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New *p*-tolylimido rhenium(V) complexes with carboxylate-based ligands: synthesis, structures and their catalytic potential in oxidations with peroxides

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Table S1. Crystal data and structure refinement for compounds **1**–**3.**

	1	**2**	**3**
Empirical formula	$C_{32}H_{28}Cl_2N_4O_2PRe$	$C_{30}H_{25}Cl_2N_3O_2PRe$	$C_{30}H_{25}Br_2\ N_3O_2PRe$
Formula weight [g·mol^{-1}]	788.65	747.60	836.52
Temperature [K]	293.0(2)	293.0(2)	293.0(2)
Wavelength [Å]	0.71073	0.71073	0.71073
Crystal system	Triclinic	triclinic	triclinic
Space group	P-1	P-1	P-1
Unit cell dimensions [Å,°]	a = 10.4006(4)	a = 9.7169(5)	a = 10.3301(10)
	b = 10.5121(4)	b = 14.4692(9)	b = 11.3982(10
	c = 16.4277(6)	c = 22.3218(12)	c = 13.2930(10)
	α = 86.211(3)	α = 97.987(5)	α = 92.707(6)
	β = 81.558(3)	β = 90.759(5)	β = 104.825(8)
	γ = 63.957(4)	γ = 105.589(5)	γ = 104.406(8)
Volume [Å^3]	1596.22(10)	2989.4(3)	1455.2(2)
Z	2	4	2
Density (calculated) [Mg/m^3]	1.641	1.661	1.909
Absorption coefficient [mm^{-1}]	4.059	4.329	7.009
F(000)	776	1464	804
Crystal size [mm]	0.141 x 0.073 x 0.047	0.091 x 0.076 x 0.016	0.153 x 0.102 x 0.085
θ range for data collection [°]	3.55 to 25.05	3.38 to 25.05	3.50 to 25.05
Index ranges	-12 ≤ h ≤ 10	-11 ≤ h ≤ 11	-12 ≤ h ≤ 10
	-12 ≤ k ≤ 11	-17 ≤ k ≤ 17	-13 ≤ k ≤ 13
	-13 ≤ l ≤ 19	-26 ≤ l ≤ 26	-14 ≤ l ≤ 15
Reflections collected	11251	28030	9640
Independent reflections	5657 (R_{int}=0.0219)	10568 [R_{int} =0.0712]	5134 [R_{int} = 0.0396]
Completeness to 2θ=25°	99.7%	99.7%	99.5%
Min. and max. transm.	0.785 and 1.000	0.712 and 1.000	0.341 and 1.000
Data/restraints/parameters	5657 / 0 / 381	10568 / 0 / 705	5134 / 0 / 353
Goodness-of-fit on F^2	0.953	0.988	1.073
Final R indices [I>2σ(I)]	R1 = 0.0222	R1 = 0.0492	R1 = 0.0545
	wR2 = 0.0448	wR2 = 0.1057	wR2 = 0.1541
R indices (all data)	R1 = 0.0306	R1 = 0.0785	R1 = 0.0675
	wR2 = 0.0459	wR2 = 0.1166	wR2 = 0.1590
Largest diff. peak and hole [e Å^{-3}]	0.679 and -0.655	2.038 and -1.144	3.346 and -2.388

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Table S2. Crystal data and structure refinement for compounds **4**–**7**.

	4	**5**	**6**	**7**
Empirical formula	$C_{35}H_{35}Cl_2N_3O_4PRe$	$C_{68}H_{57}Cl_4N_7O_4P_2Re_2$	$C_{67}H_{58}Br_4N_6O_5P_2Re_2$	$C_{68}H_{57}Br_4N_7O_4P_2Re_2$
Formula weight [g·mol^{-1}]	849.73	1612.35	1781.17	1790.19
Temperature [K]	293.0(2)	293.0(2)	293.0(2)	293.0(2)
Wavelength [Å]	0.71073	0.71073	0.71073	0.71073
Crystal system	triclinic	monoclinic	monoclinic	monoclinic
Space group	P-1	$P2_1/c$	$P2_1/c$	$P2_1/c$
Unit cell dimensions [Å,°]	a = 8.2043(4) b = 11.1956(5) c = 18.7854(8) α = 84.911(4) β = 84.904(4) γ = 85.397(4)	a = 11.0285(4) b = 20.8676(7) c = 28.0720(10) β = 90.662(3)	a = 11.1469(3) b = 21.1457(6) c = 28.1190(7) β = 91.056(2)	a = 11.1434(3) b = 21.1471(6) c = 28.1174(6) β = 91.062(2)
Volume [Å^3]	1707.32(13)	6460.0(4)	6626.8(3)	6624.8(3)
Z	2	4	4	4
Density (calculated) [Mg/m^3]	1.653	1.658	1.785	1.795
Absorption coefficient [mm^{-1}]	3.805	4.013	6.163	6.165
F(000)	844	3176	3448	3464
Crystal size [mm]	0.836 x 0.502 x .384	0.325x 0.093 x 0.052	0.115 x 0.079 x 0.019	0.259 x 0.053 x 0.026
θ range for data collection [°]	3.49 to 25.05	3.45 to 25.00	3.40 to 25.05	3.40 τo 25.00
Index ranges	-9 ≤ h ≤ 9 -13 ≤ k ≤ 13 -22 ≤ l ≤ 22	-13 ≤ h ≤ 13 -21 ≤ k ≤ 24 -33 ≤ l ≤ 31	-13 ≤ h ≤ 11 -25 ≤ k ≤ 25 -33 ≤ l ≤ 33	-13<=h<=13 -25<=k<=25 -33<=l<=33
Reflections collected	15987	32105	48598	39796
Independent reflections	6031 [R_{int} = 0.0525]	11339 [R_{nt} = 0.0521]	11720 [R_{int} = 0.1008]	11642 [R_{int} = 0.0633]
Completeness to 2θ=25°	99.7%	99.8%	99.8%	99.8%
Min. and max. transm.	0.392 and 1.000	0.531 and 1.000	0.381 and 1.000	0.293 and 1.000
Data/restraints/parameters	6031 / 0 / 400	11339 / 0 / 784	11720 / 0 / 779	11642 / 0 / 787
Goodness-of-fit on F^2	1.017	1.016	0.998	1.030
Final R indices [I>2σ(I)]	R1 = 0.0320 wR2 = 0.0688	R1 = 0.0380 wR2 = 0.0823	R1 = 0.0575 wR2 = 0.1194	R1 = 0.0428 wR2 = 0.0830
R indices (all data)	R1 = 0.0389 wR2 = 0.0716	R1 = 0.0567 wR2 = 0.0893	R1 = 0.0947 wR2 = 0.1330	R1 = 0.0640 wR2 = 0.0888
Largest diff. peak and hole [e Å^{-3}]	0.986 and -1.031	1.337 and -1.181	1.874 and -1.200	1.320 and -1.568

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Table S3. Short intra- and intermolecular contacts detected in structures **1**–**7**.

D—H•••A	D—H[Å]	H•••A[Å]	D•••A[Å]	D—H•••A[°]
1				
C(2)–H(2)•••Cl(2)	0.93	2.72	3.233(3)	115.6
C(22)–H(22) •••Cl(2) #1	0.93	2.74	3.589(4)	152.3
C(30)–H(30B) •••Cl(2) #2	0.96	2.98	3.770(4)	140.7
C(98)–H(98A)•••O(1)	0.96	2.55	3.428(5)	152.6
2				
C(12)—H(12)•••Cl(2)	0.93	2.64	3.336(9)	132.0
C(22)—H(22)•••O(4)#3	0.93	2.44	3.053(10)	123.2
C(48)—H(48)•••O(3)	0.93	2.38	3.224(10)	151.2
C(53)—H(53)•••O(2)#4	0.93	2.53	3.318(10)	142.2
3				
C(8)—H(8)•••Br(1)	0.93	2.90	3.562(10)	129.1
C(21)—H(21)•••O(2)#5	0.93	2.41	3.056(14)	126.8
4				
N(3)—H(3A)•••O(98)	0.86	2.05	2.751(5)	137.8
O(99)—H(99)•••O(1)	0.82	1.98	2.776(5)	162.8
C(6)—H(6)•••Cl(1)	0.93	2.70	3.335(5)	126.6
C(6)—H(6)•••O(1)	0.93	2.48	3.107(6)	125.2
C(9)—H(9)•••O(1)#3	0.93	2.41	3.056(14)	126.8
5				
N(3)—H(3A)•••O(4)#6	0.86	2.10	2.772(5)	134.0
N(6)—H(6A)•••O(4)	0.86	2.02	2.771(5)	146.0
C(4)—H(4)•••Cl(3)#7	0.93	2.83	3.692(7)	155.2
C(18)—H(18)•••Cl(1)	0.93	2.64	3.315(7)	130.5
C(51)—H(51)•••O(3)	0.93	2.53	3.317(7)	142.0
C(56)—H(56)•••Cl(3)#8	0.93	2.78	3.565(8)	143.2
C(99)—H(99B)•••Cl(4)#2	0.96	2.82	3.637(9)	143.5
6				
N(3)—H(3)•••O(4)	0.86	1.99	2.753(9)	147.6
N(6)—H(6)•••O(2)#9	0.86	2.15	2.802(9)	132.7
C(12)—H(12)•••O(1)	0.93	2.53	3.304(12)	141.1
C(23)—H(23)•••Br(1)#10	0.93	2.87	3.642(12)	141.0
C(37)—H(37)•••Br(1)#7	0.93	2.89	3.753(11)	154.8
C(45)—H(45)•••Br(3)	0.93	2.75	3.382(11)	126.1
7				
N(3)—H(3)•••O(4)#6	0.86	2.12	2.792(6)	134.1
N(6)—H(6)•••O(2)	0.86	2.02	2.781(6)	147.4
C(8)—H(8)•••Br(2)	0.93	2.76	3.392(7)	125.9
C(16)—H(16)•••Br(3)#7	0.93	2.91	3.770(7)	154.0
C(39)—H(39)•••O(3)	0.93	2.54	3.321(8)	142.2
C(56)—H(59)•••Br(3)#10	0.93	2.81	3.622(8)	145.8
C(99)—H(99B)•••Br(2)#2	0.96	2.93	3.666(10)	134.5

Symmetry transformations used to generate equivalent atoms: #1: 1-x,-y,-1-z; #2:-1+x,y,z; #3: x+1,y,z; #4: x,y+1,z; #5: -x,-y+1,-z; #6: x,-y+3/2,z-1/2; #7: x+1,-y+3/2,z-1/2; #8: -x,y+1/2,-z+3/2; #9: x,-y+3/2,z-1/2; #10: -x+1,y+1/2,-z+3/2;

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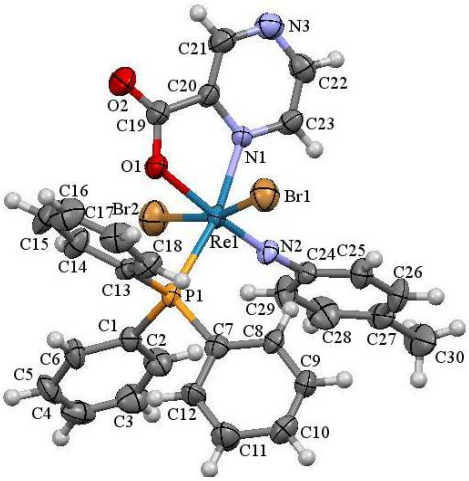

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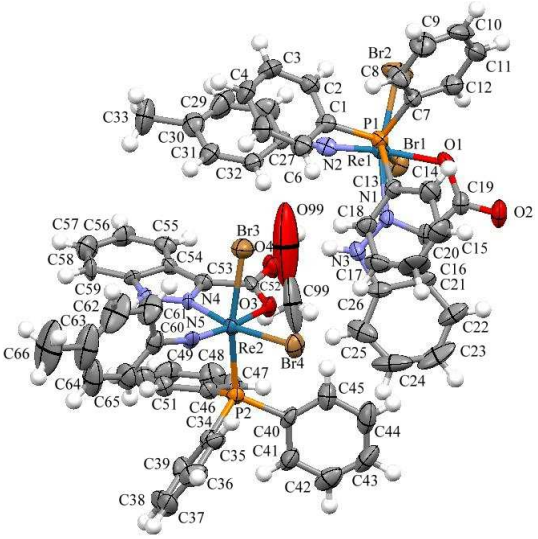

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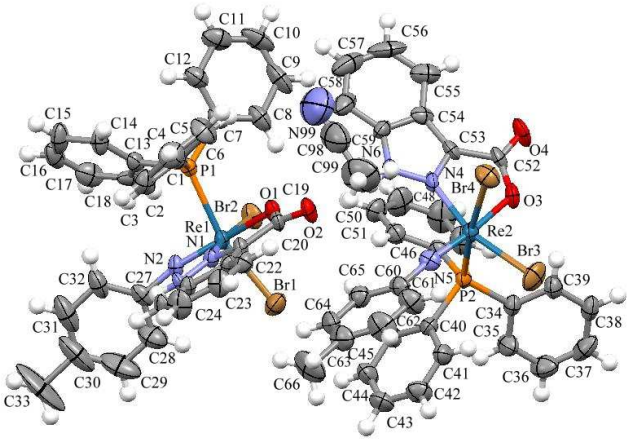

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Figure S1. The asymmetric units of the bromo imido rhenium(V) complexes. Displacement ellipsoids are drawn at 50% probability.