Electronic Supplementary Material (ESI) for RSC Advances

Electronic Supplementary Information

Mononuclear palladium and heterodinuclear palladium-ruthenium complexes of semicarbazone ligands. Synthesis, characterization, and application in C-C cross-coupling reactions

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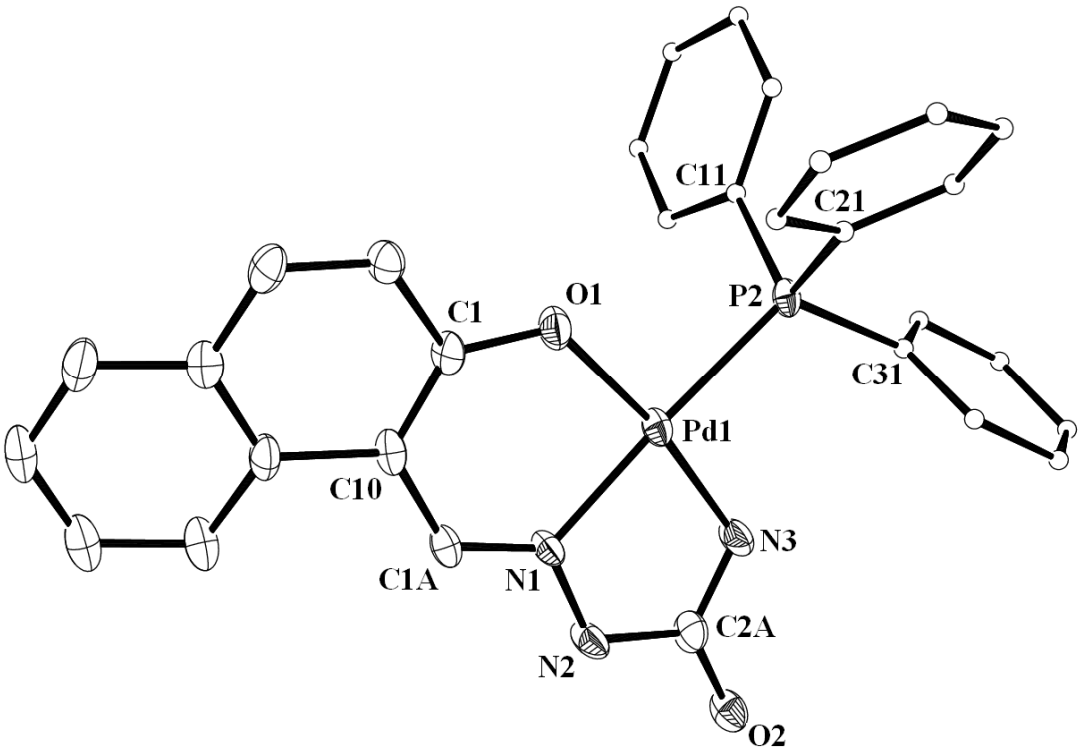

Fig. S1 View of the **1c** complex.

Table S1 Selected bond distances and bond angles for **1c** and **2c**

1c			
Bond distances (Å)			
Pd1-P(2)	2.262(2)	C(1)-O(1)	1.323(11)
Pd1-O(1)	2.005(8)	C(1A)-N(1)	1.290(11)
Pd1-N(1)	1.979(7)	N(1)-N(2)	1.373(11)
Pd1-N(3)	1.965(9)	N(2)-C(2A)	1.376(14)
		C(2A)-N(3)	1.336(5)
		C(2A)-O(2)	1.251(13)
Bond angles (º)			
N(1)-Pd(1)-P(2)	174.5(3)	N(1)-Pd(1)-O(1)	91.3(3)
O(1)-Pd(1)-N(3)	171.2(3)	N(1)-Pd(1)-N(3)	80.1(3)
2c			
Bond distances (Å)			
Pd(1)-P(3)	2.281(4)	C(51)-O(3)	1.317(18)
Pd(1)-O(3)	1.994(11)	C(41)-N(3)	1.281(17)
Pd(1)-N(3)	2.014(10)	N(2)-N(3)	1.364(16)
Pd(1)-N(1)	1.967(11)	N(2)-C(40)	1.376(17)
Ru(1)-P(2)	2.392(4)	C(40)-N(1)	1.318(18)
Ru(1)-P(1)	2.381(4)	C(40)-O(2)	1.299(18)
Ru(1)-Cl(1)	2.414(4)	C(39)-O(1)	1.191(19)
Ru(1)-C(39)	1.763(15)		
Ru(1)-N(2)	2.054(11)		
Ru(1)-O(2)	2.221(8)		
Bond angles (º)			
N(3)-Pd(1)-P(3)	175.7(3)	N(2)-Ru(1)-Cl(1)	162.8(3)
O(3)-Pd(1)-N(1)	172.0(4)	O(2)-Ru(1)-C(39)	164.0(5)
N(3)-Pd(1)-O(3)	91.6(4)	P(1)-Ru(1)-P(2)	171.7(15)
N(1)-Pd(1)-N(3)	80.3(5)	O(2)-Ru(1)-N(2)	61.5(4)

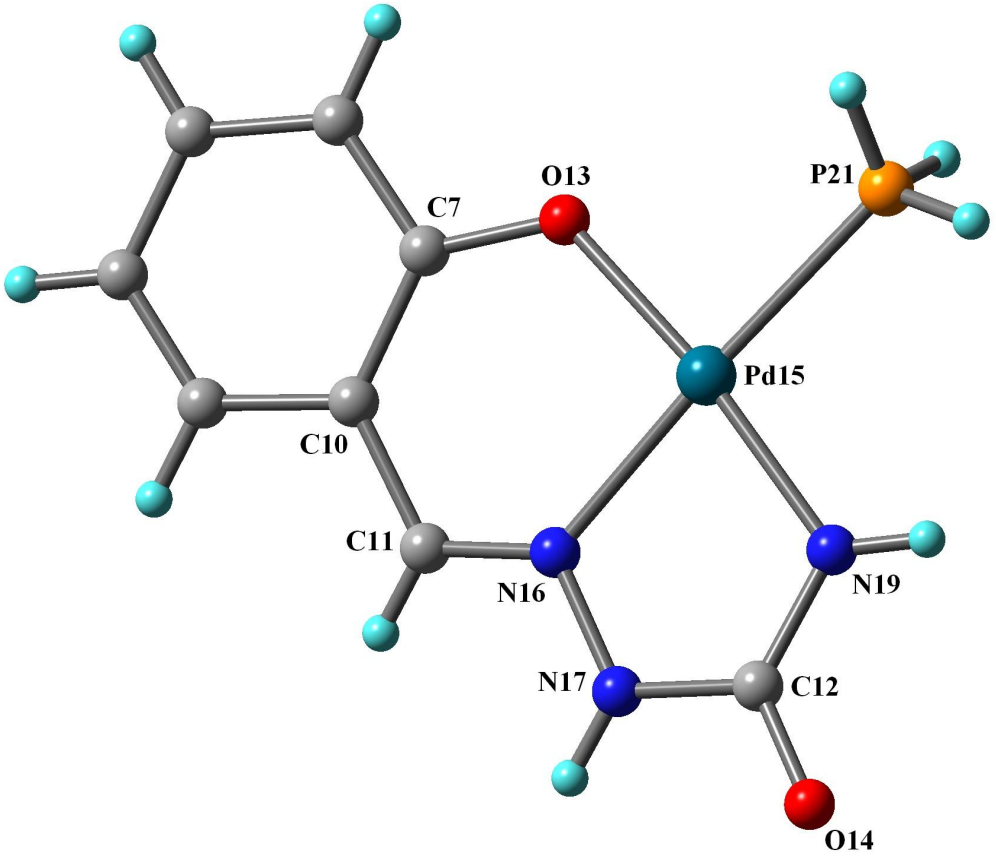

Fig. S2 DFT optimized structure of complex **1a**.

Table S2 Selected bond distances and bond angles for **1a** and **2b**

1a			
Bond distances (Å)			
Pd15-P21	2.269	C7-O13	1.312
Pd15-O13	1.983	C11-N16	1.292
Pd15-N16	2.033	N16-N17	1.386
Pd15-N19	1.976	N17-C12	1.378
		C12-N19	1.337
		C12-O14	1.244
Bond angles (º)			
N16-Pd15-P21	176.62	O13-Pd15-N16	92.62
O13-Pd15-N19	173.00	N16-Pd-N19	80.66
2b			
Bond distances (Å)			
Pd2-P3	2.265	C14-O6	1.312
Pd2-O6	1.998	C24-N9	1.287
Pd2-N9	2.018	N9-N10	1.360
Pd2-N11	1.986	N10-C25	1.353
Ru1-P5	2.385	C25-N11	1.324
Ru1-P4	2.379	C25-O7	1.294
Ru1-Cl13	2.399	C26-O8	1.148
Ru1-C26	1.823		
Ru1-N10	2.065		
Ru1-O7	2.243		
Bond angles (º)			
N9-Pd2-P3	176.58	N10-Ru1-Cl13	161.21
O6-Pd2-N11	172.92	O7-Ru1-C26	165.39
O6-Pd2-N9	93.08	P5-Ru-P4	176.83
N9-Pd2-N11	80.50	O7-Ru-N10	60.94

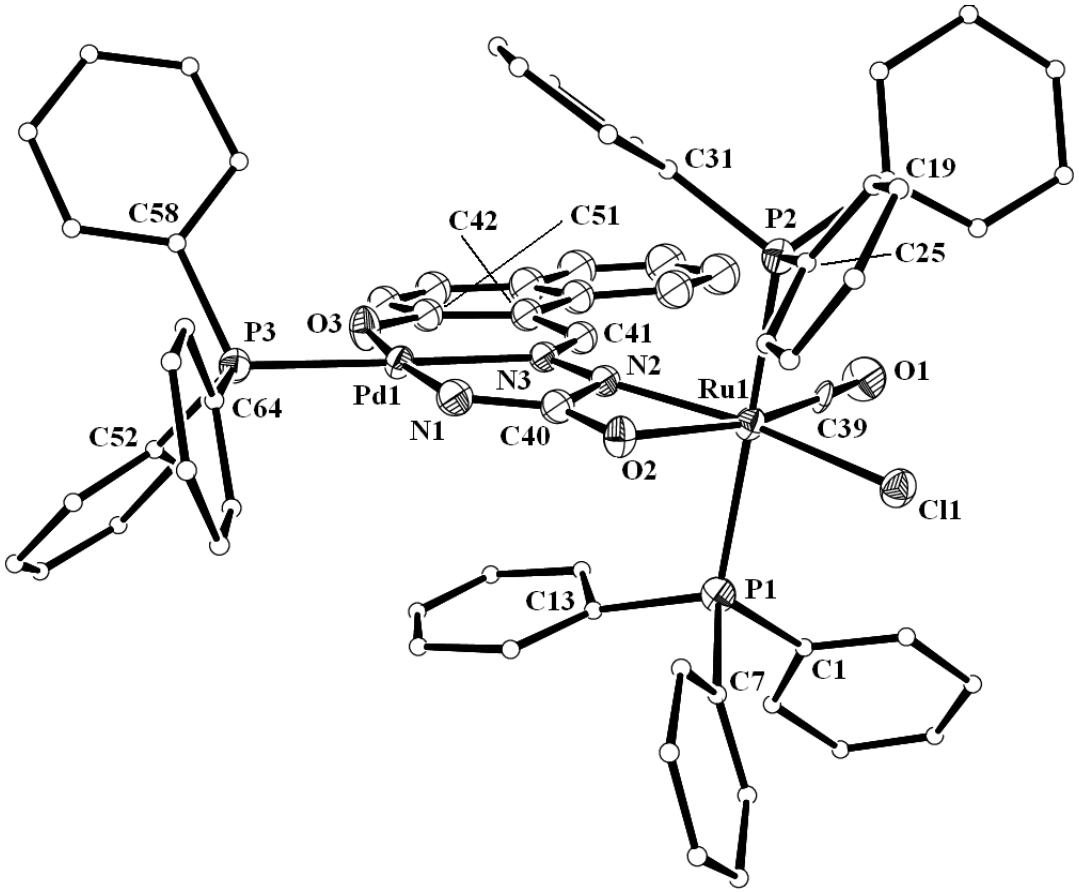

Fig. S3 View of the **2c** complex (one of the four independent molecules is shown here).

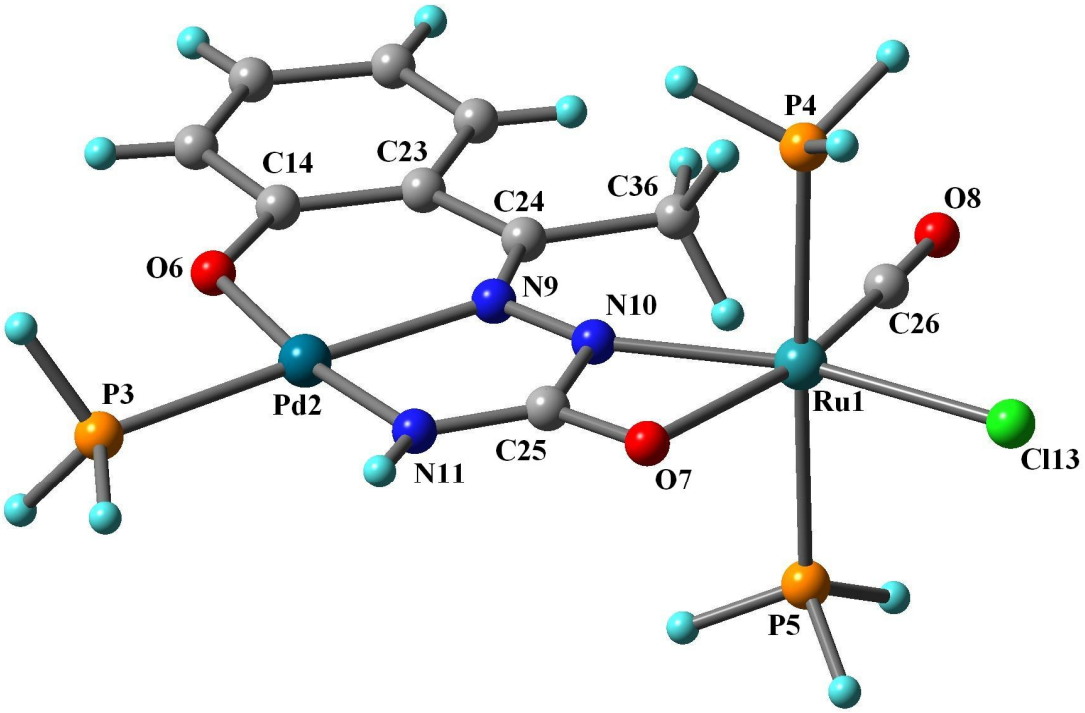

Fig. S4 DFT optimized structure of complex **2b**.

Table S3 Crystallographic data for **1c** and **2c**

	1c.H_2O	**2c**
empirical formula	$C_{30}H_{26}N_3O_3PPd$	$C_{67}H_{53}ClN_3O_3P_3PdRu$
fw	613.89	1283.98
space group	monoclinic, $P2_1/n$	triclinic, $P\bar{1}$
a / Å	9.1000(18)	19.145(4)
b / Å	16.700(3)	26.722(6)
c / Å	18.400(4)	26.880(6)
α / º	90	66.307(6)
β / º	100.39(3)	89.242(7)
γ / º	90	87.514(7)
V / Å ^{3}	2750.4(10)	12581(5)
Z	4	8
λ / Å	0.71073	0.71073
cryst size / mm^3	0.37 x 0.42 x 0.59	0.14× 0.15 × 0.16
T / K	294	293
μ / mm^{-1}	0.769	0.691
R1[a]	0.0731	0.0599
wR2[b]	0.2253	0.1497
GOF[c]	1.08	1.03

[a] $R1 = \Sigma \, | \, |F_o| - |F_c| \, | \, / \Sigma \, |F_o|$.

[b] $wR2 = [\Sigma\{w(F_o^2-F_c^2)^2\}/\Sigma\{w(F_o^2)\}]^{1/2}$.

[c] $GOF = [\Sigma(w(F_o^2-F_c^2)^2)/(M-N)]^{1/2}$, where M is the number of reflections and N is the number of parameters refined.