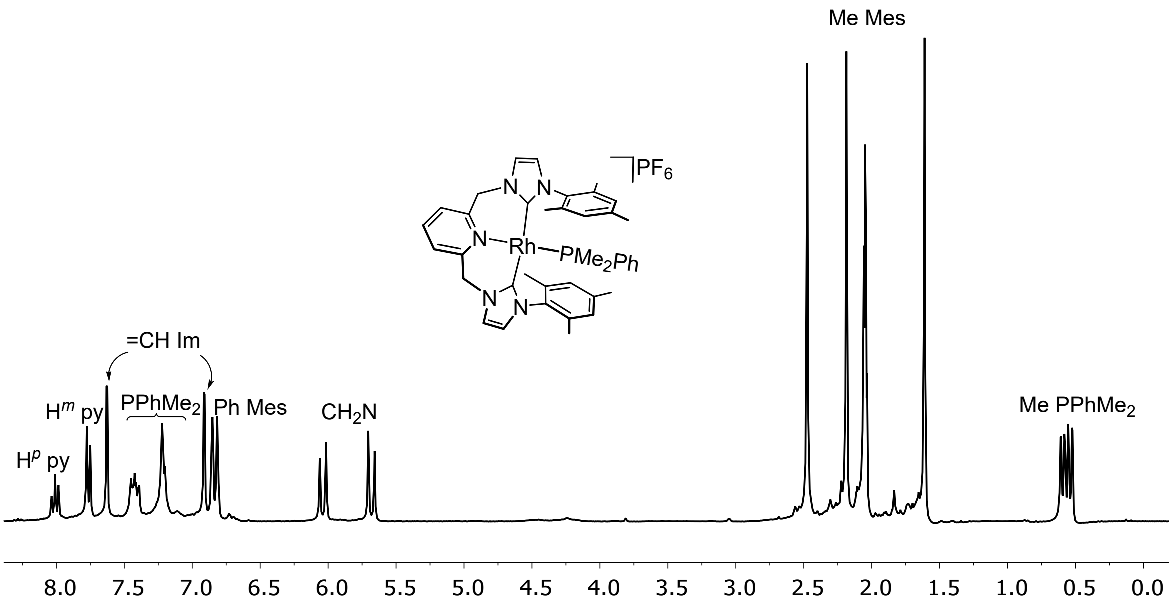


Figure S7. ^1H NMR spectrum of **1** in CD_3CN at 298 K.

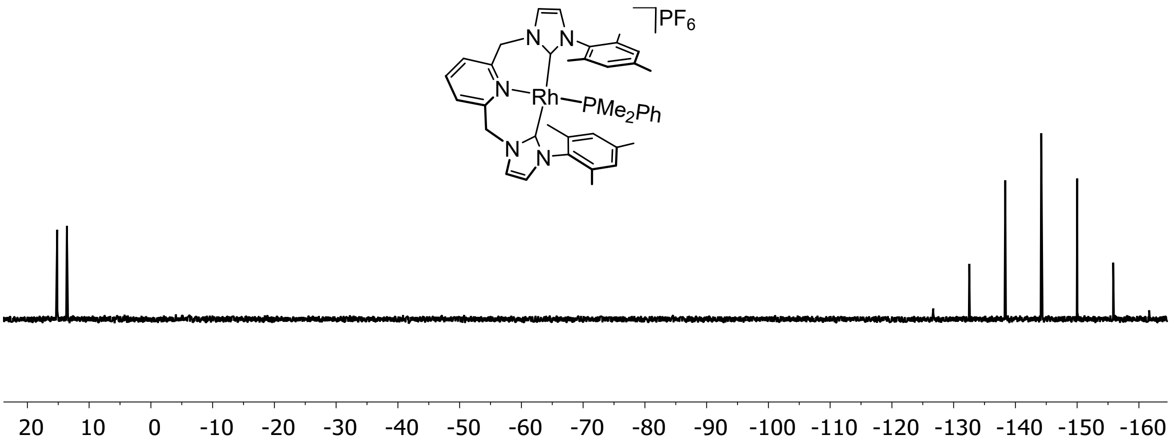


Figure S8. $^{31}P\{^1H\}$ NMR spectrum of **1** in CD_3CN at 298 K.

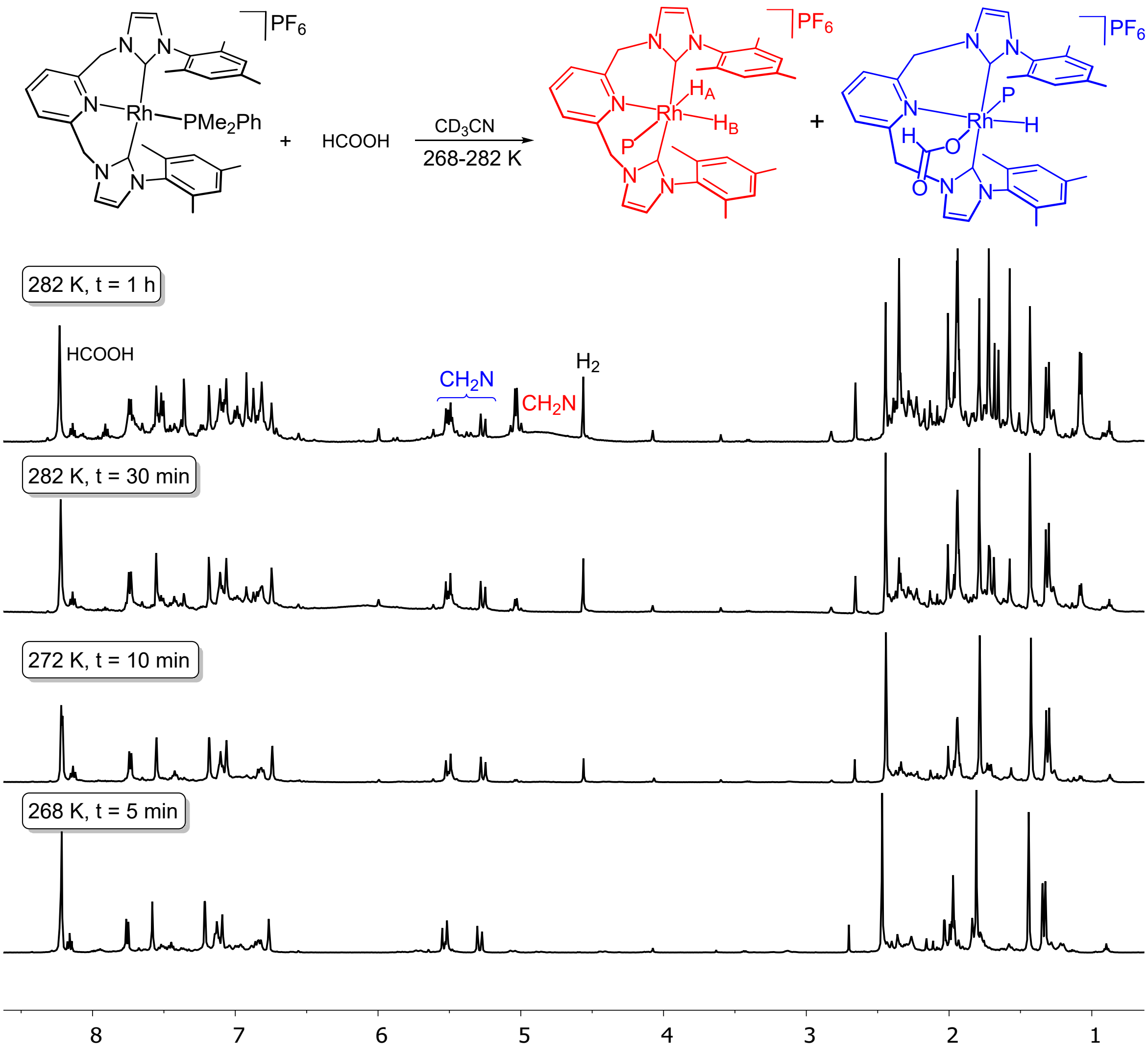


Figure S9. ^{1}H NMR spectra of the reaction of **1** (17 mg, 0.018 mmol) with 3 molar-equiv. of FA (2.58 mg, 0.056 mmol, 2.12 μL) in CD_3CN at 268-282 K.

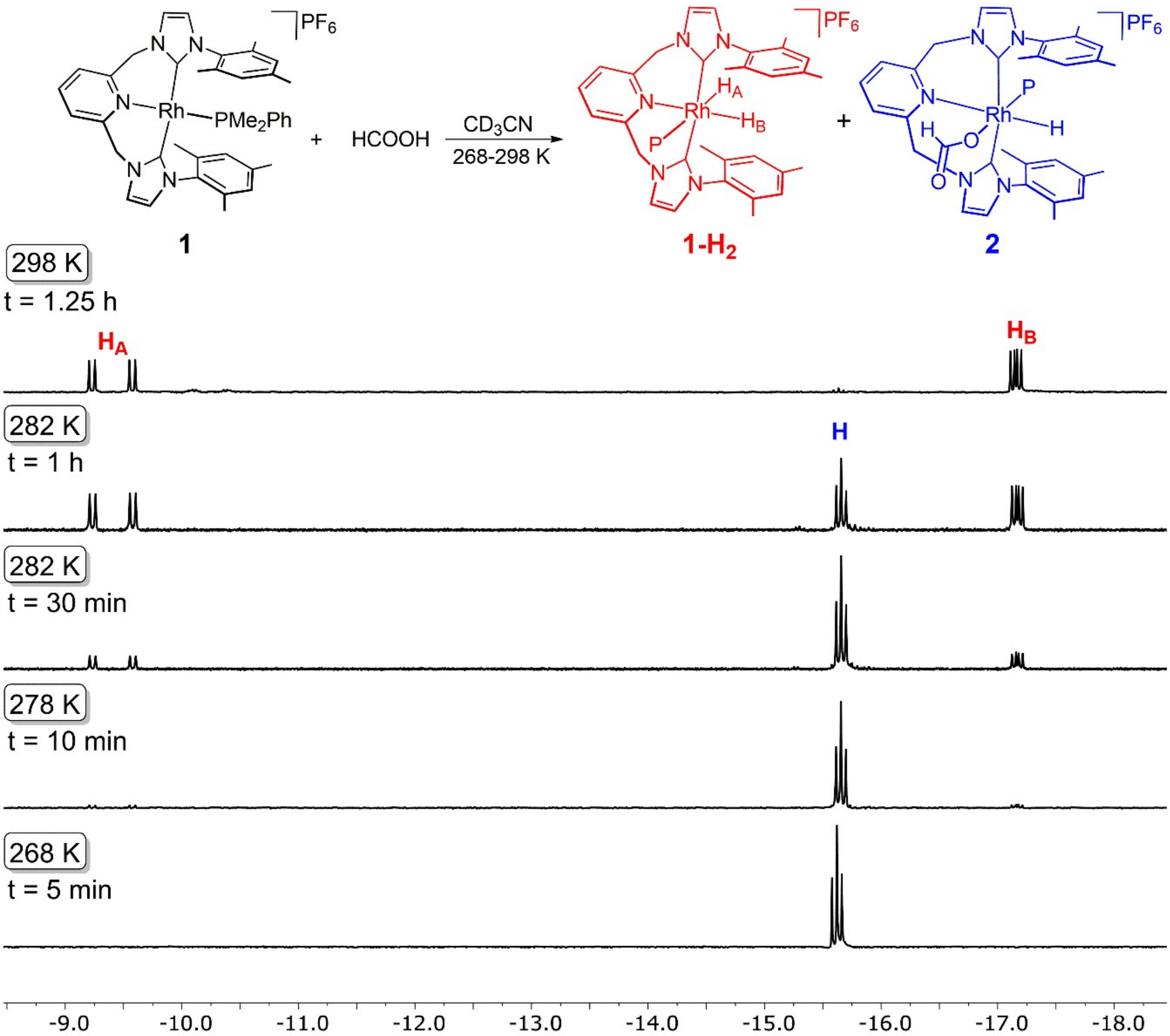


Figure S10. Selected hydride area of the ^{1}H NMR spectra of the reaction of **1** (17 mg, 0.018 mmol) with 3 molar-equiv. of FA (2.58 mg, 0.056 mmol, 2.12 μL) in CD_3CN at 268-298 K.

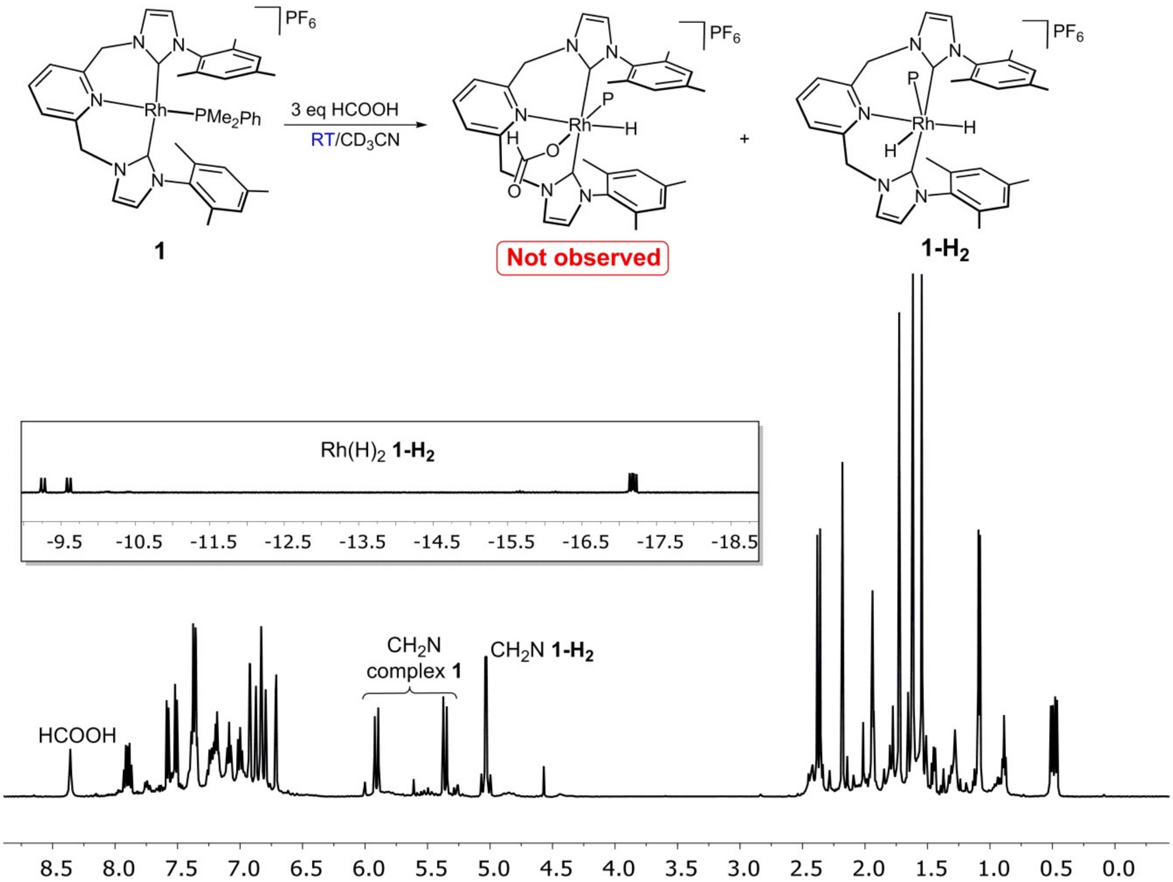


Figure S11. ^{1}H NMR spectrum of the reaction of **1** (17 mg, 0.018 mmol) with 3 molar-equiv. of FA (2.58 mg, 0.056 mmol, 2.12 µL) in CD_3CN at RT.

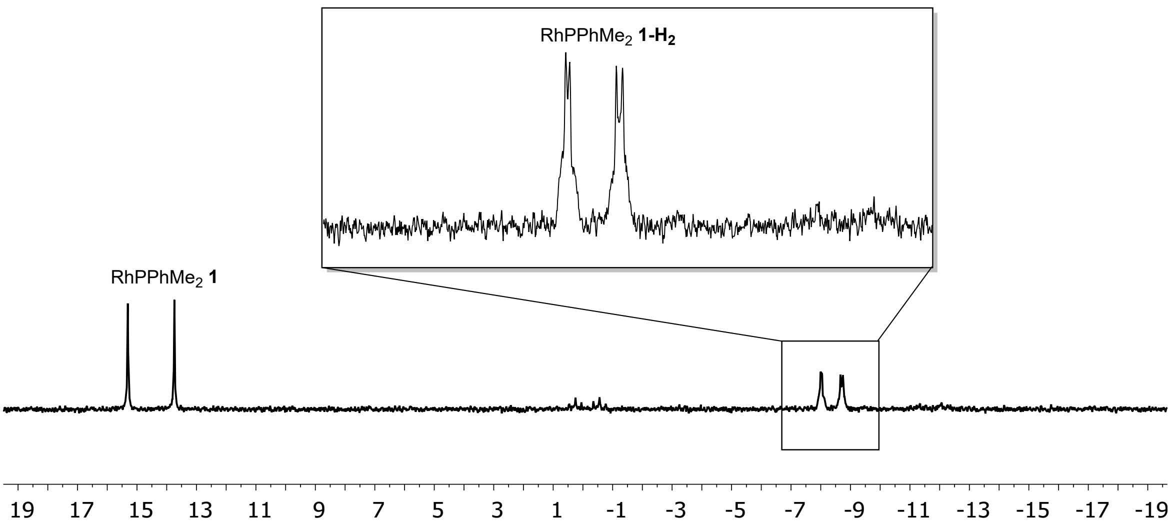


Figure S12. $^{31}P\{^{1}H\}$ NMR spectra of the reaction of **1** (17 mg, 0.018 mmol) with 3 molar-equiv. of FA (2.58 mg, 0.056 mmol, 2.12 µL) in CD_3CN at RT.

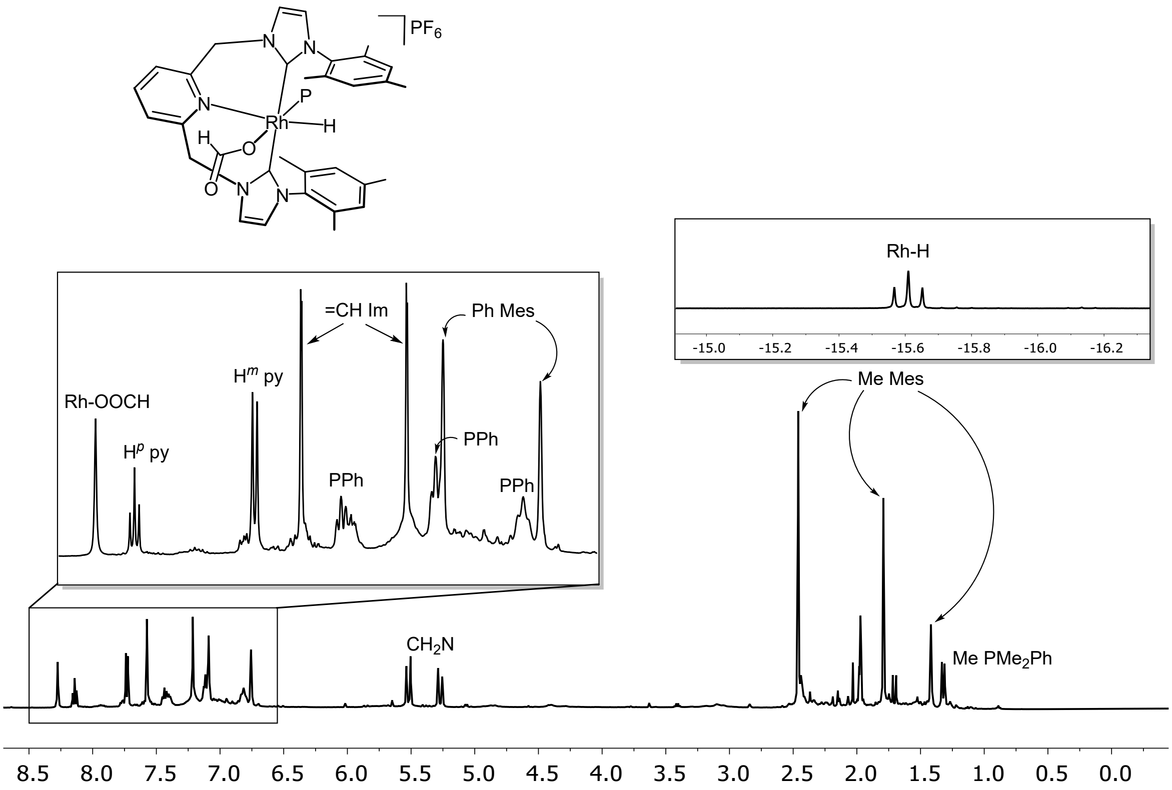


Figure S13. ^{1}H NMR spectrum of $[(CNC)^{Mes}Rh(\kappa^{O}\text{-}OC(O)H)(PMe_2Ph)H]PF_6$ (**2**) in CD_3CN at 263 K.

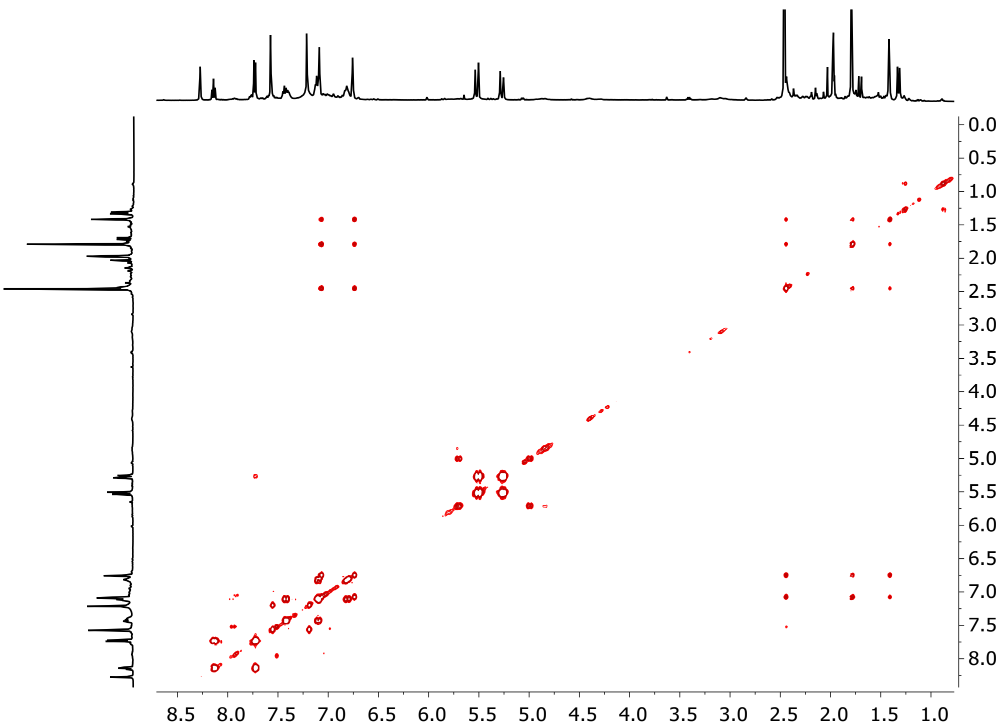


Figure S14. ^{1}H-^{1}H COSY NMR spectrum of $[(CNC)^{Mes}Rh(\kappa^{O}\text{-}OC(O)H)(PMe_2Ph)H]PF_6$ (**2**) in CD_3CN at 263 K.

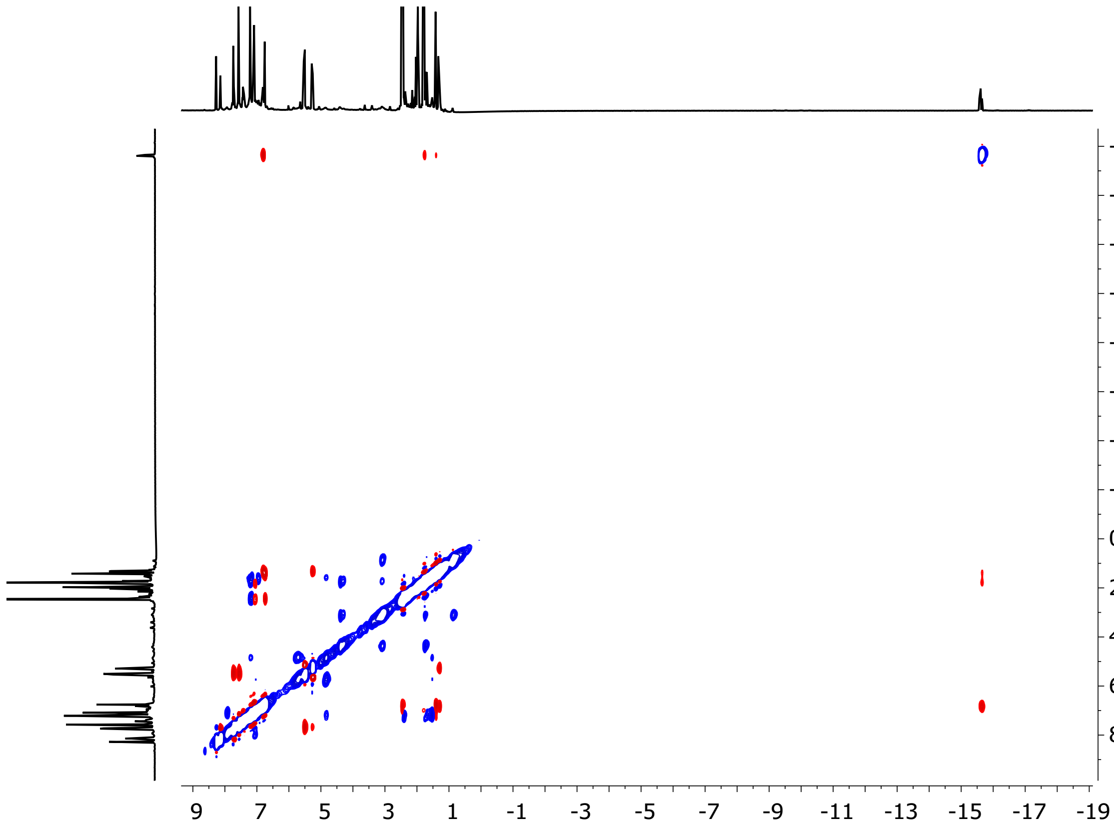


Figure S15. ^{1}H-^{1}H NOESY NMR spectrum of $[(CNC)^{Mes}Rh(\kappa^{O}$-$OC(O)H)(PMe_2Ph)H]PF_6$ (**2**) in CD_3CN at 263 K.

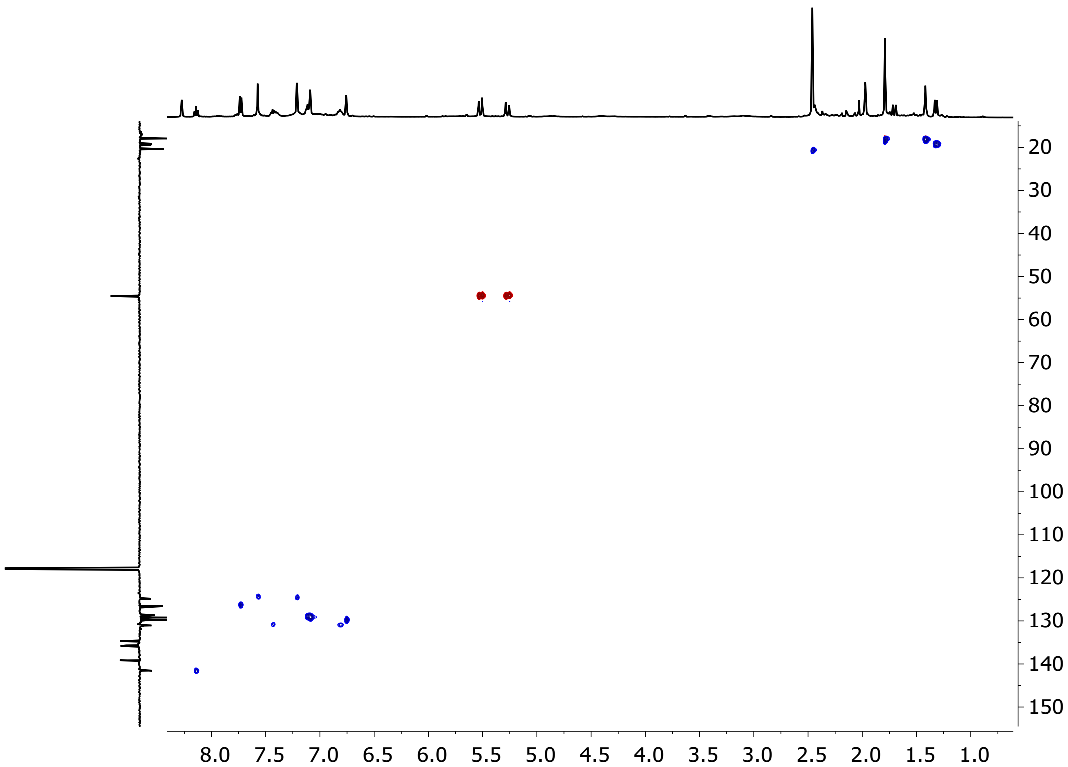


Figure S16. ^{1}H-^{13}C HSQC NMR spectrum of $[(CNC)^{Mes}Rh(\kappa^{O}$-$OC(O)H)(PMe_2Ph)H]PF_6$ (**2**) in CD_3CN at 263 K.

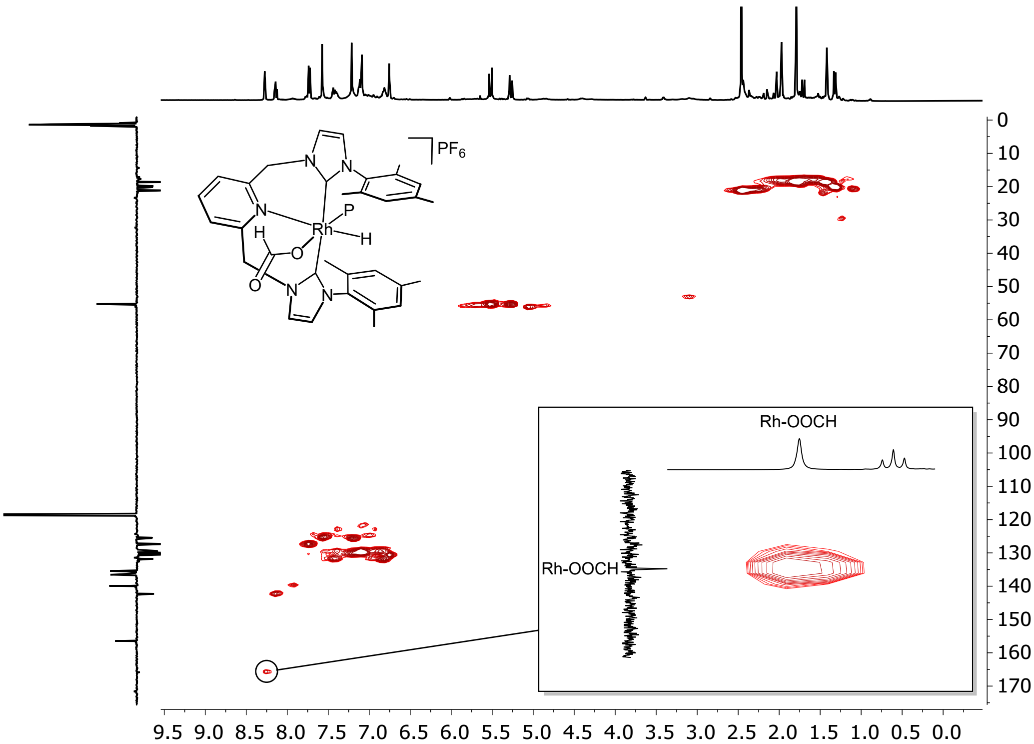


Figure S17. ^{1}H-^{13}C HMBC NMR spectrum of [(CNC)^{Mes}Rh(κ^{O}-OC(O)H)(PMe_2Ph)H]PF_6 (**2**) in CD_3CN at 263 K.

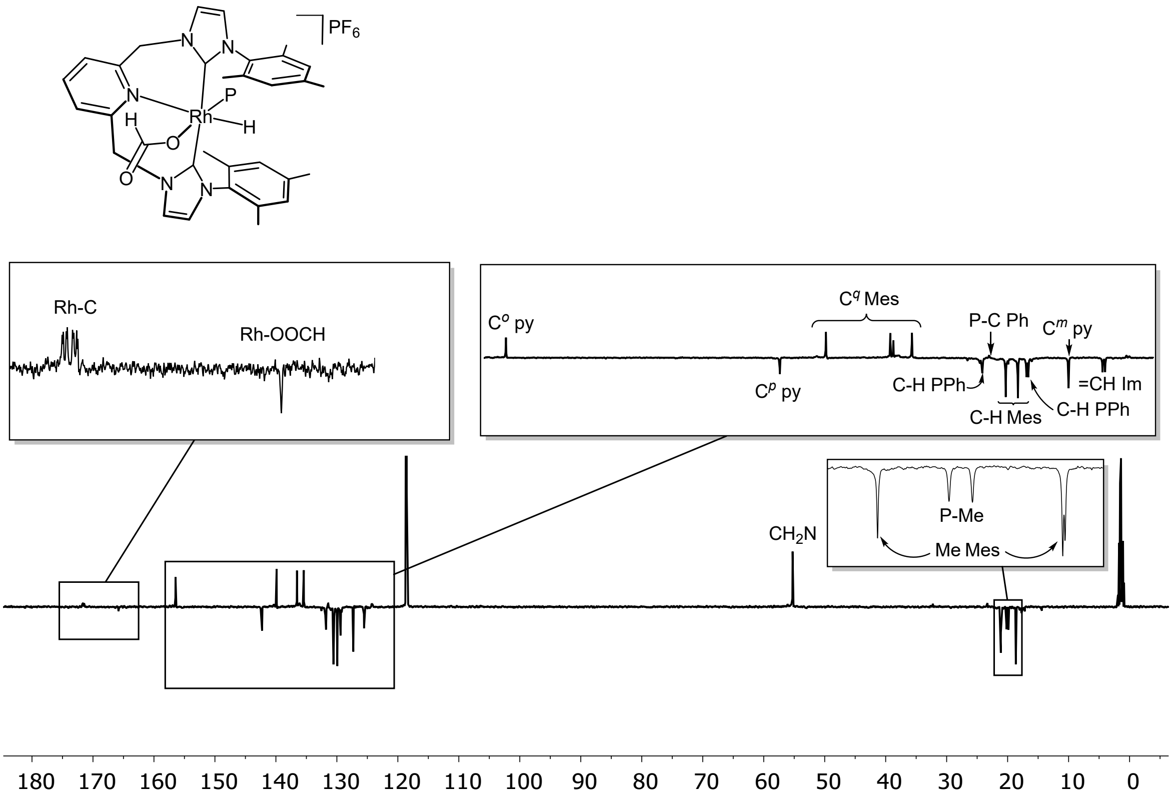


Figure S18. $^{13}C\{^{1}H\}$-APT NMR spectrum of [(CNC)^{Mes}Rh(κ^{O}-OC(O)H)(PMe_2Ph)H]PF_6 (**2**) in CD_3CN at 263 K.

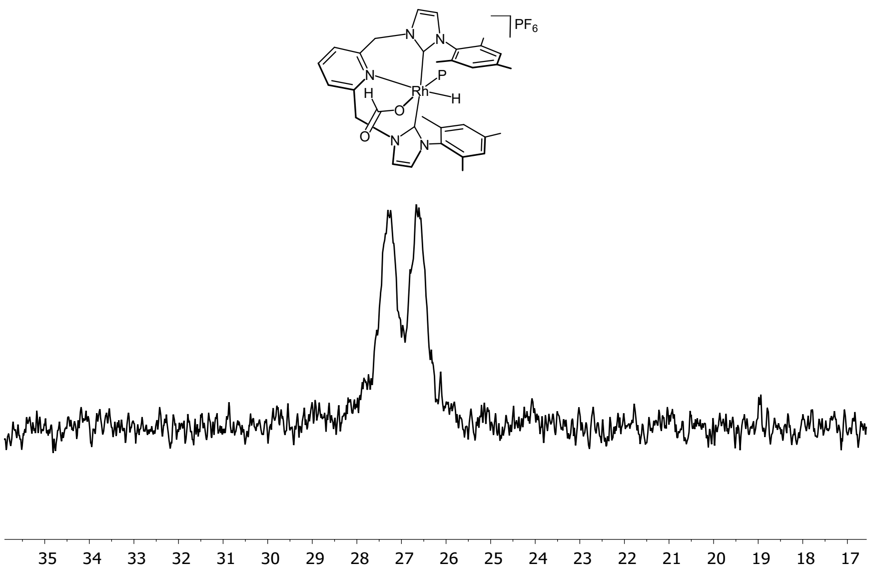


Figure S19. $^{31}P\{^{1}H\}$ NMR spectrum of $[(CNC)^{Mes}Rh(\kappa^{O}\text{-}OC(O)H)(PMe_2Ph)H]PF_6$ (**2**) in CD_3CN at 26 K.

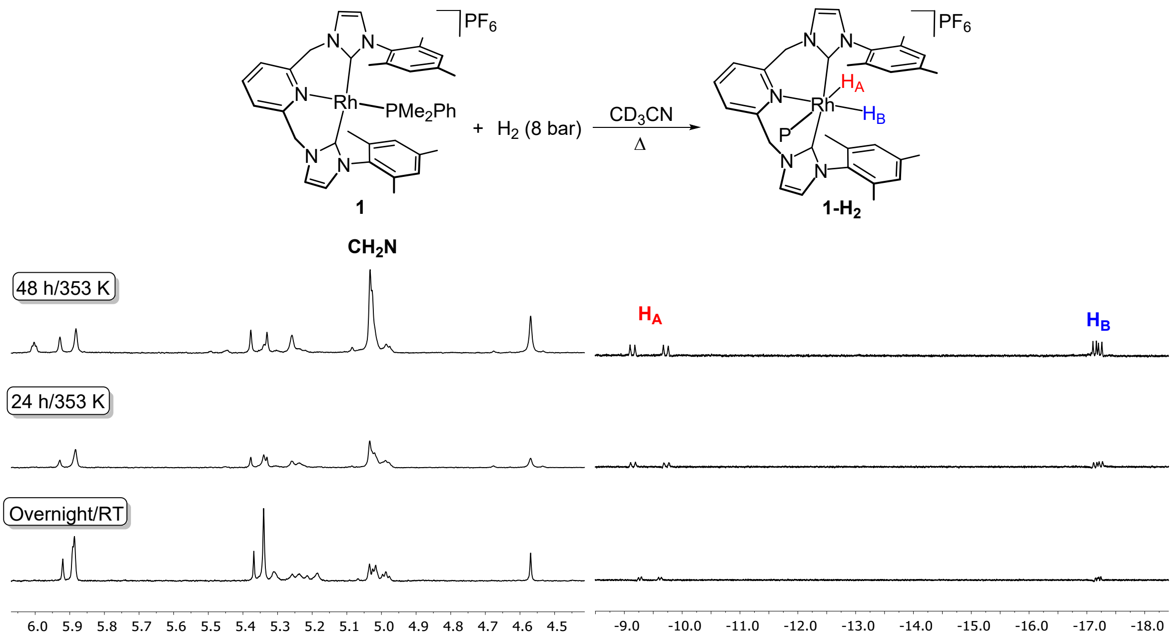
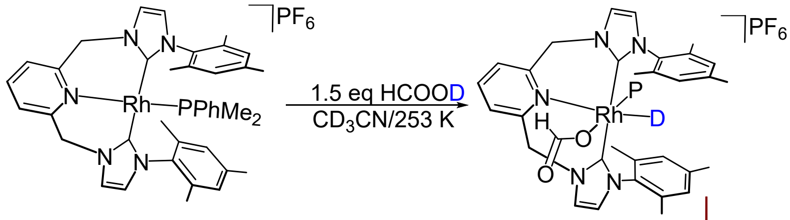


Figure S20. Selected areas of the ^{1}H NMR spectra of the reaction of **1** with H_2 (8 bar) in CD_3CN at 353 K in a J Young NMR tube.

6. Deuterium labelling experiments.

A J-Young NMR tube was charged with **1** (6 mg, 3.7 μmol), sodium formate (25 mg, 367 μmol), deuterated formic acid (5.55 mmol) and 0.3 mL of CD_3CN.



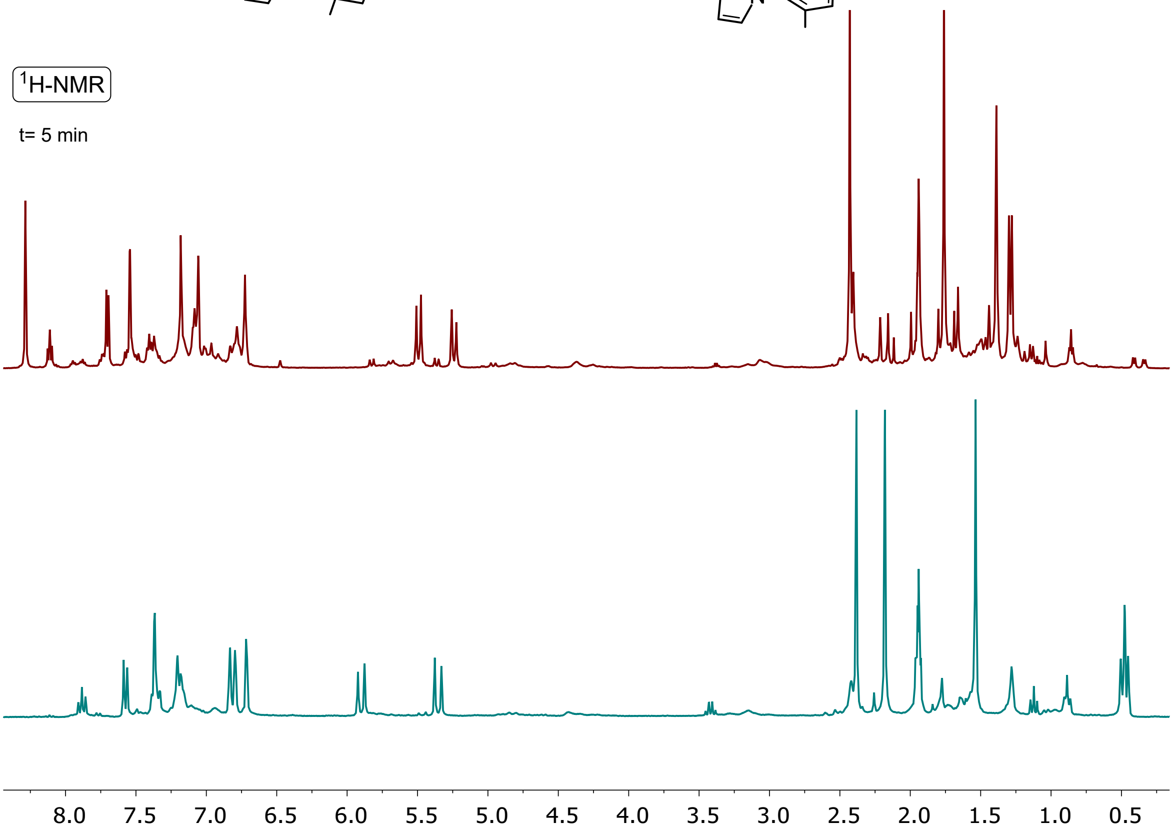


Figure S21. ^1H NMR comparative spectra of $[(CNC)^{Mes}Rh(\kappa^O\text{-}OC(O)D)(PMe_2Ph)D]PF_6$ (up) and **1** (bottom) in CD_3CN at 253 K.

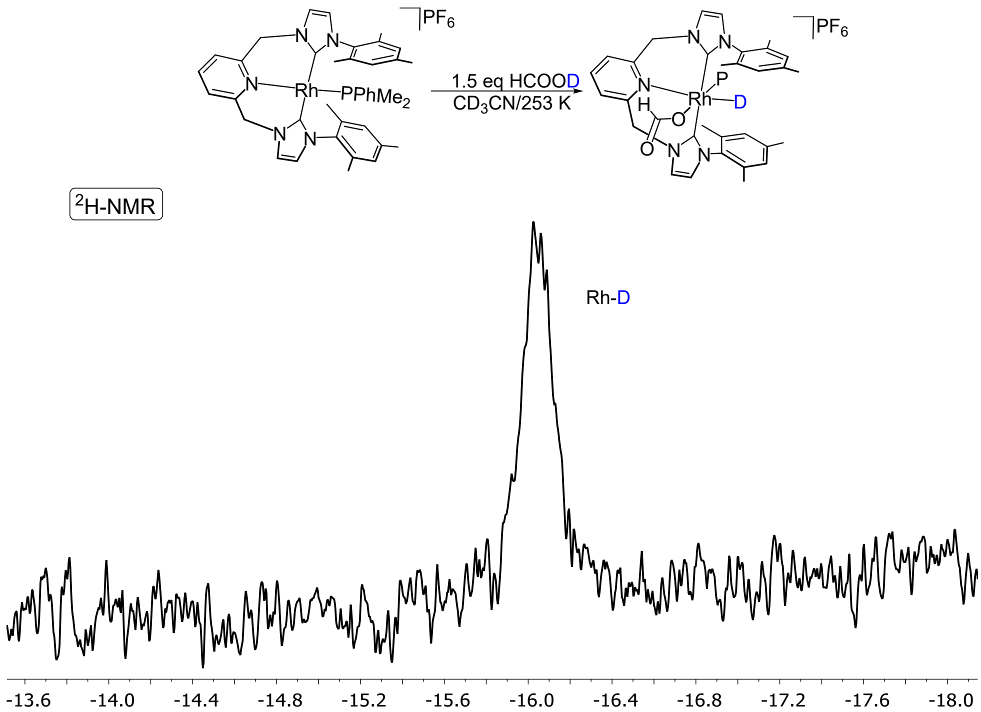


Figure S22. ^{2}H NMR spectrum (hydride area) of the *in situ* reaction of **1** with HCOOD, affording deuteride $[(CNC)^{Mes}Rh(\kappa^{O}\text{-}OC(O)H)(PMe_2Ph)D]PF_6$ (**2-D**) in CD_3CN at 253 K.

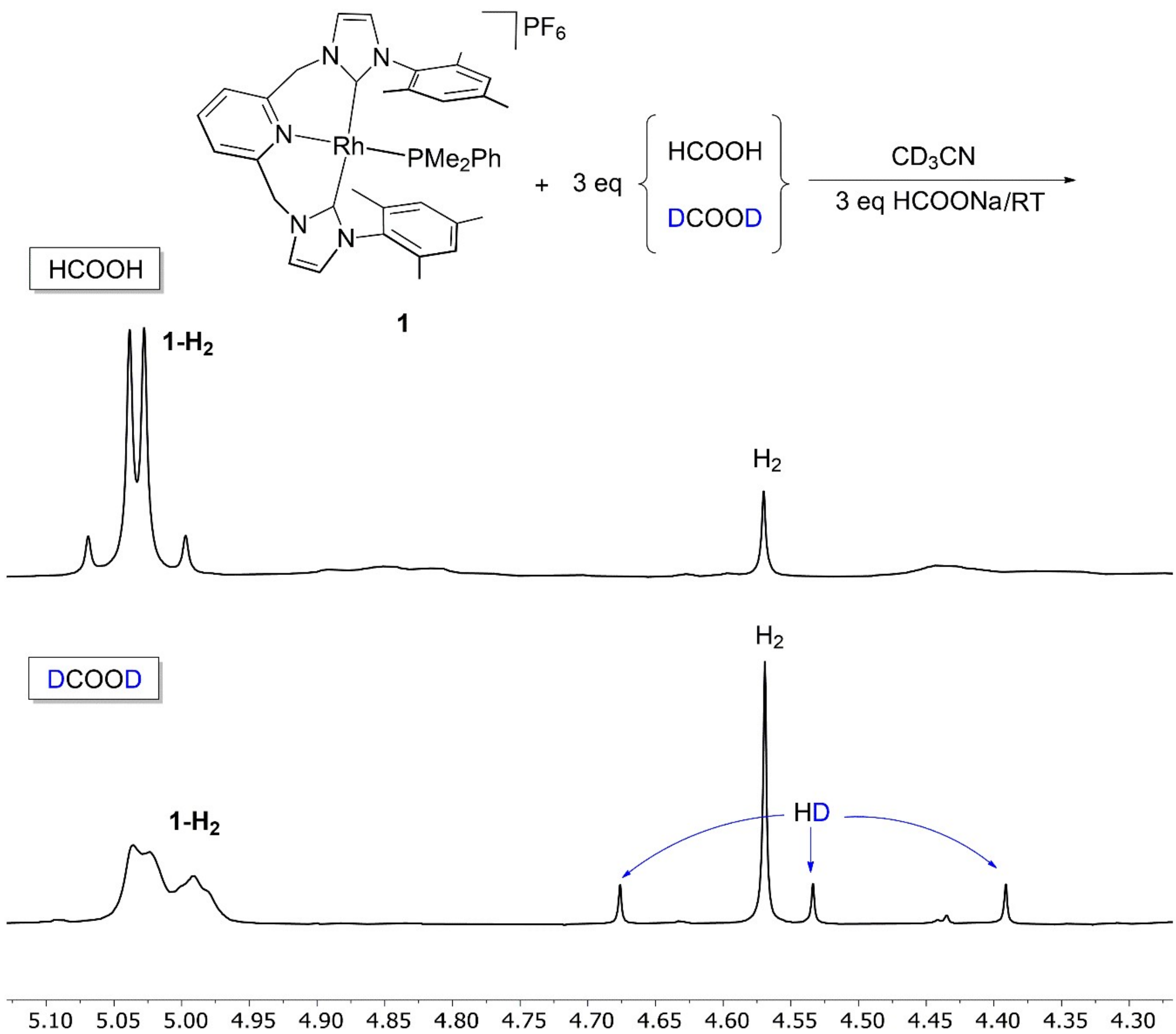


Figure S23. ^{1}H NMR spectra of the reaction of **1** with HCOOH in CD_3CN at RT (top), and with DCOOD (bottom).

7. Kinetic isotopic effect (KIE).

To obtain values for k_H/k_D, the initial rates of dehydrogenation using formic acid (HCOOH), and deuterium-labelled DCOOD, HCOOD, and DCOOH, and sodium formate-d_1 (DCOONa), were obtained. Inside a drybox, rhodium catalyst **1** (1.71 mg, 2.1 µmol, 0.075 % mol) and sodium formate (30 %, 270 mg, 3.97 mmol) were weighted and transferred to the reactor vessel, and then heated to 353 K. At this point, under an argon atmosphere, 500 µL of neat, deuterated formic acid was injected into the reactor vessel. Instantaneously, from the resulting yellow solution a high amount of gas bubbles appeared, generating an increasing pressure that was monitored by a pressure transducer connected to the reactor. The catalytic reaction was left under stirring until complete catalyst deactivation.

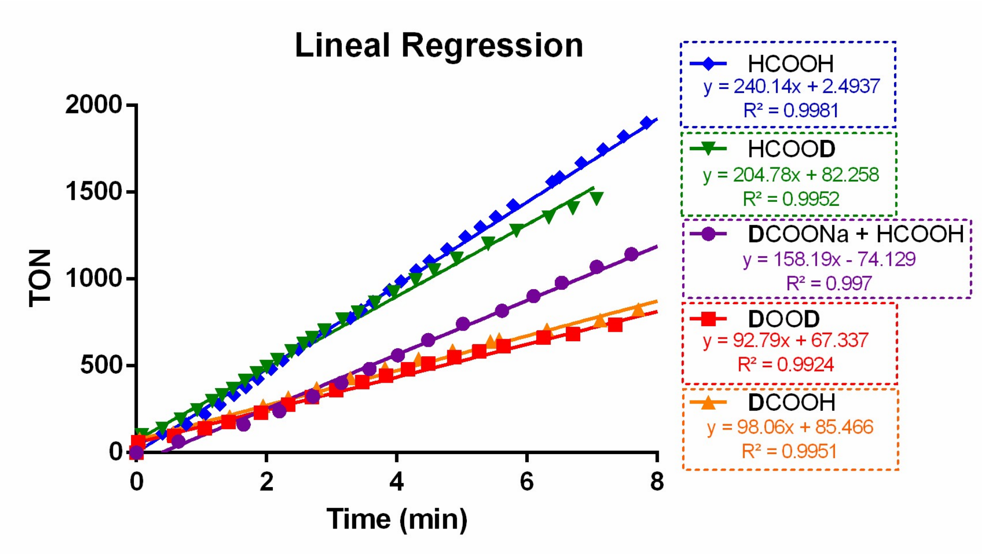


Figure S24. Kinetic profiles (lineal section) of moles of FA decomposed *vs* time for the kinetic isotope effect experiments on the catalytic dehydrogenation of isotopomers of FA with catalyst **1**.

FA derivative	K	K_{rel}	KIE effect
HCOOH	240.14	1	k_{DCOOD}/k_{DCOOH} = 1.05
DCOOD	92.79	2.58	k_{DCOOD}/k_{HCOOD} = 2.21
DCOOH	98.058	2.45	k_{HCOOH}/k_{HCOOD} = 1.17
HCOOD	204.78	1.17	k_{HCOOH}/k_{DCOOD} = 2.58
30% DCOONa/HCOOH	158.19	1.52	k_{HCOOH}/k_{DCOOH} = 2.44
			k_{HCOONa}/k_{DCOONa} = 1.51

8. Eyring plot.

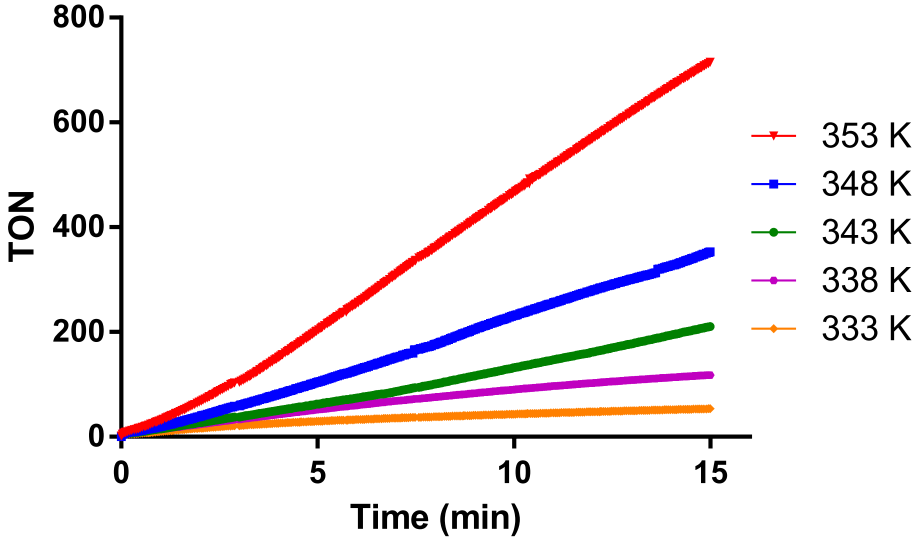


Figure S25. Representation of TON (mol of H_2 per mol of Rh) *vs.* time of the solventless FA dehydrogenation catalysed by **1** (0.016 % mol) in the presence of HCOONa (30 % mol) at different temperatures.

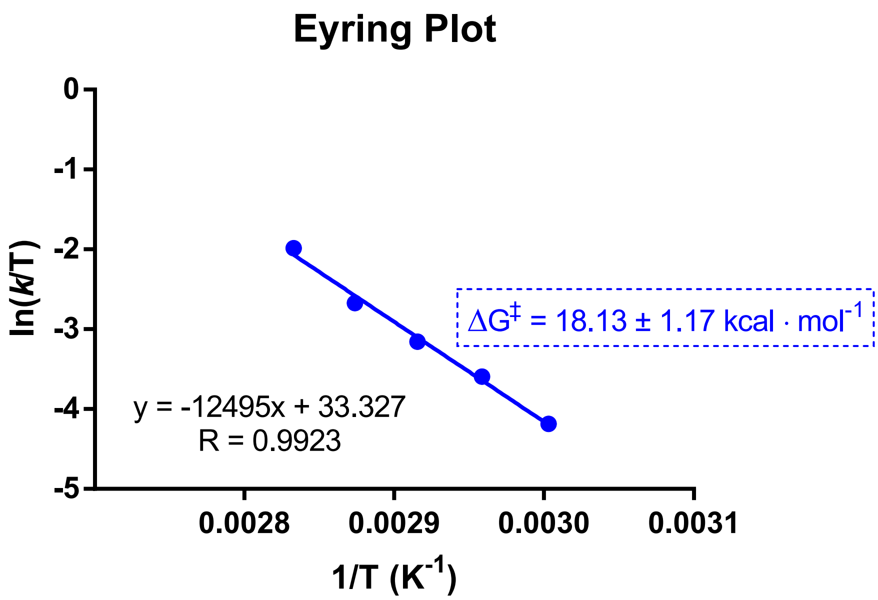


Figure S26. Eyring plot representation of ln(K/T) *vs* 1/T.

9. DFT studies.

Table S1. Energetic data of DFT calculated species. Total energies for all optimized structures calculated at B3LYP-D3(PCM)/def2SVP level (E(B3LYP-D3(PCM)/DZ)), thermodynamic corrections calculated at the same level to obtain Gibbs free energies including quasi-harmonic corrections, at 1M and 353.15K (Gcorr(B3LYP-D3(PCM)/DZ,1M,353K)-qh). Single point energies calculated at M06L(SMD)/def2TZVP level (E(M06L(SMD)/TZ)). All absolute energies in atomic units. Relative Gibbs free energies (to **A** and isolated molecules, in kcal mol^{-1}) ∆G(M06L(SMD)/TZ,1M,353K)-qh calculated using E(M06L(SMD)/TZ energy plus Gcorr(B3LYP-D3(PCM)/DZ,1M,353K)-qh correction.

	E(B3LYP-D3(PCM)/DZ)	Gcorr(B3LYP-D3(PCM)/DZ,1M,353K)-qh	E(M06L(SMD)/TZ)	∆G(M06L(SMD)/TZ, 1M,353K)-qh
A	-2237.2042	0.6639	-2238.8012	0
B	-2426.843	0.6934	-2428.6244	9.1
TSBC	-2426.8417	0.6902	-2428.6207	9.4
C	-2426.8815	0.6933	-2428.658	-12.1
C+FA	-2616.5389	0.723	-2618.4923	-9.9
TSCD	-2616.5035	0.7219	-2618.4727	1.7
D	-2616.514	0.7207	-2618.4741	0.1
TSDE	-2616.5049	0.7159	-2618.4617	4.8
E	-2616.5205	0.7147	-2618.4765	-5.2
TSEC	-2617.6984	0.7346	-2619.6475	0.2
C+H_2+CO_2	-2617.713	0.7224	-2619.6631	-17.3
A+HCOO-	-2426.3774	0.6801	-2428.1646	6.7
B'	-2426.3695	0.6811	-2428.1607	9.8
TSBC'	-2426.3482	0.6749	-2428.1264	27.4
C'	-2426.3549	0.6749	-2428.1422	17.5
F	-2616.4744	0.7146	-2618.4446	14.7
CO_2	-188.4472	-0.0098	-188.6387	
H_2	-1.1741	-0.0006	-1.1708	
HCOOH	-189.629	0.0082	-189.8163	
$HCOO^-$	-189.1464	-0.0049	-189.353	

Full citation for reference 22:

Gaussian 09, Revision D.01, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, G. A. Petersson, H. Nakatsuji, X. Li, M. Caricato, A. Marenich, J. Bloino, B. G. Janesko, R. Gomperts, B. Mennucci, H. P. Hratchian, J. V. Ortiz, A. F. Izmaylov, J. L. Sonnenberg, D. Williams-Young, F. Ding, F. Lipparini, F. Egidi, J. Goings, B. Peng, A. Petrone, T. Henderson, D. Ranasinghe, V. G. Zakrzewski, J. Gao, N. Rega, G. Zheng, W. Liang, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, K. Throssell, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, J. M. Millam, M. Klene, C. Adamo, R. Cammi, J. W. Ochterski, R. L. Martin, K. Morokuma, O. Farkas, J. B. Foresman, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2016.

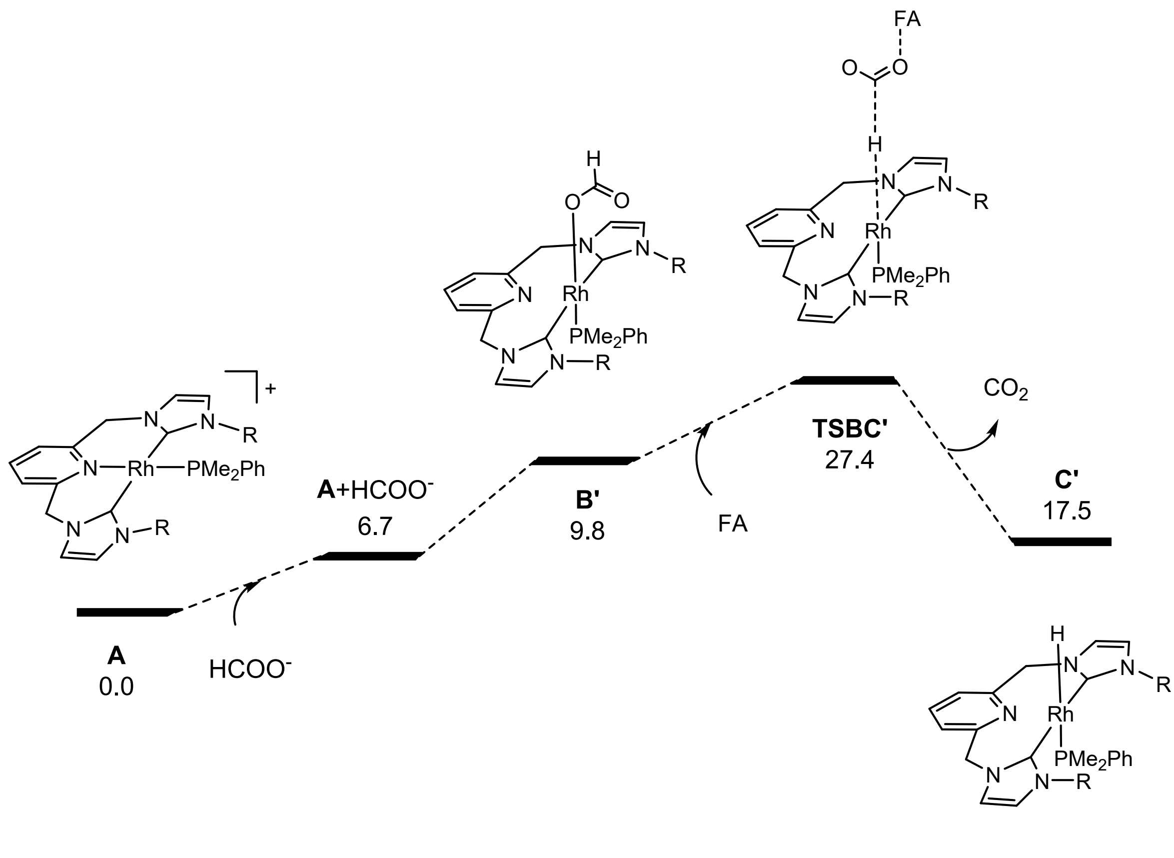


Figure S27. DFT energetic profile (ΔG in kcal mol^{-1}, relative to **A** and isolated molecules) for the CO_2 release from formate anion by complex **1** via hydrogen abstraction.

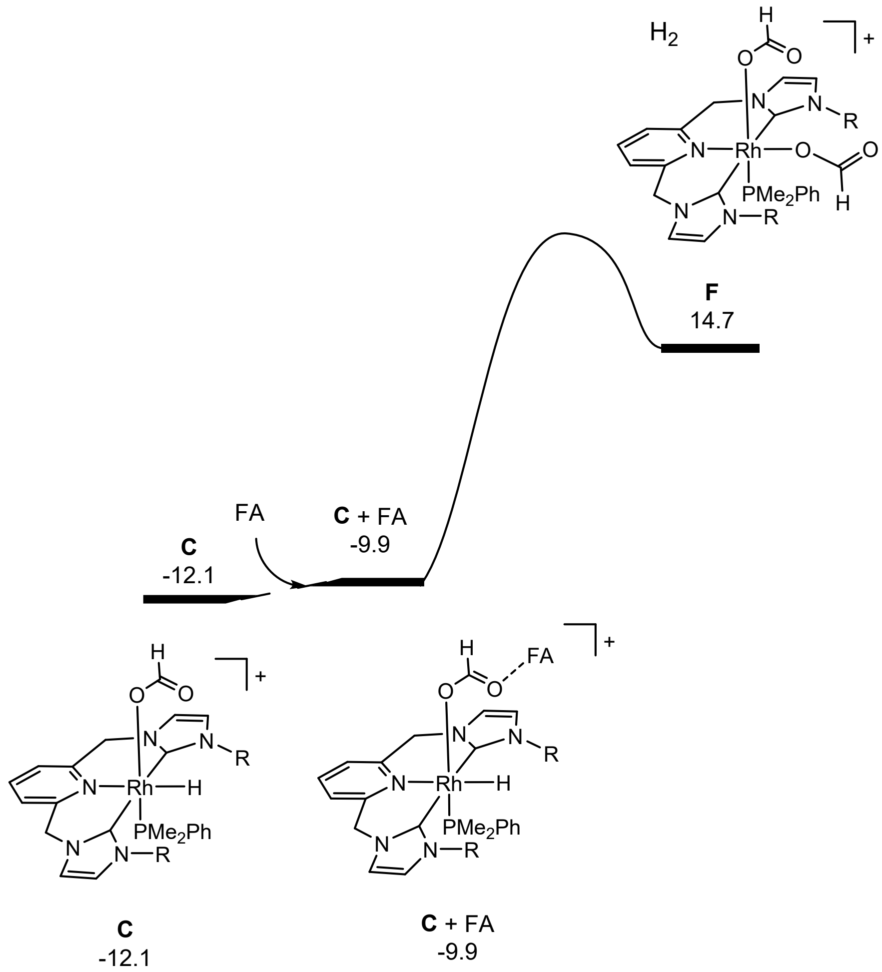
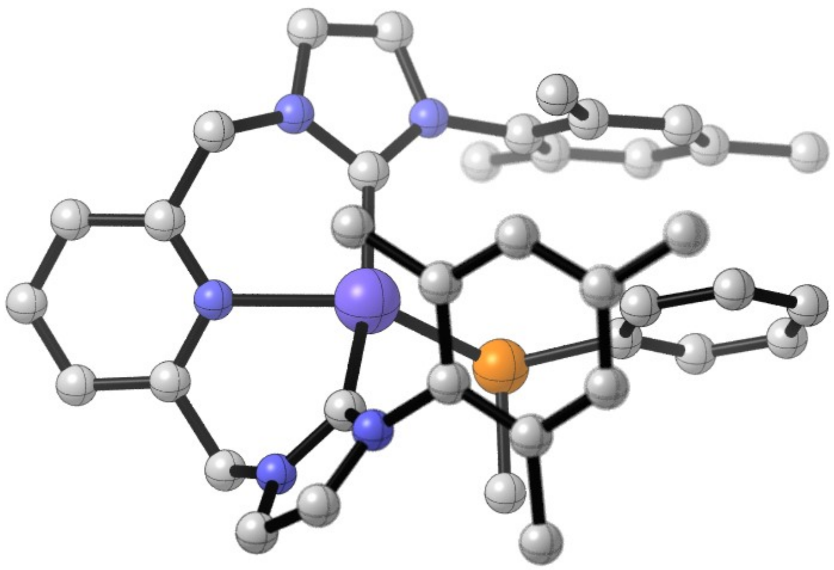
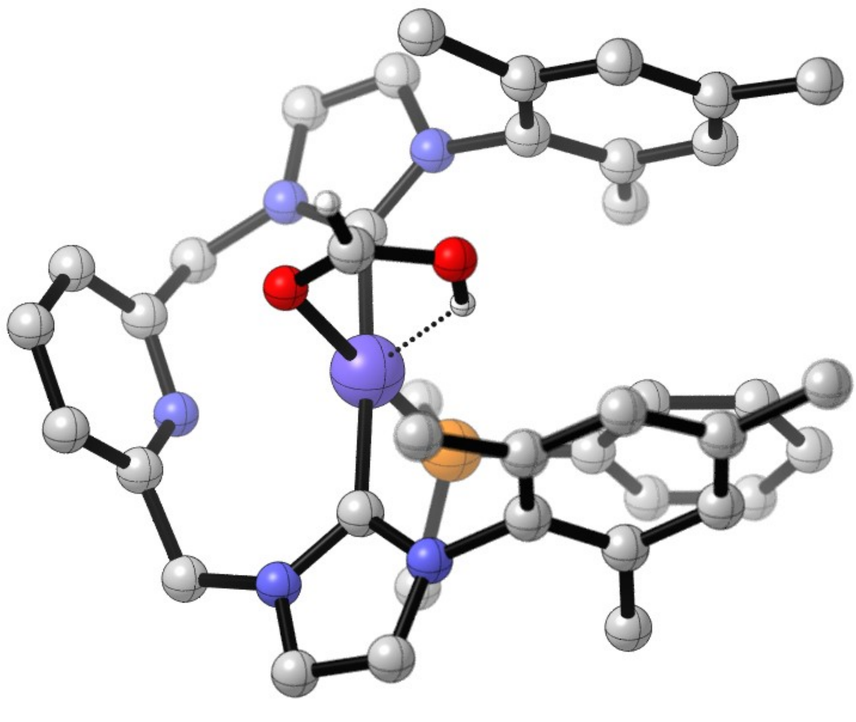


Figure S28. DFT energetic profile (ΔG in kcal mol^{-1}, relative to **A** and isolated molecules) for the formation of H_2 from intermediate **C**.

Figure S29. Geometrical representation of DFT optimized structures. Key distances in Å. Hydrogen atoms of the $(CNC)^{Mes}$ and PMe_2Ph ligands omitted for clarity.



A



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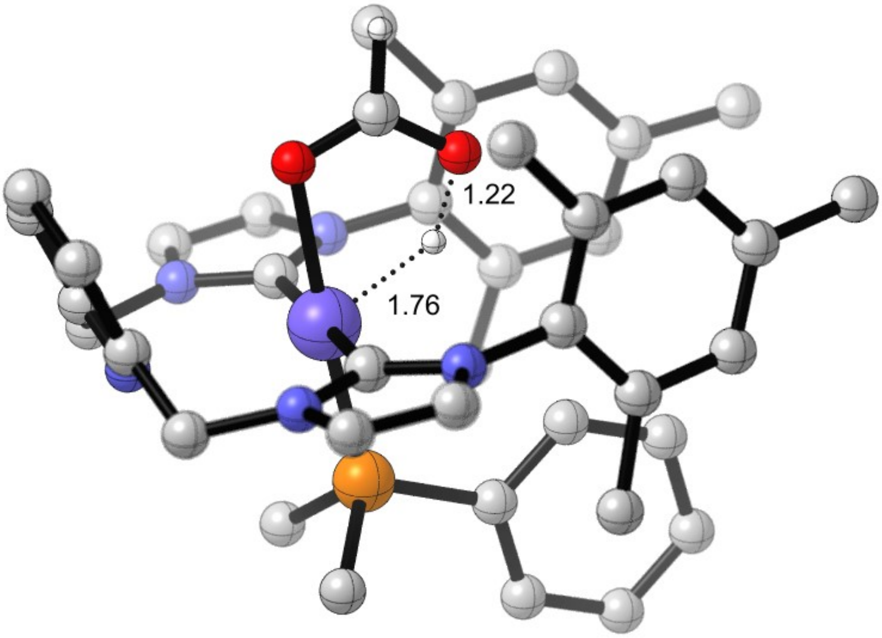

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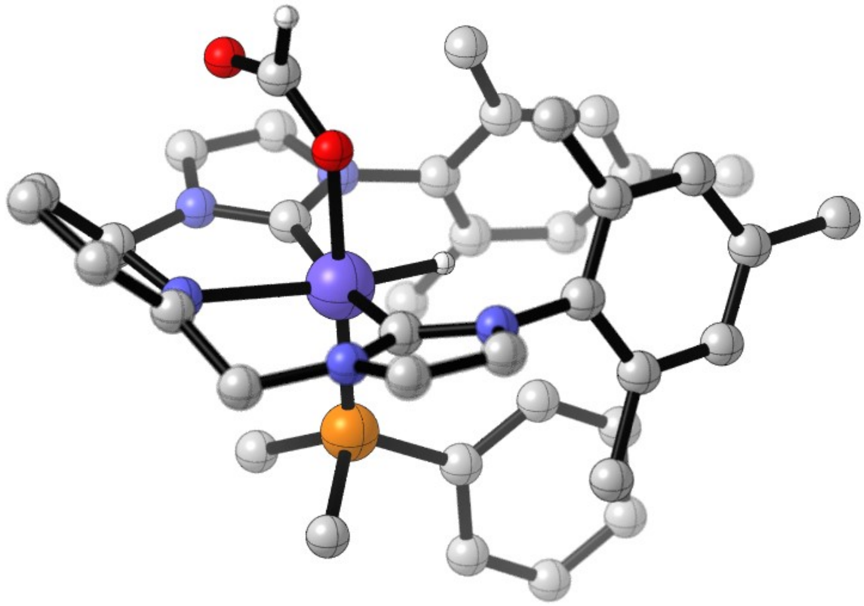

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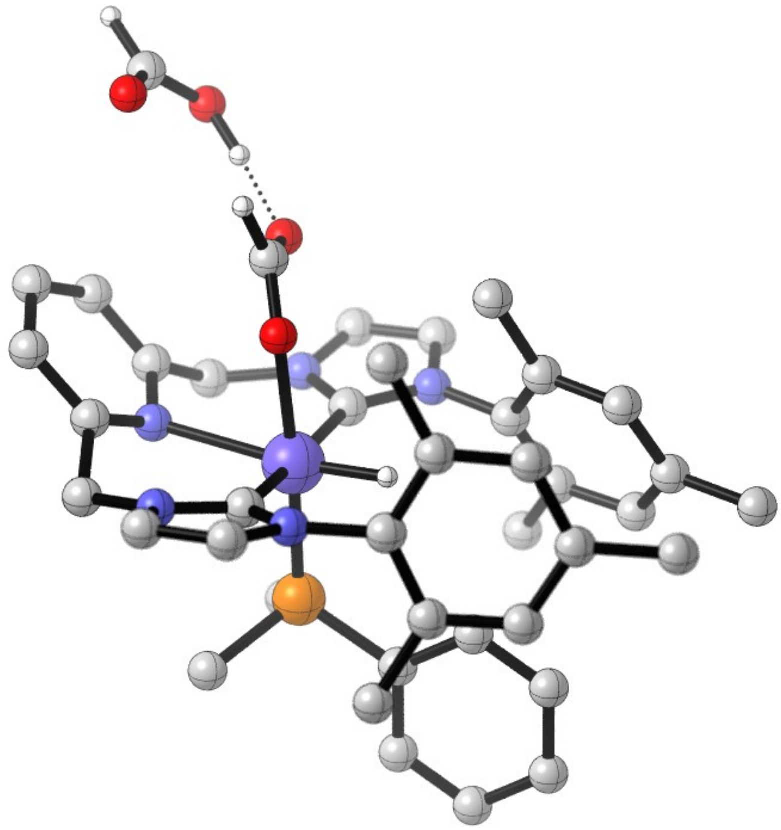

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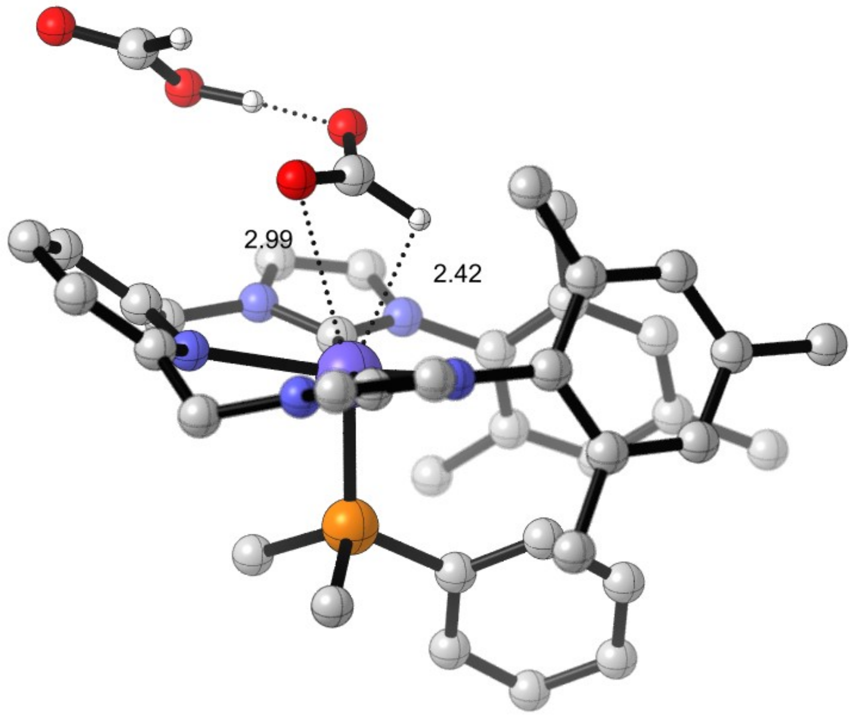

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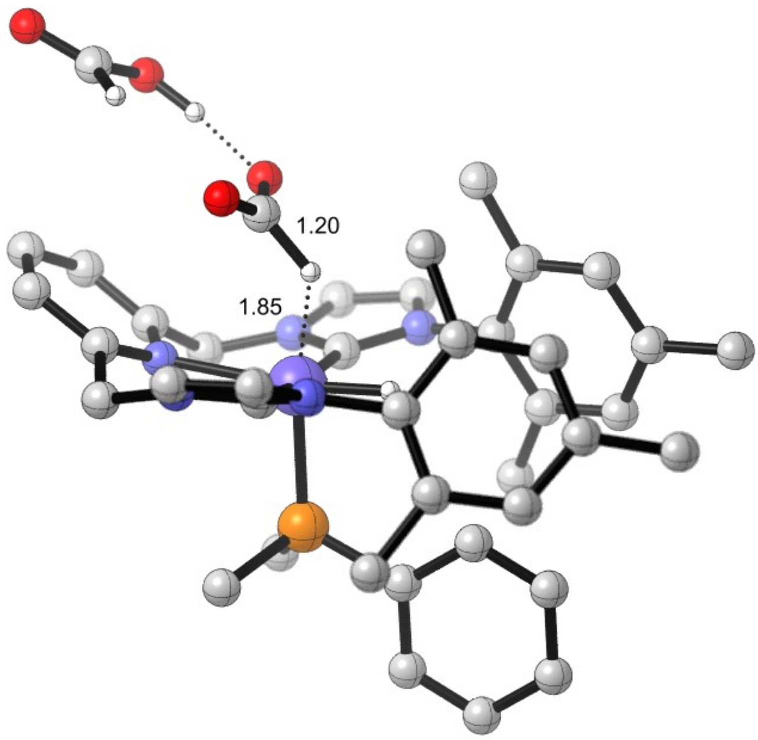

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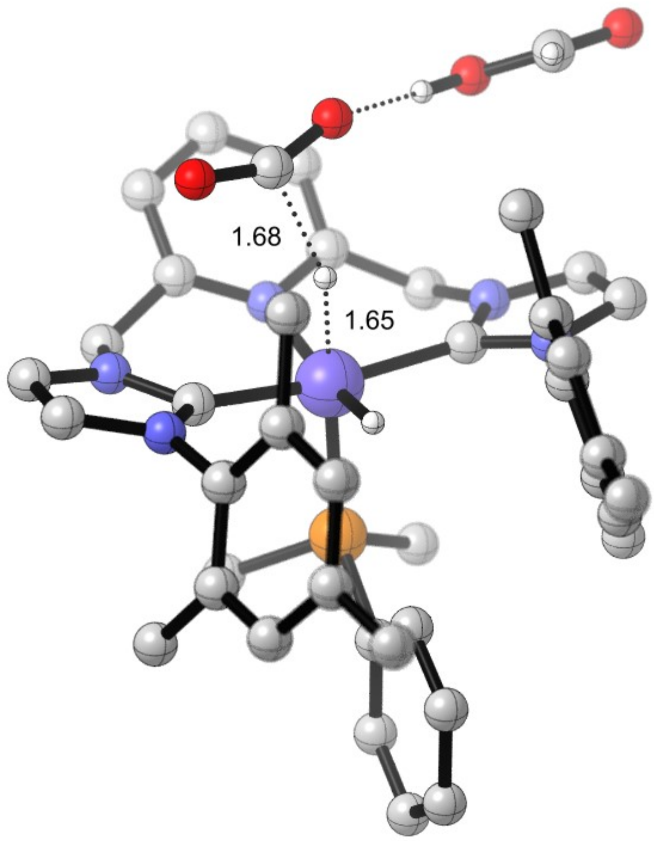

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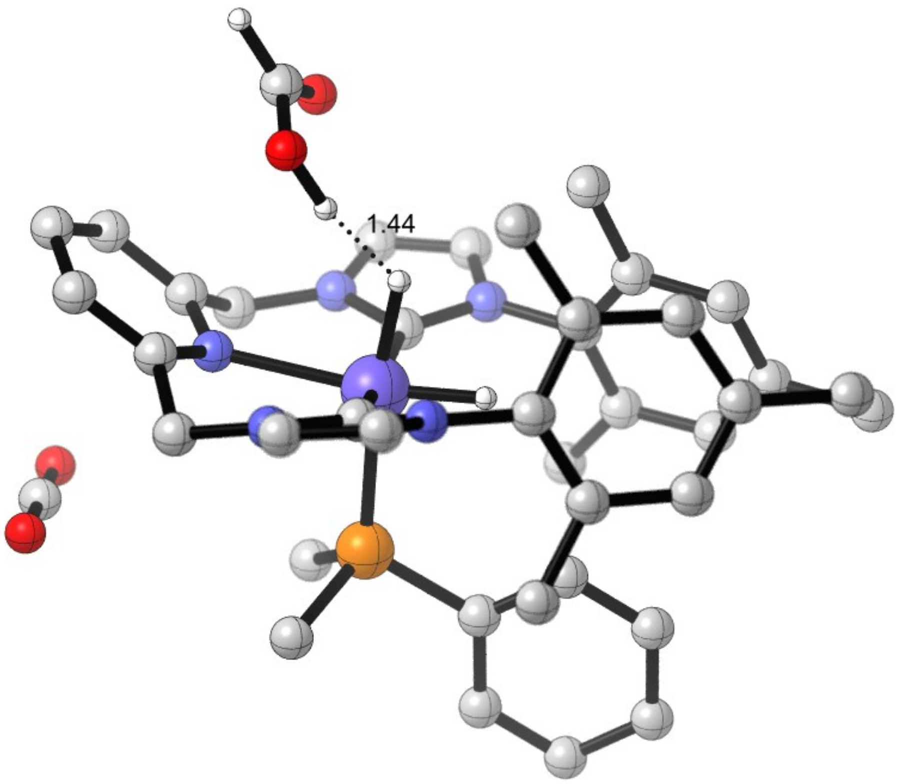

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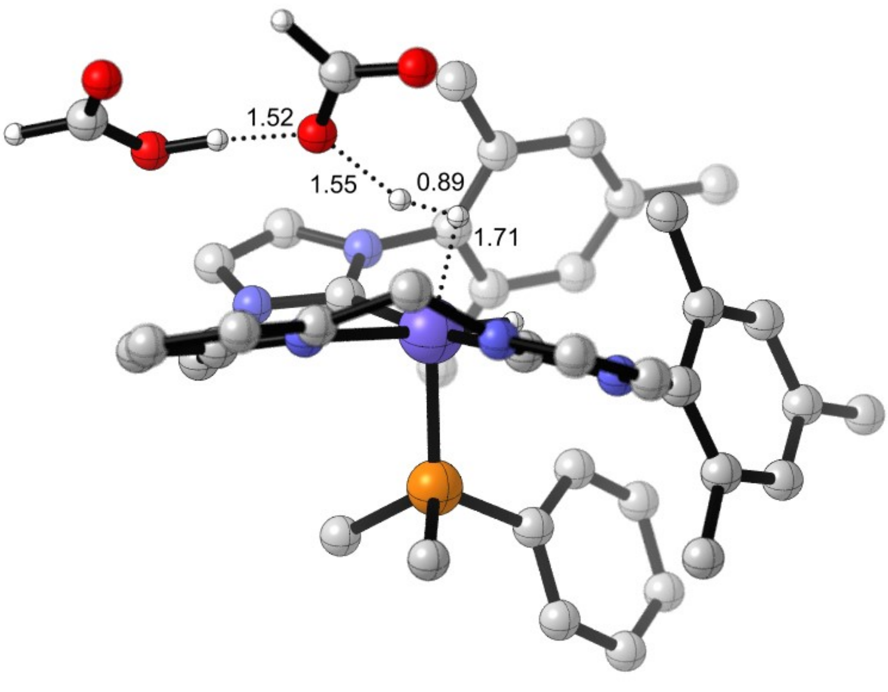

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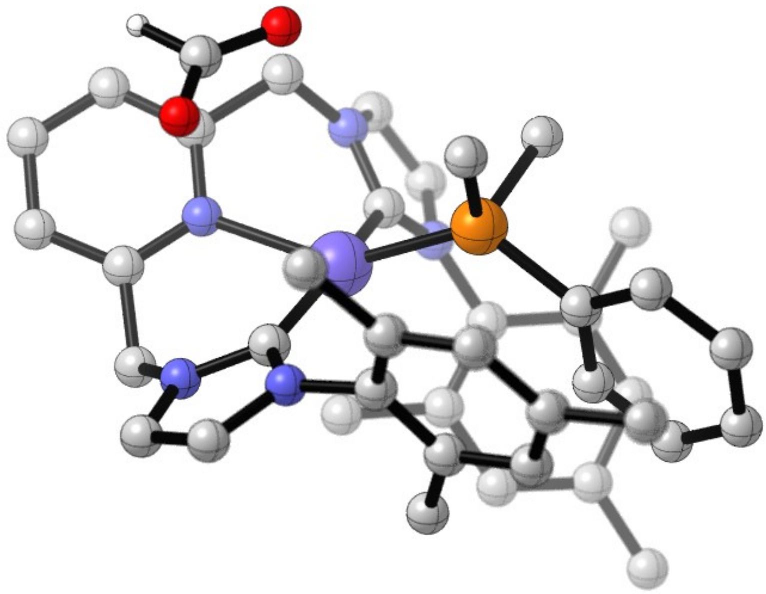

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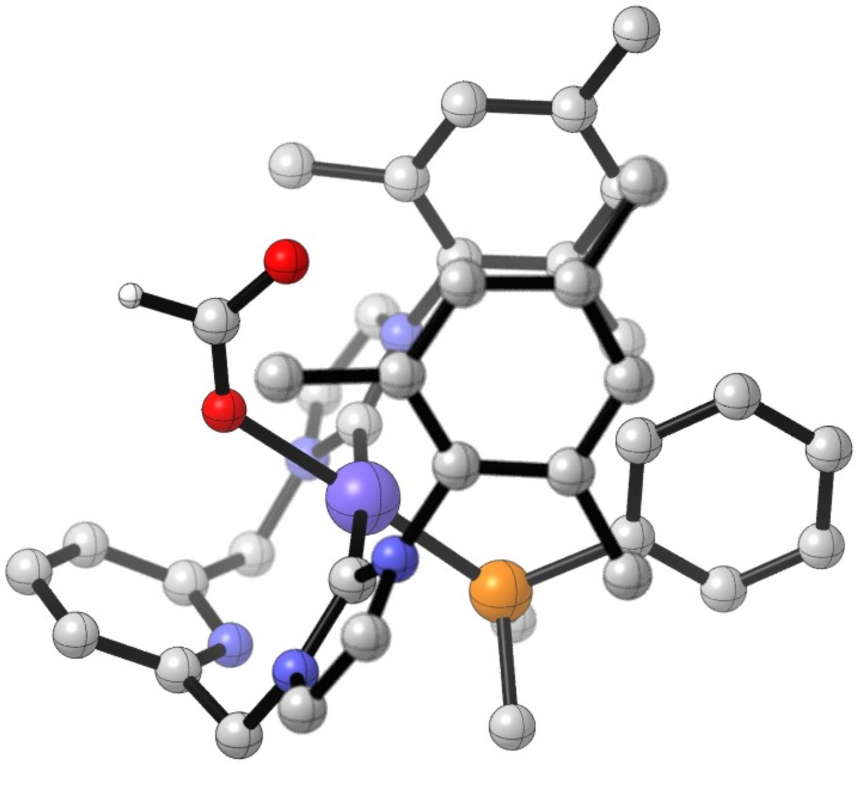

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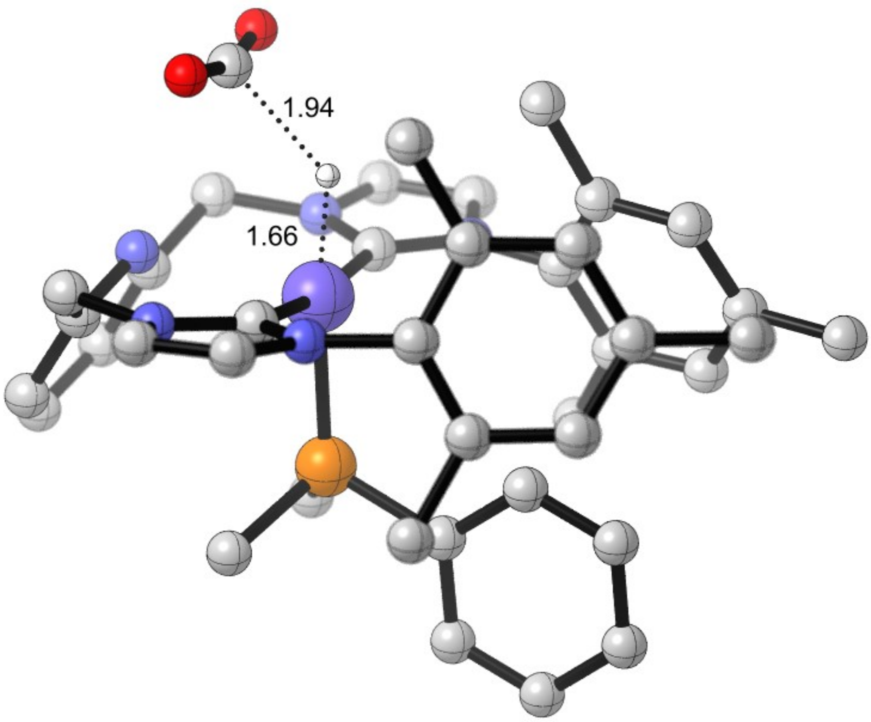

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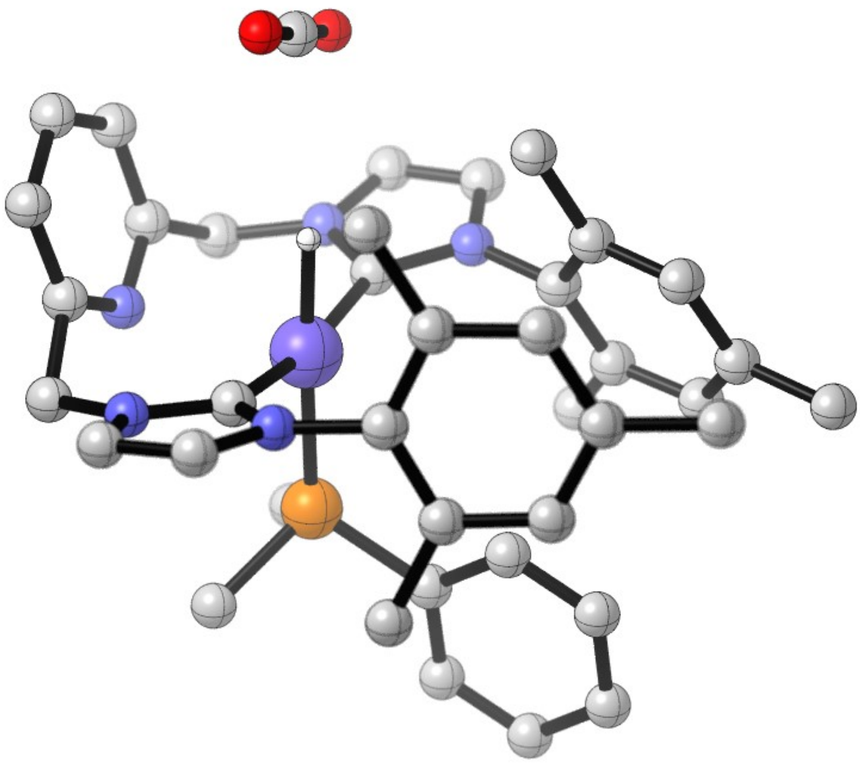

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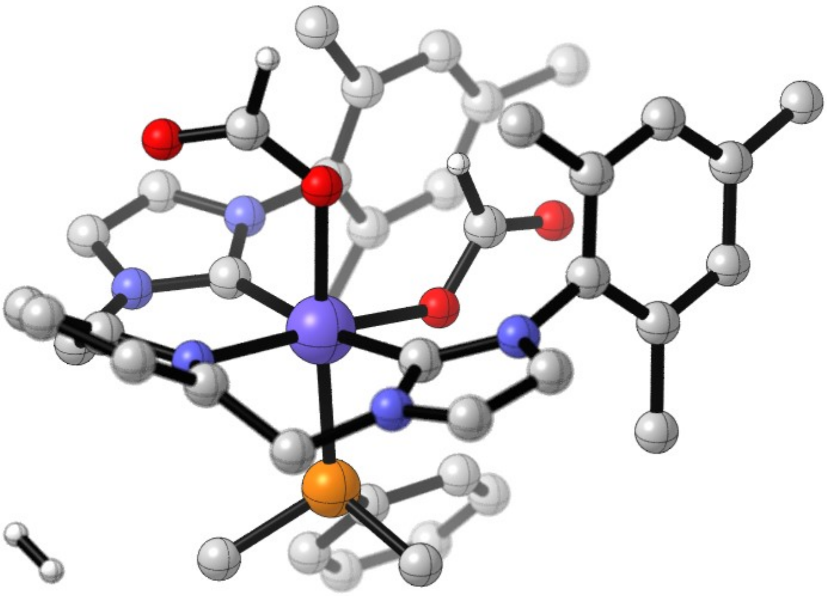

F

Table S2. Cartesian coordinates (in Å) for all DFT optimized structures.

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7	1.455449	-2.642955	1.748016
7	-0.543262	-2.867373	1.009710
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1	2.970830	4.524763	-0.489528
6	3.692184	2.683632	-1.515010
6	3.645695	0.206384	-2.086746
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1	5.210163	-3.141435	1.770374
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