crystals

MDPI

Supplementary Information

S1. Crystal Structure Solution for Compound 1

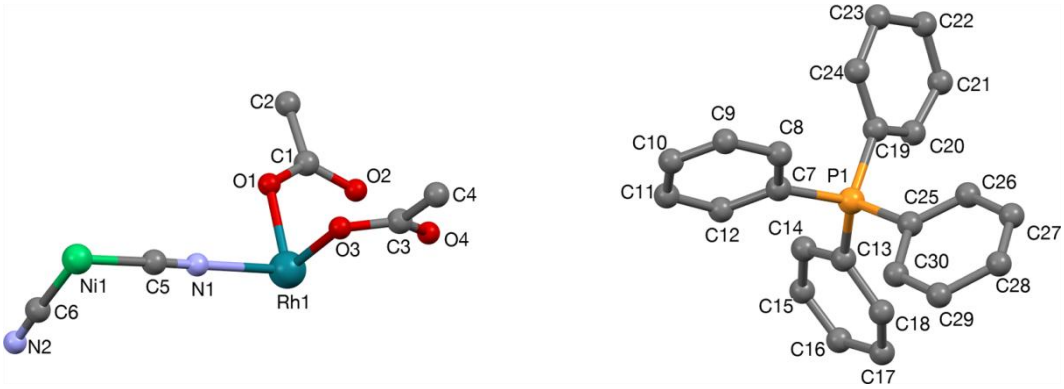

Figure S1. Asymmetric unit of compound **1**. Hydrogen atoms have been omitted for clarity.

Table S1. Sample and crystal data for 1.

CCDC Number	1954793
Chemical formula	$C_{60}H_{52}N_4NiO_8P_2Rh_2$
Formula weight	1283.52 g/mol
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	*P* -1
Unit cell dimensions	*a* = 12.669(2) Å α = 84.405(5)° *b* = 12.8404(8) Å β = 67.765(7)° *c* = 14.640(1) Å γ = 70.795(7)°
Volume	2080.7(3) Å^3
Z	1
Density (calculated)	1.024 g/cm^3
Absorption coefficient	0.694 mm^{-1}
F(000)	652

Table S2. Data collection and structure refinement for 1.

Theta range for data collection	3.27 to 25.00°
Index ranges	–15<=h<=15, –14<=k<=15, –17<=l<=17
Reflections collected	15,447
Independent reflections	7322 [R(int) = 0.0550]
Refinement method	Full-matrix least-squares on F^2
Refinement program	SHELXL-2014/7 (Sheldrick, 2014)
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$
Data/restraints/parameters	7322/0/349
Goodness-of-fit on F^2	1.017
Final R indices	4505 data; I>2σ(I) R_1 = 0.0740, wR_2 = 0.2276

	all data	R_1 = 0.1104, wR_2 = 0.2500
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.1420P)^2]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.815 and –0.752 eÅ^{-3}	
R.M.S. deviation from mean	1.125 eÅ^{-3}	

S2. Crystal Structure Solution for Compound 2

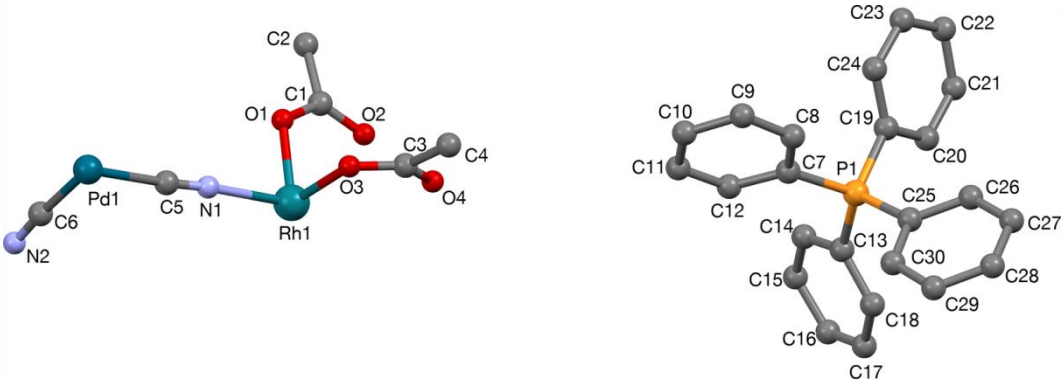

Figure S2. Asymmetric unit of compound **2**. Hydrogen atoms have been omitted for clarity.

Table S3. Sample and crystal data for 2.

CCDC Number	1954794
Chemical formula	$C_{60}H_{52}N_4O_8P_2PdRh_2$
Formula weight	1331.21 g/mol
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	*P* -1
Unit cell dimensions	*a* = 12.636(1) Å α = 85.137(9)°
	b = 13.095(2) Å β = 68.479(9)°
	c = 14.524(2) Å γ = 70.436(9)°
Volume	2104.7(4) Å^3
Z	1
Density (calculated)	1.050 g/cm^3
Absorption coefficient	0.676 mm^{-1}
F(000)	670

Table S4. Data collection and structure refinement for 2.

Theta range for data collection	3.21 to 26.00°
Index ranges	–15 <= h <= 15, –15 <= k <= 16, –17 <= l <= 17
Reflections collected	17,912
Independent reflections	8262 [R(int) = 0.0526]
Refinement method	Full-matrix least-squares on F^2
Refinement program	SHELXL-2014/7 (Sheldrick, 2014)
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$
Data/restraints/parameters	8262/0/343

Goodness-of-fit on F^2	1.005	
Final R indices	4648 data; I>2σ(I)	R_1 = 0.0662, wR_2 = 0.1699
	all data	R_1 = 0.1057, wR_2 = 0.1854
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0846P)^2]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	1.279 and –0.772 eÅ^{-3}	
R.M.S. deviation from mean	0.117 eÅ^{-3}	

S3. Crystal Structure Solution for Compound 3

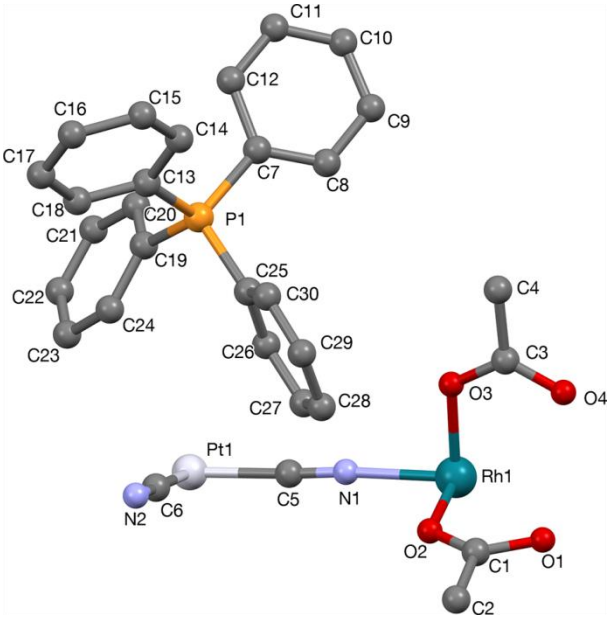

Figure S3. Asymmetric unit of compound **3**. Hydrogen atoms have been omitted for clarity.

Table S5. Sample and crystal data for 3.

CCDC Number	1954794	
Chemical formula	$C_{60}H_{52}N_4O_8P_2PtRh_2$	
Formula weight	1419.90 g/mol	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 13.0959(4) Å	α = 90°
	b = 11.7615(4) Å	β = 101.613(4)°
	c = 25.1321(11) Å	γ = 90°
Volume	3791.8(2) Å^3	
Z	2	
Density (calculated)	1.244 g/cm^3	
Absorption coefficient	2.356 mm^{-1}	
F(000)	1404	

Table S6. Data collection and structure refinement for 3.

Theta range for data collection	3.27 to 26.00°	
Index ranges	–15<=h<=16, –13<=k<=14, –22<=l<=30	
Reflections collected	20,720	
Independent reflections	7438 [R(int) = 0.0435]	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-2014/7 (Sheldrick, 2014)	
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$	
Data/restraints/parameters	7438/0/351	
Goodness-of-fit on F^2	1.008	
Final R indices	4025 data; I>2σ(I)	R_1 = 0.0395, wR_2 = 0.1046
	all data	R_1 = 0.0788, wR_2 = 0.1179
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0430P)^2]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.616 and –0.565 eÅ^{-3}	
R.M.S. deviation from mean	0.110 eÅ^{-3}	

S4. Coordination Environment Parameters

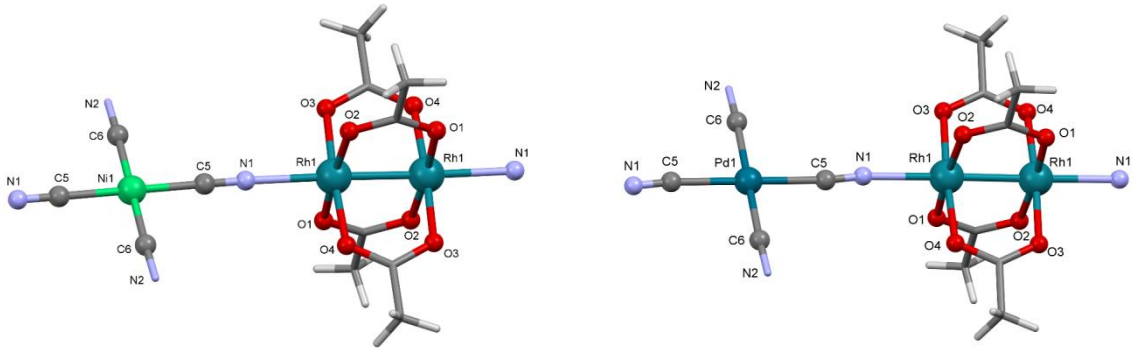

Figure S4. Coordination environments of metal atoms in **1** (left) and **2** (right).

Table S7. Coordination environment distances (Å) in 1 and 2.

	1 (M = Ni)	**2 (M = Pd)**
Rh1-O1	2.035(5)	2.020(4)
Rh1-O2^{i}	2.024(5)	2.040(4)
Rh1-O3	2.019(5)	2.031(4)
Rh1-O4^{i}	2.041(5)	2.052(4)
Rh1-N1	2.275(6)	2.246(5)
Rh1-Rh1^{i}	2.394(1)	2.3968(9)
M1-C5	1.888(8)	2.080(6)
M1-C6	1.850(8)	1.960(8)

(i) = –x + 2, –y + 1, –z

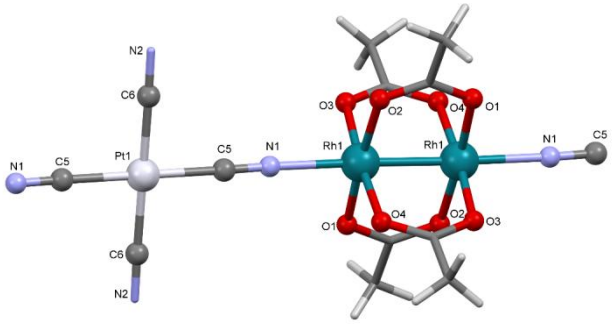

Figure S5. Coordination environments of metal atoms in **3**.

Table S8. Coordination environment distances (Å) in 3.

3	
Rh1-O1^{i}	2.026(4)
Rh1-O2	2.029(4)
Rh1-O3	2.042(4)
Rh1-O4^{i}	2.035(4)
Rh1-N1	2.209(5)
Rh1-Rh1^{i}	2.4007(8)
Pt1-C5	1.998(6)
Pt2-C6	1.979(8)
(i)−*x*+2, −*y*+1, −*z*+1	

Table S9. Coordination environment angles (°) in 1 and 2.

	1 (M = Ni)	**2 (M = Pd)**
O1-Rh1-O2^{i}	175.5(2)	175.26(2)
O1-Rh1-O3	90.7(2)	90.62(2)
O1-Rh1-O4^{i}	88.5(2)	88.8(2)
O1-Rh1-N1	91.6(2)	92.0(2)
O1-Rh1-Rh1^{i}	87.5(1)	87.3(1)
O2^{i}-Rh1-O3	88.2(2)	88.7(2)
O2^{i}-Rh1-O4	92.3(2)	91.6(2)
O2^{i}-Rh1-N1	92.7(2)	92.7(2)
O2^{i}-Rh1-Rh1	88.2(1)	88.0(1)
O3-Rh1-O4^{i}	175.9 (2)	175.7(2)
O3-Rh1-N1	93.4(2)	93.1(2)
O3-Rh1-Rh1^{i}	88.0(1)	87.5(1)
O4^{i}-Rh1-N1	90.7(2)	91.1(2)
O4^{i}-Rh1-Rh1^{i}	88.0(1)	88.2(1)
N1-Rh1-Rh1^{i}	178.4(2)	179.1(1)
C5-M1-C5	180	180
C5-M1-C6	91.2(3)	91.1(2)
C6-M1-C5^{ii}	88.8(3)	88.9(2)
C6-M1-C6^{ii}	180	180
(i) = −x+2, −y + 1, −z; (ii) = −x + 2, −y + 2, −z		

Table S10. Coordination environment angles (°) in 3.

3	
O1^{i}-Rh1-O2	175.6(2)
O1^{i} -Rh1-O3	88.5(2)
O1^{i} -Rh1-O4^{i}	91.4(2)
O1^{i} -Rh1-N1	91.4(2)
O1^{i} -Rh1-Rh1^{i}	88.2(1)
O2-Rh1-O3	90.6(2)
O2-Rh1-O4^{i}	89.2(2)
O2-Rh1-N1	93.1(2)
O2-Rh1-Rh1^{i}	87.4(1)
O3-Rh1-O4^{i}	175.4 (2)
O3-Rh1-N1	94.5(2)
O3-Rh1-Rh1^{i}	87.3(1)
O4-Rh1-N1	90.1(2)
O4^{i}-Rh1-Rh1^{i}	88.2(1)
N1-Rh1-Rh1^{i}	178.2(1)
C5-Pt1-C5^{ii}	180
C5-Pt1-C6	90.1(3)
C6-Pt1-C5^{ii}	89.9(3)
C6-Pt1-C6^{ii}	180
(i)−*x* + 2, −*y* + 1, −*z*+1; (ii) −*x* + 1, −*y* + 1, −*z* + 1	

S5. Supramolecular Interactions

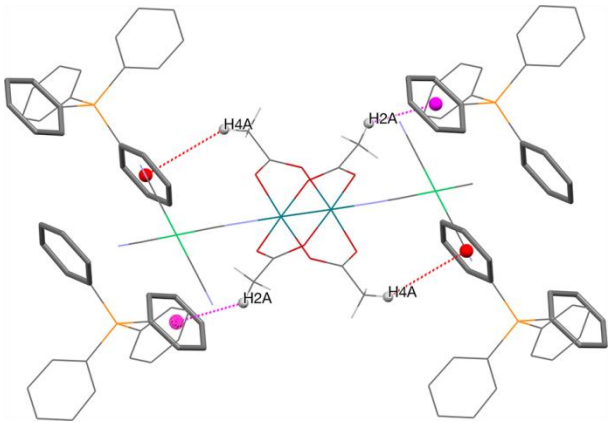

Figure S6. C-H···π interactions in **1** and **2;** the rings involved in these interactions are represented with thicker lines, the C7- C17 ring centroid is depicted in magenta and C13-C18 in red.

Table S11. Supramolecular interactions in **1** and **2**.

	1 (Å)	**2 (Å)**
C2-H2A···centroid C13-C18	3.280	3.208
C4-H4A···centroid C7-C12	3.310	3.385

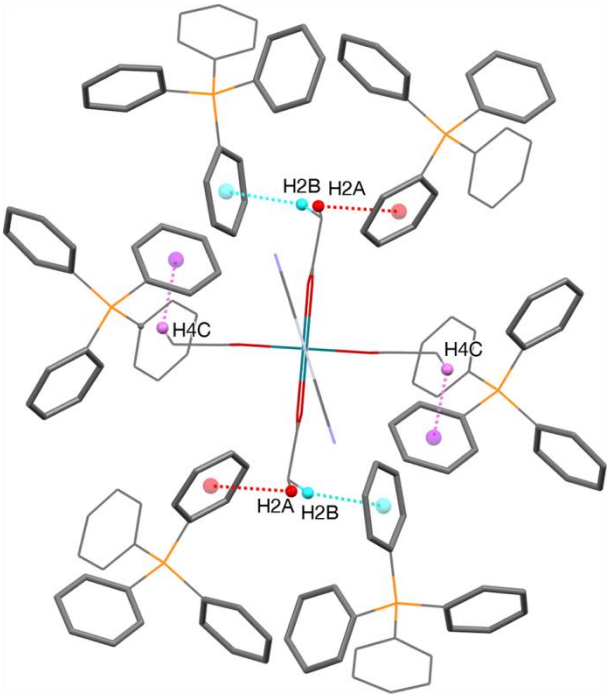

Figure S7. C-H···π interactions in **3;** the rings involved in these interactions are represented with thicker lines, the C7- C17 ring centroid is depicted in red, C13-C18 in cyan, and C25-C30 in pink.

Table S12. Supramolecular interactions in 3.

	3 (Å)
C2-H2A···centroid C7-C12	3.836
C2-H2B···centroid C13-C18	2.796
C4-H4C···centroid C25-C30	3.482